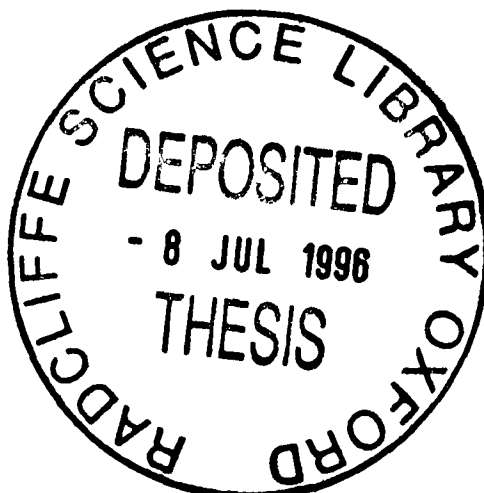


# **THE SPRAY FORMING OF Ni BASED SUPERALLOYS**

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# **ABSTRACT**

## **THE SPRAY FORMING OF Ni BASED SUPERALLOYS**

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The main aim has been to investigate the effect of process parameters on the spray forming of UDIMET 720 and in particular to understand the mechanism of grain size evolution in the deposit using a combination of experimental and computer modelling techniques.

Samples of two spray formed Ni superalloys, MAR-M-002 and UDIMET 720, have been re-heated into the solid/liquid region and the fully solid region just below the solidus temperature for a series of times to try and reproduce the situation of grain growth in a spray formed deposit. Grain growth in the solid/liquid region follows the equation:  $d^3 = d_0^3 + Kt$ , where  $d$  is the grain size,  $d_0$  is the initial grain size,  $K$  is the coarsening rate constant and  $t$  is the time. Coarsening rate constants have been determined for temperatures in the solid/liquid region and they increase with increasing temperature/decreasing solid fraction.

Existing spray forming equipment for Al alloys has been modified to manufacture UDIMET 720 deposits. Process conditions were monitored continually during spray forming, in particular the temperature of the deposit by embedded thermocouples and infra red thermal imaging of the deposit top surface.

Above a deposit temperature of  $\approx 1250^\circ\text{C}$  the microstructure consists of equiaxed fine grains ( $20\mu\text{m}$ - $35\mu\text{m}$ ) and the porosity is low ( $<1\%$ ). Below this deposit temperature the microstructure consists of droplet "splats" and the porosity is higher ( $2\text{-}4\%$ ). The measure grain size increases with increasing deposit temperature and solidification time and agrees reasonably well with the predicted grain size using the above equation.

A commercial finite difference based fluid dynamics software program, FLUENT, has been used to model the 2-dimensional dynamic and thermal behaviour of UDIMET 720 droplets during gas atomisation and spray forming. The effect of atomising gas pressure, spray distance and melt mass flow rate on the equilibrated droplet spray temperature has been examined and shows similar variations with process parameters as the measured maximum deposit temperature. The predicted spray temperature at the substrate is always higher than the measured maximum deposit temperatures under all conditions, and increases with (i) decreasing gas pressure, (ii) decreasing spray distance and (iii) increasing MFR. Mean droplet temperatures and velocities are strongly dependent on droplet size, the mean droplet temperature decreases and mean droplet axial velocities increases with decreasing droplet size.

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The work described is original and no part of this work has been submitted for a degree at any other university. Where the work of others has been drawn upon, it is acknowledged in the text and a list of references is included at the end of the thesis.

I would like to thank Prof. Richard Brook for the provision of laboratory facilities and Dr. Brian Cantor, Dr. PS Grant and Mr DJ Bryant for their combined supervision which provided a good mixture of experienced advice, helpful discussions, encouragement and technical advice.

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Some of the work in this thesis has been published or presented at conferences as follows:

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## **CHAPTER 1 - INTRODUCTION**

The current manufacturing route for non-critical stationary ring components in jet engines involves piercing a cast billet followed by ring rolling. The need for increased engine temperature capabilities and a continuing demand to reduce manufacturing costs in a competitive aerospace market has led several jet engine manufacturers to examine spray forming as a cost effective, alternative manufacturing route for these types of components.

### **1.1 Spray Forming**

In spray forming, a stream of molten metal is atomised by a high pressure inert gas jet to form a spray of droplets. The droplet spray is carried away from the atomisation region by the atomising gas to be deposited on a rotating collector plate placed in the spray plume, to build up a large deposit (Singer 1970). A variety of shapes of spray formed deposits can be produced depending on the geometry of the process, including cylinder rolls, tubes and strip forms. Fig 1.1 is a schematic diagram of the spray forming process indicating two of the geometries that are used to produce ring and billet forms.

Spray forming has several advantages over the conventional cast and wrought manufacturing route including: (i) a refined equiaxed microstructure, due to a high nucleation density on the deposit top surface arising from large amounts of fully solidified droplets and broken dendrites (Mathur *et al* 1989, Bewlay and Cantor 1990); (ii) an extension of solid solubility for some alloy systems because of relatively high solidification rates (Liang and Lavernia 1994, De Sanctis 1991); (iii) the facility for the injection of particulate ceramics into the gas stream during spray forming which co-

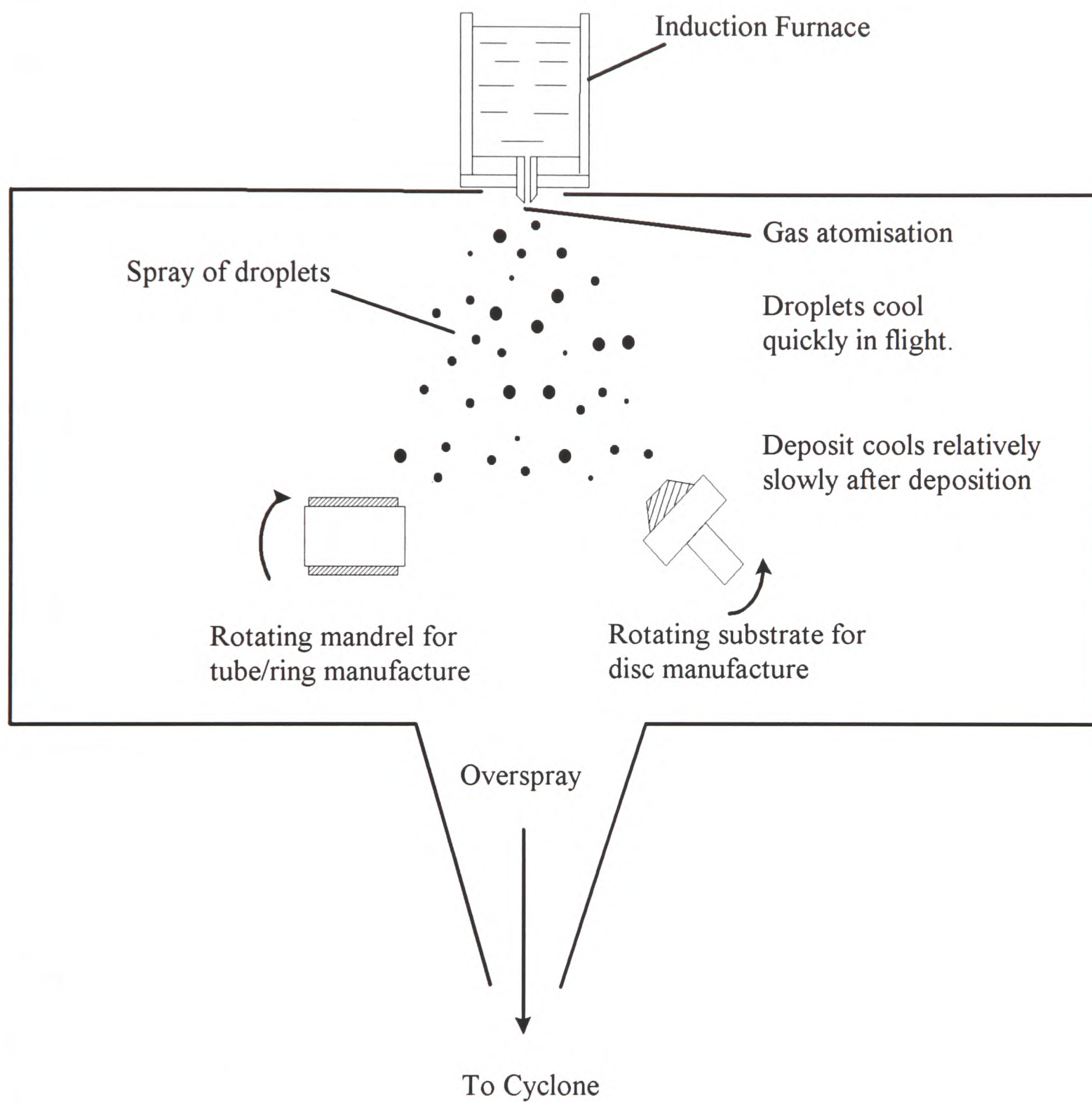


Fig 1.1 Schematic diagram of the spray forming process.

deposit with the metal droplets to produce a homogeneous metal matrix composite (MMC) deposit (White *et al* 1987, Singer and Ozbek 1985); and (iv) the production of near net shape billet, strip and ring forms directly from the melt (Leatham *et al* 1995, Higginbotham 1993).

Microstructural evolution in spray formed deposits is thought to be strongly dependent on the thermal condition of the deposit top surface during spraying. If the deposit is too hot then a large liquid fraction develops on the deposit top surface and this leads to a large grain size and excess amounts of gas porosity caused by whipping up of the semi-solid layer by the atomising gas. If the deposit is too cold, then individual droplets solidify completely before other droplets arrive and a splat structure develops with lots of prior particle boundaries present and high inter particle porosity. The optimum spray forming condition is somewhere between these two extremes i.e. a small liquid fraction on the deposit top surface is necessary to remelt any small solidified droplets and remove prior particle boundaries, but the fraction of liquid must not be too high or entrapped gas porosity will result.

## **1.2 Historical Perspective**

Spray forming had its beginnings in the invention of the spray rolling process by Professor ARE Singer at Swansea University during the late sixties (Singer 1970). Singer developed and patented other spray deposition processes over the following years including centrifugal spray deposition (Singer *et al* 1983), simultaneous shot peening (Singer 1984) and co-spray nozzles for the production of metal matrix composites (Singer 1991, Singer and Ozbek 1985). Subsequent commercial development of the spray forming



process to produce large billet and ring preforms was led by Osprey Metals who licence their own spray forming technology, known as the 'Osprey Process' (Leatham *et al* 1990). Current licensees of the Osprey process are shown in table 1.1 (Leatham 1993). Other workers in this field use their own spray forming technology. For example, a spray forming derivative known as liquid dynamic compaction is used at the University of California at Irvine (Lavernia *et al* 1989).

| Material          | Licences                                                      |
|-------------------|---------------------------------------------------------------|
| Al alloys         | Peak, Sumitomo Light Metals, Pechiney, Alusuisse              |
| Cu alloys         | Olin, Boillat, Wieland                                        |
| Special steels    | Sandvik, SHIFF, Kobe Steel, Chaparral Steel, CRM, Rautaruukki |
| Ni superalloys    | General Electric, Howmet                                      |
| Research purposes | US Navy, Drexel University, Billiton                          |
| Machine builders  | Manessmann Demag, Sumitomo Heavy Industries                   |

Table 1.1 Current licences of the Osprey process. (As of September 1995)

**1.3 This Project**

The application of the spray forming process to produce superalloy billet and ring forms is now quite well established. Although it is known that the deposit top surface thermal condition is important in controlling the deposit microstructure, there is still little detailed understanding of how the deposit microstructure, and in particular the grain size is formed. The objective of this project is to investigate the relationship between spray forming thermal conditions and Ni superalloy deposit microstructure under a range of spray forming conditions using a combination of (i) on line monitoring; (ii) computer

modelling; (iii) deposit re-heating experiments; and (iv) optical, electron probe microanalysis (EPMA) and image analysis.

The thesis is divided into the following chapters: chapter 2 reviews the literature relevant to spray forming; chapter 3 describes the spray forming procedure, the monitoring of thermal conditions and the re-heating experiments on two Ni superalloys; chapter 4 describes the results of the reheating experiments; chapter 5 describes the results of the spray forming experiments; chapter 6 describes the results of the computer modelling of spray forming; chapter 7 compares the experimental and modelling results and discusses the microstructural evolution in spray formed deposits; chapter 8 contains the conclusions and chapter 9 contains suggestions for further work.

## **CHAPTER 2 - LITERATURE SURVEY**

### **2.1 Introduction**

In spray forming, a stream of molten metal is atomised by a high pressure inert gas jet to form a spray of droplets. The droplet spray is carried away from the atomisation region by the atomising gas to be deposited on a rotating collector plate placed in the spray plume, to build up a large deposit (Singer 1970). A variety of shapes of spray formed deposits can be produced depending on the geometry of the process, including cylinder rolls, tubes and strip deposits.

There has been much recent interest from the major jet engine manufacturers in spray forming superalloys for non-rotating parts of jet engines. If the technology could be proven for non-critical components such as casings then other components such as disks could be future candidates for manufacture by spray forming. Spray forming is often seen as a competitor to PM (powder metallurgy) because PM is relatively expensive and has inherent problems such as large amounts of oxide inclusions and high gas content, whereas spray forming retains many of the rapid solidification benefits of PM such as a refined equiaxed microstructure, low segregation and wider possible alloy compositions, without the problems of high oxide and gas content (Higginbotham 1993). However, spray forming is also competing with traditional processing routes where its near net shape capability and improved mechanical properties is a significant economic advantage.

The main physical processes in spray forming will be reviewed in the following sections as well as modelling studies relating to spray forming.

## **2.2 Rapid Solidification**

Rapid solidification processing (RSP) is now a well established route for producing materials with benefits over conventionally processed material. The main benefits of RSP as given by Davies (1990) and Cohen *et al* (1980) are: (i) refinement of microstructure, including grains, subgrains, second phase particles, dendrites and non metallic inclusions; (ii) extension of solid solubility for primary and intermediate solid solutions; and (iii) reduced macrosegregation.

In order to achieve RS, the solid/liquid interface must move rapidly through the solidifying liquid. This can be achieved by high undercooling prior to solidification, or by the rapid extraction of heat from the melt by good contact of a sufficiently thin layer of liquid material with an efficient heat sink (Jones 1982). A cooling rate in excess of  $10^2\text{K/s}$  is the lower limit necessary to achieve RS benefits (Cohen *et al* 1980). Jones (1978) pointed out that heat flow theory predicts an upper limit to the cooling rate attainable for a given solidifying thickness for an assumed effective heat transfer coefficient in a given system, as shown in table 2.1. Spray forming falls within the range necessary to achieve RS benefits for the droplets in flight, however the resulting deposit then solidifies at conventional low cooling rates (Mathur *et al* 1989). Spray formed deposits do exhibit RS benefits though, including refined equiaxed grains and low macrosegregation.

Jones (1982) identified three main commercial processes used to produce RS material: (i) atomisation methods which produce particulate material; (ii) chill methods which produce continuous material; and (iii) weld methods, using electron or laser beam melting. Here, we are mainly concerned with atomisation, as it is not only the method by

which the melt is broken down in the initial stage of spray forming, but is used more widely in powder metallurgy (PM) to produce Ni superalloy powders for subsequent consolidation.

| Range of Cooling Rate ( $\epsilon$ ) |             | Typical Production Processes                                     | Typical Limiting Thickness* (2z) |
|--------------------------------------|-------------|------------------------------------------------------------------|----------------------------------|
| Limits K/s                           | Designation |                                                                  |                                  |
| $10^{-3}$ -10                        | Low         | Standard castings, ingots & strands.                             | 6-0.2m                           |
| 10- $10^3$                           | Med-High    | Thin strip and die casting, normal atomisation.                  | 200-6mm                          |
| $10^3$ - $10^6$                      | High        | Fine powder atomisation, melt extrusion/extraction.              | 6-0.2mm                          |
| $10^6$ - $10^9$ & above              | Ultra-High  | Spray deposition, melt spinning, electron beam or laser glazing. | 200-6 $\mu$ m                    |

\* For a metal slab cooled on both sides by contact with a cold relatively extensive heat sink giving  $\epsilon \approx 10^4/z^2$  (z in mm).

Table 2.1 Some typical ranges of cooling rates in RS and their characteristics. (after Jones 1982).

**2.3 Atomisation**

Atomisation can be defined as the catastrophic breakup of a liquid into fine droplets typically smaller than 150 $\mu$ m (Klar & Schaffer 1984). The use of fluid such as water or inert gas to break up the liquid is known as twin fluid atomisation. Centrifugal force may also be used to break up the liquid. Other atomisation methods include vacuum or soluble gas atomisation, roller atomisation, vibrating electrode atomisation and the melt drop, technique and are described in more detail by Lawley (1977) and Jones (1982). Several authors have reviewed current atomisation technology, some with regard to the specific issues of rapid solidification benefits including Lawley & Doherty (1985), Lawley

(1977 & 1981), Savage & Froes (1984) and Grant (1983). In this review only gas atomisation is considered.

### **2.3.1 Gas Atomisation**

Gas atomisation is achieved by the transfer of kinetic energy from a high velocity atomising fluid into a stream of the liquid metal which causes catastrophic break up of the metal. Small and Bruce (1968) compared water and gas atomisation and concluded that water atomisation increased the oxide content of the powders by an order of magnitude over inert gas atomised powder and produced a higher proportion of irregularly shaped particles. Water atomisation is therefore ignored in this review.

In gas atomisation a high velocity inert gas (usually Ar or N<sub>2</sub>) exits the atomiser either through a continuous annular ring or by a series of discrete jets arranged concentrically around the axis of the metal stream. The jets are equally inclined to the metal stream and intersect it at an impingement point where atomisation occurs. Atomisers can be further subdivided into either open (free fall) or closed (confined) types, as shown in fig 2.1.

Fig 2.1(a) shows that in confined atomisation, the liquid metal is atomised at the tip of the melt delivery nozzle. Therefore, the gas velocity is higher at the impingement point and atomisation is more efficient. Fig 2.1(b) shows that in free fall atomisation, the liquid is allowed to fall under gravity for about 5-20cm before it is impacted by the gas. Confined atomisers are much more sensitive to metal "freeze off" in the delivery nozzle because of the chilling effects of the gas jets and careful nozzle design is needed, especially for high melting point metals (Klar & Schaffer 1984).

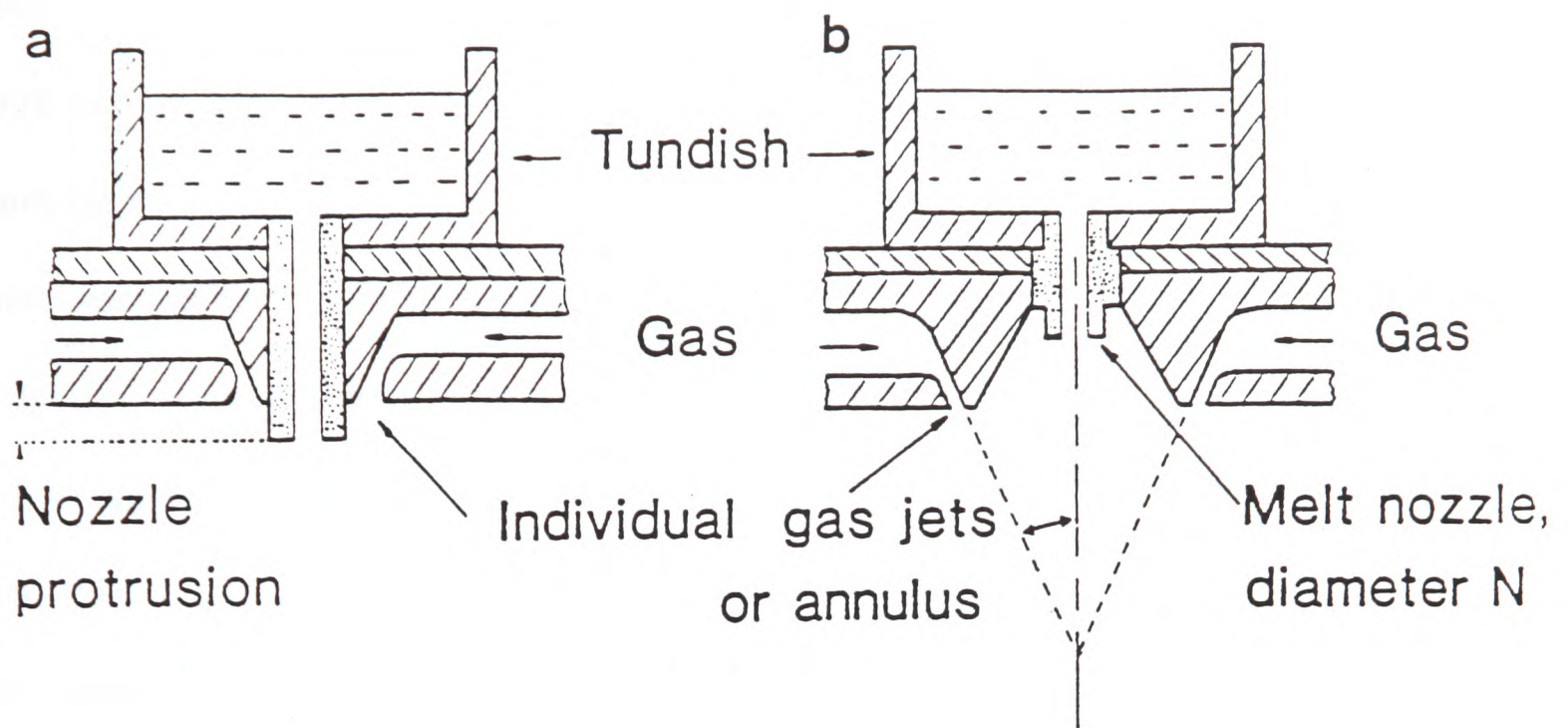


Fig 2.1 Schematic diagram of (a) closed (confined) atomiser, and (b) open (free fall) atomiser (after Grant 1991).

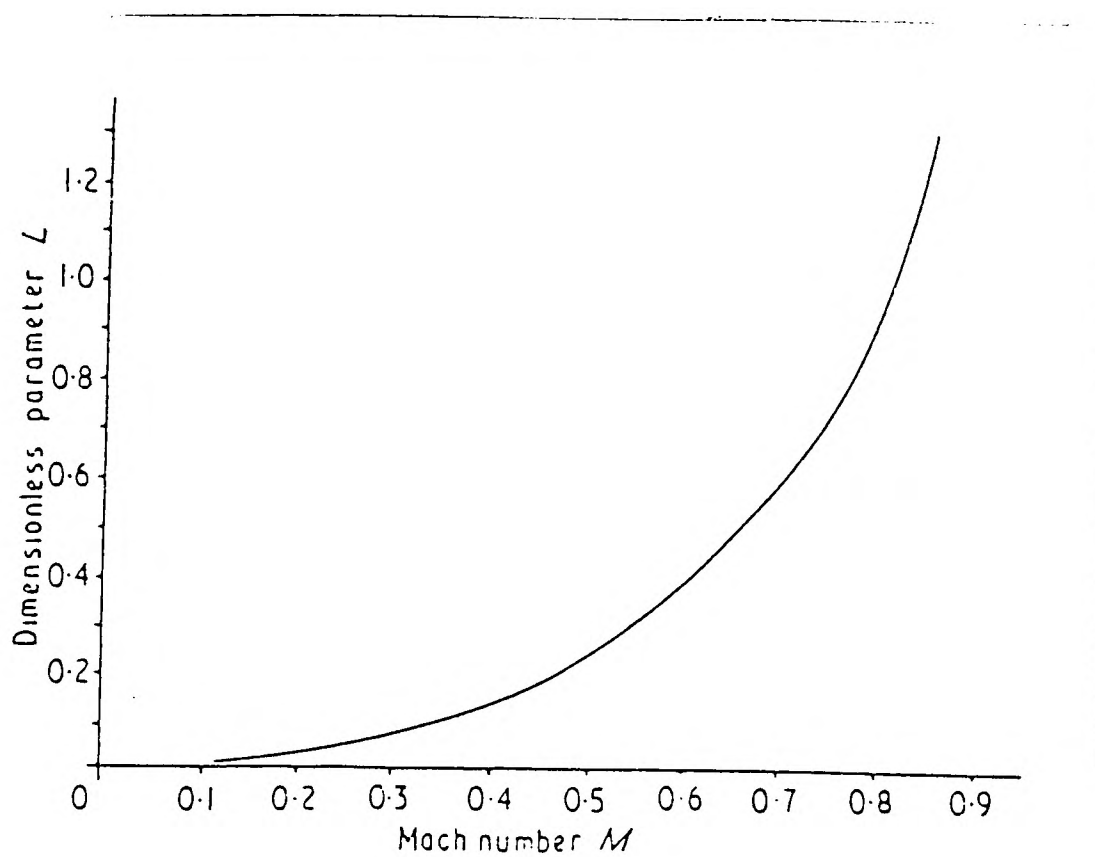


Fig 2.2 Universal curve of  $L$  against  $M$  (after Bradley 1973).

The physical mechanism of gas atomisation is not completely understood, although several models have been presented (Castleman 1931, Fraser *et al* 1962, See & Johnston 1978 and Bradley 1973). Most of the models are based on Castleman's work on the atomisation of liquids in internal combustion engines, in which it was suggested that a fine ligament is torn from the liquid stream and is then, following Rayleigh's (1878) instability criterion, broken into a series of spherical droplets. Bradley (1973) developed a more rigorous mathematical model of the atomisation process and this is the only model that takes into account gas compressibility and liquid metal viscosity. Previous assumptions of gas incompressibility are only valid for gases at low Mach numbers. Bradley modelled the atomisation process as a high speed compressible gas flowing over an initially stationary liquid layer of infinite depth with uniform gas velocity parallel to the liquid surface. Bradley extended Taylor's (1940) analysis of wave growth on a liquid surface and determined the value of the wave number with the fastest growing amplitude (fastest growing wavelength)  $k_{\max}$ . Bradley then assumed (following Castleman 1931) that when this fastest growing wavelength reached a critical amplitude, tears occurred so that the crests formed approximately cylindrical ligaments, which then collapsed by Rayleigh instability to form spherical droplets.

The mean droplet size ( $D$ ) then only depends on the sonic gas velocity ( $U_s$ ), liquid metal surface tension ( $T$ ) and a dimensional parameter  $L$  (which is a function of the Mach number of the gas,  $Ma$ ) as shown below:

$$D=2R=\frac{2.95T}{L\rho_g U_s^2} \quad (2.1)$$

where  $\rho_g$  is the density of the gas. A plot of  $L$  against  $Ma$  is shown in fig 2.2 and is a



universal curve for the atomisation of all liquid metals, although only valid for sonic gas velocities of  $0.1 < Ma < 0.9$ . Lawley and Doherty (1985) compared Bradley's model with the results of See and Johnston (1978) and Rao and Mehrotra (1981), and showed reasonable agreement. In general, not enough reliable data was available to give a detailed assessment of accuracy. Limiting assumptions of Bradley's analysis were that the liquid stream was modelled as a flat sheet, and that no account was given of the angle between the gas jets and metal stream i.e. experimental geometries could not be modelled.

See and Johnston (1978) rigorously investigated the disintegration of liquid Pb and Sn by nitrogen atomisation using high speed photography, and concluded that atomisation was a three stage process:

- (i) Primary Disintegration. The metal stream spreads radially to form a hollow cone, followed by disintegration of this cone by a mechanism of ligament formation resembling the model developed by See *et al* (1973) based upon that of Dombroski & Johns (1963).
- (ii) Secondary Disintegration. Larger droplets formed by primary disintegration of ligaments are not stable and undergo further disintegration. Unal (1987) suggested a critical value for the Weber number ( $W$ ) of  $\approx 13$  below which larger droplets will not undergo secondary break up.
- (iii) Solidification. In this stage the shape of the droplets is determined by the relative times for solidification and spherodisation.

Because of the difficulty in physically modelling the atomisation process, several investigators (Lubanska 1970 and Small & Bruce 1968) adopted an empirical approach to predict the mean droplet sizes of atomised liquids. Correlations are usually only valid for a particular atomiser and provide no insight into the mechanism of droplet formation,

although they are convenient for predicting the mean droplet size as a function of experimental variables.

The most widely used expression is that of Lubanska (1970), who investigated the atomisation of iron and steel using a spray ring atomiser. Wigg (1964) had recognised the importance of the atomising gas kinetic energy in determining the droplet size, and Lubanska modified Wigg's expression empirically for the atomisation of liquid metals to obtain the following expression for the mean droplet size:

$$\frac{d_m}{D} = k \left[ \left( 1 + \frac{M_l}{M_g} \right) \frac{w_l}{w_g W} \right]^{\frac{1}{2}} \quad (2.2)$$

where  $d_m$  is the mass median diameter of powder,  $D$  is the nozzle diameter,  $k$  is a constant in the range 40-50,  $M_l$  and  $M_g$  are the mass flow rates of liquid and solid respectively and  $w_l$  and  $w_g$  are the kinematic viscosities of the liquid and gas respectively. Although other empirical expressions for the mean droplet size have been proposed by other workers (Small & Bruce 1968 and Kim & Marshall 1971), the expression due to Lubanska gives a reasonable fit for a wide variety of metals and atomiser designs (Bewlay 1988). Rao & Mehrotra (1980) improved the fit of Lubanska's empirical correlation by allowing  $k$  and the exponent  $n=0.5$  to vary with design conditions. They used  $k$  in the range 6-10, and  $n$  in the range 0.25-0.3.

From the analytical models by Bradley and others and from empirical studies by Lubanska and others, it is possible to draw conclusions about the general effect of the experimental variables on the mean droplet size:

(i) Higher gas pressures and higher gas velocities at the atomisation point produce finer powder, so that a power law relationship exists between the mass median droplet diameter

and the gas pressure.

(ii) Decreasing the liquid/gas mass flow rate ratio also reduces the mass median droplet diameter.

(iii) The metal properties affect the mass median droplet diameter directly through the liquid surface tension in Bradley's analysis, and through the Weber number, which is dependent on the liquid surface tension in Lubanska's empirical correlation.

The spread of droplet sizes is usually described as a log normal distribution of droplet mass, i.e. a plot of the droplet mass probability density as a function of the logarithm of droplet diameter is normal or Gaussian. Numerous investigators have reported log normal droplet distributions for a wide variety of alloys (Lubanska 1970, See & Johnston 1978 and Rao & Mehrotra 1980). However, Unal (1989A and 1989B) showed strongly bimodal Al and Mg droplet distributions produced by a confined atomiser and postulated that bimodality arose from primary and secondary break up mechanisms. Bewlay (1988) showed bimodal distributions for a Sn-38wt%Pb alloy atomised at low gas/metal flow rate ratios, with a shift to mono-modal distributions when the gas/metal flow rate ratio was increased and atomisation efficiency correspondingly increased. Bewlay and Cantor (1990) also showed that droplet size data on Sn-15wt%Pb and Sn-38-wt%Pb alloys atomised with nitrogen in a confined type atomiser and previous data from other authors (Shinde & Tendolkar 1977 & Date *et al* 1967) could not be represented using a log normal distribution and went on to point out that the existence of bimodality was often masked on cumulative probability plots. However, despite its shortcomings, the log normal approximation remains a popular way of presenting droplet size data for gas atomised alloys.

## **2.4 Single and Dual Phase Flows**

In gas atomisation, the gas flow and droplet dynamic behaviour are intimately linked. Although the single phase gas flow will be affected by the presence of metal droplets formed by atomisation, it is easier to measure single phase flows, though it is now possible to also measure dual phase flows with a reasonable degree of accuracy.

Accurate measurements of gas velocities in the flow field are generally obtained either using intrusive static Pitot tubes or by LDA (laser Doppler anemometry). Pitot tubes measure the pressure difference developed between an orifice placed normal to the flow and an orifice placed parallel to the flow, which can then be related to the gas velocity. However Pitot tubes are intrusive, and may disturb the flow in the measurement region. In LDA and an associated technique, PDA (phase Doppler anemometry), a laser beam is split into two beams of equal intensity, which are then focused to cross and form an interference pattern within a measurement volume in the flow field. When a small particle entrained in the flow passes through the measurement volume, the interference pattern, or standing wave, is scattered and shifted (doppler shift) by an amount related to the particle velocity. The scattered light frequency is measured by a photo multiplier in the receiving optics. Provided the particles are small enough that momentum exchange with the gas can be neglected, e.g alumina particles or talcum powder (Durst *et al* 1979), the measured particle velocity is then equal to the gas velocity. Droplet sizes can be determined in PDA by relating the differential phase shift of two corresponding identical signals measured by two photomultiplier detectors, placed at different locations (Bachalo 1980 and Bauckhage *et al* 1987). The advantages of LDA/PDA are rapid response time and non-intrusive measurements.

### **2.4.1 Single Phase Flows**

In early experiments to characterise the flow pattern of a free sonic jet, Anderson & Johns (1955) measured subsonic gas velocities using static Pitot tubes. They concluded that the decrease in axial gas velocity with axial distance was exponential, and the variation in axial gas velocity with radial distance was approximately normal, with jet spreading rates of 4-6°.

Bewlay & Cantor (1989) measured the N<sub>2</sub> gas flow field from a confined annular atomiser in an enclosed chamber using a variety of different techniques including Ar ion LDA, He/Ne LDA, Pitot tubes and smoke marker visualisation experiments, and compared the results with computer modelling calculations. They concluded that the axial decay in axial gas velocity was once again exponential with Gaussian radial symmetry and a gas plume divergence semi-angle of  $\approx 15^\circ$ . Turbulent intensities of  $\approx 30\%$  were measured near the spray axis and  $\approx 65\%$  near the spray periphery.

### **2.4.2 Dual Phase Flows**

Popper *et al* (1974) studied the two phase flow of oil droplets injected upstream into the flow field of a free turbulent air jet. They measured the velocities of the oil droplets using LDA having previously determined the velocity profile of the single phase jet. When oil drops were injected upstream: (i) the axial decay in axial gas velocity was once again exponential with Gaussian radial symmetry, however the droplets had an initial velocity lower than the gas velocity and then decayed at a lower rate; and (ii) the two phase jet was narrower than the single phase jet.

Bewlay & Cantor (1990) measured Sn-15wt%Pb and Sn-38-wt%Pb droplets

velocities produced by a N<sub>2</sub> confined atomiser using fast exposure photography and LDA, and found that the fast flowing gas accelerated the droplets until they reached the gas velocity at some distance from the atomiser. At this point the gas/droplet slip velocity was zero, and thereafter the gas was flowing slower than the droplets, and the droplets experienced a retarding drag force. Bewlay & Cantor suggested that the gravitational acceleration and retarding forces on the droplet were almost balanced in this region as the droplet velocity decreased only slightly. Measured mean axial droplet velocities were in the range 3-32m/s for corresponding mean axial gas velocities of 11-84m/s. Bauckhage *et al* (1987) measured gas atomised steel droplet velocities which agreed qualitatively with the results of Bewlay & Cantor (1990), i.e. droplets were accelerated until they reached the gas velocity and were then subjected to decelerating forces.

Bewlay & Cantor (1989) measured the streak lengths of droplets on short exposure film and related them to droplet velocities. These were always lower than corresponding LDA measurements because of: (i) difficulties in establishing the beginning and end of the streaks; (ii) the larger droplets were detected preferentially as they reflected more light; and (iii) droplet velocities away from the spray axis were preferentially measured as they were easier to identify, giving an overall error estimated to be  $\approx 25\%$ . Mathur (1986) pointed out the following problems with this technique: (i) the plane of droplet trajectory is not necessarily parallel to the plane of the film; (ii) the droplet size cannot be determined from the streak size; and (iii) the droplet velocity is not constant over the droplet streak. However, Mathur *et al* (1989) used fast exposure photography to measure Ni-20wt%Cr droplet velocities from an Osprey gas atomiser. They presented no detailed results of this technique and roughly estimated the mean measured droplet velocity to be

$\approx 100\text{m/s}$ . Although estimates of mean droplet velocity and mean droplet sizes are available, the mean droplet velocity ( $v_m$ ) does not always correspond to the mean droplet size ( $d_m$ ), because they do not always have the same distribution functions.

## **2.5 Modelling of Gas and Droplet Velocities**

A large number of computer models have been developed to predict the behaviour of the gas flow, droplets in flight and the effect of the droplet dynamic history on droplet solidification behaviour.

Numerical models of the single phase gas flow solve the appropriate steady state conservation equations for mass, momentum and energy for mean gas velocities ( $V_g$ ), pressure (P) and enthalpy (H), as well as similar conservation equations for the kinetic energy of turbulence (k) and dissipation rate ( $\epsilon$ ). All conservation equations have the generalised form:

$$\text{div}(\rho_g V_g \Phi - \Gamma \Delta \Phi) = S_\Phi \quad (2.3)$$

where  $\rho_g$  and  $V_g$  are the gas density and velocity respectively,  $\Phi$  is the dependent variable,  $\Gamma$  is the effective viscosity or thermal diffusivity and  $S_\Phi$  is a source term for  $\Phi$  per unit volume.

In the modelling of two phase flows the atomisation process is usually ignored and droplets are injected into the gas flow field at an appropriate point and their subsequent behaviour modelled. Most of the models simplify the dynamics of gas/droplet interaction, and concentrate on the droplet cooling and solidification. The dynamic behaviour of droplets in flight based on the previous LDA experimental evidence is: (i) droplets are initially subject to an accelerating force due to the gas/droplet slip velocity; (ii) droplets

increase their velocity until they reach the gas velocity and are then subject to a decelerating drag force.

Two different approaches to the modelling of two phase flows have been used. The first is a 1-d formulation which uses an assumed variation of the gas velocity profile that is unaffected by the presence of droplets and all droplets experience the same axial gas velocity profile along the spray axis. The expression for the drag force acting on a sphere in a 1-d continuum flow is given by Ranger & Nicholls (1969), other assumptions usually made are: (i) droplets are spherical; (ii) droplets do not interact with each other; and (iii) effects of turbulence in the gas flow are ignored. Models of this type have been formulated by Mathur *et al* (1989), Grant (1991) and Lavernia *et al* (1988) and agree reasonably well with experimental results described previously. Fig 2.3 shows the gas and Al-4wt%Cu droplet axial velocity profiles as a function of axial distance from the model by Grant. Small droplets are accelerated and decelerated faster than large droplets, whose higher inertia make them relatively insensitive to gradual decreases in the gas velocity.

The other approach is more computationally demanding and couples 2-d or 3-d calculations of the gas flow in the chamber using a Eulerian frame of reference with Lagrangian tracking of individual droplets e.g. FLUENT<sup>1</sup> or PHOENICS<sup>2</sup>. Two way coupling of momentum, mass and energy is achieved by alternately solving the droplet trajectories and the gas flow equations at each cell in the chamber until the solutions of both sets of equation no longer change.

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<sup>1</sup> - FLUENT Inc, Centerra Resource Park,  
10 Cavendish Court Lebanon, NH 03766 U.S.A.

<sup>2</sup> - CHAM Ltd. London SW19 5AU, U.K



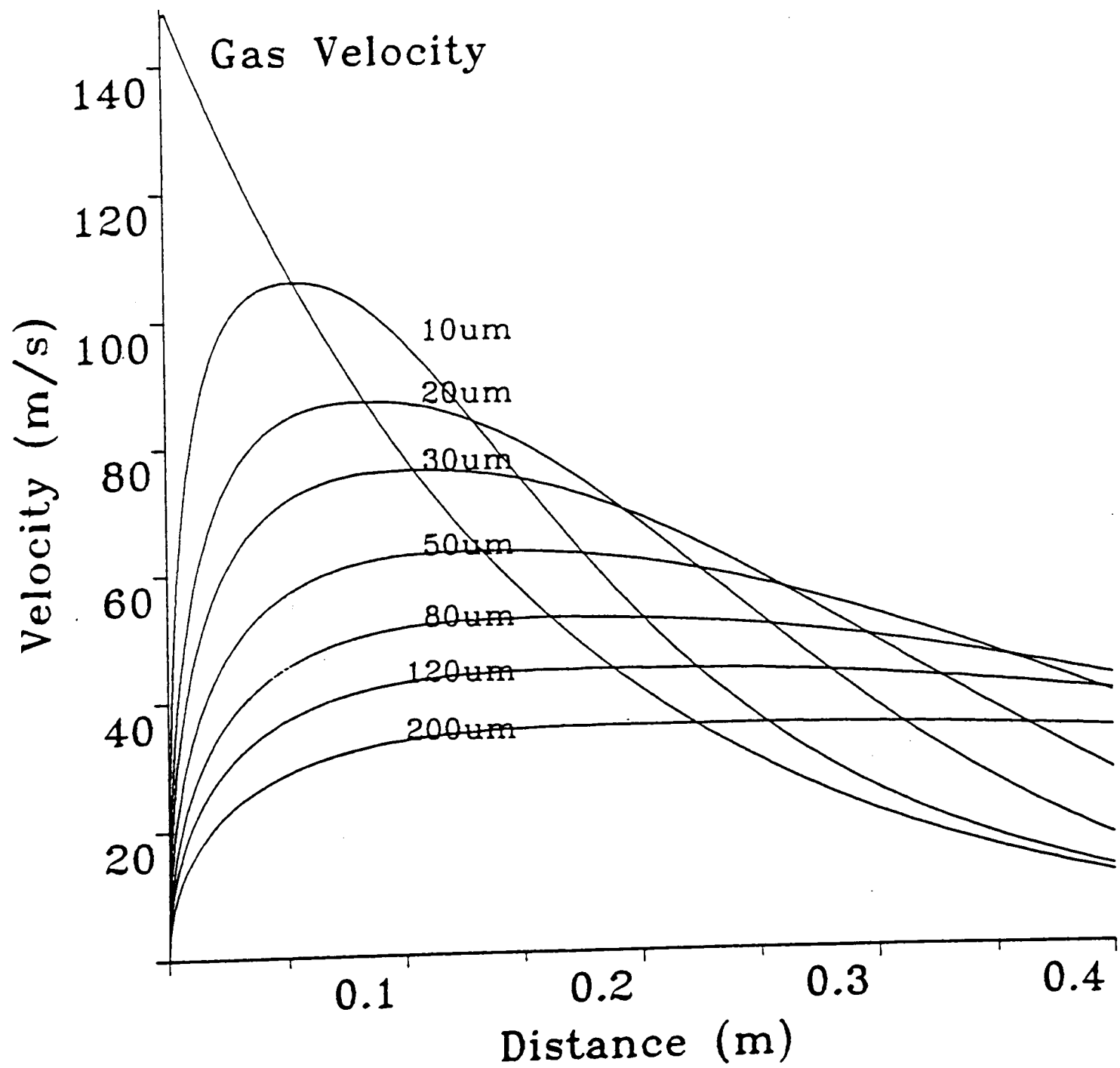


Fig 2.3 Gas and Al-4wt%Cu droplet axial velocity profiles as a function of axial distance for an inlet gas velocity of  $150\text{ms}^{-1}$  (after Grant 1991).

The accuracy of all the models of two phase flow are limited by the difficulties in simulating the atomisation process and assume droplets appear in the gas flow near the real atomisation region. However, models of gas and droplet behaviour are a useful comparative tool and provide insight into the effect of process parameters on the spray forming process.

## **2.6 Cooling and Solidification of Droplets**

Levi and Mehrabian (1980, 1982A and 1982B) conducted a comprehensive study into the heat flow during the solidification of undercooled metal droplets. They modelled the solidification of undercooled spherical aluminium droplets using both a Newtonian (uniform droplet temperature) and non-Newtonian (non-uniform) enthalpy model. The assumption of Newtonian conditions was generally valid for Biot numbers ( $Bi$ )  $< 0.1$  but became invalid at large undercoolings. Droplet thermal histories were conveniently represented on a dimensionless plot of enthalpy versus temperature, as shown in fig 2.4. Fig 2.4 shows three possible solidification paths: Path 1 show isothermal solidification with no droplet undercooling. Path 2 shows isenthalpic or adiabatic solidification, if a system is undercooled below path 2 then it is known as hypercooling. The undercooling is large enough such that on recalescence, the droplet reheats to a temperature below the system solidus, i.e. an undercooling of  $\Delta T > L/C$  is necessary for hypercooling ( $L$  is the latent heat and  $C$  is the specific heat of the system). Path 3 is the general case. The system is undercooled to a temperature  $\theta_N$ , then undergoes recalescence to some temperature close to the melting point, before completing solidification at a rate determined by the external rate of heat extraction.

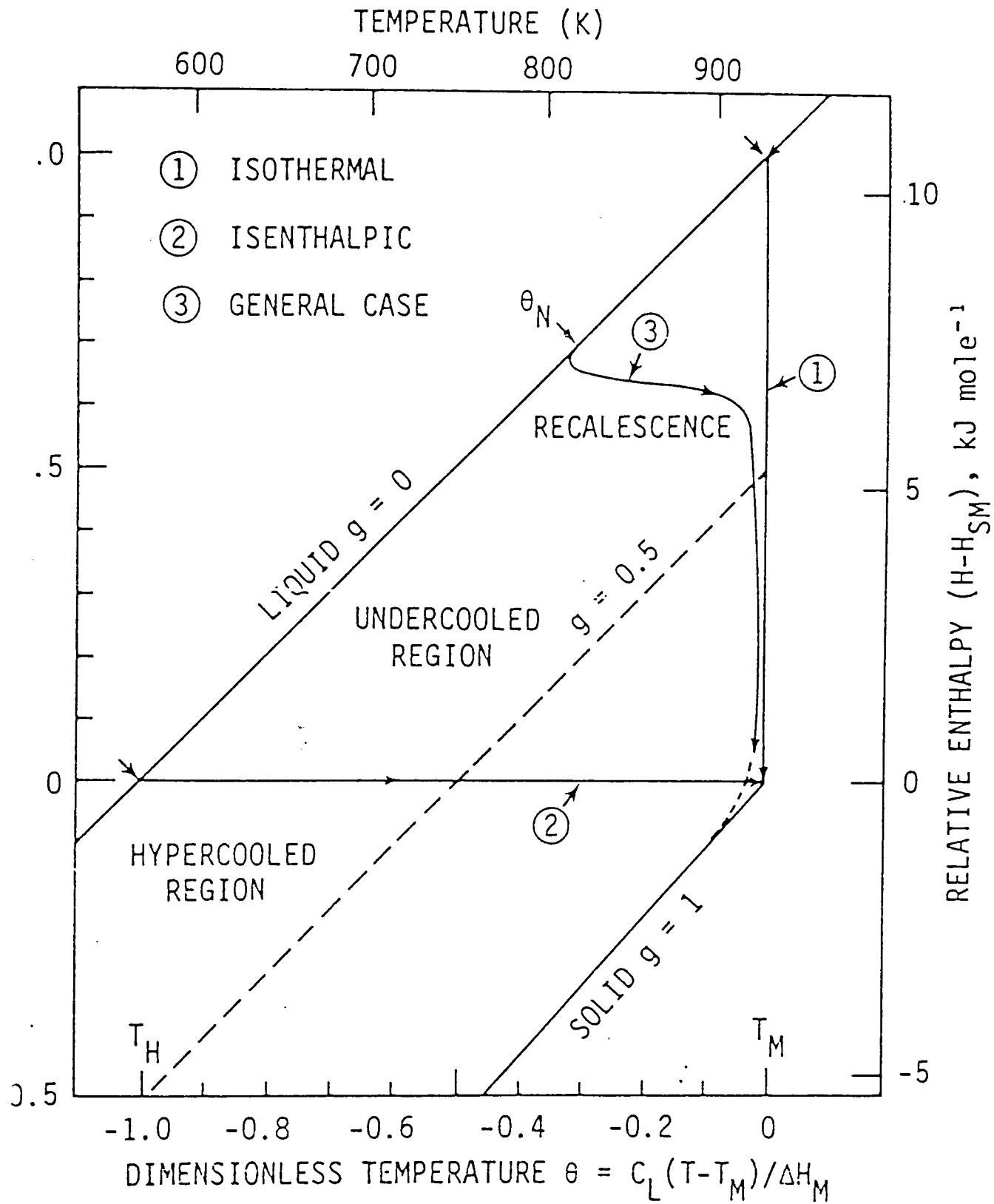


Fig 2.4 Enthalpy-temperature diagram showing possible solidification paths.  $\theta_N$  is the dimensionless temperature at the moment of nucleation (after Levi and Mehrabian (1982B)).

Levi & Mehrabian (1982B) identified two regimes of droplet thermal history associated with the general solidification case: (i) recalescence or rapid solidification, where the supercooled droplet absorbed most of the latent heat of fusion, heat loss to the surroundings was largely irrelevant, and the solid/liquid interface moved very quickly which gave rapid solidification conditions; and (ii) a regime of slower growth after the maximum recalescence temperature was reached, where solidification was largely determined by the imposed external heat transfer conditions, and solidification was approximately isothermal. The thermal history of the droplets reflected the competition between these two regimes. Levi & Mehrabian (1982A) also showed results of a TEM study into the solidification behaviour of electro-hydrodynamic atomised sub-micron pure aluminium droplets which appeared to agree qualitatively with the above results.

Several numerical models which simplified the rapid solidification effects, but determined the solid fraction of representative droplets as a function of axial distance from atomisation point have been developed by Lavernia *et al* (1988), Mathur *et al* (1989) and Grant (1991). These models were more instructive for spray forming as they gave an indication of the spray solid fraction arriving at the deposit top surface during spray forming. Convective cooling was assumed to be dominant over radiative cooling (Levi & Mehrabian 1982B), and an empirical relation for the surface heat transfer coefficient ( $h$ ) given by the Ranz-Marshall relation (1952A and 1952B) was also usually assumed. Five possible solidification regimes were identified in these models as shown schematically in fig 2.5: (i) cooling and nucleation; (ii) recalescence; (iii) segregated solidification; (iv) eutectic temperature arrest; and (v) cooling in the solid phase.

Typical results from the model by Grant showing droplet temperatures and solid

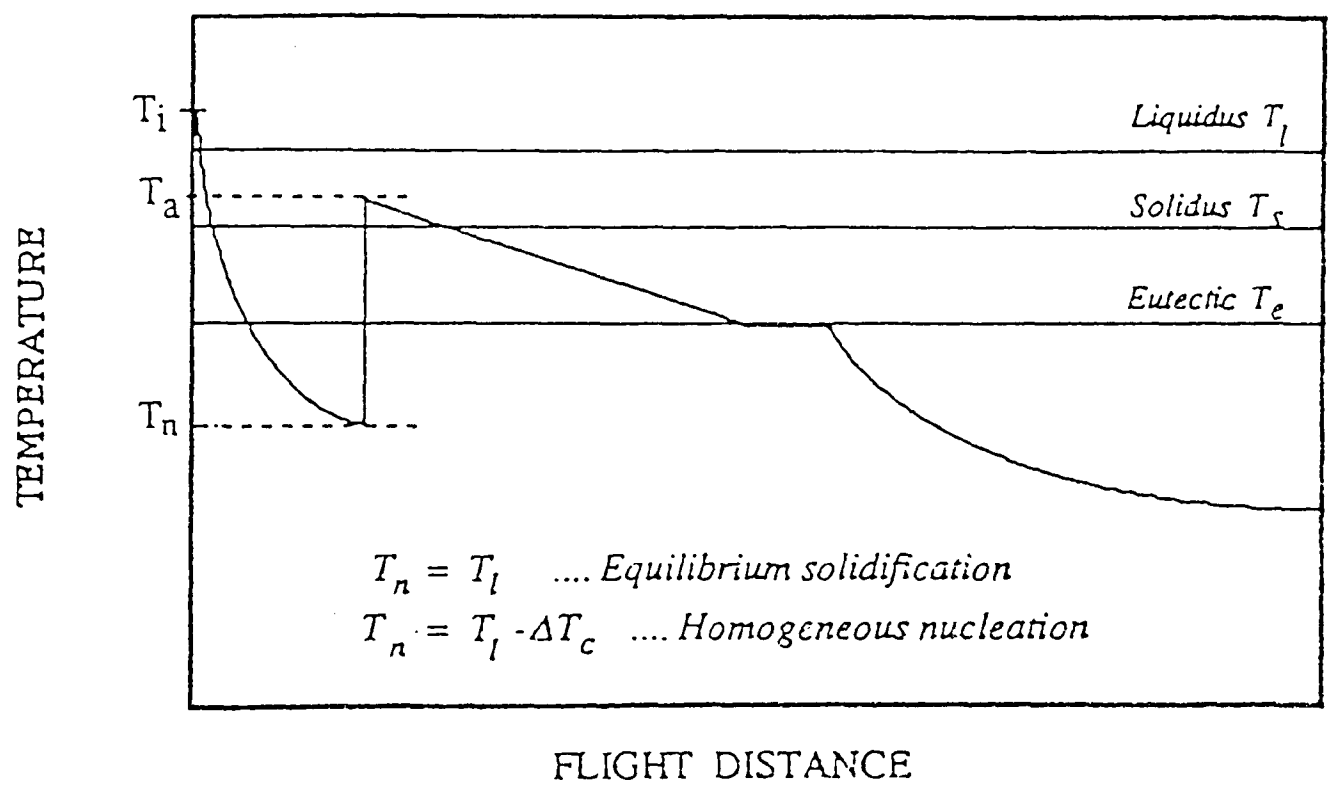


Fig 2.5 Schematic temperature profile of a droplet in flight for an alloy composition with a mushy region (after Mathur *et al* 1989).

fractions as a function of axial distance are shown in figs 2.6 and 2.7 respectively, which show that small (10-30 $\mu\text{m}$ ) droplets are fully solid at a substrate 0.3m from the atomisation point while large (>200 $\mu\text{m}$ ) are fully liquid at the same point. These modelling studies concluded droplet cooling and solidification was: (i) very dependent on droplet size; (ii) relatively independent of the assumptions made about droplet solidification; and (iii) strongly dependent on the assumptions made about gas and droplet dynamics and the mechanisms of heat transfer such as the Ranz-Marshall empirical correlation (Grant *et al* 1993A and 1993B).

## **2.7 Droplet Microstructure**

It is possible to determine the cooling rate of atomised alloy droplets from their microstructure by measuring the dendrite arm spacing or cell size. The relationship between cooling rate ( $\omega$ ) and secondary dendrite arm spacing (DAS) is expressed as:

$$\lambda = a\omega^{-n} \quad (2.4)$$

where  $\lambda$  is the DAS,  $a$  is a constant and  $n$  is an experimentally determined constant, (Mehrabian 1977). This relationship was validated for a range of cooling rates and alloys by Predecki *et al* (1965) and Bower *et al* (1966), although it was difficult to measure cooling rates experimentally for  $\omega > 10^3 \text{K/s}$ . Observations of dendrite coarsening during isothermal holding in the freezing range of Al-Cu alloys by Kattamis *et al* (1967) showed that the DAS ( $\lambda$ ) was proportional to the holding time in this region:

$$\lambda = bt_0^n \quad (2.5)$$

for continuous cooling through the cooling range ( $\Delta T$ ), the holding time  $t_0 = \Delta T / \omega$ , and  $b = a\Delta T^n$  is a constant. Jones (1982) suggested that  $n$  should be equal to 1/3 as the situation

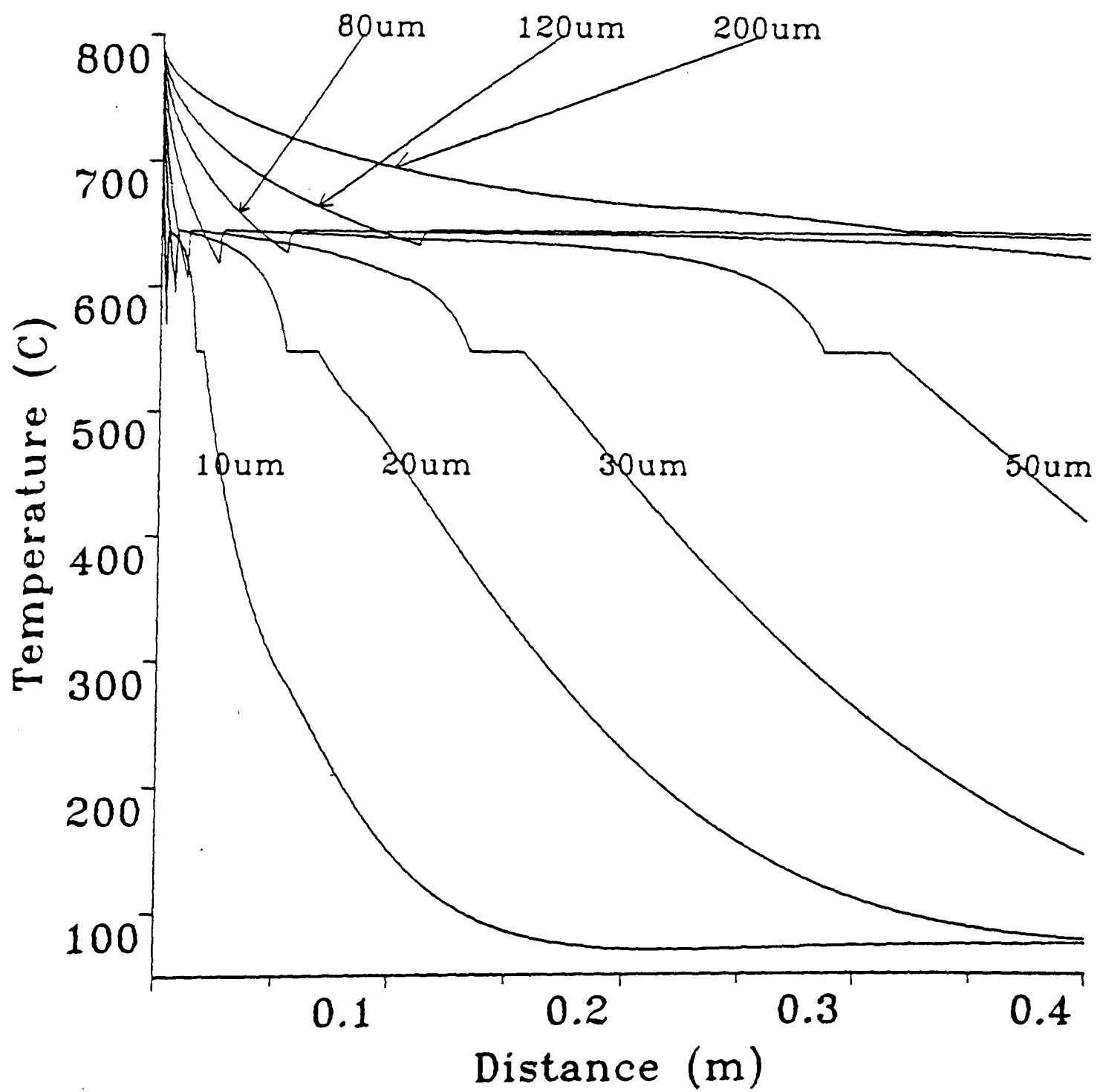


Fig 2.6 Al-4wt%Cu droplet temperatures as a function of axial distance for an initial axial gas velocity of  $150\text{ms}^{-1}$  and melt superheat of  $150^\circ\text{C}$  (after Grant 1991).

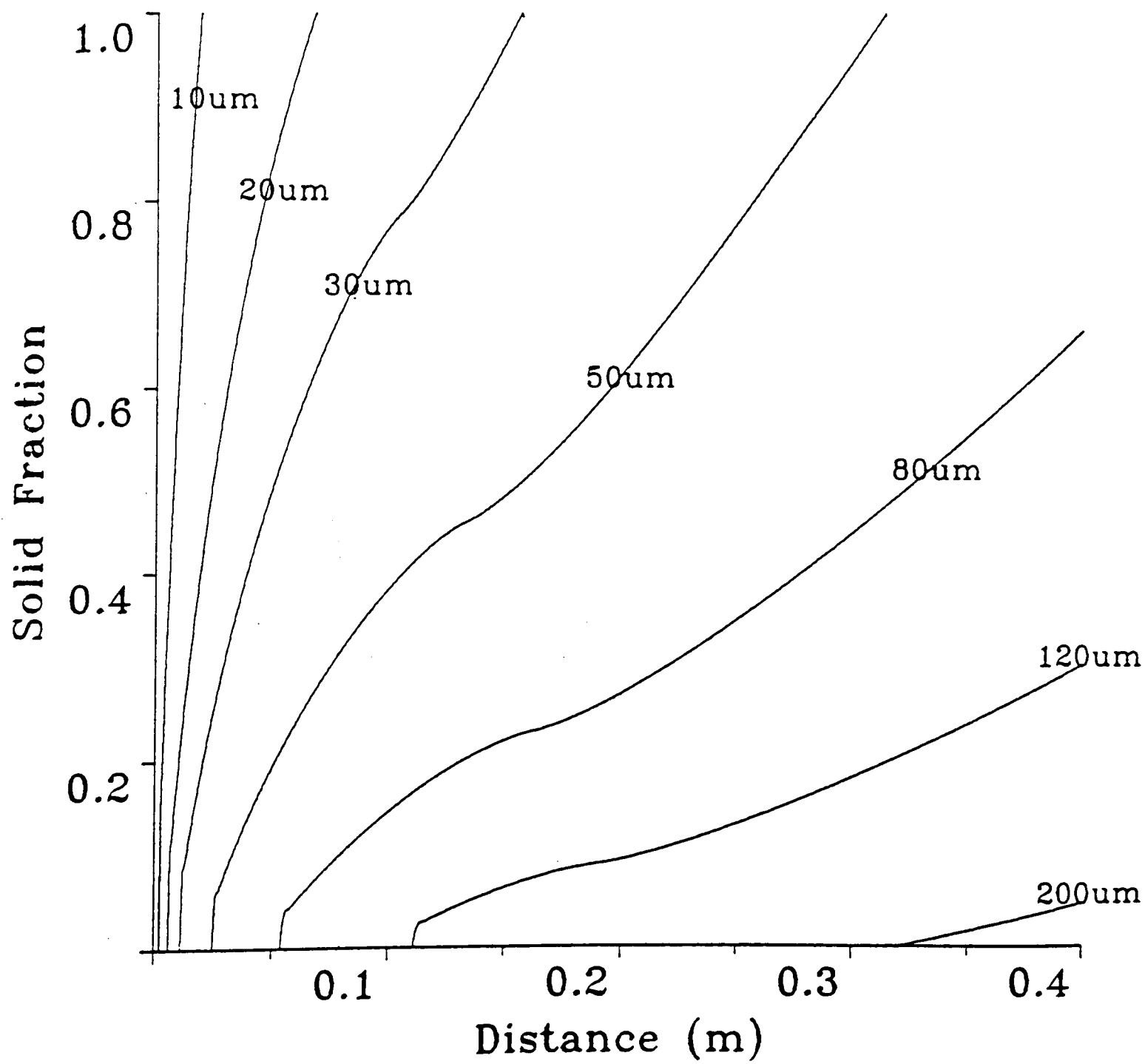


Fig 2.7 Al-4wt%Cu droplet solid fractions as a function of axial distance for an initial axial gas velocity of  $150\text{ms}^{-1}$  and melt superheat of  $150^{\circ}\text{C}$  (after Grant 1991).



was similar to Ostwald ripening. In practice deviations from  $n=1/3$  were measured and  $n$  typically varied from 0.25-0.4. Although equation (2.4) should be used with some care when extrapolating to high cooling rates, it can be used for gas atomised droplets to estimate the cooling rate from droplet DAS measurements.

## **2.8 Metal Deposition**

During spray forming fully solid, semi-solid or fully liquid droplets impact the deposit top surface. Mathur *et al* (1989) used two parameters to describe the metal deposition process: (i) the metal deposition rate ( $D$ ) and (ii) the deposit solidification rate ( $S$ ). However, as pointed out by Bewlay & Cantor (1991), the deposition rate takes no account of the actual enthalpy content of the droplets arriving at the deposit surface, and they described metal deposition using the enthalpy arrival rate ( $H_a$ ) due to the spray of droplets and the enthalpy removal rate ( $H_r$ ) due to conduction into the substrate, conduction through the solidifying deposit and convection from the gas flow across the deposit top surface. Two extreme conditions of spray forming were identified:

- (i)  $H_a > H_r$ . Droplets did not completely solidify before the next droplet impacted and a solid/liquid region developed on the deposit top surface. The solid/liquid region ensured that pores were continually fed with liquid and acted to reduce porosity in the deposit.
- (ii)  $H_r > H_a$ . Each droplet splatted on impact and was able to solidify before the next droplet arrived, and the deposit contained interstitial porosity due to insufficient liquid feeding. The deposit consisted essentially of an agglomeration of splats with a lot of prior droplet boundaries.

Attempts to relate the deposit thermal history to the resulting microstructure were

made by Mathur *et al* (1989), Bewlay & Cantor (1991), Grant (1991), Gutierrez-Miravete *et al* (1988) and Liang *et al* (1992), using a combination of measurements from embedded thermocouples in the deposit and numerical modelling. Numerical models of deposit heat flow are reviewed in the next section

### **2.8.1 Deposit Heat Flow**

Mathur *et al* (1989) developed an explicit finite difference model of deposit heat flow, using an expanding computational grid to model the deposit growth. The average heat content of the spray arriving at the substrate ( $H_a$ ), as determined by numerical modelling of droplet solidification and the experimental metal deposition rate  $D$  was used to determine the total heat input into the deposit. The rate of heat removal from the deposit ( $H_r$ ) is determined by the deposit/substrate and gas/deposit heat transfer coefficients ( $h_s$  and  $h_g$  respectively), as approximated from experimental correlations from the literature (Martin 1977). Mathur *et al* (1989) predicted two layers in the deposit: (i) a layer of fully solidified material next to the substrate where heat extraction was dominated by heat conduction into the substrate i.e. similar to regime (ii) described above where  $H_r > H_a$ ; and (ii) a partially liquid layer which contained decreasing amounts of fraction solid from bottom to top i.e. similar to regime (i) described above where  $H_a > H_r$ .

Mathur *et al* (1989) also predicted a local solidification time  $t_f$  and temperature profile for a given point in the deposit in good agreement with the measured temperature profiles from embedded thermocouples. Bewlay & Cantor (1991) used analytical and numerical solutions of heat transfer in the deposit to calculate a deposit/substrate heat transfer coefficient of  $\approx 10^4 \text{Wm}^{-2}\text{K}^{-1}$ . Similar models by Gutierrez-Miravete *et al* (1988) and Grant

(1991) also predicted that heat transfer in the early stages of deposition was dominated by heat conduction into the substrate. As the deposit subsequently thickened, heat transfer into the substrate decreased, due to the larger thermal mass of the deposit. The deposit top surface temperature increased until it reached approximate steady state conditions. In this region, deposit cooling was now controlled by convection into the cool atomising gas. Grant & Cantor (1992) reinforced these findings by measuring the deposit top surface temperatures of Al-4wt%Cu during spray forming using infrared thermal imaging and found that the deposit top surface temperature increased initially and then reached approximate steady state conditions where the top surface temperature remained approximately constant.

## **2.9 Microstructural Evolution**

Distinctive fine grained equiaxed and low porosity microstructures in spray formed deposits have been reported by almost all investigators, although a complete understanding of the mechanism of microstructural evolution has not yet been developed. Most qualitative models of microstructural evolution involve the growth/coarsening of nuclei in the partially liquid layer (Mathur *et al* 1989, Bewlay & Cantor 1991 Grant *et al* 1993 and Annavarapu and Doherty 1995).

### **2.9.1 Grain Size**

Singer & Evans (1983) suggested that a fine grain size was produced because of a large amount of nuclei present on the deposit top surface due to seeding with pre-solidified droplets and fragmentation of dendrite arms in partially solidified droplets on

impact.

Mathur *et al* (1989) compared the measured cell sizes of spray formed Fe-20wt%Mn with the predictions of an empirical relationship (Jones 1978) for conventionally solidified material, and found that the measured cell sizes were an order of magnitude smaller than predicted by the empirical relationship. Mathur *et al* suggested that the difference was due to a higher nucleation density in the partially liquid layer due to a large number of solid droplets arriving from the spray and fragmented dendrite arms from partially solidified droplets. Bewlay & Cantor (1991) agreed with this suggestion from their observations of Sn-38wt% Pb and Sn-15wt%Pb alloys, although they suggested that fully solid droplets would be much more potent nucleation sites than fragmented dendrite arms. Mathur *et al* (1989) gave the following steps in the evolution of the final microstructure: (i) droplet remelting and interdendritic remelting on deposit top surface; (ii) the remaining solid droplets act as nucleation sites, and a high nucleation density leads to a fine grain size; and (iii) solid state coarsening of the resulting structure. Lavernia (1989) suggested that the equiaxed grain structure was a result of three processes: (i) dendrite arm fragmentation, due to mechanical fracture and dendrite arm remelting; (ii) nucleation/grain multiplication; and (iii) constrained growth. This was in general agreement with both Mathur *et al* (1989) and Bewlay & Cantor (1991).

Generally the final grain size is determined by: (i) the size, heat content and rate of arrival of droplets at the deposit top surface i.e. the density of solid nuclei controlled by heat transfer in the droplet spray and subsequent to deposition by the deposit macroscopic heat flow; (ii) the extent of grain growth which takes place in the solid/liquid region on the deposit top surface; and (iii) the extent of additional grain growth which

takes place during cooling in the fully solid region below the deposit top surface.

Recently, Liang *et al* (1992), Grant *et al* (1993C) and Annavarapu and Doherty (1995) have investigated grain growth in the solid/liquid region and fully solid region just below the liquidus by re-heating spray formed samples. Liang *et al* suggested that the fine grain size in Ni<sub>3</sub>Al was as a result of two mechanisms: (i) the homogenisation of dendrites that did not deform extensively during deposition; and (ii) the growth and coalescence of the deformed or fractured dendrite arms. Support to this suggestion was provided by experimental and numerical evidence which showed that the deposit was exposed to a high temperature anneal below the solidus temperature during deposition. However, Annavarapu and Doherty (1993) suggested that the numerical model (Lavernia *et al* 1988) used by Liang *et al* (1992) was only valid for the initial transient region of spray forming when substrate conduction was significant and typical splat or layered microstructures present.

Annavarapu and Doherty (1995) suggested that grain coarsening in spray formed deposits occurred by the migration of liquid-wetted grain boundary films via solution and reprecipitation mechanism and that minority insoluble second phase particles observed in many samples were responsible for inhibiting grain growth and therefore the resulting fine grain size typical of spray formed deposits. Grain growth in the solid/liquid region was proposed to follow cubic coarsening kinetics of the form:

$$d^3 = d_0^3 + Kt \quad (2.6)$$

where  $d$  is the grain size at time  $t$ ,  $d_0$  is the initial grain size and  $K$  is a coarsening rate constant. Similar behaviour was observed by Grant *et al* (1993C) from experiments on several spray formed Al alloys.

### **2.9.2 Porosity**

Porosity is always present in spray formed deposits and is usually closed by subsequent forging, rolling, extrusion or HIPing. Mathur *et al* (1989) identified three different causes of porosity: (i) gas entrapment; (ii) solidification shrinkage; and (iii) insufficient liquid feeding. Porosity due to gas entrapment occurs as the atomising gas becomes entrapped in liquid on the deposit top surface. Bewlay & Cantor (1991) suggested that an excessive amount of liquid on the top surface led to large amounts of entrapped gas porosity. Lavernia (1989) suggested that porosity due to solidification shrinkage occurred when large amounts of liquid were present on the deposit top surface after the atomising gas was turned off (similar to gravity casting). Insufficient liquid feeding is likely to lead to interstitial irregularly shaped pores between grains, especially in the early stages of spray forming where liquid flow is limited by the fast cooling associated with effective heat transfer into the substrate i.e.  $H_r > H_a$ . Lavernia (1989) suggested that the small micron sized pores at grain boundaries, often observed in aluminium alloys were due to insufficient liquid feeding.

### **2.10 Spray Forming of Superalloys**

Refined grain sizes and low segregation make spray forming an attractive manufacturing route for Ni superalloys where small grain sizes give high strength and good forgeability, and reduced macrosegregation allows alternative more highly alloyed Ni materials to be used and/or developed. Much of the published work on spray formed Ni superalloys has focused on the mechanical properties of the spray formed deposits after consolidation by forging/HIPing and heat treatment. However, there are some reports of

the variation of the initial spray formed microstructure with process conditions.

Many investigators (Bricknell 1986, Chang & Fiedler 1988 and Dalal & Pritchard 1992) have reported as-sprayed densities of  $\geq 98\%$  for a wide range of spray formed superalloys (René 80, René 95 AF115, AF2-1DA, Astroloy, MERL 76, IN718, René 41 and Waspaloy) atomised with both Ar and N<sub>2</sub>. Densities increased to 100% after subsequent consolidation.

Bricknell (1986) investigated spray formed René 80 atomised with both Ar and N<sub>2</sub>. The Ar atomised material exhibited a larger proportion of spherical pores, typically  $\approx 50\mu\text{m}$  which were suggested to be stabilised by Ar, whereas the N<sub>2</sub> atomised material had a higher density. Fiedler *et al* (1987) also found that spray formed IN718 and René 95 had a lower density when atomised with Ar compared with N<sub>2</sub>, due to the presence of a larger number of small spherical pores. Bricknell (1986), Fiedler *et al* (1987) and Chang & Fiedler (1988) all found that N pick up in the deposit increased substantially when N<sub>2</sub> was used as the atomising gas. Bricknell's data on SF René 80 is shown in table 2.2, which shows that the O and N content of N<sub>2</sub> atomised material was an order of magnitude greater than Ar atomised material. Bricknell (1986) and Fiedler *et al* (1987) both speculated that the higher deposit density for N<sub>2</sub> atomised deposits was because of the tendency to form nitrides and carbonitrides rather than porosity.

Benz *et al* (1993) suggested that for N<sub>2</sub> atomised René 95, N present at levels  $>10\text{ppm}$  should precipitate as nitrides or carbonitrides. From TEM observations of SF IN718 and René 95 they observed clusters of sub-micron (Ti,Nb)(C,N) carbonitrides which, they suggested precipitated while the matrix was in the liquid state and that they clustered together by a liquid state agglomeration process. Benz *et al* (1993) therefore

concluded that N pickup during atomisation resulted in an increased number of micron sized Ti and Nb carbonitride agglomerates in the microstructure. However, the precipitates were sufficiently small so that they did not appear to influence low cycle fatigue (LCF) life.

| Material          | Oxygen Content (ppm) | Nitrogen Content (ppm) |
|-------------------|----------------------|------------------------|
| Starting material | 19.17                | 12.14                  |
| Argon atomised    | 40.44                | 18.13                  |
| Nitrogen atomised | 99.102               | 185.146                |

Table 2.2 Oxygen and nitrogen contents of spray formed René 80 (after Bricknell 1986).

Benz *et al* (1991) spray formed IN718 at flow rates significantly lower ( $<20\text{cm}^3/\text{s}$ ) than previously (Fiedler *et al* 1987), using Ar as the atomising gas. They found that the lower flow rates did not significantly increase the porosity ( $<2\%$ ) as would be anticipated from previous work (Fiedler *et al* 1985), which could then be eliminated by a 75% forge reduction. The microstructure did not exhibit a layered structure typical of "cold" deposition conditions (i.e.  $H_r > H_a$ ), which they suggested was because the lower heat capacity of Ar did not cool the droplets in flight as much.

The choice of Ar or  $\text{N}_2$  as the atomising gas is dependent upon several factors: (i) the flow rates used; (ii) forging reductions possible; (iii) acceptable amount of N pick up; and (i) cost of the gas and the possibility of gas recycling. Ar atomisation is expensive and has slightly higher deposit porosities ( $<2\%$ ), although there is no increase in the number of carbonitride precipitates. While  $\text{N}_2$  atomisation is cheaper and has lower deposit porosities, although there is an increase in the number of carbonitride precipitates



which may affect subsequent processing routes.

Bricknell (1986), Fiedler *et al* (1987) and Benz *et al* (1990) all reported fine grained equiaxed microstructures for a wide variety of superalloys. Grain sizes were typically in the range 25-40 $\mu\text{m}$  (Chang & Fiedler 1988), although grain sizes as low as 12 $\mu\text{m}$  were observed in René 80 by Bricknell (1986).

The mechanical properties of as-sprayed material is degraded by the 1-2% porosity present and is therefore usually subjected to heat treatments and a forging reduction or HIP treatment before mechanical testing. Fiedler *et al* (1987) showed that the tensile properties at 650°C of N<sub>2</sub> atomised, SF IN718 were slightly less than the equivalent cast and wrought material. The properties were improved by HIP consolidation and further improved by suitable forging. Stress rupture properties were also improved by HIPing or forging. Ar atomised IN718 had lower tensile and stress rupture properties even after HIPing or forging because of incomplete densification. Similar trends in tensile and stress rupture properties were observed for René 95 by the same authors. In a later paper, Benz *et al* (1991) showed that the tensile and stress rupture properties for Ar atomised IN718 at lower flow rates of <20cm<sup>3</sup>/s and a press forging reduction of 75%, were equivalent to conventional cast and wrought material.

Fiedler *et al* (1987) stated that SF Ni superalloy low cycle fatigue life (LCF) properties were of prime importance, reflecting typical applications for which these superalloys are being considered. Fiedler *et al* (1987) measured the low cycle fatigue life of N<sub>2</sub> atomised SF René 95 after press forging, and concluded that the LCF life from SF forgings was similar to that reported for fine grain size, powder consolidated specimens. Fracture sites in these specimens were frequently associated with particles of Mg and Al

or Al and Si when analysed using EDS (energy dispersive spectrometers). Since magnesia crucibles were used, these particles were assumed to be debris from the crucible and from cement used in the melting arrangement.

The alloys to be studied in this project are UDIMET<sup>3</sup> 720 and MAR-M-002. Their nominal compositions are given in table 2.3. UDIMET 720 is a cast-wrought Ni base superalloy which was originally developed for use in land based gas turbine blades, and later modified and developed for use in jet engine disks (Sczerzenie & Maurer 1984). MAR-M-002 is another cast-wrought high strength Ni base superalloy, which was originally developed for use as a directionally solidified (DS) blade alloy.

|           | Ni  | Al  | Cr | Co | Mo | Ti  | W   | Hf  | Ta  | C    | B    | Zr  |
|-----------|-----|-----|----|----|----|-----|-----|-----|-----|------|------|-----|
| U720      | Bal | 2.5 | 18 | 15 | 3  | 5   | 1.5 | -   | -   | .035 | .033 | .03 |
| MAR-M-002 | Bal | 5.5 | 9  | 10 | -  | 1.5 | 10  | 1.5 | 2.5 | 0.15 | .015 | .03 |

Table 2.3 Nominal composition of UDIMET 720 and MAR-M-002 in wt%.

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<sup>3</sup>UDIMET is a registered trade name of the Special Metals Corporation.

## **CHAPTER 3 - EXPERIMENTAL**

This chapter describes design modifications made to the existing spray forming system at Oxford University, and the key features of the new spray forming system; the experimental details of spray forming the Ni superalloy UDIMET 720 and the monitoring of process parameters; the procedure for re-heating spray formed samples to investigate the effect of time and temperature on the grain size; and the microstructural characterisation of spray formed deposits and re-heated spray formed samples.

### **3.1 Spray Forming**

This section describes modifications that were necessary to the spray forming melting arrangement required for the spray forming of Ni superalloys, and a description of the resulting spray forming system and procedure for spray forming.

#### **3.1.1 Modifications to the Spray Forming System**

The spray forming system in use at Oxford was designed for the spray forming of Al alloys (Grant 1991) and some modifications to the melting arrangement and melt delivery systems were required to spray much higher melting point superalloys. The previous design for spray forming Al alloys is shown in fig 3.1(a) and the modified design for spray forming superalloys is shown in fig 3.1(b). The modified system was designed in consultation with Rolls-Royce to melt  $\approx 3\text{-}4\text{kg}$  of Ni superalloy at  $\approx 1450^\circ\text{C}$ . The key features of the new melting design were:

1. An alumina ( $\text{Al}_2\text{O}_3$ ) crucible surrounded by an alumina tube, packed tightly with 'Pog Ram Mix' and situated on an  $\text{Al}_2\text{O}_3$  plate which had a 30mm central hole to allow the

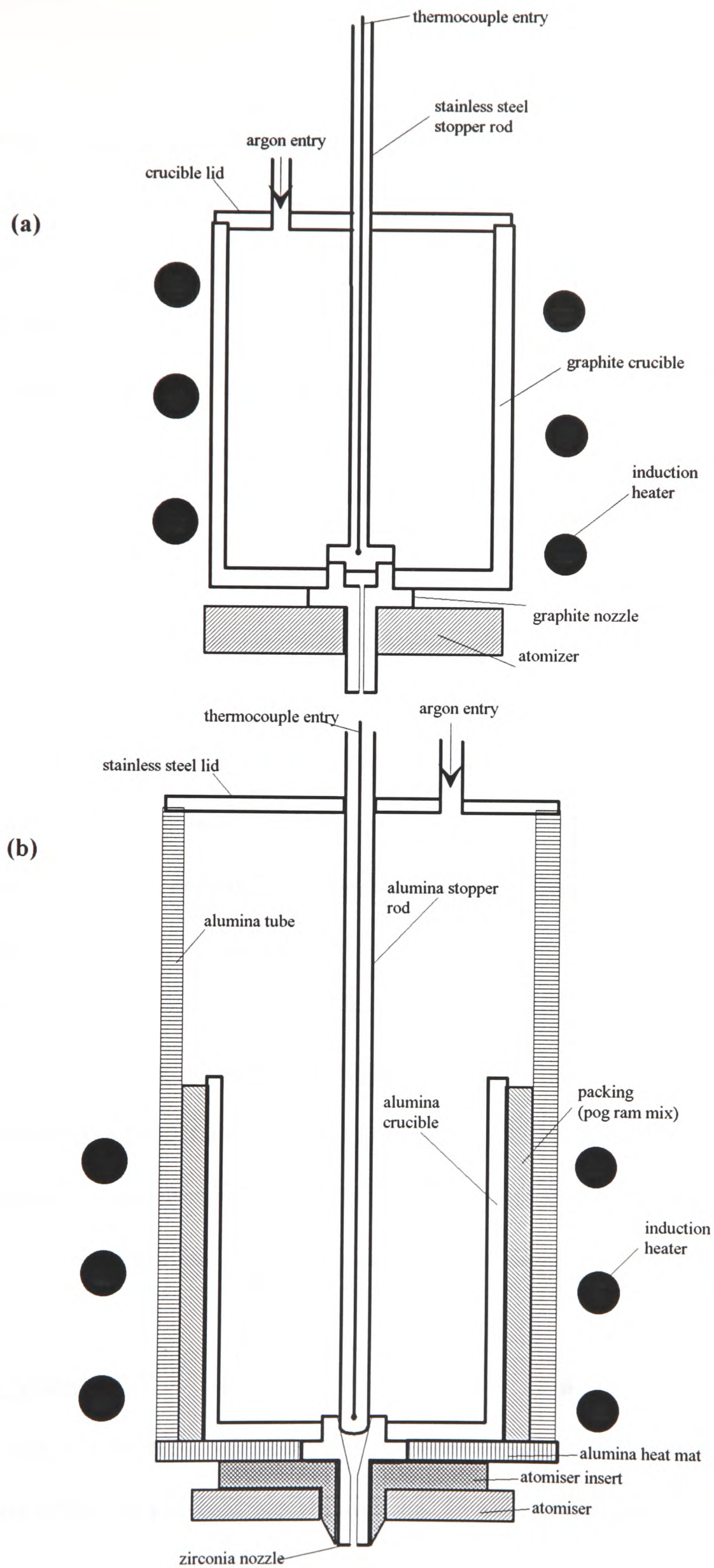


Fig 3.1(a) Previous melting arrangement for Al alloys and (b) modified melting arrangement for Ni superalloys.

melt delivery nozzle to pass through.

2. An  $\text{Al}_2\text{O}_3$  plate to support the crucible arrangement, placed on a stainless steel atomiser insert, which fitted through a central hole in the atomiser. The atomiser insert acted as an additional heat shield for the atomiser and shielded the nozzle from the atomiser gas jets until the metal exited from the nozzle in the atomisation region. Fig 3.2 is a schematic diagram of the atomiser insert.
3. A  $\text{Zr}_2\text{O}_3$  melt delivery nozzle which fitted tightly into a central hole in the bottom of the crucible. The nozzle delivered molten metal through the atomiser insert into the atomisation region. Fig 3.3 is a schematic diagram of the nozzle.
4. A stainless steel lid which fitted onto the top of the  $\text{Al}_2\text{O}_3$  tube, and had a central hole for an  $\text{Al}_2\text{O}_3$  stopper rod with a thermocouple inserted in the rod. The bottom of the rod was located in the top of melt delivery nozzle.
5. A pneumatic actuator supported on top of the stainless steel lid to raise the stopper rod at the beginning of spray forming.
6. A Cheltenham Induction 12kW microprocessor controlled induction heater modified to provide maximum power to melt the superalloy charge by a suitable induction coil.
7. A Eurotherm 815 temperature controller using a type R feedback thermocouple located in the stopper rod was used to control the voltage supplied to the induction heater and therefore control the heating rate and superheat of the superalloy charge.

### **3.1.2 Gas Atomiser**

A new gas atomiser was designed as shown in fig 3.4 which had 225% bigger holes by area than the previous gas atomiser i.e. 16 x 1.5mm diameter holes instead of

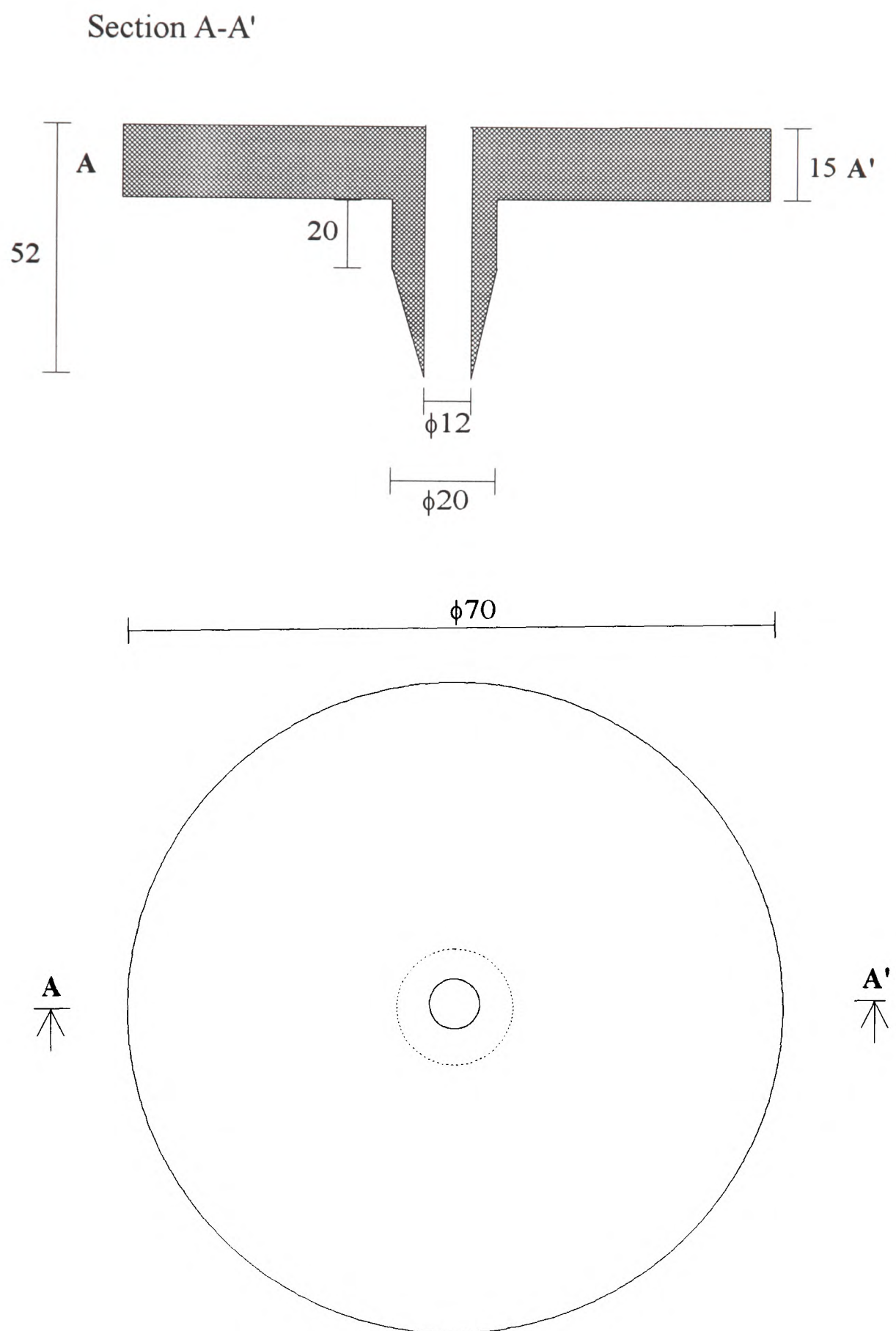


Fig 3.2 Schematic diagram of the atomiser insert.

Section A-A'

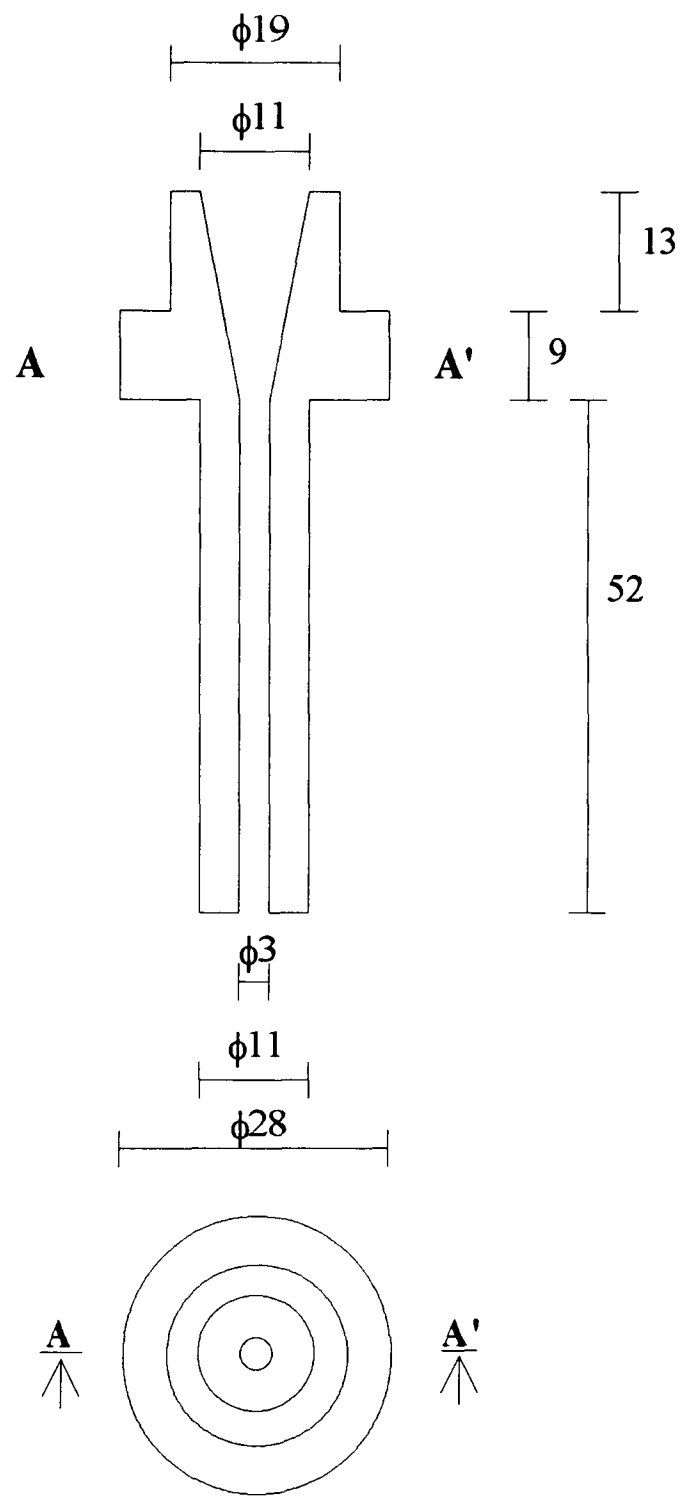


Fig 3.3 Schematic diagram of the zirconia nozzle.

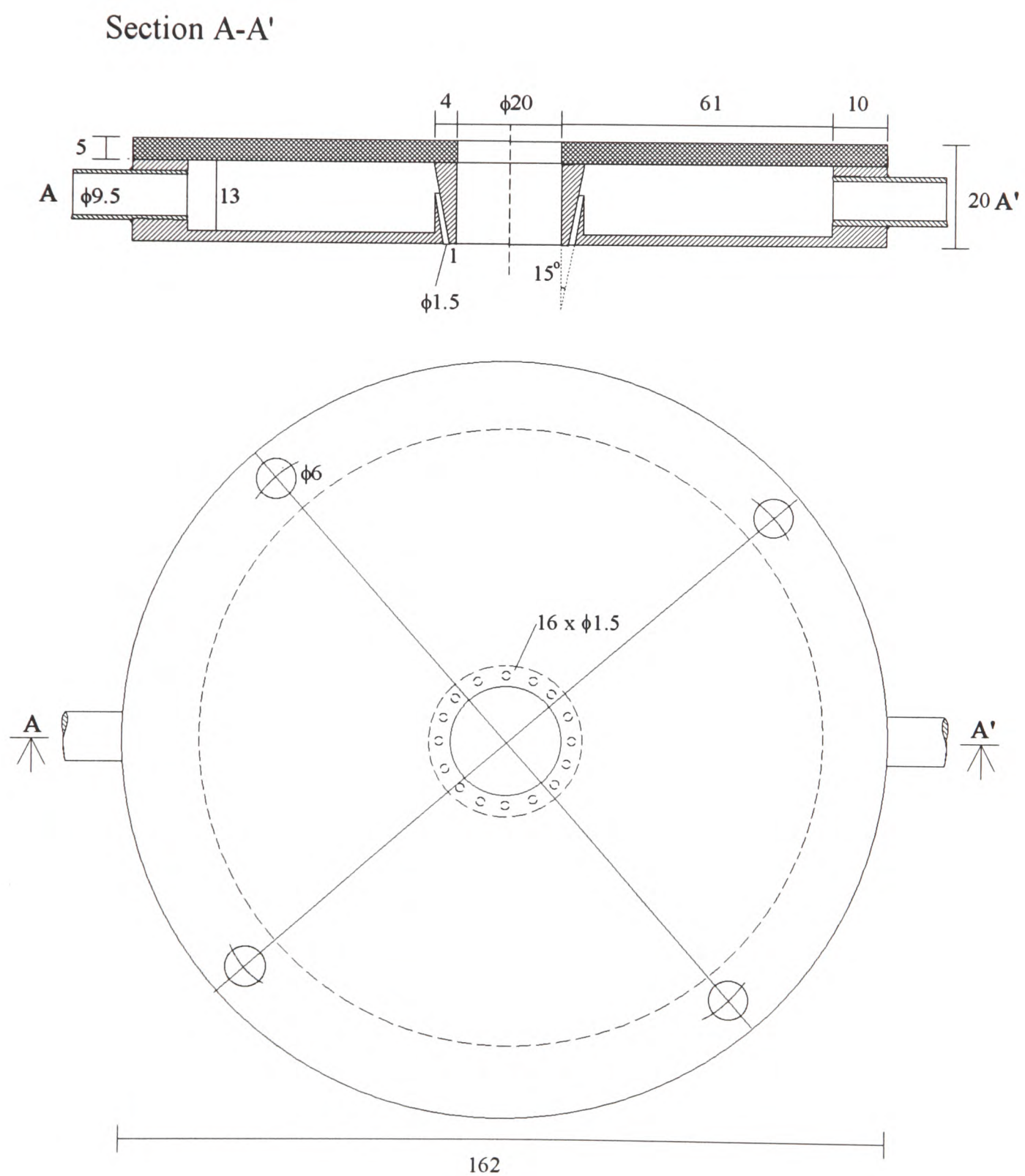


Fig 3.4 Modified gas atomiser.



16 x 1mm diameter holes. The Lubanska correlation (1970) for droplet sizes of gas atomised powders states that the mass median diameter ( $d_m$ ) is proportional to MFR:GFR where MFR and GFR are the mass flow rates of the liquid metal and gas respectively. Therefore a higher GFR through the atomiser should atomise more efficiently higher density, and therefore higher mass flow rate superalloys, leading to a smaller mass median droplet diameter ( $d_m$ ).

### **3.1.3 Description of the Spray Forming System**

Fig 3.5 is a schematic diagram of the whole spray forming system. The key features were:

1. A N<sub>2</sub> atomising gas supply provided by multiple pressurised gas bottles with a pressure gauge metered in the range 0-1.4MPa.
2. A Platon gas flow meter calibrated for N<sub>2</sub> gas volumetric flow rates in the range 0-5.5l/s.
3. An Ar overpressure gas supply provided by a similar arrangement of multiple pressurised gas bottles with a pressure gauge metered in the range 0-1.4MPa. The overpressure gas supply was connected to a gas reservoir tank fitted with a gas exit solenoid valve metered in the pressure range 0-70kPa.
4. Two Druck DPC 270 pressure transducers with digital readouts calibrated for pressures in the range 0-1.4MPa placed in the flow meter manifold and in the plenum chamber of the atomiser to measure the atomising gas pressure.
5. An 11cm diameter, 4cm thick roughened degreased stainless steel substrate attached to a rotating substrate table that could also be positioned at an angle to the spray chamber

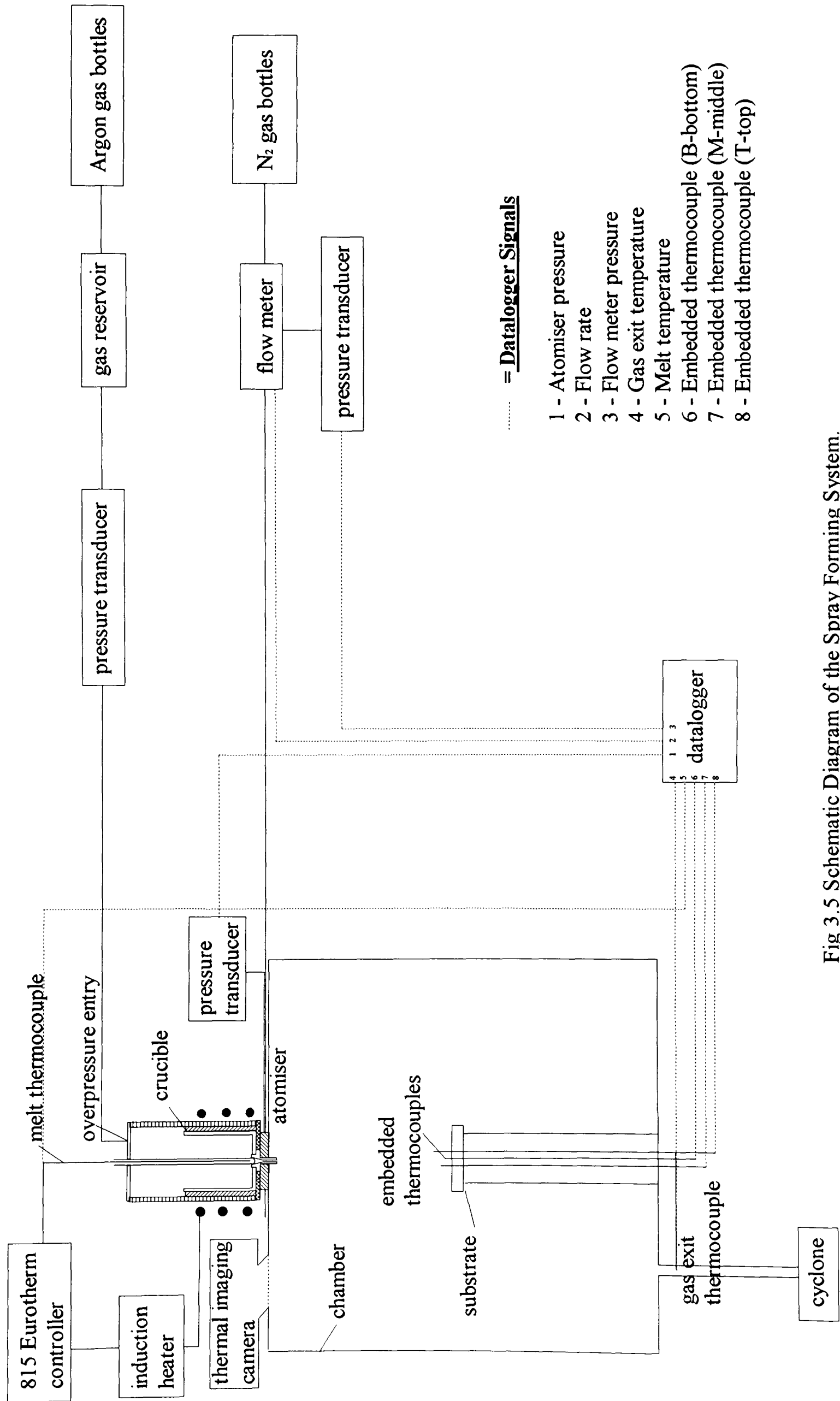


Fig 3.5 Schematic Diagram of the Spray Forming System.

axis by means of adjusting the height of three tripod legs connecting the substrate table to the chamber base. Three thermocouples could also be positioned above the substrate to allow deposit temperature measurement during spray forming.

6. 4-20mV output signals taken from each of the two Druck DPC 270 pressure transducers and the Platon gas flow meter were connected to a Schlumberger 3531F datalogger to continuously measure the atomising gas pressure and flow rate during spray forming. Type K and R thermocouples were also connected to the datalogger to allow continuous temperature measurements during spray forming.

7. A 12kW Cheltenham Induction heater controlled by a Eurotherm 815 temperature controller was used to heat the superalloy charge by a copper induction coil.

8. A cyclone was connected to an exit manifold on the spray chamber base plate by a heat resistant 4cm diameter flexible pipe. The cyclone had an outlet filter that removed particulate down to diameters of  $\approx 1\mu\text{m}$ .

Fig 3.6(a) shows a photograph of the spray forming arrangement indicating: (i) the spray forming chamber, (ii) the melting arrangement and induction coil and (iii) thermal imaging camera. Fig 3.6(b) shows a photograph of the spray forming control equipment indicating: (i) the computer to control the thermal imaging camera, (ii) the computer to control the datalogger and (iii) the instrument panel to control overpressure, gas flow rate, stopper rod and cyclone.

#### **3.1.4 Spray Forming Procedure**

To perform a spray forming run the following procedure was followed:

1. The atomiser insert was supported on two secure metal blocks. The melt delivery





**Fig 3.6(a) Photograph of the spray forming arrangement showing; (i) the spray forming chamber, (ii) the melting arrangement and induction coil and (iii) thermal imaging camera.**



**Fig 3.6(b) Photograph of the spray forming control equipment showing; (i) computer to control the thermal imaging camera, (ii) computer to control the datalogger and (iii) instrument panel to control overpressure, gas flow rate, stopper rod and cyclone.**



nozzle was then placed through the atomiser insert and the  $\text{Al}_2\text{O}_3$  plate on top of the atomiser insert, so that the top of the melt delivery nozzle projected through the hole in the  $\text{Al}_2\text{O}_3$  plate.

2. The  $\text{Al}_2\text{O}_3$  crucible was placed onto the  $\text{Al}_2\text{O}_3$  plate so that the top of the melt delivery nozzle fitted into the hole in the bottom of the  $\text{Al}_2\text{O}_3$  crucible. The  $\text{Al}_2\text{O}_3$  tube was then placed around the crucible as shown in fig 3.1(b).

3. The space between the tube and crucible was packed tightly with 'Pog Ram Mix' to provide support of the crucible during melting of the charge.

4. The entire crucible arrangement was transferred to the spray forming chamber so that the atomiser insert fitted through the hole in the atomiser.

5. A 15mm hole was drilled through a 76cm diameter,  $\approx 100\text{cm}$  high and  $\approx 3\text{kg}$  UDIMET 720 bar; the bar was then placed in the crucible and an  $\text{Al}_2\text{O}_3$  stopper rod located in the top of the melt delivery nozzle with a thermocouple inserted in the stopper rod. The stainless steel lid was secured onto the top of the  $\text{Al}_2\text{O}_3$  tube and the top of the stopper rod fixed onto the pneumatic actuator.

6. The substrate table was set to the required height and angle by appropriate adjustment of the tripod legs and the substrate table connected to an electric motor which rotated at 50rpm.

7. The chamber base plate was bolted to the bottom of the spray chamber and glass windows fitted into the chamber ports.

8. All thermocouples, pressure transducers and the flow meter output signals were connected to the datalogger which was set to record data at 1.5Hz.

9. The heating rate of the charge controlled by the Eurotherm 815 controller was set to

heat up at 14°C/min to the superheat temperature (typically  $\approx 1445^{\circ}\text{C}$ ). The melt was then held at the superheat temperature for  $\approx 7$  mins until spraying began.

10. At the superheat temperature the induction heater was turned off and the cyclone and substrate rotation turned on. The argon overpressure and the stopper rod were operated simultaneously to allow metal to flow through the nozzle for 1-2s to heat the nozzle up and help prevent 'freeze off' of the metal in the nozzle.

11. The atomising gas was turned on and spray forming continued until all the metal had flowed through the nozzle.

12. After spraying the cyclone, substrate rotation, Ar overpressure and  $\text{N}_2$  atomising gas were turned off and the apparatus left to cool for several hours.

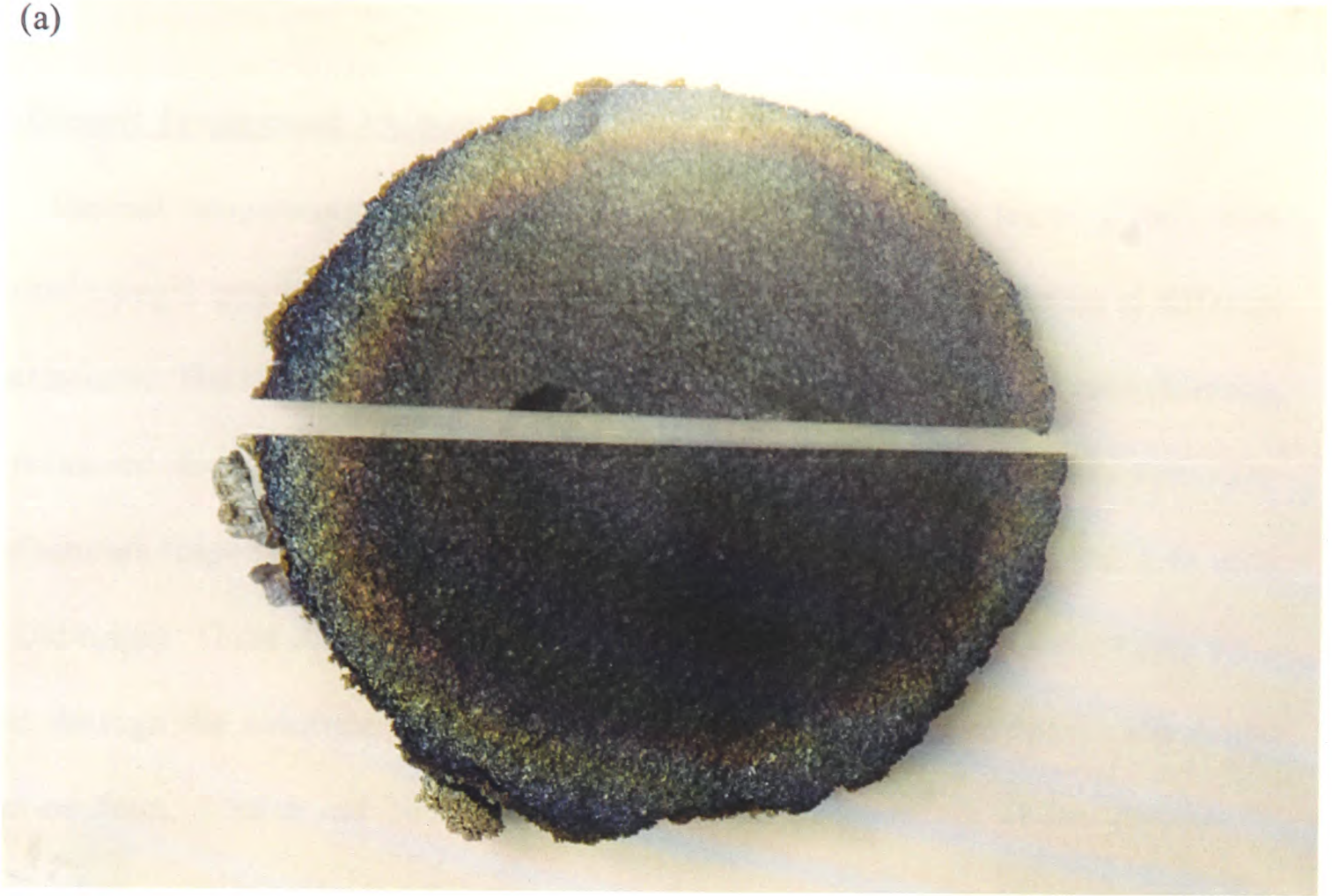
13. The base plate and chamber windows were removed, while wearing dust masks, and any overspray powder remaining in the chamber was collected in the cyclone.

Fig 3.7 shows a typical spray formed deposit in (a) plan view and (b) cross section. The cross sectional shape of spray formed deposits was approximately Gaussian.

### **3.2 Spray Forming of UDIMET 720**

To produce deposits with a range of microstructures the following process parameters were varied independently: (i) atomising gas flow rate; (ii) spray distance (atomiser to substrate distance) and (iii) substrate angle. All the process conditions were carefully monitored during spraying by the Schlumberger datalogger and an infra-red thermal imaging camera. The UDIMET 720 starting material was provided by Rolls-Royce plc from cast VIM (vacuum induction melted) stock.

(a)



(b)

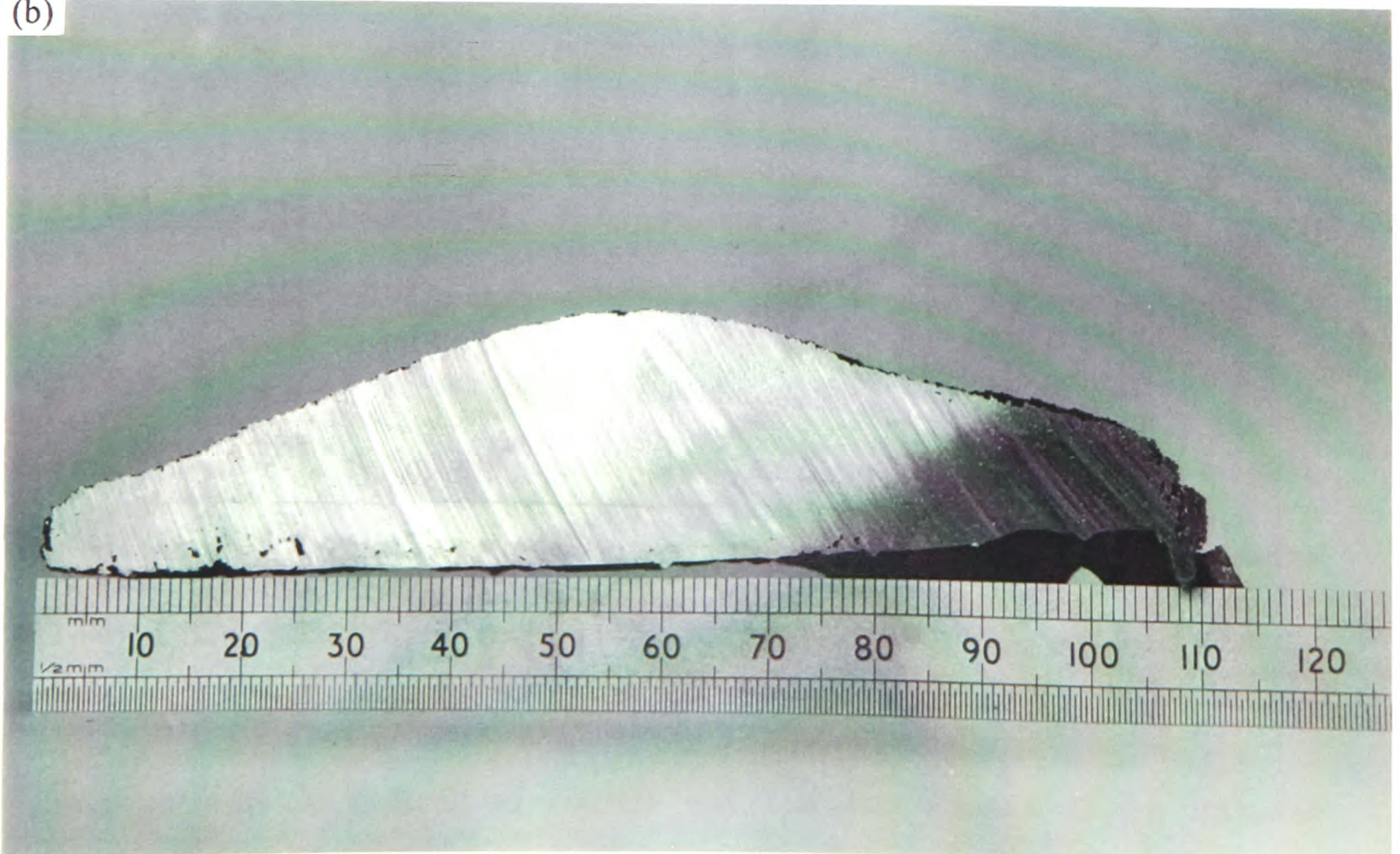


Fig 3.7 Typical spray formed deposit (a) in plan view and (b) in cross section

### **3.2.1 Deposit Temperature Measurement**

Deposit temperatures during manufacture for a set of spray forming runs were measured using 3 type K thermocouples projecting above the substrate surface at different pre-set heights. The thermocouples became embedded in the deposit during spray forming and measured the instantaneous deposit temperature and deposit cooling rate. The manufacturers response time for the above thermocouples was 0.4s (TC Ltd, P.O. Box 130, Uxbridge). Three 3mm holes equidistant from each other at a 20mm radius were drilled through the substrate and thermocouples fixed at heights of 5mm, 10mm and 15mm or 5mm, 7.5mm and 10mm, depending on the likely height of the deposit, by carborundum ceramic paste. Fig 3.9 shows typical embedded thermocouple measurements recorded at 5mm, 7.5mm and 10mm above the substrate. The trailing thermocouple wires exited the chamber through a 3cm diameter manifold in the chamber base. It was not possible to rotate the substrate during these runs because of the trailing thermocouple wires.

### **3.2.2 Infra Red Thermal Imaging**

Infra-red thermal imaging is a technique for measuring the spatial distribution of thermal radiation intensity in the infra-red range (1-30 $\mu$ m). Given suitable calibration, thermal intensity can be converted into temperature (Grant 1991 and Grant *et al* 1989). The advantages of using thermal imaging are that (i) it requires no external illumination and (ii) it is a real time and non-intrusive technique. However, absolute temperature measurements by infra-red thermal imaging are problematical because the infra-red thermal intensity is difficult to convert into real temperatures. If the infra-red



characteristics of the imaging camera have been determined by calibration at manufacture then:

$$I_o = I_e + I_r + I_a \quad (3.1)$$

where  $I_o$  is the intensity of infra-red radiation received at the detector,  $I_e$  is the intensity of infra-red radiation emitted by the object,  $I_r$  is the intensity of infra-red radiation reflected by the object and  $I_a$  is the intensity of infra-red radiation emitted by the intervening atmosphere. For spray forming  $I_r$  is negligible compared with  $I_e$  because of the relatively high temperature of the deposit top surface. The infra-red contribution from the intervening atmosphere  $I_a$  is accessible from standard calibration tables. For 100% transmittance and  $I_r=0$  then

$$I_o = \epsilon_o(T)I_{bb} + I_a \quad (3.2)$$

where  $I_{bb}$  is the intensity of infra-red radiation emitted by a black body at temperature  $T_o$  and  $\epsilon_o$  is the surface emissivity (assumed constant over the infra-red detection wavelength). Therefore, in order to determine the object temperature from measured infra-red thermal intensity, the object surface emissivity ( $\epsilon_o$ ) and the transmittance of any intervening ports or windows must be known.  $\epsilon_o$  depends on (i) the object material, (ii) the surface condition of the object and (iii) temperature.

### 3.2.2.1 Thermal Imaging Procedure and Equipment

An Agema Thermovision 870 thermal imaging camera was used to take thermal images of the deposit top surface during spray forming and the resulting images were analysed and manipulated using Agema CATS E software running on a Compaq 286 Deskpro computer. The computer was fitted with an Agema TIC-4000 interface board which provided in real time: (i) digitised and colourised thermal images; (ii) conversion

of measured infra-red thermal intensities to object surface temperatures using an assumed  $\epsilon_o$ ; and (iii) computer control of the thermal imaging camera settings (range and level).

The thermal imaging camera was fixed securely to a mounting arrangement on the frame supporting the spray forming chamber so that it could view the deposit through a port in the chamber top plate fitted with a silicon window as shown in fig 3.6(a). The interior surface of the silicon window was coated with anti-abrasive and anti-reflective coatings. Recording of the thermal images was started a few seconds before spraying and images were recorded at 1Hz. Each image was obtained over a period of 0.04s and up to a maximum of 100 images could be recorded in one run. A typical sequence of thermal images is shown in fig 3.8(a) for run 30 using  $\epsilon_o=0.3$ .

### 3.2.2.2 Calibration of Deposit Top Surface Temperatures

Images showing the deposit top surface temperature during spray forming were obtained using an assumed object surface emissivity of 0.3, which produced temperatures in the range 800°C-1900°C. To obtain more quantitative measurements of the deposit top surface temperature, calibration of the thermal images was performed using the embedded thermocouple temperature measurements.

Surface temperatures were measured by averaging from a central circular region of the image as shown in fig 3.8(b) for run 10 assuming  $\epsilon_o=0.3$ . The same region was used for all the images of a spray forming run. A typical plot of top surface temperature using  $\epsilon_o=0.3$  and the corresponding embedded thermocouple temperatures is shown in fig 3.9. In order to calibrate the thermal images, for each spray run the maximum temperature of a fully embedded thermocouple as it was embedded by the growing deposit top surface was plotted against the average top surface temperature measured by thermal imaging at



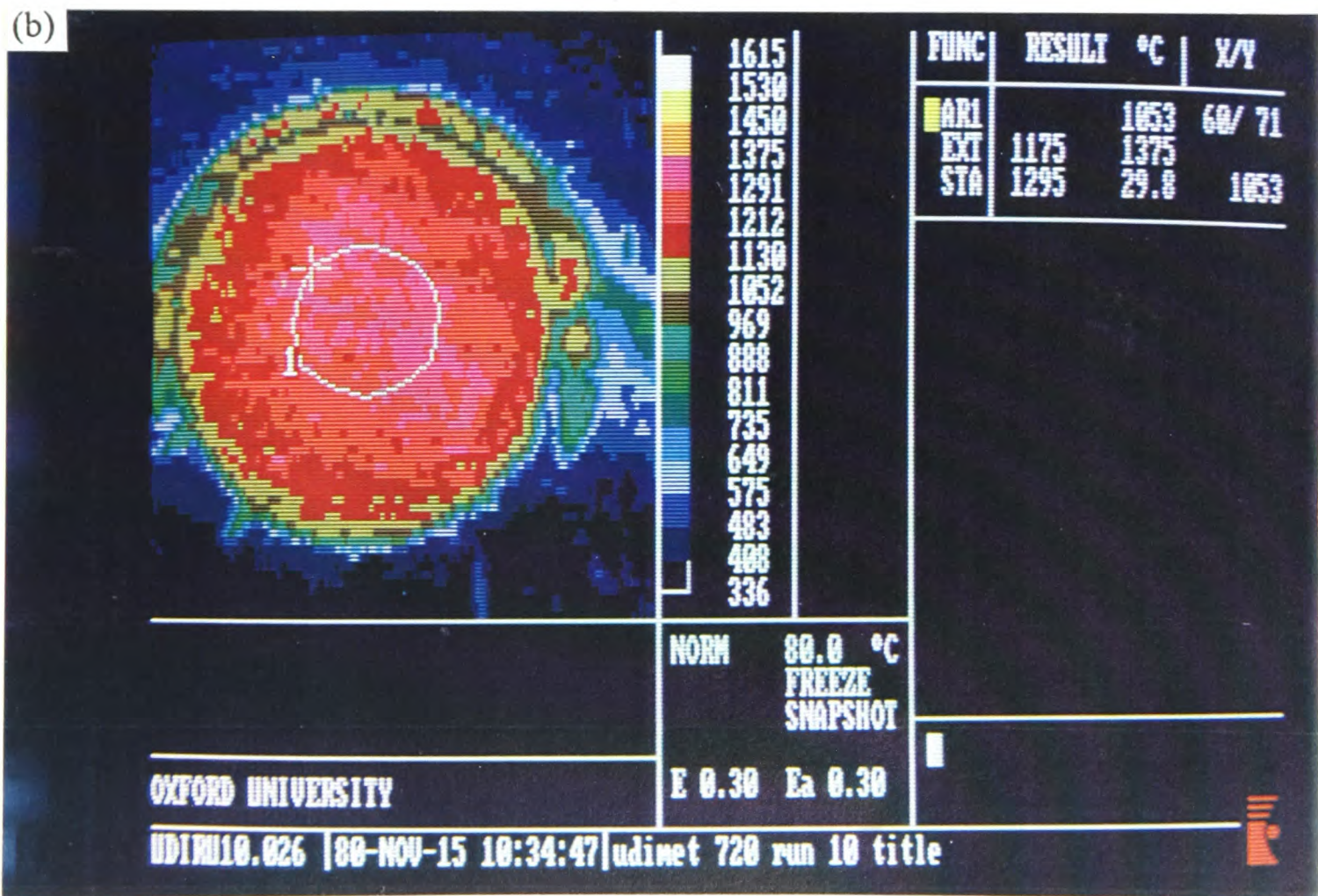
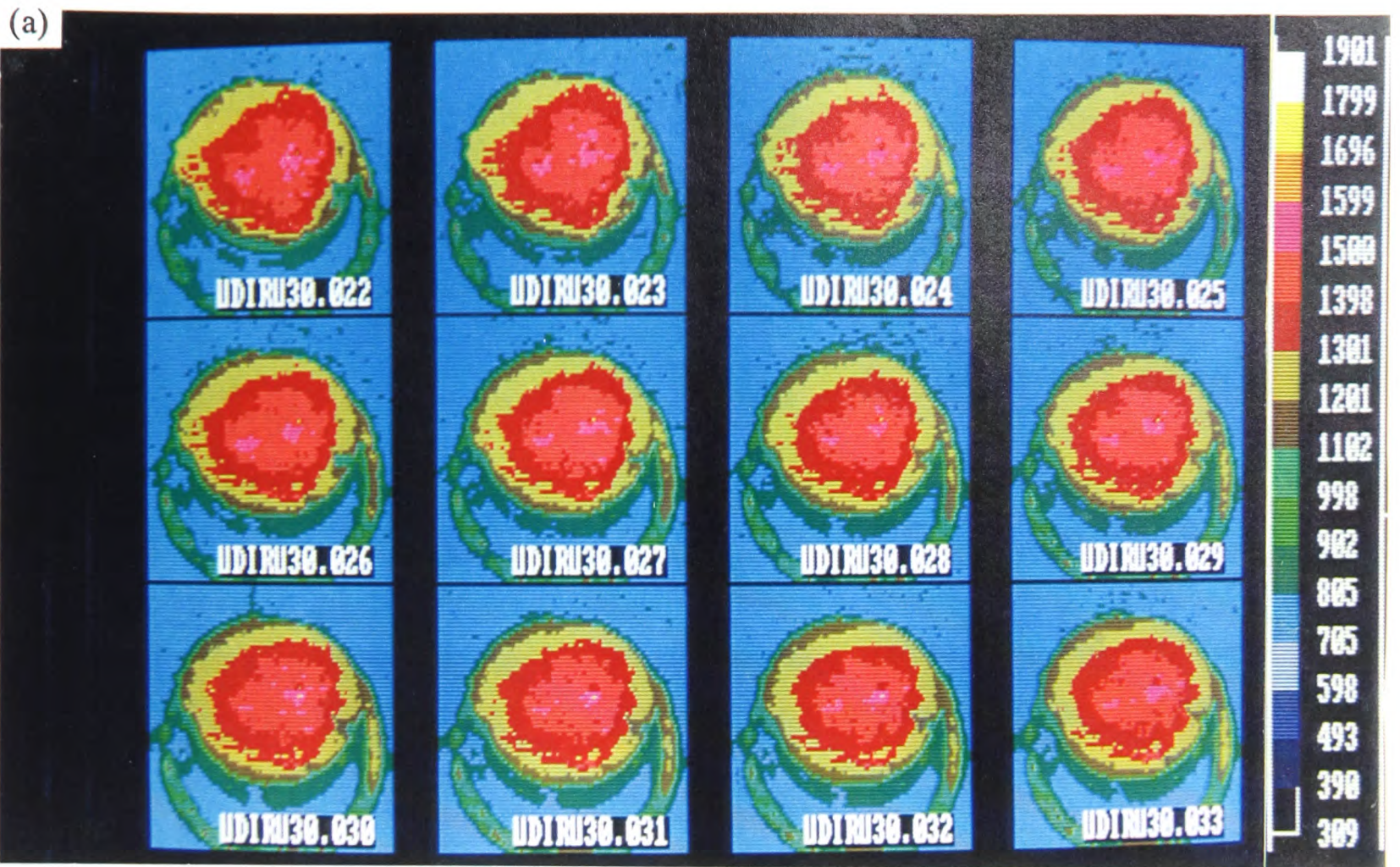


Fig 3.8 Typical (a) sequence of thermal images of the deposit top surface from run 30 and (b) individual thermal image showing the region where average temperature measurements were taken from for run 10.



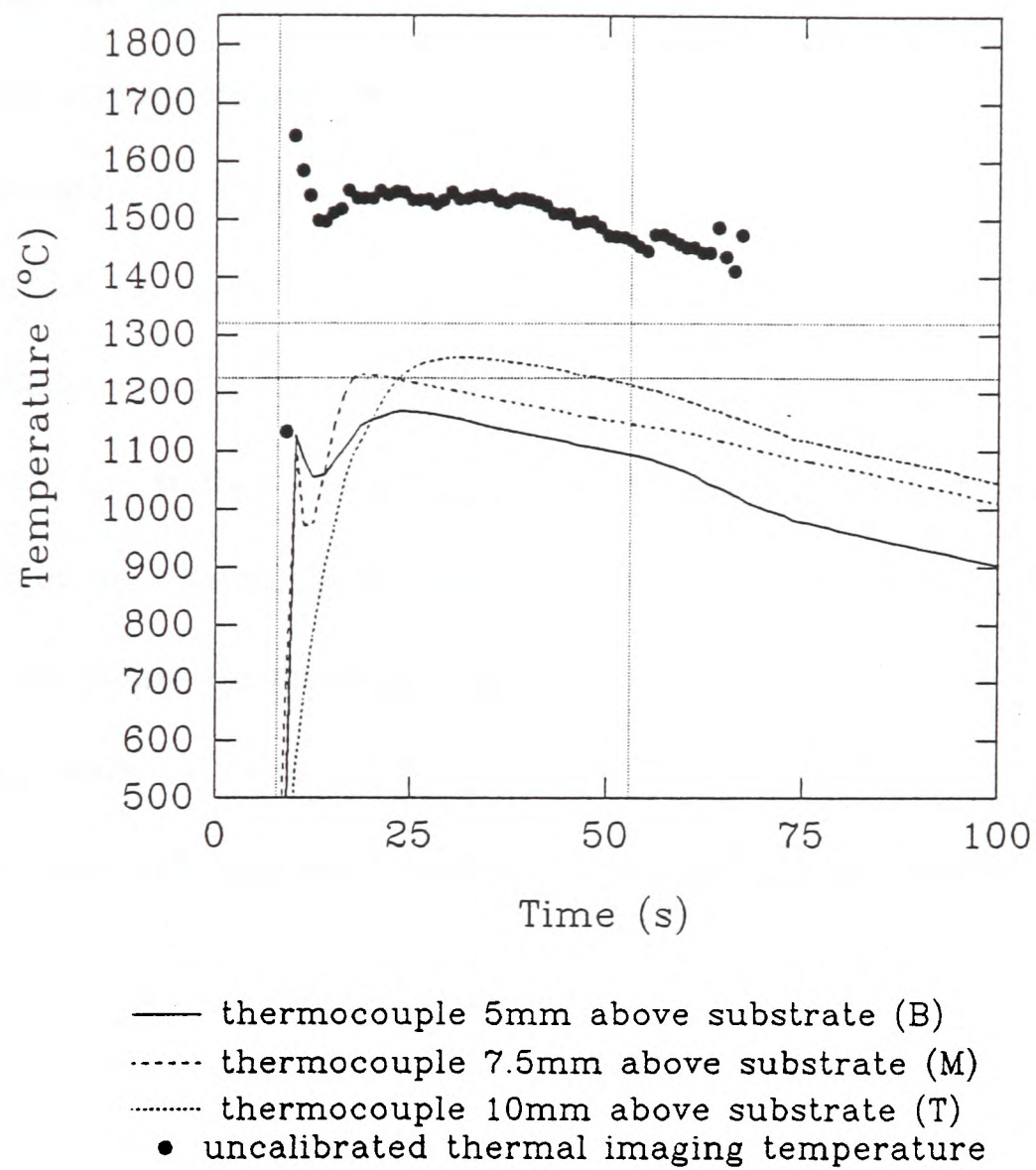


Fig 3.9 Typical top surface temperature from thermal imaging measurements using  $\epsilon_0=0.3$  and embedded thermocouple measurements during a spray run.

that point. In this way a calibration curve was obtained which related measured top surface temperature to the actual top surface temperature throughout the run. This calibration technique assumes that: (i) the object surface emissivity remains constant during the spray run; and (ii) the maximum embedded thermocouple temperature is the same as the top surface temperature. (i) is reasonably as long as temperatures do not significantly deviate from the calibration temperature i.e. cooling below the alloy solidus or oxidation of the top surface which significantly alter  $\epsilon_o$  will cause inaccuracies; and (ii) is approximately true for the instant just as the thermocouple becomes fully embedded below the deposit top surface i.e. reductions in thermocouple response time because of prior deposition on the exposed thermocouple beads will introduce some inaccuracies. The results of this calibration are shown in detail in chapter 5, section 5.3.2.

Previous work by Grant (1991) showed that the transmittance of the silicon window was at least 98% and this conclusion was supported by Newbery (1994). The effects of the spray and dust during spray forming on the measured infra-red intensity was ignored due to the difficulty of calibrating this effect. Qualitative inspection of thermal images shows that the thermal intensity contribution from the spray is negligible in comparison with the deposit thermal intensity.

### **3.2.3 Overspray Powder**

Overspray powder consists of droplets that do not land on the deposit or bounce off to be collected in the chamber. Fig 3.10 shows the formation of a real deposit during spray forming. The bright droplet streaks indicate that many of the droplets do not land on the deposit and some of the streaks appear to show droplets bouncing off the deposit



Fig 3.10 Photograph of the formation of a deposit during spray forming.

top surface. The overspray powder was collected in the cyclone after spray forming and sieved by an automatic shaker into size fractions using: 22, 32, 45, 63, 90, 125, 180, 250, 355, 500, 710 and 1000 $\mu\text{m}$  sieves. The powder size distribution was analysed by plotting the normalised weight per unit  $\ln(\mu\text{m})$  in each size range against the natural log of the mid point of the size range and comparing the resulting distribution to a best fit log-normal distribution using SigmaPlot software (SigmaPlot, Jandel Scientific GmbH, Schimmelbuschstr. 25, D-4006 Erkrath, Germany). The log-normal distribution is described by equation 3.3:

$$f(x) = \left(\frac{k}{s}\right) \exp\left(-\frac{(\ln(x)-\ln(m))^2}{2s^2}\right) \quad (3.3)$$

where  $f$  is the best fit normalised weight per unit  $\ln(\mu\text{m})$  in each size range,  $x$  is the mid point of the sieve size range,  $m$  is the mean diameter,  $s$  is the standard deviation and  $k$  is a normalising constant.

An alternative method was also used to analyse the overspray powder by plotting the normalised weight fraction above a given sieve size against that sieve size, and comparing the resulting distribution to a best fit Rosin-Rammler distribution using SigmaPlot software. This gave another measure of the mean size, the mass median diameter ( $d_m$ ). The Rosin-Rammler distribution is described by the equation:

$$f(x) = \exp\left(-\left(\frac{x}{m}\right)^n\right) \quad (3.4)$$

where  $f(x)$  is the normalised weight fraction above a given sieve size,  $x$  is the sieve size,  $m$  is the mean diameter and  $n$  is the range. The mass median diameter is when  $f(x)=0.5$ .

### **3.3 Grain Growth of Spray Formed Deposits**

The final grain size of spray formed deposits is determined by: (i) the sizes and heat contents of droplets arriving at the deposit surface i.e. the density of solid nuclei (Bewlay and Cantor 1990, Mathur *et al* 1989); (ii) the extent of grain growth which takes place in the solid plus liquid region on the deposit top surface during times of  $\approx 1-2$  min (Annavarapu and Doherty 1993, Grant *et al* 1993C); and (iii) the extent of additional grain growth which takes place during cooling in the fully solid region below the deposit top surface.

To investigate the conditions present in (ii) samples of two commercial spray formed Ni superalloys, MAR-M-002 and UDIMET 720, were re-heated into the solid plus liquid region and into the fully solid region as determined by thermal analysis for a series of times shown in table 3.1. The grain growth occurring in this simple isothermal reheating process may differ from the actual detailed conditions on the top surface of a spray formed deposit, where the continuous arrival of partially solidified droplets, and the associated thermal and momentum fields also influence grain growth. However, the predominant situation of grain growth in the solid/liquid region is simulated reasonably well.

#### **3.3.1 Spray Forming Conditions**

Spray formed UDIMET 720 and MAR-M-002 deposits were provided by Rolls-Royce plc with nominal compositions given in table 3.2 and were spray formed using a standard OSPREY spray forming system. A 15kg charge weight was induction melted and bottom poured at 0.242kg/s melt flow rate with an overpressure ramp rate of 34.1KPa in



120s into a 1.02MPa, 153l/s nitrogen standard OSPREY scanning atomiser. The resulting atomised droplet spray was collected on a rotating corderite collector plate positioned at an angle of 35° to the spray axis and an axial distance of 0.525m from the atomiser.

| Alloy      | Heat Treatment Temperature (°C) | Solid Fraction | Heat Treatment Time (min) |
|------------|---------------------------------|----------------|---------------------------|
| MAR-M-002  | 1200                            | 1.0            | 66, 126, 246, 486         |
|            | 1300                            | 0.96           | 16, 26, 46, 96, 186       |
|            | 1315                            | 0.93           | 16, 26, 46, 96, 186       |
|            | 1330                            | 0.90           | 16, 26, 46, 96, 186       |
|            | 1343                            | 0.87           | 16, 26, 46, 96, 186       |
| UDIMET 720 | 1150                            | 1.0            | 66, 126, 246, 486         |
|            | 1180                            | 1.0            | 66, 126, 246, 486         |
|            | 1245                            | 0.98           | 16, 26, 46, 96, 186       |
|            | 1260                            | 0.95           | 16, 26, 46, 96, 186       |
|            | 1270                            | 0.90           | 16, 26, 46, 96, 186       |
|            | 1280                            | 0.84           | 16, 26, 46, 96, 186       |
|            | 1290                            | 0.78           | 16, 26, 46, 96, 186       |

Table 3.1 Heat treatment times, temperatures and corresponding solid fractions for MAR-M-002 and UDIMET 720.

|           | Ni  | Al  | Cr | Co | Mo | Ti  | W   | Hf  | Ta  | C    | B    | Zr  |
|-----------|-----|-----|----|----|----|-----|-----|-----|-----|------|------|-----|
| MAR-M-002 | Bal | 5.5 | 9  | 10 | -  | 1.5 | 10  | 1.5 | 2.5 | 0.15 | .015 | .03 |
| U720      | Bal | 2.5 | 18 | 15 | 3  | 5   | 1.5 | -   | -   | .035 | .033 | .03 |

Table 3.2 Composition of MAR-M-002 and UDIMET 720 in wt%.

### **3.3.2 Heat Treatment Conditions**

Approximately  $0.5\text{cm}^3$  samples were cut from the central region of the spray formed deposits using a Bennet's MOS abrasive wheel on a Microfine abrasive wheel cutter, sealed in quartz tube capsules which were evacuated to  $10^{-4}$ - $10^{-5}$  Torr, and then heat treated for times in the range 16-486min at temperatures in the range 1150-1343°C as shown in table 3.1. After heat treatment the samples were quenched in water. The heat treatment times include the warm up time in the furnace which was  $\approx 6$ mins

### **3.3.3 Determination of Solid Fraction**

The heat treatment temperatures (T) and corresponding solid fractions (f) for UDIMET 720 were determined at Rolls-Royce by differential scanning calorimetry (DSC). Similar data for MAR-M-002 was taken from Willis *et al* (1987). The variation of f with T is shown in fig 3.11 for MAR-M-002 and UDIMET 720. The data for UDIMET 720 was from a spray formed sample and for MAR-M-002 from an investment cast sample i.e. in both cases this is likely to be non-equilibrium data, as there was no equilibrium data available for these alloys. The best fit Scheil equation (Flemings 1974) for UDIMET 720 had a k value of 0.85 as shown in fig 3.12. The remaining solid fraction at the solidus temperature was 0.975 and this was assumed to completely solidify within 3°C of the solidus temperature.

## **3.4 Microstructural Examination**

Examination of the spray formed deposits and the heat treated spray formed deposits were performed using the following techniques.

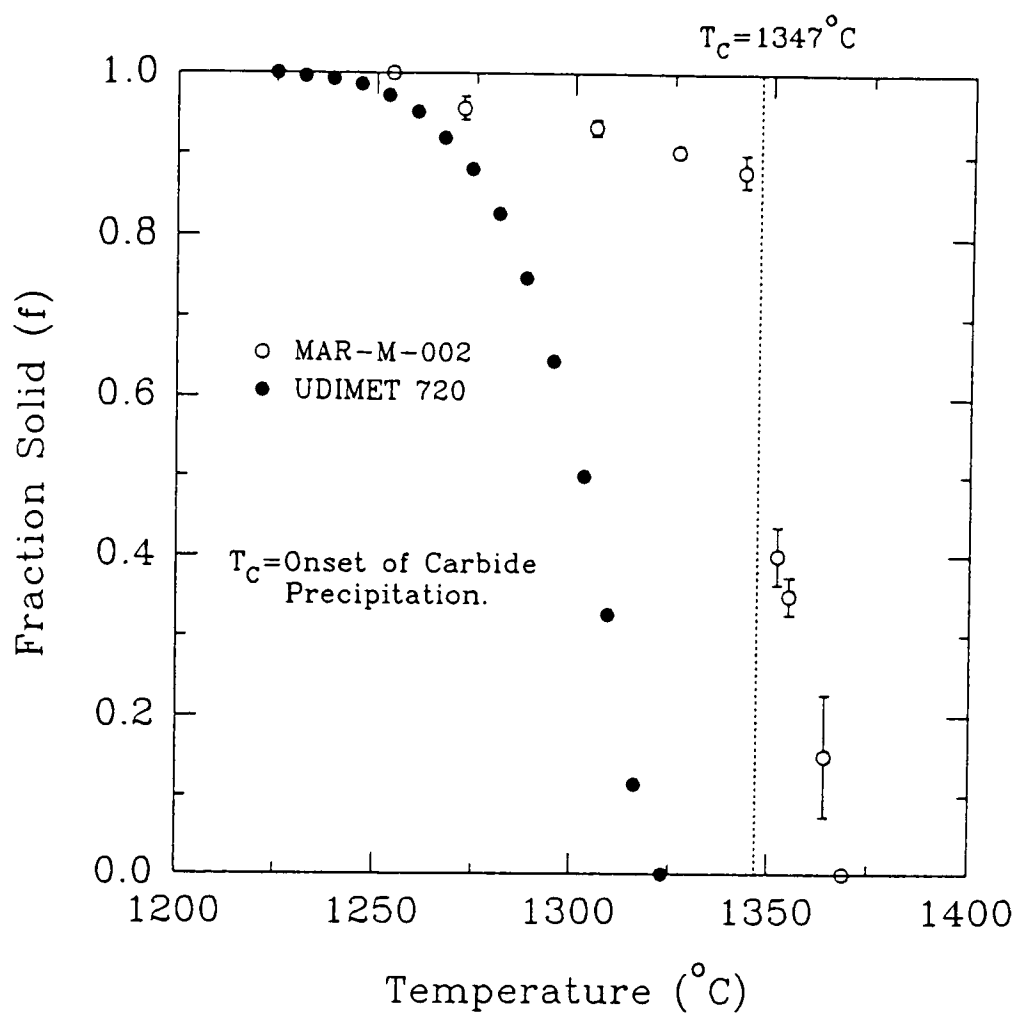


Fig 3.11 Variation of solid fraction ( $f$ ) with temperature ( $T$ ) for (i) MAR-M-002 and (ii) UDIMET 720

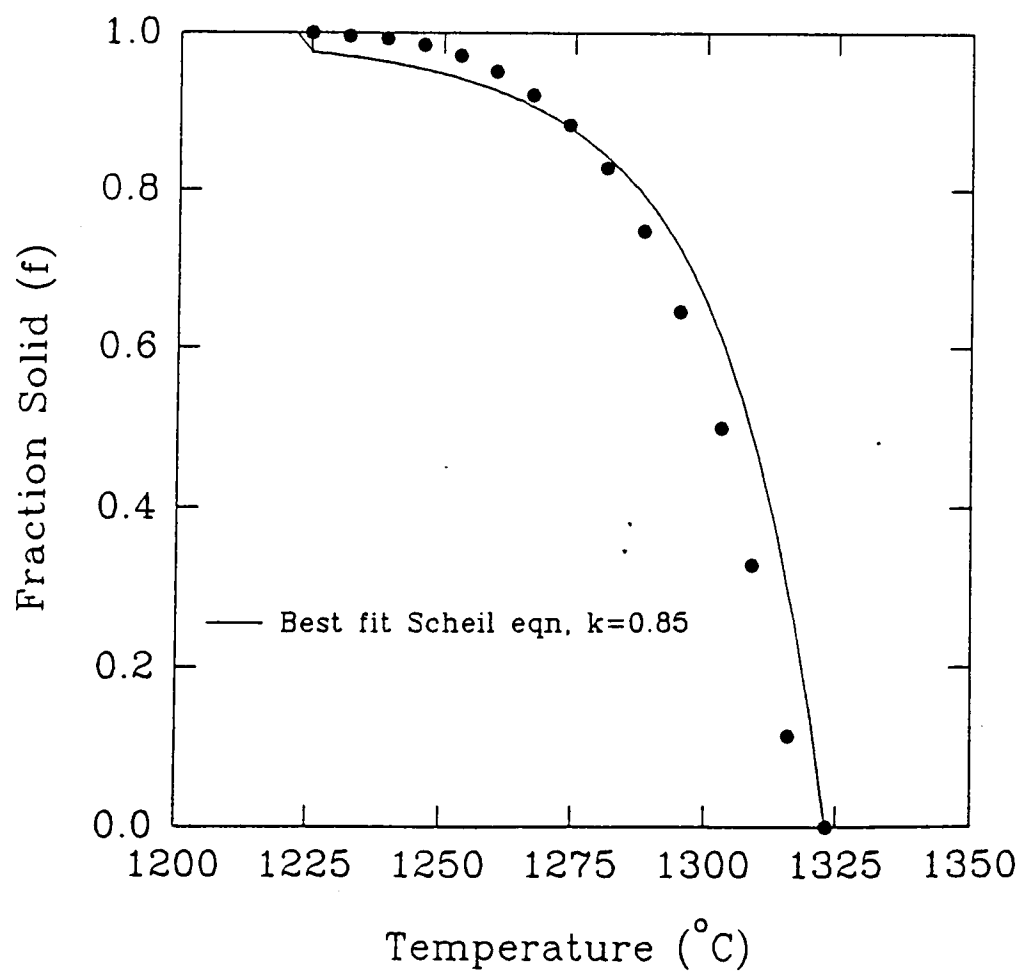


Fig 3.12 Variation of solid fraction ( $f$ ) with temperature ( $T$ ) for UDIMET 720 showing the best fit Scheil equation.

**3.4.1 Optical Microscopy**

Small central sections (approx. 15mm x 15mm x deposit height) of the spray formed deposits were cut using a Struers 81 EXO abrasive wheel on a Buehler Powermet 1 abrasive wheel cutter. All samples were mounted in conductive bakelite and metallographically prepared on a Buehler Metaserv Motopol 8 automatic polisher using the routine detailed in table 3.3. In between each stage of the routine, the samples were thoroughly cleaned with detergent to ensure no cross contamination of the polishing wheels and diamond suspensions occurred.

| Surface          | Lubricant     | Abrasive Type/Size                 | Time (min)  | Force Per Sample | Speed (rpm) | Relative Motion |
|------------------|---------------|------------------------------------|-------------|------------------|-------------|-----------------|
| SiC Paper        | Water         | 400 grit                           | Until Plane | 5lbs             | 150         | Comp.           |
| Metlap® 4 Platen |               | 6µm Water Based Diamond Suspension | 5           | 5lbs             | 120         | Comp.           |
| Textmet® Cloth   | Metadi® fluid | 3µm Water Based Diamond Suspension | 6           | 5lbs             | 250         | Comp.           |
| Microcloth®      |               | Colloidal Silica (OPS)             | 2           | 3lbs             | 100         | Contra.         |
| Microcloth®      | Water         | -                                  | 2           | 3lbs             | 100         | Contra.         |

Table 3.3 Metallographic Preparation Routine For MAR-M-002 and UDIMET 720 using the Buehler Metaserv Motopol 8 automatic polisher. (Comp=complimentary rotation of sample to wheel, Contra=opposite rotation of sample to wheel.)

The following etchants were used to reveal different aspects of the microstructure.

Etchant A - 5% Br in Methanol. To reveal the grain structure in UDIMET 720, although contamination of the samples sometimes occurs. Immediately after final polishing samples were heated for 30-60s using a warm air dryer and then etched for 3-6s.

Etchant B - Marbles Reagent (10g CuSO<sub>4</sub>, 50cm<sup>3</sup> HCl, 50cm<sup>3</sup> H<sub>2</sub>O (distilled)). To reveal the grain structure in MAR-M-002 using a swab.

Etchant C - 10% Orthophosphoric Acid. To attack the  $\gamma$  phase in UDIMET 720 and MAR-M-002. This is an electro-etch using the following conditions: 3-5V D.C. at room temperature for 5-10s where the bakelite mount is the cathode and the sample the anode. Etching of the  $\gamma$  phase reveals grain boundaries and carbide particles.

A Reichert microscope was used for optical examination of the samples. Digital optical images were taken on a Kontron IBAS image analysis system, transferred to a Silicon Graphics workstation and printed on a Hewlet Packard 4MPlus 600dpi laser printer.

### **3.4.2 Grain Size**

The grain size in the central region of spray formed deposits and the average grain size of the heat treated spray formed samples was determined by a linear intercept technique using IBAS image analysis software. The grain size  $\lambda$  is given by:

$$\lambda = L/N \quad (3.5)$$

where L is the length of a line chosen to be parallel to the substrate surface and N is the number of grain boundaries which cross that line.

### **3.4.3 Porosity**

A Kontron IBAS image analysis system using a Reichert optical microscope was used to determine the through thickness variation in area fraction porosity for spray formed deposits. The grey level contrast for each sample was set manually and an average value of area fraction porosity was determined for a series of heights above the substrate.

### **3.4.4 Electron Probe Microanalysis (EPMA)**

EPMA was used to give semi-quantitative compositional information about small precipitate particles in heat treated MAR-M-002 samples prepared in the same way as for optical microscopy, using a CAMECA CAMEBAX SEM microprobe operating at an accelerating voltage of 20kV. The following elements were analysed: Al, Co, Cr, Hf, Ni, Ti, and W; and C was calculated by difference. Table 3.4 shows the x-ray lines and crystal spectrometers used for the semi-quantitative analysis of MAR-M-002.

The ratio of the sample x-ray intensity of a given element peak, above background, in the sample to that of the pure element is corrected to give the corrected elemental wt% by a ZAF programme which takes into account the atomic weight (Z), absorption (A) and fluorescence (F) (Wells 1974). Although the incident electron beam can be focused to a point  $1\mu\text{m}^2$ , inside the material the characteristic x-rays are generated from a pear drop shaped volume greater than  $1\mu\text{m}^2$ , therefore point measurements ( $1\mu\text{m}^2$ ) of the precipitate particles identified optically were obtained.

EPMA analysis is only semi-quantitative due to assumptions in the ZAF correction that the excited volume and x-ray exit path are of the same composition, this gives a minimum error of 2%. However, for the analysis of a large number of elements at low

concentrations in a single system the accuracy is reduced to 10% (Wells 1974).

| Element | Crystal                  | x-ray line |
|---------|--------------------------|------------|
| Al      | TAP                      | K alpha    |
| Co      | LIF                      | K alpha    |
| Cr      | LIF                      | K alpha    |
| Hf      | LIF                      | K alpha    |
| Ni      | LIF                      | L alpha    |
| Ti      | LIF                      | L alpha    |
| W       | LIF                      | L alpha    |
| C       | calculated by difference |            |

Table 3.4 Crystal spectrometers and x-ray lines used for semi-quantitative EPMA analysis of heat treated MAR-M-002 and UDIMET 720 samples.

### **3.5 Compositional Analysis**

Samples from as supplied UDIMET 720 and 3 typical spray formed deposits were sent to Rolls-Royce plc for a compositional check on the elements present and to see if there was any macrosegregation in the spray formed deposits using a Philips PW 1400 x-ray fluorescence spectrometer. Approximately 55g samples were button melted and prepared to a 90µm diamond finish prior to analysis. The elements analysed were Ni, Al, Co, Cr, Mo, Ti and W, the measurement error for elements present in concentrations up to 25wt% was  $\approx 0.05\text{wt}\%$ . Fig 3.13 shows a schematic cross sectional diagram of spray formed deposits from: (a) run 20, (b) run 30 and (c) run 34 indicating the position of the samples used for compositional analysis. As shown in fig 3.13 samples from the central and edge regions were analysed (labelled A and B respectively). The thickness of the samples was  $\approx 13\text{mm}$ .

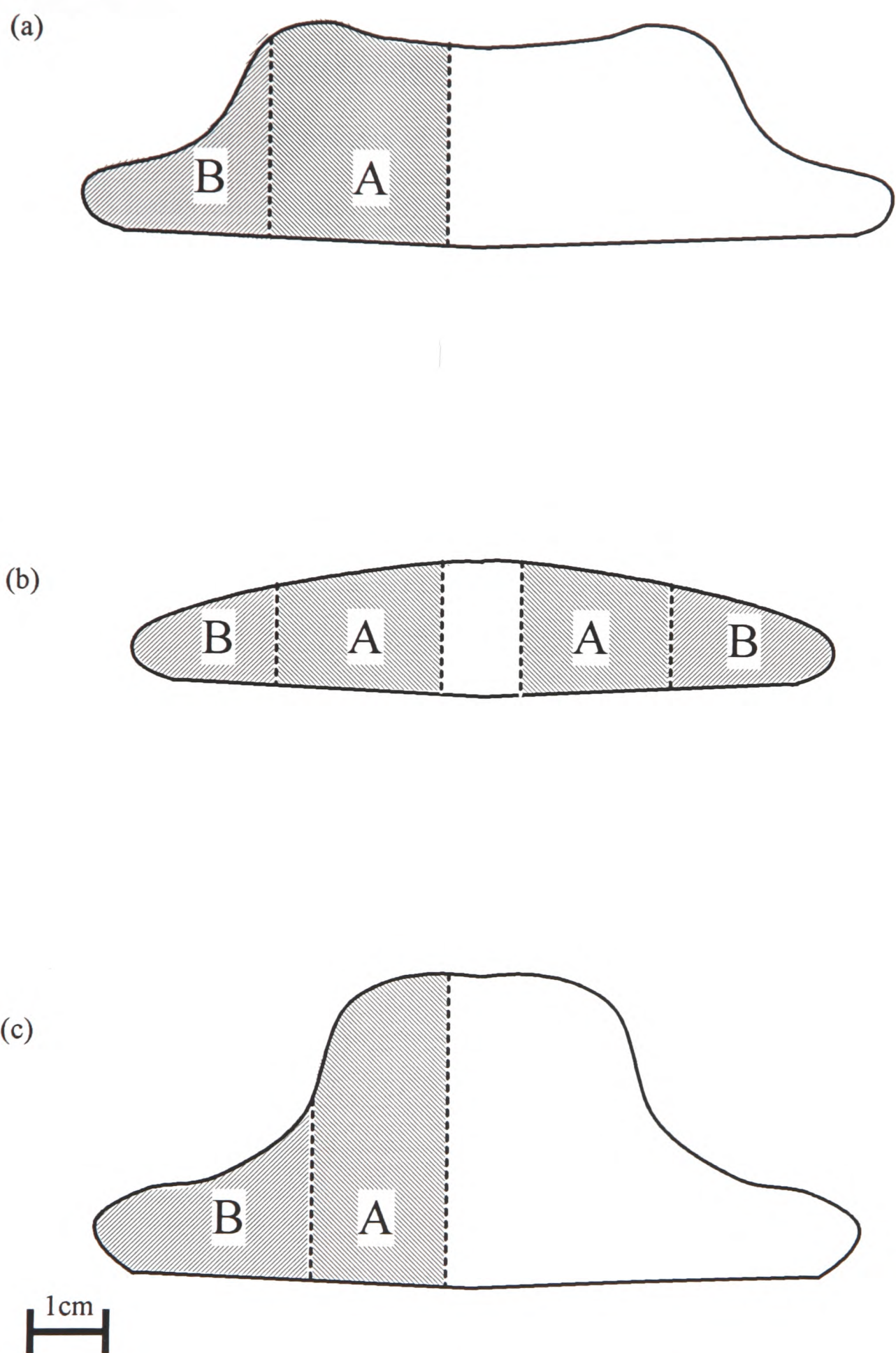


Fig 3.13 Schematic cross sectional diagram of spray formed deposits from: (a) run 20, (b) run 30 and (c) run 34 indicating the positions where approx 13mm thick samples were taken from for compositional analysis. A=central region and B=edge region.



### **3.5.1 Gas Analysis**

Samples from as supplied UDIMET 720 and 3 typical spray formed deposits were sent to Rolls-Royce plc for analysis of the O and N gas content using a Leco TC-436 inert gas (Ar) fusion determinator. The measurement error for gas contents up to 50ppm was  $\approx \pm 4$ ppm. Fig 3.14 shows a schematic cross sectional diagram of spray formed deposits from: (a) run 19, (b) run 30 and (c) run 35 indicating the position of the samples used for gas analysis. As shown in fig 3.14 samples from 3 different radial positions ( $r \approx 0.5$ cm, 3cm and 5.5cm respectively) were analysed and the average O and N content in the deposit calculated.

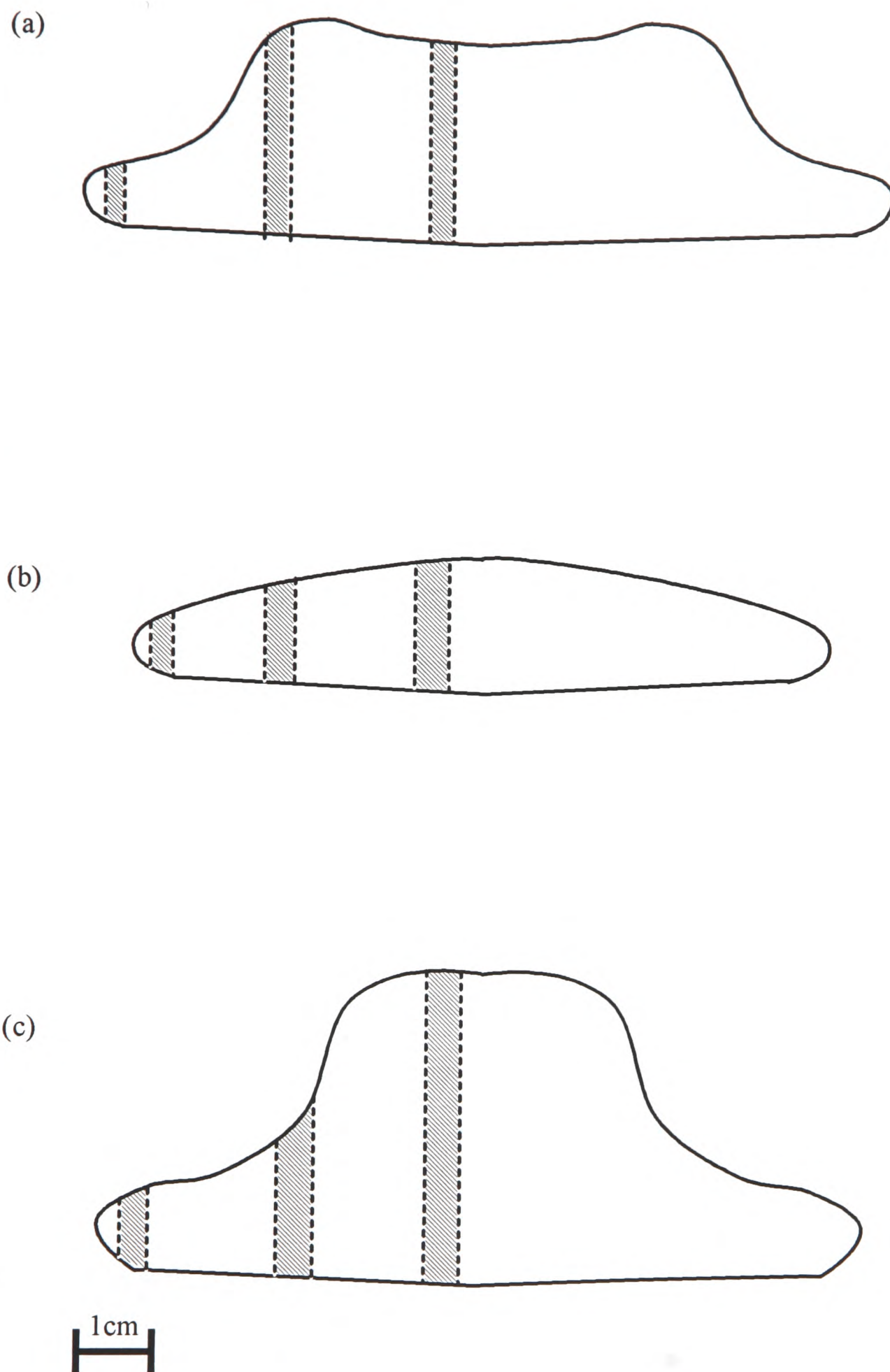


Fig 3.14 Schematic cross sectional diagrams of spray formed deposits from: (a) run 19, (b) run 30 and (c) run 35 indicating the positions where approx 4mm thick samples were taken from for gas analysis.

## **CHAPTER 4 - GRAIN GROWTH OF SPRAY FORMED Ni SUPERALLOYS**

Numerical modelling calculations (Mathur *et al* 1989, Grant *et al* 1993A and 1993B) and deposit top surface temperature measurements (chapter 5, Grant *et al* 1989) both show that a mixture of fully solid, partially solid and fully liquid droplets arrive at the deposit top surface to create a top surface solid/liquid region ( $>0.8$  fraction solid) for short periods of time ( $\approx 1$ -2mins). Qualitative models of microstructural evolution (Bewlay & Cantor 1990, Mathur *et al* 1989) propose that fully solid droplets seed the semi-solid top surface layer, giving a high nucleation density which leads to the observed equiaxed, fine grain structures. The final grain size is determined by: (i) the size, heat content and rate of arrival of droplets at the deposit top surface i.e. the density of solid nuclei controlled by heat transfer in the droplet spray and subsequent to deposition by the deposit macroscopic heat flow; (ii) the extent of grain growth which takes place in the solid/liquid region on the deposit top surface; and (iii) the extent of additional grain growth which takes place during cooling in the fully solid region below the deposit top surface.

This chapter describes the results of experiments which simulate conditions corresponding to (ii) and (iii), i.e. grain growth in the fully solid and solid/liquid region by reheating samples of spray formed Ni superalloys.

### **4.1 Grain Growth of MAR-M-002**

Historically MAR-M-002 and derivatives have been used by Rolls-Royce as a DS (directionally solidified) turbine blade alloy due to their high temperature capability and high volume fraction of  $\gamma'$  precipitates (Willis *et al* 1987).

Fig 4.1 shows a typical MAR-M-002 as-sprayed microstructure which consisted of homogeneous, equiaxed grains, with an average size of  $10\mu\text{m}$ . Figs 4.2(a) and 4.2(b) show etched microstructures of MAR-M-002 heat treated for 186mins at  $1300^\circ\text{C}$  ( $f\approx 0.96$ ) and  $1343^\circ\text{C}$  ( $f\approx 0.87$ ) respectively. At  $1300^\circ\text{C}$  ( $f\approx 0.96$ ), there was a small amount of quenched grain boundary liquid present during heat treatment, whereas at  $1343^\circ\text{C}$  ( $f\approx 0.87$ ) a "dendritic necklace" of quenched liquid surrounding the grain boundaries indicated that more grain boundary liquid was present during heat treatment. Annavarapu and Doherty (1995), from similar studies of spray formed alloys heat treated in the solid/liquid region, attributed this necklace effect, to the quench not being rapid enough to prevent a breakdown of the solid liquid interface into small grain boundary dendrites. Fig 4.2(c) shows a typical unetched microstructure of MAR-M-002 heat treated for 186mins at an intermediate temperature of  $1330^\circ\text{C}$  ( $f\approx 0.90$ ) showing segregation of precipitate particles to grain boundaries.

Fig 4.3 shows the variation in MAR-M-002 grain size at all heat treatment temperatures and times given in table 3.1. Grain growth in the fully solid region at  $1200^\circ\text{C}$  ( $f=1.0$ ) was negligible for times up to 486mins. In the solid/liquid region, however, grain growth was in all cases rapid initially ( $t<10^3\text{s}$ ). The grain size then either approached approximate steady state values (at  $1300^\circ\text{C}$ ,  $f\approx 0.96$  and  $1315^\circ\text{C}$ ,  $f\approx 0.93$ ) or continued to increase, but at a slower rate (at  $1330^\circ\text{C}$ ,  $f\approx 0.90$  and  $1343^\circ\text{C}$ ,  $f\approx 0.87$ ). For example, at  $1300^\circ\text{C}$  ( $f\approx 0.96$ ) and  $1343^\circ\text{C}$  ( $f\approx 0.82$ ), the average grain size increased from  $10\mu\text{m}$  to  $\approx 31\mu\text{m}$  and  $\approx 75\mu\text{m}$  respectively after 46min, and then increased to  $\approx 34\mu\text{m}$  and  $\approx 107\mu\text{m}$  respectively after 186min.

Tables 4.1-4.3 show the results of EPMA point analyses of the matrix, grain



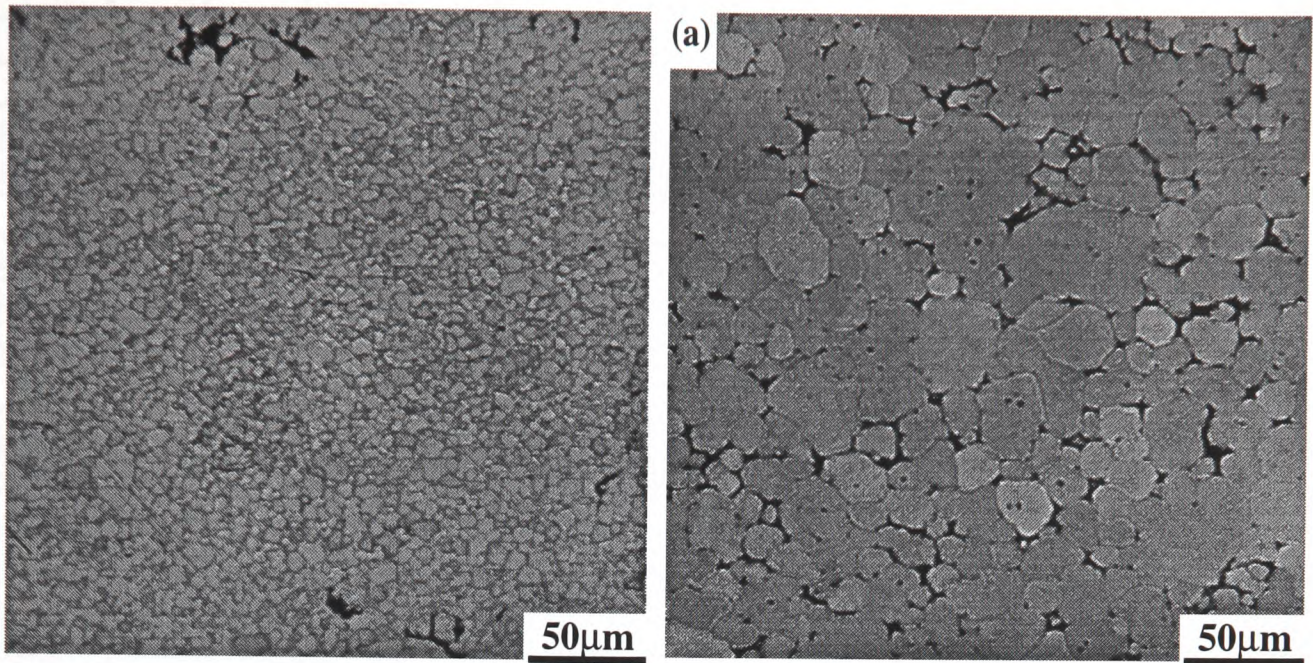


Fig 4.1 As sprayed MAR-M-002.  
Etchant Marbles reagent.

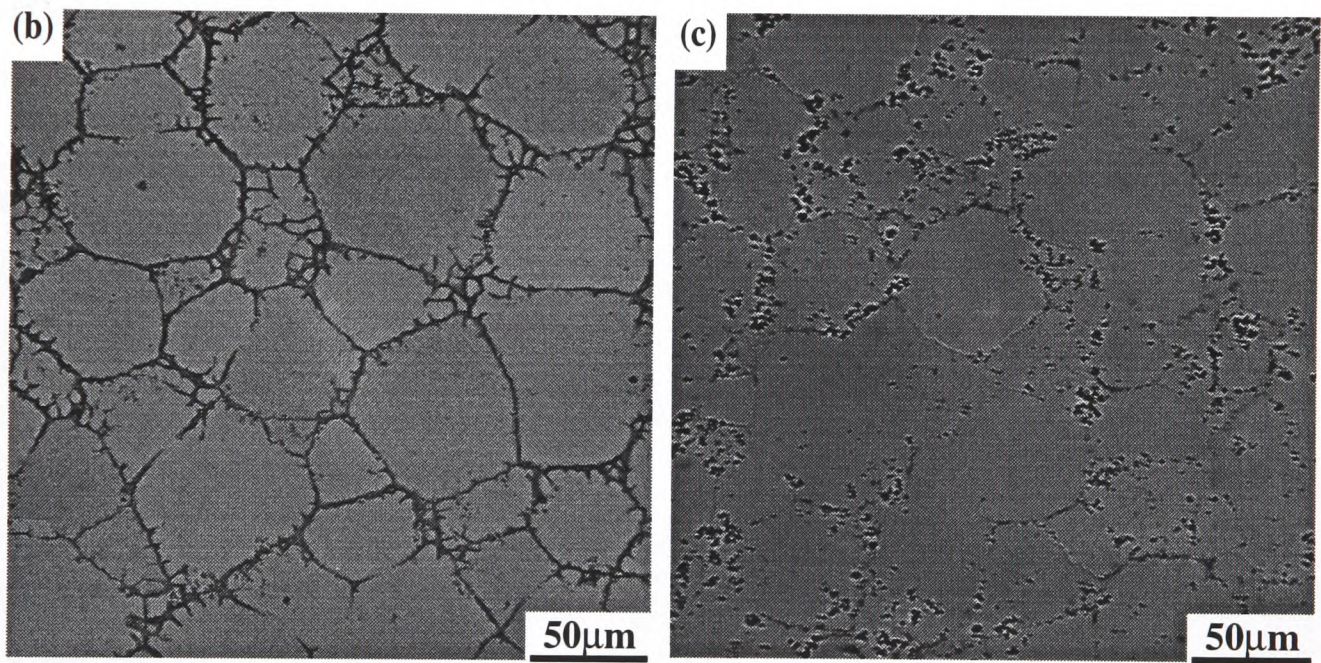


Fig 4.2 MAR-M-002 heat treated for 186mins at (a) 1300C, (b) 1343C and (c) 1330C. (a) and (b) etchant Marbles reagent, (c) unetched.

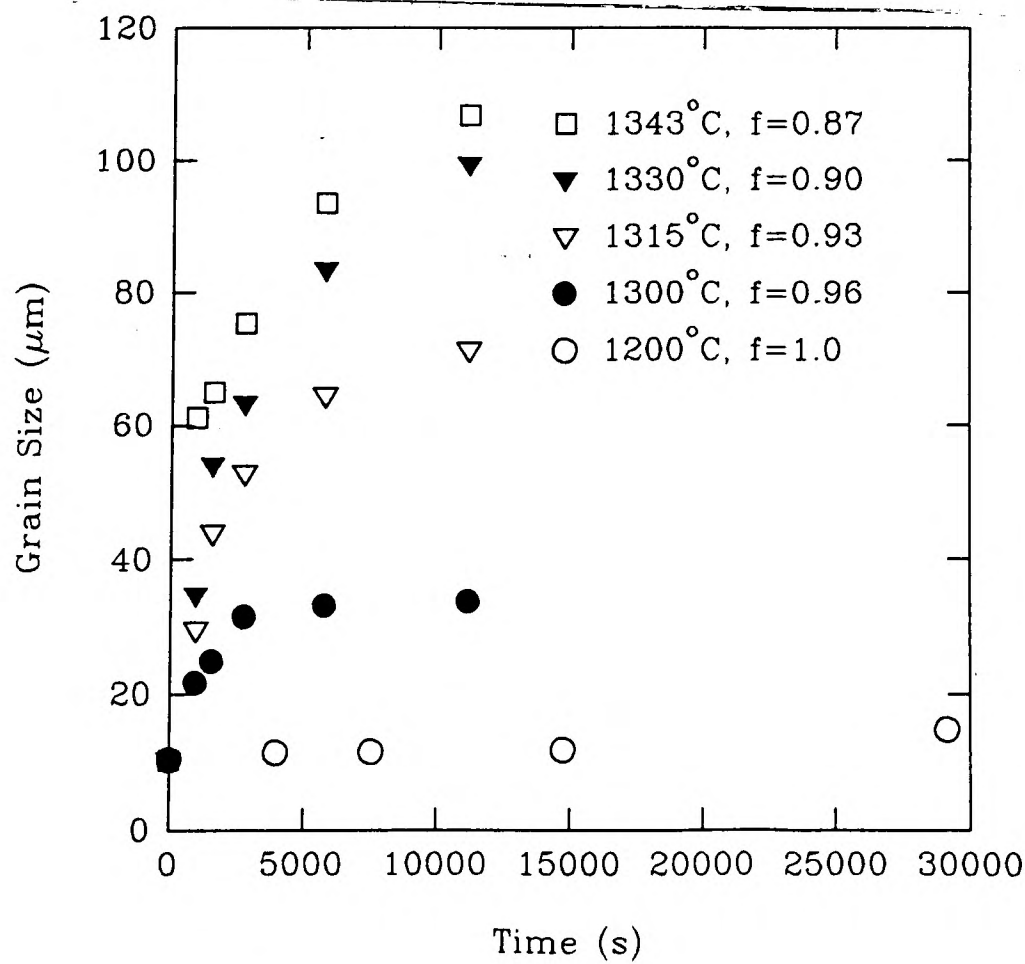


Fig 4.3 Grain size against time for MAR-M-002.



|               | Al   | Ti   | Cr   | Co   | Ni    | Hf   | Ta   | W    | C    | B            | Zr   | Proposed Formula |
|---------------|------|------|------|------|-------|------|------|------|------|--------------|------|------------------|
| Nominal Comp. | 5.5  | 1.5  | 9    | 10   | 59.8  | 1.5  | 2.5  | 10   | 0.15 | 0.015        | 0.03 |                  |
|               |      |      |      |      |       |      |      |      |      |              |      |                  |
| Matrix        | 5.99 | 2.71 | 9.63 | 9.15 | 64.07 | 0.95 | 3.09 | 3.11 | 1.3  | not analysed |      |                  |
| Matrix        | 5.82 | 2.39 | 9.48 | 9.07 | 63.69 | 0.56 | 2.81 | 4.77 | 1.4  | "            | "    |                  |
|               |      |      |      |      |       |      |      |      |      | "            | "    |                  |
| G.B Liquid    | 4.14 | 2.93 | 11.2 | 7.25 | 48.33 | 22.9 | 1.65 | 0.83 | 0.72 | "            | "    |                  |
| G.B Liquid    | 3.7  | 2.78 | 10.4 | 7.33 | 46.43 | 26.1 | 1.46 | 0.62 | 1.19 | "            | "    |                  |
| G.B Liquid    | 4.21 | 2.82 | 11.4 | 7.37 | 46.99 | 24.2 | 1.38 | 1.05 | 0.58 | "            | "    |                  |
|               |      |      |      |      |       |      |      |      |      | "            | "    |                  |
| G.B Particle  | 0.58 | 2.33 | 1.93 | 1.93 | 10.05 | 73   | 2.23 | 0.54 | 7.47 | "            | "    | HfC1.5           |
| G.B Particle  | 1.32 | 2.39 | 2.54 | 2.74 | 15.15 | 63.9 | 2.37 | 1.43 | 8.13 | "            | "    | HfC1.9           |
| G.B Particle  | 1.24 | 6.54 | 5.05 | 3.13 | 18.69 | 37.1 | 14.7 | 8.6  | 5.04 | "            | "    | HfC2             |
| G.B Particle  | 0.86 | 3.5  | 3.11 | 3.06 | 18.63 | 58.9 | 1.6  | 1.74 | 8.57 | "            | "    | HfC2.2           |
| G.B Particle  | 0.33 | 21.2 | 1.78 | 1.35 | 7.98  | 11.8 | 40   | 12.1 | 3.5  | "            | "    | (Ti,Ta)2.3C      |
| G.B Particle  | 0.42 | 11.4 | 2.93 | 1.66 | 9.02  | 21.2 | 36.2 | 12.4 | 4.83 | "            | "    | (Ti,Ta)1.1C      |

Table 4.1 EPMA point analysis of matrix, grain boundary liquid and grain boundary particles for MAR-M-002 heat treated at 1343C for 186mins. (Carbon calculated by difference)

|               | Al   | Ti   | Cr   | Co   | Ni    | Hf   | Ta   | W    | C    | B            | Zr   | Proposed Formula |
|---------------|------|------|------|------|-------|------|------|------|------|--------------|------|------------------|
| Nominal Comp. | 5.5  | 1.5  | 9    | 10   | 59.8  | 1.5  | 2.5  | 10   | 0.15 | 0.015        | 0.03 |                  |
|               |      |      |      |      |       |      |      |      |      |              |      |                  |
| Matrix        | 4.53 | 0.98 | 9.13 | 10.7 | 62.75 | 0    | 0.94 | 8.79 | 2.08 | not analysed |      |                  |
|               |      |      |      |      |       |      |      |      |      | "            | "    |                  |
| G.B Liquid    | 2.27 | 2.74 | 9.19 | 8.2  | 50.33 | 23.1 | 0.61 | 0.07 | 3.41 | "            | "    |                  |
| G.B Liquid    | 2    | 2.23 | 19.2 | 7.12 | 41.51 | 18.6 | 0.61 | 5.13 | 3    | "            | "    |                  |
|               |      |      |      |      |       |      |      |      |      | "            | "    |                  |
| G.B Particle  | 1.19 | 2.41 | 3.56 | 3.93 | 21.21 | 47.4 | 10.6 | 4.09 | 5.55 | "            | "    | HfC1.8           |
| G.B Particle  | 0.4  | 16.3 | 1.87 | 1.77 | 9.58  | 5.59 | 47.4 | 11.8 | 5.23 | "            | "    | (Ti,Ta)1.4C      |
| G.B Particle  | 0.75 | 15.3 | 2.55 | 2.1  | 12.99 | 9.7  | 41.8 | 9.15 | 5.56 | "            | "    | (Ti,Ta)1.2C      |
| G.B Particle  | 0.3  | 16.9 | 1.87 | 1.56 | 8.88  | 6.74 | 50.6 | 8.12 | 4.96 | "            | "    | (Ti,Ta)1.5C      |
| G.B Particle  | 0.18 | 18.1 | 1.14 | 1.32 | 6.09  | 5.79 | 52.9 | 9.6  | 4.82 | "            | "    | (Ti,Ta)1.6C      |
| G.B Particle  | 1.21 | 13.1 | 3.51 | 3.8  | 21.39 | 5.03 | 38.4 | 7.15 | 6.37 | "            | "    | (Ti,Ta)0.9C      |
| G.B Particle  | 0.93 | 13.9 | 3.56 | 4.25 | 23.14 | 6.58 | 36.1 | 7.41 | 4.22 | "            | "    | (Ti,Ta)1.4C      |

Table 4.2 EPMA point analysis of matrix, grain boundary liquid and grain boundary particles for MAR-M-002 heat treated at 1330C for 186mins. (Carbon calculated by difference)

|               | Al   | Ti   | Cr   | Co   | Ni    | Hf   | Ta   | W    | C    | B            | Zr   | Proposed Formula |
|---------------|------|------|------|------|-------|------|------|------|------|--------------|------|------------------|
| Nominal Comp. | 5.5  | 1.5  | 9    | 10   | 59.8  | 1.5  | 2.5  | 10   | 0.15 | 0.015        | 0.03 |                  |
|               |      |      |      |      |       |      |      |      |      |              |      |                  |
| Matrix        | 5.03 | 1.81 | 9.8  | 9.98 | 61.89 | 0.38 | 1.8  | 8    | 1.26 | not analysed |      |                  |
|               |      |      |      |      |       |      |      |      |      | "            | "    |                  |
| G.B Liquid    | 2.55 | 3.15 | 14.5 | 8.62 | 51.54 | 13.7 | 0.57 | 1.46 | 3.71 | "            | "    |                  |
| G.B Liquid    | 2.61 | 3    | 12.1 | 8.67 | 52.8  | 14   | 0.52 | 1.25 | 4.77 | "            | "    |                  |
|               |      |      |      |      |       |      |      |      |      |              |      |                  |
| G.B Particle  | 0.43 | 16.8 | 1.27 | 1.34 | 7.29  | 23.6 | 39.1 | 5.05 | 5.13 | "            | "    | (Ti,Ta)1.4C      |
| G.B Particle  | 0.55 | 15.4 | 2.25 | 2.01 | 11.95 | 6.81 | 47.8 | 7.88 | 5.18 | "            | "    | (Ti,Ta)C         |
| G.B Particle  | 0.99 | 13.3 | 5.7  | 4.28 | 29.34 | 10.3 | 24   | 6.44 | 5.64 | "            | "    | (Ti,Ta)0.9C      |
| G.B Particle  | 0.88 | 14.5 | 3.84 | 4.19 | 29.15 | 4.59 | 30   | 7.24 | 5.63 | "            | "    | (Ti,Ta)C         |
| G.B Particle  | 0.16 | 17.8 | 1.3  | 1.37 | 6.91  | 7.86 | 53.9 | 6.97 | 3.8  | "            | "    | (Ti,Ta)2.1C      |
| G.B Particle  | 0.07 | 17.8 | 0.94 | 1.08 | 5.54  | 9.97 | 54   | 6.66 | 4.01 | "            | "    | (Ti,Ta)2C        |

Table 4.3 EPMA point analysis of matrix, grain boundary liquid and grain boundary particles for MAR-M-002 heat treated at 1315C for 186mins. (Carbon calculated by difference)

boundary liquid and segregated grain boundary particles at 1343°C, 1330°C and 1315°C respectively. At all temperatures the matrix compositions were approximately the same as the nominal compositions, the quenched grain boundary liquid was enriched greatly in Hf, enriched slightly in Ti and Cr and depleted in Ni, W, Ta, Co and Al. The particles present in all the samples analysed were probably carbides. C, B and Zr were not analysed and the composition difference was assumed to be all C, since the amount of B and Zr present was very small. The proposed formula of the carbides present is also shown in tables 4.1-4.3. The majority of the particles analysed were of the formula  $(\text{Ti,Ta})_x\text{C}$ , where  $x$  varied from 0.8-2.1 i.e. they were probably MC type carbides. A number of particles rich in Hf and C were observed, especially for MAR-M-002 heat treated at 1343°C for 186mins, these were probably of the type  $\text{HfC}_2$ .

## **4.2 Grain Growth of UDIMET 720**

UDIMET 720 was originally used as a turbine blade alloy in land based gas turbine engines and has subsequently been developed for jet engine disk applications (Sczerzenie and Maurer 1984). UDIMET 720 has a  $\gamma'$  solvus temperature below the equilibrium solidus temperature and therefore a lower temperature capability than alloys like MAR-M-002.

Fig 4.4 shows a typical as sprayed microstructure of UDIMET 720. As sprayed UDIMET 720 consisted of homogeneous equiaxed grains with an average grain size of 37 $\mu\text{m}$ . Figs 4.5(a)-(e) show electro-etched microstructures of UDIMET 720 (a) heat treated for 486mins at 1150°C ( $f \approx 1.0$ ) and for 186mins at (b) 1245°C ( $f \approx 0.98$ ), (c) 1270°C ( $f \approx 0.90$ ), (d) 1290°C ( $f \approx 0.78$ ) and (e) 1280°C ( $f \approx 0.84$ ). For UDIMET 720 heat treated at



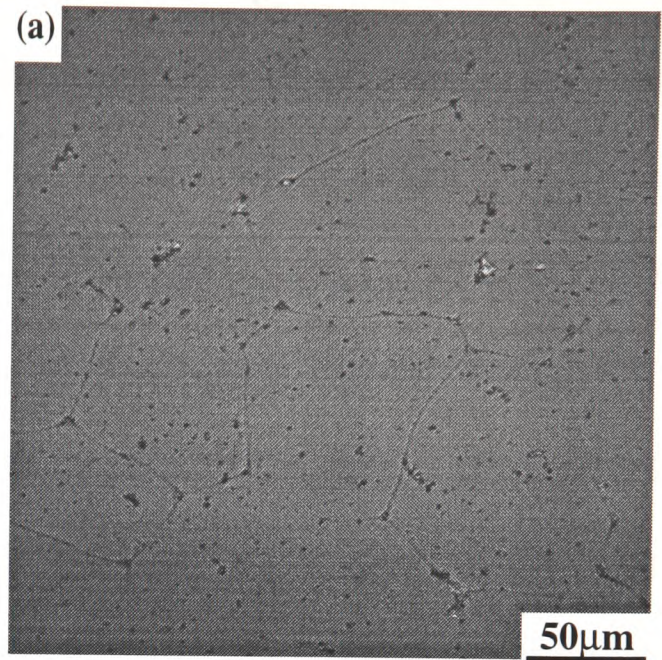
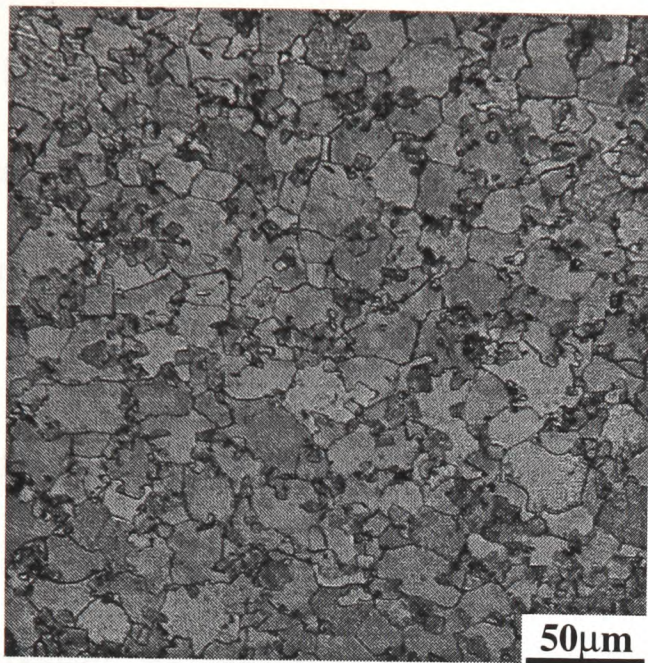


Fig 4.4 As sprayed UDIMET 720.  
Etchant 10% orthophosphoric acid.

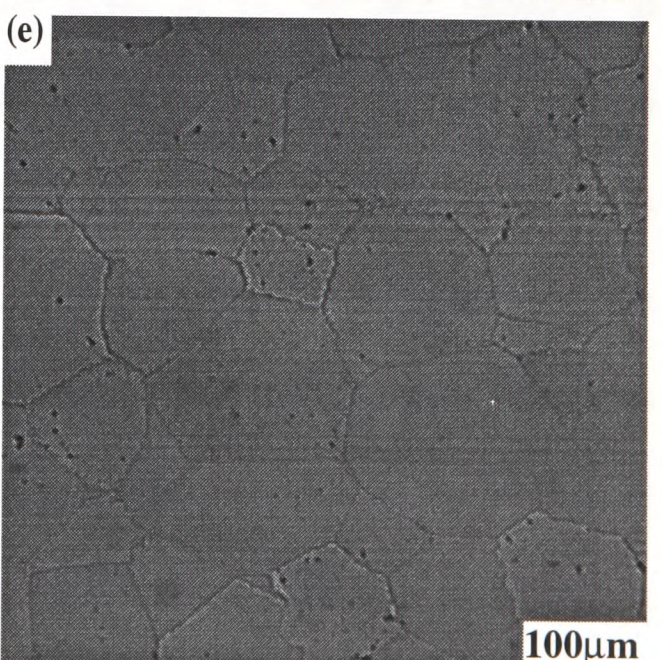
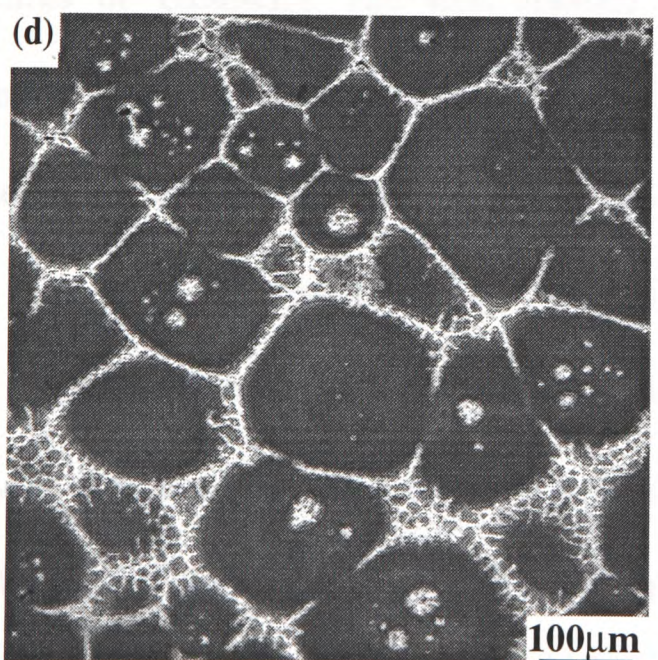
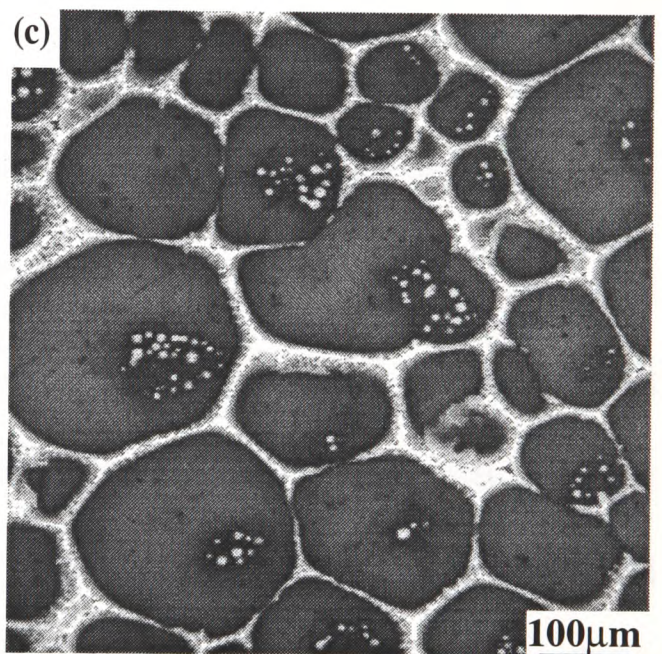
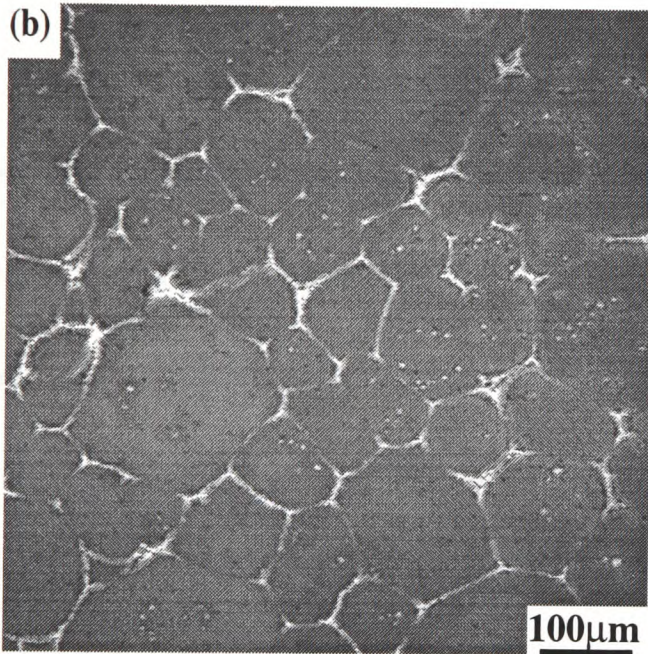


Fig 4.5 UDIMET 720 heat treated for (a) 486mins at 1180C and for 186mins at (b) 1245C, (c) 1270C, (d) 1290C and (e) 1280C. (a)–(d) etchant 10% orthophosphoric acid, (e) unetched.,



1180°C ( $f_s \approx 1.0$ ) for 486mins in fig 4.5(a), there was an extensive distribution of particles (probably carbides) at grain boundaries, triple points and also within grains. There was no evidence of any grain boundary liquid present during heat treatment. For UDIMET 720 heat treated for 186mins at temperatures of 1245°C ( $f_s \approx 0.98$ ), 1270°C ( $f_s \approx 0.90$ ) and 1290°C ( $f_s \approx 0.78$ ) in figs 4.5(b)-(d) respectively. There was an increase in the amount of quenched liquid at the grain boundaries and the size of grain boundary triple points as the solid fraction decreased. At 1290°C ( $f_s \approx 0.78$ ) a "dendritic necklace" of quenched liquid, also reported by Annavarapu and Doherty (1995) for Al alloys, surrounded the grain boundaries. Large isolated liquid droplets within the grains were also present at all temperatures, which Annavarapu and Doherty (1995) suggested were left behind when the liquid wetted grain boundaries moved rapidly and left behind isolated liquid droplets. Fig 4.5(e) is a typical unetched microstructure of UDIMET 720 heat treated in the solid/liquid region (186mins at 1280°C ( $f_s \approx 0.84$ )) and shows that unlike MAR-M-002 relatively few particles were present at the grain boundaries. Figs 4.6(a)-(f) show electro-etched microstructures of UDIMET 720 heat treated for times of 16, 46 and 186mins at temperatures of 1245°C ( $f_s \approx 0.98$ ) and 1290°C ( $f_s \approx 0.78$ ) respectively. They show an increase in grain size and a reduction in the number of isolated liquid droplets within grains with increasing time at both temperatures. All the microstructures heat treated in the solid/liquid region exhibited low curvature grain boundaries, and at higher solid fractions the majority of liquid was present at grain boundary triple points because liquid drained from grain boundary edges into triple points during heat treatment because of the higher curvature of the triple points, as previously shown by Annavarapu and Doherty (1995).

Fig 4.7 shows the variation in UDIMET 720 grain size at all heat treatment



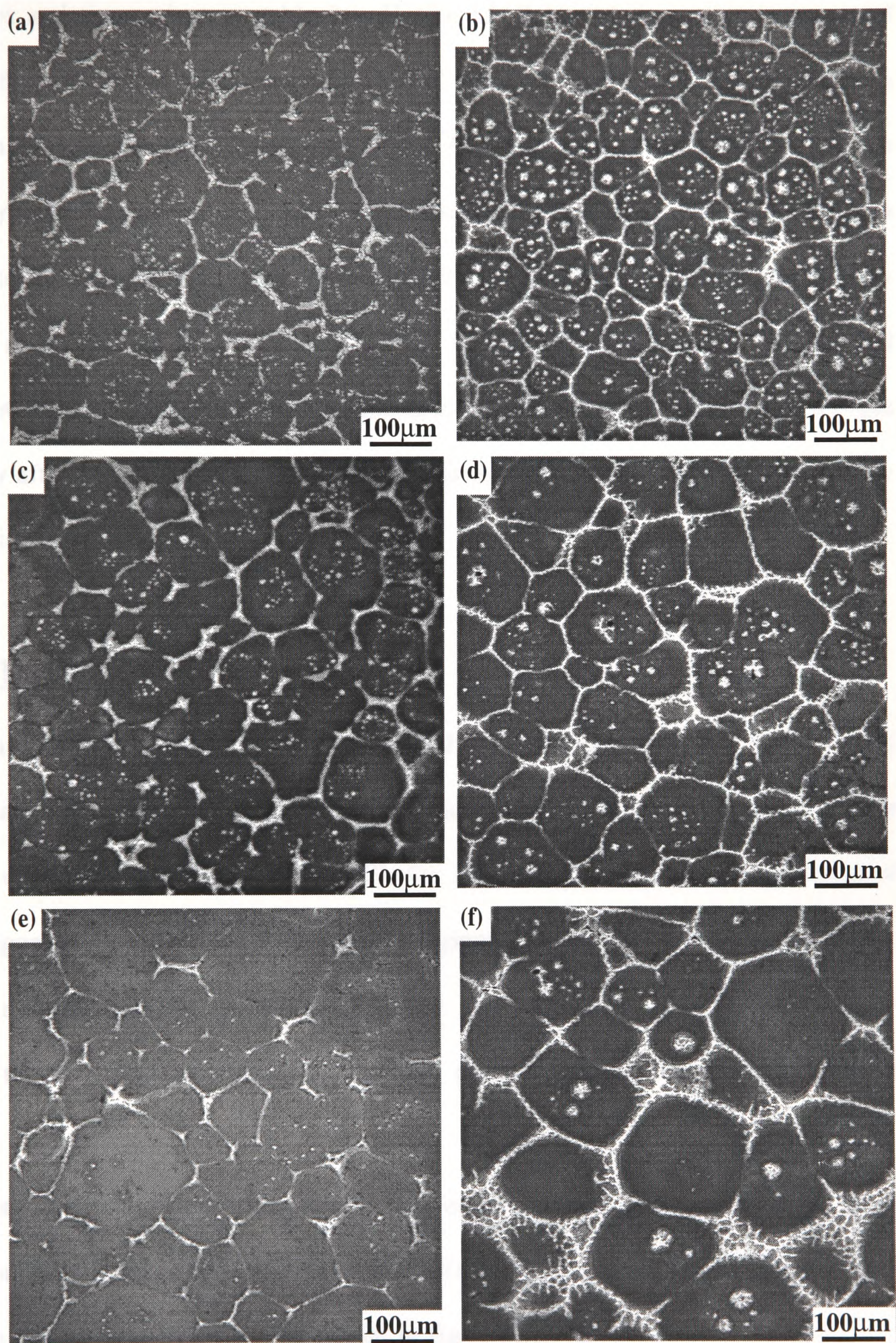


Fig 4.6 UDIMET 720 heat treated at 1245C for (a) 16mins, (c) 46mins and (e) 186mins and at 1290C for (b) 16mins, (d) 46mins and (f) 186mins. Etchant 10% orthophosphoric acid.



temperatures and times given in table 3.1. Unlike MAR-M-002 there was some initial grain growth in the fully solid region at 1150°C from 37µm to ≈50µm followed by a stabilisation of grain size for  $t > 66$  mins at ≈55µm. At 1180°C, in the fully solid region but above the  $\gamma'$  solvus temperature (1150°C, Courtesy of Rolls-Royce plc), the grain size showed a steady increase at all times. In the solid/liquid region grain growth was initially very rapid at  $t < 10^3$ s, but then slowed at  $t > 10^3$ s. For example, at 1245°C ( $f \approx 0.98$ ) and 1290°C ( $f \approx 0.78$ ), the average grain size increased from 37µm to ≈82µm and ≈87µm respectively after 46min, and then increased further to ≈113µm and ≈132µm respectively after 186min.

### 4.3 DISCUSSION

Non-equilibrium solidification conditions during spray forming (De Sanctis 1991) lead to small amounts of low melting point grain boundary eutectic phases in the as sprayed microstructure (Grant *et al* 1993C). On re-heating of the samples Grant *et al* (1993C) suggested that there are three possible regions of heat treatment temperatures for a simple binary alloy as shown in figs 4.8(a)-(b):

- I. Below the eutectic temperature,  $T_E$  the alloy remains solid at all times during heat treatment.
- II. Between  $T_E$  and the equilibrium solidus temperature,  $T_S$  any low melting point grain boundary eutectic is metastable and so melts initially and then re-solidifies as the alloy equilibrates during heat treatment, as shown in fig 4.8(b).
- III. Above  $T_S$ , liquid is always present although the amount reduces as the alloy equilibrates, as shown in fig 4.8(b).

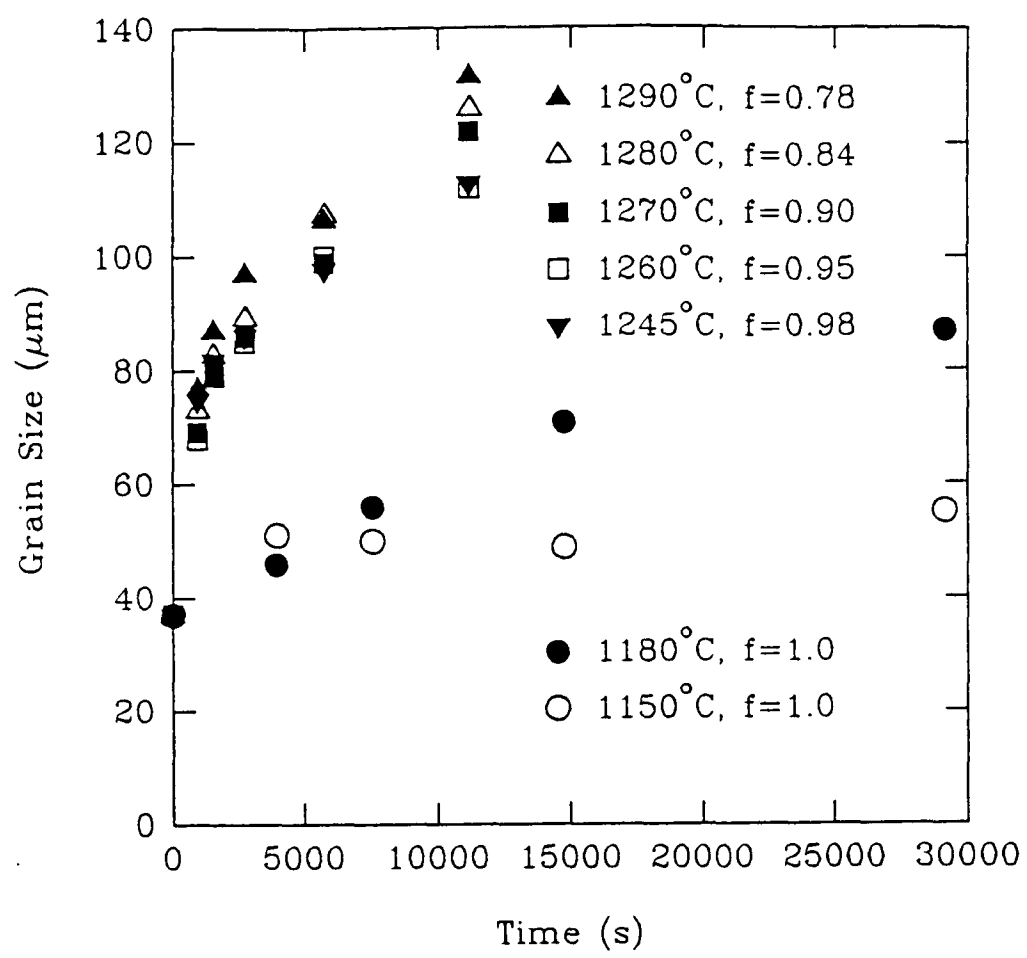


Fig 4.7 Grain size against time for UDIMET 720.

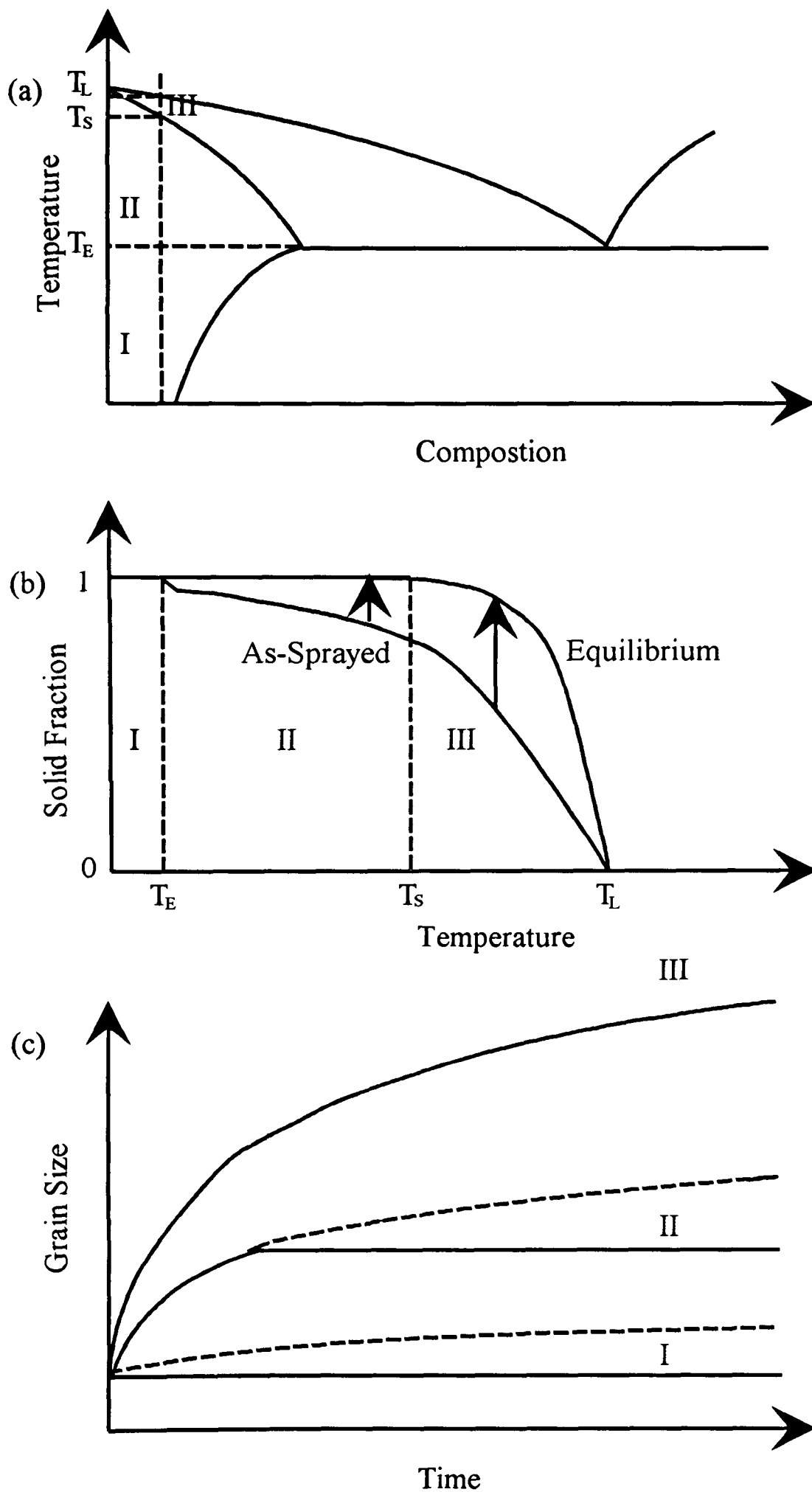


Fig 4.8 Schematic diagram of (a) binary phase diagram, (b) spray formed and equilibrium solid fractions and (c) expected grain growth behaviour from heat treatment in regions I, II and III in (a) and (b). In fig 4.8(c) the solid lines indicate the expected grain growth behaviour when there is negligible grain growth in region I and the dashed lines indicate the expected grain growth behaviour when there is some limited grain growth in region I. (After Grant *et al* 1993C)

The corresponding grain growth behaviour is shown schematically in fig 4.8(c), and the modified behaviour for alloys which undergo some grain growth in the fully solid region is indicated by the dashed lines. Grain growth is negligible or increases steadily in region I; grain growth is initially rapid followed by stabilisation or a reduced rate of steady state growth after equilibration in region II; and grain growth is initially rapid followed by a reduced rate of steady state growth in region III. Regions II and III are similar for alloys where there is some grain growth in the fully solid region.

The heat treated microstructures and grain growth behaviour in figs 4.3 and 4.7 can be related to the solidification data in fig 3.11 and the heat treatment regions I-III in fig 4.8. MAR-M-002 at 1200°C is in region I with negligible grain growth in the fully solid region. Solidification in MAR-M-002 is terminated by the onset of a coupled eutectic reaction involving  $\gamma$ ,  $\gamma'$  and carbide (Willis *et al* 1987) therefore, there is no reduction in grain boundary pinning effect and subsequent increase in grain size for MAR-M-002 heat treated below its solidus temperature. From fig 4.3 for MAR-M-002 the heat treatment temperatures 1300°C and 1315°C are in region II, with initial rapid growth followed by equilibration and a fully solid microstructure with only slight grain growth at  $t > 20$  min. The heat treatment temperatures 1330°C and 1343°C are in region III, with initial accelerated grain growth followed by equilibration and an increased solid fraction with a reduced rate of steady state growth at  $t > 20$  min. Unfortunately no data for the equilibrium solidus of this alloy system is available to confirm this interpretation. The rapid increase in solid fraction for MAR-M-002 in fig 3.11(a) at 1347°C was due principally, to the formation of large amounts of  $\gamma$  phase which accompanied the precipitation of carbides in the interdendritic liquid (Willis *et al* 1987).



Unlike MAR-M-002, UDIMET 720 shows a steady increase in grain size in the fully solid region above the  $\gamma'$  solvus at 1180°C, but below the solidus, probably because of solutioning of the  $\gamma'$  phase and some carbides and carboroborides (Sczerzenie *et al* 1988) that would otherwise pin grain boundaries. The equilibrium solidus temperature from computer modelling studies for UDIMET 720 is 1259°C (Courtesy of Rolls-Royce plc), therefore according to fig 4.8(a) the heat treatment temperatures 1245°C and 1260°C are in the modified region II i.e. fully solid after equilibration with a reduced rate of steady state growth in the solid state. The heat treatment temperatures 1270°C, 1280°C and 1290°C are in region III, with initial accelerated grain growth followed by equilibration and an increased solid fraction with a reduced rate of steady state growth at  $t > 20$  min.

The reason for the initial rapid increase in grain size observed in both superalloys is not clear, although the non-equilibrium liquid to equilibrium liquid or solid transformation may be important. The presence of grain boundary particles in MAR-M-002 which persisted to relatively high temperatures as shown in fig 4.2(c) may also explain the stabilisation of grain size for MAR-M-002 at  $f \approx 0.93$  and  $f \approx 0.96$  not seen in UDIMET 720. The solutioning of grain boundary particles in UDIMET 720 as shown in fig 4.6 and supported by Sczerzenie *et al* (1987) removes effective grain boundary pinning agents and grain growth can occur at reasonable rates in the fully solid region above the  $\gamma'$  solvus temperature but below the solidus temperature. Solid fractions obtained by thermal analysis and modelling studies may not be accurate because they don't represent the real spray forming solidification behaviour. Therefore, the values of  $f$  should only be treated as approximate values and the transitions between regions I, II and III cannot be predicted accurately.

Figs 4.9-4.12 show plots of  $d^2-d_0^2$  and  $d^3-d_0^3$  ( $d_0$  is the initial grain size and  $d$  is the grain size at time  $t$ ) versus time for MAR-M-002 and UDIMET 720 respectively, excluding the data where the grain size was stable for MAR-M-002 at 1300°C and 1315°C. Figs 4.9-4.12 also show first order regression lines through the data points. The regression coefficients and  $d_0$  values obtained from the y-intercept shown in table 4.4.

| Alloy      | Heat Treatment Temperature (°C) | $d^2-d_0^2$ | $d_0$ from y-intercept | $d^3-d_0^3$ | $d_0$ from y-intercept |
|------------|---------------------------------|-------------|------------------------|-------------|------------------------|
| MAR-M-002  | 1200                            | -           | -                      | -           | -                      |
|            | 1300                            | 0.90301     | 14                     | 0.93051     | 16                     |
|            | 1315                            | 0.96973     | 18                     | 0.99107     | -10                    |
|            | 1330                            | 0.97419     | 30                     | 0.99543     | 8                      |
|            | 1343                            | 0.94356     | 47                     | 0.98612     | 47                     |
| UDIMET 720 | 1150                            | -           | -                      | -           | -                      |
|            | 1180                            | 0.99518     | 9                      | 0.99767     | -28                    |
|            | 1245                            | 0.91860     | 52                     | 0.96879     | 60                     |
|            | 1260                            | 0.93024     | 49                     | 0.97144     | 55                     |
|            | 1270                            | 0.96568     | 46                     | 0.99190     | 49                     |
|            | 1280                            | 0.95792     | 50                     | 0.99030     | 55                     |
|            | 1290                            | 0.94967     | 54                     | 0.98450     | 59                     |

Table 4.4 Regression coefficients and  $d_0$  from y-intercept values for MAR-M-002 and UDIMET 720 when fitted to parabolic and cubic laws.

In general, it is not easy to discriminate clearly between parabolic and cubic grain growth kinetics. However, from table 4.4 the regression coefficients for the cubic law are all  $>0.98$  except MAR-M-002 at 1300°C, while for the parabolic law five

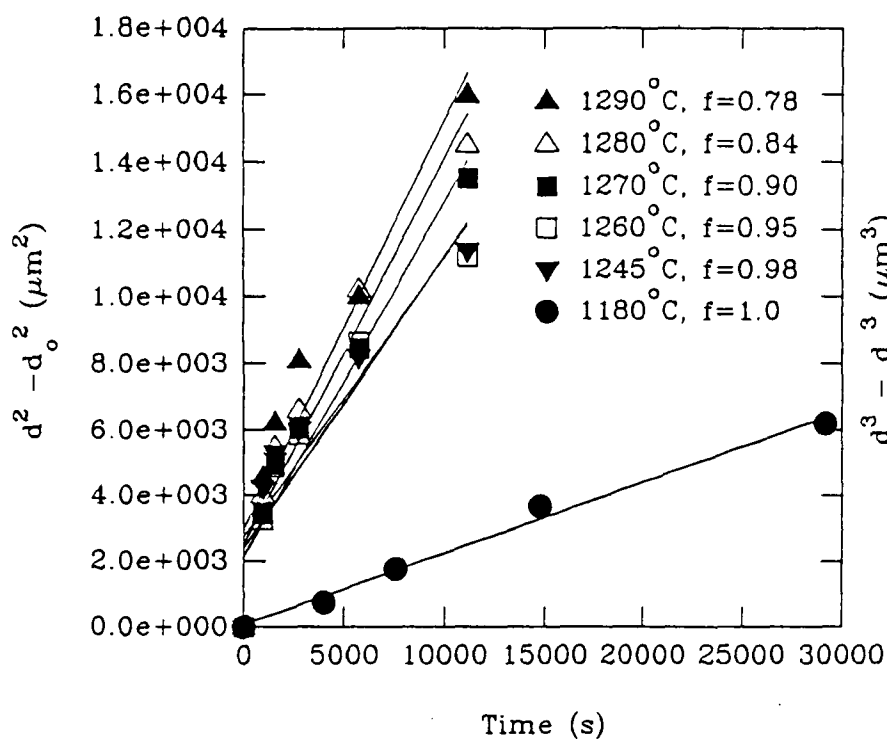


Fig 4.9  $d^2 - d_0^2$  against time for UDIMET 720.

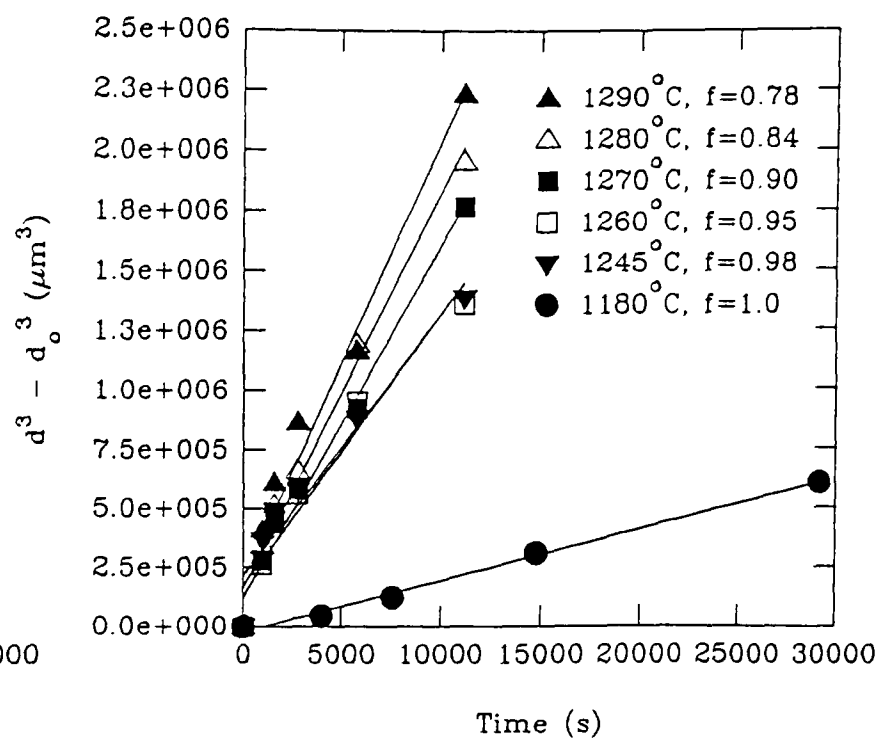


Fig 4.10  $d^3 - d_0^3$  against time for UDIMET 720.

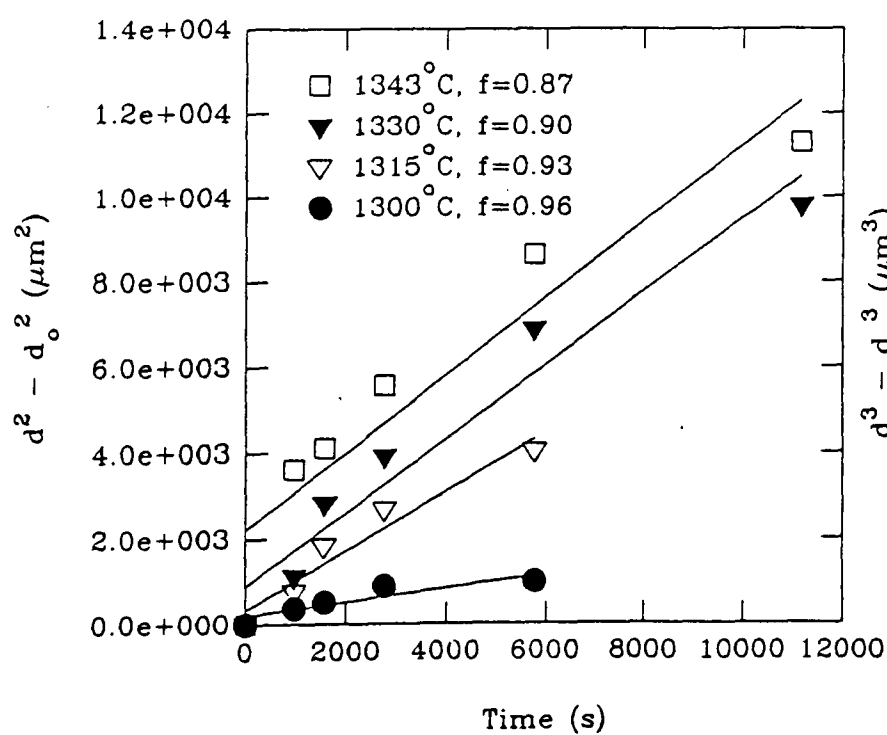


Fig 4.11  $d^2 - d_0^2$  against time for MAR-M-002

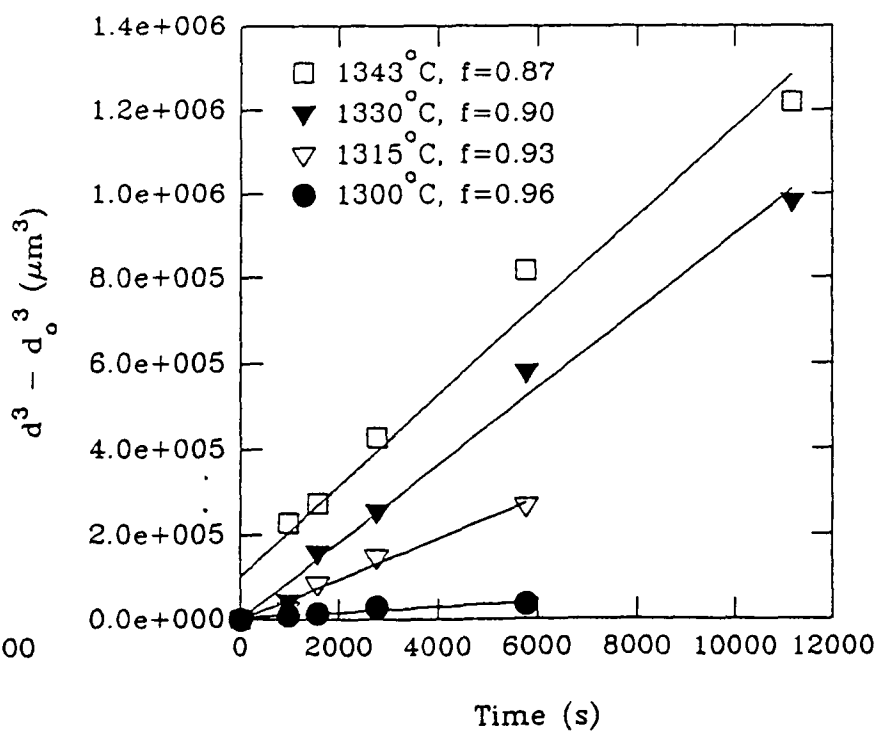


Fig 4.12  $d^3 - d_0^3$  against time for MAR-M-002.

regression coefficients were less than 0.95. From the  $d_0$  intercept values the cubic law again gave a slightly better fit for MAR-M-002 heat treated in the solid/liquid region at 1315°C and 1330°C. From the  $d_0$  intercept values for UDIMET 720 heat treated in the solid/liquid region there was no appreciable difference between parabolic and cubic kinetics, although for UDIMET 720 heat treated in the fully solid region above the  $\gamma'$  solvus (1180°C) parabolic kinetics appeared to give a better fit. Therefore, on the basis of the regression coefficients and  $d_0$  values in table 4.4 the diffusion limited cubic law was a better fit for MAR-M-002 and UDIMET 720 heat treated in the solid/liquid region, while for UDIMET 720 heat treated at 1180°C parabolic grain growth kinetics seemed to give a better fit. Annavarapu and Doherty (1995) and Grant *et al* (1993C) also reported cubic kinetics from similar experiments on spray formed Cu, Ni, and Al alloys heat treated in the solid/liquid region while Liang *et al* (1992) reported parabolic kinetics from similar experiments of spray formed Ni<sub>3</sub>Al heat treated in the fully solid region.

Cubic grain growth kinetics follow an equation of the form:

$$d^3 - d_0^3 = Kt \quad (4.1)$$

where  $d$  is the grain size after a time  $t$ ,  $d_0$  is the initial grain size and  $K$  is a coarsening rate constant. From similar experiments on spray formed Cu, Ni and Al alloys Annavarapu and Doherty (1995) reported values of the coarsening rate constant ( $K$ ) in the range  $6-393 \times 10^{-18} \text{m}^3/\text{s}$  for solid fractions of between 0.58-0.95. Fig 4.13(a) shows a plot of coarsening rate constant ( $K$ ) against heat treatment temperature for MAR-M-002 and UDIMET 720. Fig 4.13(b) shows the same data as in fig 4.13(a) plotted against solid fraction, as well as data from Annavarapu and Doherty (1995) and Grant *et al* (1993C). All the data shows that  $K$  decreased as the solid fraction increased and the  $K$  values were

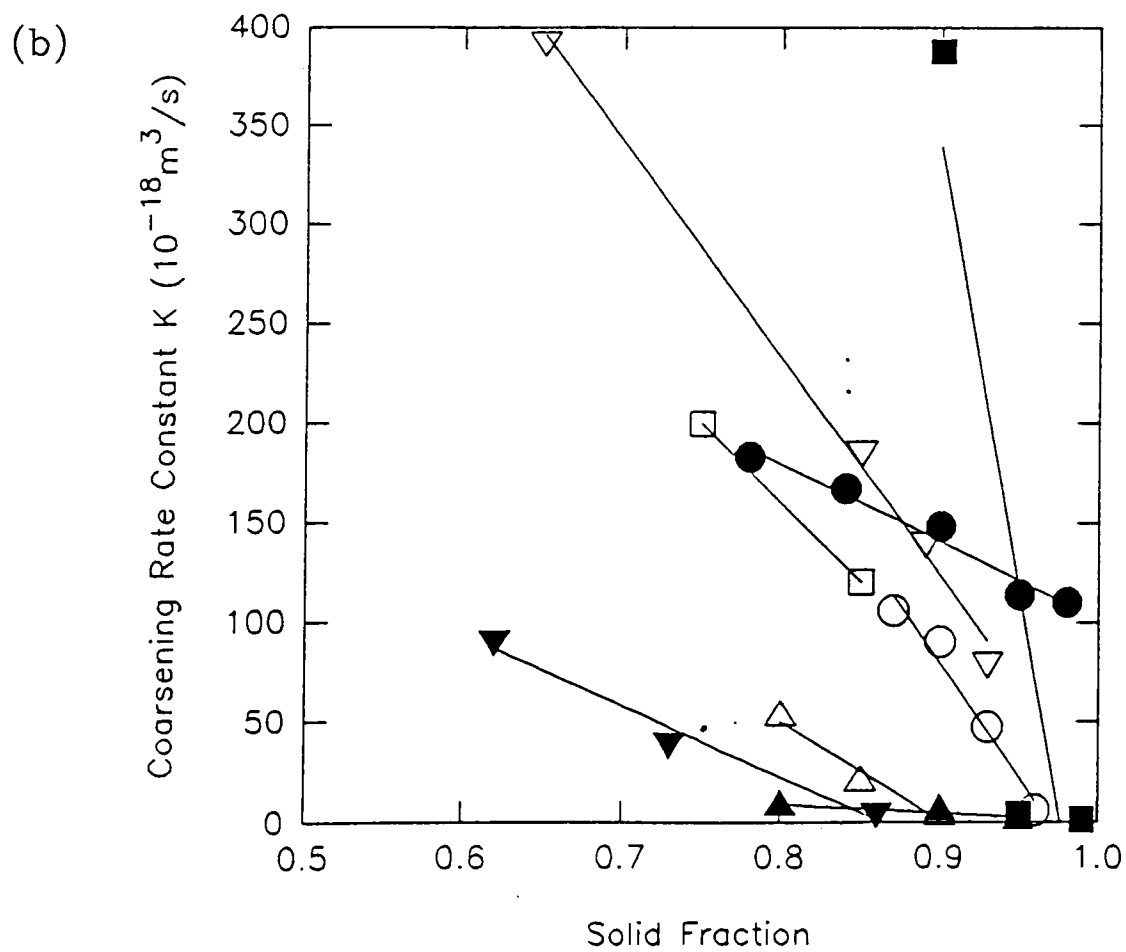
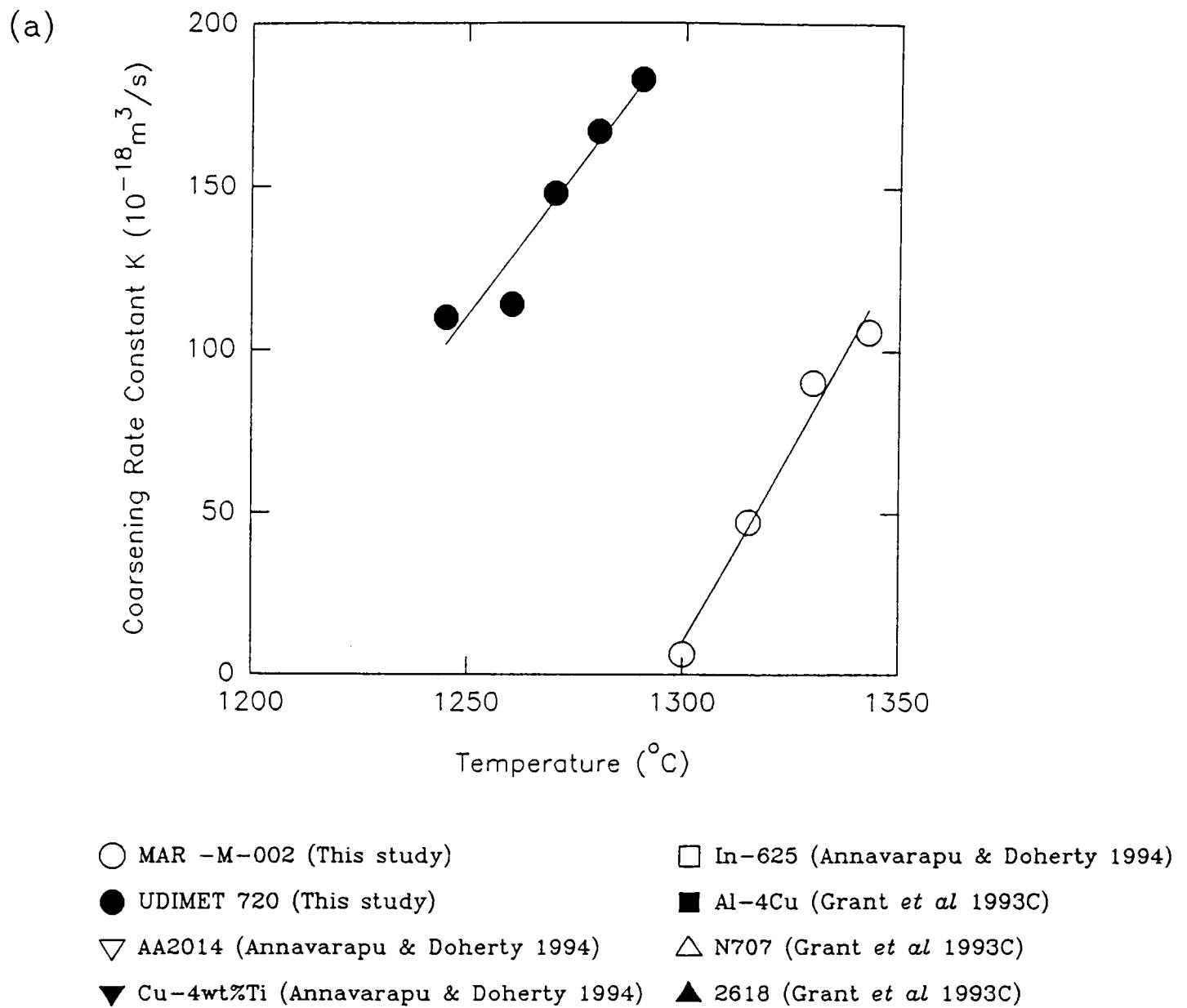


Fig 4.13 Coarsening rate constant ( $K$ ) against (a) temperature and (b) solid fraction.

of a similar magnitude. This variation is against the well established trend for LPS (liquid phase sintering) of binary alloys as shown in fig 4.14, because of a shortening of the diffusion distance between grains (Kang and Yoon 1982, Hardy and Voorhees 1988 and Niemi and Courtney 1981), although in all cases for the data in fig 4.14 the solid fraction was varied by changing the composition and not temperature. Annavarapu and Doherty (1995) proposed that a minority of insoluble second phase particles were responsible for inhibited coarsening rates observed in spray formed alloys. Therefore, carbide particles in MAR-M-002, clearly present at liquid wetted grain boundaries in fig 4.15, and a smaller amount of similar grain boundary carbide particles for UDIMET 720 shown in fig 4.5(e) may be responsible for the lower than expected coarsening rates as proposed by Annavarapu and Doherty (1995).

#### **4.4 Summary**

When heat treated in the solid/liquid region there was an initial rapid increase in grain size for both MAR-M-002 and UDIMET 720. For MAR-M-002 the grain size then either stabilised if  $f \geq 0.93$  or continued to increase but at a slower rate if  $f \leq 0.90$ . For UDIMET 720 there was no stabilisation in the grain size at any temperature in the solid/liquid region. The stabilisation of grain size for MAR-M-002 when  $f \geq 0.93$  was probably caused by the re-solidification of non-equilibrium liquid present during the early stages of heat treatment, since MAR-M-002 showed virtually no increase in grain size when heat treated in the fully solid region probably due to the presence of grain boundary carbide particles. However, UDIMET 720 showed a steady increase in grain size when heat treated in the fully solid region above the  $\gamma'$  solvus temperature probably due to the



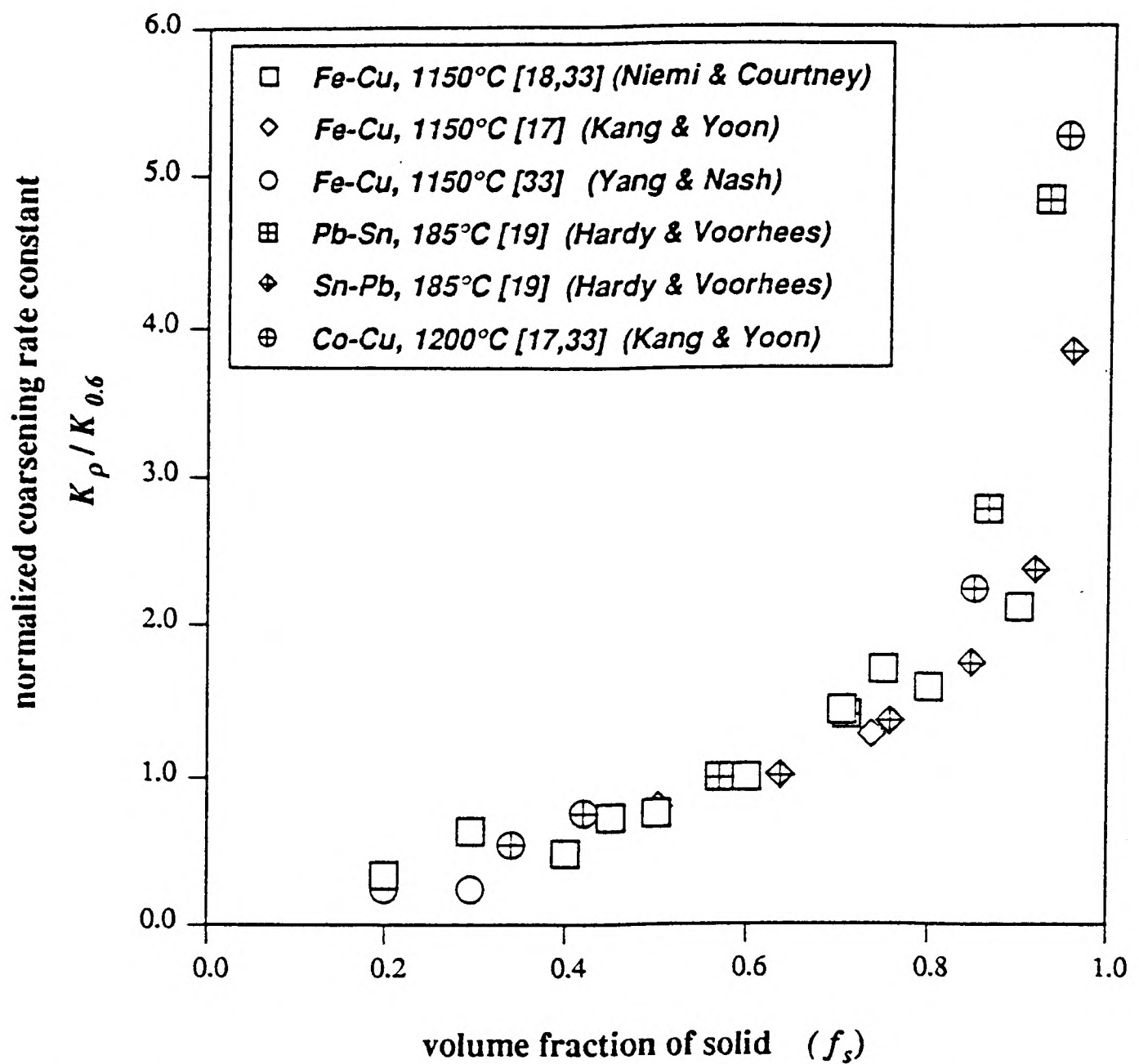


Fig 4.14 Measured dependence of coarsening rate constant ( $K$ ) on solid fraction during isothermal LPS of various binary alloys. For ease of comparison the data was normalised to the measured  $K$  value for  $f_s \approx 0.6$ . In all cases  $f_s$  was varied by changing the composition. (After Annavarapu and Doherty 1995).

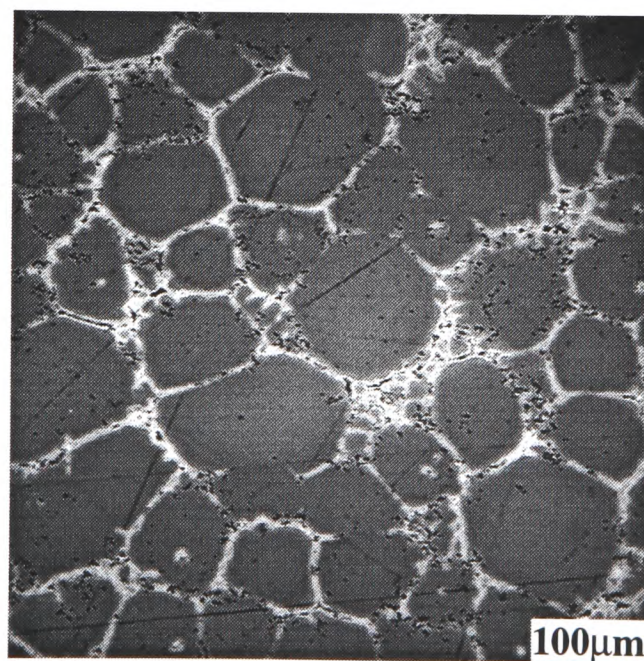


Fig 4.15 MAR-M-002 heat treated at 1330C for 186mins. Etchant 10% orthophosphoric acid.

dissolution of carbide particles (Sczerzenie *et al* 1988).

Cubic kinetics gave a better fit to the grain size data for MAR-M-002 and UDIMET 720 in the solid/liquid region, while parabolic kinetics were a better fit for UDIMET 720 heat treated in the fully solid region at 1180°C. The cubic coarsening rate constant (K) for MAR-M-002 and UDIMET 720 decreased with increasing solid fraction, against the trend for LPS of binary alloys. The presence of small grain boundary carbide particles observed in MAR-M-002 and UDIMET 720, as proposed by Annavarapu and Doherty (1995) may account for the decrease of K with solid fraction.

## **CHAPTER 5 - SPRAY FORMING OF UDIMET 720**

This chapter describes the results of the spray forming of UDIMET 720 under a range of different process parameters designed to isolate the effect of individual process parameters. The changes in deposit porosity and grain size with process parameters are described and their relationship with deposit thermal conditions as measured by infra-red thermal imaging and embedded thermocouples.

### **5.1 Introduction**

In total 38 spray forming runs were performed of which 25 were considered to be successful i.e. a deposit was produced under known conditions which could be sectioned and examined metallographically. The experiments were designed so that only one independent variable was changed between runs. The independent variables studied were: (i) GFR (gas flow rate) and (ii) spray distance (atomiser-substrate) distance. The GFR and spray distance could be altered by appropriate manipulation of the starting process parameters. Occasionally during spray forming it was discovered that the melt delivery nozzle broke either partially or fully, giving measurable changes in the MFR (melt flow rate), determined after spraying by dividing the mass which flowed through the nozzle by the run time. Therefore, this variation inadvertently provided another independent variable: (iii) MFR, although its magnitude could not be preset. The error in the measurement of MFR was estimated to be between 2 and 4g/s (3-9%) because of variation in the time between when metal first starts flowing through the nozzle and when spraying starts (1-2s). The variation in the deposition rate, a dependent variable defined as the mass of the deposit divided by the run time, was also measured. The error in the measurement of

deposition rate was estimated to be 0.5g/s (1-4%) due to errors in the measurement of spraying time. Errors in the measurement of other process parameters are given in tables 5.1 and 5.2 respectively.

Two sets of spray forming runs were performed: (i) a preliminary set with a rotating substrate at an angle of  $22^\circ$  to identify the best combination of process parameters and the broad effect of changing individual parameters and (ii) a more extensive set of runs with a stationary horizontal substrate using embedded thermocouples to investigate the variation of microstructure with process parameters and deposit temperature.

## **5.2 Preliminary Spray Forming Results**

Table 5.1 shows a summary of all the process parameters for runs 0-15 in the preliminary set of spray forming runs. Unsuccessful runs are left blank. Runs 0-3 were performed using the previous atomiser for spray forming Al alloys (Grant 1991) for which the GFR could not be increased beyond  $\approx 3.31/\text{s}$ . The overspray powder distributions for runs 1 and 2 are plotted in figs 5.1(a) and (b) and fitted well to a log-normal distribution with relatively large mean diameters of  $175\mu\text{m}$  and  $164\mu\text{m}$  respectively. Fig 5.2 shows typical microstructures taken from the middle of the corresponding deposit for run 2. Fig 5.2(a) shows a large amount of irregular porosity between the droplet splat boundaries probably because insufficient liquid was present during spray forming. Fig 5.2(b) shows the splat internal grain structure with grain boundaries perpendicular to the splat boundaries, i.e. parallel to the deposit growth direction. This splatted microstructure arises because at a GFR of  $3.31/\text{s}$  atomisation is inefficient giving large droplets which remain liquid until deposition. The droplets deform extensively on impact and solidify before the

| Process Parameters                 | Error | Run Number |       |       |       |   |   |   |       |   |   |       |       |       |    |    |       |
|------------------------------------|-------|------------|-------|-------|-------|---|---|---|-------|---|---|-------|-------|-------|----|----|-------|
|                                    |       | 0          | 1     | 2     | 3     | 4 | 5 | 6 | 7     | 8 | 9 | 10    | 11    | 12    | 13 | 14 | 15    |
| Gas flow rate (l/s)                | 0.1   | 3.3        | 3.4   | 3.3   | 3.3   |   |   |   | 3.8   |   |   | 4.3   | 4.8   | 5.3   |    |    | 5.3   |
| Flow meter pressure (MPa)          | 0.02  |            | 0.75  | 0.77  | 0.74  |   |   |   | 0.31  |   |   | 0.36  | 0.43  | 0.51  |    |    | 0.52  |
| Overspressure (Bar)                | 0.02  | 0.2        | 0.15  | 0.15  | 0.15  |   |   |   | 0.2   |   |   | 0.2   | 0.2   | 0.2   |    |    | 0.2   |
| Stopper rod temperature (C)        | 7     | >1400      | 1438  | 1448  | 1460  |   |   |   | 1416  |   |   | 1446  | 1438  | 1446  |    |    | 1437  |
| Substrate angle                    | 1     |            | 22    | 22    | 22    |   |   |   | 22    |   |   | 22    | 22    | 22    |    |    | 22    |
| Atomiser-Substrate distance (m)    | 0.005 |            | 0.4   | 0.35  | 0.3   |   |   |   | 0.3   |   |   | 0.3   | 0.3   | 0.3   |    |    | 0.2   |
| Gas exit temperature (C)           | 2     |            | -     | 245   | 360   |   |   |   | 340   |   |   | 320   | 250   | 290   |    |    | 270   |
| Run time (s)                       | 0.5-1 | data-      | 40-45 | 51    | 58    |   |   |   | 47.5  |   |   | 48    | 53    | 60    |    |    | 58    |
| Melt flow rate (g/s)               | 2-4   | logger     | -     | 74    | 64    |   |   |   | 59    |   |   | 55    | 48    | 48    |    |    | 52    |
| MFR:GFR                            | 0.5   |            |       | 18.7  | 16.2  |   |   |   | 12.9  |   |   | 10.7  | 8.2   | 7.5   |    |    | 8.2   |
| Deposition rate at substrate (g/s) | 0.5   | not        | 13-15 | 15    | 13    |   |   |   | 12    |   |   | 12    | 11    | 13    |    |    | 21    |
| Initial charge wt (kg)             | 0.002 | working    | 3.7   | 3.99  | 4.227 |   |   |   | 3.248 |   |   | 2.94  | 3.213 | 3.236 |    |    | 3.422 |
| Skull weight (kg)                  | 0.002 |            | -     | 0.233 | 0.508 |   |   |   | 0.432 |   |   | 0.3   | 0.832 | 0.363 |    |    | 0.424 |
| Deposit weight (kg)                | 0.002 |            | 0.587 | 0.772 | 0.766 |   |   |   | 0.58  |   |   | 0.586 | 0.587 | 0.784 |    |    | 1.243 |
| Yield through atomiser (%)         | 1     |            | -     | 20.5  | 20.5  |   |   |   | 18    |   |   | 20    | 18    | 24    |    |    | 36    |
| Total yield (%)                    | 1     |            | 16    | 19    | 18    |   |   |   | 20    |   |   | 22    | 22    | 27    |    |    | 41    |
| Thermal imaging (yes/no)           |       | n          | y     | y     | y     |   |   |   | y     |   |   | y     | n     | n     |    |    | y     |

Table 5.1 Spray forming process parameters for preliminary runs 0-15.

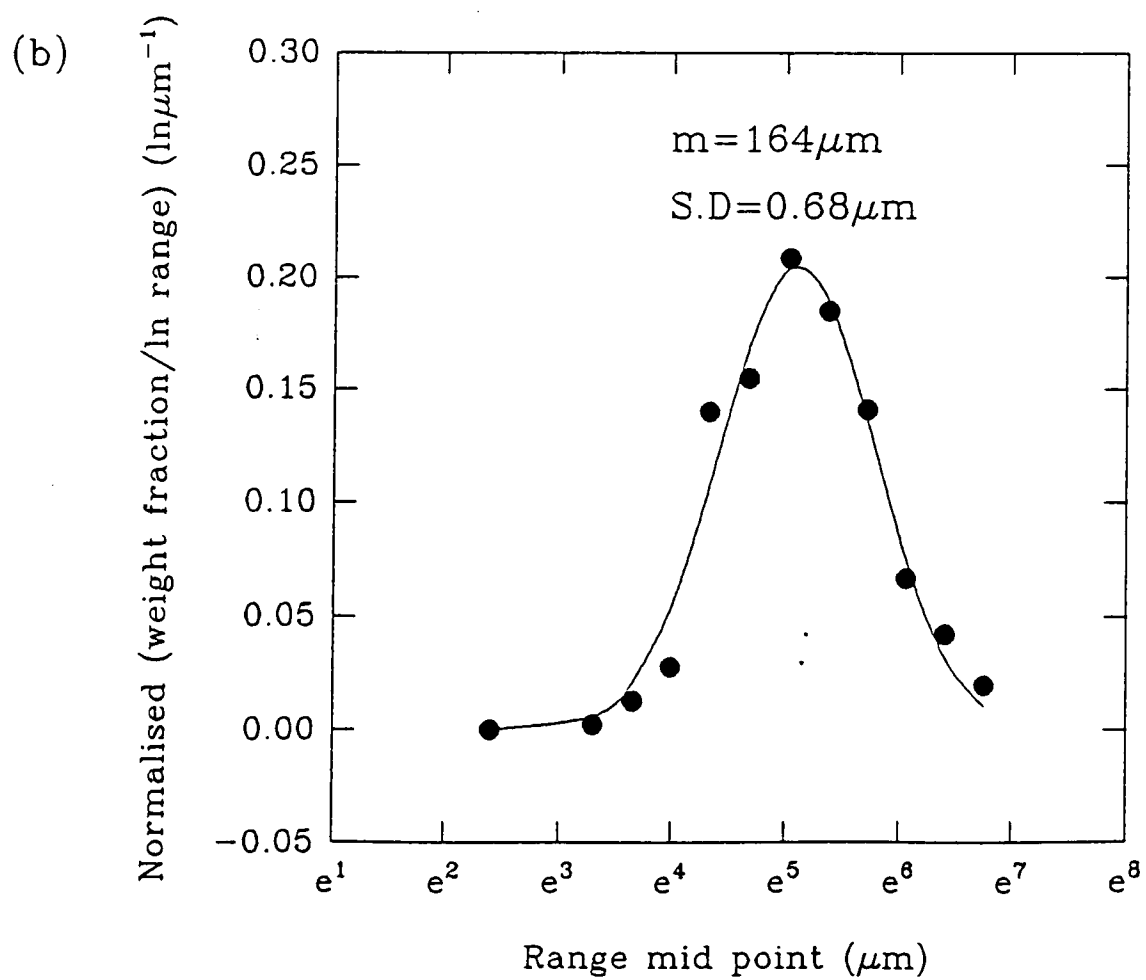
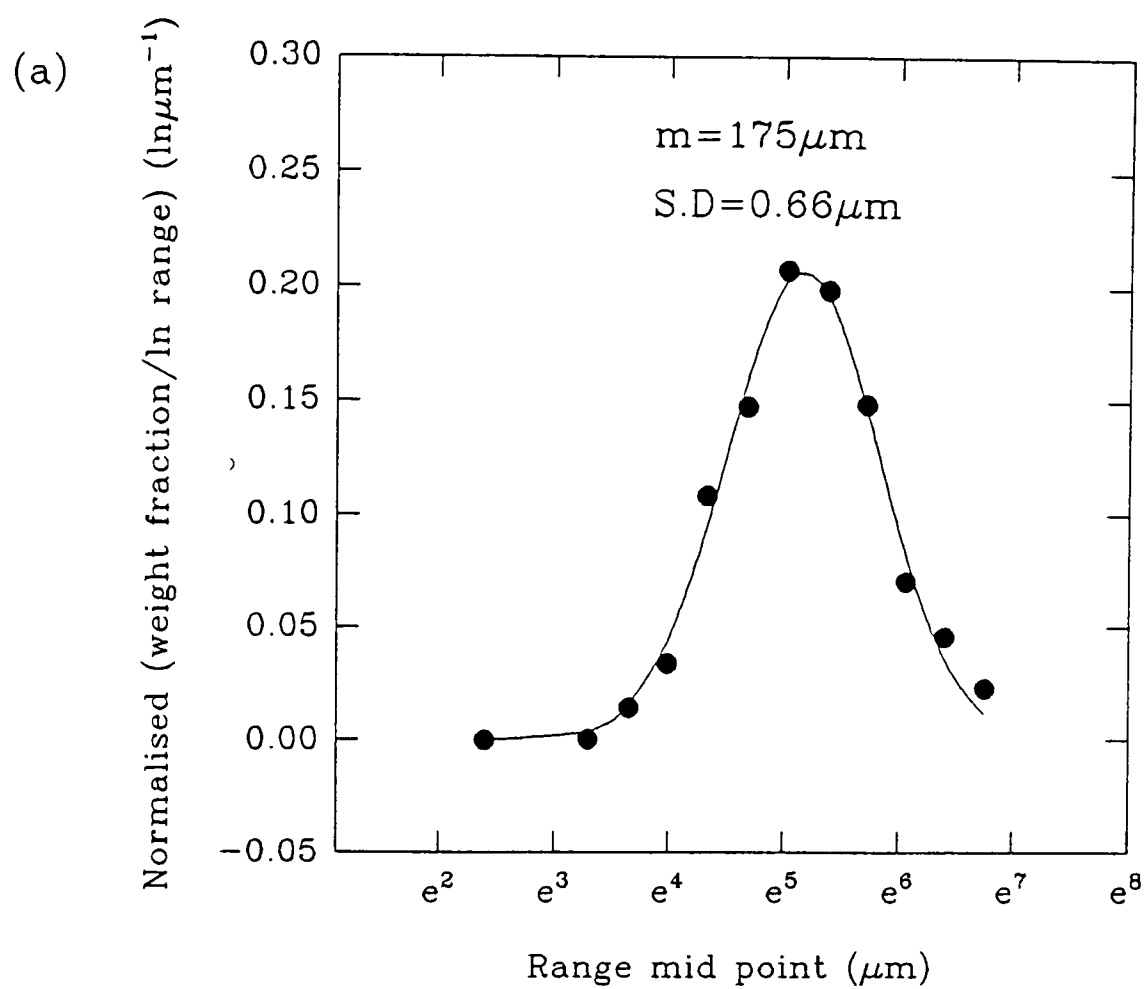


Fig 5.1 Overspray powder distribution fitted to a log-normal distribution for (a) run 1 and (b) run 2.



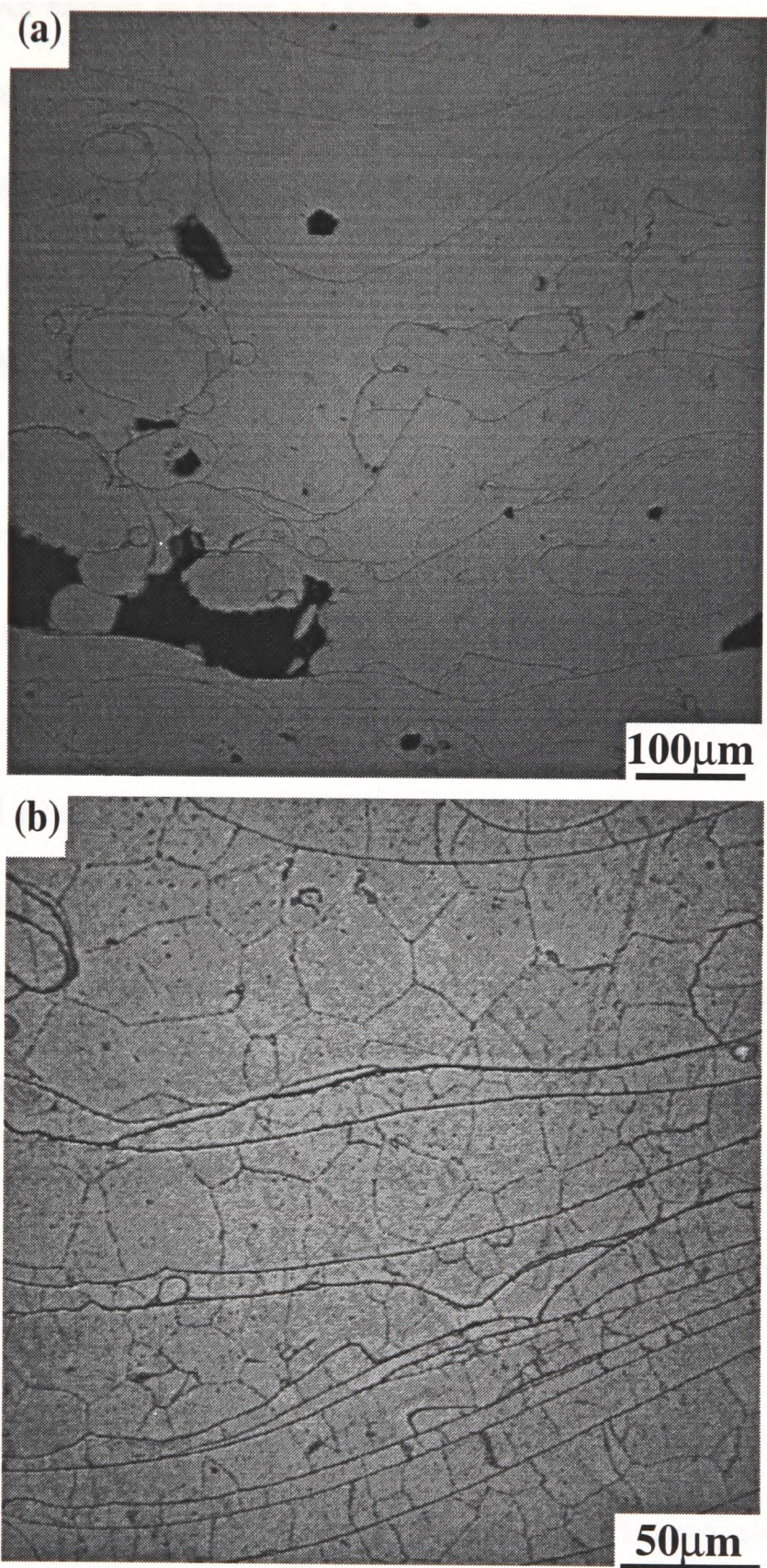


Fig 5.2 Micrographs taken from the middle of run 2: (a) unetched at a magnification of x88 and (b) etched at a magnification of x200. Etchant 10% Orthophosphoric acid.

next droplets arrive. As shown in table 5.1 for runs 0-3 deposit yields were also very low ( $\approx 20\%$ ). The combination of large liquid droplets and low deposition rates ( $\approx 14\text{g/s}$ ) leads to the splatted microstructure in fig 5.2, more characteristic of thermal spray processes rather than spray formed microstructures. Therefore, subsequent experiments used the modified higher GFR atomiser in an attempt to reduce droplet sizes, restrict spray divergence and therefore increase deposition rates.

For runs 7-15 the GFR was increased steadily from  $\approx 3.8\text{l/s}$  to  $\approx 5.3\text{l/s}$ , while attempting to keep the other process parameters constant, although there were slight variations in MFR. Figs 5.3(a)-(e) show the variation in the overspray powder distribution, fitted to a log-normal distribution for runs 7-15 respectively, as the MFR:GFR (ratio of melt to gas mass flow rates) was decreased. Figs 5.3(a)-(e) show that the mean diameter decreased from  $237\mu\text{m}$  for run 7 to  $157\mu\text{m}$  for run 12 as the MFR:GFR decreased from 12.9 to 7.5. A secondary peak between  $63\mu\text{m}$  and  $90\mu\text{m}$  also appeared and became progressively larger, i.e. there were more smaller droplets in the overspray distribution at lower MFR:GFR ratios. The GFR could not be increased beyond  $5.3\text{l/s}$  using the current flow rate meter.

Figs 5.4(a)-(e) show the microstructures of runs 7-15 respectively taken from the middle of the deposit. They all show evidence of splat boundaries where droplets impacted the substrate. Figs 5.4(a), (c) and (d) for runs 7, 11 and 12 respectively show that some of the splats had a fine dendritic structure whereas figs 5.4(b) and (e) for runs 10 and 15 respectively show that the splats had a fine internal equiaxed grain structure similar to run 2 in fig 5.2(b). Some grain boundaries were continuous through the splats.

Fig 5.5 shows the through thickness variation in average area fraction porosity for



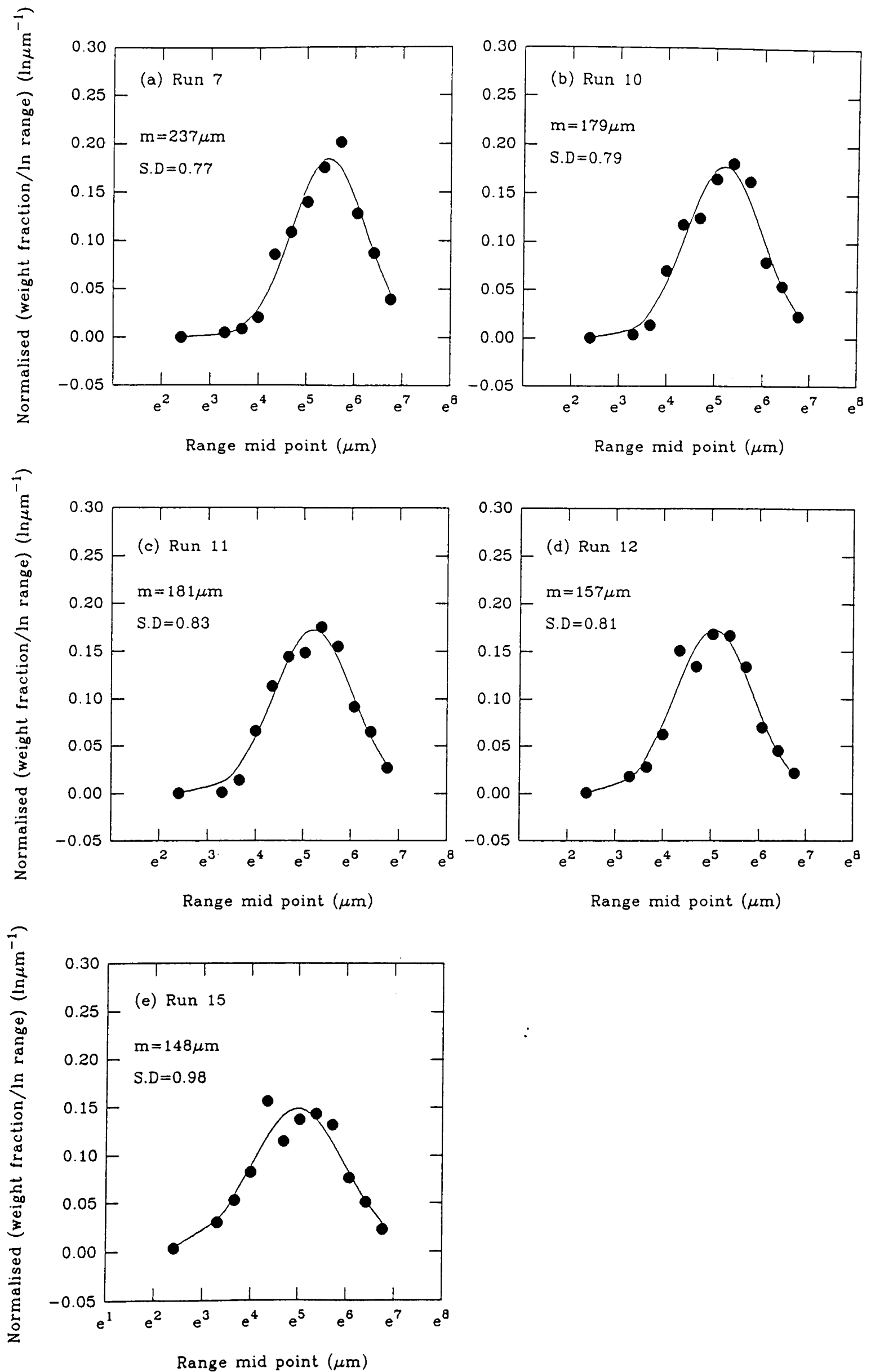


Fig 5.3 Overspray powder distribution fitted to a log-normal distribution for (a) run 7, (b) run 10, (c) run 11, (d) run12 and (e) run 15.



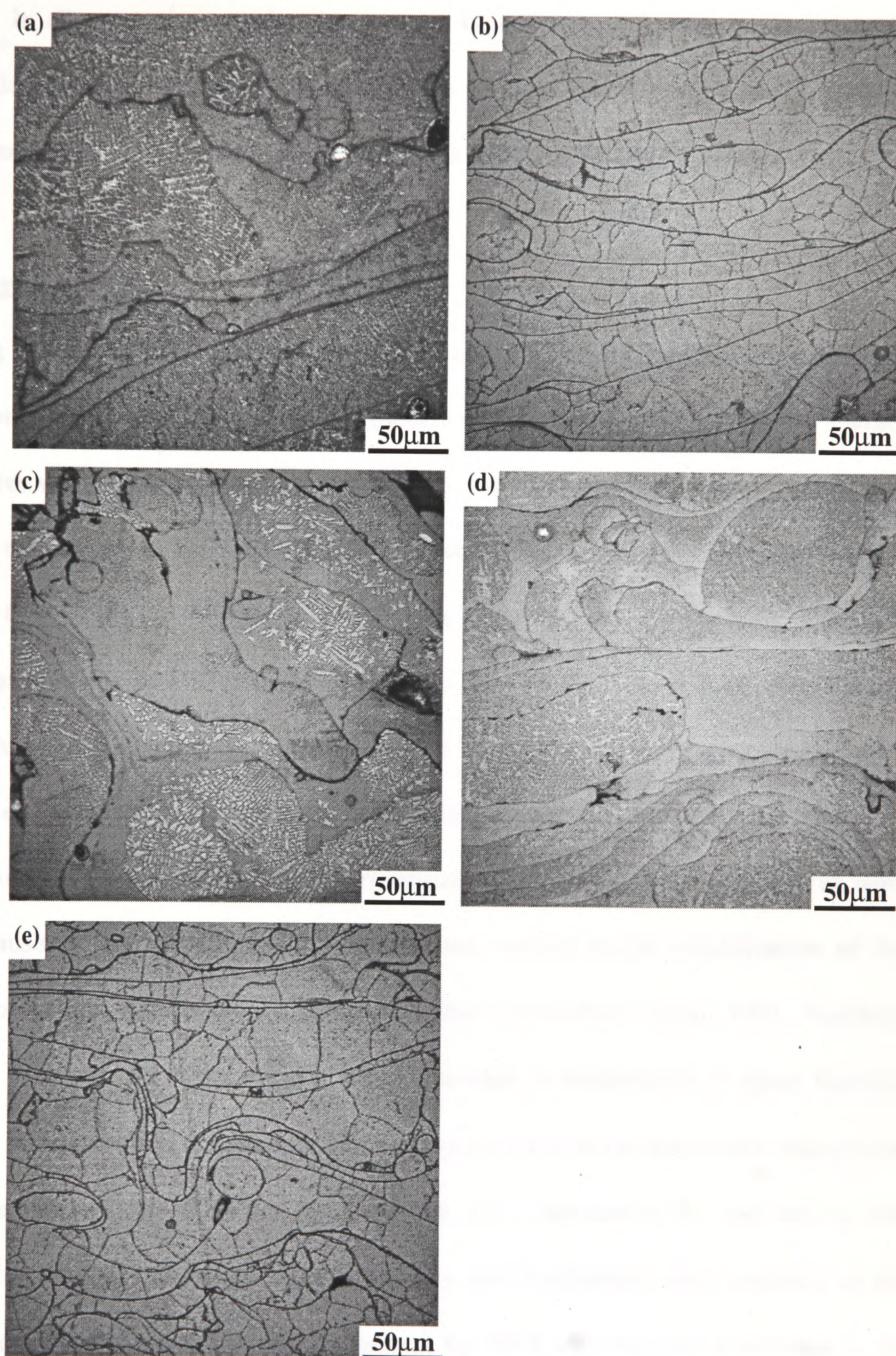


Fig 5.4 Micrographs taken from the middle of the deposit for (a) run 7, (b) run 10, (c) run 11 (d) run 12 and (e) run 15. Etchant 10% Orthophosphoric acid.



runs 7-15. In all cases, the porosity was initially high, up to 18% close to the substrate, and then decreased to between 5-8%. There was no clear relationship between porosity and process parameters. Typical mean grain sizes for runs 10 and 15 were  $\approx 25\mu\text{m}$ .

### **5.2.1 Thermal Imaging**

Fig 5.6 shows the top surface temperature against normalised run time from the thermal imaging of runs 7, 10 and 15 respectively assuming  $\epsilon_o=0.3$ . The top surface temperatures were mean temperatures taken from a central, circular region of the deposit similar to that shown in fig 3.8(b). Fig 5.7 shows 3 sequences of thermal images taken from run 10 assuming  $\epsilon_o=0.3$ ; (a) at the beginning of the run; (b) in the middle of the run; and (c) just after the end of the run respectively. For each run, very high temperatures were measured at the beginning and near the end. Initial high temperatures corresponded to when metal was first poured through the nozzle and splashed onto the substrate before the atomising gas was turned on. Rapid temperature increases near the end of the run were either because of: (i) oxidation of the deposit top surface or (ii) solidification of the deposit top surface, both of which result in higher emissivities (Grant 1991, Newbery 1993). All 3 runs in fig 5.6 showed a steady decrease in temperature as spray forming proceeded. A reduction in temperature can be because the heat extraction rate was greater than the heat arrival rate. This can be caused by: (i) a decrease in the heat arrival rate from the droplet spray: a steady reduction in the melt hydrostatic head pressure as the melt was consumed leads to a steady decrease in the MFR and therefore a decrease in the heat arrival rate from the droplet spray; or (ii) an increase in the heat extraction rate, through the deposit and substrate or gas convection from the deposit top surface.

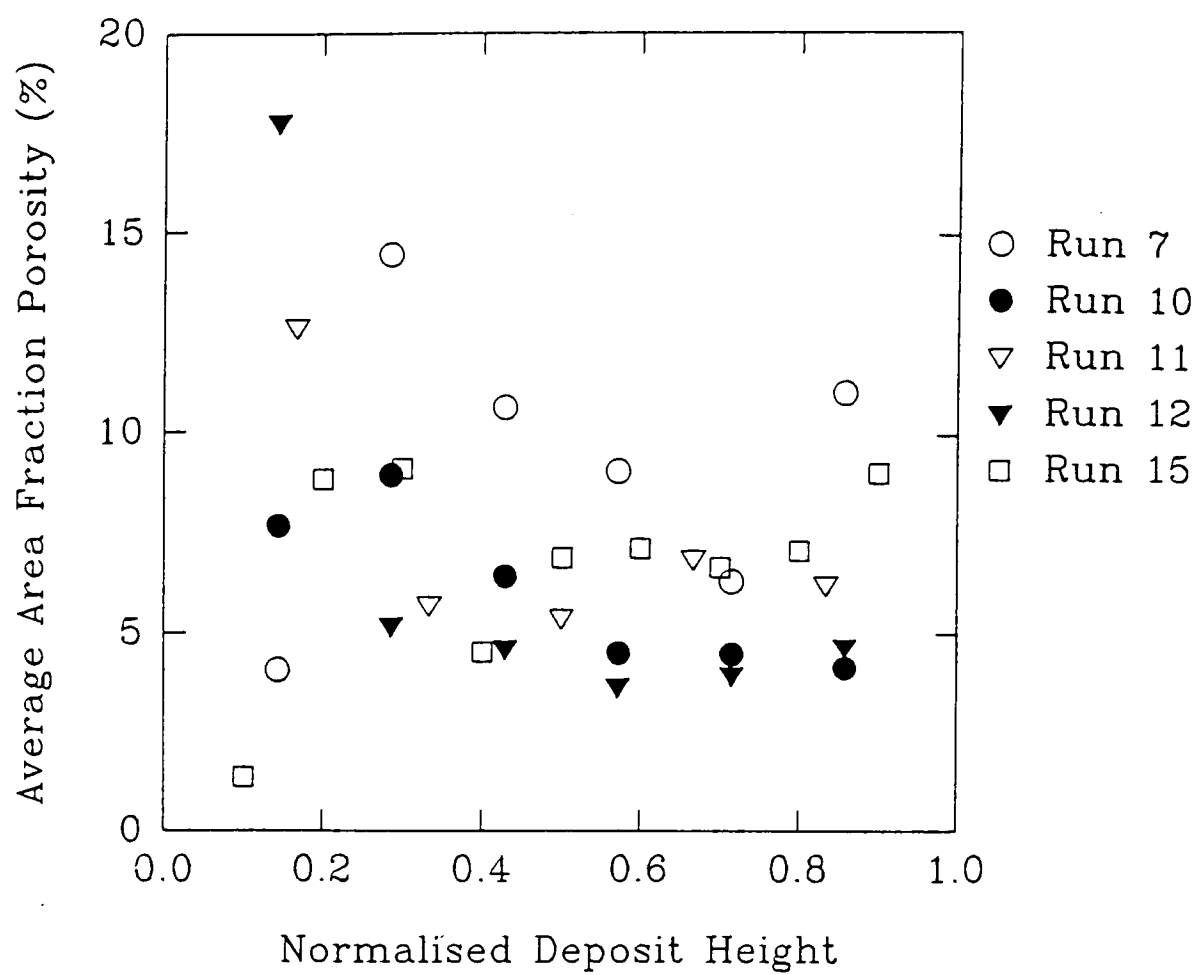


Fig 5.5 Average area fraction porosity against normalised deposit height for runs 7-15.

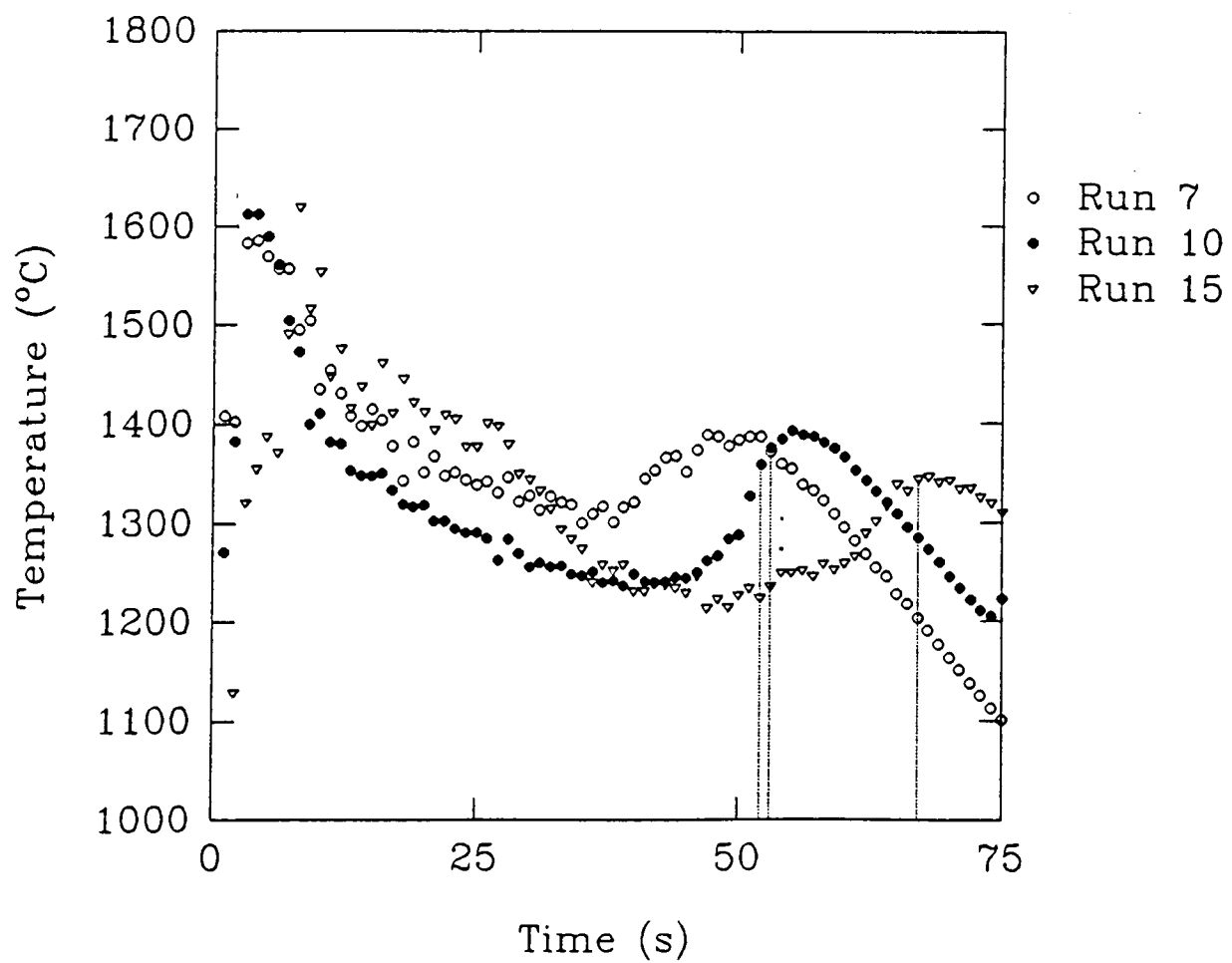


Fig 5.6 Thermal imaging temperature, assuming  $\epsilon_0=0.3$  against run time for runs 7, 10 and 15.





Fig 5.7 Sequence of thermal images taken from run 10 assuming  $\epsilon_0=0.3$ ; (a) at the beginning of the run; (b) in the middle of the run; and (c) just after the end of the run respectively.



(c)



cont... Fig 5.7 Sequence of thermal images taken from run 10 assuming  $\epsilon_0=0.3$ ; (a) at the beginning of the run; (b) in the middle of the run; and (c) just after the end of the run respectively.

Unfortunately all the deposits in this set of spray forming runs had a splat structure with corresponding high porosities. Slight microstructural changes with process parameters were observed though, and a reasonable level of process parameter control was achieved. Therefore, a second set of more extensive runs with a stationary horizontal substrate using embedded thermocouples was embarked upon to try and produce deposits with a low porosity and a more desirable equiaxed grain structure.

### **5.3 Spray Forming Using Embedded Thermocouples**

Table 5.2 shows a summary of all the process parameters used for spray forming runs 16-37 employing embedded thermocouples. Again those runs that were unsuccessful are left blank. The melt overpressure, substrate angle and superheat were kept constant throughout at 0.2Bar, 0° and 1445°C respectively with the exception of run 33 where the superheat was  $\approx 40^\circ\text{C}$  lower at 1403°C. The three independent variables: GFR, spray distance and MFR ranged from 4.8-5.3l/s, 0.2-0.3m, and 38-101g/s respectively. This gave a variation in deposition rate of 14-48g/s. Initial charge weights varied from 2.621-3.258kg, with deposit weights ranging from 0.777-1.318kg, giving yields as a percentage of metal that flowed through the atomiser of 24-44%. The gas exit temperatures and run times ranged from 150-370°C and 23-58s respectively.

#### **5.3.1 Embedded Thermocouples**

Embedded thermocouples were placed at three heights above the substrate prior to spraying: (i) B=bottom, a thermocouple at the lowest height which was 5mm above the substrate for all deposits; (ii) M=middle, a thermocouple at the median height of 10mm

| Process Parameters                 | Error | Run Number |       |    |       |       |       |    |       |       |       |    |       |       |    |       |       |       |       |       |       |    |       |
|------------------------------------|-------|------------|-------|----|-------|-------|-------|----|-------|-------|-------|----|-------|-------|----|-------|-------|-------|-------|-------|-------|----|-------|
|                                    |       | 16         | 17    | 18 | 19    | 20    | 21    | 22 | 23    | 24    | 25    | 26 | 27    | 28    | 29 | 30    | 31    | 32    | 33    | 34    | 35    | 36 | 37    |
| Gas flow rate (l/s)                | 0.1   | 5.3        | 5.3   |    | 5.3   | 5.3   | 5.3   |    | 5.3   | 4.8   | 4.8   |    | 4.8   | 4.8   |    | 5.3   | 5.3   | 5.3   | 4.8   | 5.3   | 4.8   |    | 4.8   |
| Flow meter pressure (MPa)          | 0.02  | 0.52       | 0.5   |    | 0.51  | 0.51  | 0.5   |    | 0.51  | 0.41  | 0.41  |    | 0.41  | 0.41  |    | 0.5   | 0.5   | 0.5   | 0.413 | 0.5   | 0.413 |    | 0.413 |
| Overspressure (Bar)                | 0.02  | 0.2        | 0.2   |    | 0.2   | 0.2   | 0.2   |    | 0.2   | 0.2   | 0.2   |    | 0.2   | 0.2   |    | 0.2   | 0.2   | 0.2   | 0.2   | 0.2   | 0.2   |    | 0.2   |
| Stopper rod temperature (C)        | 7     | 1448       | 1444  |    | 1443  | 1445  | 1444  |    | 1444  | 1441  | 1445  |    | 1443  | 1439  |    | 1445  | 1446  | 1444  | 1403  | 1440  | 1445  |    | 1444  |
| Substrate angle                    | 1     | 0          | 0     |    | 0     | 0     | 0     |    | 0     | 0     | 0     |    | 0     | 0     |    | 0     | 0     | 0     | 0     | 0     | 0     |    | 0     |
| Atomiser-Substrate distance (m)    | 0.005 | 0.3        | 0.3   |    | 0.25  | 0.25  | 0.25  |    | 0.2   | 0.2   | 0.25  |    | 0.25  | 0.3   |    | 0.3   | 0.25  | 0.2   | 0.2   | 0.2   | 0.2   |    | 0.25  |
| Gas exit temperature (C)           | 2     | 260        | 250   |    | 325   | 370   | 260   |    | 250   | 210   | 240   |    | 255   | 290   |    | 225   | 240   | 150   | 220   | 225   | 245   |    | 290   |
| Run time (s)                       | 0.5-1 | 31         | 50    |    | 25    | 23    | 51    |    | 28    | 56    | 58    |    | 57    | 45    |    | 56    | 54    | 50    | 44    | 45    | 49    |    | 47    |
| Melt flow rate (g/s)               | 2-4   | 64         | 59    |    | 101   | 99    | 59    |    | 59    | 50    | 42    |    | 43    | 44    |    | 39    | 39    | 45    | 41    | 38    | 41    |    | 50    |
| MFR:GFR                            | 0.5   | 9.9        | 9.1   |    | 15.9  | 15.6  | 9.3   |    | 9.3   | 8.7   | 7.3   |    | 7.5   | 7.6   |    | 6.1   | 6.1   | 7.1   | 7.1   | 6     | 7.1   |    | 8.7   |
| Deposition rate at substrate (g/s) | 0.5   | 25         | 23    |    | 48    | 47    | 27    |    | 32    | 25    | 15    |    | 16    | 14    |    | 14    | 16    | 25    | 21    | 21    | 20    |    | 17    |
| Initial charge wt (kg)             | 0.002 | 3.106      | 3.258 |    | 2.751 | 3.313 | 3.324 |    | 3.091 | 3.077 | 3.228 |    | 2.689 | 2.688 |    | 2.662 | 2.677 | 2.696 | 2.77  | 2.689 | 2.712 |    | 2.621 |
| Skull weight (kg)                  | 0.002 | 1.12       | 0.33  |    | 0.22  | 1.036 | 0.307 |    | 1.45  | 0.288 | 0.77  |    | 0.241 | 0.712 |    | 0.505 | 0.581 | 0.422 | 0.946 | 0.977 | 0.702 |    | 0.282 |
| Deposit weight (kg)                | 0.002 | 0.777      | 1.128 |    | 1.197 | 1.081 | 1.391 |    | 0.901 | 1.318 | 0.855 |    | 0.909 | 0.654 |    | 0.793 | 0.851 | 1.254 | 0.912 | 0.946 | 0.971 |    | 0.785 |
| Yield through atomiser (%)         | 1     | 25         | 35    |    | 44    | 33    | 42    |    | 29    | 43    | 27    |    | 34    | 24    |    | 30    | 32    | 47    | 33    | 35    | 36    |    | 30    |
| Total Yield (%)                    | 1     | 39         | 39    |    | 47    | 47    | 46    |    | 55    | 47    | 35    |    | 37    | 33    |    | 37    | 41    | 55    | 50    | 55    | 48    |    | 33    |
| Thermal imaging (yes/no)           | -     | y          | n     |    | n     | n     | n     |    | n     | y     | y     |    | y     | y     |    | y     | y     | y     | y     | y     | y     |    | y     |

Table 5.2 Spray forming process parameters for embedded thermocouple runs 16-37.

or 7.5mm above the substrate depending on the expected deposit thickness; and (iii) T=top, a thermocouple at 15mm or 10mm above the substrate, again depending on the expected deposit thickness.

Figs 5.8(a)-(c) show embedded thermocouple measurements from runs 20, 24 and 27 respectively. For runs 24 and 27 in figs 5.8(b) and (c), the results from the B thermocouple showed some variation initially until it became fully embedded. The temperature of the M and T thermocouples increased rapidly soon after spraying started until they each became fully embedded and then the temperature slowly decreased. Occasionally thermocouples were not fully embedded as shown for the T thermocouple for runs 24 and 27 in figs 5.8(b) and (c) respectively, because (i) the deposit was too thin to embed the thermocouple fully or (ii) excessive porosity developed around the thermocouple impeding intimate deposit contact. Figs 5.8(b) and (c) show that the initial rate of increase in temperature of the T thermocouple was not the same as for the B or M thermocouples and it did not reach the same or higher temperature as the M thermocouple which would be expected from its higher position in the deposit indicating that it was not fully embedded.

Table 5.3 shows the maximum B, M and T thermocouple temperatures reached during spraying for each of runs 16-37. Table 5.3 indicates that several T and M thermocouples were not fully embedded since their temperatures were much lower than the other thermocouples in that deposit. The  $T_{max}$  temperature for a deposit is defined as the maximum temperature reached during spray forming as measured by B, M or T thermocouples and the  $T_{max}$  temperature for each deposit is indicated in table 5.3 in bold type. The measurement error of the thermocouples was  $\approx \pm 7^{\circ}\text{C}$ . Table 5.3 also contains

| Parameters            | Run Number |      |      |      |      |      |      |      |      |      |      |      |      |      |      |      |      |
|-----------------------|------------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|
|                       | 16         | 17   | 19   | 20   | 21   | 23   | 24   | 25   | 27*  | 28*  | 30*  | 31*  | 32*  | 33** | 34*  | 35*  | 37*  |
| Thermal Imaging       | y          | n    | n    | n    | n    | n    | y    | y    | y    | y    | y    | y    | y    | y    | y    | y    | y    |
| Spray distance (m)    | 0.3        | 0.3  | 0.25 | 0.25 | 0.25 | 0.2  | 0.2  | 0.25 | 0.25 | 0.3  | 0.3  | 0.25 | 0.2  | 0.2  | 0.2  | 0.2  | 0.25 |
| Gas flow rate (l/s)   | 5.4        | 5.4  | 5.3  | 5.3  | 5.3  | 5.3  | 4.8  | 4.8  | 4.8  | 4.8  | 5.3  | 5.3  | 5.3  | 4.8  | 5.3  | 4.8  | 4.8  |
| Melt flow rate (g/s)  | 64         | 59   | 101  | 99   | 59   | 59   | 50   | 42   | 43   | 44   | 39   | 39   | 45   | 41   | 38   | 41   | 50   |
| Deposition rate (g/s) | 25         | 23   | 48   | 47   | 27   | 32   | 25   | 14   | 16   | 14   | 14   | 16   | 25   | 21   | 21   | 20   | 17   |
| Max temp-B (C)        | -          | -    | -    | -    | 1264 | 1263 | 1256 | 1262 | 1229 | 1194 | 1150 | 1185 | 1205 | 1237 | 1168 | 1218 | 1180 |
| Max temp-M (C)        | 1061       | 1260 | 1244 | 1276 | 1278 | 1241 | 1272 | 1145 | 1239 | 1235 | 1160 | 1165 | 1268 | 1259 | 1232 | 1250 | 1213 |
| Max temp-T (C)        | 1246       | 1270 | 1295 | 1297 | 1278 | 1199 | 1170 | 911  | 1096 | 1190 | 1180 | 1210 | 1245 | 1153 | 1261 | 1252 | 1165 |
| Mass median size (um) | 164        | 147  | 144  | 153  | 144  | 157  | 140  | 145  | 140  | 140  | 133  | 122  | 122  | 134  | 115  | 136  | 146  |

B=5mm, M=10mm & T=15mm except \* - B=5mm, M=7.5mm and T=15mm  
 Superheat=1445C except \*\* - superheat=1403C

Table 5.3 Summary of spray forming process parameters, B, M and T thermocouple maximum temperatures and mass median overspray size for runs 16-37. (Tmax temperatures indicated in bold type).



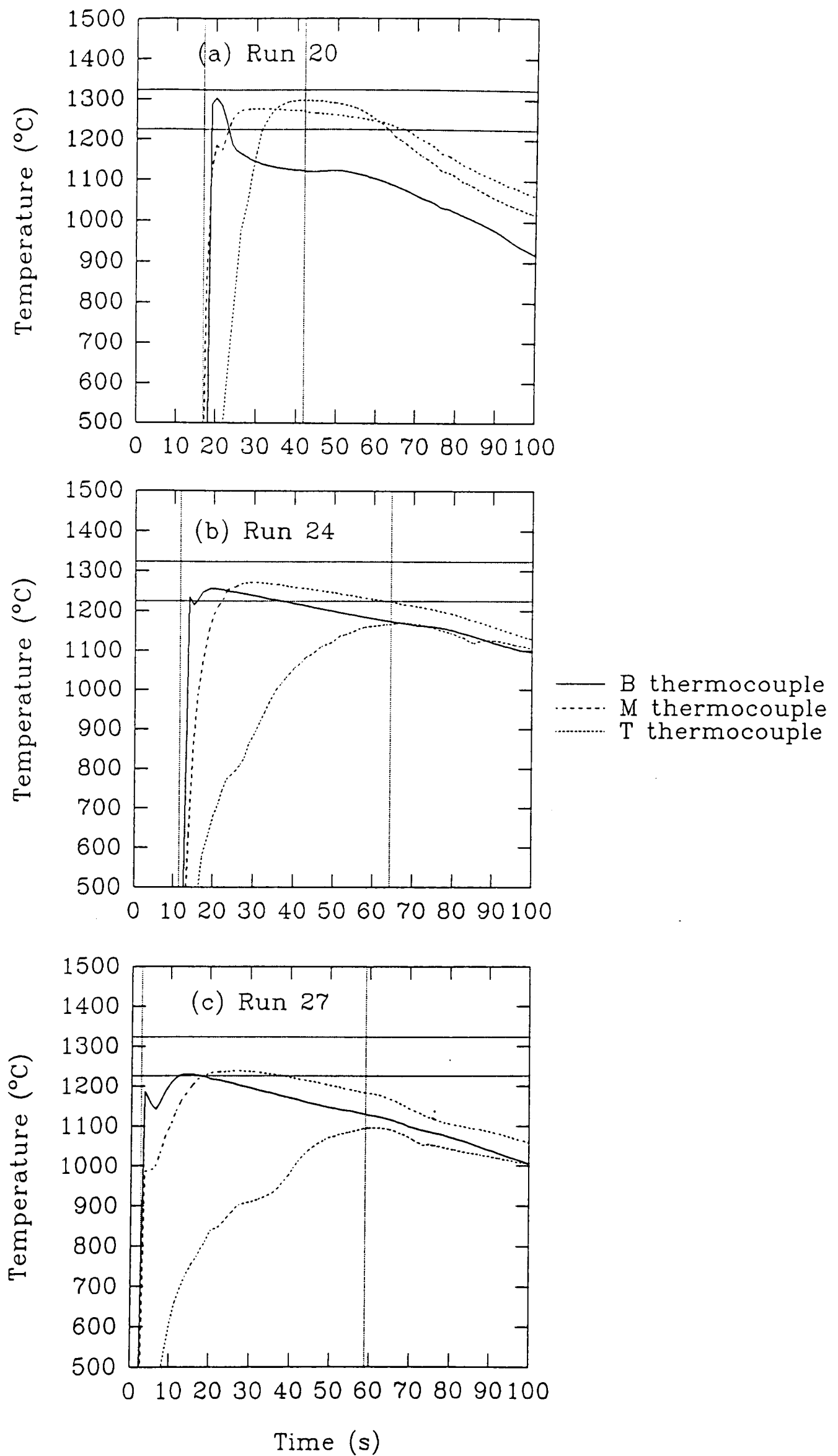


Fig 5.8 Typical variation of embedded thermocouple temperature with time for: (a) run 20, (b) run 24 and (c) run 27. The vertical dashed lines indicate the start and end of the run respectively and the horizontal dashed lines indicate the solidus and liquidus temperature of UDIMET 720 respectively.

a more concise summary for each of runs 16-37 of the independent variables spray distance, GFR and MFR, together with the deposition rate and the mass median diameter of the overspray powder distribution when fitted to a Rosin-Rammler distribution.

### **5.3.2 Thermal Imaging**

Fig 5.9 shows typical mean temperatures assuming  $\epsilon_0=0.3$  taken from the deposit centre for runs 30, 32 and 34 against run time. The 3 dashed vertical lines indicate the end of each run respectively. Excluding the very start and end of the runs for reasons described in section 5.2.1, fig 5.9 shows that: (a) for run 30 the top surface temperature was approximately constant for  $\approx 15$ s and then decreased towards the end of the run; (b) for run 32 the top surface temperature also remained approximately constant at a higher temperature for  $\approx 35$ s and then increased at the very end of the run; and (c) for run 34 the top surface temperature remained approximately constant for  $\approx 25$ s and then decreased slightly towards the end of the run. All 3 runs showed an apparent rapid increase in temperature either just before or after the run finished as described above for runs 7, 10 and 15.

Fig 5.10 shows typical individual thermal images assuming  $\epsilon_0=0.3$  taken from the region of the run where the temperature was approximately constant for: (a) run 30, (b) run 32 and (c) run 34. They show that the thermal intensity distribution was approximately symmetrical around the centre of the deposit and the thermal intensity decreased towards the edges of the deposit. Figs 5.11 and 5.12 show typical sequences of thermal images taken from run 30 and run 34 respectively: (a) at the beginning of the run; (b) in the middle of the run; and (c) just after the end of the run assuming that

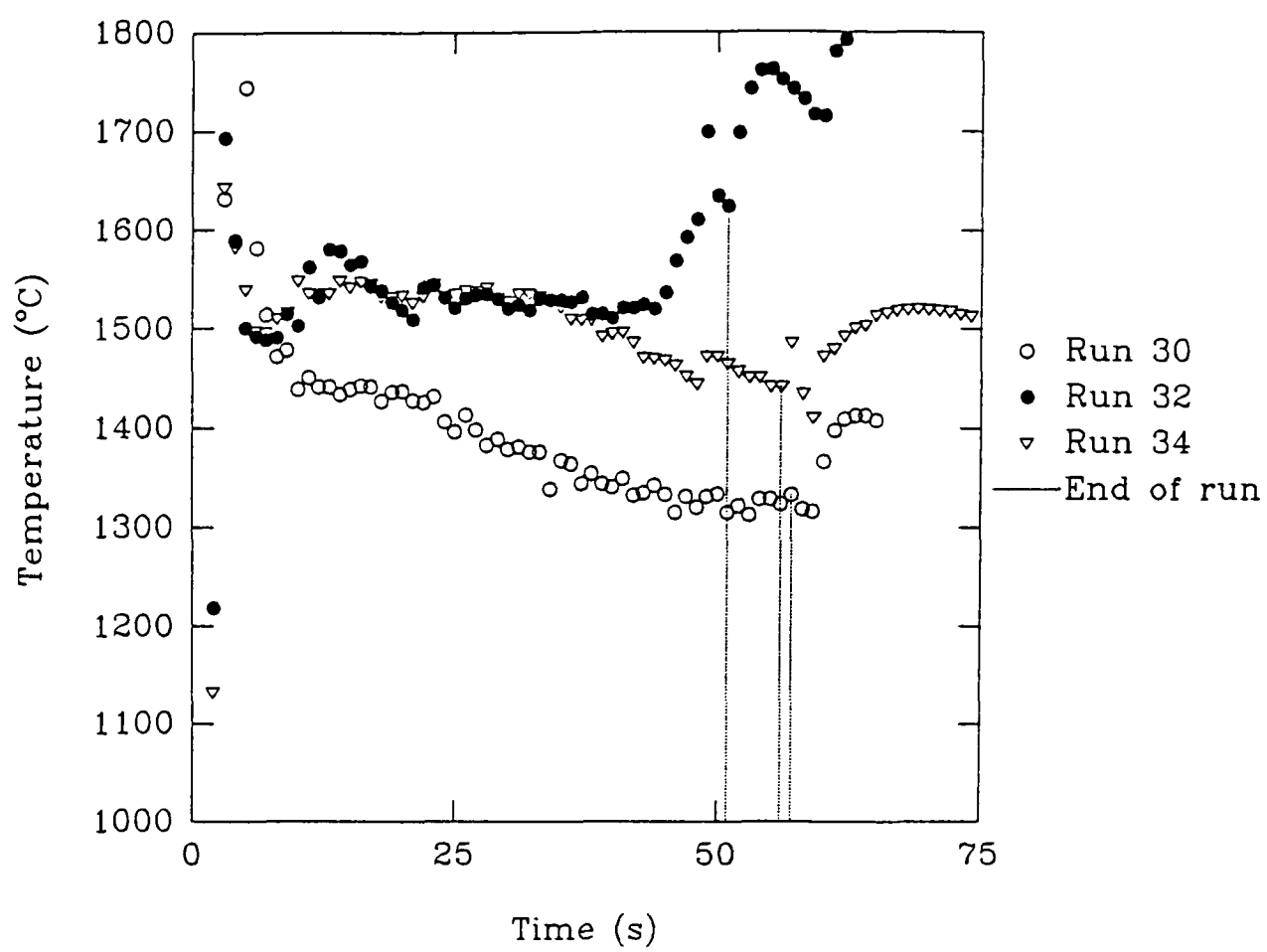


Fig 5.9 Typical mean thermal imaging temperatures, assuming  $\epsilon_o=0.3$  against time for runs 30, 32 and 34.



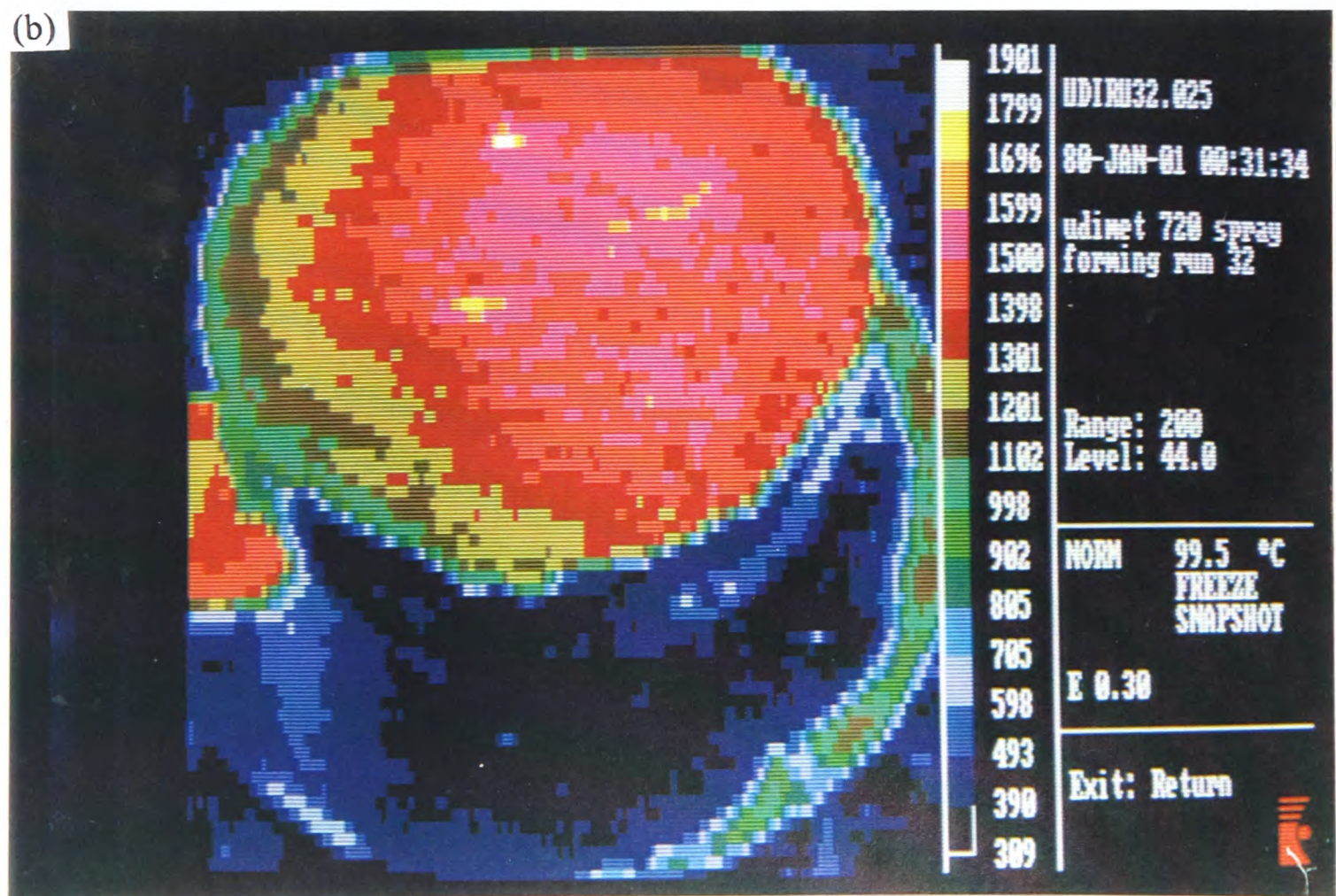
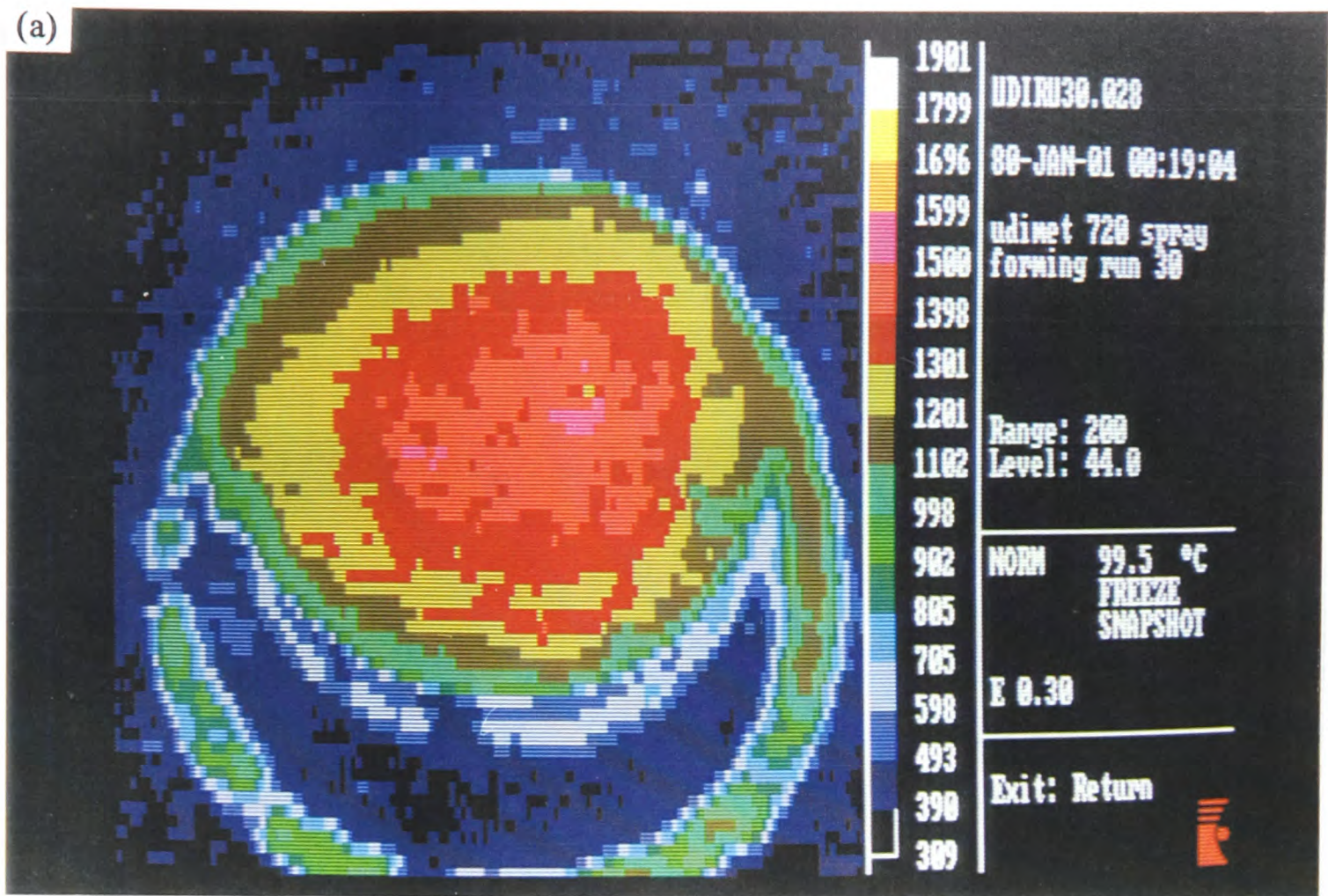
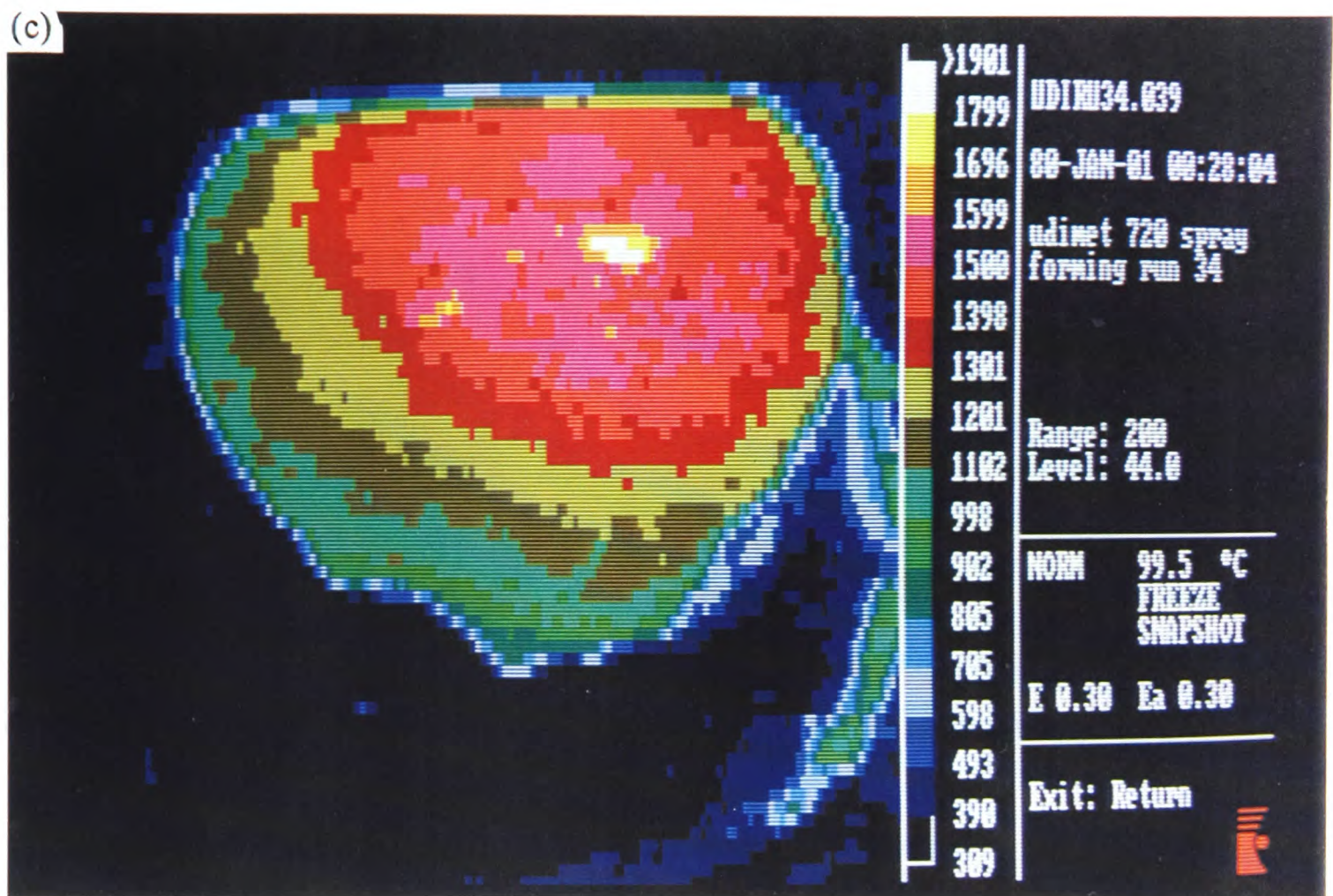


Fig 5.10 Individual thermal images taken from the middle of the run for: (a) run 30, (b) run 32 and (c) run 34.





cont... Fig 5.10 Individual thermal images taken from the middle of the run for: (a) run 30, (b) run 32 and (c) run 34.



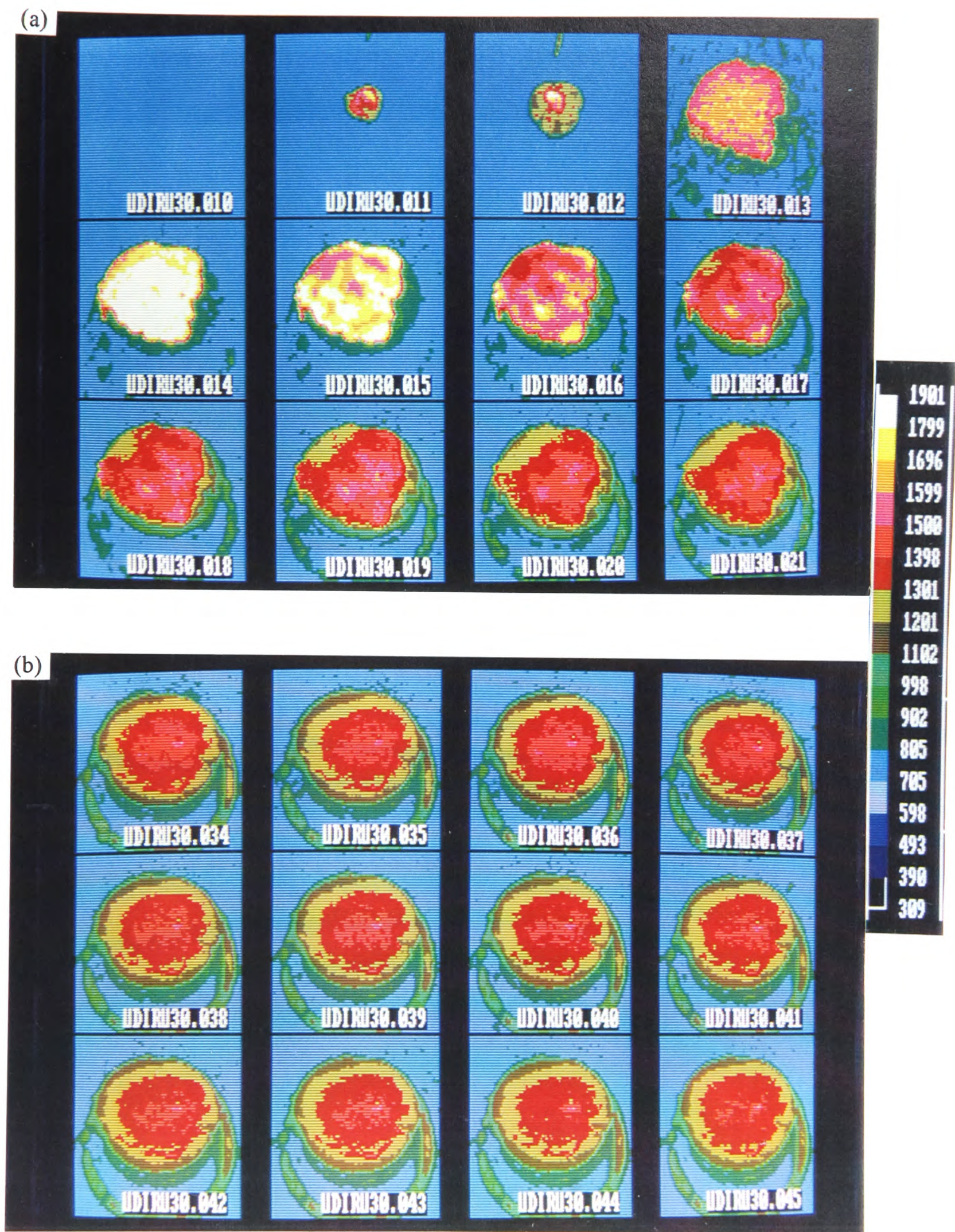
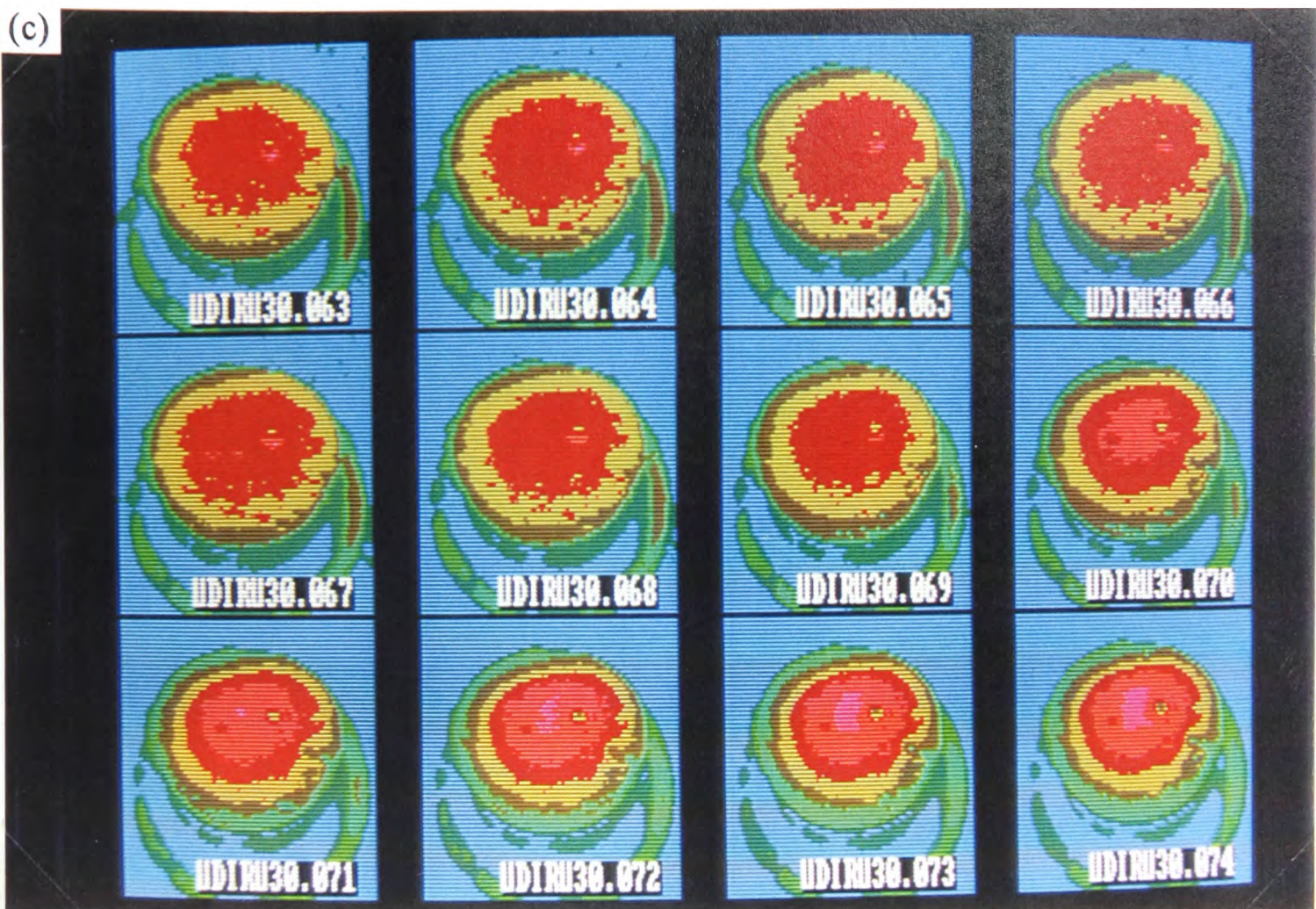


Fig 5.11 Typical sequence of thermal images, assuming  $\epsilon_0=0.3$  for run 30: (a) at the beginning of the run, (b) in the middle of the run and (c) just after the end of the run.



(c)



cont... Fig 5.11 Typical sequence of thermal images, assuming  $\epsilon_o=0.3$  for run 30: (a) at the beginning of the run, (b) in the middle of the run and (c) just after the end of the run.



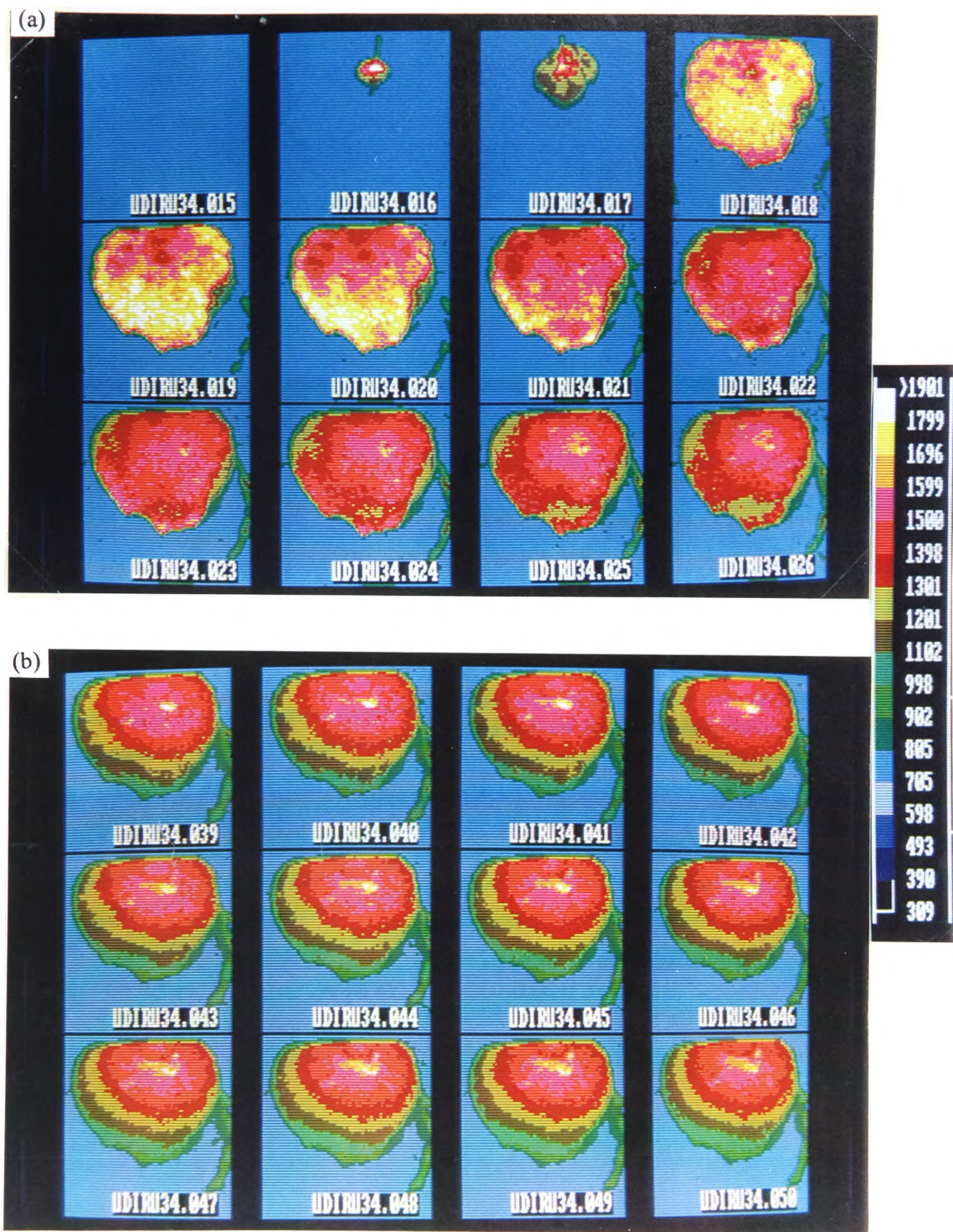


Fig 5.12 Typical sequence of thermal images, assuming  $\epsilon_0=0.3$  for run 34: (a) at the beginning of the run, (b) in the middle of the run and (c) just after the end of the run.



(c)



cont... Fig 5.12 Typical sequence of thermal images, assuming  $\epsilon_0=0.3$  for run 34: (a) at the beginning of the run, (b) in the middle of the run and (c) just after the end of the run.

$\epsilon_o=0.3$ . They show that the thermal intensities at the beginning and end of the runs were much higher than during the middle of the run for the reasons discussed in section 5.2.1, and that the temperature during the middle part of the run for run 30 in fig 5.11(b) decreased slightly and for run 34 in fig 5.12(b) was approximately constant as also shown in fig 5.9.

Calibration of the thermal imaging temperatures was performed using the procedure described in section 3.2.2.2. i.e. correlating the maximum temperatures of fully embedded thermocouples from each run with the respective mean temperatures for that time assuming  $\epsilon_o=0.3$ . Fig 5.13 shows the mean uncalibrated thermal imaging temperature against the maximum embedded thermocouple temperature of fully embedded thermocouples using runs 16 and 24-37. A best fit straight line is shown through the data points which followed the equation:

$$T_{th} = 2.214T_{em} - 1270.3 \quad (5.1)$$

where  $T_{th}$  is the uncalibrated thermal imaging temperature and  $T_{em}$  is the embedded thermocouple temperature. This equation was then used to determine the calibrated thermal imaging temperature,  $T_{cal}$  for all temperatures:

$$T_{cal} = (T_{th} + 1270.3)/2.214 \quad (5.2)$$

Figs 5.14(a)-(l) show calibrated and uncalibrated thermal imaging temperatures and embedded thermocouple measurements for all runs in which thermal imaging was used. The solidus and liquidus temperatures and the start and end of the runs are indicated by horizontal and vertical lines respectively. The maximum temperatures of fully embedded thermocouples agree well with the corresponding thermal imaging temperatures as indicated by the calibration curve in fig 5.13



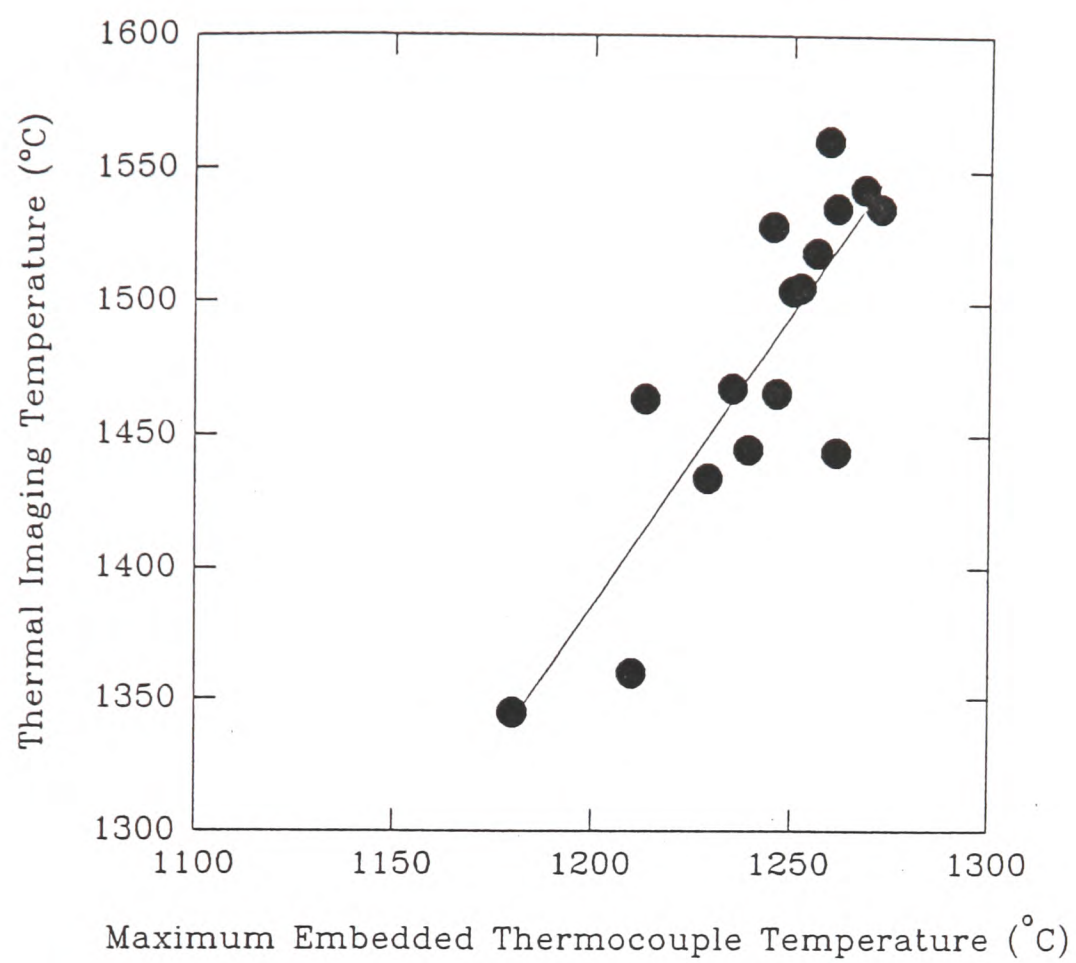


Fig 5.13 Calibration curve of, thermal imaging temperature at the position of  $T_{\max}$  against  $T_{\max}$  at that point.

Figs 5.14(a), (b) and (h)-(k) for runs 16, 24 and 32-35 respectively show that excluding the very start of the run the calibrated top surface thermal imaging temperature was always in the solid/liquid region. In figs 5.14(b), (i) and (j) for runs 24, 33 and 34 respectively, the temperature was approximately constant for most of the run, decreased slightly towards the end of the run and then increased rapidly either just before or after the run finished. In figs 5.14(a), (h) and (k) for runs 16, 32 and 35 respectively the temperature was approximately constant for most of the run and then increased rapidly either just before or after the run finished.

Figs 5.14(c)-(g) and (l) for runs 25-31 and 37 respectively show that excluding the very start of the run, the calibrated top surface thermal imaging temperature was always very close to  $T_{\text{sol}}$  (solidus temperature-1225°C). In figs 5.14(d), (f), (g) and (l) for runs 27, 30, 31 and 37 the top surface temperature was approximately constant for half of the run, decreased towards the end of the run and then increased rapidly either just before or after the run finished. In fig 5.14(e) for run 28 the top surface temperature was approximately constant for most of the run and then increased rapidly just as the run finished, similar to figs 5.14(a), (h) and (k) for runs 16, 32 and 35 respectively but at a lower temperature.

### **5.3.3 Deposit Temperatures**

Embedded thermocouple measurements give the thermal history of a point in the deposit while thermal imaging gives the variation in deposit top surface temperature. Combining the two measurements gives a more complete picture of the deposit thermal history. Since thermal imaging data was not available for all of the deposits in this study, embedded thermocouple data was used to illustrate the variation of process parameters and

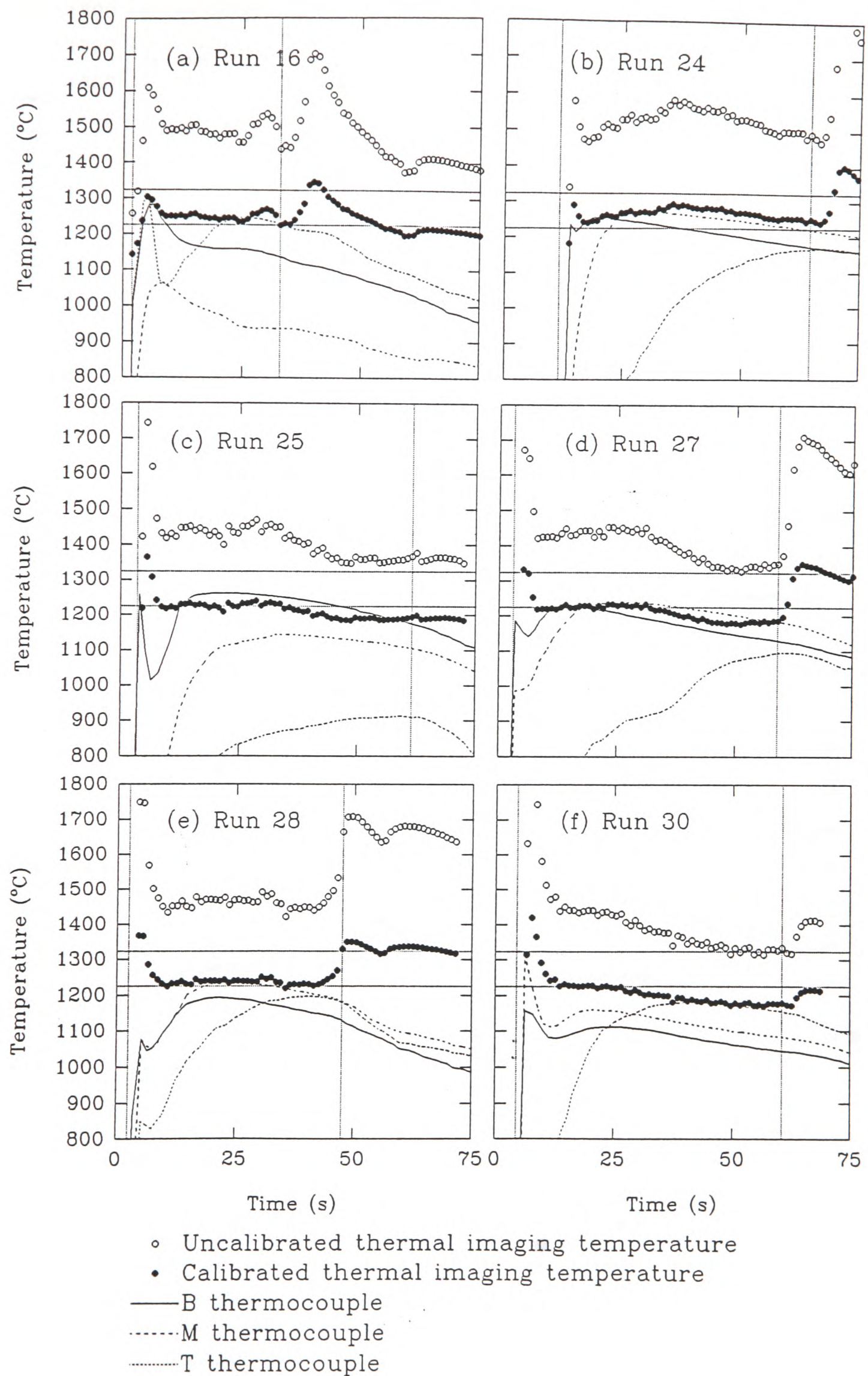
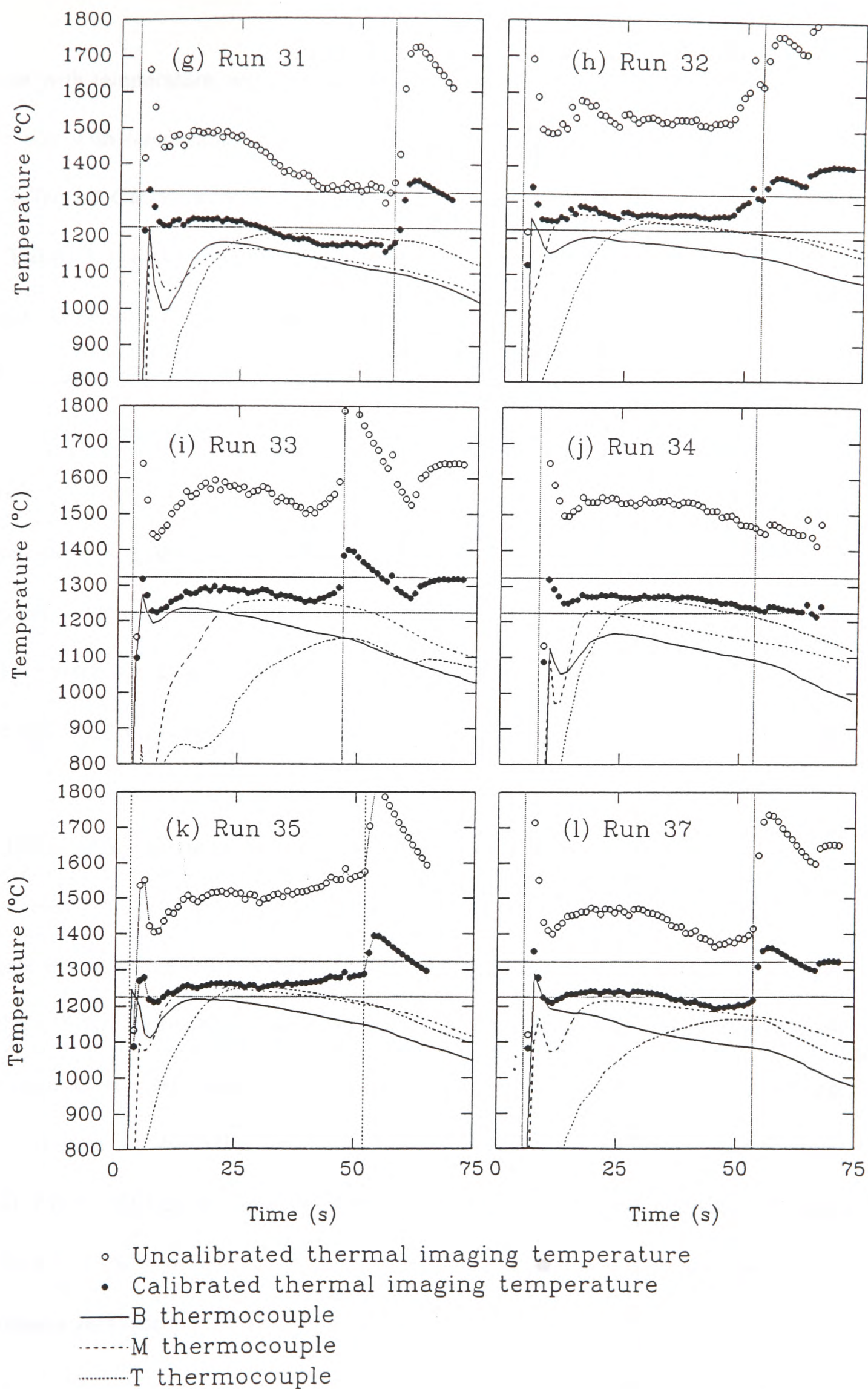


Fig 5.14 Uncalibrated and calibrated thermal imaging temperature and embedded thermocouple temperature against time for: (a) run 16, (b) run 24, (c) run 25, (d) run 27, (e) run 28, (f) run 30.





cont... Fig 5.14 Uncalibrated and calibrated thermal imaging temperature and embedded thermocouple temperature against time for: (g) run 31, (h) run 32, (i) run33, (j) run 34, (k) run 35 and (l) run 37.

grain size with temperature, and thermal imaging data was used to illustrate the variation of grain size with temperature through the thickness of deposits.

### 5.3.3.1 Effect of Gas Flow Rate

Tables 5.4(a)-(c) show the details of 3 groups of runs taken from table 5.3 to investigate the effect of GFR at spray distances and MFR's of: 0.2m and  $\approx 41\text{g/s}$ ; 0.25m and  $\approx 41\text{g/s}$ ; and 0.3m and  $\approx 41\text{g/s}$  respectively. For each table all process parameters were constant except GFR, although there was some variation in the MFR, and table 5.4(a) contains run 33 which had a lower superheat. Fig 5.15(a) shows the variation of  $T_{\max}$  (indicated by bold type in tables 5.4-5.7) against GFR for tables 5.4(a)-(c).  $T_{\text{sol}}$  is marked by a dashed horizontal line. At spray distances and MFR's of 0.25m and  $\approx 41\text{g/s}$  and 0.3m and  $\approx 41\text{g/s}$  there was a decrease in temperature as the GFR increased whereas at a spray distance and MFR of 0.2m and  $\approx 41\text{g/s}$  there was little change in temperature as the GFR increased.

### 5.3.3.2 Effect of Spray Distance

Tables 5.5(a)-(d) show the details of 4 groups of runs taken from table 5.3 to investigate the effect of spray distance at GFR's and MFR's of: 5.3l/s and  $\approx 59\text{g/s}$ ; 4.8l/s and  $\approx 41\text{g/s}$ ; 5.3l/s and  $\approx 39\text{g/s}$ ; and 4.8l/s and  $\approx 50\text{g/s}$  respectively. For each table all process parameters were constant except spray distance, although once again there was a slight variation in the MFR and table 5.5(b) contains run 33 which had a lower superheat. Fig 5.15(b) again shows the variation of  $T_{\max}$  against spray distance for tables 5.5(a)-(d). Fig 5.15(b) shows that in all cases there was a decrease in temperature as the spray distance increased, with a stronger effect at high spray distances.



(a)

| Parameters            | Run No.     |             |             |             |
|-----------------------|-------------|-------------|-------------|-------------|
|                       | 33**        | 32*         | 35*         | 34*         |
| Spray distance (m)    | 0.2         | 0.2         | 0.2         | 0.2         |
| Gas flow rate (l/s)   | 4.8         | 5.3         | 4.8         | 5.3         |
| Melt flow rate (g/s)  | 41          | 45          | 41          | 38          |
| Deposition rate (g/s) | 21          | 25          | 20          | 21          |
| Max temp-B (C)        | 1237        | 1205        | 1218        | 1168        |
| Max temp-M (C)        | <b>1259</b> | <b>1268</b> | 1250        | 1232        |
| Max temp-T (C)        | 1153        | 1245        | <b>1252</b> | <b>1261</b> |
| Mass median size (um) | 134         | 122         | 136         | 115         |

(b)

| Parameters            | Run No.     |             |             |
|-----------------------|-------------|-------------|-------------|
|                       | 25          | 27*         | 31*         |
| Spray distance (m)    | 0.25        | 0.25        | 0.25        |
| Gas flow rate (l/s)   | 4.8         | 4.8         | 5.3         |
| Melt flow rate (g/s)  | 42          | 43          | 39          |
| Deposition rate (g/s) | 14          | 16          | 16          |
| Max temp-B (C)        | <b>1262</b> | 1229        | 1185        |
| Max temp-M (C)        | 1145        | <b>1239</b> | 1165        |
| Max temp-T (C)        | 911         | 1096        | <b>1210</b> |
| Mass median size (um) | 145         | 140         | 122         |

(c)

| Parameters            | Run No.     |             |
|-----------------------|-------------|-------------|
|                       | 28*         | 30*         |
| Spray distance (m)    | 0.3         | 0.3         |
| Gas flow rate (l/s)   | 4.8         | 5.3         |
| Melt flow rate (g/s)  | 44          | 39          |
| Deposition rate (g/s) | 14          | 14          |
| Max temp-B (C)        | 1194        | 1150        |
| Max temp-M (C)        | <b>1235</b> | 1160        |
| Max temp-T (C)        | 1190        | <b>1180</b> |
| Mass median size (um) | 140         | 133         |

B=5mm, M=10mm & T=15mm except: \*-B=5mm, M=7.5mm & T=10mm.  
 Superheat=1445C except: \*\* - superheat =1403C.

Tables 5.4(a)-(c) Summary of spray forming process parameters, B, M and T thermocouple maximum temperatures and mass median overspray size for 3 groups of runs where gas flow rate was the only independent variable. (Tmax temperatures indicated in bold type)

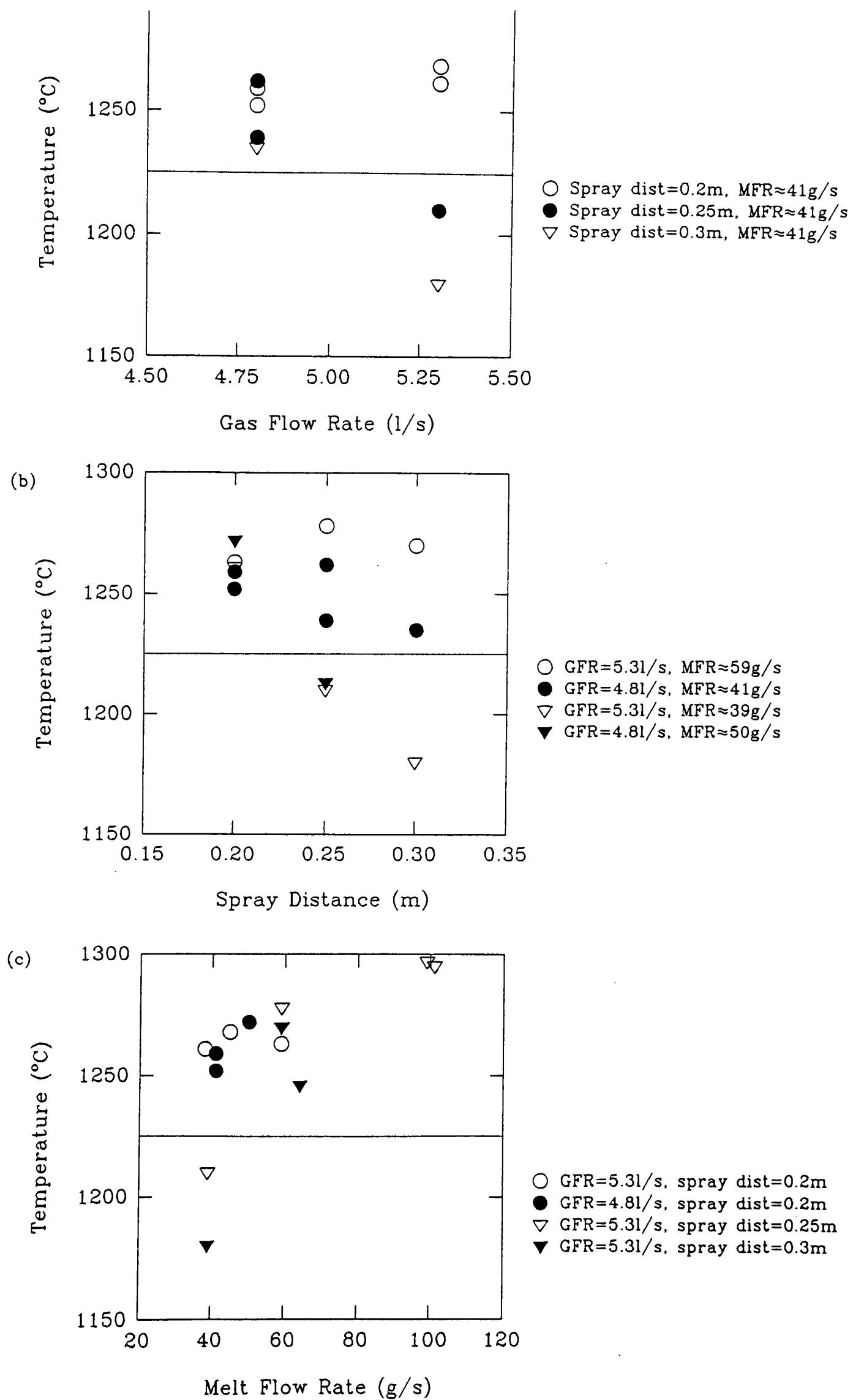


Fig 5.15  $T_{\max}$  against: (a) gas flow rate, (b) spray distance, and (c) melt flow rate. The dashed horizontal line indicates the solidus temperature.

(a)

| Parameters            | Run No.     |             |             |
|-----------------------|-------------|-------------|-------------|
|                       | 17          | 21          | 23          |
| Spray distance (m)    | 0.3         | 0.25        | 0.2         |
| Gas flow rate (l/s)   | 5.4         | 5.3         | 5.3         |
| Melt flow rate (g/s)  | 59          | 59          | 59          |
| Deposition rate (g/s) | 23          | 27          | 32          |
| Max temp-B (C)        | -           | 1264        | <b>1263</b> |
| Max temp-M (C)        | 1260        | 1278        | 1241        |
| Max temp-T (C)        | <b>1270</b> | <b>1278</b> | 1199        |
| Mass median size (um) | 147         | 144         | 157         |

(b)

| Parameters            | Run No.     |             |             |             |             |
|-----------------------|-------------|-------------|-------------|-------------|-------------|
|                       | 28*         | 25          | 27*         | 33**        | 35*         |
| Spray distance (m)    | 0.3         | 0.25        | 0.25        | 0.2         | 0.2         |
| Gas flow rate (l/s)   | 4.8         | 4.8         | 4.8         | 4.8         | 4.8         |
| Melt flow rate (g/s)  | 44          | 42          | 43          | 41          | 41          |
| Deposition rate (g/s) | 14          | 14          | 16          | 21          | 20          |
| Max temp-B (C)        | 1194        | <b>1262</b> | 1229        | 1237        | 1218        |
| Max temp-M (C)        | <b>1235</b> | 1145        | <b>1239</b> | <b>1259</b> | 1250        |
| Max temp-T (C)        | 1190        | 911         | 1096        | 1153        | <b>1252</b> |
| Mass median size (um) | 140         | 145         | 140         | 134         | 136         |

(c)

| Parameters            | Run No.     |             |             |
|-----------------------|-------------|-------------|-------------|
|                       | 30*         | 31*         | 34*         |
| Spray distance (m)    | 0.3         | 0.25        | 0.2         |
| Gas flow rate (l/s)   | 5.3         | 5.3         | 5.3         |
| Melt flow rate (g/s)  | 39          | 39          | 38          |
| Deposition rate (g/s) | 14          | 16          | 21          |
| Max temp-B (C)        | 1150        | 1185        | 1168        |
| Max temp-M (C)        | 1160        | 1165        | 1232        |
| Max temp-T (C)        | <b>1180</b> | <b>1210</b> | <b>1261</b> |
| Mass median size (um) | 133         | 122         | 115         |

(d)

| Parameters            | Run No.     |             |
|-----------------------|-------------|-------------|
|                       | 24*         | 37*         |
| Spray distance (m)    | 0.2         | 0.25        |
| Gas flow rate (l/s)   | 4.8         | 4.8         |
| Melt flow rate (g/s)  | 50          | 50          |
| Deposition rate (g/s) | 25          | 17          |
| Max temp-B (C)        | 1256        | 1180        |
| Max temp-M (C)        | <b>1272</b> | <b>1213</b> |
| Max temp-T (C)        | 1170        | 1165        |
| Mass median size (um) | 140         | 146         |

B=5mm, M=10mm & T=15mm except: \*-B=5mm, M=7.5mm & T=10mm.  
 Superheat=1445C except: \*\* - superheat =1403C.

Tables 5.5(a)-(d) Summary of spray forming process parameters, B, M and T thermocouple maximum temperatures and mass median overspray size for 4 groups of runs where spray distance was the only independent variable.

(a)

| Parameters            | Run No.     |             |             |
|-----------------------|-------------|-------------|-------------|
|                       | 23          | 32*         | 34*         |
| Spray distance (m)    | 0.2         | 0.2         | 0.2         |
| Gas flow rate (l/s)   | 5.3         | 5.3         | 5.3         |
| Melt flow rate (g/s)  | 59          | 45          | 38          |
| Deposition rate (g/s) | 32          | 25          | 21          |
| Max temp-B (C)        | 1263        | 1205        | 1168        |
| Max temp-M (C)        | <b>1241</b> | <b>1268</b> | 1232        |
| Max temp-T (C)        | 1199        | 1245        | <b>1261</b> |
| Mass median size (um) | 157         | 122         | 115         |

(b)

| Parameters            | Run No.     |             |             |
|-----------------------|-------------|-------------|-------------|
|                       | 24          | 33**        | 35*         |
| Spray distance (m)    | 0.2         | 0.2         | 0.2         |
| Gas flow rate (l/s)   | 4.8         | 4.8         | 4.8         |
| Melt flow rate (g/s)  | 50          | 41          | 41          |
| Deposition rate (g/s) | 25          | 21          | 20          |
| Max temp-B (C)        | 1256        | 1237        | 1218        |
| Max temp-M (C)        | <b>1272</b> | <b>1259</b> | 1250        |
| Max temp-T (C)        | 1170        | 1153        | <b>1252</b> |
| Mass median size (um) | 140         | 134         | 136         |

(c)

| Parameters            | Run No.     |             |             |             |
|-----------------------|-------------|-------------|-------------|-------------|
|                       | 19          | 20          | 21          | 31*         |
| Spray distance (m)    | 0.25        | 0.25        | 0.25        | 0.25        |
| Gas flow rate (l/s)   | 5.3         | 5.3         | 5.3         | 5.3         |
| Melt flow rate (g/s)  | 101         | 99          | 59          | 39          |
| Deposition rate (g/s) | 48          | 47          | 27          | 16          |
| Max temp-B (C)        | -           | -           | 1264        | 1185        |
| Max temp-M (C)        | 1244        | 1276        | 1278        | 1165        |
| Max temp-T (C)        | <b>1295</b> | <b>1297</b> | <b>1278</b> | <b>1210</b> |
| Mass median size (um) | 144         | 153         | 144         | 122         |

(d)

| Parameters            | Run No.     |             |             |
|-----------------------|-------------|-------------|-------------|
|                       | 16          | 17          | 30*         |
| Spray distance (m)    | 0.3         | 0.3         | 0.3         |
| Gas flow rate (l/s)   | 5.3         | 5.3         | 5.3         |
| Melt flow rate (g/s)  | 64          | 59          | 39          |
| Deposition rate (g/s) | 25          | 23          | 14          |
| Max temp-B (C)        | -           | -           | 1150        |
| Max temp-M (C)        | 1061        | 1260        | 1160        |
| Max temp-T (C)        | <b>1246</b> | <b>1270</b> | <b>1180</b> |
| Mass median size (um) | 164         | 147         | 133         |

B=5mm, M=10mm & T=15mm except: \*-B=5mm, M=7.5mm & T=10mm.  
 Superheat=1445C except: \*\* - superheat =1403C.

Tables 5.6(a)-(d) Summary of spray forming process parameters, B, M and T thermocouple maximum temperatures and mass median overspray size for 4 groups of runs where melt flow rate was the only independent variable.  
 (Tmax temperatures indicated in bold type)



### 5.3.3.3 Effect of Melt Flow Rate

Tables 5.6(a)-(d) show the details of 4 groups of runs taken from table 5.3 to investigate the effect of MFR at spray distances and GFR's of: 0.2m and 5.3l/s; 0.2m and 4.8l/s; 0.25m and 5.3l/s; and 0.3m and 5.3l/s. For each table all process parameters were constant except MFR, although table 5.6(b) contains run 33 which had a lower superheat. Fig 5.15(c) again shows the variation of  $T_{\max}$  against MFR for tables 5.6(a)-(d). Fig 5.15(c) shows that in all cases there was an increase in temperature as the MFR increased.

### 5.3.3.4 Effect of Deposition Rate

Fig 5.16(a) is a summary of all the data in figs 5.15(a)-(c) to show the variation of  $T_{\max}$  with deposition rate despite the variation in the other process parameters. Again,  $T_{\text{sol}}$  is marked by a dashed horizontal line. There was a parabolic increase in temperature with increasing deposition rate.  $T_{\max}$  was converted to a solid fraction using a best fit Scheil equation to the solid fraction against temperature curve as shown in fig 3.12. Fig 5.16(b) then shows that the solid fraction increased approximately linearly with decreasing deposition rate.

## 5.3.4 Porosity

The variation of porosity in spray formed deposits was characterised in two ways: (i) as a function of normalised deposit thickness and (ii) as a function of spray forming conditions e.g. temperature and deposition rate.

Figs 5.17(a) and (b) show the variation in average area fraction porosity with normalised deposit height for runs 16-27 and 28-37 respectively. For all spray runs the porosity was high initially ( $\approx 10\%$ ), corresponding to the region close to the substrate at

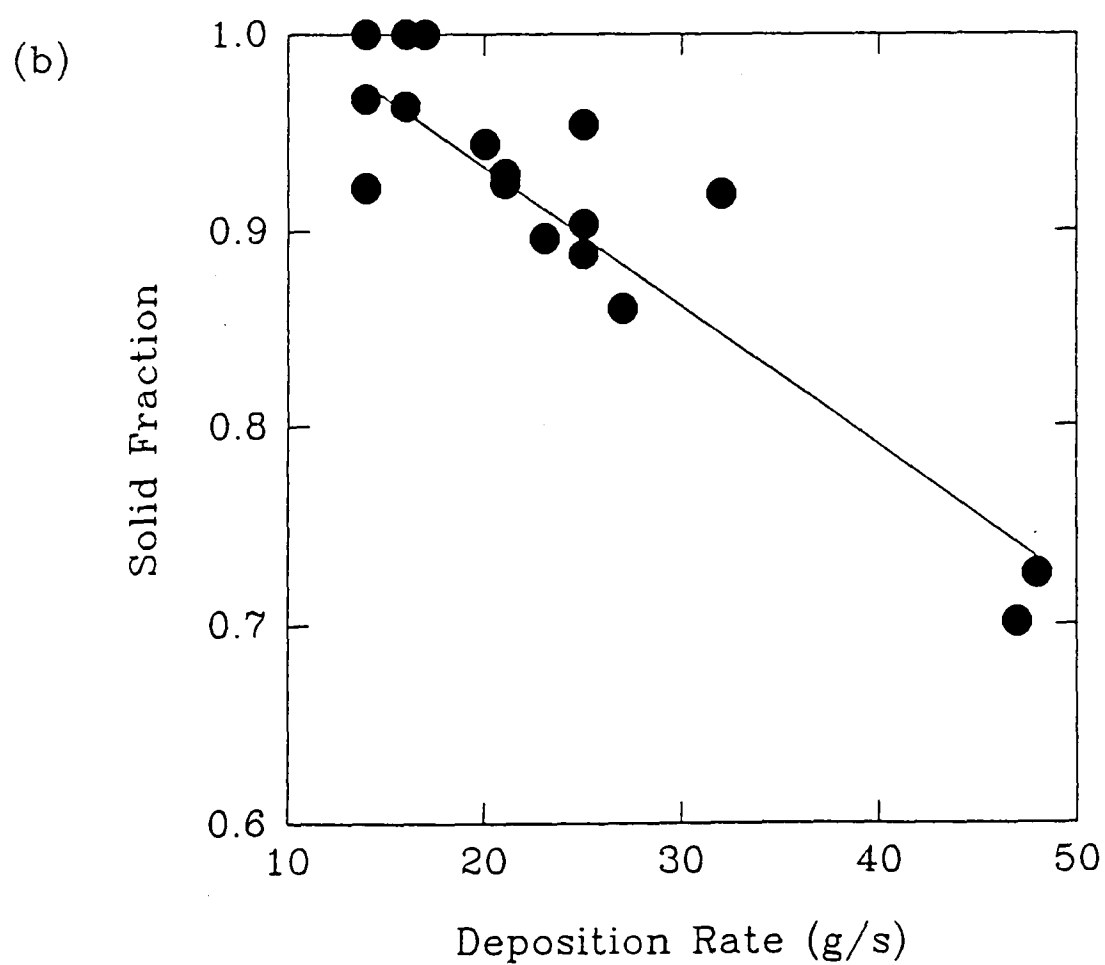
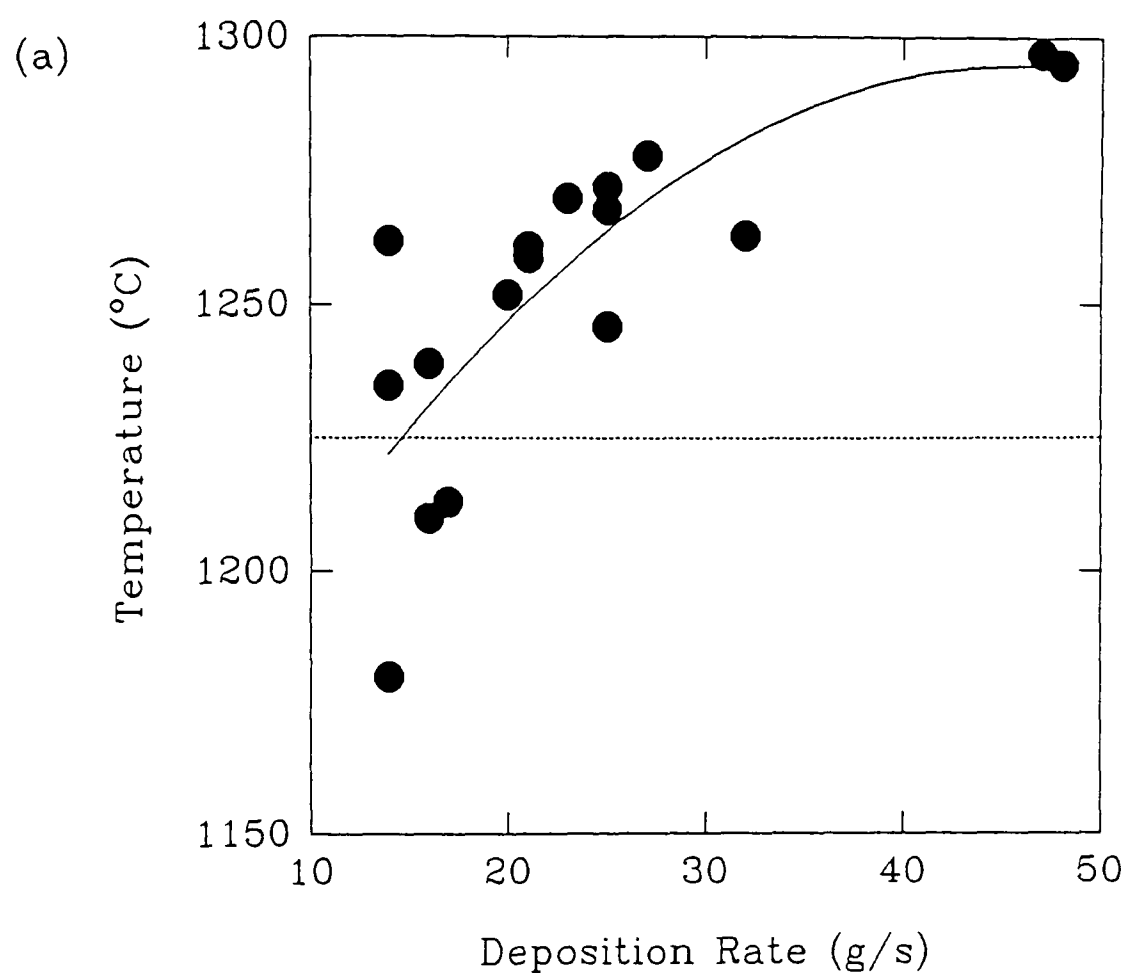


Fig 5.16 Deposition rate against: (a)  $T_{\max}$  and (b) solid fraction.

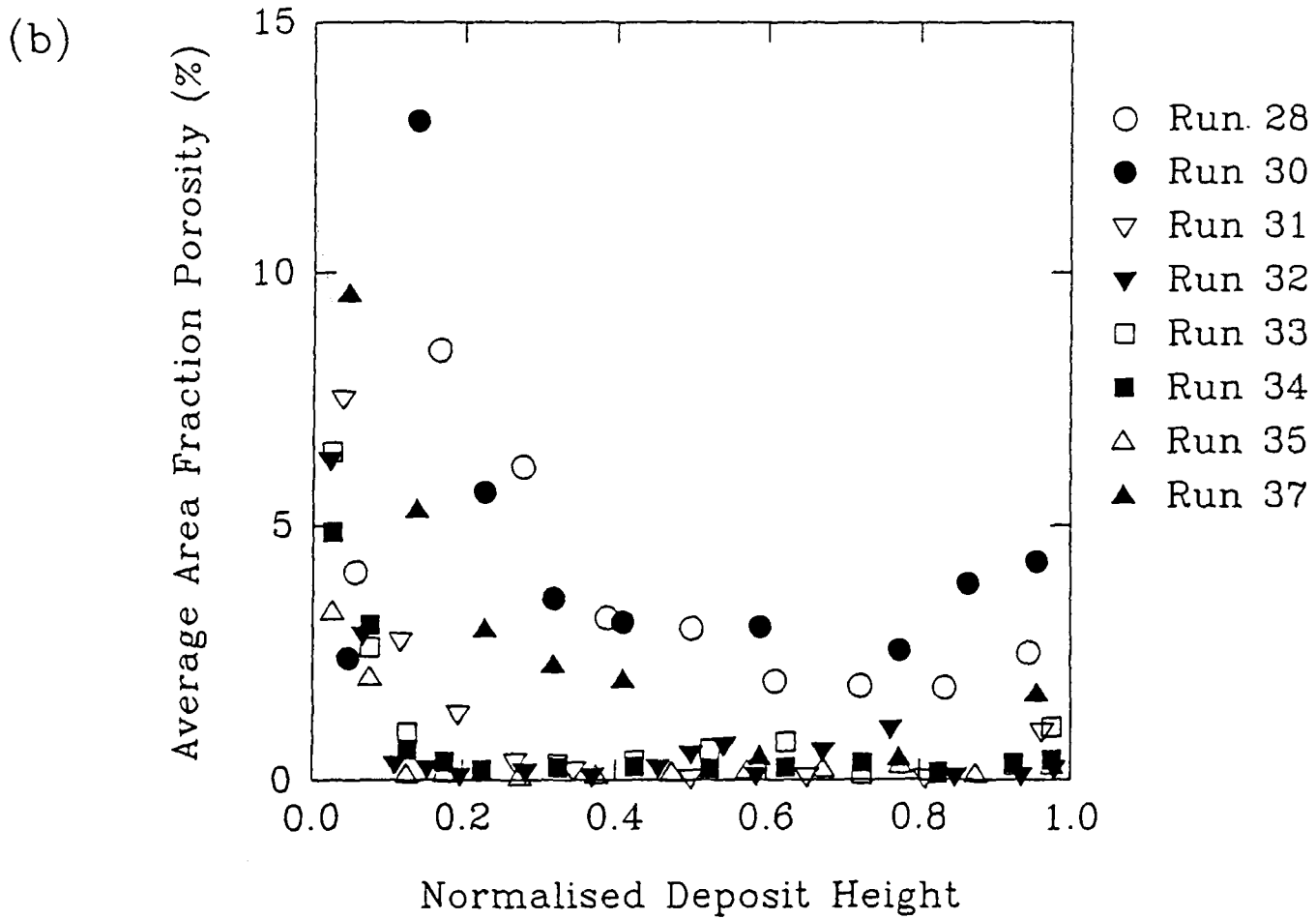
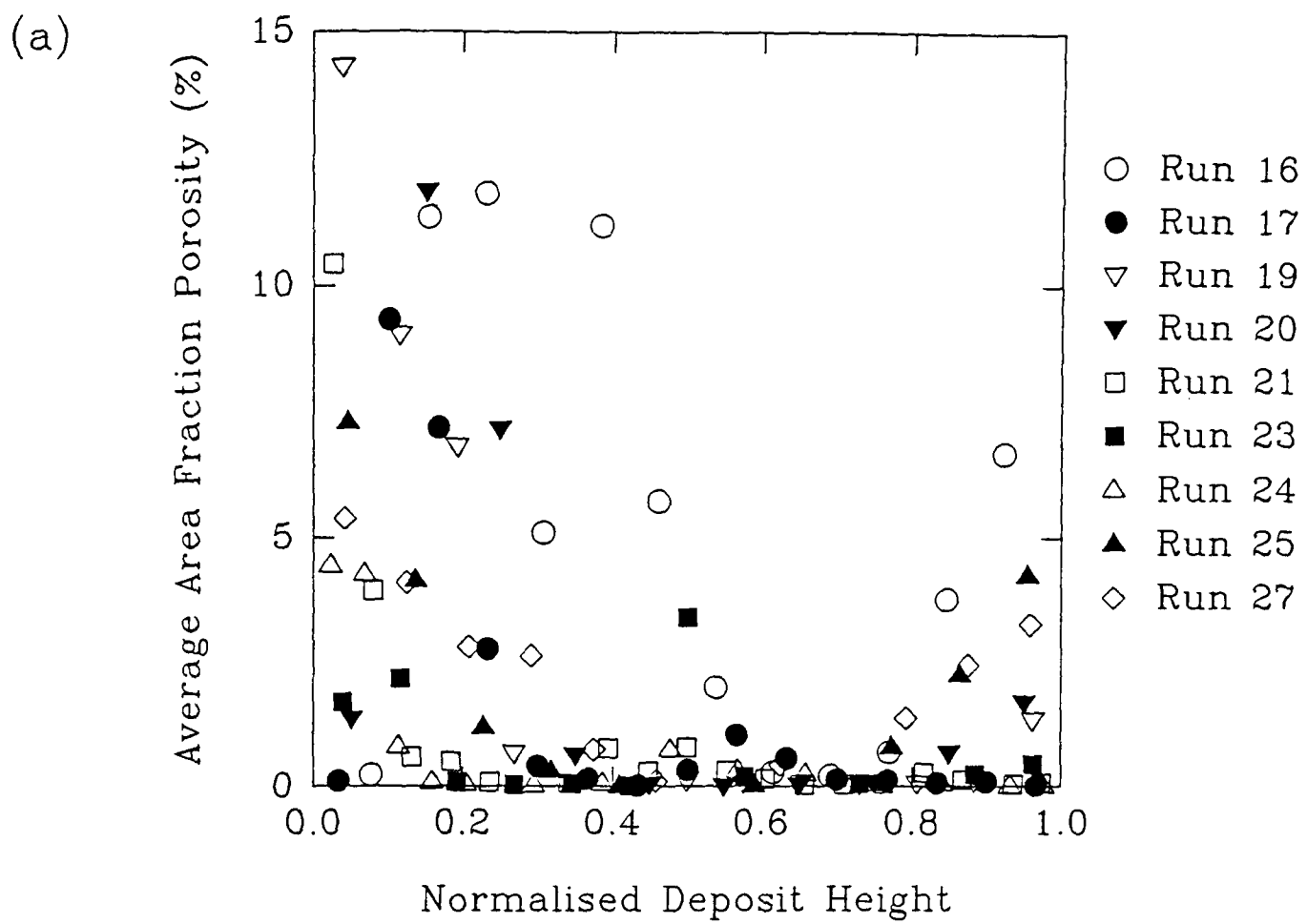


Fig 5.17 Average area fraction porosity against normalised deposit height for: (a) runs 16-27 and (b) runs 28-37.

the beginning of spray forming. As spray forming proceeded, either the porosity became very low (e.g. <1% for runs 17-24) or fell to moderate levels (e.g. 2-4% for runs 27-31) for the major part of the run. In most cases, the porosity increased slightly again near the end of the run close to the deposit top surface. For example, for run 19 the average area fraction porosity was 14.3% initially, 0.13% in the middle of the deposit and 1.4% at the end of the deposit. As another example for run 30 the average area fraction porosity was 13% initially, 3.1% in the middle of the deposit and 4.3% at the end of the deposit.

Fig 5.18 shows the average area fraction porosity, measured at the position of  $T_{\max}$  against (a) deposition rate and (b)  $T_{\max}$ . Fig 5.18(a) shows that below a deposition rate of  $\approx 20\text{g/s}$  the porosity was relatively high (2-4%) whereas above  $\approx 20\text{g/s}$  the porosity was <1%. For example, at 25g/s the porosity was 0.18% (run 32) and at 14g/s the porosity was 3.18% (run 28). Correspondingly, fig 5.18(b) shows that below a temperature of  $\approx 1250^\circ\text{C}$  ( $f > \approx 0.95$ ) the porosity was 2-4%, and reduced to <1% at temperatures above  $\approx 1250^\circ\text{C}$  ( $f < \approx 0.95$ ). For example, at  $1268^\circ\text{C}$  the porosity was 0.18% (run 32) and at  $1235^\circ\text{C}$  the porosity was 3.18% (run 28). The relationship of porosity with temperature was more closely obeyed than with deposition rate. This is illustrated by runs 16 and 25 which had porosities of 2.02% and 0.75% at deposition rates of 25g/s and 15g/s respectively. However,  $T_{\max}$  was  $1246^\circ\text{C}$  and  $1262^\circ\text{C}$  respectively. However, run 31 still didn't fit in with the above variation as its porosity was <1% at a deposition rate of 16g/s and  $T_{\max}$  of  $1210^\circ\text{C}$ .

Fig 5.19 shows typical unetched deposit microstructures exhibiting: (a)-(b) low (<1%) porosity; (c)-(d) moderate (2-4%) porosity all taken at the position of  $T_{\max}$ ; and (f) high porosity ( $\approx 10\%$ ) taken from the region at the beginning of spray forming. Figs

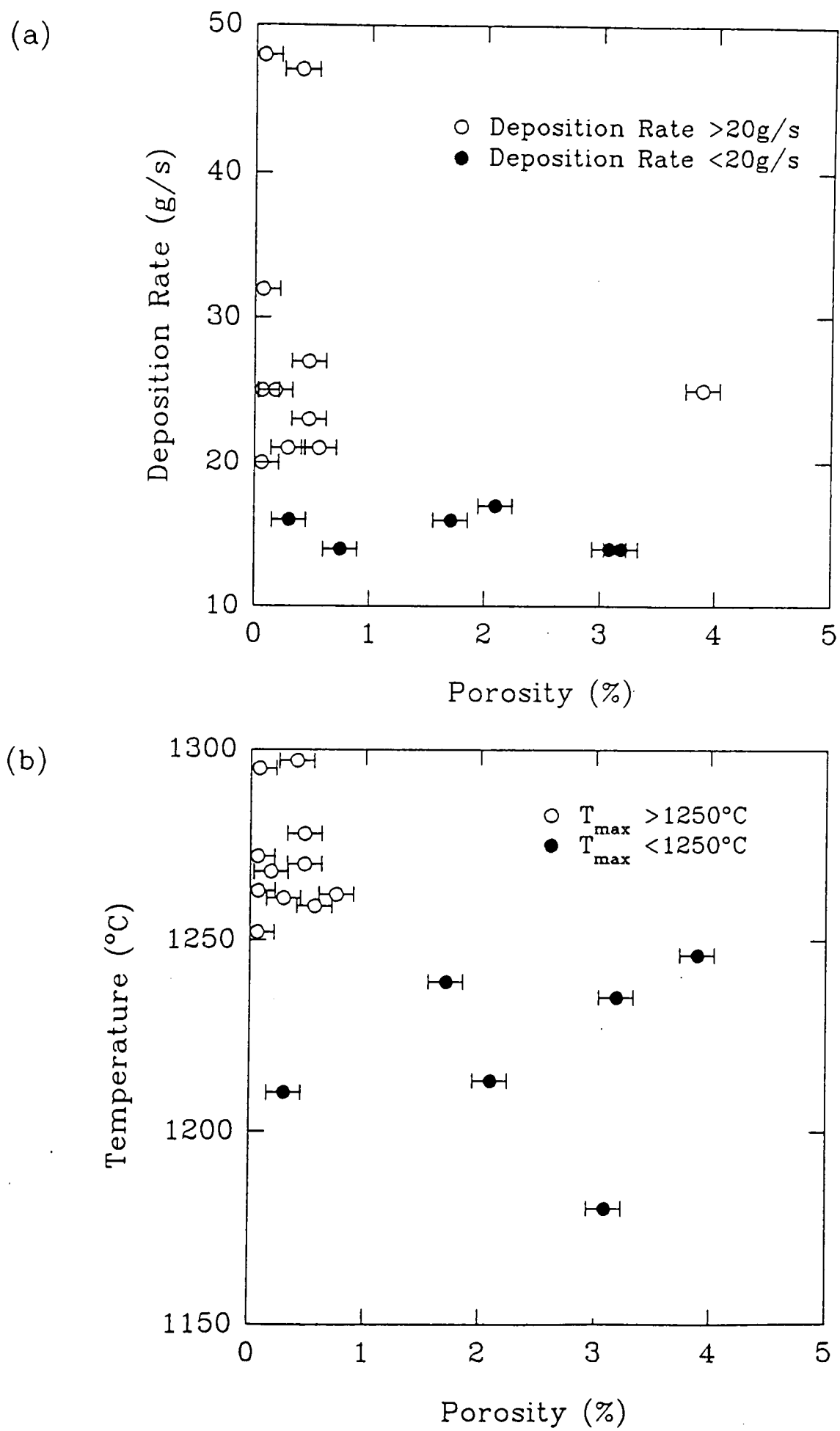


Fig 5.18 Mean average area fraction porosity against (a) deposition rate and (b)  $T_{\max}$ .



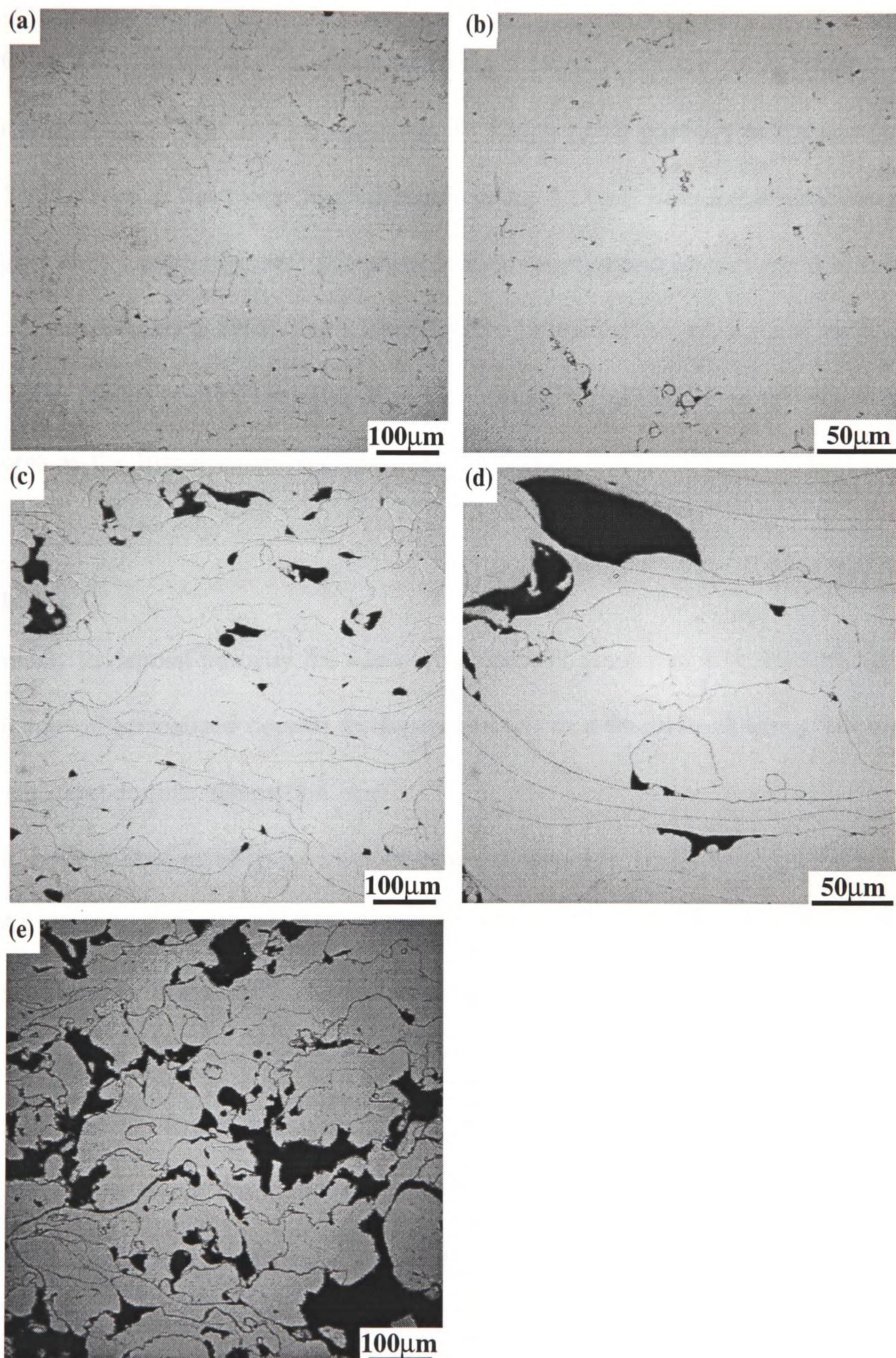


Fig 5.19 Micrographs taken at the position of  $T_{\max}$  for: (a) run 32 at a magnification of x88, (b) run 32 at a magnification of x200, (c) run 30 at a magnification of x88, (d) run 30 at a magnification of x200 and (e) at the beginning of spray forming for run 19.



5.19(a) and (b) show run 32 which had an average area fraction porosity at the position of  $T_{\max}$  of 0.2%. Only at higher magnification in fig 5.19(b) could the small amount of porosity be seen. Figs 5.19(c) and (d) show run 30 which had a porosity at the position of  $T_{\max}$  of 3.1%. Even at the lower magnification in fig 5.19(c), irregular shaped, large pores ( $\approx 50\mu\text{m}$ ) were visible together with pre-solidified droplets and smaller pores at splat boundaries as shown in fig 5.19(d). Fig 5.19(e) for run 19 is a typical example of the high porosity ( $\approx 10\%$ ) region at the beginning of spray forming which consisted of very large irregular pores as the first droplets splatted onto the deposit.

### **5.3.5 Grain Size**

Similarly to deposit porosity the variation of deposit grain size was characterised:

(i) as a function of normalised deposit thickness and (ii) as a function of spray forming conditions e.g. temperature, deposition rate.

Four distinct regions of grain microstructure in deposits 16-37 were identified.

**Region I.** As described in section 3.1.4, metal was poured through the nozzle prior to atomisation to prevent "freeze off" of metal in the nozzle. This created a thin (2mm) hot region at the start of each run. Fig 5.20(a) shows the dendritic microstructure of this region next to the substrate and was typical of all deposits, indicating the highly directional heat flow away from the deposit/substrate interface. The breakdown and coarsening of the columnar dendrites is shown in fig 5.20(b).

**Region II.** Fig 5.21(a) for run 24, shows the transition that occurred as the atomising gas was turned on. The first droplets splatted onto region I giving a high level of intersplat porosity and small grains within the splats. Fig 5.21(b) for run 24 shows the typical



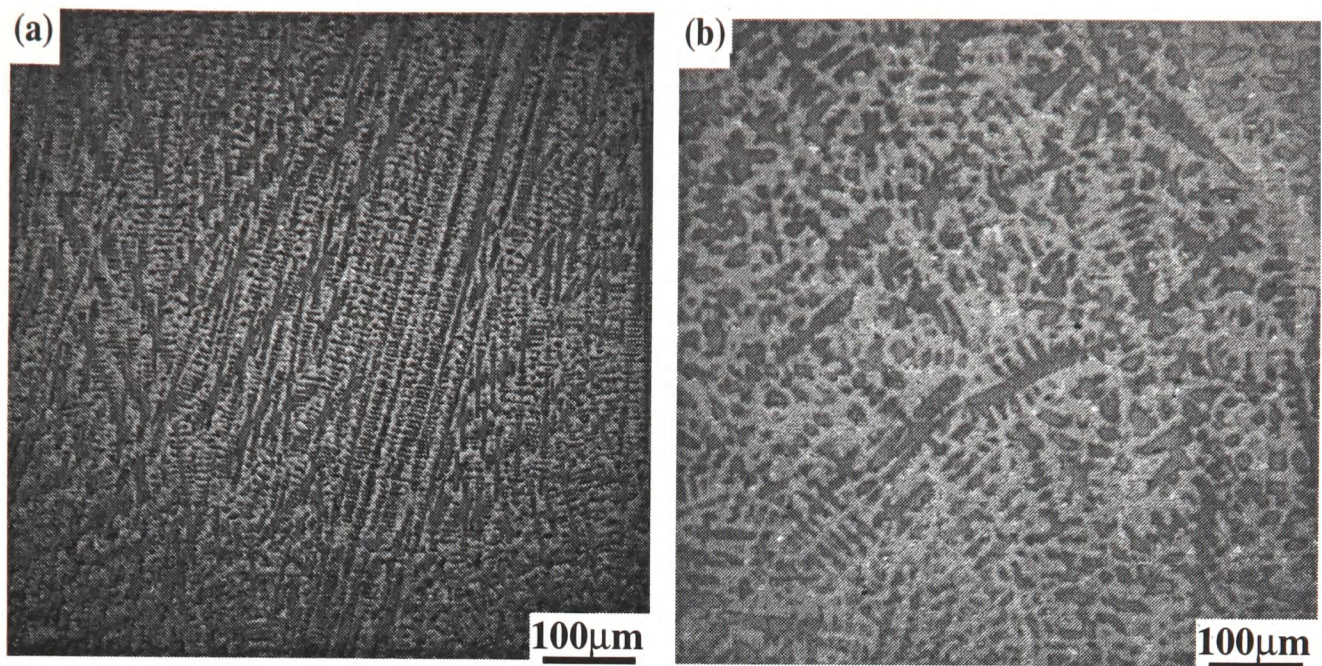


Fig 5.20 Micrograph of region I before spraying started for (a) run 37 and (b) run 17. Etchant 10% Orthophosphoric acid.

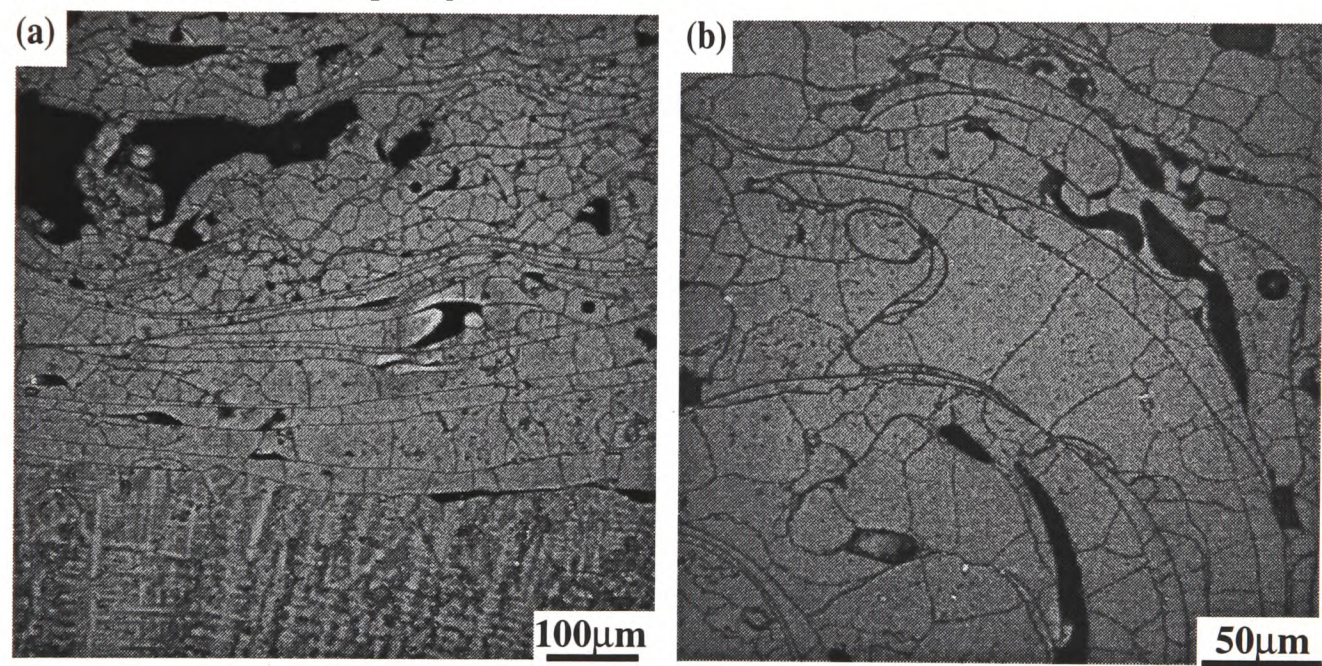


Fig 5.21 Micrograph of (a) the transition from region I to region II for run 24 and (b) region II for run 24. Etchant 10% Orthophosphoric acid.

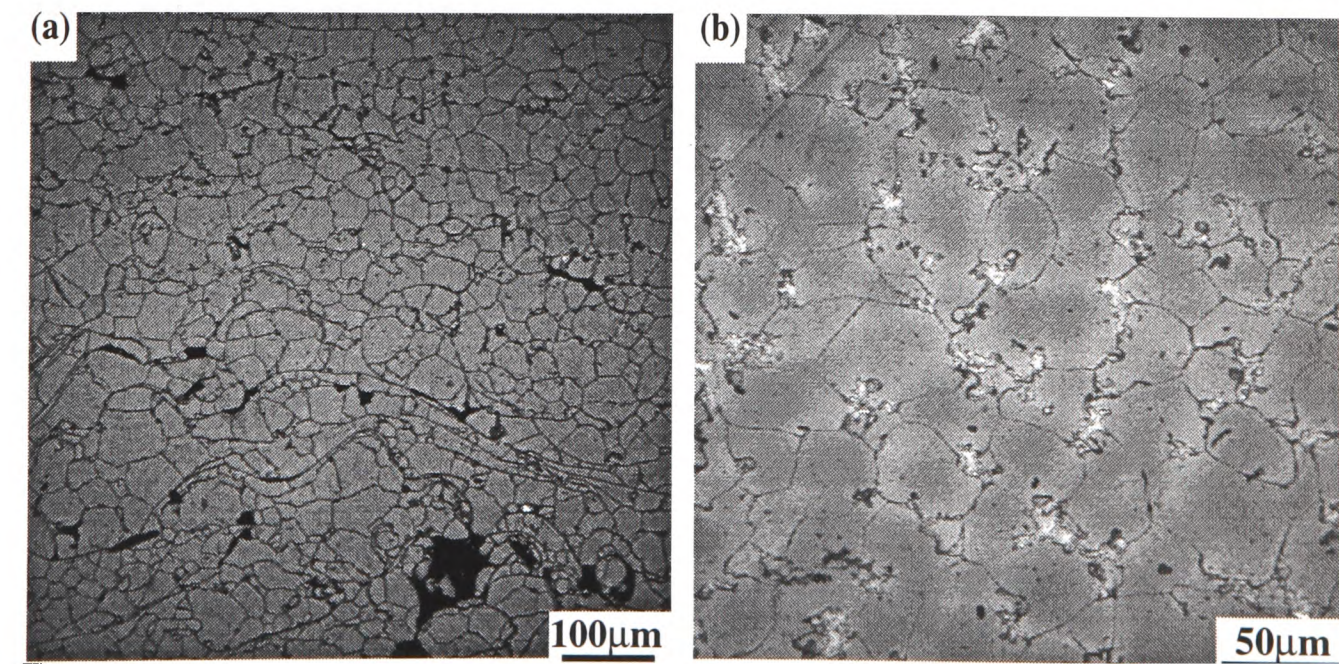


Fig 5.22 Micrograph of (a) the transition from region II to region III for run 19 and (b) region III for run 19 showing an equiaxed grain structure. Etchant 10% Orthophosphoric acid.



microstructure of region II. It consisted of splats with an internal equiaxed grain structure and a high level of porosity. Region II was typically 3mm thick and was also similar for all deposits.

Region III. This was the largest region in the deposit and accounted for approximately 85% of the deposit by thickness. The microstructure in this region was either equiaxed or remained as a splat structure similar to region II, although the porosity was lower. Fig 5.22(a) shows the transition from the splat structure in region II to the equiaxed structure in this case for run 19. Splat boundaries and a relatively high level of porosity were present at the bottom of the micrograph, while towards the top small equiaxed grains were observed and there was a decrease in porosity. The equiaxed grains in region III also showed small grain boundary particles, probably carbides, as shown in fig 5.22(b). Towards the end of this region the grain size was smaller. Figs 5.23(a) and (b), for run 27 show the non-equiaxed region III microstructure with large droplet splats similar to region II, although with a lower porosity. Small grain boundaries particles similar to those in fig 5.22(b) were also present when the sample was defocused. Towards the end of this region the grains within the splats became smaller.

Region IV This region corresponded to the end of the spray run and was typically 2mm thick. Similarly to region III, region IV comprised either smaller equiaxed grains, with a slightly higher porosity, as shown in fig 5.24(a) for run 34, or a mixture of splats and pre-solidified droplets as shown in fig 5.24(b) for run 30, for those deposits that had a splat structure in region III.

Figs 5.25(a)-(e) and figs 5.26(a)-(e) show composite micrographs of the typical through thickness variation in microstructure for runs 24 and 34 respectively. Both



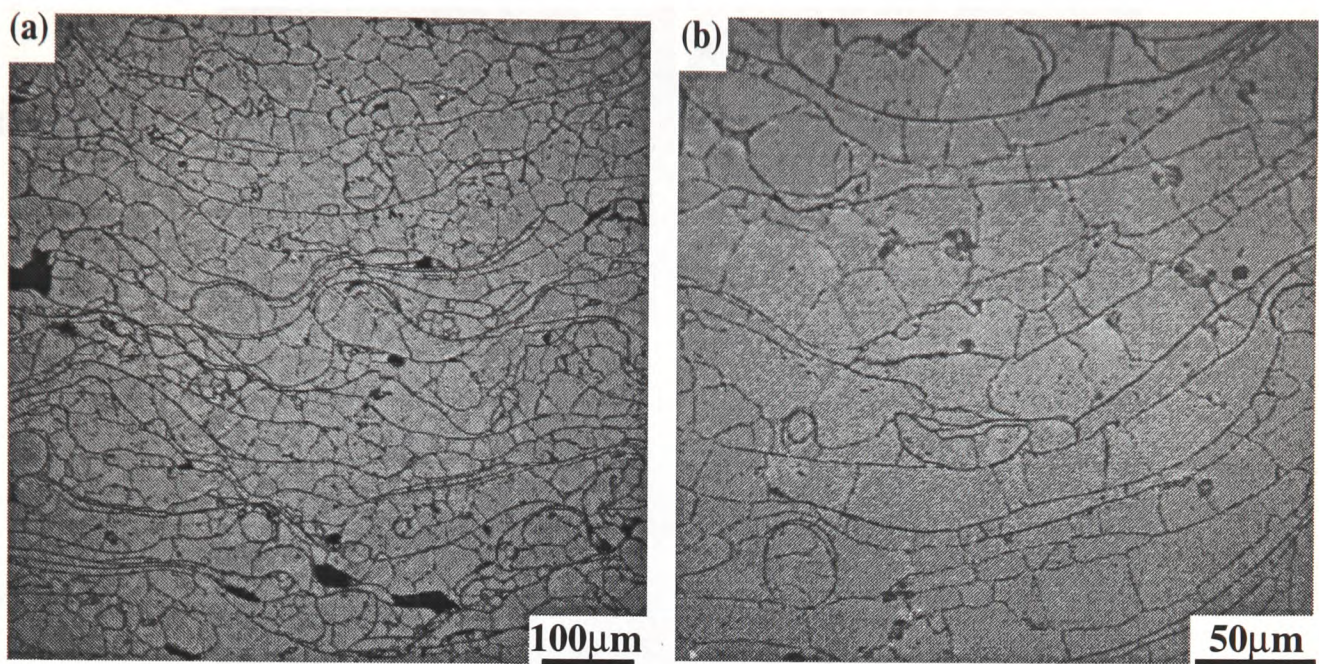


Fig 5.23 Micrograph of (a) region III for run 27 at a magnification of x88 and (b) region III for run 27 at a magnification of x200 showing a splat structure. Etchant 10% Orthophosphoric acid.

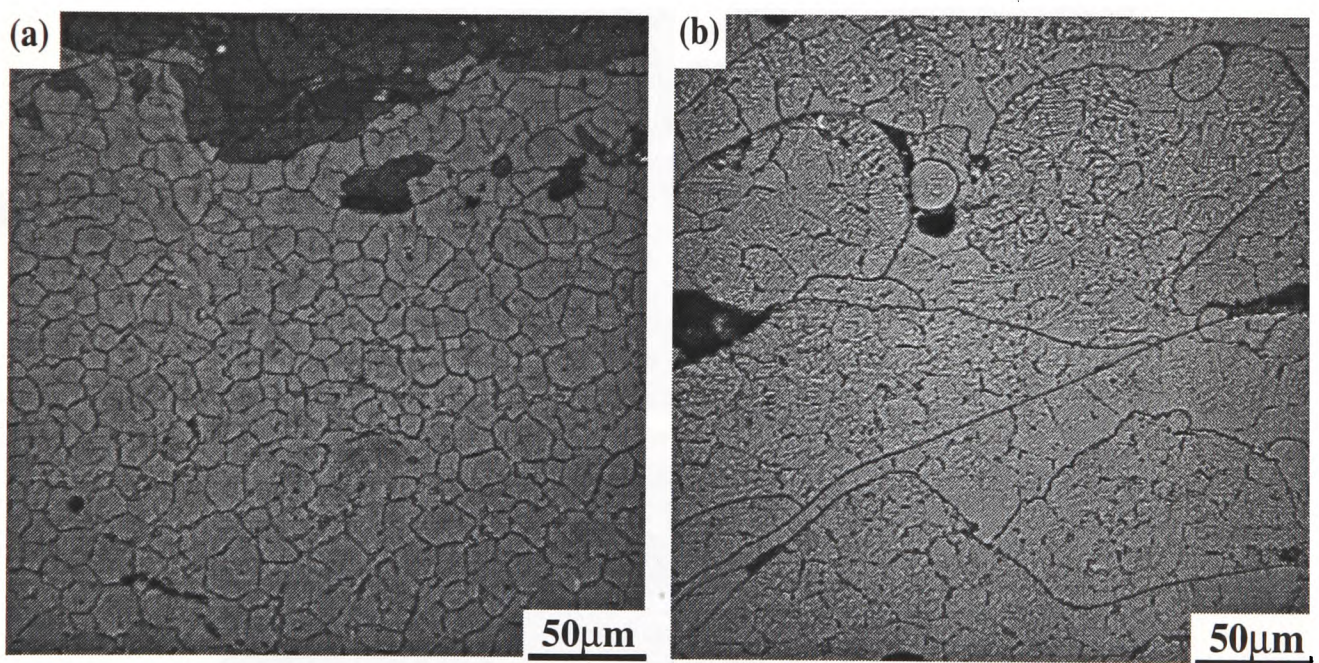
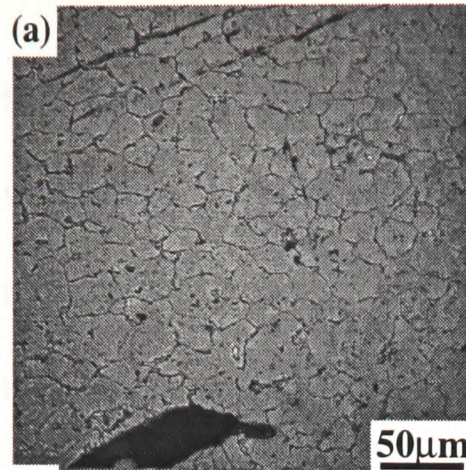


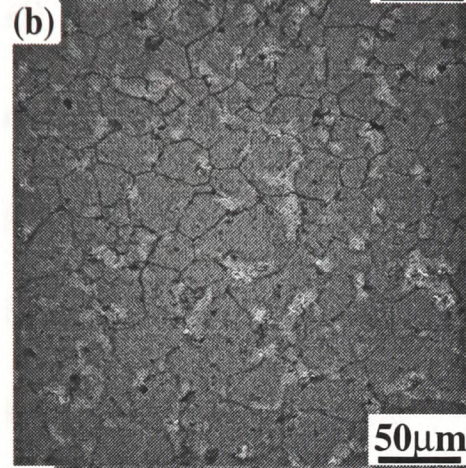
Fig 5.24 Micrograph of (a) region IV for run 34 showing an equiaxed grain structure and (b) region IV for run 30 showing a splat structure. Etchant 10% Orthophosphoric acid.



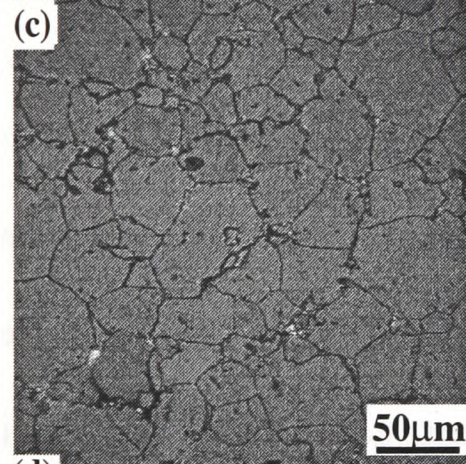
**Region IV**



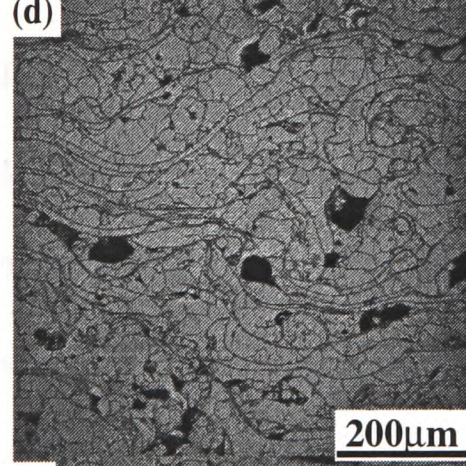
**Region III**



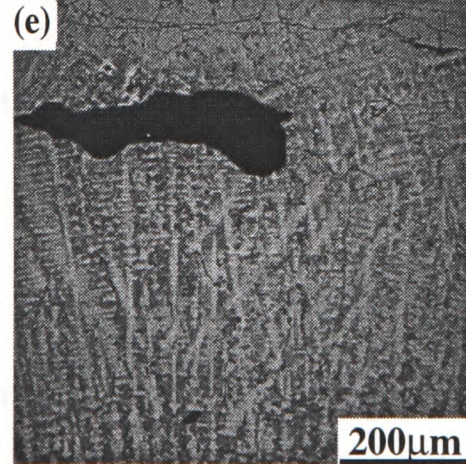
**Region III**



**Region II**

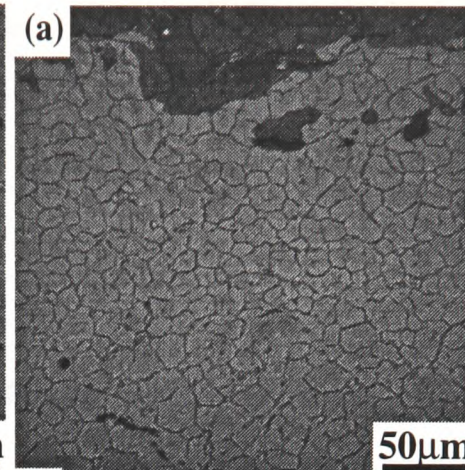


**Region I**

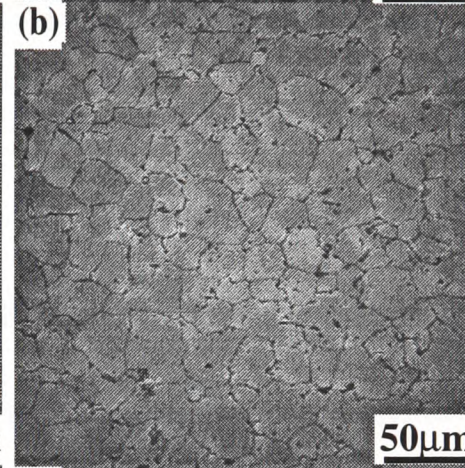


Figs 5.25(a)–(e) Through thickness variation in microstructure for run 24

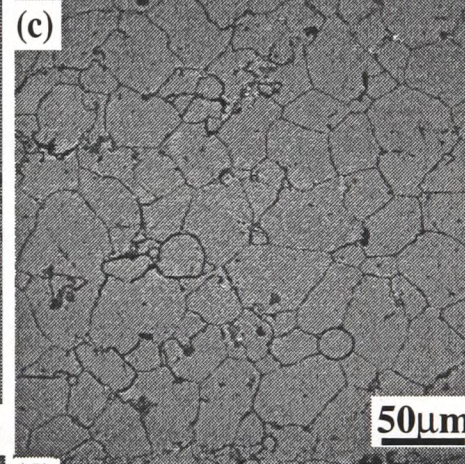
**Region IV**



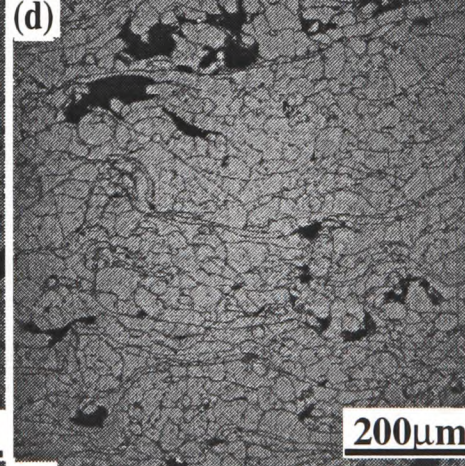
**Region III**



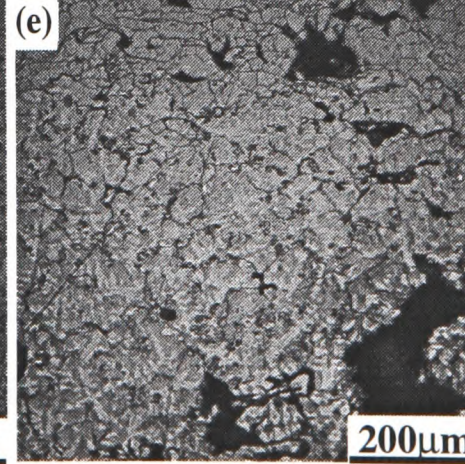
**Region III**



**Region II**



**Region I**



Figs 5.26(a)–(e) Through thickness variation in microstructure for run 34



deposits had an equiaxed grain structure in region III. Figs 5.27(a)-(e) and figs 5.28(a)-(e) show similar through thickness microstructures for runs 28 and 30 respectively which had a splat structure in region III.

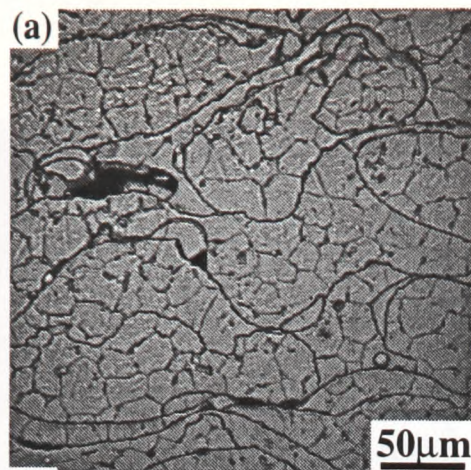
Figs 5.29(a)-(q) show the microstructure at the position of  $T_{\max}$  for runs 16-37 respectively and were all taken from region III. The microstructures either consisted of equiaxed grains e.g. runs 17-23 or a splat structure e.g. runs 27-30. Figs 5.29(c) and (d) of runs 19 and 20 also show quite large particles that were present at the grain boundaries and are likely to be carbides (Sczerzenie *et al* 1988). These particles were observed in all the deposits, although they varied in size and were seen most clearly when the deposit was defocused slightly.

Similar to the variation of either low (<1%) or high (2-4%) porosity in figs 5.18 and 5.19, the grain morphology also showed a transition from splat microstructure if  $T_{\max}$  was  $<\approx 1250^{\circ}\text{C}$  and deposition rate  $<\approx 20\text{g/s}$  to an equiaxed microstructure at higher temperatures or deposition rates. Again this transition was more reliable in terms of temperature. For example, run 16 had a splat structure at a  $T_{\max}$  of  $1246^{\circ}\text{C}$  although its deposition rate was  $25\text{g/s}$ , and run 25 had an equiaxed grain structure at a  $T_{\max}$  of  $1262^{\circ}\text{C}$  although its deposition rate was  $15\text{g/s}$ . Run 31 again was an exception to both rules though with an equiaxed microstructure at a  $T_{\max}$  and deposition rate of  $1210^{\circ}\text{C}$  and  $16\text{g/s}$  respectively.

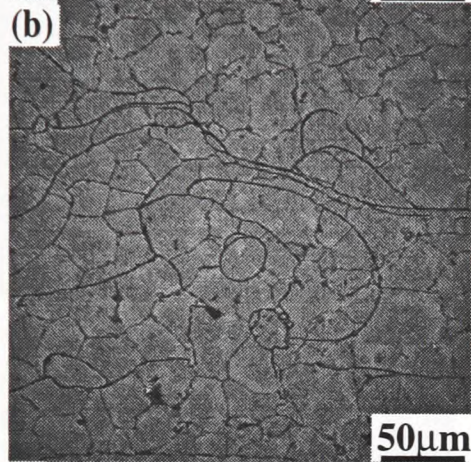
Figs 5.30(a)-(e) show the variation of grain size in region III with normalised deposit height for runs 17-23 (i.e. with an equiaxed grain structure). The vertical dashed line marks the position of the  $T_{\max}$  thermocouple in the deposit (either B, M or T). Figs 5.30(a)-(e) show that in each case the grain size was roughly constant until approximately



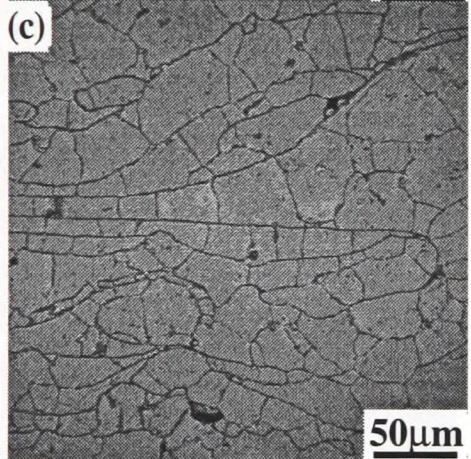
**Region IV**



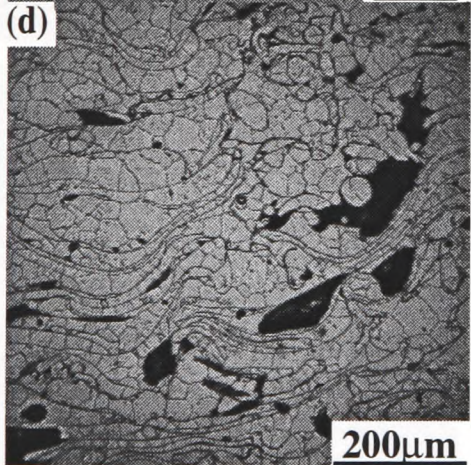
**Region III**



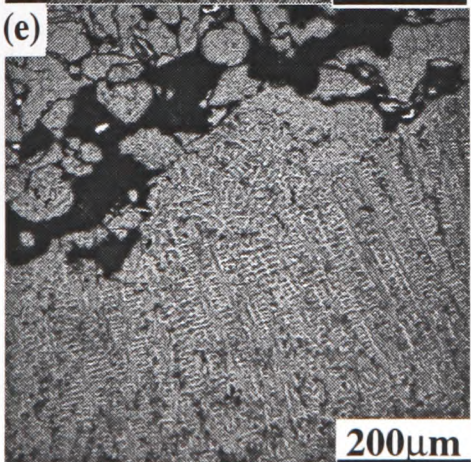
**Region III**



**Region II**

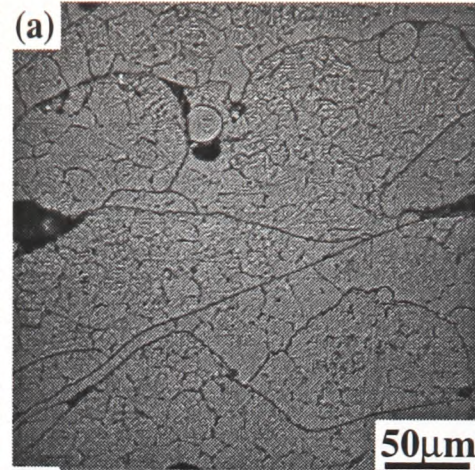


**Region I**

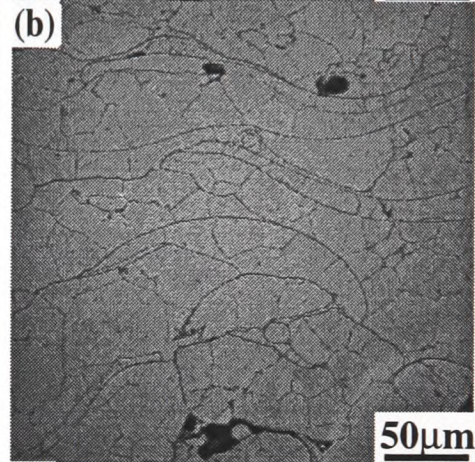


Figs 5.27(a)–(e) Through thickness variation in microstructure for run 28

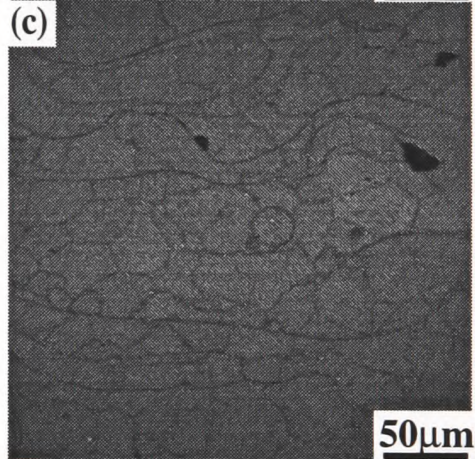
**Region IV**



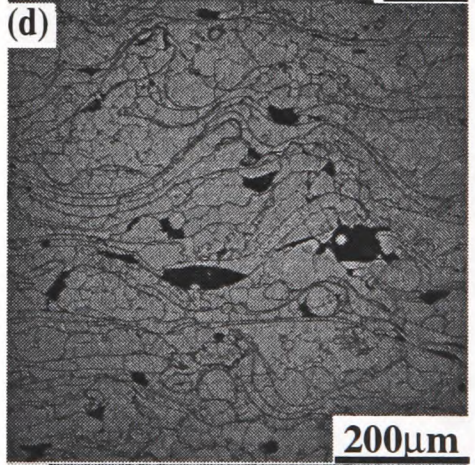
**Region III**



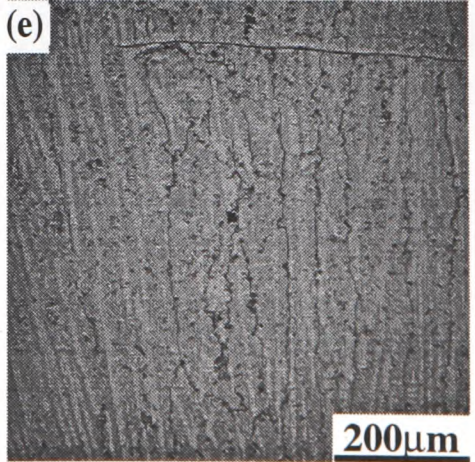
**Region III**



**Region II**



**Region I**



Figs 5.28(a)–(e) Through thickness variation in microstructure for run 30



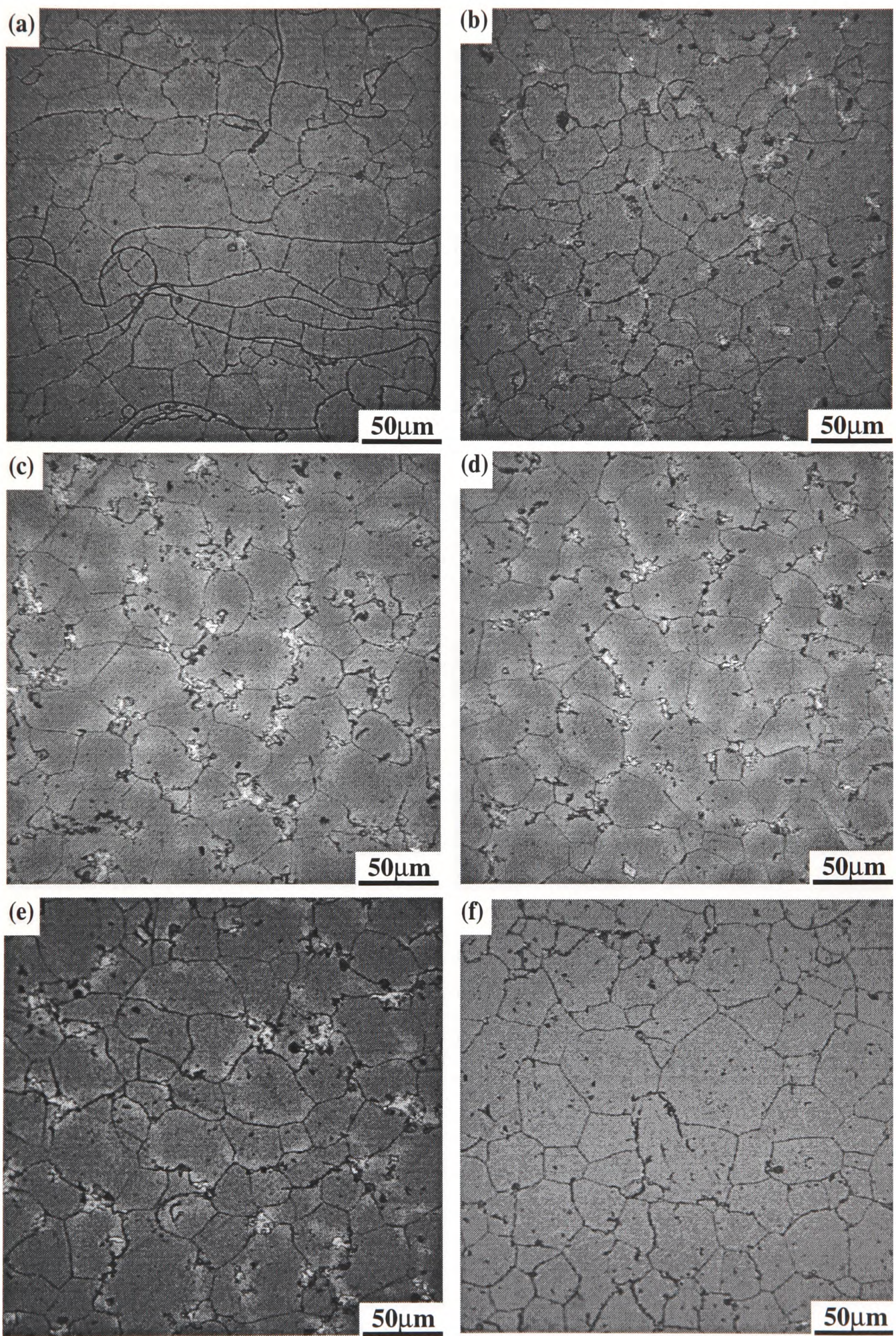
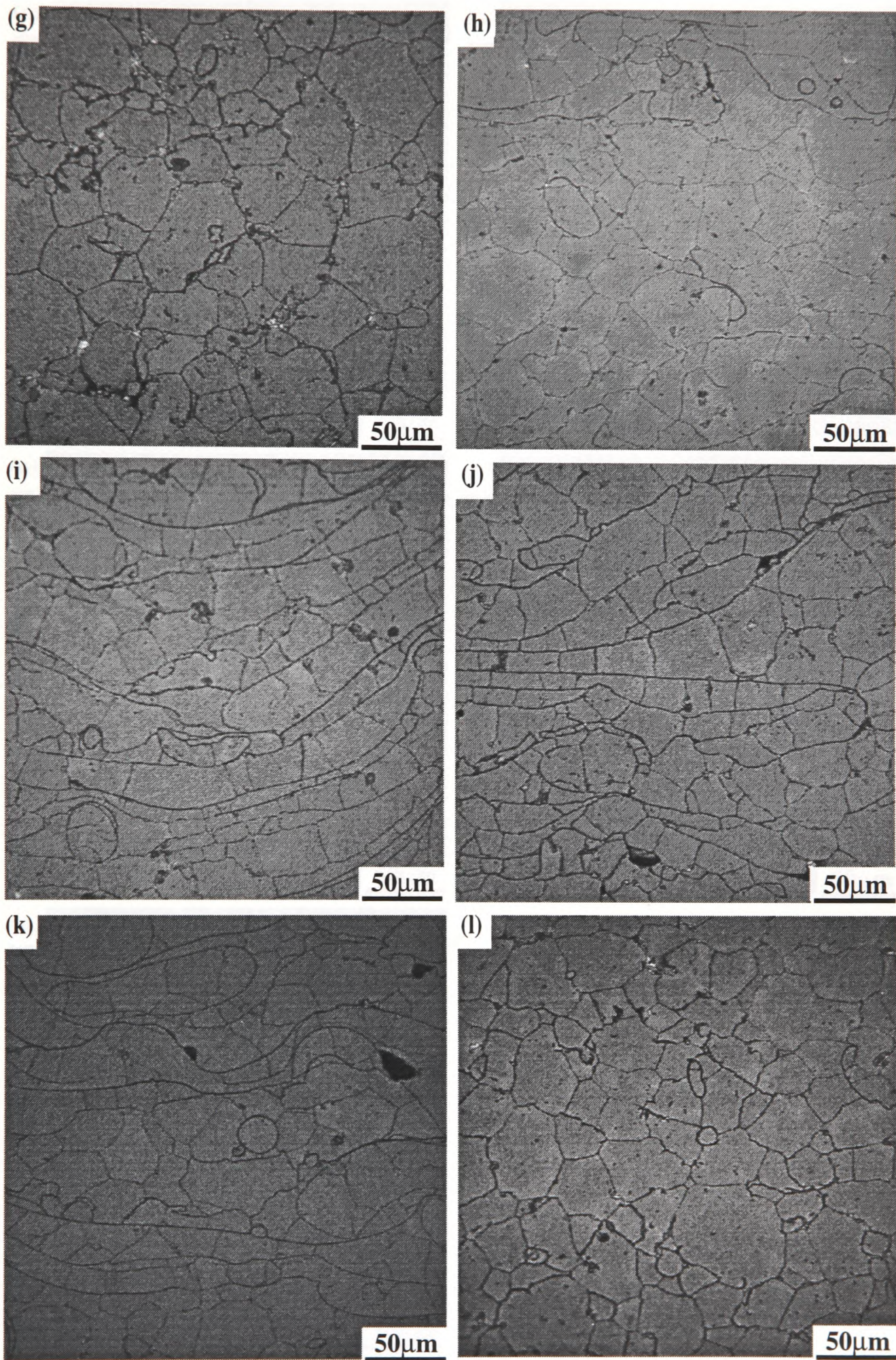


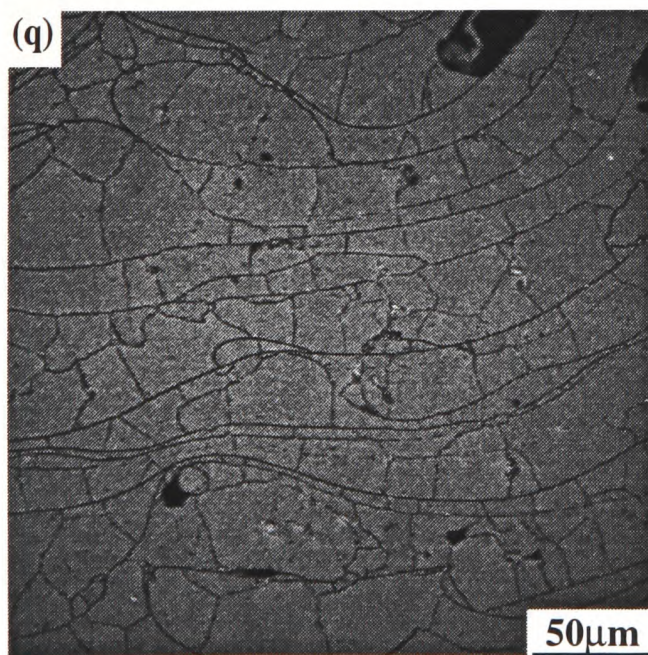
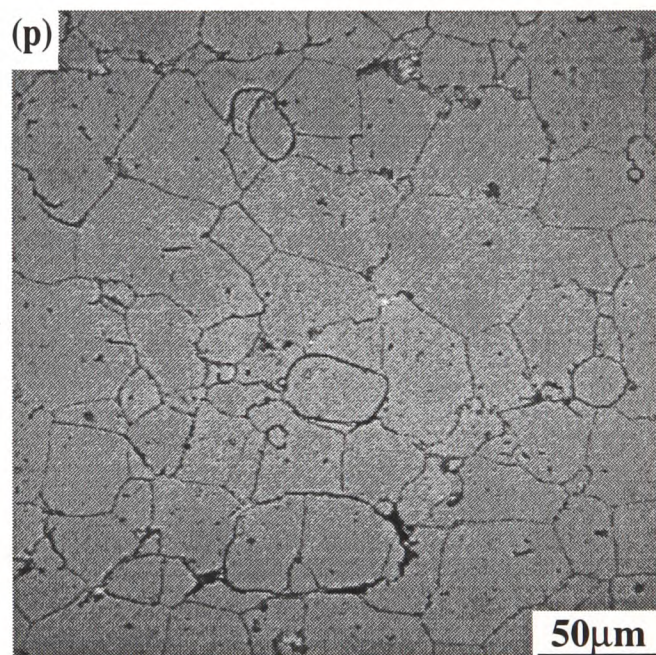
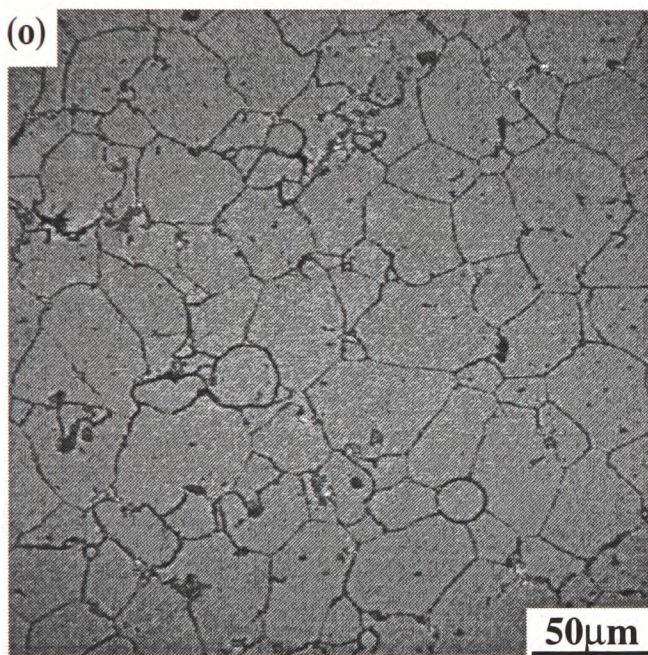
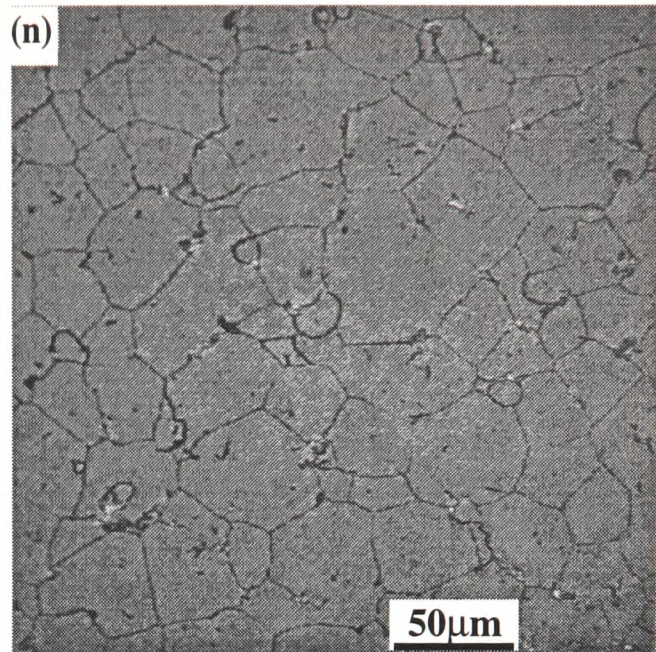
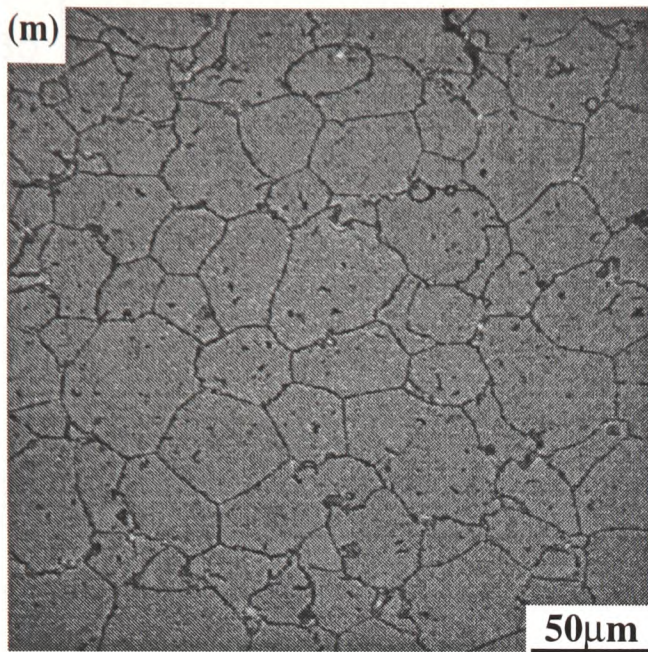
Fig 5.29 Micrographs taken at the position of maximum temperature of (a) Run 16, (b) Run 17, (c) Run 19, (d) Run 20, (e) Run 21 and (f) Run 23. Etchant 10% Orthophosphoric acid. cont...





...cont Fig 5.29 Micrographs taken at the position of maximum temperature of (g) Run 24, (h) Run 25, (i) Run 27, (j) Run 28, (k) Run 30 and (l) Run 31. Etchant 10% Orthophosphoric acid. cont...





...cont Fig 5.29 Micrographs taken at the position of maximum temperature of (m) Run 32, (n) Run 33, (o) Run 34, (p) Run 35 and (q) Run 37. Etchant 10% Orthophosphoric acid.



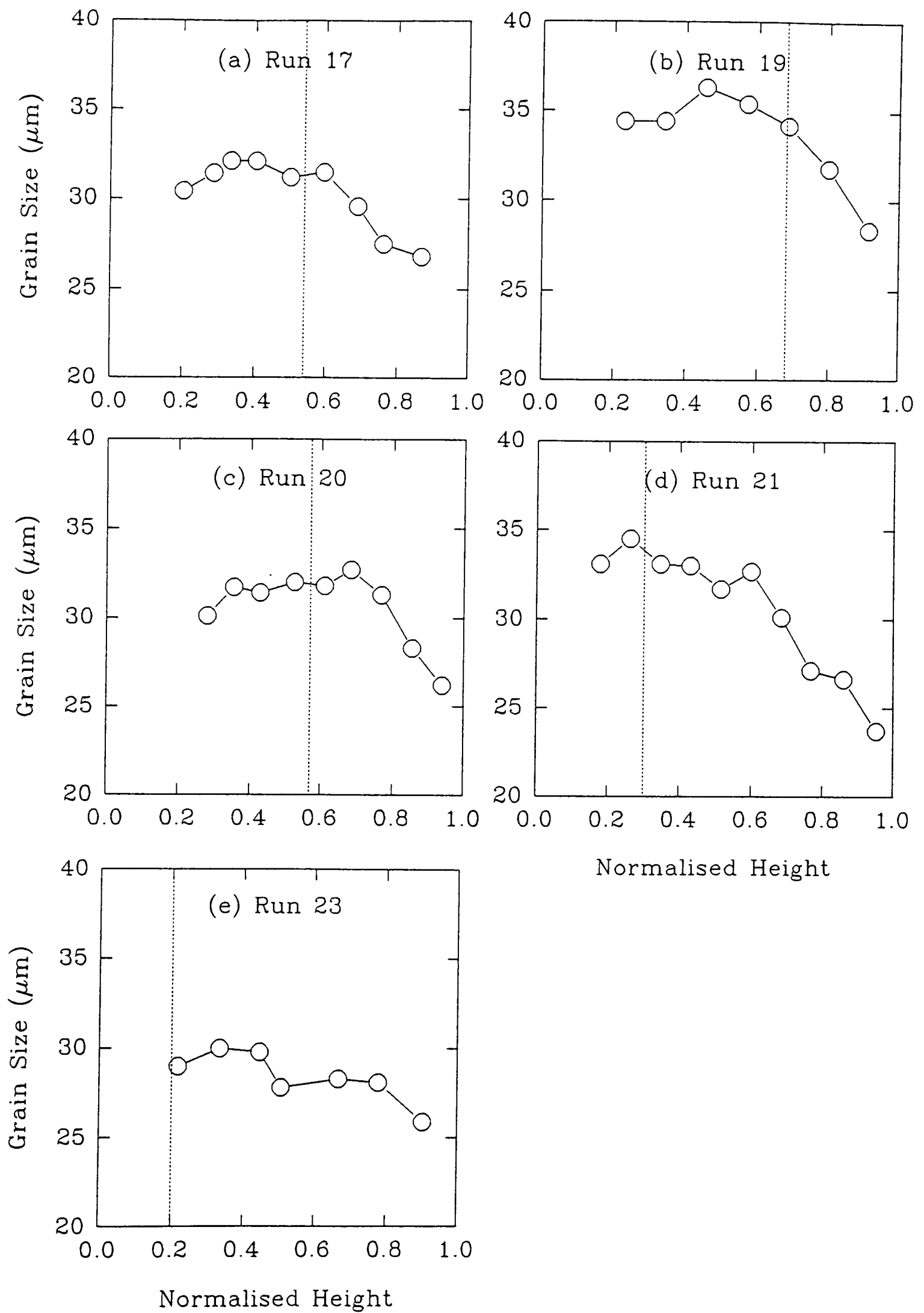


Fig 5.30 Grain size against normalised deposit height for: (a) run 17, (b) run 19, (c) run 20, (d) run 21, (e) run 23.

three quarters of the way through the deposit after which it decreased. For example, in run 21 the grain size was  $33.5\mu\text{m}$  at 0.3 of the deposit height, stayed approximately constant until 0.65, and then decreased to  $23.7\mu\text{m}$  at 0.95 of the deposit height.

Figs 5.31(a)-(f) show the variation of both grain size in region III, and calibrated top surface temperature with normalised deposit height/time for runs 16, 24 and 32-35. The position of the  $T_{\text{max}}$  thermocouple is marked as in fig 5.30 and the solidus of UDIMET 720 ( $1225^{\circ}\text{C}$ ) is marked by a horizontal dashed line. Figs 5.31(a)-(f) show the following features: (i) there was good correlation between temperature (excluding the anomalous temperatures at the beginning and end of deposition) and the through thickness grain size i.e. a reduction in top surface temperature gave a reduction in grain size; and vice versa (ii) increased average top surface temperatures led to increased final grain sizes. For example, in fig 5.31(e) for run 34, the grain size and temperature were  $31.8\mu\text{m}$  and  $1274^{\circ}\text{C}$  respectively at 0.31 of deposit height, were approximately constant until  $\approx 0.5$  of the deposit height, and decreased to  $20.5\mu\text{m}$  and  $1248^{\circ}\text{C}$  respectively at 0.91 of deposit height.

Figs 5.32(a)-(f) show the variation of grain size and calibrated top surface temperature with normalised deposit height/time for runs 25-31 and run 37. The grain size for splat structures was again determined by a linear intercept method, except that splat boundaries nearly parallel to the measurement line were not counted. The positions of the  $T_{\text{max}}$  thermocouple and  $T_{\text{sol}}$  are again marked by vertical and horizontal dashed lines respectively. For each deposit the grain size in region III was again approximately constant for half to three quarters of the deposit and then decreased quite rapidly for the rest of the deposit when there was a corresponding decrease in temperature. In comparison



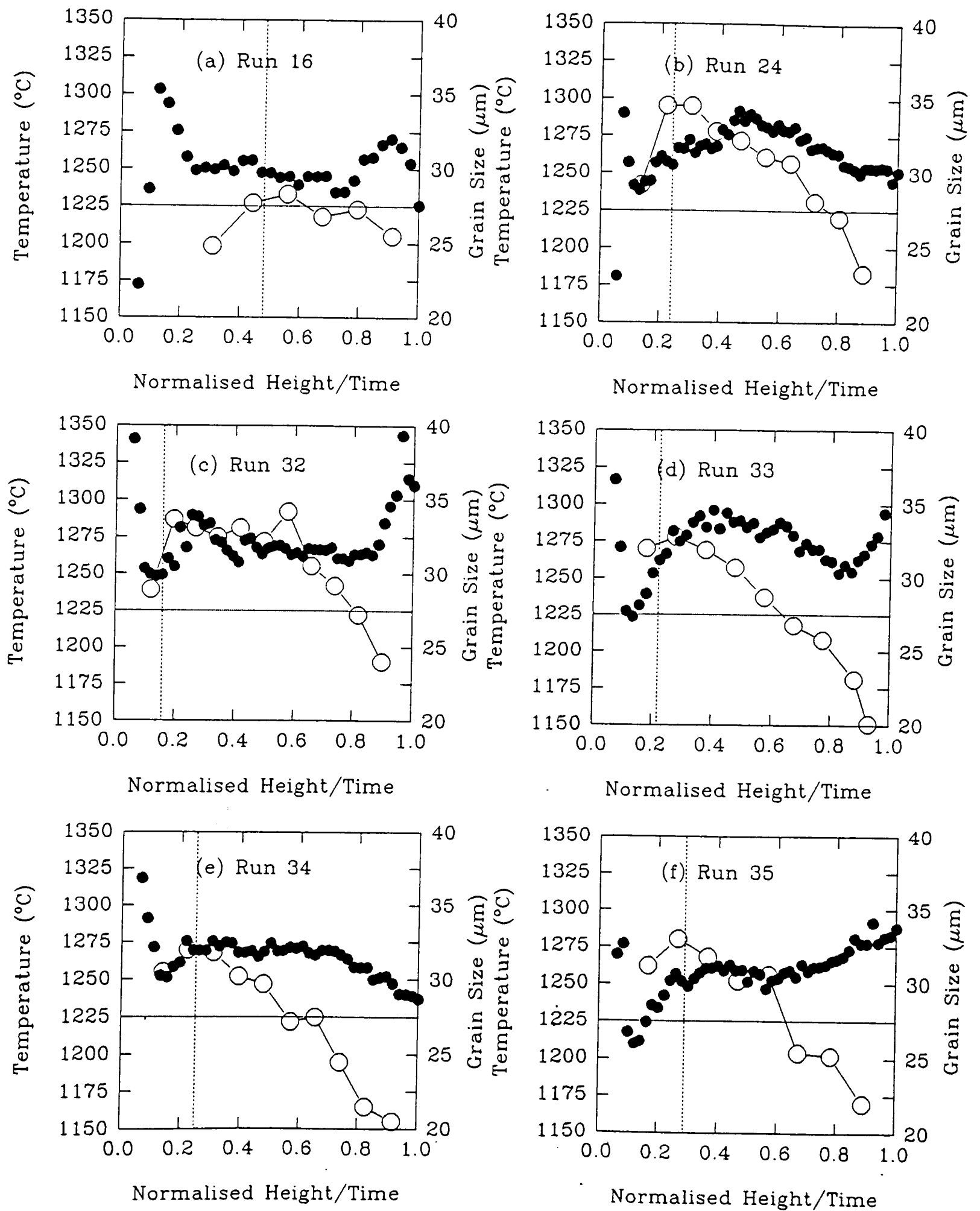


Fig 5.31 Grain size and calibrated thermal imaging temperature against normalised deposit height/run time for: (a) run 16, (b) run 24, (c) run 32, (d) run 33, (e) run 34, (f) run 35.

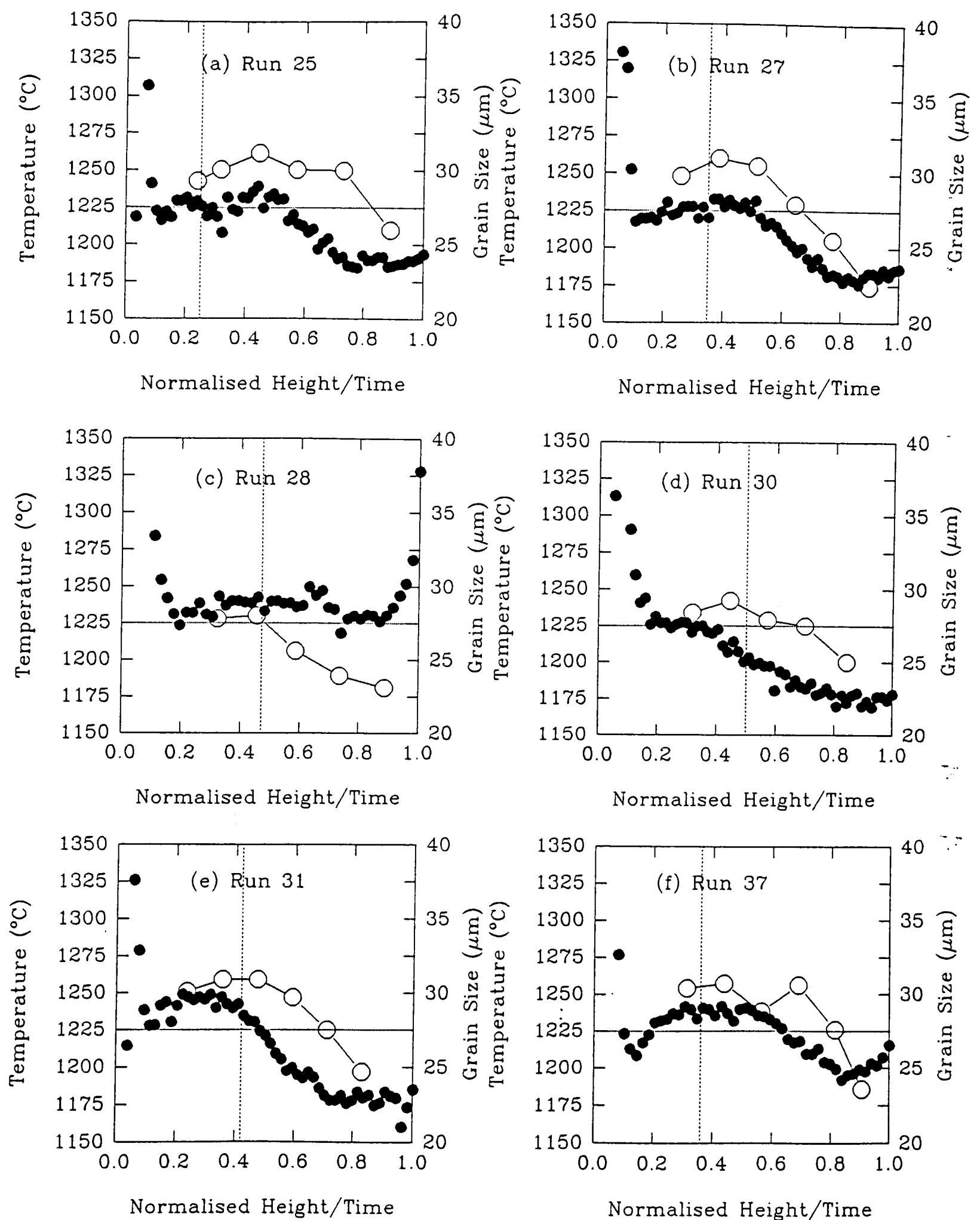


Fig 5.32 Grain size and calibrated thermal imaging temperature against normalised deposit height/run time for: (a) run 25, (b) run 27, (c) run 28, (d) run 30, (e) run 31, (f) run 37.

with figs 5.31(a)-(f), temperatures were generally lower and consequently grain sizes were also somewhat smaller, the correlation between a decrease in temperature and lower grain sizes was also more reliable than in figs 5.31(a)-(f). For example, in fig 5.32(a) for run 25 the grain size and temperature were  $30.4\mu\text{m}$  and  $1240^\circ\text{C}$  respectively at 0.31 of the deposit height, were approximately constant until  $\approx 0.5$  of the deposit height and then decreased to  $23.6\mu\text{m}$  and  $1198^\circ\text{C}$  respectively at 0.91 of the deposit height.

Fig 5.33 shows the grain size at the position of  $T_{\text{max}}$  against (a) deposition rate and (b)  $T_{\text{max}}$  respectively for runs 16-37 with an equiaxed grain structure in region III. The error bars are the standard deviation from the linear intercept method used and were quite large indicating the relatively large spread of as-sprayed grain sizes. The grain size increased slightly with increasing deposition rate in fig 5.33(a) from  $31.8\mu\text{m}$  at 20g/s to  $33.5\mu\text{m}$  at 27g/s. Fig 5.33(b) shows that the grain size increased slightly from  $31.8\mu\text{m}$  at  $1252^\circ\text{C}$  to  $34.5\mu\text{m}$  at  $1295^\circ\text{C}$ . Similarly to the variation of porosity with deposition rate and temperature, grain size showed a more reliable correlation with temperature.

The local solidification time for deposits with an equiaxed grain structure was calculated from the total time spent above  $T_{\text{sol}}$  by the  $T_{\text{max}}$  thermocouple. Fig 5.34 shows the measured grain size at the position of the  $T_{\text{max}}$  thermocouple against the local solidification time. Fig 5.34 also shows  $T_{\text{max}}$  as a function of grain size. Run 31 was not included because although it had an equiaxed grain structure its maximum embedded thermocouple temperature was below  $T_{\text{sol}}$ . The grain size increased steadily with temperature as shown before in fig 5.33(b), but there was also a steady increase in grain size with increasing local solidification time. For example the grain size increased from  $31.8\mu\text{m}$  at a local solidification time of 24.8s to  $34.5\mu\text{m}$  at 39.9s.



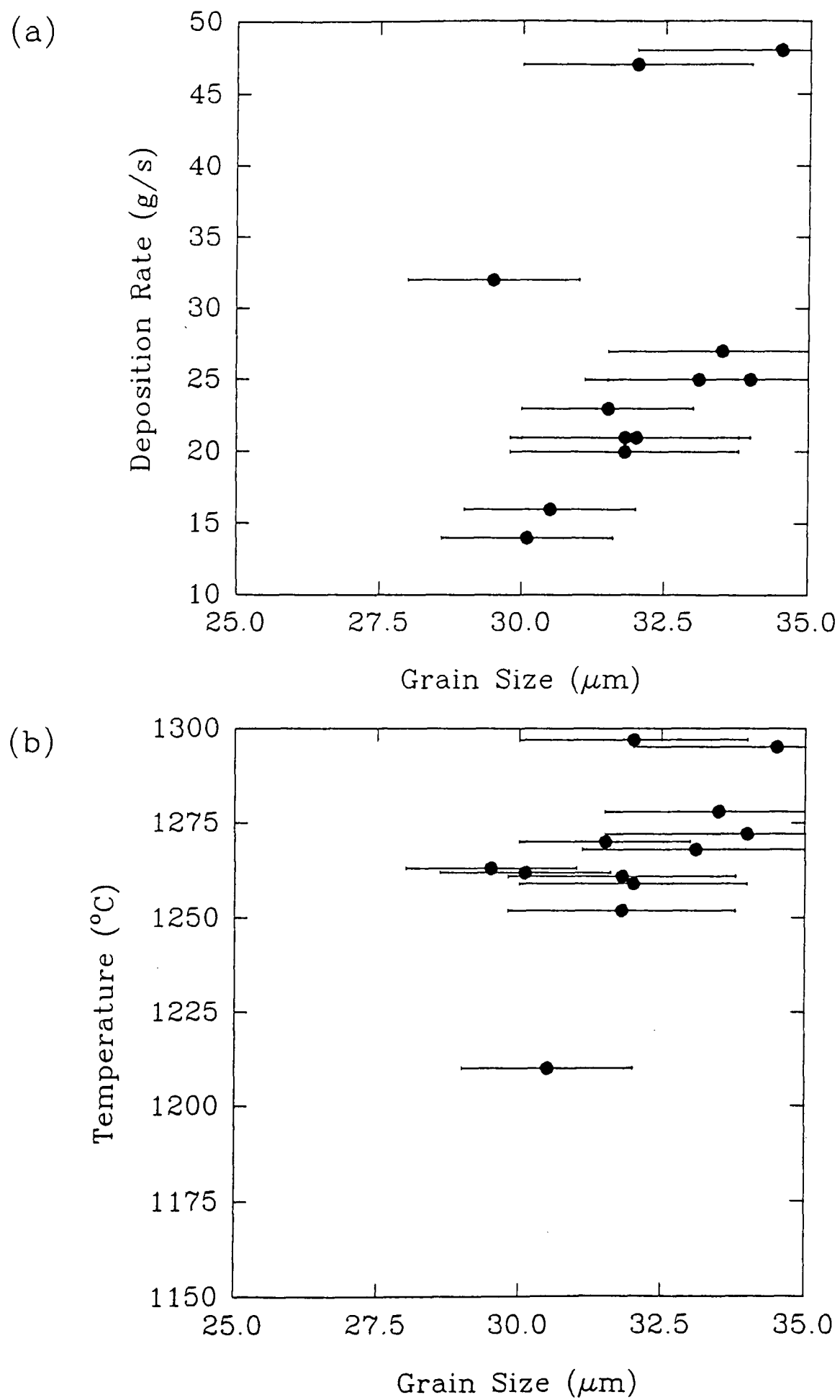


Fig 5.33 Grain size against (a) deposition rate and (b)  $T_{\text{max}}$  for runs 16-37 which had an equiaxed grain structure.

Section 4.3 showed that by assuming grain growth in the solid/liquid region followed equation 4.1, then the coarsening rate constants (K) for UDIMET 720 at temperatures ranging from 1245°C-1290°C could be determined and are shown in table 5.7. Assuming that the solid component of the droplet spray arising from fractured or complete dendrite arms of solid or semi-solid droplets after impact on the deposit top surface leads to an initial grain size  $d_0 \approx 5\mu\text{m}$  (Annavarapu and Doherty 1993, Bewlay and Cantor 1989), using the values of K from table 5.7 and the time (t) from the local solidification time then the final equiaxed grain size of spray formed deposits can be predicted. Only those deposits with an equiaxed grain size and  $T_{\text{max}} > T_{\text{sol}}$  were analysed.

| Temperature (°C) | Coarsening Rate<br>Constant K ( $10^{-18}\text{m}^3/\text{s}$ ) |
|------------------|-----------------------------------------------------------------|
| 1245             | 110                                                             |
| 1260             | 114                                                             |
| 1270             | 148                                                             |
| 1280             | 166                                                             |
| 1290             | 183                                                             |

Table 5.7 Coarsening rate constant (K) at temperatures in the solid/liquid region for UDIMET 720.

Fig 5.35 shows this predicted grain size against the measured local solidification times and  $T_{\text{max}}$ . Fig 5.35 shows that the predicted increase in grain size with solidification time and  $T_{\text{max}}$  and the predicted range of grain sizes were broadly in agreement with measured grain sizes.

Fig 5.36 shows measured grain size against predicted grain size, with the predicted grain size calculated in three ways using (i)  $d_0 = 5\mu\text{m}$  and the values of K from table 5.7;

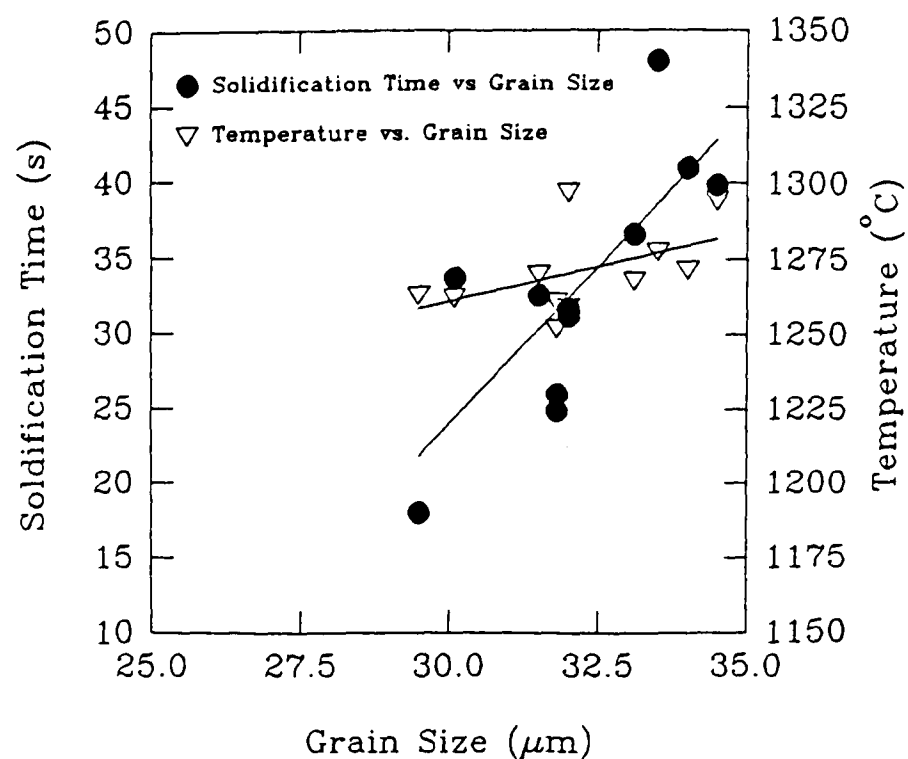


Fig 5.34 Solidification time and  $T_{\max}$  against grain size for runs 16-37 which had an equiaxed grain structure and a  $T_{\max}$  temperature above the solidus.

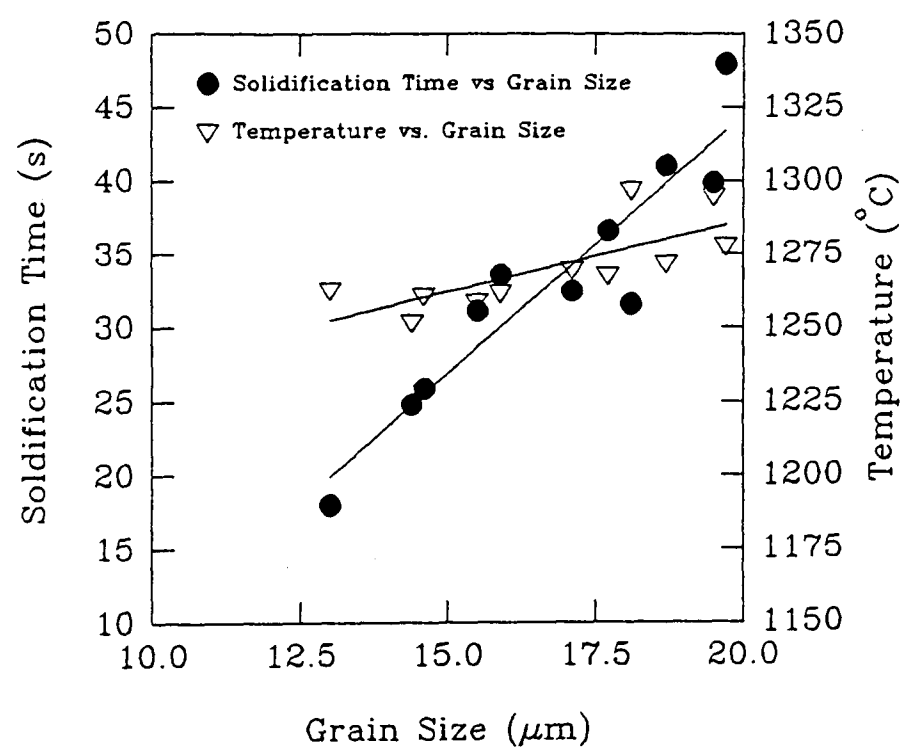


Fig 5.35 Solidification time and  $T_{\max}$  against predicted grain size for runs 16-37 which had an equiaxed grain structure and a  $T_{\max}$  temperature above the solidus.

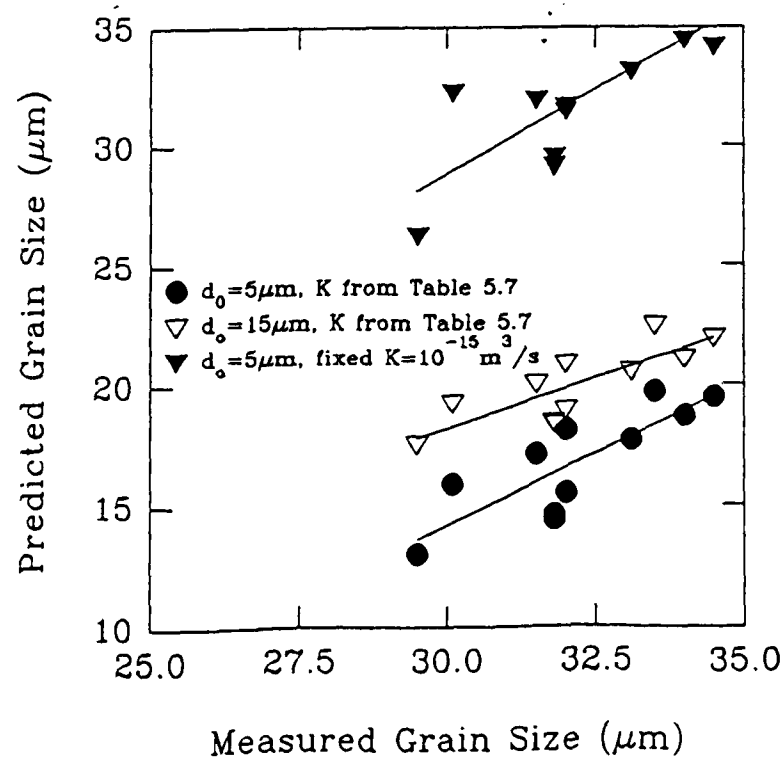


Fig 5.36 Measured against predicted grain size for runs 16-37 which had an equiaxed grain



(ii)  $d_0=15\mu\text{m}$  and the values of  $K$  from table 5.7; and (iii)  $d_0=5\mu\text{m}$  and an assumed constant value of  $K=10^{-15}\text{m}^3/\text{s}$ . In all cases there was good general agreement between measured and predicted grain sizes, although measured grain sizes were underestimated by  $\approx 15\mu\text{m}$  and  $\approx 12\mu\text{m}$  for cases (i) and (ii) respectively. An initial grain size of  $d_0=5\mu\text{m}$  and an assumed constant  $K=10^{-15}\text{m}^3/\text{s}$  gave the best agreement with the measured grain sizes. For example, the predicted grain size was  $29.2\mu\text{m}$  compared with a measured value of  $31.8\mu\text{m}$  at  $1252^\circ\text{C}$ ,  $24.8\text{s}$  and  $34.2\mu\text{m}$  compared with a measured value of  $34.5\mu\text{m}$  at  $1295^\circ\text{C}$ ,  $39.9\text{s}$ . This indicates that the measured  $K$  values were probably lower than they actually were during solidification of the deposit.

### **5.3.6 Solidification Defects**

Fig 5.37 shows typical cross sectional views of spray formed deposits from: (a) run 28 with a  $T_{\text{max}}$  of  $1235^\circ\text{C}$  and (b) run 37 with a  $T_{\text{max}}$   $1213^\circ\text{C}$ . They show that the cross sectional shape was approximately Gaussian. However, no major defects were visible. Figs 5.38 shows typical cross sectional views of spray formed deposits from: (a) run 21 with a  $T_{\text{max}}$  of  $1278^\circ\text{C}$ , (b) run 21 with a  $T_{\text{max}}$  of  $1263^\circ\text{C}$  and (c) run 32 with a  $T_{\text{max}}$   $1259^\circ\text{C}$ . They show that again the cross sectional shape was approximately Gaussian. However, at these higher temperatures several major defects were present including: (i) broken pieces of the zirconia nozzle, (ii) excess porosity around embedded thermocouples and (iii) contraction cracks.

As described in section 5.1 the zirconia melt delivery nozzle occasionally broke during spraying, increasing the MFR. Fig 5.38(a) shows that a piece of the zirconia nozzle was embedded in the growing deposit. When pieces of broken zirconia were observed

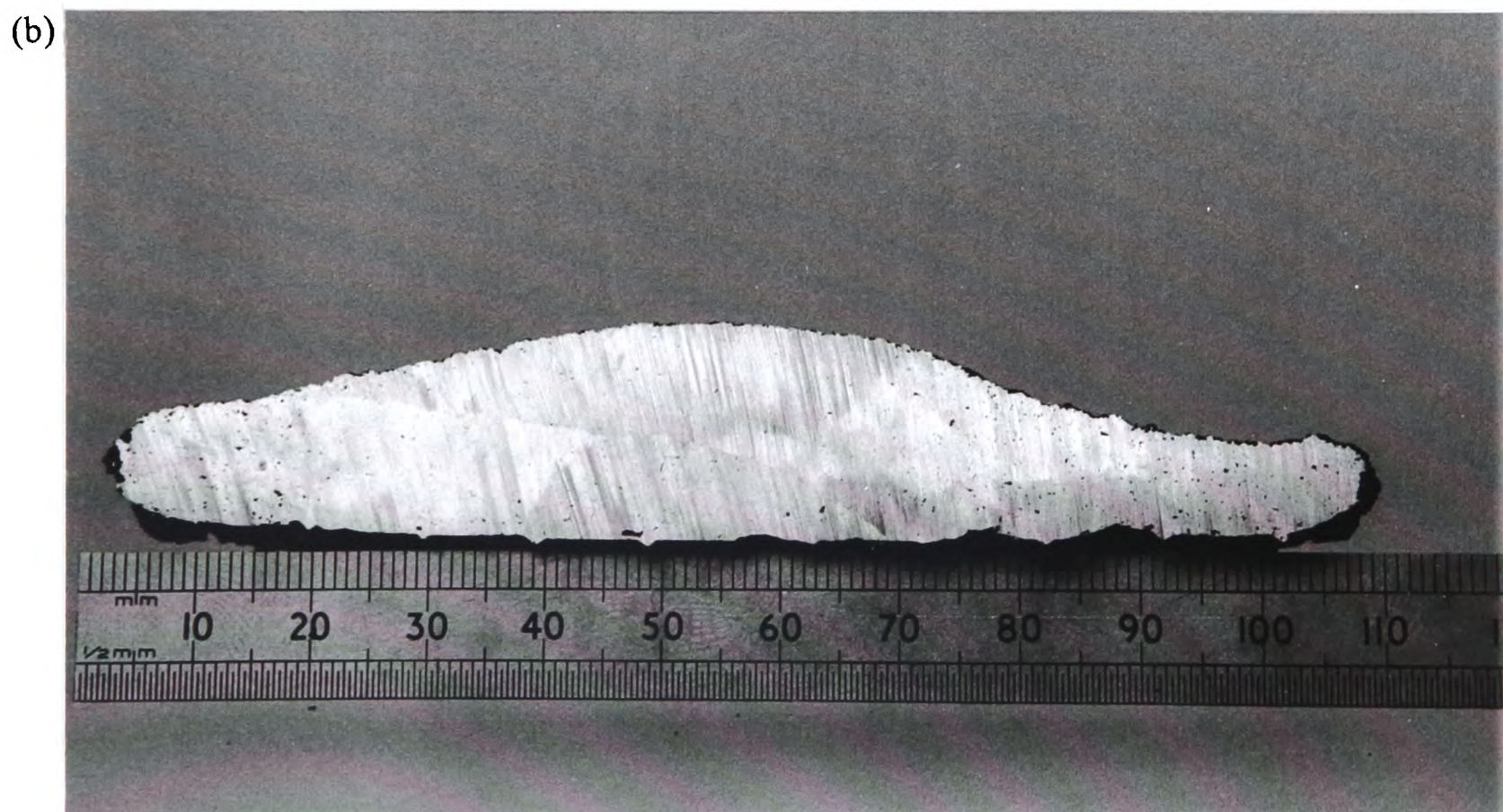
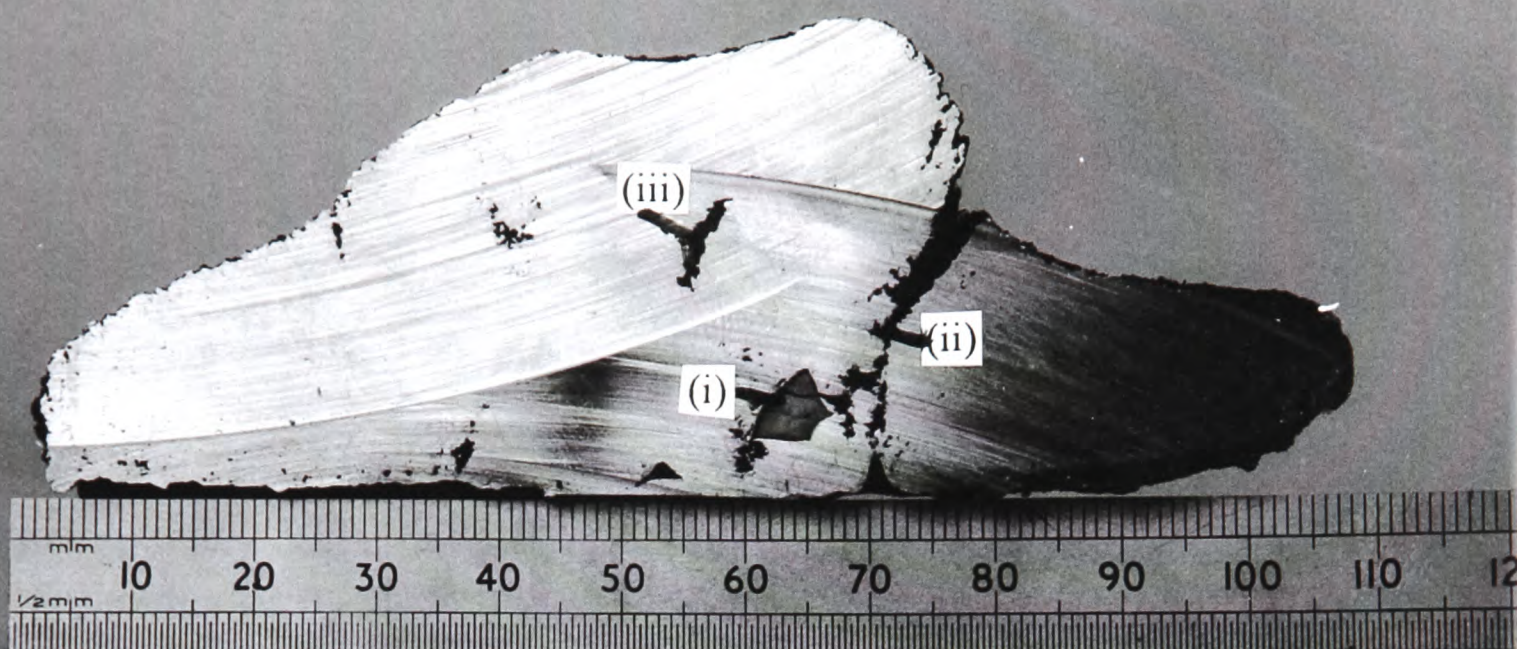


Fig 5.37 Typical cross sectional views of spray formed deposits from: (a) run 28 with a  $T_{\max}$  of  $1235^{\circ}\text{C}$  and (b) run 37 with a  $T_{\max}$   $1213^{\circ}\text{C}$ .



(b)



(c)

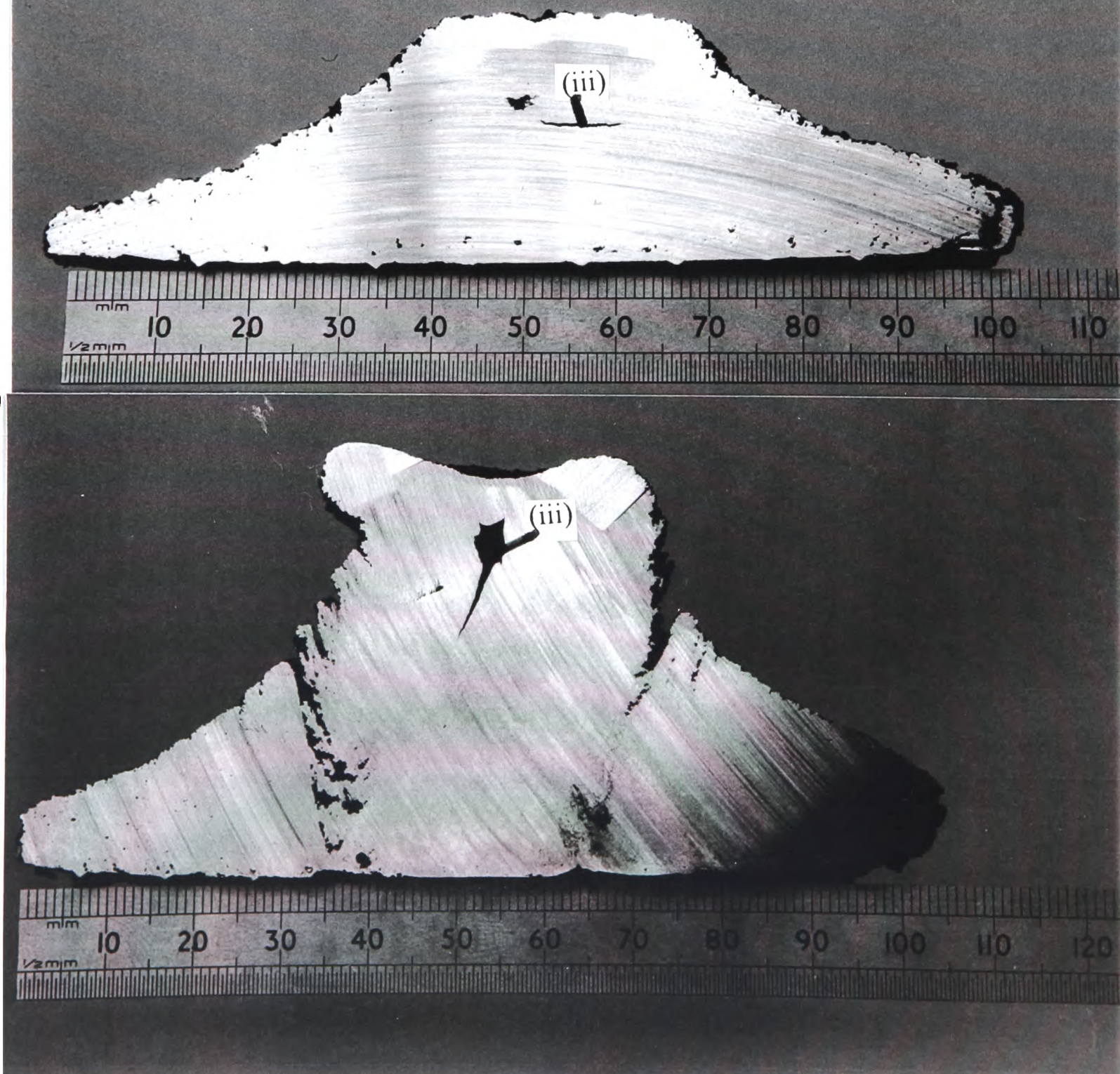


Fig 5.38 Typical cross sectional views of spray formed deposits from: (a) run 21 with a  $T_{\max}$  of  $1278^{\circ}\text{C}$ , (b) run 21 with a  $T_{\max}$  of  $1263^{\circ}\text{C}$  and (c) run 32 with a  $T_{\max}$   $1259^{\circ}\text{C}$ , showing the following defects: (i) broken pieces of the zirconia nozzle. (ii) excess



they were always at the bottom of the deposit indicating that the nozzle broke at the beginning of spray forming.

As described in section 5.3.1 and fig 5.8 thermocouples were not always fully embedded. Figs 5.38(a) and (c) show that excessive porosity sometimes developed around the tip of thermocouples due to the geometry of the growing deposit, which prevented thermocouples becoming fully embedded.

As shown in figs 5.38(a)-(c) contraction cracks (hot tears) were present in the top half of deposits with a  $T_{\max} > \approx 1250^{\circ}\text{C}$ . Hot tearing is more common in alloys with a wide freezing range and occurs when regions of isolated liquid are subjected to thermal stresses during cooling (Davies 1973). This indicates that in deposits with a  $T_{\max} > \approx 1250^{\circ}\text{C}$  i.e.  $> \approx 5\%$  liquid fraction, the top layer of the deposit probably solidified due to the convective cooling of the atomising gas leaving regions of isolated liquid in the top half of the deposit, similar to conventional castings, which then cracked on cooling.

### **5.3.7 Compositional Analysis**

Table 5.8 shows the nominal composition of UDIMET 720 and measured compositions from central and edge regions of 3 typical spray formed deposits and the as supplied UDIMET 720. It shows that the as supplied UDIMET 720 had approximately the same composition as the nominal composition. Spray formed deposits retained the same composition as the as supplied material, and there was no evidence of any macrosegregation of elements within the deposit for the samples analysed.

Table 5.9 shows the average O and N content of as supplied UDIMET 720 and 3 typical spray formed deposits. Table 5.9 shows that the O and N pickup during spray



forming was  $\approx 700\text{-}1070\text{ppm}$  and  $\approx 60\text{-}116\text{ppm}$  respectively. The high O pickup is probably because melting was not carried out under vacuum and the N pickup agrees reasonably well with values reported by Bricknell (1986) and Benz *et al* (1993) of  $\approx 170\text{ppm}$  and  $\approx 150\text{ppm}$  from  $\text{N}_2$  atomised spray formed René 80 and René 95 respectively.

| Sample                 | Ni  | Al   | Co    | Cr    | Mo   | Ti   | W    | C            | B    | Zr  |
|------------------------|-----|------|-------|-------|------|------|------|--------------|------|-----|
| Nominal UDIMET 720     | Bal | 2.5  | 15    | 18    | 3    | 5    | 1.5  | .035         | .033 | .03 |
| As supplied UDIMET 720 | Bal | 2.32 | 14.86 | 17.85 | 2.99 | 4.82 | 1.39 | not analysed |      |     |
| Run 20 centre          | Bal | 2.25 | 14.79 | 17.72 | 3.01 | 5.04 | 1.37 |              |      |     |
| Run 20 edge            | Bal | 2.22 | 14.76 | 17.70 | 3.03 | 5.13 | 1.36 |              |      |     |
| Run 30 centre          | Bal | 2.22 | 14.79 | 17.67 | 3.02 | 5.08 | 1.34 |              |      |     |
| Run 30 edge            | Bal | 2.27 | 14.84 | 17.72 | 3.02 | 5.06 | 1.32 |              |      |     |
| Run 35 centre          | Bal | 2.28 | 14.79 | 17.70 | 3.02 | 5.06 | 1.31 |              |      |     |
| Run 35 edge            | Bal | 2.25 | 14.76 | 17.69 | 3.02 | 5.09 | 1.35 |              |      |     |

Table 5.8 Nominal composition of UDIMET 720 and measured composition (wt%) from central and edge regions of 3 typical spray formed deposits and as supplied UDIMET 720.

|                        | O wt (ppm) | N wt (ppm) |
|------------------------|------------|------------|
| As supplied UDIMET 720 | 297        | 28         |
| Run 19                 | 1365       | 144        |
| Run 30                 | 1260       | 94         |
| Run 35                 | 1008       | 88         |

Table 5.9 Average O and N content of as supplied UDIMET 720 and 3 typical spray formed deposits.

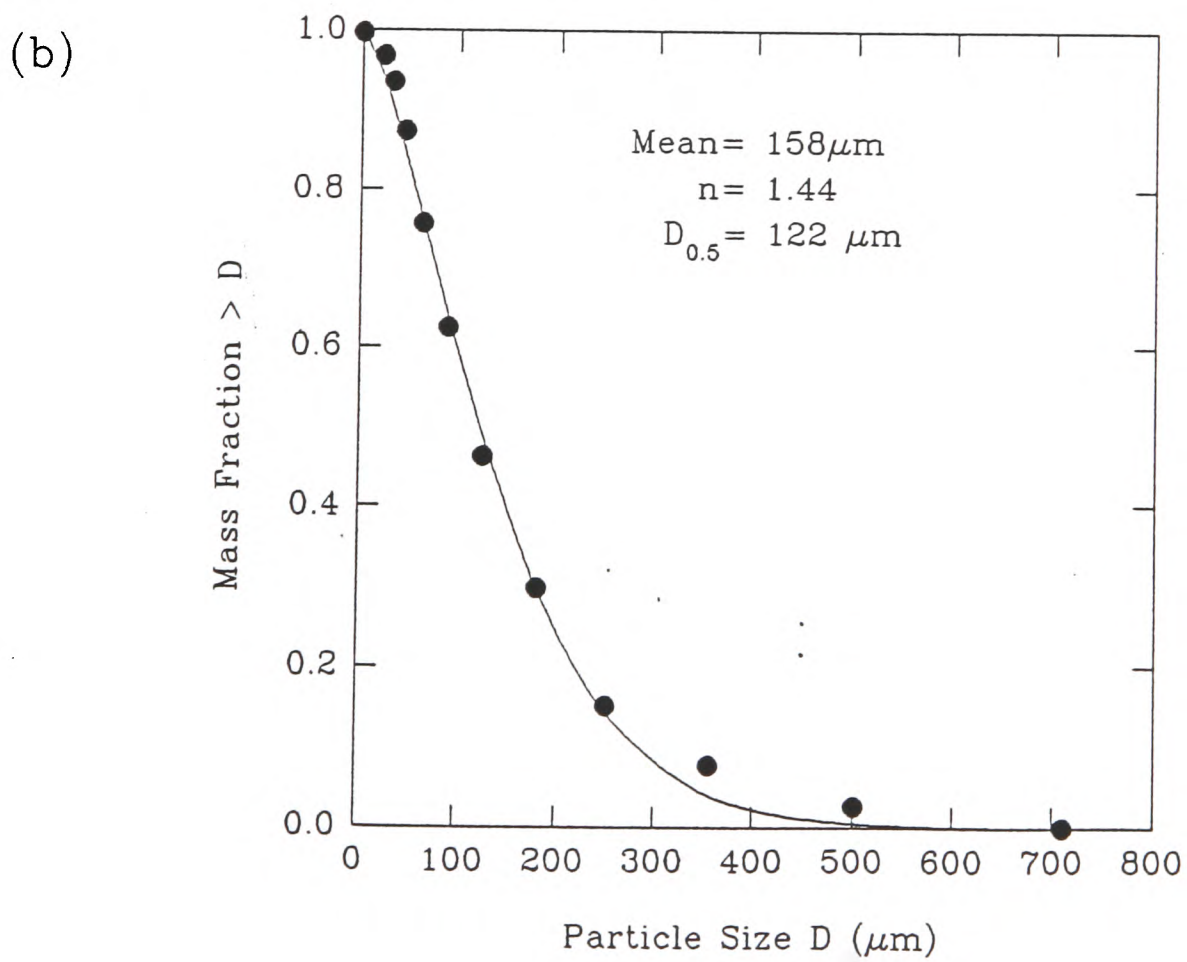
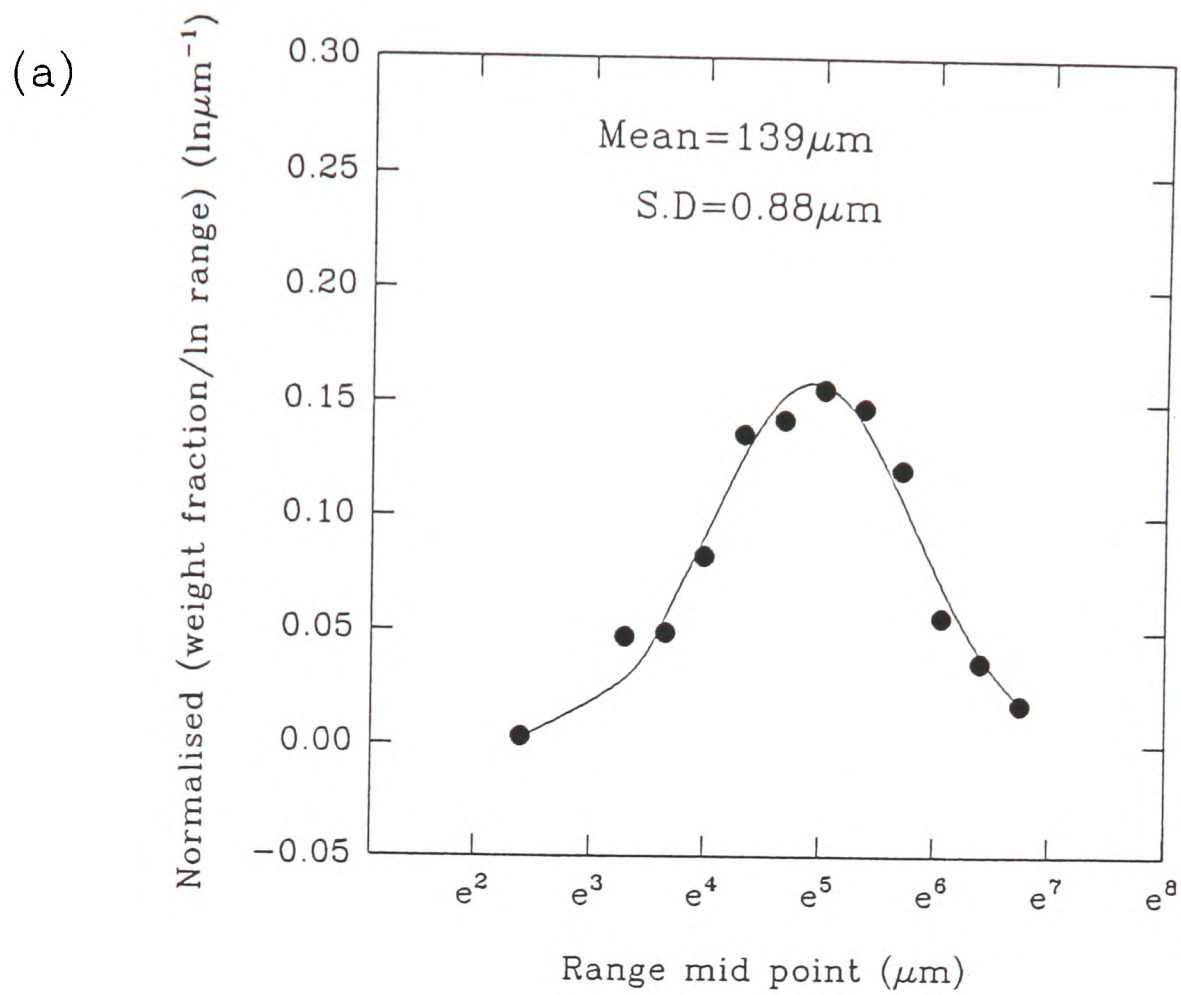
### **5.3.8 Overspray Powder**

Typical overspray powder distributions fitted to log-normal and Rosin-Rammler distributions respectively are shown in figs 5.39(a)-(f) for runs 31, 33 and 19 respectively, spray formed at MFR:GFR ratios of 6.1, 7.1 and 15.9 respectively. The data in all three cases fitted quite well to both log-normal and Rosin-Rammler distributions. Figs 5.39(a) and (c) for runs 31 and 33 both exhibited a bimodal distribution with a main peak at 125-180 $\mu$ m and a secondary peak at 63-90 $\mu$ m. Fig 5.39(e) for run 19 had a single peak at 125-180 $\mu$ m size range.

Fig 5.40 shows the variation of the log-normal mean and the mass median diameter ( $D_{0.5}$ ) for the Rosin-Rammler distribution with MFR:GFR for runs 16-37. Fig 5.38 shows that  $D_{0.5}$  increased from 122 $\mu$ m at a MFR:GFR of 6.1 to 153 $\mu$ m at a MFR:GFR of 9.3 then remained approximately constant as the MFR:GFR was increased further to  $\approx 16$ . Although runs 19 and 20 did not show the expected increase in mean droplet size there was a disappearance of the secondary peak between 60-90 $\mu$ m for these runs as shown in fig 5.39(e) for run 19.

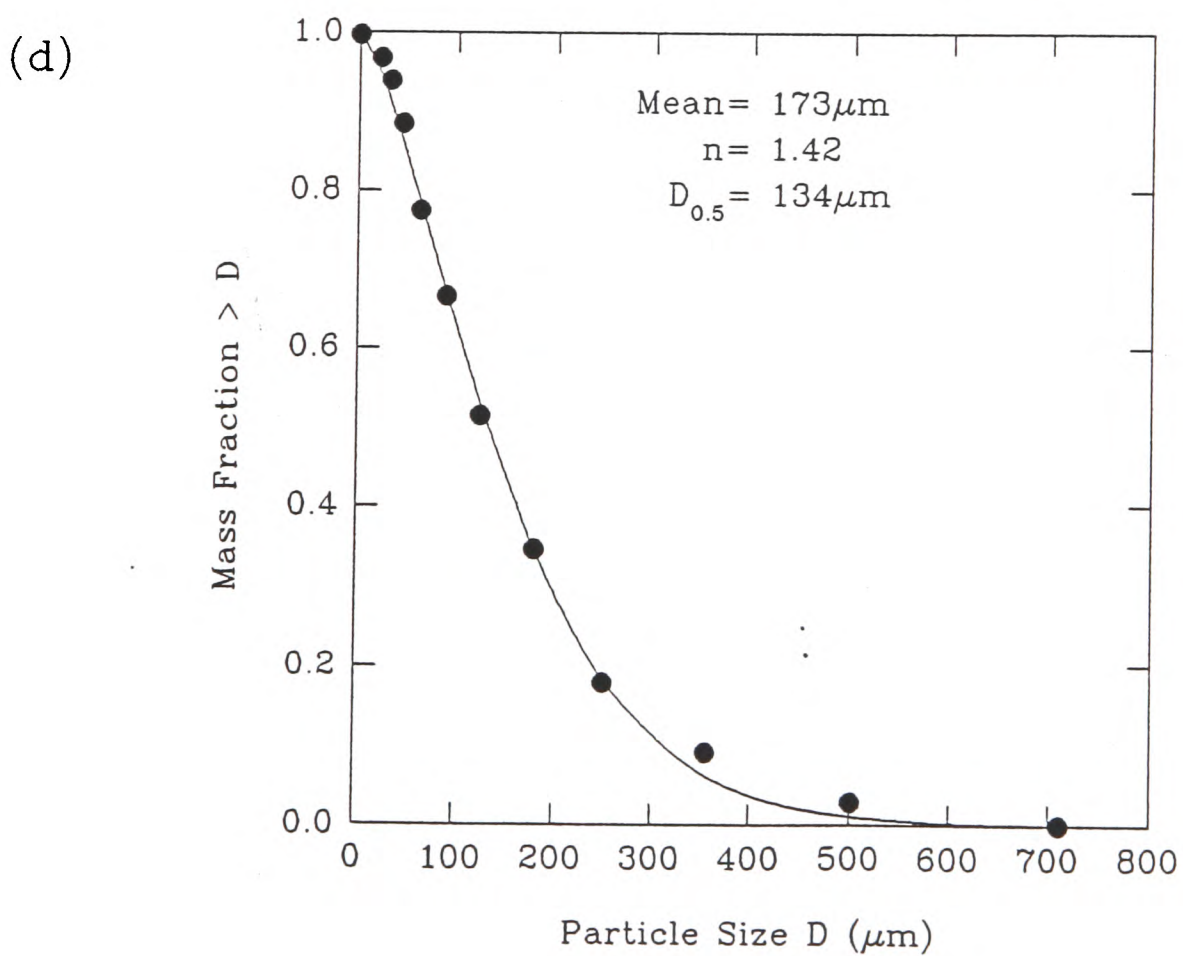
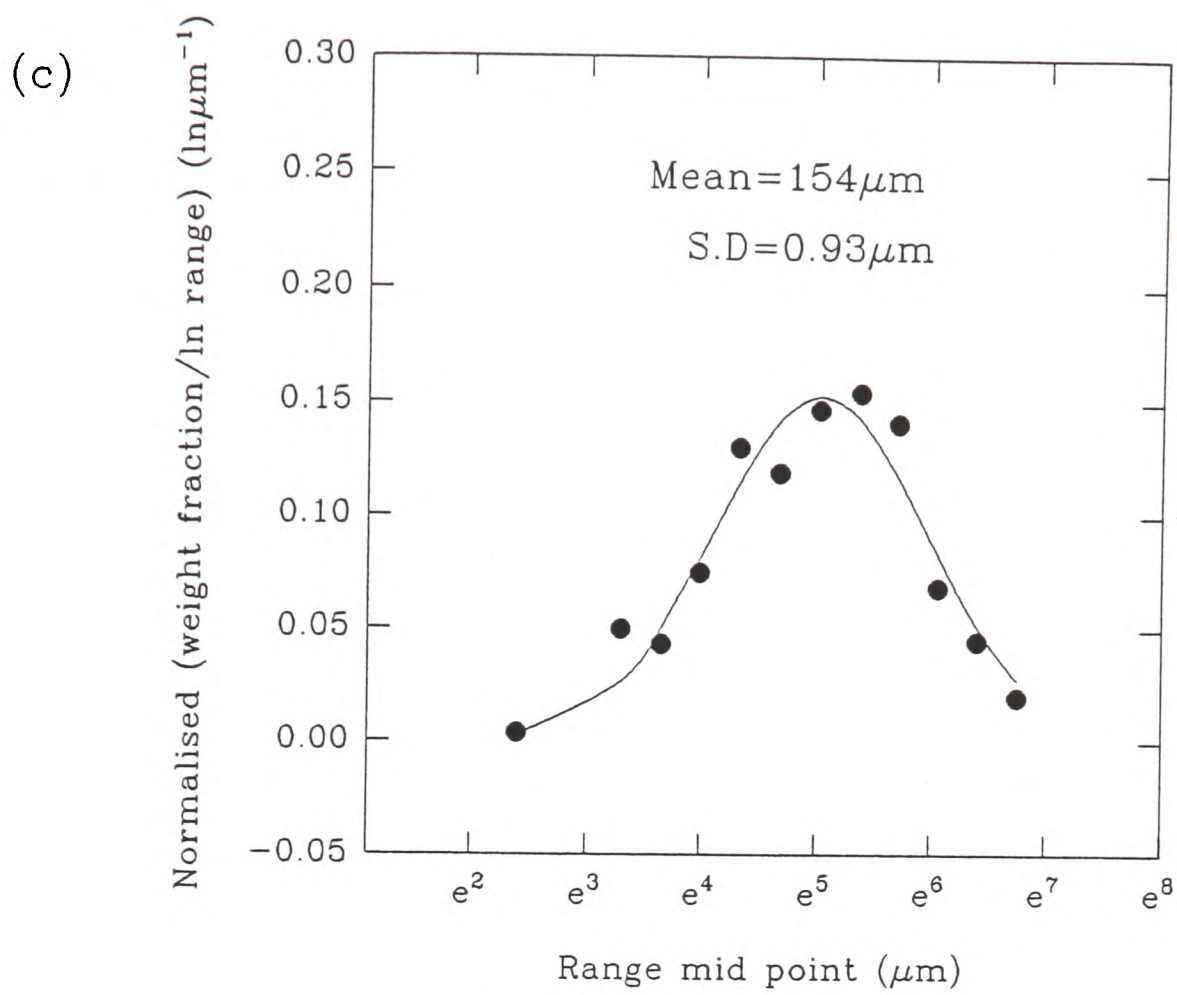
## **5.4 Summary**

A range of deposit microstructures were observed from high porosity splat structures to low porosity equiaxed grain structures. The microstructure was correlated with deposit temperature as measured by embedded thermocouples and infra-red thermal imaging. The transition from high porosity (2-4%) splat to low porosity (<1%) equiaxed grain microstructures was observed to occur above a temperature of  $\approx 1250^{\circ}\text{C}$  and deposition rate of  $\approx 20\text{g/s}$ . The transition in terms of temperature was more reliable.

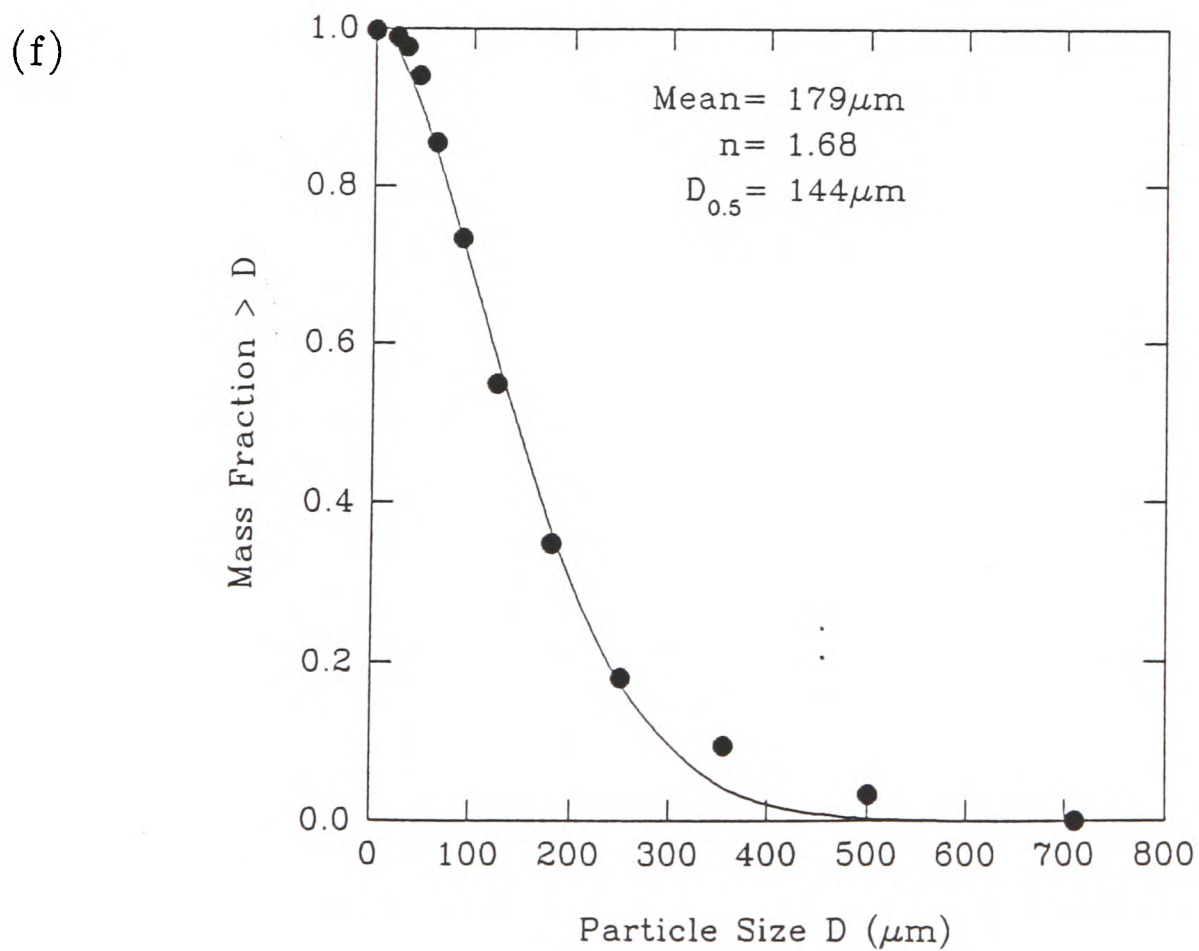
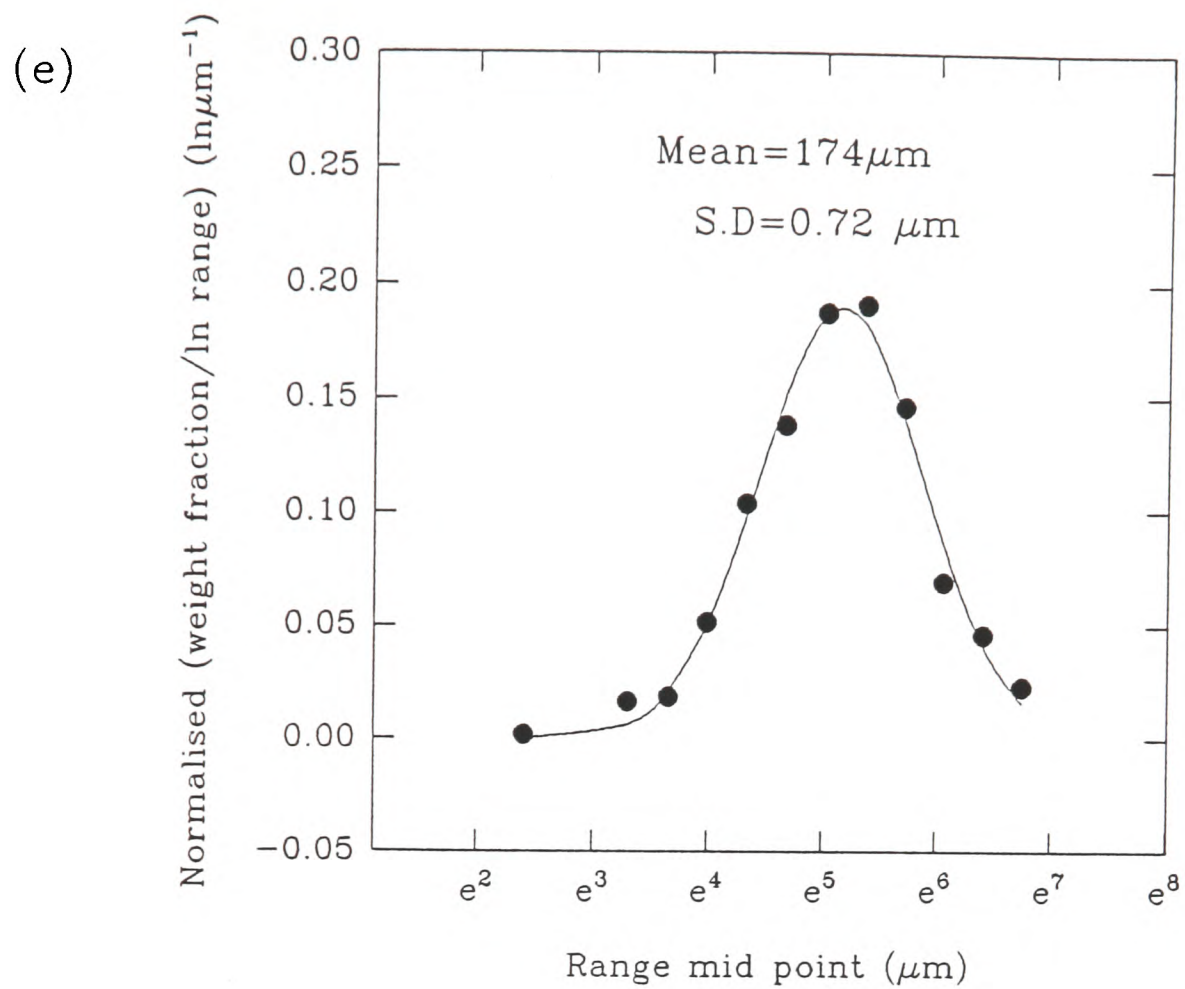


Figs 5.39(a)-(b) Typical overspray powder distributions from run 31 fitted to log-normal and Rosin-Rammler distributions respectively.





cont... Figs 5.39(c)-(d) Typical overspray powder distributions from run 33 fitted to log normal and Rosin-Rammler distributions respectively.



cont... Figs 5.39(e)-(f) Typical overspray powder distributions from run 19 fitted to log normal and Rosin-Rammler distributions respectively.

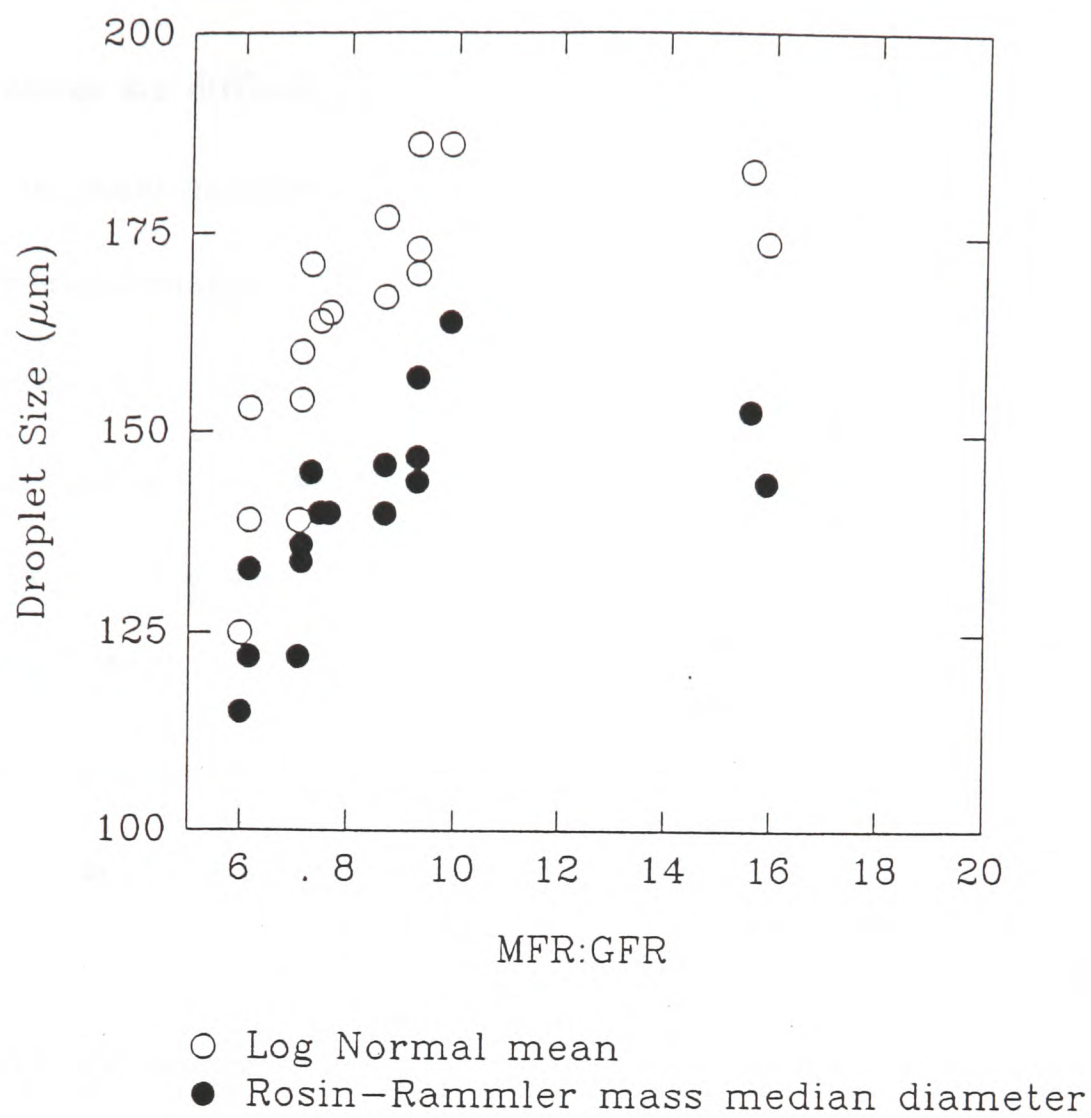


Fig 5.40 Log-normal mean size and Rosin-Rammler mass median size against MFR:GFR for runs 16-37.



However, temperatures are difficult to obtain when spray forming real components and deposition rate is an easier parameter to measure.

Infra-red thermal imaging was used to measure deposit top surface temperatures during spray forming. The temperatures were successfully calibrated using the embedded thermocouple measurements and were correlated with changes in grain size.

Grain size increased with increasing temperature and solidification time. There was reasonable agreement between measured and predicted grain size using previously measured values of  $K$  and an assumed  $d_0=5\mu\text{m}$  and excellent agreement between measured and predicted grain size using a constant value of  $K=10^{-15}\text{m}^3/\text{s}$  and an assumed  $d_0=5\text{m}$ . The mass median size of overspray powder increased with increasing MFR:GFR.

Contraction cracks similar to those observed in conventional castings were present in deposits with a  $T_{\text{max}} > \approx 1250^\circ\text{C}$  i.e.  $> \approx 5\%$  liquid fraction.

## **CHAPTER 6-RESULTS OF THE COMPUTER MODELLING OF DROPLETS IN FLIGHT USING FLUENT<sup>1</sup>**

This chapter presents the computer modelling results obtained using a commercial 2-dimensional finite difference based program, FLUENT, to calculate the behaviour of UDIMET 720 droplets during spray forming under the same range of experimental conditions as described in chapter 5. The dynamic and thermal behaviour of individual droplets in the spray forming chamber is modelled as well as post processing analysis to determine the thermal condition of the droplet spray and mass distribution arriving at the substrate.

### **6.1 The FLUENT Code**

FLUENT V4.22 is a general purpose computer program for modelling fluid flow, heat transfer and chemical reaction. It models this range of phenomena by solving the appropriate steady state conservation equations for mass, momentum and energy for mean gas velocities, pressure and enthalpy, as well as similar conservation equations for the kinetic energy of turbulence and its dissipation rate. All conservation equations have the generalised form:

$$\text{div}(\rho_g V_g \Phi - \Gamma \Delta \Phi) = S_\Phi - S_{\Phi d} \quad (6.1)$$

where  $\rho_g$  and  $V_g$  are the gas density and velocity respectively,  $\Phi$  is the dependent variable,  $\Gamma$  is either the remaining effective viscosity or thermal diffusivity,  $S_\Phi$  is a source term for  $\Phi$  per unit volume and  $S_{\Phi d}$  is a source term for coupling to the dispersed phase.

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<sup>1</sup> FLUENT is a registered trademark of FLUENT Inc. Centerra Resource Park, 10 Cavendish Court, Lebanon, NH 03766 U.S.A.

The values of  $S_\Phi$ ,  $S_{\Phi_d}$  and  $\Gamma$  for the dependent variables ( $\Phi$ ) of axial gas velocity ( $u_g$ ), radial gas velocity ( $v_g$ ), enthalpy ( $H$ ), turbulent kinetic energy ( $k$ ) and its dissipation ( $\epsilon$ ) are given in appendix A for the 2-dimensional axisymmetric form of equation 6.1 (FLUENT manual 1993, Fritsching *et al* 1993, Bewlay and Cantor 1989).

The conservation equations are discretised on a curvilinear grid to enable computations in complex/irregular geometries (FLUENT manual 1993, pp1-6). A complete description of the theoretical basis of FLUENT is given in the manual (FLUENT manual 1993, pp831-880). FLUENT consists of two parts: (i) a preprocessor, preBFC V4 to define the geometry and structured grid for the problem; and (ii) the FLUENT main module to define physical models, fluid/material properties, boundary conditions for the problem and to solve the steady state fluid flow in the defined computational domain. The basic program structure of FLUENT is shown in fig 6.1.

## **6.2 Problem Setup**

The aim in defining the problem in terms of the FLUENT program structure is to reproduce as realistically as possible the actual conditions of the droplet spray in the spray forming chamber. Obviously, limitations of the program hinder the exact experimental situation, however, a reasonable approximation can be achieved.

### **6.2.1 Geometry and Boundary Conditions**

Fig 6.2 shows the physical geometry representing (a) the whole spray forming chamber and atomiser for a spray distance of 0.3m and (b) an enlarged view of the atomiser region. The real substrate arrangement is simplified to a 0.05m thick, 0.2m diameter disk and the atomiser which consists of 16 circular holes arranged around the



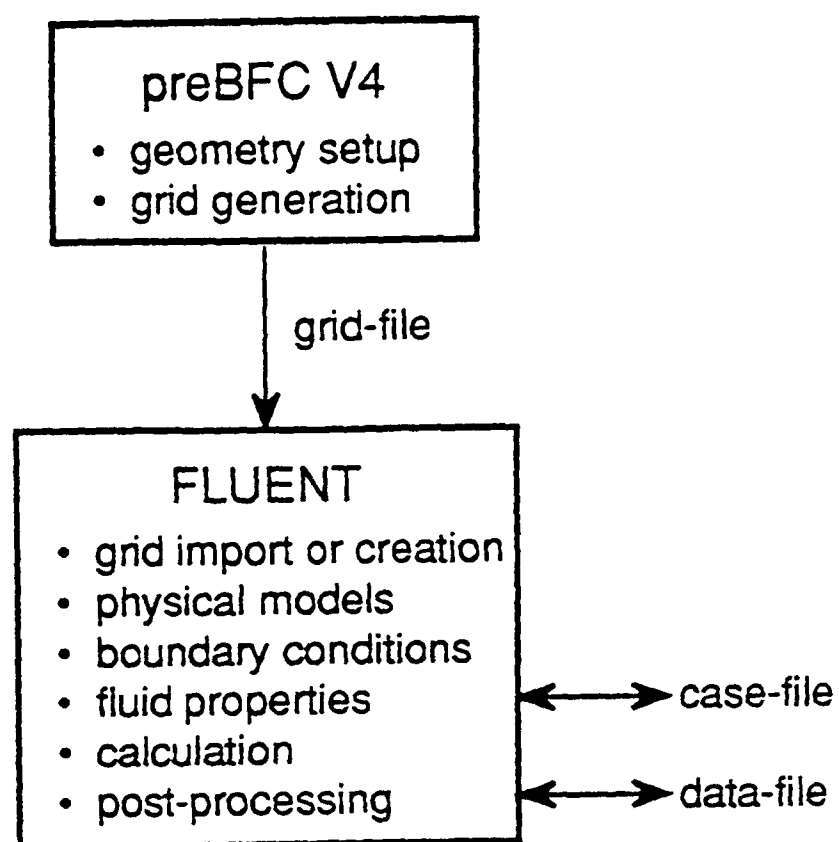


Fig 6.1 Fluent V4.22 Basic program structure.

melt nozzle is simplified as a continuous annular slit with the same total exit area. The geometric boundaries labelled in fig 6.2 are described below and the associated boundary conditions summarised in table 6.1:

- (i) S=symmetry axis.
- (ii) I1=gas inlet has a fixed pressure of  $P_{gas}$ , the measured gas pressure (relative to atmospheric pressure), assumed turbulent intensity of 10% and temperature of 27°C.
- (iii) I2=outlet boundary has a fixed pressure of atmospheric pressure, assumed turbulent intensity of 10% and temperature of 250°C, the approximate measured gas exit temperature.
- (iv) W2=atomiser insert which surrounds the melt nozzle has a constant temperature of 727°C, the approximate measured atomiser insert temperature.
- (v) W3=melt exit from the nozzle has a constant temperature 1445°C the measured melt superheat temperature.
- (vi) W1=all other boundaries i.e. chamber wall, substrate and atomiser have a constant temperature of 70°C, the approximate measured chamber wall temperature.

|    | axial vel.     | radial vel. | Pressure*  | Turbulent Intensity (%) | Characteristic Length (m) | Temperature (°C) |
|----|----------------|-------------|------------|-------------------------|---------------------------|------------------|
| W1 | 0              | 0.          | -          | -                       | -                         | 70               |
| W2 | 0              | 0           | -          | -                       | -                         | 727              |
| W3 | 0              | 0           | -          | -                       | -                         | 1445             |
| I  | Fixed Pressure |             | $P_{flow}$ | 10                      | 0.1                       | 25               |
| O  | Fixed Pressure |             | 0          | 10                      | 0.1                       | 250              |
| S  | -              |             |            |                         |                           |                  |

Table 6.1 Summary of boundary conditions for the continuous phase.

\* - pressures are relative to atmospheric pressure.

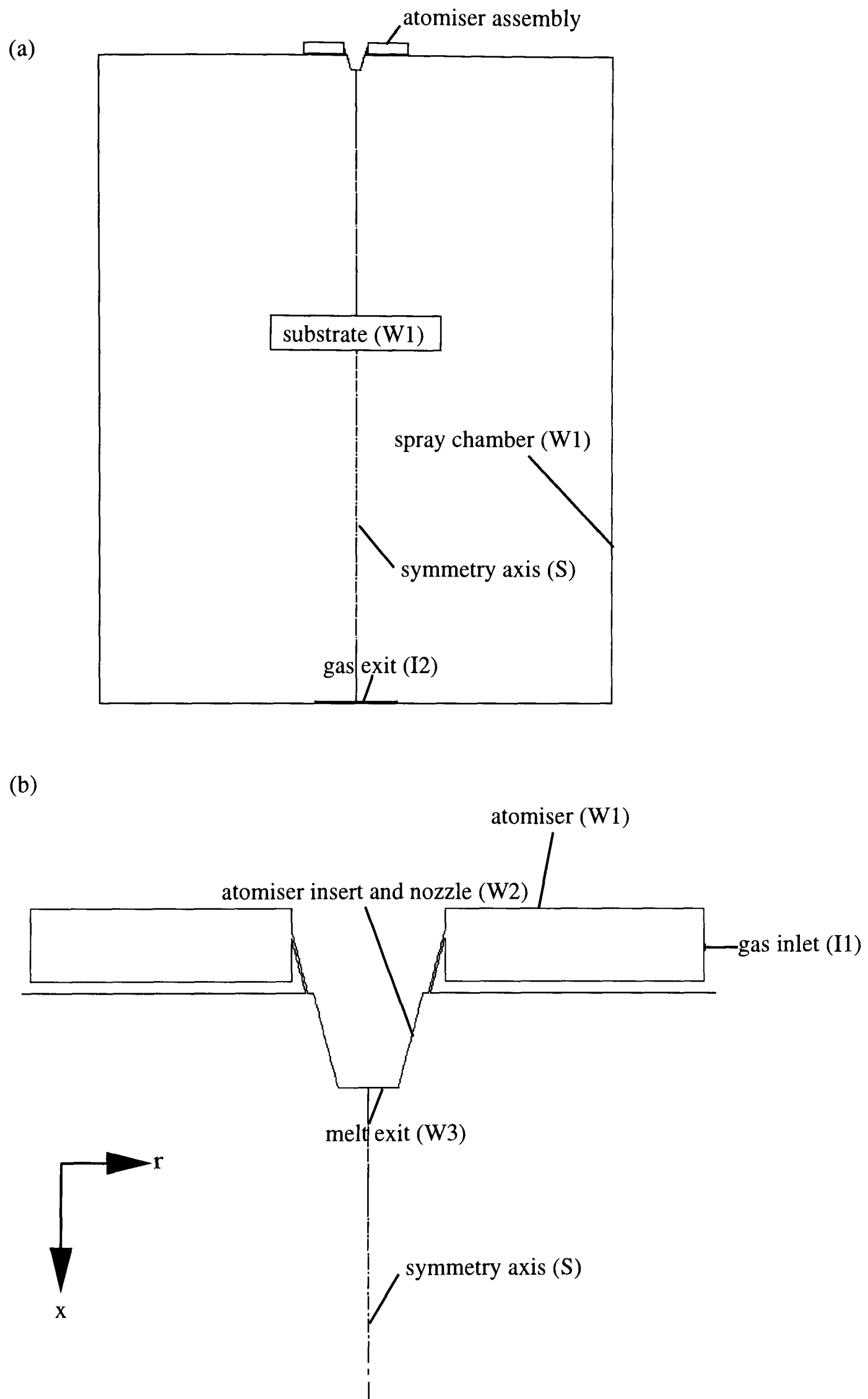


Fig 6.2 Outline of the spray chamber and atomiser geometry showing: (a) the whole spray chamber and atomiser and (b) an enlarged view of the atomiser. The boundaries are labelled as follows: S=symmetry axis, I1=inlet boundary, I2=exit boundary, W2=atomiser insert and nozzle boundary, W3=melt exit boundary and W1=remaining boundaries i.e. spray chamber, substrate and atomiser boundaries.

Droplets are reflected (perfectly elastic) at S, W2 and W3 boundaries, trapped at the W1 (chamber wall and substrate) boundary and escape at I1 and I2 boundaries. However, in practice droplets only impact the S, W1 and I2 boundaries.

### **6.2.2 Grid**

A body fitted coordinate (BFC) system is used to create a structured grid in the above geometry. Wherever possible the grid is created so that cell boundaries are orthogonal to each other which aids the accuracy of the solver. Also the density of cells in regions where flow gradients are high, such as the atomiser exit and atomisation region, is increased to improve the flow resolution in these regions. Fig 6.3 shows the physical grid: (a) for the whole of the chamber and (b) an enlarged view of the atomiser and atomisation region for a spray distance of 0.3m indicating the high density of grid points in the atomiser and atomisation region.

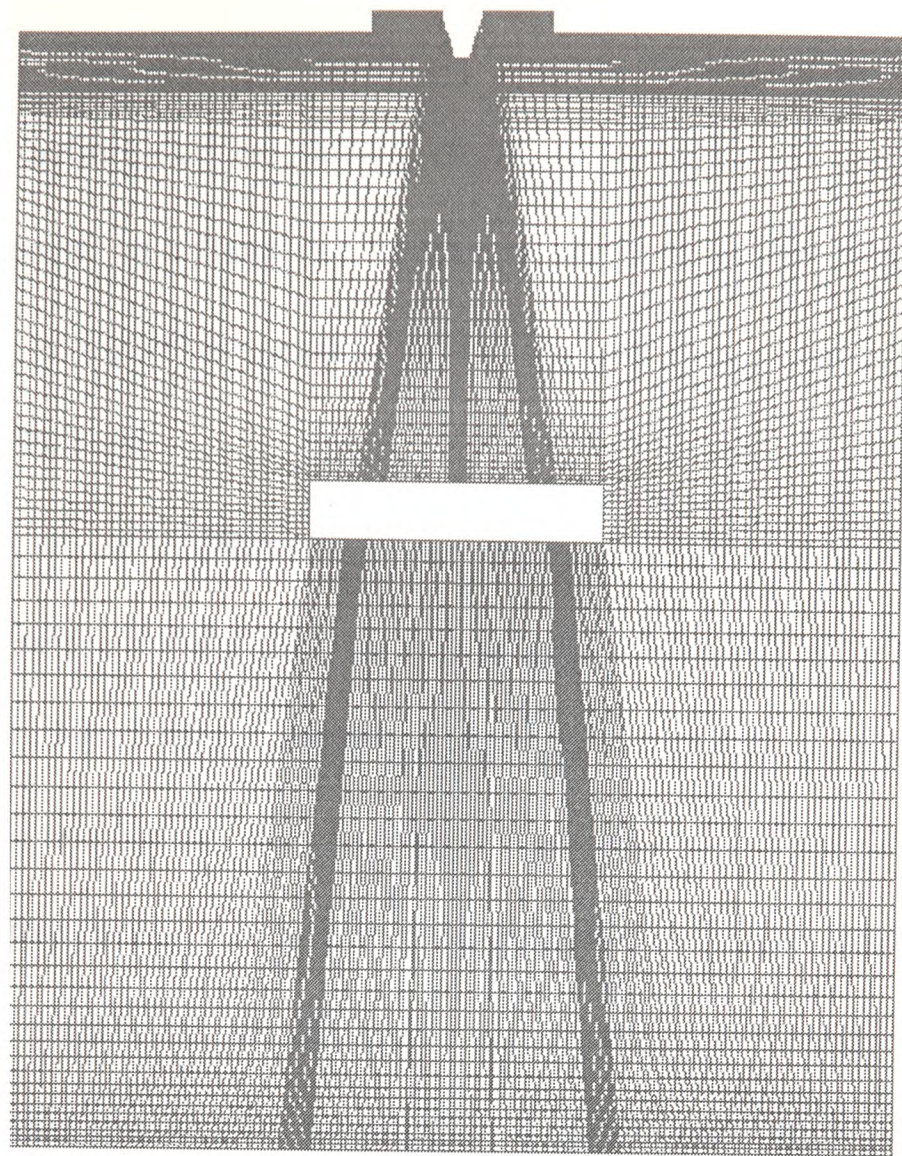
### **6.2.3 Physical Models**

The solution of the flow of droplets in a gas flow field involves two steps: (i) the single phase flow of gas in the chamber is modelled using a Eulerian formulation i.e. a cartesian frame coordinate system; and (ii) the dynamic and thermal behaviour of droplets moving in the above gas flow field is modelled using a Lagrangian formulation i.e. a droplet based coordinate system to allow for momentum and heat exchange between gas and droplets.

For solution of the gas flow field three of FLUENT's physical models are activated: (i) the turbulent k- $\epsilon$  model (Launder and Spalding 1974); (ii) the heat transfer



(a)



(b)

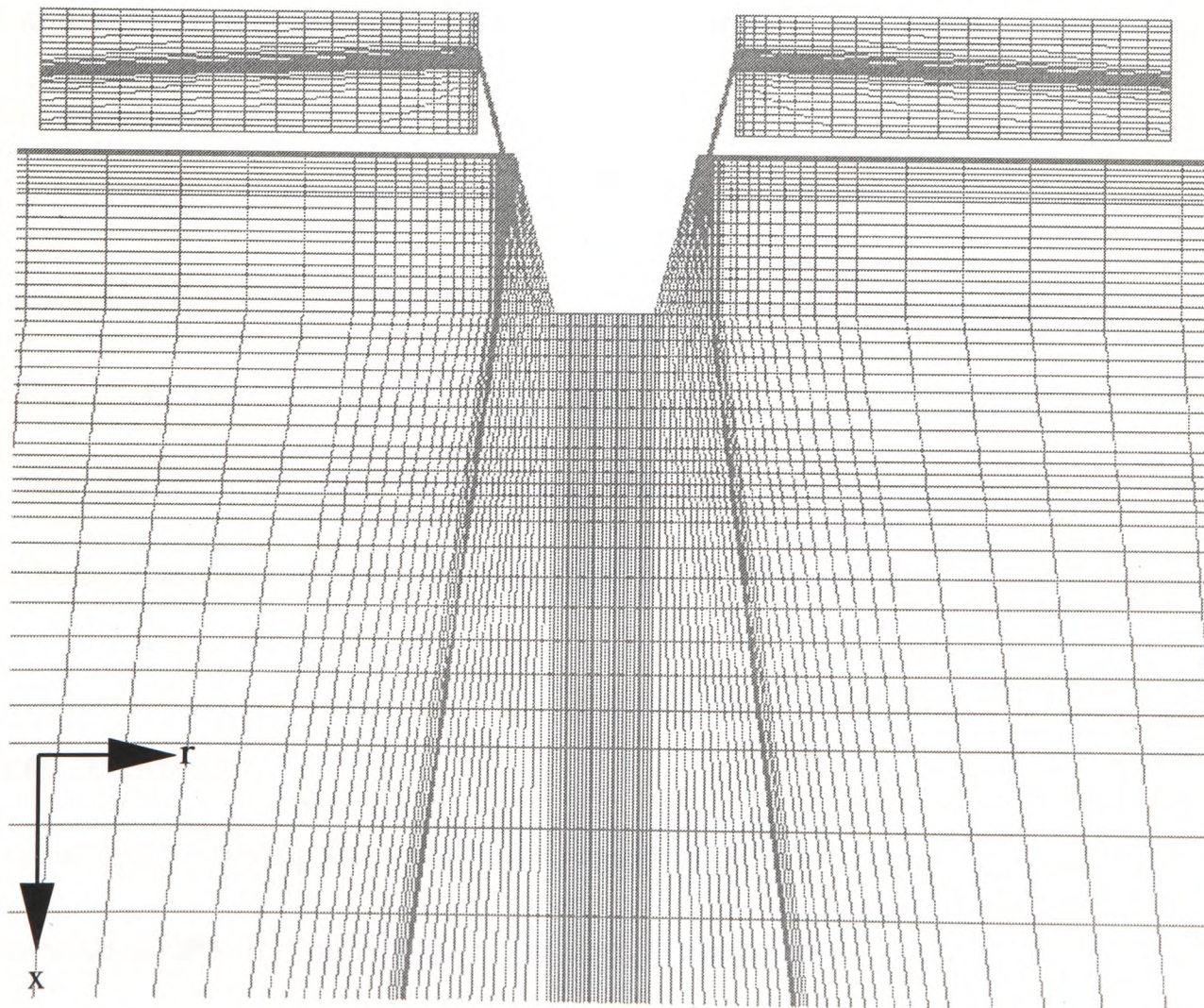


Fig 6.3 The physical grid used showing: (a) the whole spray chamber and atomiser and (b) an enlarged view of the atomiser region.



model which activates solution of the energy equation; and (iii) the compressible flow model which defines density as a function of pressure and is important when the gas flow velocity approaches or exceeds the speed of sound, for instance on exiting the atomiser (FLUENT manual pp175-340).

In the solution of two phase flows involving a dispersed second phase the following physical models are activated: (i) calculation of the second phase trajectory using a Lagrangian formulation that includes the second phase inertia, hydrodynamic drag and gravity; (ii) cooling and solidification of the second phase; and (iii) coupling of the continuous phase flow field prediction to the second phase calculations. Appendix A gives further details of these models. As the droplets move through the chamber, momentum and energy transfer from the gas to the droplet at each grid point is retained and subsequently incorporated in re-calculations of the gas flow field. Coupling of the continuous and second phases is accomplished by alternately solving the continuous and second phase equations until the solutions in both phases have ceased to change. When this occurs a converged two phase solution is accomplished. Typically this is when the sum of the normalised enthalpy residuals is  $<10^{-6}$  and the sum of the remaining residuals of pressure ( $p$ ), axial velocity ( $u_g$ ), radial velocity ( $v_g$ ), turbulent kinetic energy ( $k$ ) and dissipation rate ( $\epsilon$ ) is  $<10^{-3}$ . Fig 6.4 shows a schematic diagram of the interphase exchange of heat, mass and momentum between the continuous and second phases.

#### **6.2.4 Physical Properties**

For solutions involving flow and heat transfer the following properties are required for the continuous phase: (i) density ( $\rho_g$ ), for compressible flow this is defined using the

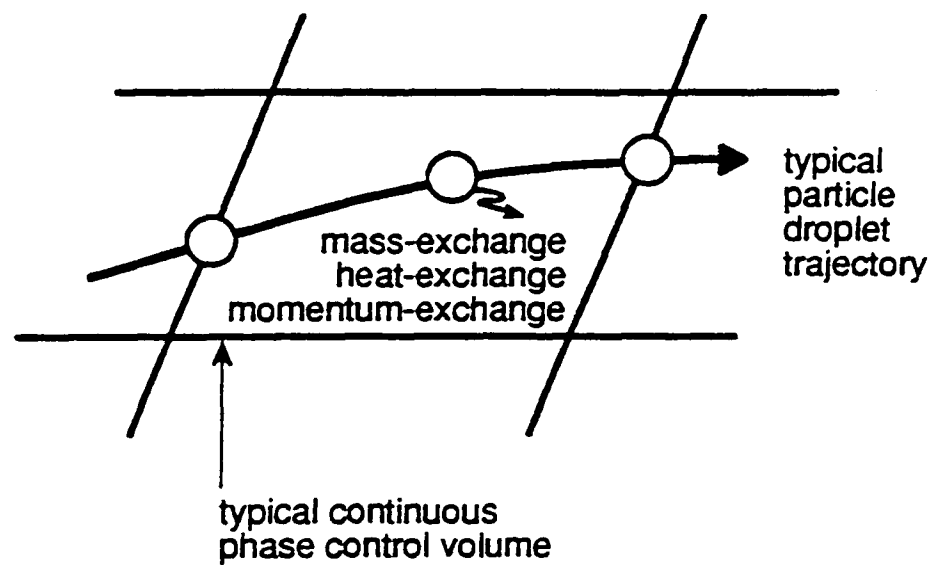


Fig 6.4 Heat, mass and momentum transfer between the second and continuous phases.

ideal gas law; (ii) viscosity ( $\mu$ ); (iii) specific heat capacity ( $c_p$ ); and (iv) thermal conductivity ( $k$ ). The value of these properties for  $N_2$  are shown in table 6.2.

|            | Density<br>(kg/m <sup>3</sup> ) | Latent<br>Heat<br>(Jkg <sup>-1</sup> ) | Viscosity<br>(kgm <sup>-1</sup> s <sup>-1</sup> ) | Thermal<br>Conductivity<br>(Wm <sup>-1</sup> K <sup>-1</sup> ) | Specific Heat<br>Capacity<br>(Jkg <sup>-1</sup> K <sup>-1</sup> ) |
|------------|---------------------------------|----------------------------------------|---------------------------------------------------|----------------------------------------------------------------|-------------------------------------------------------------------|
| $N_2$      | Use Ideal<br>Gas Law            | -                                      | $1.67 \times 10^{-5}$                             | $2.43 \times 10^{-2}$                                          | 1040                                                              |
| UDIMET 720 | $8.08 \times 10^3$              | $145 \times 10^3$                      | -                                                 | -                                                              | $c_{eq}$                                                          |

Table 6.2 Physical property data for continuous ( $N_2$ ) and second phase (UDIMET 720).

The following property inputs are required for the second phase: (i) initial density ( $\rho$ ); (ii) latent heat ( $L$ ); and (iii) specific heat ( $c_p$ ), throughout the temperature range. The effect of latent heat is included by using an equivalent specific heat for the droplets ( $c_{eq}$ ). Assuming that the variation of solid fraction with temperature is given by the Scheil equation (Flemings 1974) as shown in fig 3.12 then:

$$c_{eq} = -L \frac{df}{dT} + c_p(T) \tag{6.2}$$

where  $c_{eq}$  is the equivalent specific heat,  $L$  is the latent heat,  $f$  is the solid fraction and  $c_p(T) = a + bT$  Jkg<sup>-1</sup>K<sup>-1</sup>, where  $a$  and  $b$  are constants determined by Neumann and Kopp's rule to be 425Jkg<sup>-1</sup>K<sup>-1</sup> and 0.19Jkg<sup>-1</sup> respectively (Smithells Metals Reference Book 1992). Table 6.2 shows a summary of the physical properties for the second phase (UDIMET 720). Fig 6.5 shows the equivalent specific heat against temperature for UDIMET 720 calculated using equation 6.2. Grant (1991) suggested that the exact shape of the  $c_{eq}$  curve is not important as long as the latent heat ( $L$ ) is correct. Therefore to improve the resolution near the liquidus temperature and to try and minimise numerical enthalpy losses by overshooting the liquidus, the data are modified, somewhat arbitrarily, so that the large



increase in  $c_{eq}$  is assumed to take place over  $3^{\circ}\text{C}$  instead of  $0.1^{\circ}\text{C}$ , while keeping  $L$  constant. The variation of  $c_{eq}$  with temperature is input to FLUENT as a piecewise linear variation, the linear regions chosen for input are indicated by the symbols in fig 6.5.

### **6.2.5 Solver Parameters**

A detailed description of the FLUENT solver is given in the manual pp638-690. However, an outline of the method used to obtain a fully converged two phase solution is shown in fig 6.6. The case file stores all the information on the geometry, physical property and boundary conditions, and the data file stores all the solutions for the gas flow field and droplet trajectories.

### **6.2.6 Droplet Injection**

The actual atomisation process is not modelled because of its complicated nature (Bradley 1973, See and Johnston 1978). Therefore, droplets are injected into the gas flow at certain points in the approximate atomisation region and their subsequent dynamic and thermal behaviour calculated. From the examination of the frozen metal at the tip of the nozzle after spray forming, atomisation probably occurred  $\approx 0.03\text{m}$ - $0.038\text{m}$  from the top of the chamber and over a radial distance of  $\approx 0.006\text{m}$  from the spray axis. Analysis of the overspray powder indicates that the droplet size range was  $\approx 10\mu\text{m}$ - $700\mu\text{m}$  as shown in fig 5.39. Droplets are therefore injected in groups of 20 droplets with diameters spread linearly across the droplet size range of  $10\text{-}700\mu\text{m}$  i.e. the first droplet has a diameter of  $10\mu\text{m}$  and the 20th droplet a diameter of  $700\mu\text{m}$  and each droplet is injected at a different point. Each group of droplets has a set of specified properties and the shape of the droplet

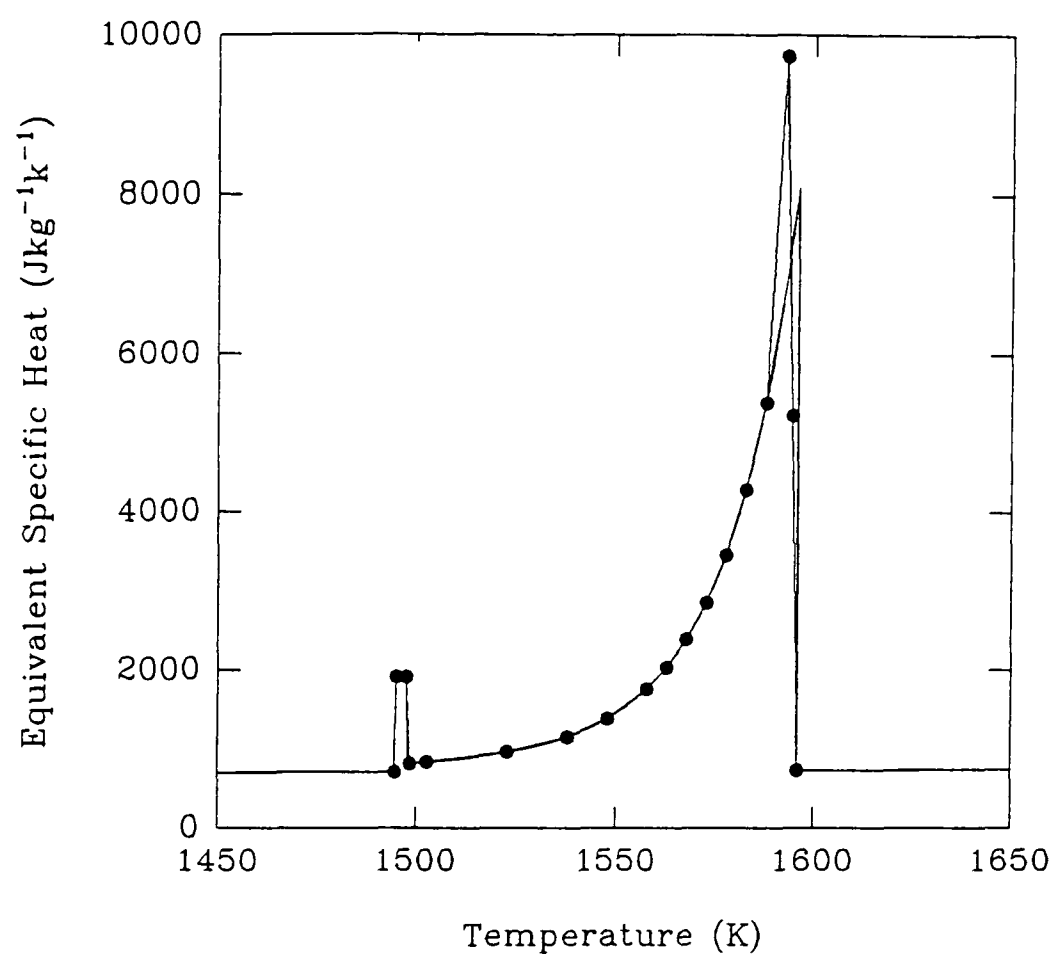


Fig 6.5 Equivalent specific heat against temperature for UDIMET 720.

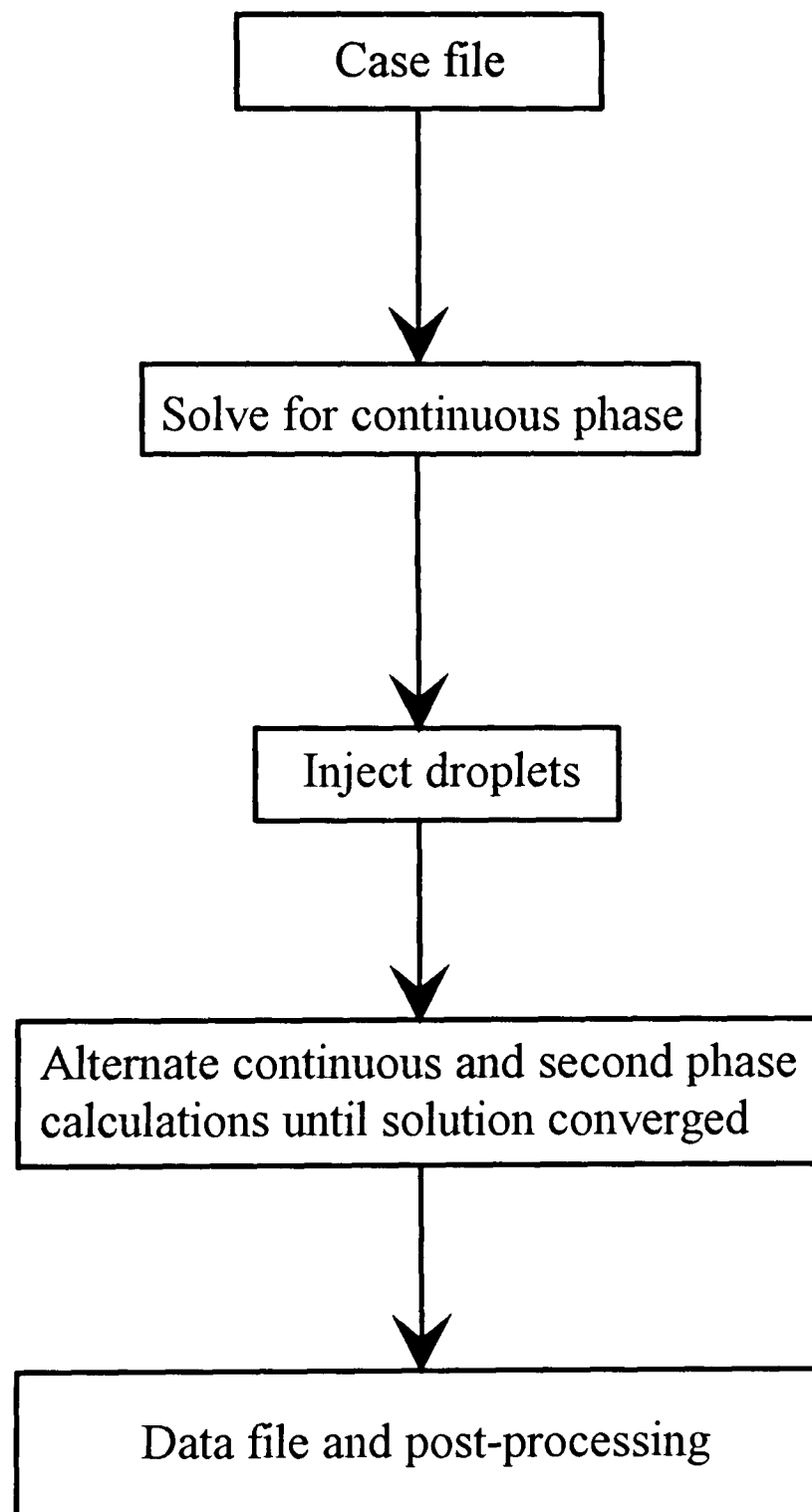


Fig 6.6 Outline of the solver manipulation to obtain a converged two phase solution.

size distribution from 10-700µm is described by the Rosin-Rammler distribution. The injection properties of a typical group of 20 droplets is given in table 6.3. The droplets are injected at equal distances along the line (x1,r1)-(x2,r2) i.e. droplet 1 of size d1 is injected at (x1,r1) and droplet 20 of size d2 is injected at (x2,r2) with all other droplets distributed equally in between. The droplets all have the same initial temperature and velocity. The initial droplet temperature is the same as the melt superheat. The initial radial droplet velocity ( $v_d$ ) is zero and the initial axial droplet velocity ( $u_d$ ) is related to the experimental MFR (melt flow rate) as shown below:

$$u_d = \text{MFR} / \rho A_d \tag{6.3}$$

where MFR is the experimental melt flow rate,  $\rho$  is the density of UDIMET 720 and  $A_d$  is the exit area of the melt delivery nozzle.

|                                     |         |
|-------------------------------------|---------|
| Droplet Label                       | 1-20    |
| x1 (m)                              | 0.034   |
| x2 (m)                              | 0.034   |
| r1 (m)                              | 0.006   |
| r2 (m)                              | 0.00405 |
| axial velocity (ms <sup>-1</sup> )  | 1.03    |
| radial velocity (ms <sup>-1</sup> ) | 0       |
| Temperature (°C)                    | 1445    |
| d1 (µm)                             | 10      |
| d2 (µm)                             | 700     |
| Rosin-Rammler Mean Size (µm)        | 183     |
| Rosin-Rammler Range (µm)            | 1.66    |
| Total MFR (gs <sup>-1</sup> )       | 59      |

Table 6.3 Injection conditions for a typical group of 20 droplets.



When droplets are injected in more than one group the MFR of each group of droplets is adjusted so that the total MFR is distributed equally between each of the droplet groups injected. The sensitivity of the results to the precise location of where droplets are injected is analysed in more detail in section 6.3.3.

### **6.2.7 Post Processing**

A FORTRAN program written originally by Dr. PS Grant is used to analyse the droplet trajectories and temperatures (Appendix B). This program calculates:

(i) the mass arrival rate due to the droplets arriving at a series of radial bins at the substrate as shown in equation 6.4. Four 13.75mm wide radial bins are used to span the radius of the substrate.

$$M_{ar}(j) = \sum_{i=1}^n (Pr(i) * MFR) \quad (6.4)$$

where  $M_{ar}(j)$  is the total mass arrival rate ( $gs^{-1}rad^{-1}$ ) in the  $j^{th}$  radial bin,  $Pr(i)$  is the mass probability of the  $i^{th}$  droplet, given by the Rosin-Rammler distribution, MFR is the total melt flow rate of all the injected droplets and  $n$  is the total number of droplets which arrive in the  $j^{th}$  radial bin. The mass arrival rate is then converted to a total mass arrival rate/area ( $M_{ar/a} - gs^{-1}m^{-2}$ ) for each radial bin in 3-d as follows:

$$M_{ar/a}(j) = \frac{2\pi M_{ar}(j)}{A_j} \quad (6.5)$$

where  $M_{ar/a}(j)$  is the mass arrival rate/area in the  $j^{th}$  radial bin and  $A_j$  is the 3-d area of the  $j^{th}$  radial bin.

(ii) the equilibrated enthalpy ( $H_{sp}$ ) of all the droplets which arrive at the substrate as

shown below:

$$H_{sp} = \frac{\sum_{i=1}^n H(i) * Pr(i) * MFR}{\sum_{i=1}^n Pr(i) * MFR} \quad (6.6)$$

where  $H(i)$  is the enthalpy of the  $i^{th}$  droplet which arrives at the substrate,  $Pr(i)$  is the mass probability of the  $i^{th}$  droplet, given by the Rosin-Rammler distribution, MFR is the total melt flow rate of all injected droplets and  $n$  is the total number of droplets which arrive at the substrate. The equilibrated temperature ( $T_{sp}$ ) of all the droplets which arrive at the substrate is then given by the definition of enthalpy ( $H$ ):

$$H = \int_0^T c_{eq} dT \quad (6.7)$$

where  $c_{eq}$  is the equivalent droplet specific heat given by equation 6.2 and  $T$  is the temperature in Kelvin.

The following key variables are used when comparing the results using different process parameters: (i)  $T_{sp}$  at the substrate; (ii)  $M_{ar/a}$  in each of the radial bins at the substrate normalised by the maximum  $M_{ar/a}$ ; (iii) variation of mean (time averaged) droplet axial velocity ( $u_d$ ) with axial distance; (iv) variation of mean (time averaged) droplet radial distance (divergence) with axial distance; and (v) variation of mean (time averaged) droplet temperature with axial distance.

### **6.3 Model Sensitivity**

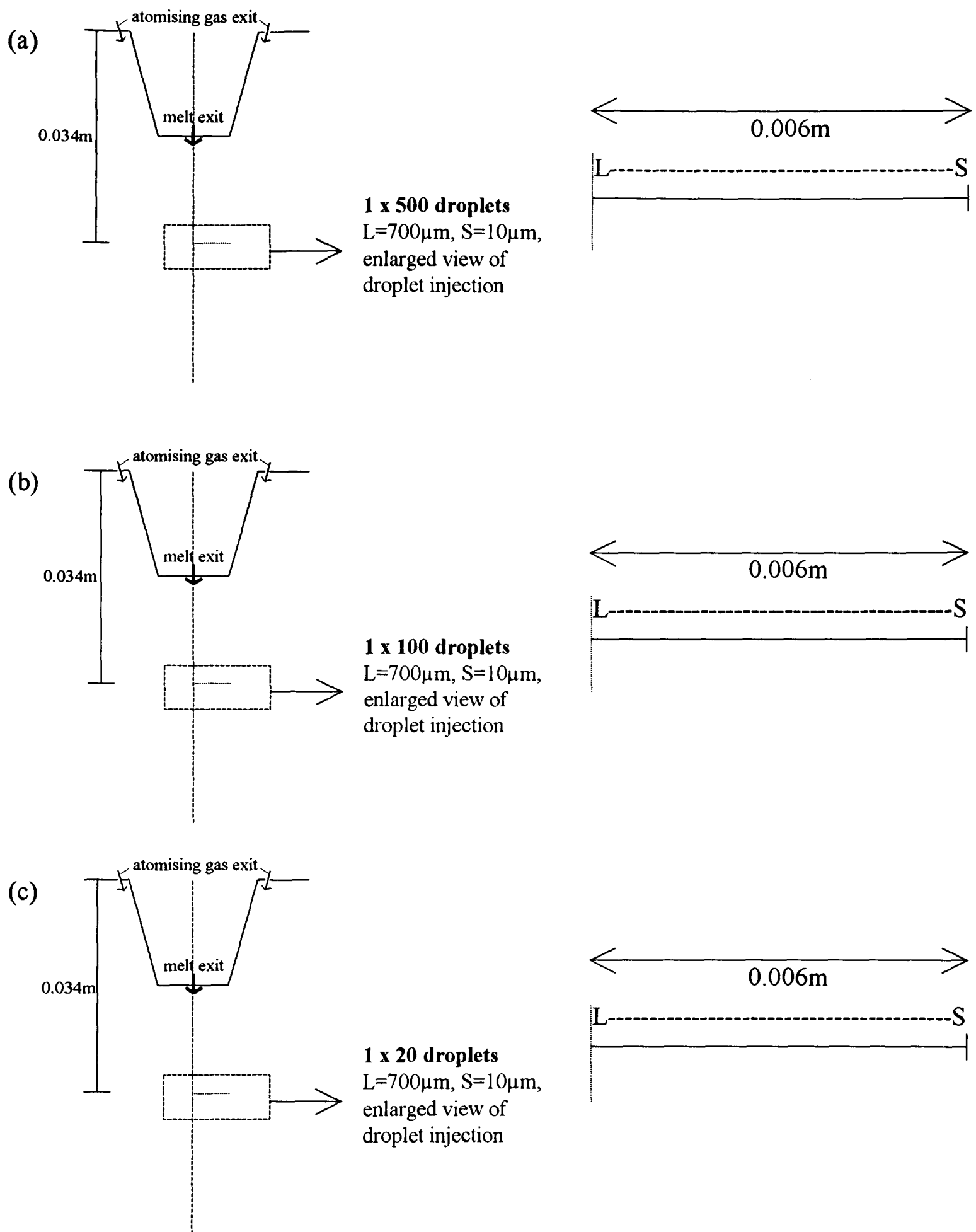
The sensitivity of the model to the following conditions was investigated: (i)

number of droplets injected; (ii) starting axial position of injection; (iii) initial turbulent intensity of gas phase; (iv) W1 thermal boundary conditions; and (v) radial injection position of different size droplets. All the other model process parameters, total MFR ( $59\text{gs}^{-1}$ ), initial droplet axial velocity ( $1.03\text{ms}^{-1}$ ), mean Rosin-Rammler droplet size and range ( $183\mu\text{m}$  and  $1.52$ ), spray distance ( $0.25\text{m}$ ), superheat ( $1445^\circ\text{C}$ ), atomising gas pressure ( $0.503\text{MPa}$ ), W2, W3, I1 and I2 temperatures ( $727^\circ\text{C}$ ,  $1445^\circ\text{C}$ ,  $27^\circ\text{C}$  and  $250^\circ\text{C}$  respectively) were kept constant throughout.

### **6.3.1 Number of Droplets**

Fig 6.7 shows schematic diagrams indicating the droplet injection positions for one group of (a) 500 droplets, (b) 100 droplets and (c) 20 droplets. In all cases droplets were injected along a single horizontal line,  $0.034\text{m}$  from the top of the chamber with the largest droplet ( $L=700\mu\text{m}$ ) at the spray axis and the smallest droplet ( $S=10\mu\text{m}$ ) at the edge of the injection group.

Fig 6.8 shows the normalised  $M_{\text{ar/a}}$  against radial distance for one group of (a) 500 droplets, (b) 100 droplets and (c) 20 droplets. Figs 6.8(a) and (b) show there is little difference in either the mass distribution or  $T_{\text{sp}}$  of  $\approx 1315^\circ\text{C}$  when the number of droplet was decreased from 500 to 100. However, for a decrease in the number of droplets injected from 100 to 20 in fig 6.8(c) there was a slight change in the shape of the normalised  $M_{\text{ar/a}}$  against radial distance bar chart with more droplets arriving nearer the spray axis. Although in all cases the distribution of mass arriving at the substrate was approximately Gaussian and  $T_{\text{sp}}$  only decreased slightly from  $1316^\circ\text{C}$  in fig 6.8(a) to  $1312^\circ\text{C}$  in fig 6.8(c).



Constant process parameters:

MFR = 59g/s

Droplet axial velocity=1.03m/s

Mean Rosin-Rammler droplet size=183μm

Rosin-Rammler range=1.52

Spray distance=0.25m

Superheat=1445C

Gas pressure=0.503MPa

Wall temperatures:

W1=70C

W2=727C

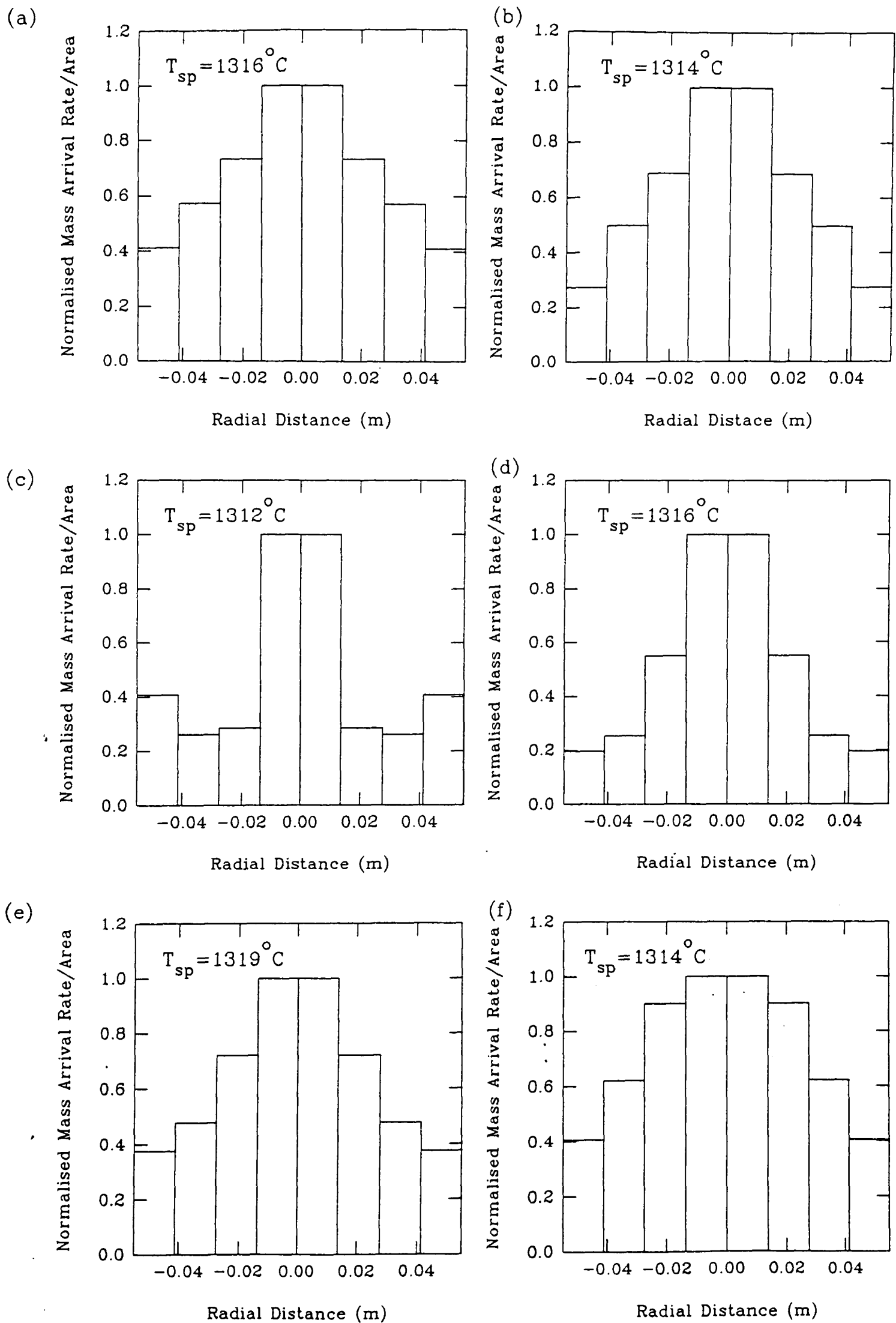
W3=1445C

I1=27C

I2=250C

Fig 6.7 Schematic diagrams indicating the droplet injection positions for one group of: (a) 500 droplets, (b) 100 droplets and (c) 20 droplets.





Figs 6.8(a)-(c) Normalised mass arrival rate/area ( $M_{ar/a}$ ) against radial distance for droplet injection conditions shown in figs 6.7(a)-(c) respectively and droplet injection conditions shown in fig 6.7(b) except with: (d) a starting axial injection position of 0.024m, (e) a turbulent intensity of 15% and (f) zero heat flux W1 boundary condition.

### **6.3.2 Starting Axial Injection Position**

Fig 6.8(d) shows the normalised  $M_{ar/a}$  against radial distance for the same injection conditions and process parameters as in fig 6.8(b) except that the starting axial injection position decreased from 0.034m to 0.024m from the top of the chamber. The shape of the normalised  $M_{ar/a}$  against radial distance bar chart in fig 6.8(d) was not significantly different from fig 6.8(b) i.e. approximately a Gaussian distribution and  $T_{sp}$  increased slightly from 1314°C in fig 6.8(b) to 1316°C in fig 6.8(d) as the starting axial injection position was decreased from 0.034m to 0.024m.

### **6.3.3 Turbulence**

Fig 6.8(e) shows the normalised  $M_{ar/a}$  against radial distance for the same injection conditions and process parameters as in fig 6.8(b) except that the gas phase turbulent intensity at I1 and I2 boundaries increased from 10% to 15%. The shape of the normalised  $M_{ar/a}$  against radial distance bar chart in fig 6.8(e) was not significantly different from fig 6.8(b) i.e. approximately a Gaussian distribution and  $T_{sp}$  increased slightly from 1314°C in fig 6.8(b) to 1319°C in fig 6.8(e) as the inlet and exit turbulent intensities were increased from 10% to 15%.

### **6.3.4 Wall Boundary Conditions**

Fig 6.8(f) shows the normalised  $M_{ar/a}$  against radial distance for the same injection conditions and process parameters as in fig 6.8(b) except that the W1 (chamber wall, substrate and atomiser wall) boundary condition was changed from a constant temperature of 80°C to a zero heat flux (adiabatic system) condition. The shape of the normalised  $M_{ar/a}$

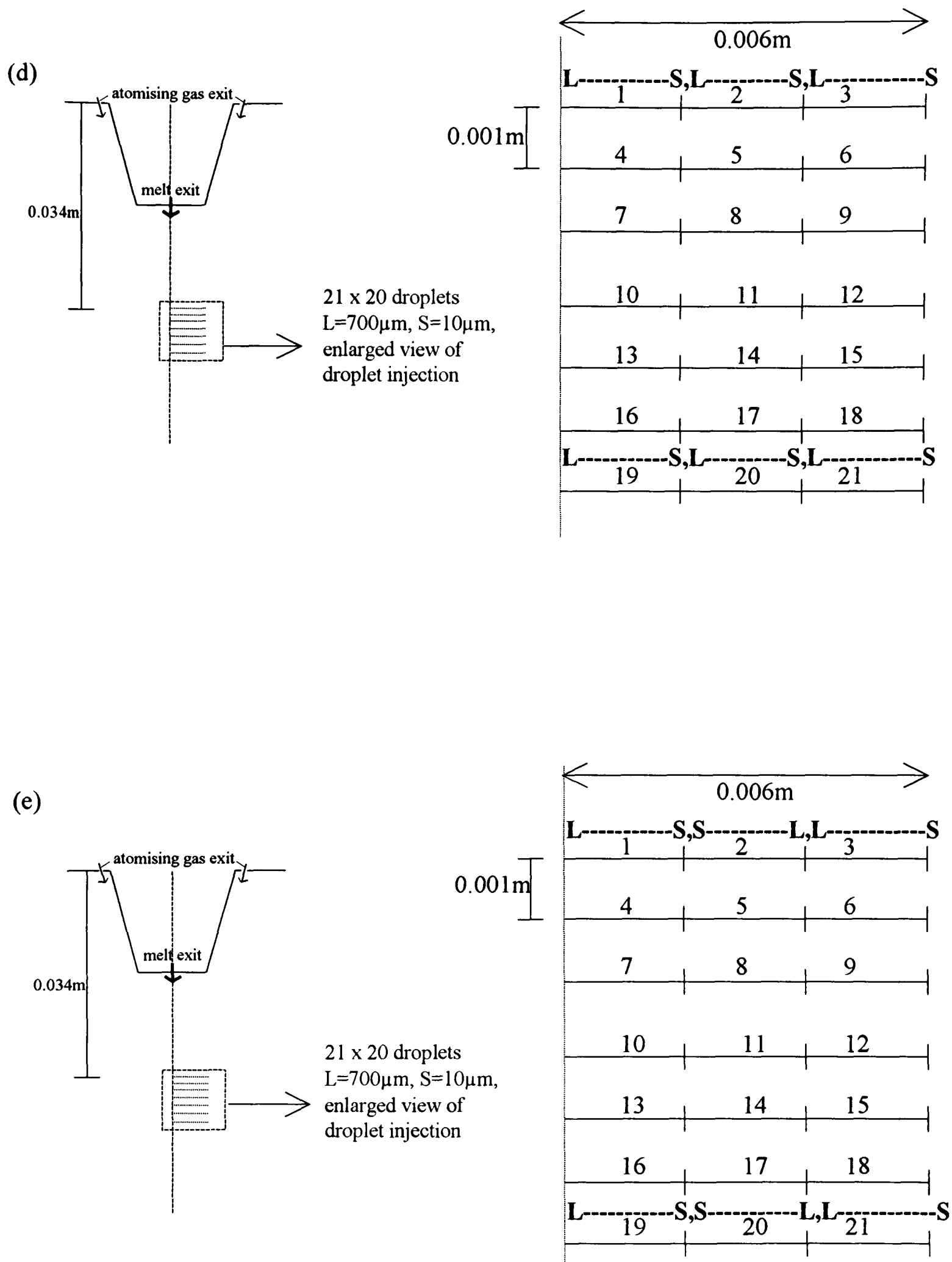
against radial distance bar chart in fig 6.8(f) was not significantly different from fig 6.8(b) i.e. approximately a Gaussian distribution, and  $T_{sp}$  was unchanged.

### **6.3.5 Radial Distribution of Different Size Droplets**

Fig 6.8(g) shows the normalised  $M_{ar/a}$  against radial distance for the same injection conditions and process parameters as in fig 6.8(b) except that the radial distribution of droplet sizes was reversed so that the largest droplet ( $L=700\mu\text{m}$ ) was injected at the edge of the group and the smallest droplet ( $S=10\mu\text{m}$ ) at the spray axis. In this case, the shape of the normalised  $M_{ar/a}$  against radial distance bar chart in fig 6.8(g), where more mass collected at greater radial distances was very much altered in comparison with fig 6.8(b). Also, there was a decrease in  $T_{sp}$  from  $1314^\circ\text{C}$  in fig 6.8(b) to  $1308^\circ\text{C}$  in fig 6.8(g) as the radial distribution of droplet sizes was reversed.

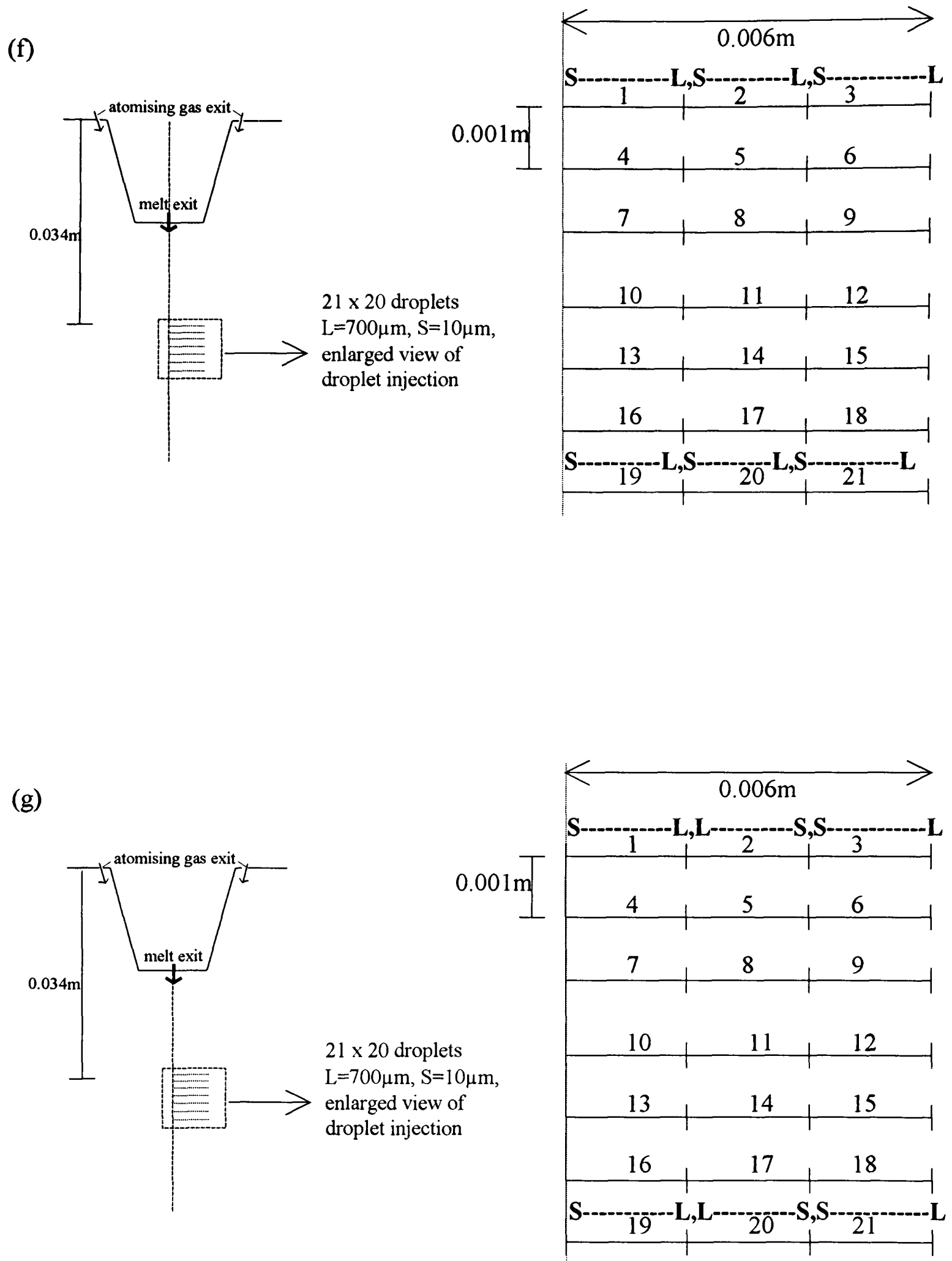
Fig 6.7(d) shows the injection conditions for 21 groups of 20 droplets (the numbers 1-21 in fig 6.7(d)-(g) indicate the respective droplet groups), each group having the same properties as in fig 6.7(b) except that the MFR of each group was adjusted so that the total MFR was distributed equally between all 21 groups. The droplets were injected along a series of 7 horizontal lines  $0.001\text{m}$  apart, with 3 groups along each line. In each group the largest droplet ( $L=700\mu\text{m}$ ) was injected nearest the spray axis and the smallest droplet ( $S=10\mu\text{m}$ ) furthest from the spray axis. Fig 6.7(e) shows the injection conditions for 21 groups of 20 droplets similar to fig 6.7(d) except that for the middle group of droplets in each horizontal line the radial distribution of droplet sizes was reversed.

Figs 6.8(h)-(i) show the normalised  $M_{ar/a}$  against radial distance for changes in the radial distribution of droplet size described in figs 6.7(d)-(e) respectively. The shape of

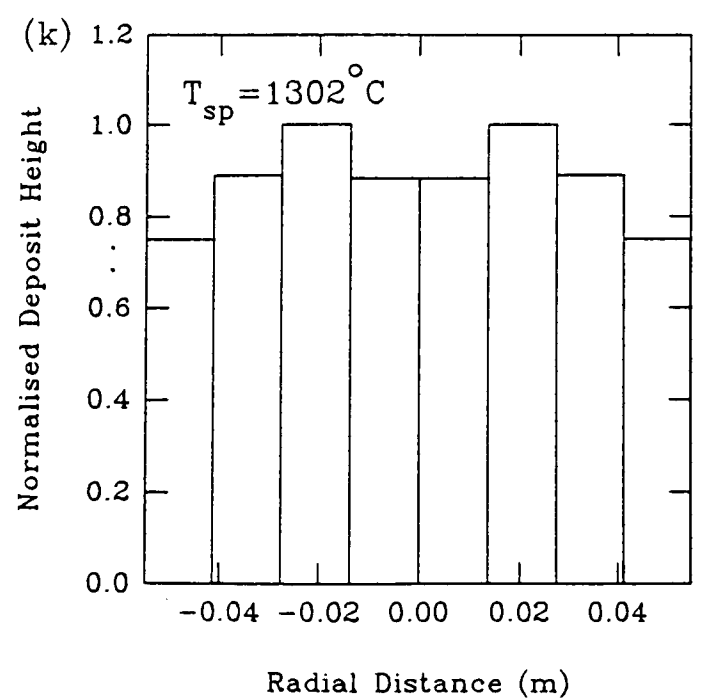
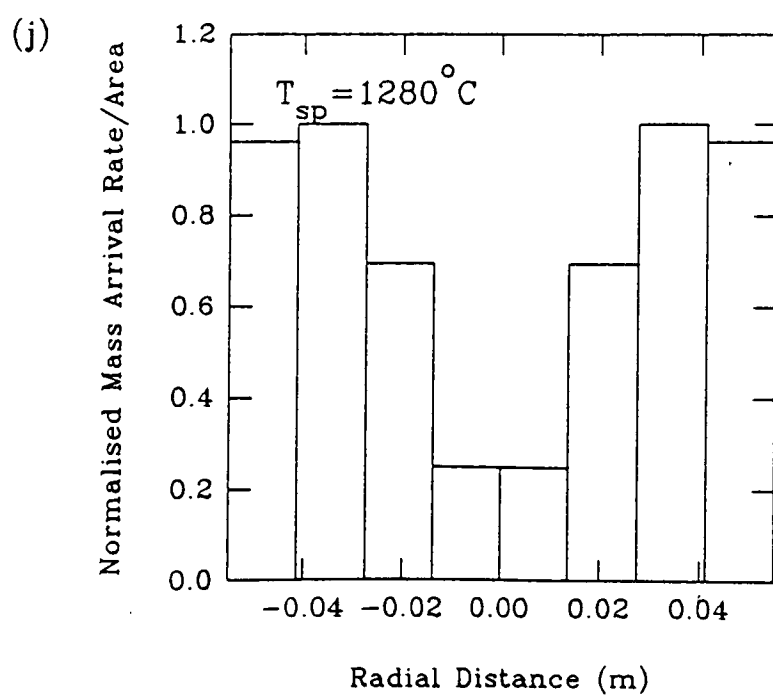
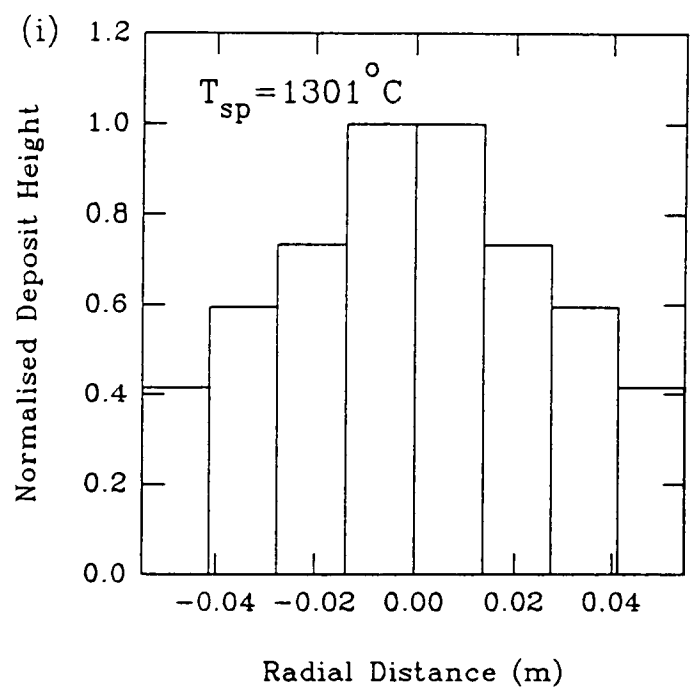
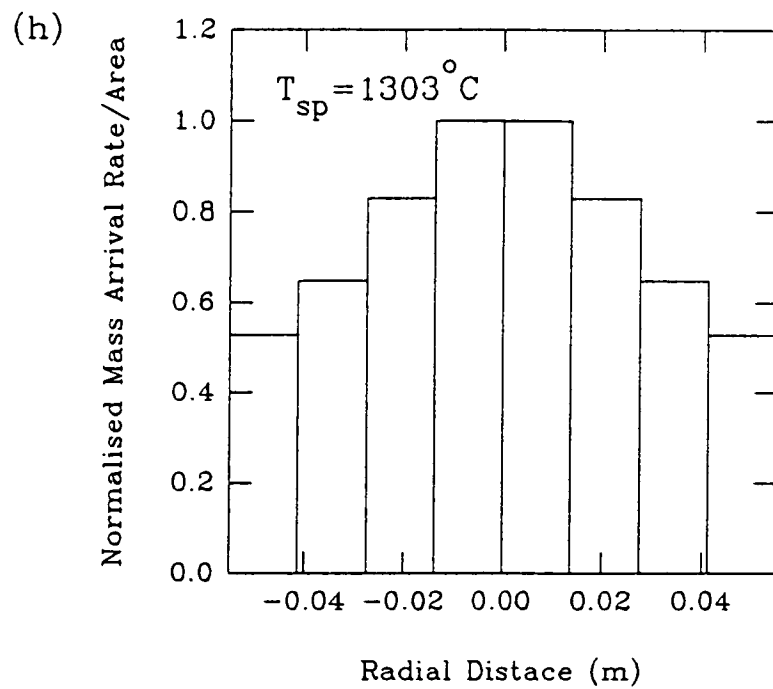
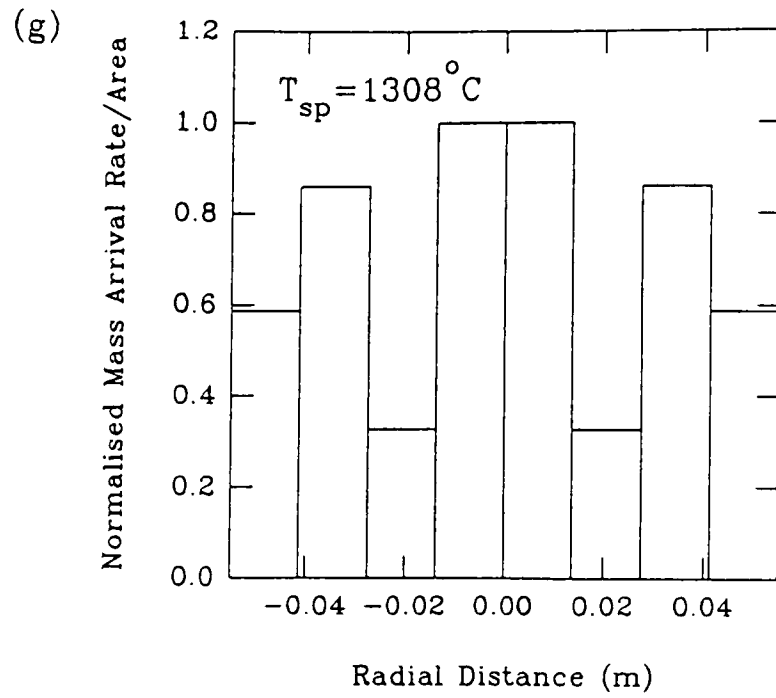


cont... Fig 6.7 Schematic diagrams indicating the droplet injection positions for 21 groups of 20 droplets with different radial distributions of droplet size: (d) in each group of droplets the largest droplet (700 $\mu$ m) was injected nearest the spray axis and (e) in the middle group of droplets along each horizontal line the smallest droplet (10 $\mu$ m) was injected nearest the spray axis.





cont... Fig 6.7 Schematic diagrams indicating the droplet injection positions for 21 groups of 20 droplets with different radial distributions of droplet size: (f) in each group of droplets the smallest droplet (10 $\mu$ m) was injected nearest the spray axis and (g) in the middle group of droplets along each horizontal line the largest droplet (700 $\mu$ m) was injected nearest the spray axis.



cont... Fig 6.8 Normalised mass arrival rate/area ( $M_{ar/a}$ ) against radial distance for: (g) droplet injection conditions shown in fig 6.8(b) except that the radial size distribution was reversed, and (h)-(k) droplet injection conditions shown in figs 6.8(d)-(g) respectively.

the normalised  $M_{ar/a}$  against radial distance bar charts in figs 6.8(h)-(i) were not significantly different from each other or from figs 6.8(a)-(f) i.e. approximately a Gaussian distribution.  $T_{sp}$  decreased slightly from 1303°C to 1301°C for the droplet injection conditions in figs 6.7(d) and (e) respectively, and in both cases  $T_{sp}$  was  $\approx 10^\circ\text{C}$  lower than in figs 6.8(a)-(f) for one group of injected droplets.

Fig 6.7(f) shows the injection conditions for 21 groups of 20 droplets, each group having the same properties as in fig 6.7(b) except that the MFR of each group was adjusted so that the total MFR was distributed equally between all 21 groups similar to fig 6.7(d). Once again, the droplets were injected along a series of 7 identical horizontal lines 0.001m apart, with 3 groups along each line. However, in this case, in each group the smallest droplet ( $S=10\mu\text{m}$ ) was injected nearest the spray axis and the largest droplet ( $L=700\mu\text{m}$ ) furthest from the spray axis. Fig 6.7(g) shows the injection conditions for 21 groups of 20 droplets similar to fig 6.7(f) except that for the middle group of droplets in each horizontal line the radial distribution of droplet sizes was reversed.

Figs 6.8(j)-(k) show the normalised  $M_{ar/a}$  against radial distance for changes in the radial distribution of droplet size described in figs 6.7(f)-(g) respectively. The shape of the normalised  $M_{ar/a}$  against radial distance bar charts in figs 6.8(j)-(k) were somewhat different from each other with more mass arriving further from the spray axis in fig 6.8(j), although fig 6.8(k) was more similar to figs 6.8(a)-(f) and (h)-(i) i.e. approximately a Gaussian distribution.  $T_{sp}$  increased from 1280°C to 1302°C for the droplet injection conditions in figs 6.7(f)-(g) respectively, again this was lower than in figs 6.8(a)-(f) for one group of injected droplets. Although  $T_{sp}$  for the droplet injection conditions in fig 6.7(g) (1302°C) was similar to  $T_{sp}$  for the droplet injection conditions in figs 6.7(d)-(e)

(1303°C and 1301°C respectively).

### **6.3.6 Summary**

In conclusion, two indicators of the model results,  $T_{sp}$  and the shape of the normalised  $M_{ar/a}$  against radial distance bar chart:

- (i) were not dependent on the number of droplets injected in the range 20-500.
- (ii) were not dependent on the starting axial injection position in the range 0.024m-0.034m from the top of the chamber.
- (iii) were not dependent on the initial (I1) gas phase turbulent intensity in the range 10-15%.
- (iv) were not dependent on whether the chamber wall (W1) boundary was either at a constant temperature of 80°C or zero heat flux (adiabatic system).
- (v) were strongly dependent on the radial distribution of particle sizes when injected as a single group along a horizontal line.  $T_{sp}$  decreased from 1314°C to 1308°C and more mass arrived further from the spray axis when the radial distribution of droplet sizes was reversed so that the largest droplet (700µm) was injected furthest from the spray axis and the smallest droplet (10µm) injected nearest the spray axis.
- (vi) were weakly dependent on the radial distribution of droplet sizes when droplets were injected along a series of 7 identical horizontal lines 0.001m apart, with 3 groups of 20 droplets along each line as described in figs 6.7(d)-(g).  $T_{sp}$  was approximately constant at 1302°C and the shape of the normalised  $M_{ar/a}$  against radial distance bar chart was approximately Gaussian.

Because there is some evidence to suggest smaller droplets segregate to the edges



of an atomised spray (Grant 1991, Bewlay and Cantor 1990), and because the approximate Gaussian shape of the normalised  $M_{ar/a}$  against radial distance bar chart was qualitatively a better prediction of observed deposit shapes; the droplet injection conditions described in fig 6.7(d) (i.e. droplets injected along a series of 7 identical horizontal lines 0.001m apart, with 3 groups along each line and in each group the largest droplet (700 $\mu$ m) was injected nearest the spray axis and the smallest droplet (10 $\mu$ m) furthest from the spray axis) were judged to be the best available approximation to the real atomisation process and to give the best consistency with experimental observations.

## **6.4 Model Results**

### **6.4.1 Single Phase Gas Flow Field**

The single phase flow of gas in the atomiser and spray chamber was solved for two different inlet gas pressures of 0.503MPa and 0.413MPa. Fig 6.9 shows the contours of Mach number (Ma) in the atomisation region for inlet gas pressures of: (a) 0.503MPa and (b) 0.413MPa, for  $Ma > 1.0$  the contours are filled in white. Fig 6.9 shows that as the gas exited into the spray chamber from the atomiser the gas velocities were supersonic ( $Ma > 1.0$ ) for a short distance and then rapidly reduce to subsonic velocities nearer the droplet injection region. The supersonic velocities were higher and the region of supersonic flow greater at an inlet gas pressure of 0.503MPa. Fig 6.10 shows the corresponding gas velocity vectors in the atomisation region for inlet gas pressures of: (a) 0.503MPa and (b) 0.413MPa. Fig 6.10 shows that at both gas pressures there was: (i) a region of low, negative velocities just below the melt delivery nozzle (recirculation); (ii) a rapid decrease in axial gas velocity as the radial distance increased; and (iii) a decrease

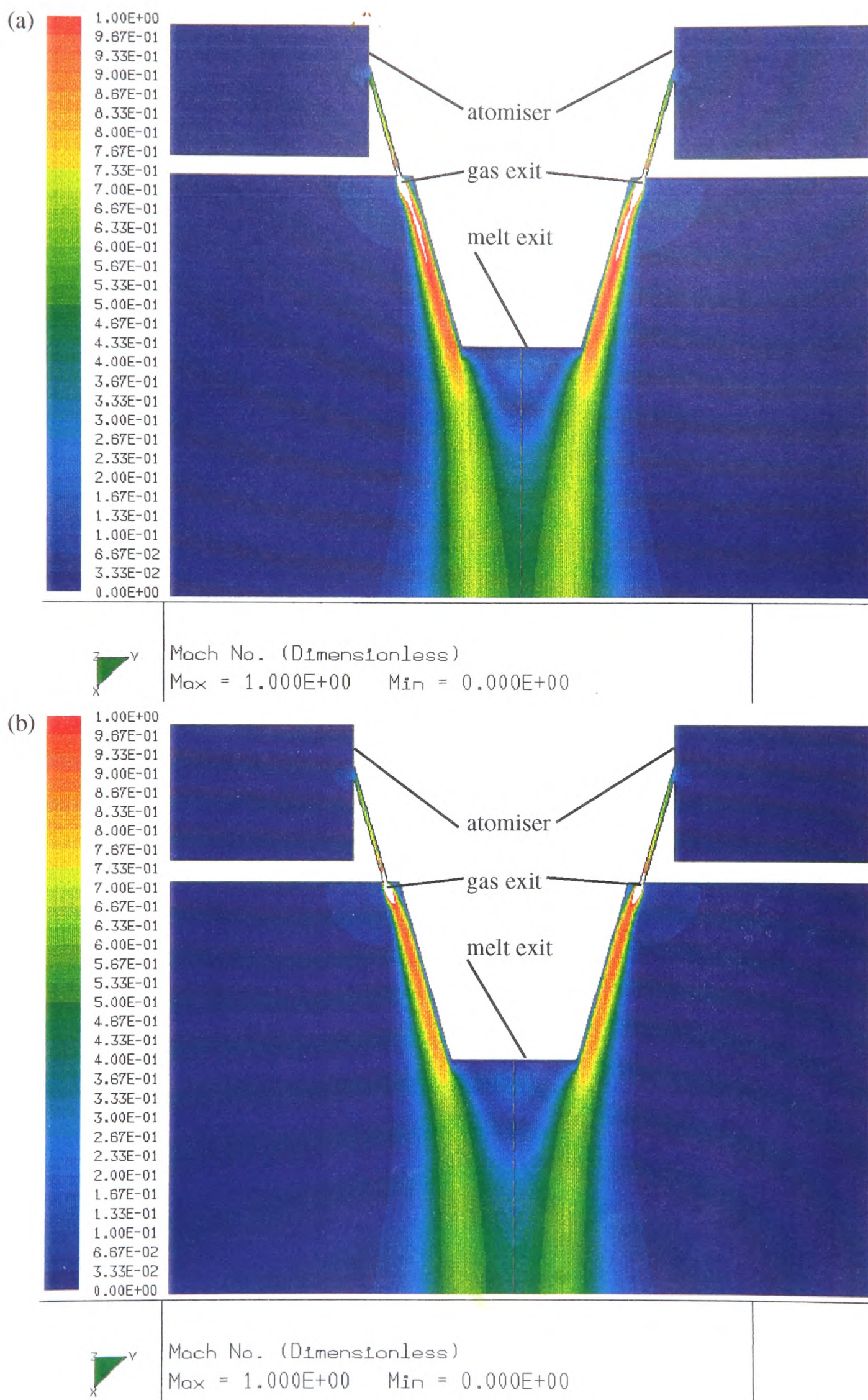


Fig 6.9 Single phase solution showing the contours of mach number between 0 and 1.0 in the atomisation region for: (a) an inlet gas pressure of 0.503MPa and (b) an inlet gas pressure of 0.413MPa.



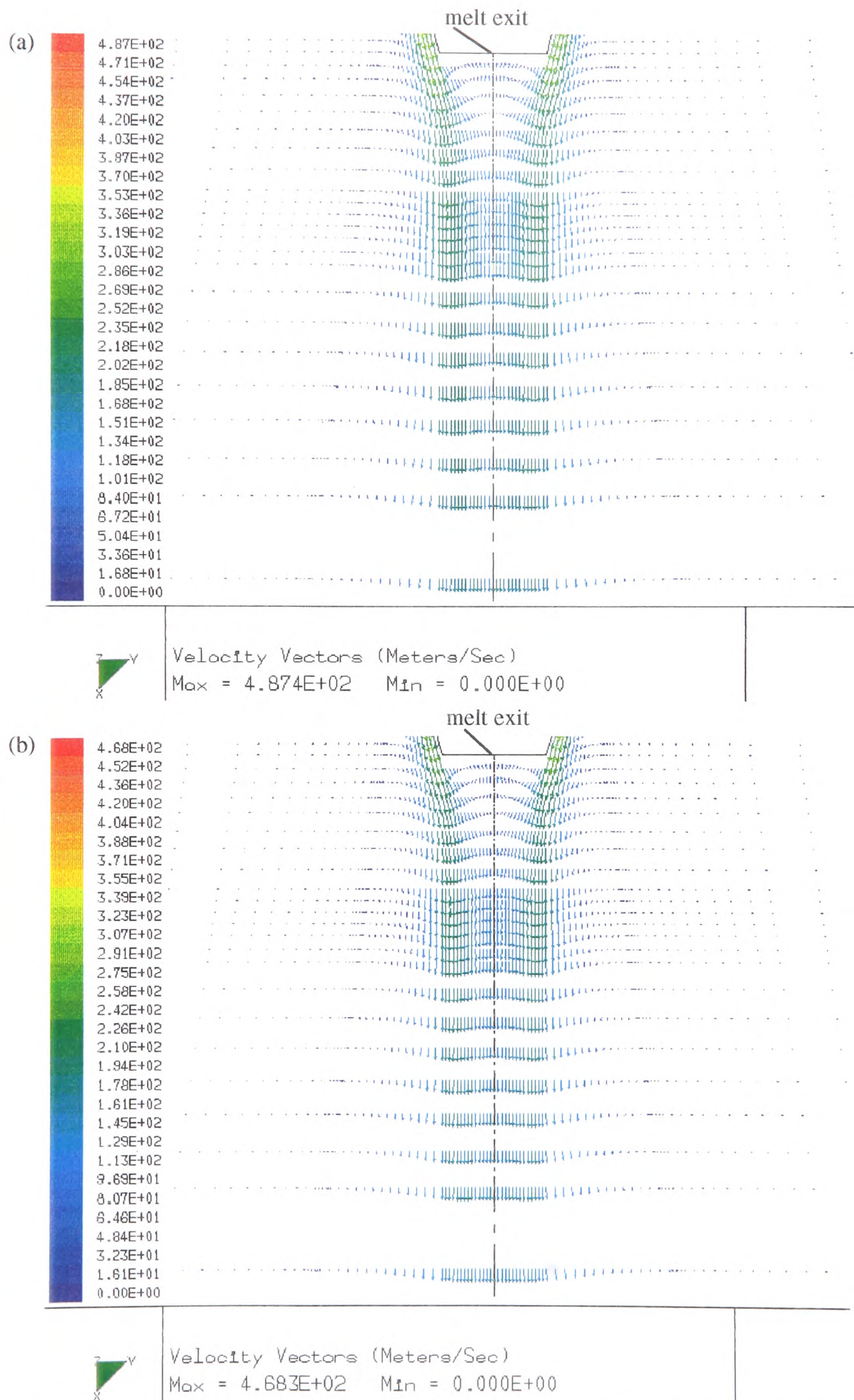


Fig 6.10 Single phase solution showing the gas velocity vectors in the atomisation region for: (a) an inlet gas pressure of 0.503MPa and (b) an inlet gas pressure of 0.413MPa.

in velocity as the axial distance increased beyond the melt exit. Overall, the gas velocities were  $\approx 20\text{ms}^{-1}$  lower in the atomisation region as the inlet gas pressure was decreased from 0.503MPa to 0.413MPa. Fig 6.11 shows the axial gas velocity against axial distance at radial distances of 0mm, 2mm, 4mm and 5.5mm from the spray axis for a gas pressure of: (a) 0.503MPa and (b) 0.413MPa. Figs 6.11(a)-(b) show that the maximum axial gas velocity increased as the radial distance increased from 0-5.5mm. For example, in fig 6.11(a) the maximum axial gas velocities were  $180\text{ms}^{-1}$  and  $206\text{ms}^{-1}$  at radial distances of 0mm and 5.5mm respectively. After reaching the point of maximum velocity, the axial gas velocities decreased approximately exponentially with axial distance and the axial position of maximum axial gas velocity decreased as the radial distance increased. The axial gas velocity then decreased quite rapidly  $\approx 5\text{cm}$  from the substrate.

Fig 6.12 shows the axial gas velocity against radial distance at axial distances of 2.5cm, 3.1cm, 4.2cm, 5.1cm, 7.2cm, 15.2cm and 25.4cm for a gas pressure of: (a) 0.503MPa and (b) 0.413MPa. Figs 6.12(a)-(b) show that as the axial distance increased the maximum axial gas velocity decreased and the radial position of maximum axial gas velocity moved closer to the spray axis. Both figs 6.12(a) and (b) show that for axial distances less than  $\approx 5\text{cm}$  there was a lower velocity region near the spray axis, the velocity increased up to a radial distance of  $\approx 5\text{mm}$  and then decreased exponentially at larger radial distances. For example, in fig 6.12(a) the maximum axial gas velocity decreased from  $225\text{ms}^{-1}$  at a radial distance of 5.1mm from the spray axis to  $138\text{ms}^{-1}$  at the spray axis as the axial distance increased from 2.5cm to 15.2cm. Once again, in general the axial gas velocities were  $\approx 10\text{-}20\text{ms}^{-1}$  lower as the gas pressure decreased from 0.503MPa to 0.413MPa. For example, in fig 6.12(b) the maximum axial gas velocity



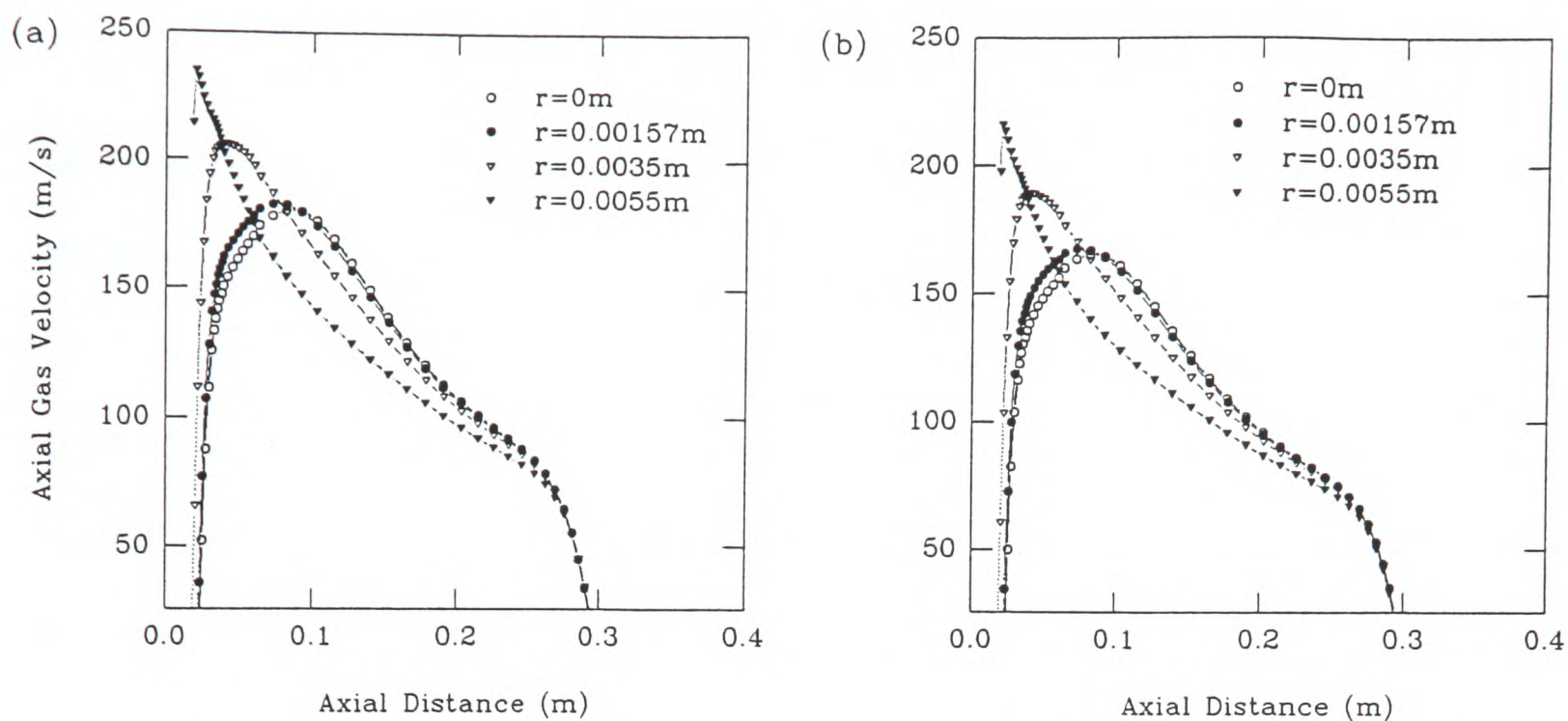


Fig 6.11 Axial gas velocity against axial distance at radial distances of 0m, 1.6mm, 3.5mm and 5.5mm for a gas pressure of (a) 0.503MPa and (b) 0.413MPa.

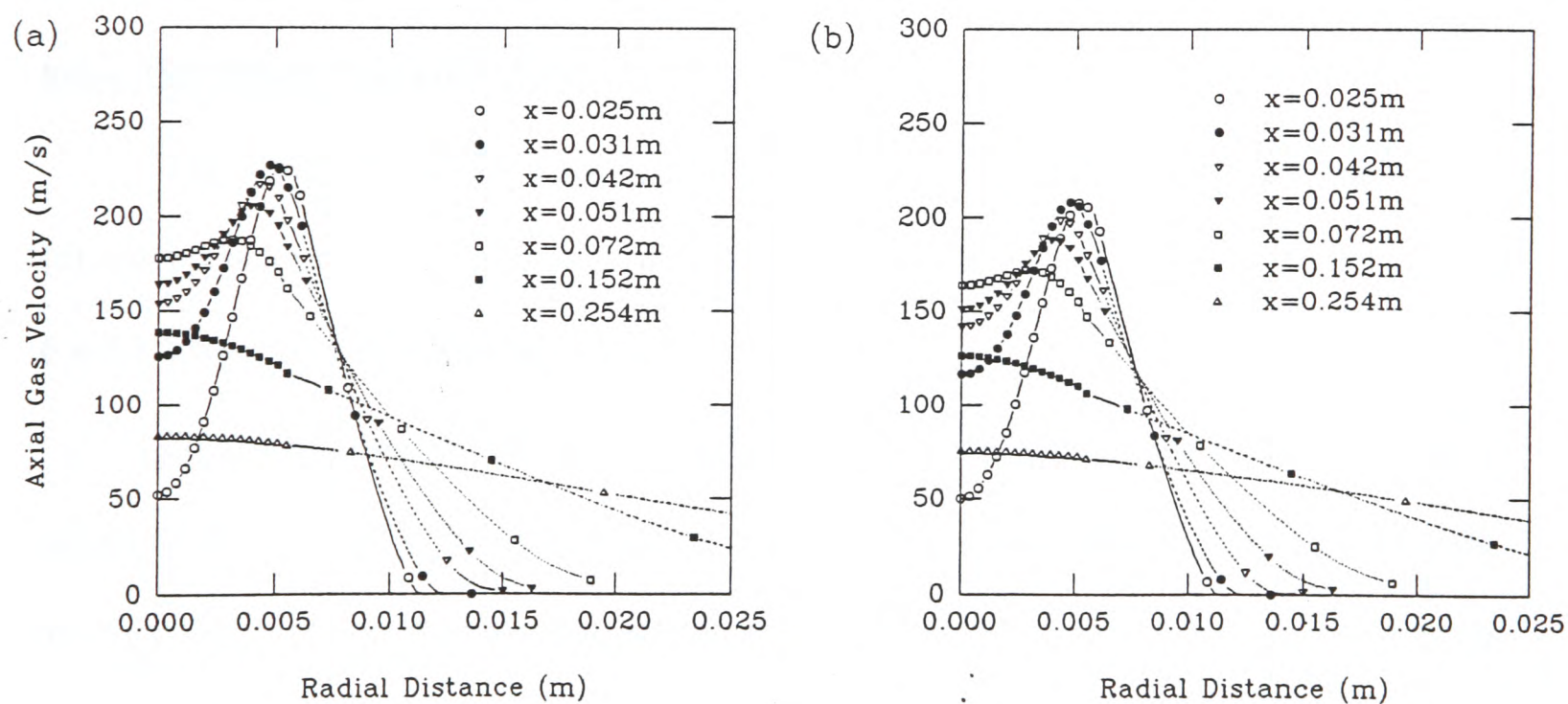


Fig 6.12 Axial gas velocity against radial distance at axial distances of 2.5cm, 3.1cm, 4.2cm, 5.1cm, 7.2cm, 15.2cm and 25.4cm for a gas pressure of (a) 0.503MPa and (b) 0.413MPa.

decreased from  $208\text{ms}^{-1}$  at a radial distance of 5.1mm from the spray axis to  $126\text{ms}^{-1}$  at the spray axis as the axial distance increased from 2.5cm to 15.2cm.

Experimental verification of the above gas velocities was attempted using PDA (phase Doppler anemometry). However, problems with the alignment of a complicated optical system and lack of time prevented any accurate measurements being made. However, Bewlay and Cantor (1989) showed good agreement between measured single phase  $\text{N}_2$  gas velocities using LDA (laser Doppler anemometry) and pitot tubes for an annular  $\text{N}_2$  gas atomiser at gas flow rates of 2.0l/s and 2.7l/s and spray distances of 0.18m and 0.36m with calculated  $\text{N}_2$  gas velocities from FLUENT.

#### **6.4.2 Two Phase Gas Flow Field**

The effect of droplets injected into the gas flow field is discussed as a function of:

(a) gas pressure and (b) melt flow rate.

##### **6.4.2.1 Effect of Gas Pressure**

Fig 6.13 shows the gas velocity vectors in the atomisation region from two phase solutions for: (a) run 30 at a gas pressure of 0.503MPa, spray distance of 0.3m and MFR of  $39\text{gs}^{-1}$  and (b) run 28 at a gas pressure of 0.413MPa, spray distance of 0.3m and MFR of  $44\text{gs}^{-1}$ . Comparing figs 6.13(a) and (b) with figs 6.10(a) and (b) respectively: (i) there was a sharp decrease in velocity as the gas impacted the droplets; and (ii) the gas flow was deflected radially as the gas impacted the droplets. Fig 6.14 shows the axial gas velocity against axial distance at radial distances of 0m, 2mm, 4mm and 5.5mm from the spray axis for: (a) run 30 at a gas pressure of 0.503MPa, spray distance of 0.3m and MFR of  $39\text{gs}^{-1}$  and (b) run 28 at a gas pressure of 0.413MPa, spray distance of 0.3m and MFR



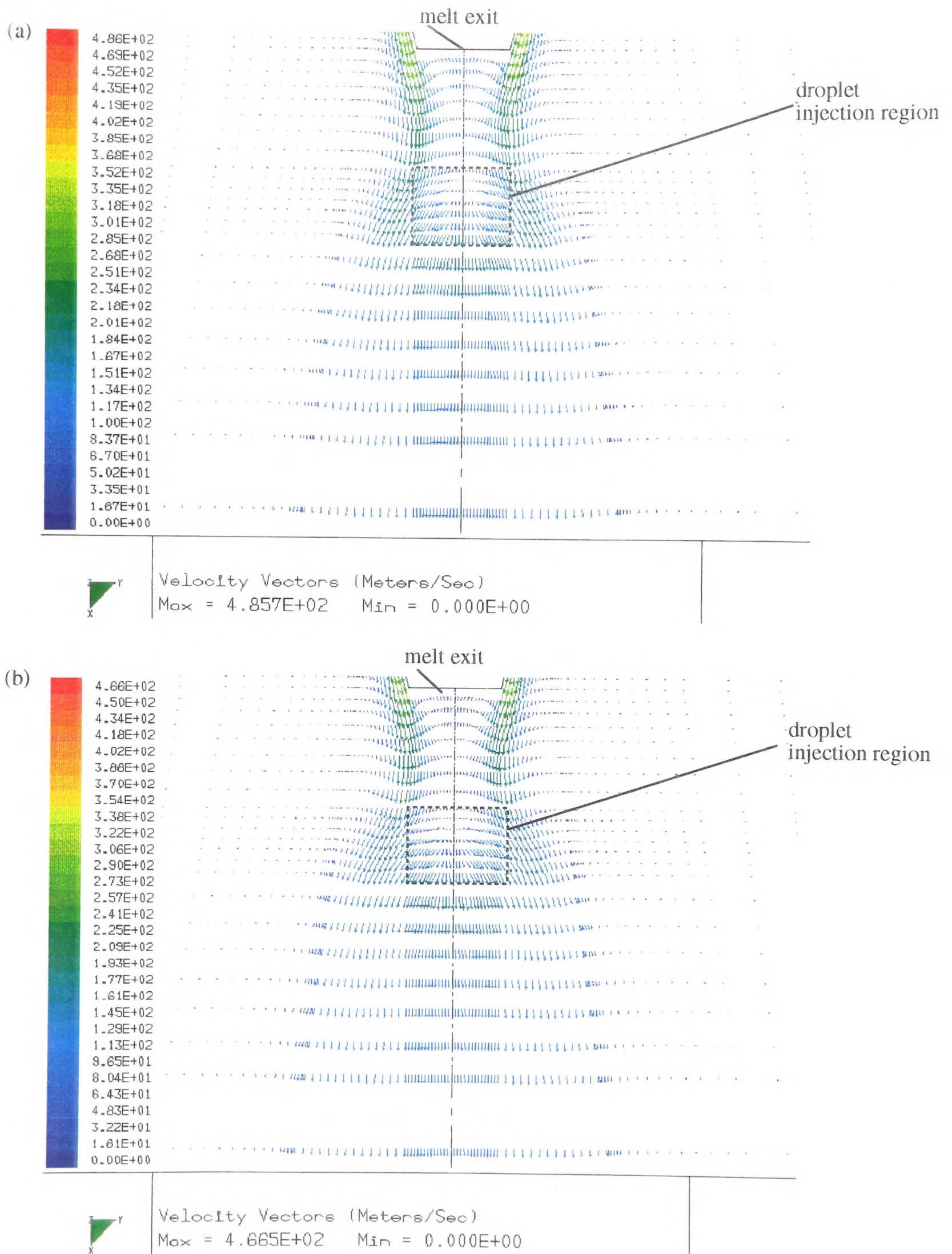


Fig 6.13 Two phase solution showing the gas velocity vectors in the atomisation region for: (a) run 30 at a gas pressure of 503MPa, spray distance of 0.3m and MFR of 39g/s and (b) run 28 at a gas pressure of 0.413MPa, spray distance of 0.3m and MFR of 44g/s

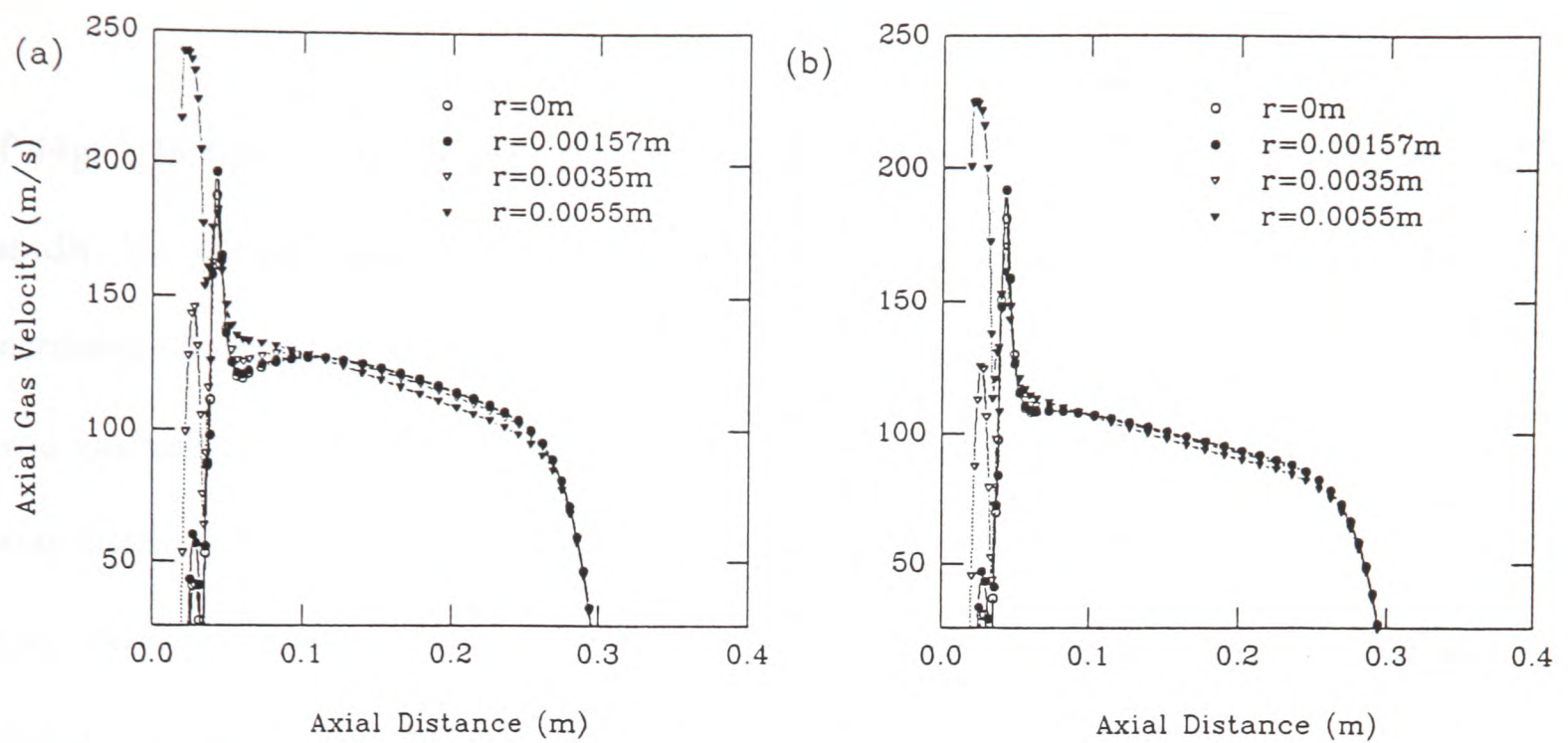


Fig 6.14 Axial gas velocity against axial distance at radial distances of 0m, 1.6mm, 3.5mm and 5.5mm for: (a) run 30 at a gas pressure of 0.503MPa, spray distance of 0.3m and MFR of  $39\text{gs}^{-1}$  and (b) run 28 at a gas pressure of 0.413MPa, spray distance of 0.3m and MFR of  $44\text{gs}^{-1}$ .

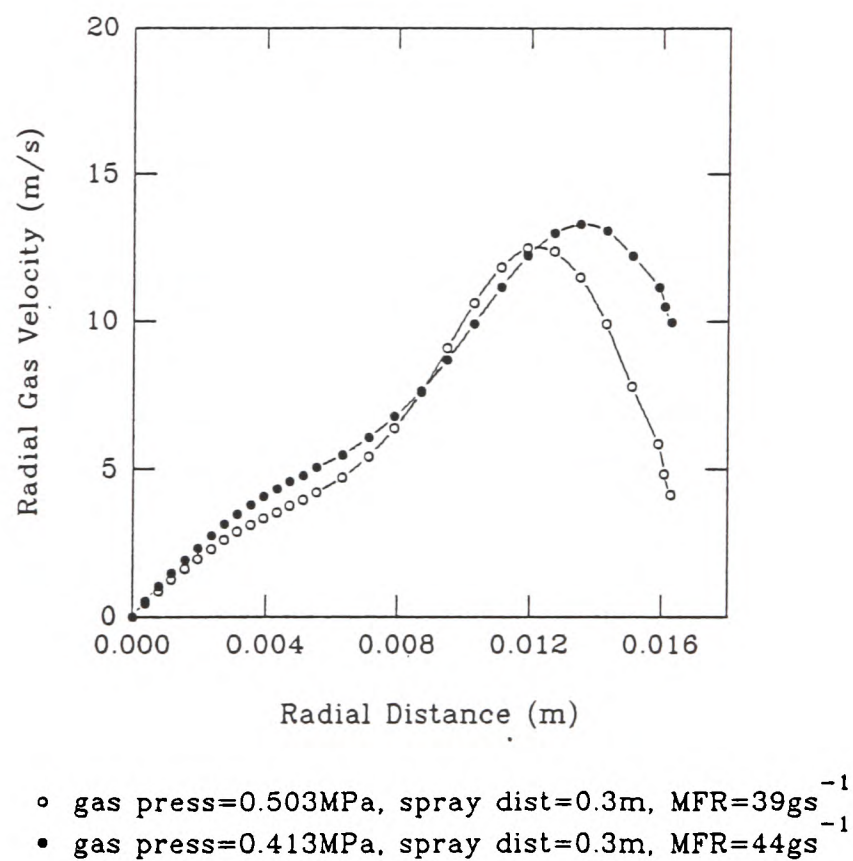


Fig 6.15 Radial gas velocity against radial distance at an axial distance of 5.1cm for: (a) run 30 at a gas pressure of 0.503MPa, spray distance of 0.3m and MFR of  $39\text{gs}^{-1}$  and (b) run 28 at a gas pressure of 0.413MPa, spray distance of 0.3m and MFR of  $44\text{gs}^{-1}$ .



of  $44\text{gs}^{-1}$ . In figs 6.14(a) and (b) for all radial distances the axial gas velocity decreased rapidly by  $\approx 60\text{ms}^{-1}$  and  $\approx 70\text{ms}^{-1}$  respectively as it impacted the droplets and then decreased relatively slowly with increasing axial distance. For example in fig 6.14(a) the axial gas velocity decreased from  $127\text{ms}^{-1}$  at an axial distance of 10cm to  $114\text{ms}^{-1}$  at an axial distance of 20cm and in fig 6.14(b) the axial gas velocity decreased from  $107\text{ms}^{-1}$  at an axial distance of 10cm to  $94\text{ms}^{-1}$  at an axial distance of 20cm i.e. the axial gas velocity decreased by  $\approx 20\text{ms}^{-1}$  as the gas pressure decreased from 0.503MPa to 0.413MPa.

Fig 6.15 shows the radial gas velocity against radial distance at an axial distance of 5.1cm for: (a) run 30 at a gas pressure of 0.503MPa, spray distance of 0.3m and MFR of  $39\text{gs}^{-1}$  and (b) run 28 at a gas pressure of 0.413MPa, spray distance of 0.3m and MFR of  $44\text{gs}^{-1}$ . Fig 6.15 shows that there was a slightly greater increase in radial gas velocity with increasing radial distance as the gas pressure decreased from 0.503MPa to 0.413MPa i.e. there was slightly greater divergence of the gas flow field at lower gas pressures. For example, at a radial distance of 4mm the radial gas velocity increased from  $3.3\text{ms}^{-1}$  to  $4.1\text{ms}^{-1}$  as the gas pressure decreased from 0.503MPa to 0.413MPa. In summary, the axial gas velocity decreased and the divergence of the gas flow field increased as the inlet gas pressure was decreased.

#### 6.4.2.2 Effect of Melt Flow Rate

Fig 6.16 shows the gas velocity vectors in the atomisation region from two phase solutions for: (a) run 16 at a MFR of  $64\text{gs}^{-1}$ , gas pressure of 0.503MPa and spray distance of 0.3m and (b) run 30 at a MFR of  $39\text{gs}^{-1}$ , gas pressure of 0.503MPa and spray distance of 0.3m. Similarly to figs 6.13(a) and (b), figs 6.16(a) and (b) show that: (i) there was a sharp decrease in velocity as the gas impacted the droplets; and (ii) the gas flow was

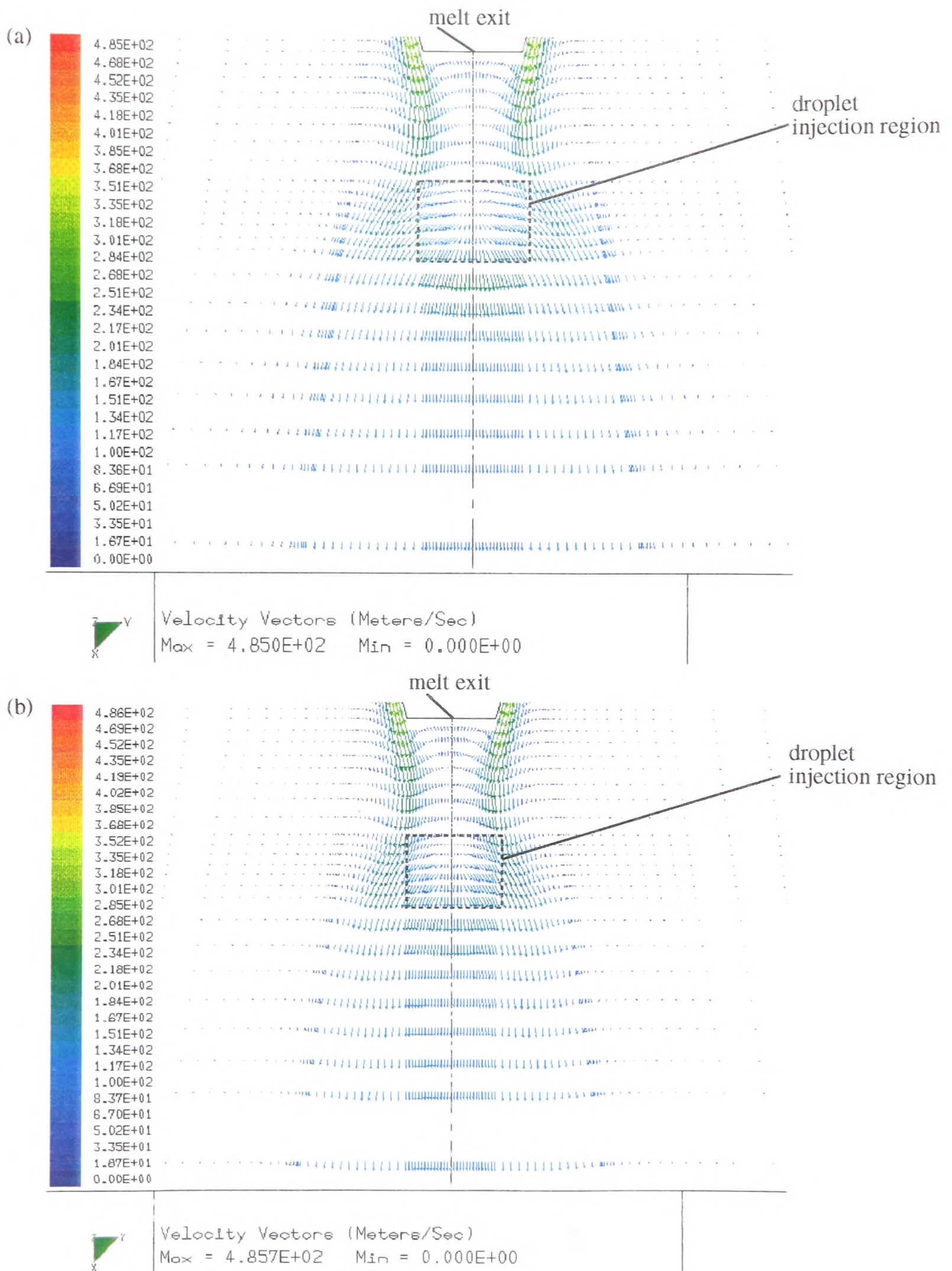


Fig 6.16 Two phase solution showing the gas velocity vectors in the atomisation region for: (a) run 16 at a MFR of 64g/s, gas pressure of 0.503MPa and spray distance of 0.3m and (b) run 30 at a MFR of 39g/s, gas pressure of 0.503MPa and spray distance of 0.3m.

deflected radially as the gas impacted the droplets. Fig 6.16 shows that there was a greater radial deflection of gas velocity as the MFR increased from  $39\text{gs}^{-1}$  in fig 6.16(b) to  $64\text{gs}^{-1}$  in fig 6.16(a). Fig 6.17 shows the axial gas velocity against axial distance at radial distances of 0m, 2mm, 4mm and 5.5mm from the spray axis for: (a) run 16 at a MFR of  $64\text{gs}^{-1}$ , gas pressure of 0.503MPa and spray distance of 0.3m and (b) run 30 at a MFR of  $39\text{gs}^{-1}$ , gas pressure of 0.503MPa and spray distance of 0.3m. In figs 6.17(a) and (b) for all radial distances the axial gas velocity decreased rapidly by  $\approx 123\text{ms}^{-1}$  and  $\approx 60\text{ms}^{-1}$  respectively as it impacted the droplets and then decreased relatively slowly with increasing axial distance. For example, in fig 6.17(a) the axial gas velocity decreased from  $101\text{ms}^{-1}$  at an axial distance of 10cm to  $87\text{ms}^{-1}$  at an axial distance of 20cm and in fig 6.17(b) the axial gas velocity decreased from  $127\text{ms}^{-1}$  at an axial distance of 10cm to  $114\text{ms}^{-1}$  at an axial distance of 20cm i.e. the axial gas velocity decreased by  $\approx 26\text{ms}^{-1}$  as the MFR increased from  $39\text{gs}^{-1}$  to  $64\text{gs}^{-1}$ .

Fig 6.18 shows the radial gas velocity against radial distance at an axial distance of 5.1cm for: (a) run 16 at a MFR of  $64\text{gs}^{-1}$ , gas pressure of 0.503MPa and spray distance of 0.3m and (b) run 30 at a MFR of  $39\text{gs}^{-1}$ , gas pressure of 0.503MPa and spray distance of 0.3m. Fig 6.18 shows that there was a greater increase in radial gas velocity with increasing radial distance as the MFR increased from  $39\text{gs}^{-1}$  to  $64\text{gs}^{-1}$  i.e. there was greater divergence of the gas flow field at higher MFR's. For example, at a radial distance of 4mm the radial gas velocity increased from  $3.3\text{ms}^{-1}$  to  $6.9\text{ms}^{-1}$  as the MFR increased from  $39\text{gs}^{-1}$  to  $64\text{gs}^{-1}$ . In summary, the axial gas velocity decreased and the divergence of the gas flow field increased as the MFR was increased.



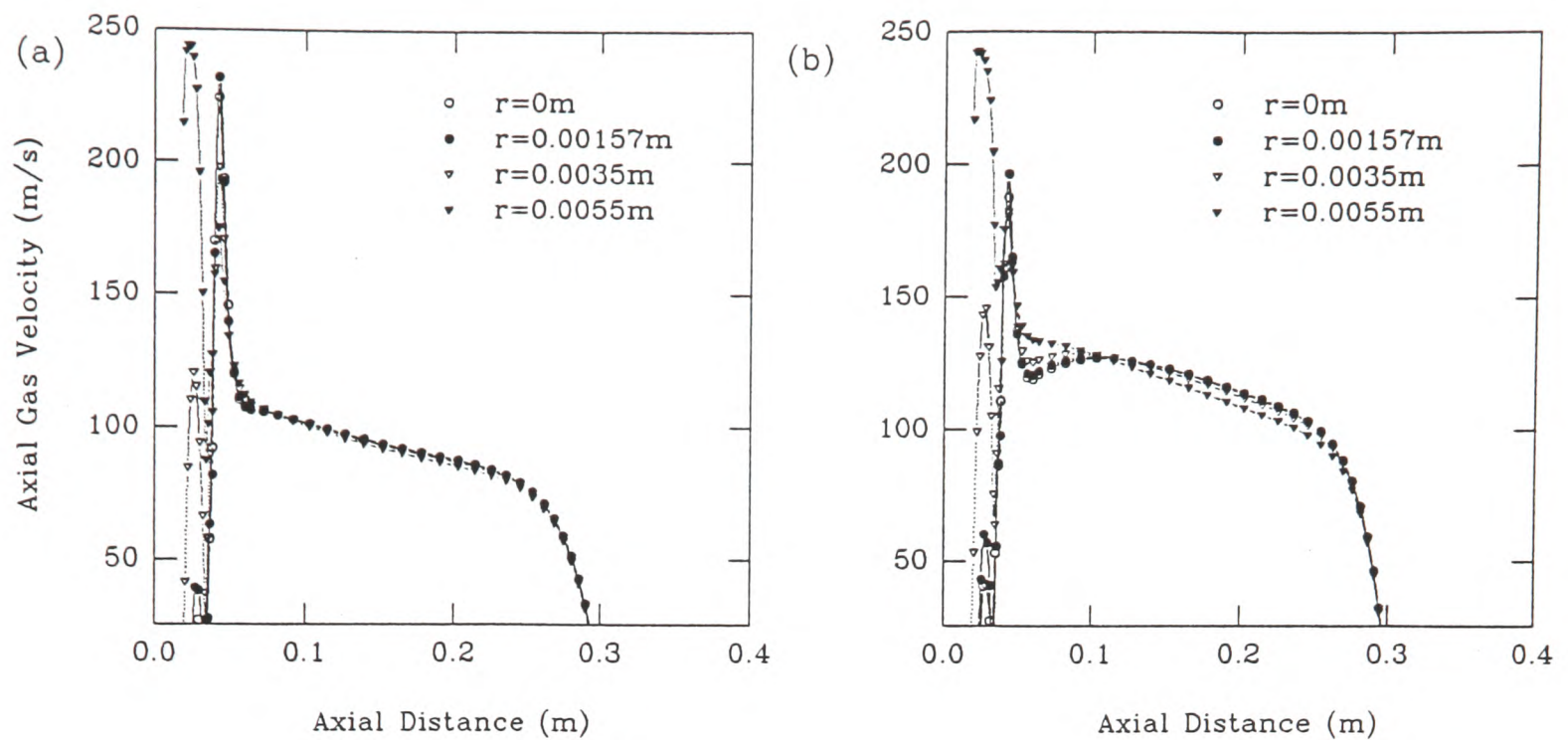


Fig 6.17 Axial gas velocity against axial distance at radial distances of 0m, 1.6mm, 3.5mm and 5.5mm for: (a) run 160 at a MFR of  $64\text{gs}^{-1}$ , gas pressure of 0.503MPa and spray distance of 0.3m and MFR of  $39\text{gs}^{-1}$  and (b) run 28 at a MFR of  $39\text{gs}^{-1}$ , gas pressure of 0.503MPa and spray distance of 0.3m.

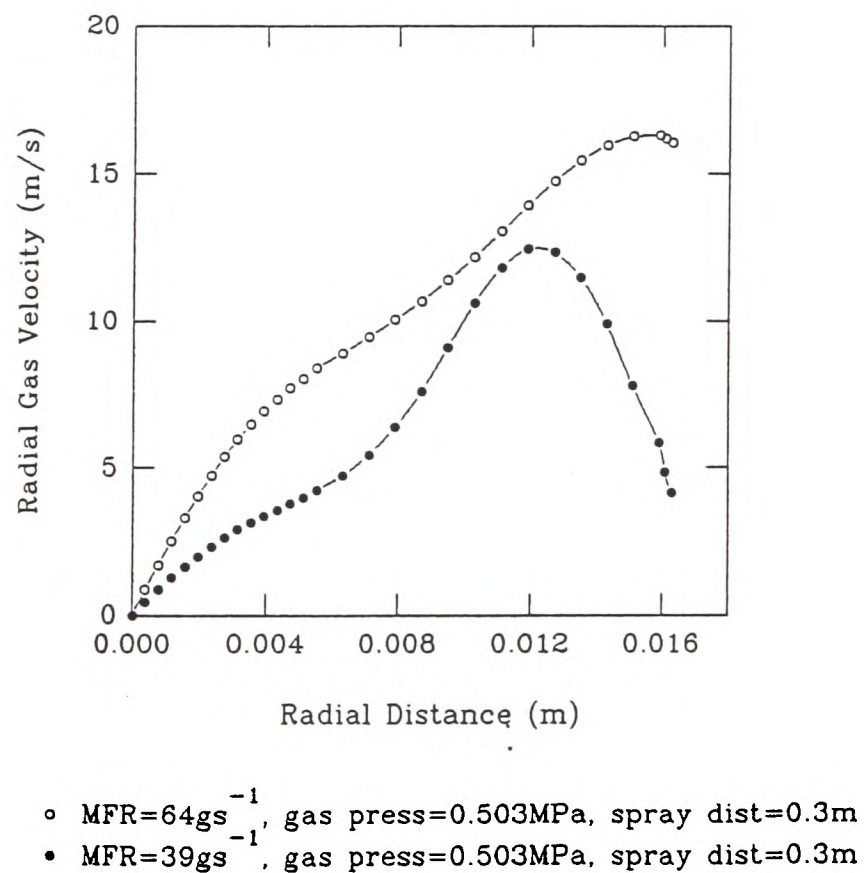


Fig 6.18 Radial gas velocity against radial distance at an axial distance of 5.1cm for: (a) run 160 at a MFR of  $64\text{gs}^{-1}$ , gas pressure of 0.503MPa and spray distance of 0.3m and MFR of  $39\text{gs}^{-1}$  and (b) run 28 at a MFR of  $39\text{gs}^{-1}$ , gas pressure of 0.503MPa and spray distance of 0.3m.



### **6.4.3 Spray Temperatures**

Table 6.5 is a summary of the model process parameters which are equivalent to the spray forming experimental process parameters shown in table 5.3, except here they are converted for use in FLUENT. Table 6.5 also shows  $T_{sp}$  temperatures calculated from Grant's post-processing program and the corresponding experimentally measured mean droplet size and range of the best fit Rosin-Rammler distribution (equation 3.4) to the overspray powder distribution. The gas pressures were measured in the flow meter, the MFR and spray distances were also equivalent to the respective measured MFR's and spray distances. The superheat was 1445°C in each case except for run 33 which had a lower superheat of 1403°C.

#### **6.4.3.1 Effect of Gas Pressure**

Tables 6.6(a)-(c) show the process parameters of 3 groups of runs taken from table 6.5 which show the effect of gas pressure in the range 0.413-0.503MPa on  $T_{sp}$  at spray distances and MFR's of: 0.2m and  $\approx 41\text{gs}^{-1}$ ; 0.25m and  $\approx 41\text{gs}^{-1}$ ; and 0.3m and  $\approx 41\text{gs}^{-1}$  respectively. For each table all process parameters were constant except gas pressure, although there was some variation in the MFR, and table 6.6(a) contains run 33 which had a lower superheat. Fig 6.19(a) shows that there was a decrease in  $T_{sp}$  as the gas pressure increased. (The dashed horizontal lines indicate the solidus ( $T_{sol}=1225^\circ\text{C}$ ) and liquidus ( $T_{liq}=1323^\circ\text{C}$ ) temperatures of UDIMET 720 respectively) For example at a spray distance and MFR of 0.25m and  $\approx 41\text{g/s}$  respectively  $T_{sp}$  decreased from 1286°C to 1269°C as the gas pressure increased from 0.413MPa to 0.503MPa.

#### **6.4.3.2 Effect of Spray Distance**

Tables 6.7(a)-(d) show the process parameters of 4 groups of runs taken from table

| Parameters                   | Run No. |       |       |       |       |       |       |       |       |  |  |
|------------------------------|---------|-------|-------|-------|-------|-------|-------|-------|-------|--|--|
|                              | 16      | 17    | 19    | 20    | 21    | 23    | 24    | 25    | 27    |  |  |
| Spray distance (m)           | 0.3     | 0.3   | 0.25  | 0.25  | 0.25  | 0.2   | 0.2   | 0.25  | 0.25  |  |  |
| Gas pressure (MPa)           | 0.503   | 0.053 | 0.503 | 0.503 | 0.503 | 0.503 | 0.413 | 0.413 | 0.413 |  |  |
| Melt flow rate (g/s)         | 64      | 59    | 101   | 99    | 59    | 59    | 50    | 42    | 43    |  |  |
| Droplet axial velocity (m/s) | 1.12    | 1.03  | 1.75  | 1.75  | 1.03  | 1.03  | 0.88  | 0.73  | 0.75  |  |  |
| Rosin-Rammler mean (um)      | 209     | 183   | 179   | 194   | 183   | 203   | 179   | 186   | 180   |  |  |
| Rosin-Rammler range          | 1.53    | 1.66  | 1.68  | 1.55  | 1.52  | 1.42  | 1.51  | 1.46  | 1.46  |  |  |
| Tspray (C)                   | 1299    | 1298  | 1303  | 1314  | 1302  | 1310  | 1304  | 1288  | 1286  |  |  |

| Parameters                   | Run No. |       |       |       |       |       |       |       |  |  |
|------------------------------|---------|-------|-------|-------|-------|-------|-------|-------|--|--|
|                              | 28      | 30    | 31    | 32    | 33**  | 34    | 35    | 37    |  |  |
| Spray distance (m)           | 0.3     | 0.3   | 0.25  | 0.2   | 0.2   | 0.2   | 0.2   | 0.25  |  |  |
| Gas pressure (MPa)           | 0.413   | 0.503 | 0.503 | 0.503 | 0.413 | 0.503 | 0.413 | 0.413 |  |  |
| Melt flow rate (g/s)         | 44      | 39    | 39    | 45    | 41    | 38    | 41    | 50    |  |  |
| Droplet axial velocity (m/s) | 0.77    | 0.68  | 0.68  | 0.79  | 0.72  | 0.66  | 0.72  | 0.88  |  |  |
| Rosin-Rammler mean (um)      | 178     | 171   | 158   | 157   | 173   | 151   | 175   | 187   |  |  |
| Rosin-Rammler range          | 1.54    | 1.45  | 1.44  | 1.44  | 1.42  | 1.35  | 1.46  | 1.5   |  |  |
| Tspray (C)                   | 1288    | 1269  | 1269  | 1288  | 1281  | 1276  | 1297  | 1299  |  |  |

Superheat=1445C except \*\* - superheat=1403C

Table 6.5 Summary of process parameters and equilibrated spray temperatures (Tspray) for runs 16-37.

(a)

| Parameters                   | Run No. |       |       |       |
|------------------------------|---------|-------|-------|-------|
|                              | 33**    | 32    | 35    | 34    |
| Spray distance (m)           | 0.2     | 0.2   | 0.2   | 0.2   |
| Gas pressure (MPa)           | 0.413   | 0.503 | 0.413 | 0.503 |
| Melt flow rate (g/s)         | 41      | 45    | 41    | 38    |
| Droplet axial velocity (m/s) | 0.72    | 0.79  | 0.72  | 0.66  |
| Rosin-Rammler mean (um)      | 173     | 157   | 175   | 151   |
| Rosin-Rammler range          | 1.42    | 1.44  | 1.46  | 1.35  |
| Tspray (C)                   | 1281    | 1288  | 1297  | 1276  |

(b)

| Parameters                   | Run No. |       |       |
|------------------------------|---------|-------|-------|
|                              | 25      | 27    | 31    |
| Spray distance (m)           | 0.25    | 0.25  | 0.25  |
| Gas pressure (MPa)           | 0.413   | 0.413 | 0.503 |
| Melt flow rate (g/s)         | 42      | 43    | 39    |
| Droplet axial velocity (m/s) | 0.73    | 0.75  | 0.68  |
| Rosin-Rammler mean (um)      | 186     | 180   | 158   |
| Rosin-Rammler range          | 1.46    | 1.46  | 1.44  |
| Tspray (C)                   | 1288    | 1286  | 1269  |

(c)

| Parameters                   | Run No. |       |
|------------------------------|---------|-------|
|                              | 28      | 30    |
| Spray distance (m)           | 0.3     | 0.3   |
| Gas pressure (MPa)           | 0.413   | 0.503 |
| Melt flow rate (g/s)         | 44      | 39    |
| Droplet axial velocity (m/s) | 0.77    | 0.68  |
| Rosin-Rammler mean (um)      | 178     | 171   |
| Rosin-Rammler range          | 1.54    | 1.45  |
| Tspray (C)                   | 1288    | 1269  |

Superheat=1445C except \*\* - superheat=1403C

Tables 6.6(a)-(c) Summary of process parameters and equilibrated spray temperatures (Tspray) for 3 groups of runs where gas pressure was the only independent variable.

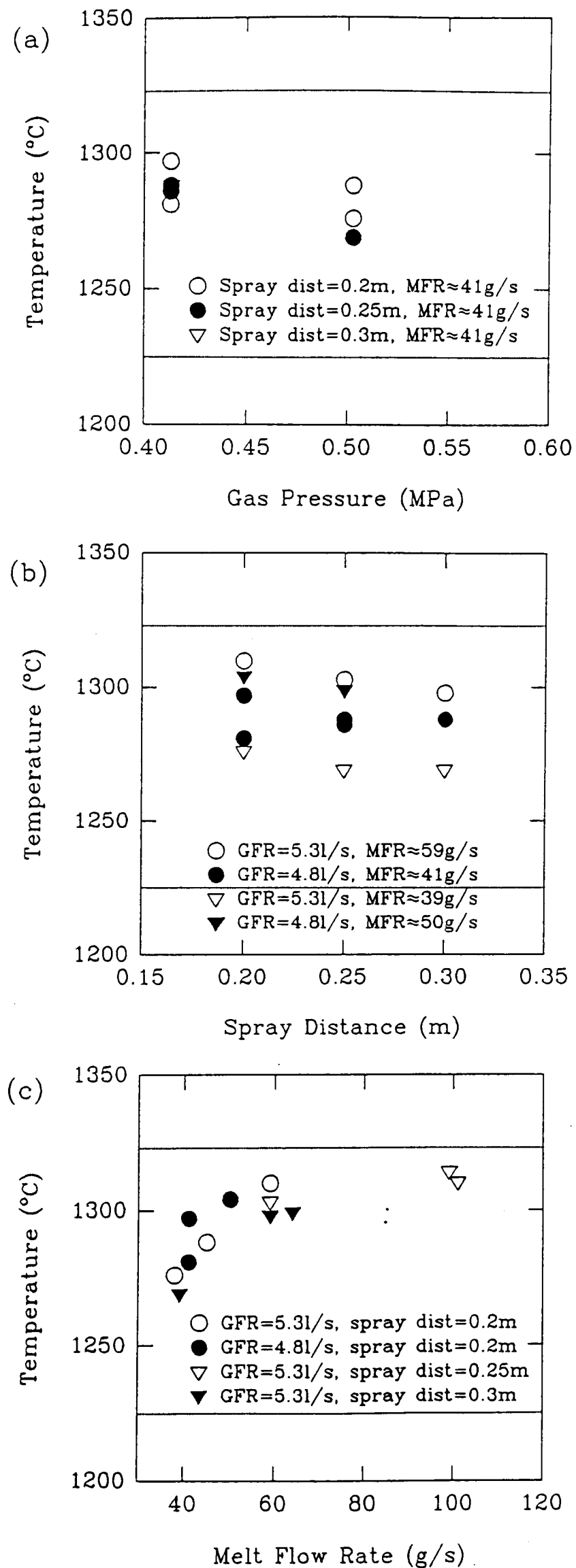


Fig 6.19 Equilibrated spray temperature ( $T_{sp}$ ) against: (a) gas pressure, (b) spray distance and (c) melt flow rate. The dashed horizontal lines indicate the liquidus and solidus temperatures.



(a)

| Parameters                   | Run No. |       |       |
|------------------------------|---------|-------|-------|
|                              | 17      | 21    | 23    |
| Spray distance (m)           | 0.3     | 0.25  | 0.2   |
| Gas pressure (MPa)           | 0.503   | 0.503 | 0.503 |
| Melt flow rate (g/s)         | 59      | 59    | 59    |
| Droplet axial velocity (m/s) | 1.03    | 1.03  | 1.03  |
| Rosin-Rammler mean (um)      | 183     | 183   | 203   |
| Rosin-Rammler range          | 1.66    | 1.52  | 1.42  |
| Tspray (C)                   | 1298    | 1303  | 1310  |

(b)

| Parameters                   | Run No. |       |       |       |       |
|------------------------------|---------|-------|-------|-------|-------|
|                              | 28      | 25    | 27    | 33**  | 35    |
| Spray distance (m)           | 0.3     | 0.25  | 0.25  | 0.2   | 0.2   |
| Gas pressure (MPa)           | 0.413   | 0.413 | 0.413 | 0.413 | 0.413 |
| Melt flow rate (g/s)         | 44      | 42    | 43    | 41    | 41    |
| Droplet axial velocity (m/s) | 0.77    | 0.73  | 0.75  | 0.72  | 0.72  |
| Rosin-Rammler mean (um)      | 178     | 203   | 180   | 173   | 175   |
| Rosin-Rammler range          | 1.54    | 1.42  | 1.46  | 1.42  | 1.46  |
| Tspray (C)                   | 1288    | 1288  | 1286  | 1281  | 1297  |

(c)

| Parameters                   | Run No. |       |       |
|------------------------------|---------|-------|-------|
|                              | 30      | 31    | 34    |
| Spray distance (m)           | 0.3     | 0.25  | 0.2   |
| Gas pressure (MPa)           | 0.503   | 0.503 | 0.503 |
| Melt flow rate (g/s)         | 39      | 39    | 38    |
| Droplet axial velocity (m/s) | 0.68    | 0.68  | 0.66  |
| Rosin-Rammler mean (um)      | 171     | 158   | 151   |
| Rosin-Rammler range          | 1.45    | 1.44  | 1.35  |
| Tspray (C)                   | 1269    | 1269  | 1276  |

(d)

| Parameters                   | Run No. |       |
|------------------------------|---------|-------|
|                              | 24      | 37    |
| Spray distance (m)           | 0.2     | 0.25  |
| Gas pressure (MPa)           | 0.413   | 0.413 |
| Melt flow rate (g/s)         | 50      | 50    |
| Droplet axial velocity (m/s) | 0.88    | 0.88  |
| Rosin-Rammler mean (um)      | 179     | 187   |
| Rosin-Rammler range          | 1.51    | 1.5   |
| Tspray (C)                   | 1304    | 1299  |

Superheat=1445C except \*\* - superheat=1403C

Tables 6.7(a)-(d) Summary of process parameters and equilibrated spray temperatures (Tspray) for 4 groups of runs where spray distance was the only independent variable.

(a)

| Parameters                   | Run No. |       |       |
|------------------------------|---------|-------|-------|
|                              | 23      | 32    | 34    |
| Spray distance (m)           | 0.2     | 0.2   | 0.2   |
| Gas pressure (MPa)           | 0.503   | 0.503 | 0.503 |
| Melt flow rate (g/s)         | 59      | 45    | 38    |
| Droplet axial velocity (m/s) | 1.03    | 0.79  | 0.66  |
| Rosin-Rammler mean (um)      | 203     | 157   | 151   |
| Rosin-Rammler range          | 1.42    | 1.44  | 1.35  |
| Tspray (C)                   | 1310    | 1288  | 1276  |

(b)

| Parameters                   | Run No. |       |       |
|------------------------------|---------|-------|-------|
|                              | 24      | 33**  | 35    |
| Spray distance (m)           | 0.2     | 0.2   | 0.2   |
| Gas pressure (MPa)           | 0.413   | 0.413 | 0.413 |
| Melt flow rate (g/s)         | 50      | 41    | 41    |
| Droplet axial velocity (m/s) | 0.88    | 0.72  | 0.72  |
| Rosin-Rammler mean (um)      | 179     | 173   | 175   |
| Rosin-Rammler range          | 1.51    | 1.42  | 1.46  |
| Tspray (C)                   | 1304    | 1281  | 1297  |

(c)

| Parameters                   | Run No. |       |       |       |
|------------------------------|---------|-------|-------|-------|
|                              | 19      | 20    | 21    | 31    |
| Spray distance (m)           | 0.25    | 0.25  | 0.25  | 0.25  |
| Gas pressure (MPa)           | 0.503   | 0.503 | 0.503 | 0.503 |
| Melt flow rate (g/s)         | 101     | 99    | 59    | 39    |
| Droplet axial velocity (m/s) | 1.75    | 1.75  | 1.03  | 0.68  |
| Rosin-Rammler mean (um)      | 179     | 194   | 183   | 158   |
| Rosin-Rammler range          | 1.68    | 1.55  | 1.52  | 1.44  |
| Tspray (C)                   | 1303    | 1314  | 1303  | 1269  |

(d)

| Parameters                   | Run No. |       |       |
|------------------------------|---------|-------|-------|
|                              | 16      | 17    | 30    |
| Spray distance (m)           | 0.3     | 0.3   | 0.3   |
| Gas pressure (MPa)           | 0.503   | 0.053 | 0.503 |
| Melt flow rate (g/s)         | 64      | 59    | 39    |
| Droplet axial velocity (m/s) | 1.12    | 1.03  | 0.68  |
| Rosin-Rammler mean (um)      | 209     | 183   | 171   |
| Rosin-Rammler range          | 1.53    | 1.66  | 1.45  |
| Tspray (C)                   | 1299    | 1298  | 1269  |

Superheat=1445C except \*\* - superheat=1403C

Tables 6.8(a)-(d) Summary of process parameters and equilibrated spray temperatures (Tspray) for 4 groups of runs where melt flow rate was the only independent variable.

6.5 which show the effect of spray distance in the range 0.2-0.3m on  $T_{sp}$  at gas pressures and MFR's of: 0.503MPa and  $\approx 59\text{gs}^{-1}$ ; 0.413MPa and  $\approx 41\text{gs}^{-1}$ ; 0.503MPa and  $\approx 39\text{gs}^{-1}$ ; and 0.413MPa and  $\approx 50\text{gs}^{-1}$  respectively. For each table all process parameters were constant except spray distance, although once again there was a slight variation in the MFR and table 6.7(b) contains run 33 which had a lower superheat. Fig 6.19(b) shows that there was a decrease in  $T_{sp}$  as the spray distance increased. For example at a MFR and gas pressure of  $39\text{gs}^{-1}$  and 0.503MPa respectively  $T_{sp}$  decreased from  $1276^{\circ}\text{C}$  to  $1269^{\circ}\text{C}$  as the spray distance increased from 0.2m to 0.3m.

#### 6.4.3.3 Effect of Melt Flow Rate

Tables 6.8(a)-(d) show the process parameters of 4 groups of runs taken from table 6.5 which show the effect of MFR in the range  $38\text{-}101\text{gs}^{-1}$  on  $T_{sp}$  at spray distances and gas pressures of: 0.2m and 0.503MPa; 0.2m and 0.413MPa; 0.25m and 0.503MPa; and 0.3m and 0.503MPa respectively. For each table all process parameters were constant except MFR although table 6.7(b) contains run 33 which had a lower superheat. Fig 6.19(c) shows that there was a decrease in  $T_{sp}$  as the MFR increased. For example, at a spray distance and gas pressure of 0.25m and 0.503MPa  $T_{sp}$  increased from  $1269^{\circ}\text{C}$  to  $1314^{\circ}\text{C}$  as the MFR increased from  $39\text{gs}^{-1}$  to  $99\text{gs}^{-1}$ .

#### 6.4.4 Mean (Time Averaged) Droplet Behaviour

From the best fit Rosin-Rammler distributions to the measured overspray powder distributions shown in fig 5.39, the range of droplet sizes  $83\mu\text{m}$ - $228\mu\text{m}$  covers  $\approx 75\%$  by mass of the droplet distributions. Therefore, predicted trajectories and temperatures of  $228\mu\text{m}$ ,  $155\mu\text{m}$  and  $83\mu\text{m}$  droplets are taken to be representative of droplet dynamic and

thermal behaviour. The injection positions of the three droplets considered is shown in fig 6.20. The droplets are injected in the middle of the atomisation region and as described in fig 6.7(d) the larger droplets were injected nearer the spray axis. Similarly to the equilibrated spray behaviour, individual droplet temperatures, axial velocities and radial distances (divergence) against axial distance were considered as a function of gas pressure, spray distance and MFR.

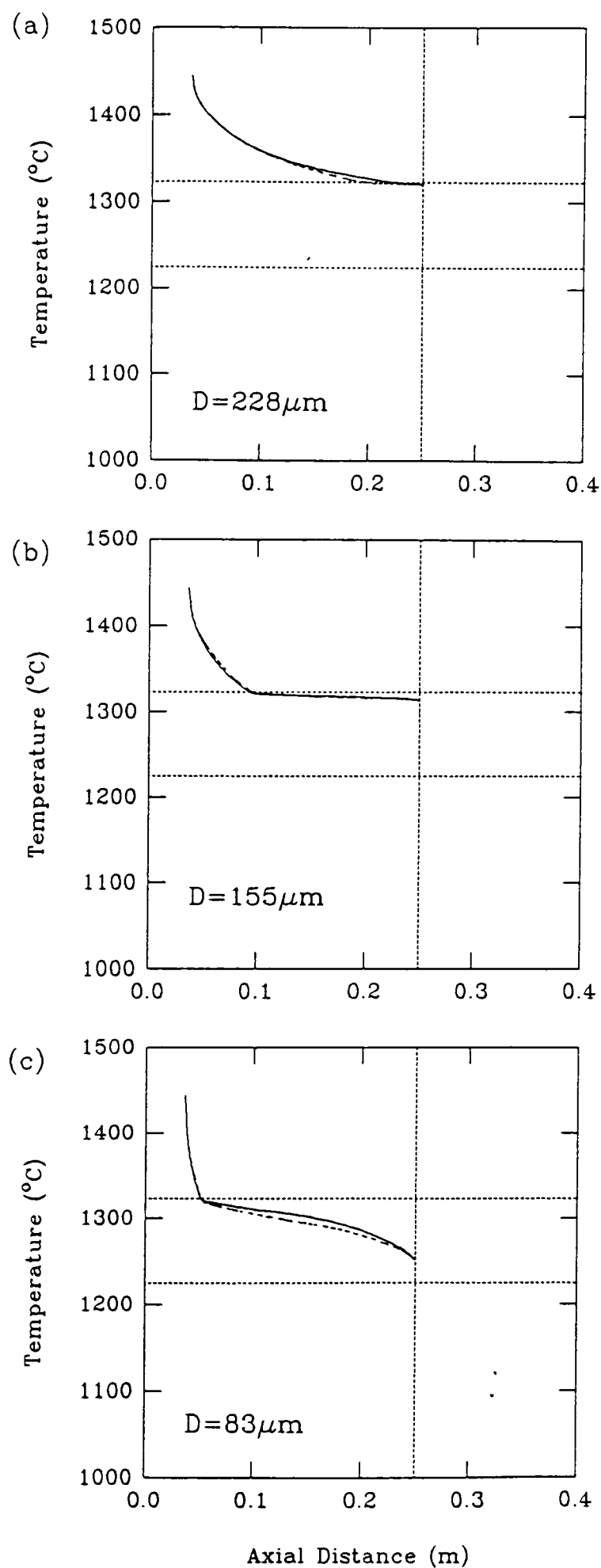
#### 6.4.4.1 Effect of Gas Pressure

Figs 6.21(a)-(c) show the effect of gas pressure in the range 0.413-0.503MPa on droplet temperature as a function of axial distance for the 228 $\mu$ m, 155 $\mu$ m and 83 $\mu$ m droplets respectively at a spray distance of 0.25m and MFR of  $\approx 41\text{gs}^{-1}$ . Once again, the dashed horizontal lines are the solidus and liquidus temperatures, and in this case the dashed vertical line indicates the axial substrate position. Figs 6.21(a)-(c) show that the temperature at the substrate decreased significantly as the droplet size decreased. For example, at a gas pressure of 0.413MPa the droplet temperature at the substrate decreased from 1321°C in fig 6.21(a) to 1251°C in fig 6.21(c) as the droplet size decreased from 228 $\mu$ m to 83 $\mu$ m. Figs 6.21(a)-(c) also show that for all 3 droplets there was virtually no change in droplet temperature at the substrate as the gas pressure increased.

Figs 6.22(a)-(c) show the effect of gas pressure on the droplet radial distance (divergence) as a function of axial distance for the 228 $\mu$ m, 155 $\mu$ m and 83 $\mu$ m droplets respectively at a spray distance of 0.25m and MFR of  $\approx 41\text{gs}^{-1}$ . The dashed vertical and horizontal lines indicate the axial position and radial edge of the substrate respectively. Figs 6.22(a)-(c) show that all 3 droplets spread to greater radial distances at lower gas pressures due to the increased divergence of the gas flow field at lower gas pressures as

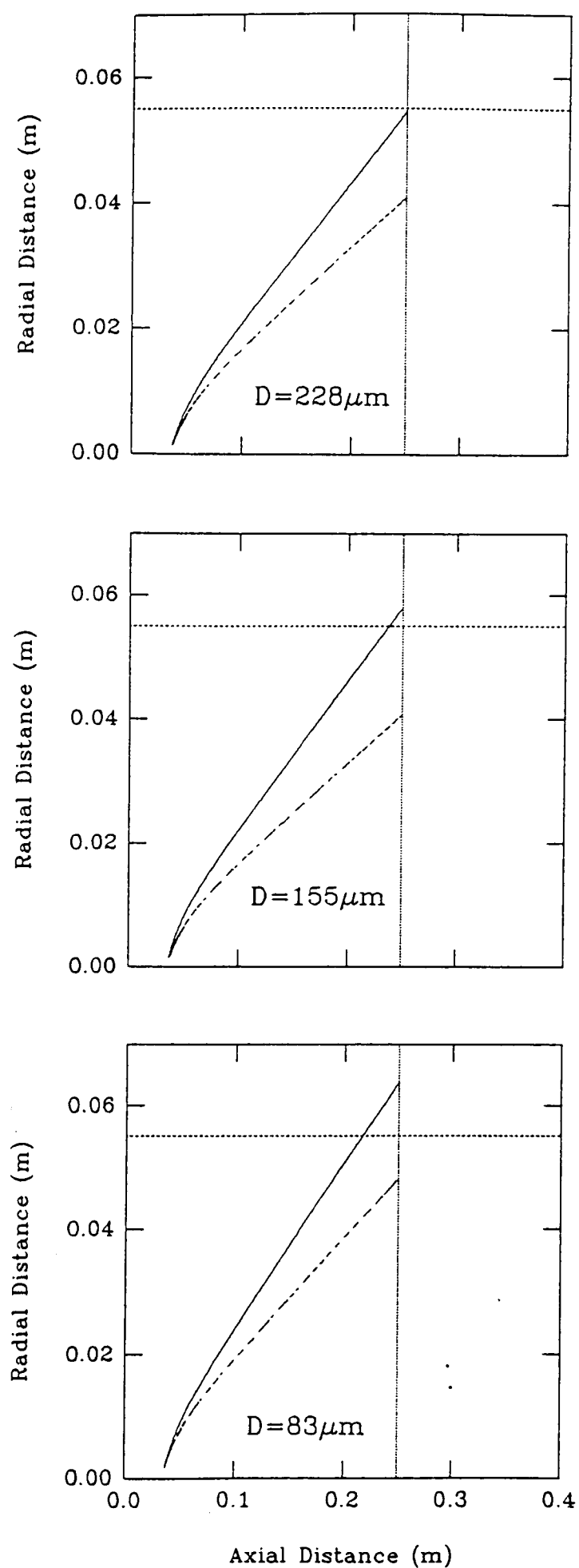






— Run 27, gas press=0.413MPa, spray dist=0.25m, MFR=43gs<sup>-1</sup>  
 ..... Run 31, gas press=0.503MPa, spray dist=0.25m, MFR=39gs<sup>-1</sup>

Fig 6.21 Mean droplet temperature against axial distance for (a) 228 $\mu\text{m}$ , (b) 155 $\mu\text{m}$  and (c) 83 $\mu\text{m}$  droplets to show the effect of gas pressure at a spray distance of 0.25m and MFR of  $\approx 41\text{gs}^{-1}$ .



— Run 27, gas press=0.413MPa, spray dist=0.25m, MFR=43gs<sup>-1</sup>  
 ..... Run 31, gas press=0.503MPa, spray dist=0.25m, MFR=39gs<sup>-1</sup>

Fig 6.22 Mean droplet radial distance against axial distance for (a) 228 $\mu\text{m}$ , (b) 155 $\mu\text{m}$  and (c) 83 $\mu\text{m}$  droplets to show the effect of gas pressure at a spray distance of 0.25m and MFR of  $\approx 41\text{gs}^{-1}$ .

shown in fig 6.15. For example, for the 155 $\mu\text{m}$  droplet in fig 6.22(b), the radial distance at the substrate increased from 0.041m to 0.058m as the gas pressure decreased from 0.503MPa to 0.413MPa. The radial distance at the substrate also increased as the droplet size decreased. For example, at a gas pressure of 0.413MPa the radial distance at the substrate increased from 0.054m in fig 6.22(a) to 0.064m in fig 6.22(c) as the droplet size decreased from 228 $\mu\text{m}$  to 83 $\mu\text{m}$ .

Figs 6.23(a)-(c) show the effect of gas pressure on droplet axial velocity as a function of axial distance for the 228 $\mu\text{m}$ , 155 $\mu\text{m}$  and 83 $\mu\text{m}$  droplets respectively at a spray distance of 0.25m and MFR of  $\approx 41\text{gs}^{-1}$ . The dashed vertical line indicates the axial position of the substrate. Figs 6.23(a)-(c) show that for all 3 droplets the maximum droplet axial velocity increased as the gas pressure increased, due to greater gas velocities and reduced divergence of the gas flow field at higher gas pressures as shown in figs 6.14 and 6.15. For example, for the 155 $\mu\text{m}$  droplet in fig 6.23(b) the maximum droplet axial velocity increased from  $19\text{ms}^{-1}$  to  $24\text{ms}^{-1}$  as the gas pressure increased from 0.413MPa to 0.503MPa. The maximum droplet axial velocity also increased as the droplet size decreased. For example, at a gas pressure of 0.413MPa the maximum droplet axial velocity increased from  $17\text{ms}^{-1}$  in fig 6.23(a) to  $25\text{ms}^{-1}$  in fig 6.23(c) as the droplet size decreased from 228 $\mu\text{m}$  to 83 $\mu\text{m}$ .

#### 6.4.4.2 Effect of Spray Distance

Figs 6.24(a)-(c) show the effect of spray distance in the range 0.2-0.3m on droplet temperature as a function of axial distance for the 228 $\mu\text{m}$ , 155 $\mu\text{m}$  and 83 $\mu\text{m}$  droplets respectively at a gas pressure of 0.503MPa and MFR of  $\approx 59\text{gs}^{-1}$ . The dashed horizontal lines are the solidus and liquidus temperatures and the dashed vertical lines indicate the



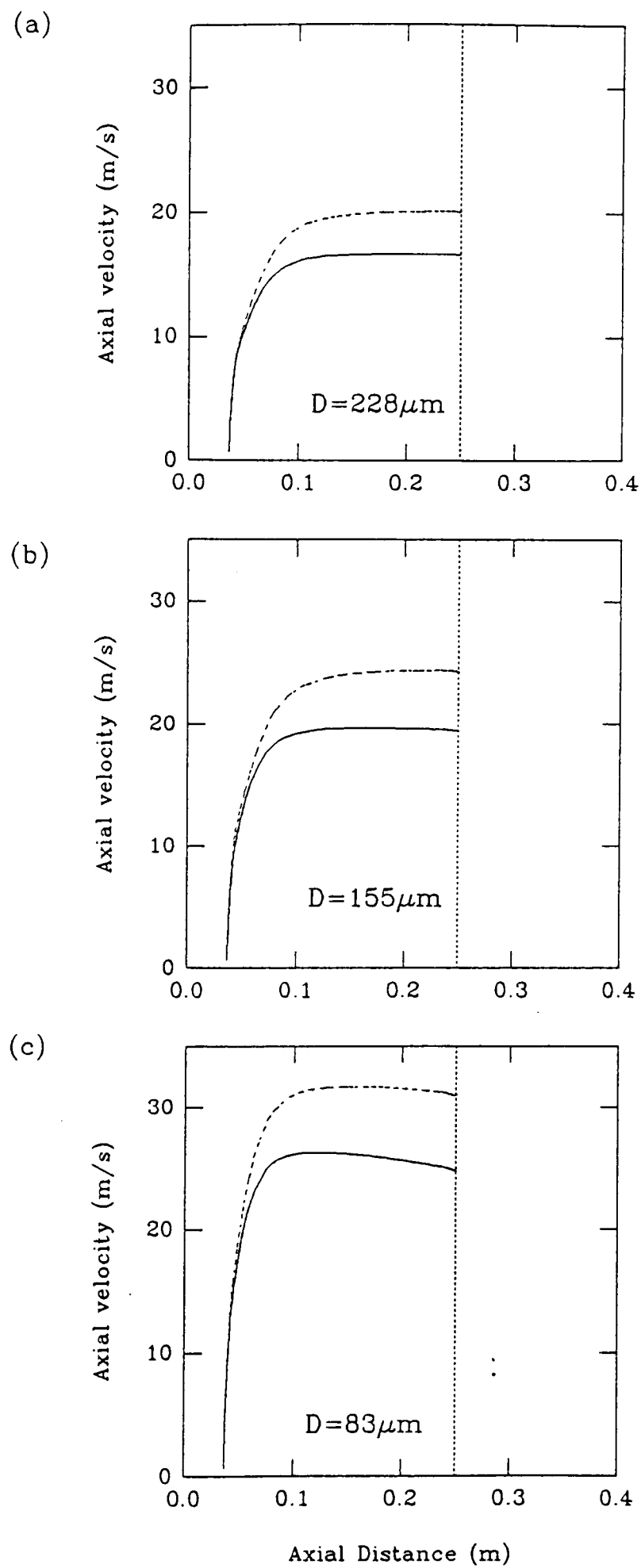
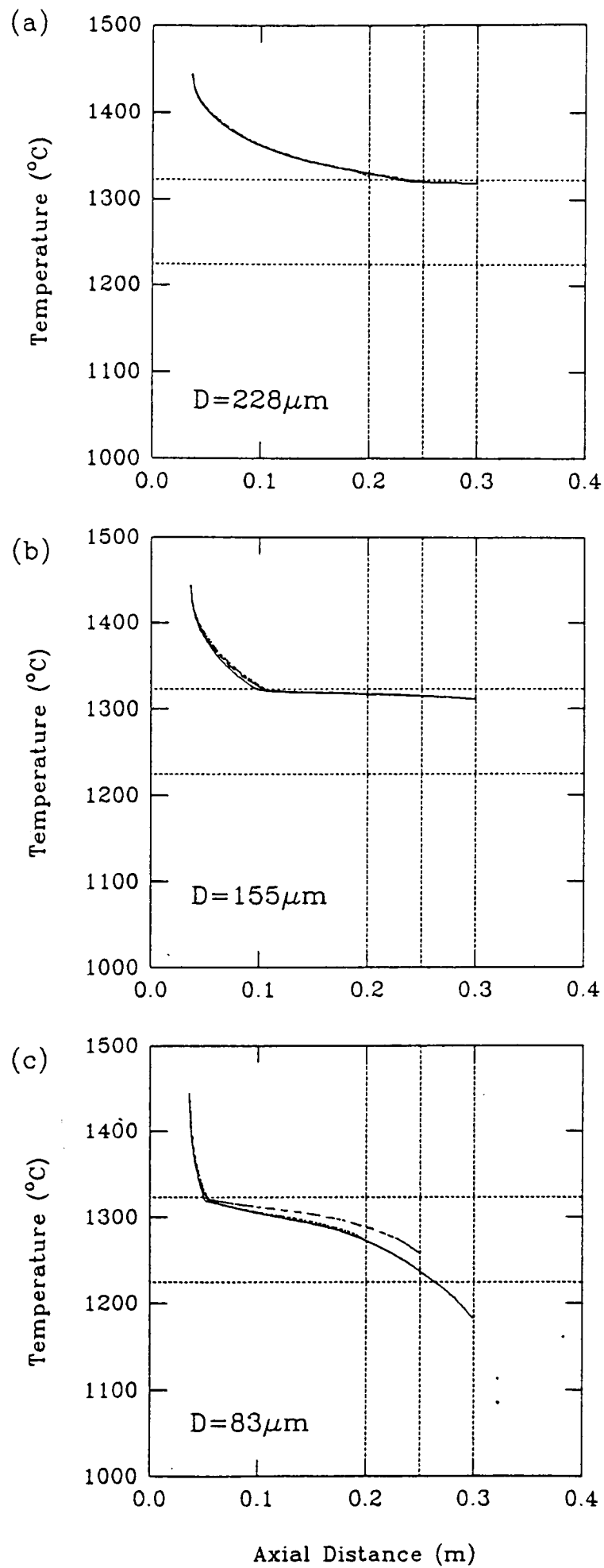


Fig 6.23 Mean droplet axial velocity against axial distance for (a) 228 $\mu\text{m}$ , (b) 155 $\mu\text{m}$  and (c) 83 $\mu\text{m}$  droplets to show the effect of gas pressure at a spray distance of 0.25m and MFR of  $\approx 41\text{gs}^{-1}$ .



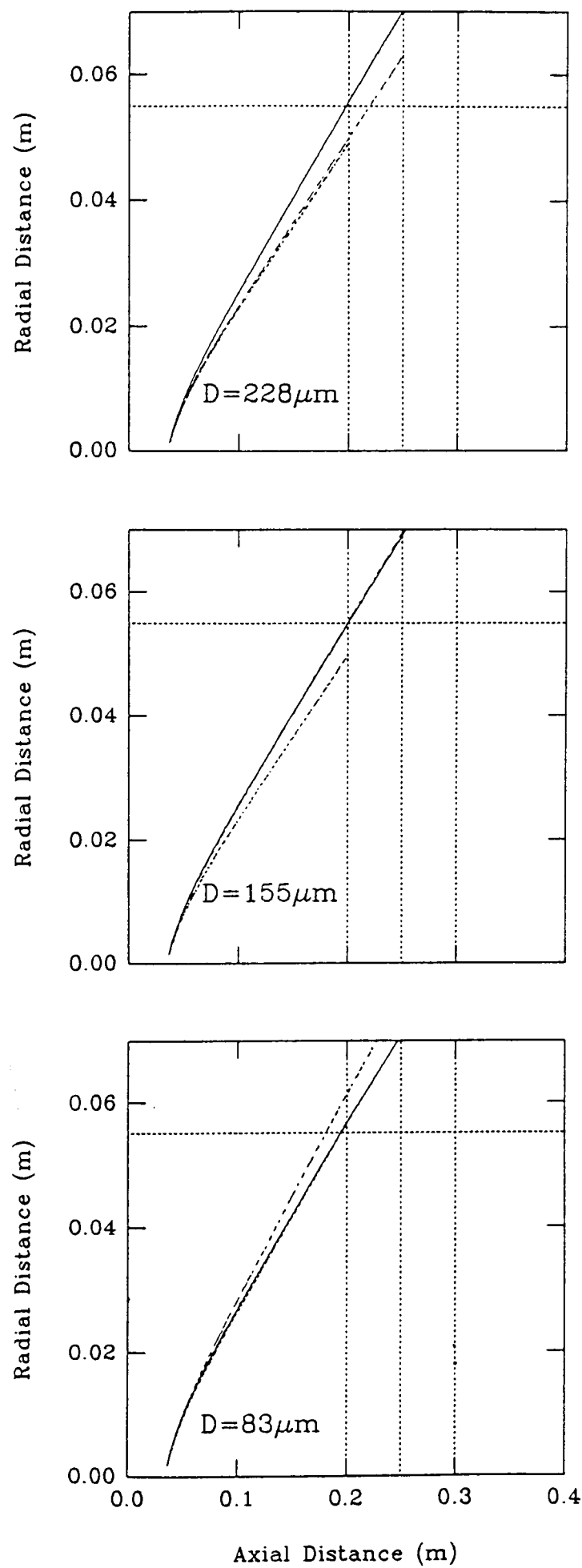
— Run 17, spray dist=0.3m, gas press=0.503MPa, MFR=59gs<sup>-1</sup>  
 - - - Run 21, spray dist=0.25m, gas press=0.503MPa, MFR=59gs<sup>-1</sup>  
 ..... Run 23, spray dist=0.2m, gas press=0.503MPa, MFR=59gs<sup>-1</sup>

Fig 6.24 Mean droplet temperature against axial distance for (a) 228 $\mu\text{m}$ , (b) 155 $\mu\text{m}$  and (c) 83 $\mu\text{m}$  droplets to show the effect of spray distance at a gas pressure of 0.503MPa and MFR of  $\approx 59\text{gs}^{-1}$ .

axial substrate positions at spray distances of 0.2m 0.25m and 0.3m respectively. Figs 6.24(a)-(c) show that the temperature at the substrate decreased significantly as the droplet size decreased. For example, at a spray distance of 0.3m the droplet temperature at the substrate decreased from 1319°C in fig 6.24(a) to 1183°C in fig 6.24(c) as the droplet size decreased from 228µm to 83µm. Figs 6.24(a)-(c) also show that for all 3 droplets the droplet temperature at the substrate decreased as the spray distance increased due to longer flight times.

Figs 6.25(a)-(c) show the effect of spray distance on the droplet radial distance as a function of axial distance for the 228µm, 155µm and 83µm droplets respectively at a gas pressure of 0.503MPa and MFR of  $\approx 59\text{gs}^{-1}$ . The dashed vertical and horizontal lines indicate the axial substrate positions and radial edge of the substrate respectively. Figs 6.25(a)-(c) show that all 3 droplets spread to greater radial distances at longer spray distances. For example, for the 155µm droplet in fig 6.25(b) the radial distance at the substrate increased from 0.050m to 0.083m as the spray distance increased from 0.2m to 0.3m. The radial distance at the substrate also increased as the droplet size decreased. For example, at a spray distance of 0.25m the radial distance at the substrate increased from 0.063m in fig 6.25(a) to 0.077m in fig 6.25(c) as the droplet size decreased from 228µm to 83µm.

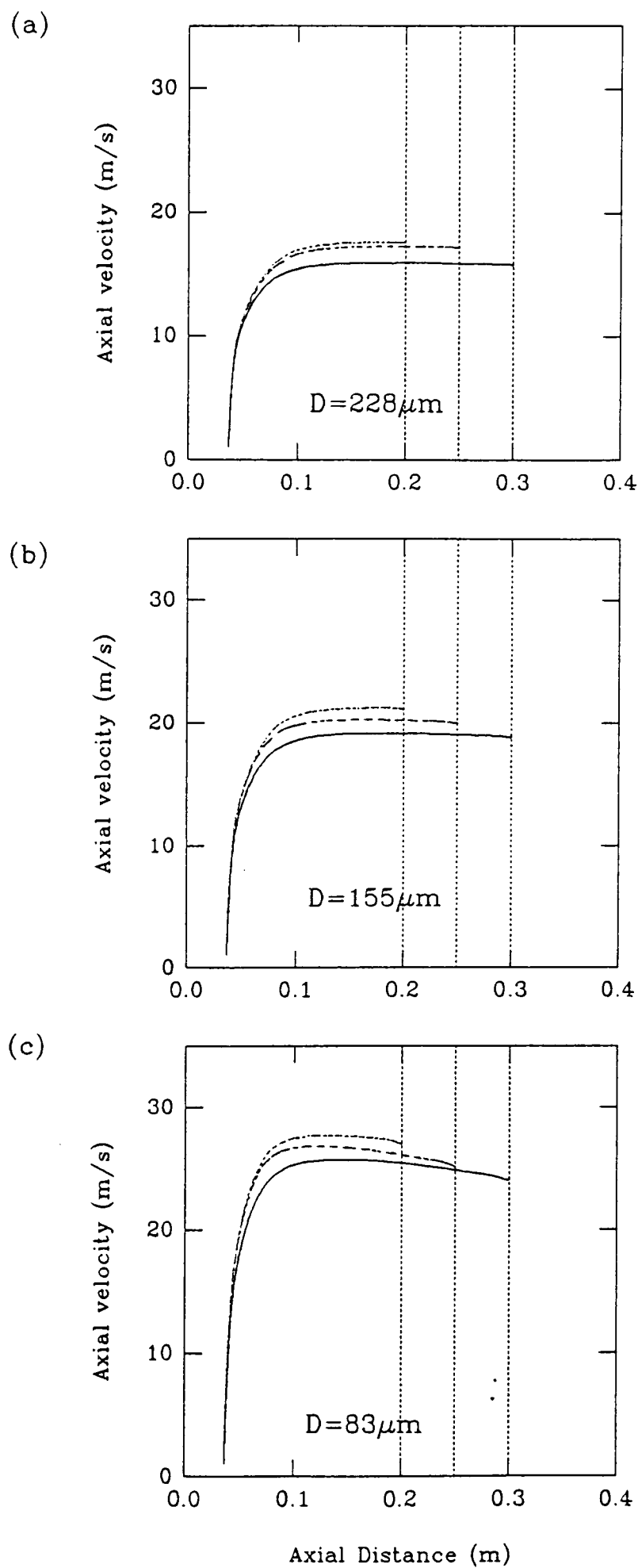
Figs 6.26(a)-(c) show the effect of spray distance on droplet axial velocity as a function of axial distance for the 228µm, 155µm and 83µm droplets respectively at a gas pressure of 0.503MPa and MFR of  $\approx 59\text{gs}^{-1}$ . The dashed vertical lines indicates the axial substrate positions. Figs 6.26(a)-(c) show that for all 3 droplets the maximum droplet axial velocity increased slightly as the spray distance decreased. For example, for the 155µm



— Run 17, spray dist=0.3m, gas press=0.503MPa, MFR=59gs<sup>-1</sup>  
 ..... Run 21, spray dist=0.25m, gas press=0.503MPa, MFR=59gs<sup>-1</sup>  
 -.-.- Run 23, spray dist=0.2m, gas press=0.503MPa, MFR=59gs<sup>-1</sup>

Fig 6.25 Mean droplet radial distance against axial distance for (a) 228 $\mu\text{m}$ , (b) 155 $\mu\text{m}$  and (c) 83 $\mu\text{m}$  droplets to show the effect of spray distance at a gas pressure of 0.503MPa and MFR of  $\approx 59\text{gs}^{-1}$ .





— Run 17, spray dist=0.3m, gas press=0.503MPa, MFR=59gs<sup>-1</sup>  
 ..... Run 21, spray dist=0.25m, gas press=0.503MPa, MFR=59gs<sup>-1</sup>  
 ..... Run 23, spray dist=0.2m, gas press=0.503MPa, MFR=59gs<sup>-1</sup>

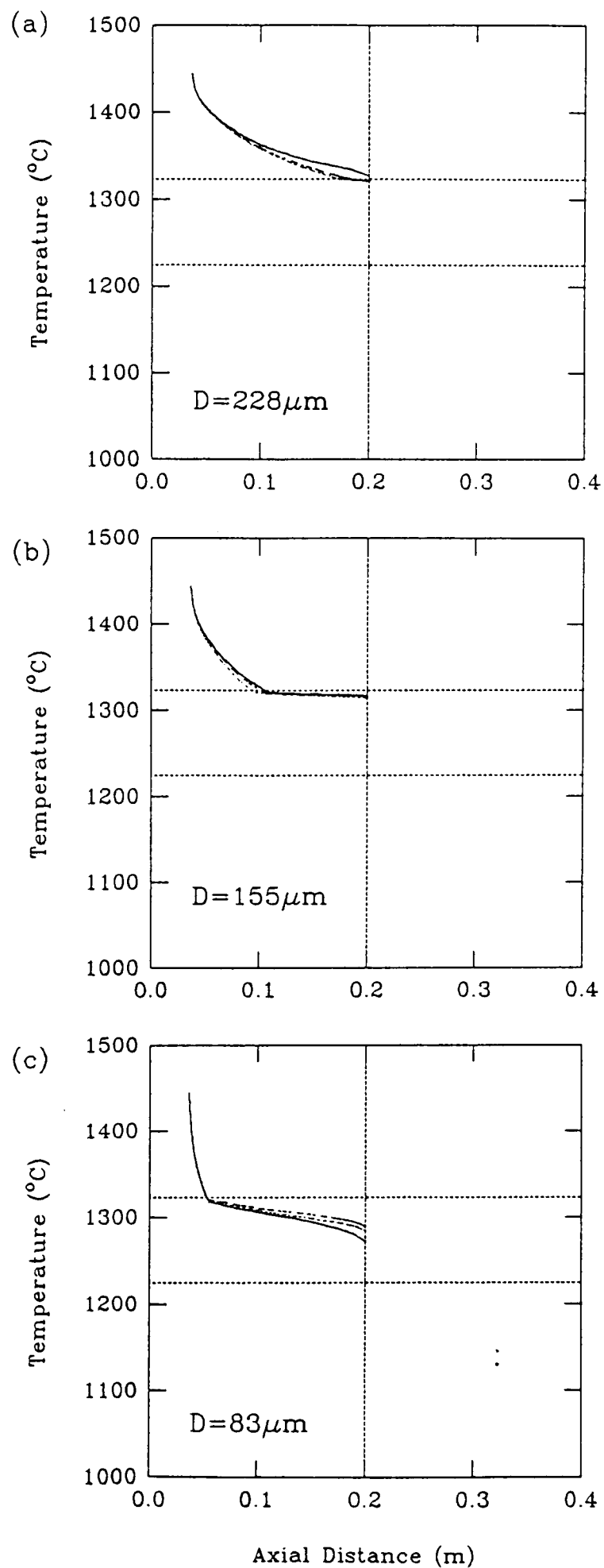
Fig 6.26 Mean droplet axial velocity against axial distance for (a) 228 $\mu\text{m}$ , (b) 155 $\mu\text{m}$  and (c) 83 $\mu\text{m}$  droplets to show the effect of spray distance at a gas pressure of 0.503MPa and MFR of  $\approx 59\text{gs}^{-1}$ .

droplet in fig 6.26(b) the maximum droplet axial velocity increased from  $19\text{ms}^{-1}$  to  $21\text{ms}^{-1}$  as the spray distance decreased from 0.3m to 0.2m. The maximum droplet axial velocity also increased as the droplet size decreased. For example, at a spray distance of 0.25m the maximum droplet axial velocity increased from  $17\text{ms}^{-1}$  in fig 6.26(a) to  $25\text{ms}^{-1}$  in fig 6.26(c) as the droplet size decreased from  $228\mu\text{m}$  to  $83\mu\text{m}$ .

#### 6.4.4.3 Effect of Melt Flow Rate

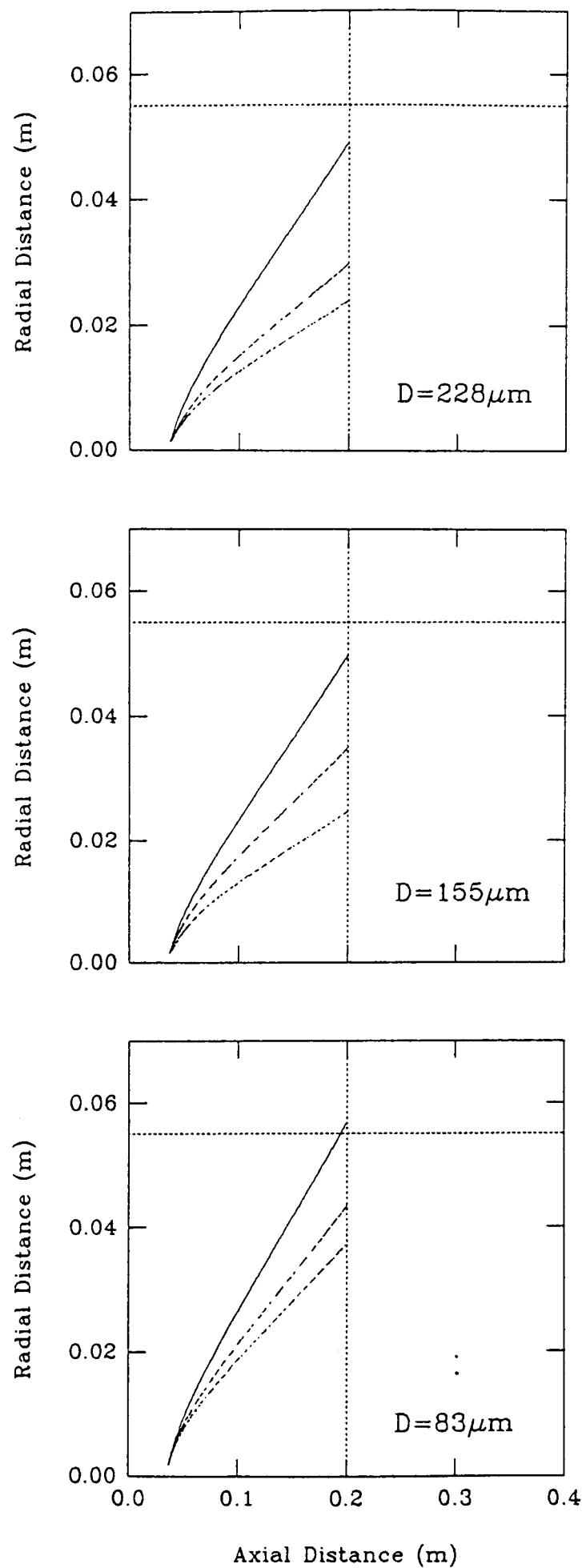
Figs 6.27(a)-(c) show the effect of MFR in the range  $38\text{-}59\text{gs}^{-1}$  on droplet temperature as a function of axial distance for the  $228\mu\text{m}$ ,  $155\mu\text{m}$  and  $83\mu\text{m}$  droplets respectively at a gas pressure of 0.503MPa and spray distance of 0.2m. The dashed horizontal lines are the solidus and liquidus temperatures and the dashed vertical line indicates the axial substrate position. Figs 6.27(a)-(c) show that the droplet temperature at the substrate decreased significantly as the droplet size decreased. For example, at a MFR of  $45\text{gs}^{-1}$  the droplet temperature at the substrate decreased from  $1321^\circ\text{C}$  in fig 6.27(a) to  $1290^\circ\text{C}$  in fig 6.27(c) as the droplet size decreased from  $228\mu\text{m}$  to  $83\mu\text{m}$ . Figs 6.27(a)-(c) also show that for all 3 droplets there was virtually no change in droplet temperature at the substrate as the MFR increased.

Figs 6.28(a)-(c) show the effect of MFR on droplet radial distance as a function of axial distance for the  $228\mu\text{m}$ ,  $155\mu\text{m}$  and  $83\mu\text{m}$  droplets respectively at a gas pressure of 0.503MPa and spray distance of 0.2m. The dashed vertical and horizontal lines indicate the axial position and radial edge of the substrate respectively. Figs 6.28(a)-(c) show that all 3 droplets spread to greater radial distances at higher MFR's due to the increased divergence of the gas flow field at higher MFR's as shown in fig 6.18. For example, for



— Run 23,  $\text{MFR}=59\text{gs}^{-1}$ , spray dist=0.2m, gas press=0.503MPa  
 ..... Run 32,  $\text{MFR}=45\text{gs}^{-1}$ , spray dist=0.2m, gas press=0.503MPa  
 ..... Run 34,  $\text{MFR}=38\text{gs}^{-1}$ , spray dist=0.2m, gas press=0.503MPa

Fig 6.27 Mean droplet temperature against axial distance for (a)  $228\mu\text{m}$ , (b)  $155\mu\text{m}$  and (c)  $83\mu\text{m}$  droplets to show the effect of MFR at a spray distance of 0.25m and gas pressure of 0.503MPa.



— Run 23, MFR=59gs<sup>-1</sup>, spray dist=0.2m, gas press=0.503MPa  
 ..... Run 32, MFR=45gs<sup>-1</sup>, spray dist=0.2m, gas press=0.503MPa  
 ..... Run 34, MFR=38gs<sup>-1</sup>, spray dist=0.2m, gas press=0.503MPa

Fig 6.28 Mean droplet radial distance against axial distance for (a) 228 $\mu$ m, (b) 155 $\mu$ m and (c) 83 $\mu$ m droplets to show the effect of MFR at a spray distance of 0.25m and gas pressure of 0.503MPa.

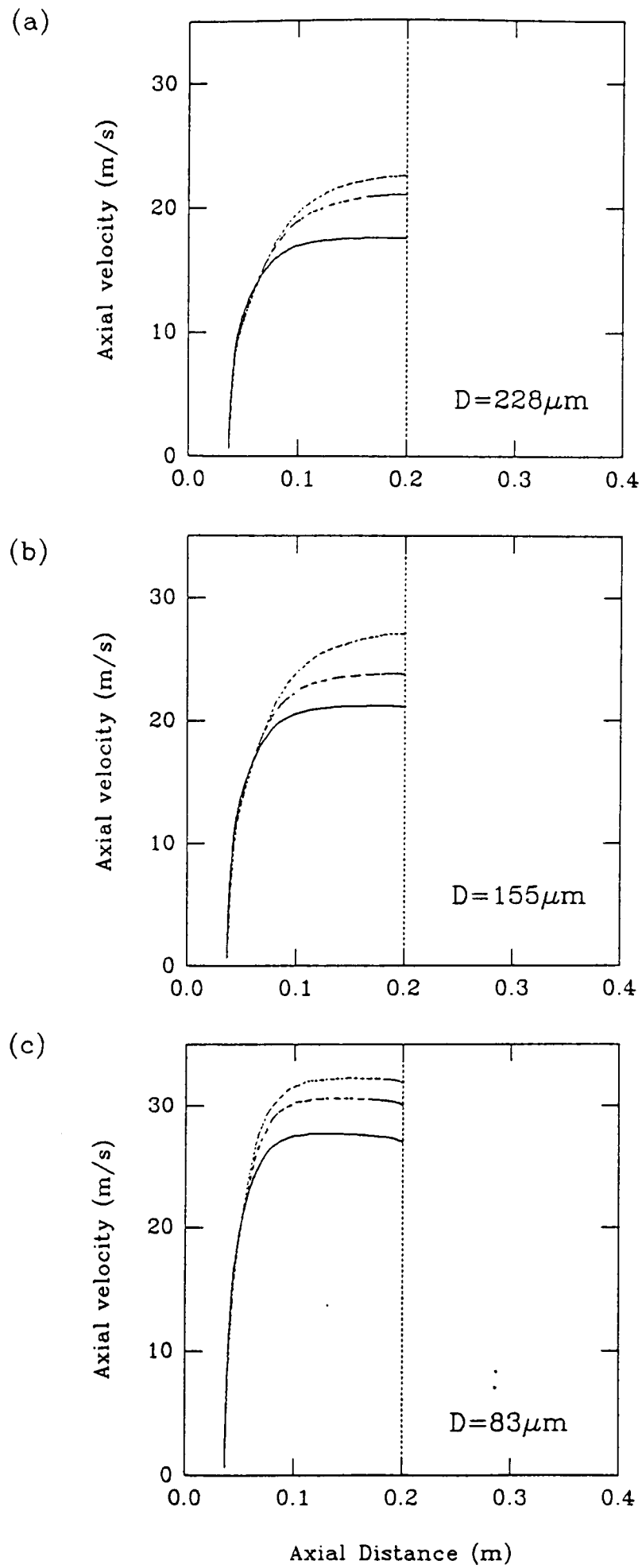


the 155 $\mu\text{m}$  droplet in fig 6.28(b) the radial distance at the substrate increased from 0.025m to 0.05m as the MFR increased from 38 $\text{gs}^{-1}$  to 59 $\text{gs}^{-1}$ . Figs 6.28(a)-(c) also show that the radial distance at the substrate increased as the droplet size decreased. For example, at a MFR of 45 $\text{gs}^{-1}$  the radial distance at the substrate increased from 0.030m in fig 6.28(a) to 0.043m in fig 6.28(c) as the droplet size decreased from 228 $\mu\text{m}$  to 83 $\mu\text{m}$ .

Figs 6.29(a)-(c) show the effect of MFR on droplet axial velocity as a function of axial distance for the 228 $\mu\text{m}$ , 155 $\mu\text{m}$  and 83 $\mu\text{m}$  droplets respectively at a gas pressure of 0.503MPa and spray distance of 0.2m. The dashed vertical line indicates the axial substrate position. Figs 6.29(a)-(c) show that for all 3 droplets the maximum droplet axial velocity decreased as the MFR increased, due to lower gas velocities and increased divergence of the gas flow field at higher MFR's as shown in figs 6.17 and 6.18. For example, for the 155 $\mu\text{m}$  droplet in fig 6.29(b) the maximum droplet axial velocity decreased from 27 $\text{ms}^{-1}$  to 21 $\text{ms}^{-1}$  as the MFR increased from 38 $\text{gs}^{-1}$  to 59 $\text{gs}^{-1}$ . Figs 6.29(a)-(c) also show that the maximum droplet axial velocity increased as the droplet size decreased. For example, at a MFR of 45 $\text{gs}^{-1}$  the maximum droplet axial velocity increased from 21 $\text{ms}^{-1}$  in fig 6.29(a) to 30 $\text{ms}^{-1}$  in fig 6.29(c) as the droplet size decreased from 228 $\mu\text{m}$  to 83 $\mu\text{m}$ .

#### **6.4.5 Deposit Profiles**

The standard deviation of the best fit Gaussian curve to the calculated normalised  $M_{\text{ar/a}}$  against radial distance from Grant's post-processing program was compared with the standard deviation of the best fit Gaussian curve to the measured normalised deposit height against radial distance from the real deposits. The standard deviations of both the



— Run 23,  $\text{MFR}=59\text{gs}^{-1}$ , spray dist=0.2m, gas press=0.503MPa  
 - - - Run 32,  $\text{MFR}=45\text{gs}^{-1}$ , spray dist=0.2m, gas press=0.503MPa  
 ..... Run 34,  $\text{MFR}=38\text{gs}^{-1}$ , spray dist=0.2m, gas press=0.503MPa

Fig 6.29 Mean droplet axial velocity against axial distance for (a)  $228\mu\text{m}$ , (b)  $155\mu\text{m}$  and (c)  $83\mu\text{m}$  droplets to show the effect of MFR at a spray distance of 0.25m and gas pressure of 0.503MPa.

calculated and measured best fit Gaussian curves were then analysed as a function of gas pressure, spray distance and MFR.

Figs 6.30(a) and (b) show typical FLUENT predictions of the normalised  $M_{ar/a}$  against radial distance and the best fit Gaussian curves at spray distances 0.25m and 0.2m respectively. In both cases the gas pressure and MFR were 0.413MPa and  $\approx 50\text{gs}^{-1}$  respectively. The data in both figures fitted well to the Gaussian curve and the best fit standard deviation decreased as the spray distance decreased. Figs 6.31(a) and (b) show typical measured normalised deposit heights against radial distance from the corresponding real deposits and the best fit Gaussian curves at spray distances of 0.25m and 0.2m. Again the data in both figures fitted well to the Gaussian curve similar to figs 6.30(a)-(b) and the best fit standard deviation decreased as the spray distance decreased.

#### 6.4.5.1 Effect of Gas Pressure

Fig 6.32 shows the standard deviation (s.d) of the best fit Gaussian curve to (a) the normalised  $M_{ar/a}$  against radial distance from FLUENT and (b) the measured normalised deposit height against radial distance as a function of gas pressure. Overall, the standard deviations in both figures were of similar order and decreased slightly as the gas pressure increased. For example, at a spray distance of 0.2m and MFR of  $\approx 41\text{g/s}$  the best fit s.d from FLUENT decreased from 0.035m to 0.034m and the best fit s.d from the real deposits decreased from 0.027m to 0.023m as the gas pressure increased from 0.413MPa to 0.503MPa.

#### 6.4.5.2 Effect of Spray Distance

Fig 6.33 shows the s.d of the best fit Gaussian curve to (a) the normalised  $M_{ar/a}$  against radial distance from FLUENT and (b) the measured normalised deposit height

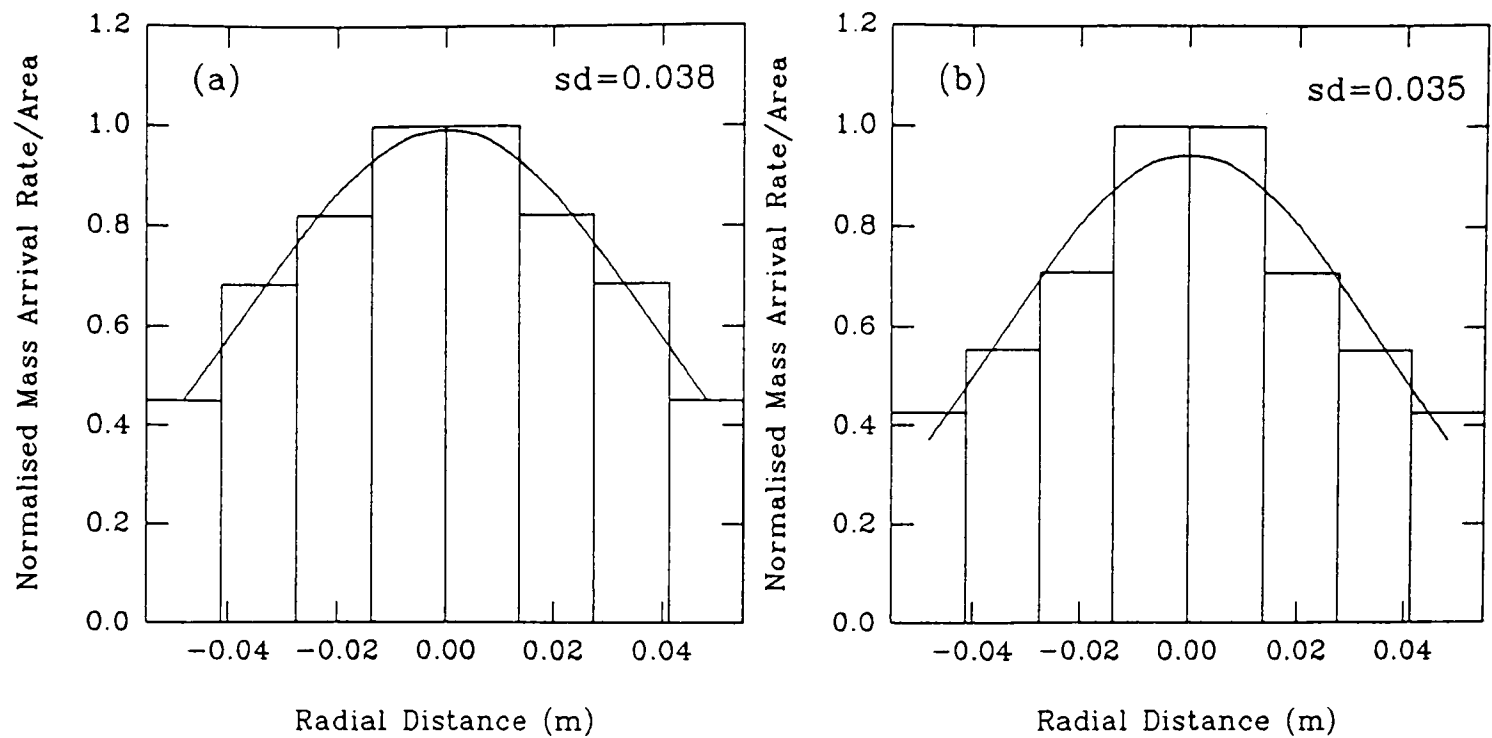


Fig 6.30 Normalised  $M_{ar/a}$  against radial distance from FLUENT for (a) run 37 at a spray distance of 0.25m, gas pressure of 0.413MPa and MFR of 50gs<sup>-1</sup> and (b) run 24 at a spray distance of 0.2m, gas pressure of 0.413MPa and MFR of 50gs<sup>-1</sup>.

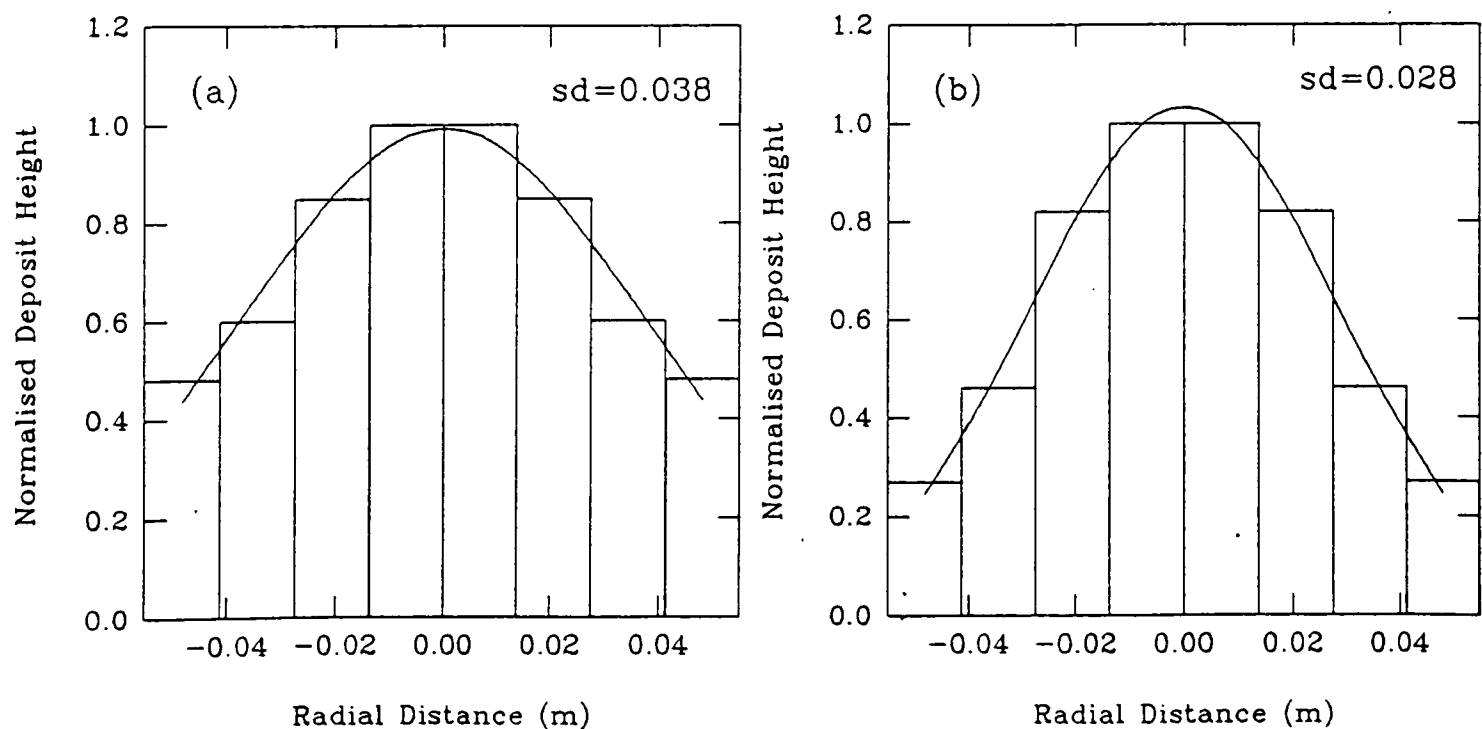


Fig 6.31 Normalised deposit height against radial distance from the real deposits for (a) run 37 at a spray distance of 0.25m, gas pressure of 0.413MPa and MFR of 50gs<sup>-1</sup> and (b) run 24 at a spray distance of 0.2m, gas pressure of 0.413MPa and MFR of 50gs<sup>-1</sup>.



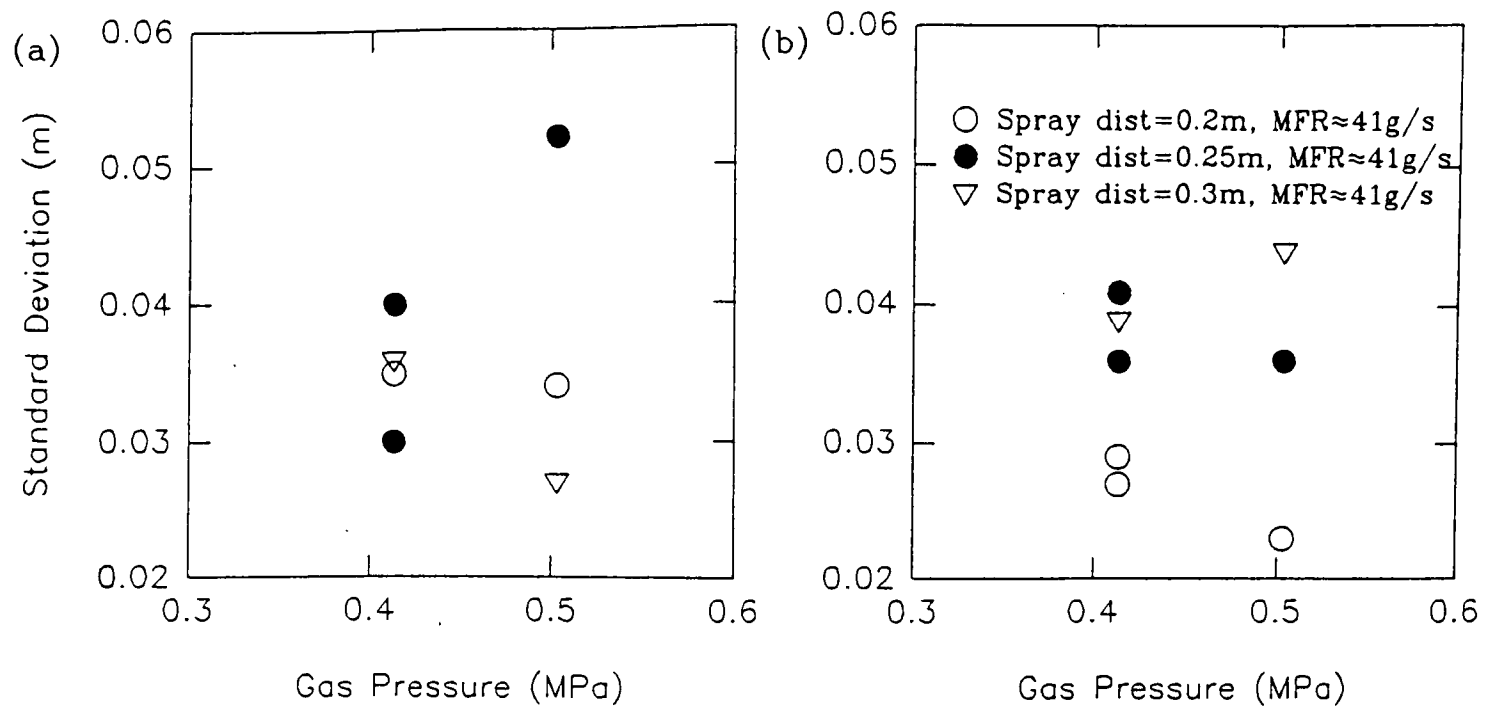


Fig 6.32 Gas pressure against standard deviation of the best fit Gaussian curve to (a) normalised  $M_{ar/a}$  against radial distance and (b) normalised deposit height against radial distance.

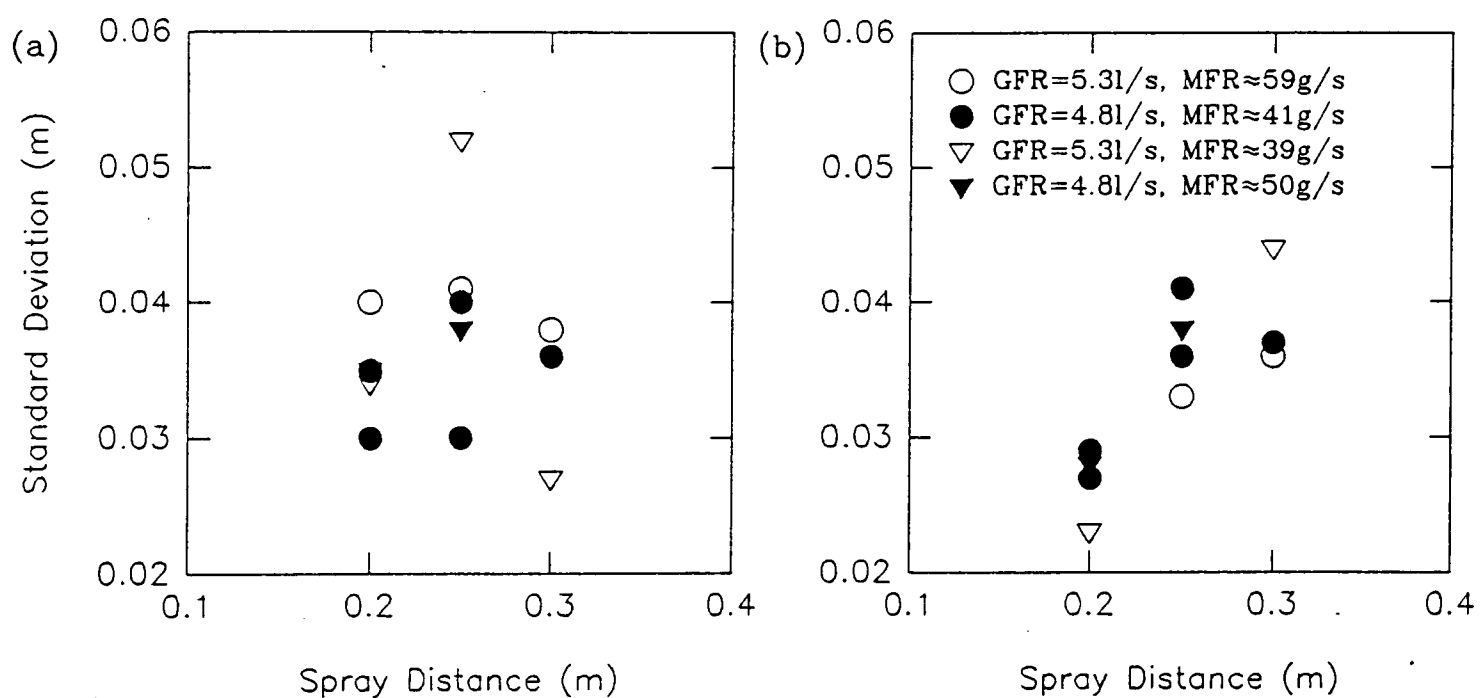


Fig 6.33 Spray distance against standard deviation of the best fit Gaussian curve to (a) normalised  $M_{ar/a}$  against radial distance and (b) normalised deposit height against radial distance.

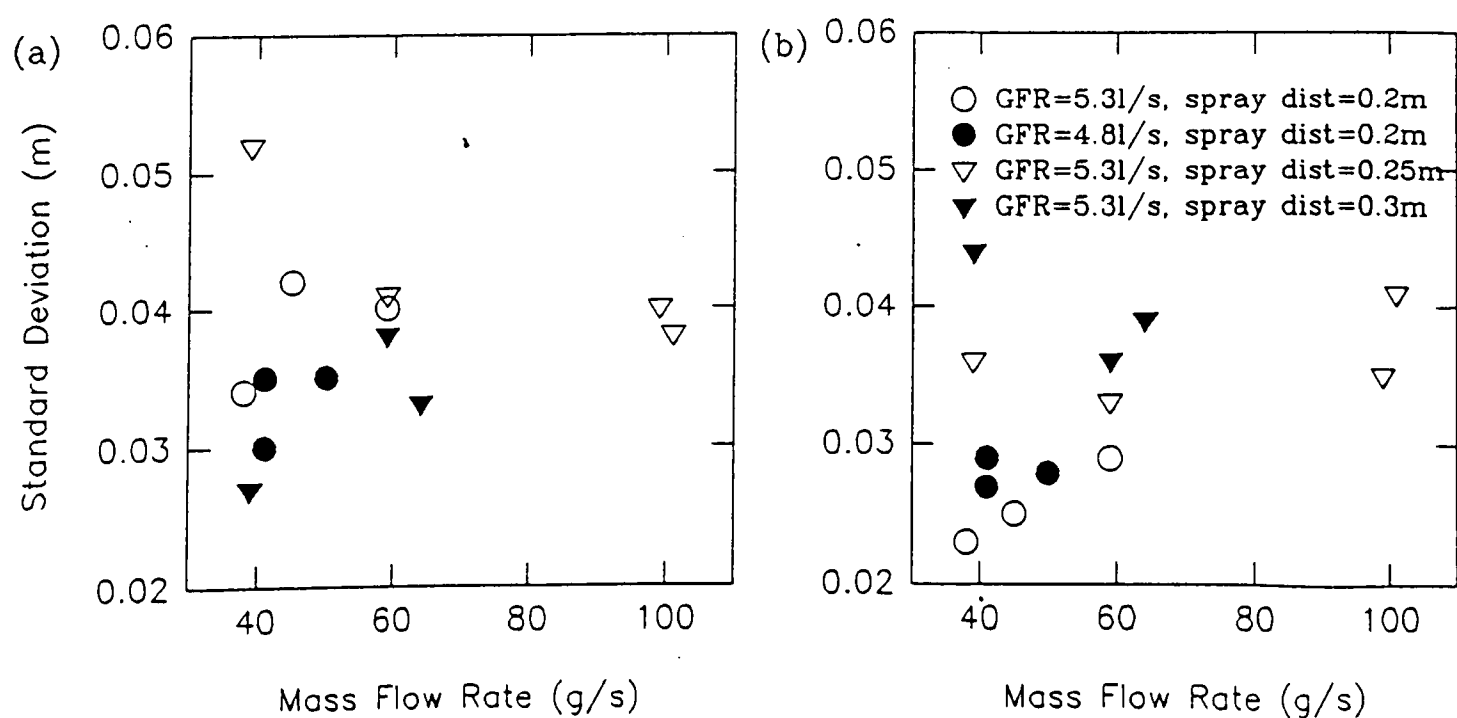


Fig 6.34 MFR against standard deviation of the best fit Gaussian curve to (a) normalised  $M_{ar/a}$  against radial distance and (b) normalised deposit height against radial distance

against radial distance as a function of spray distance. Overall, the s.d's from FLUENT and the real deposits were of a similar order. The s.d from the real deposits decreased strongly as the spray distance decreased while the s.d's from FLUENT were more variable, although there was a slight decrease as the spray distance decreased. For example, at a gas pressure and MFR of 0.413MPa and  $\approx 50\text{g/s}$  respectively the best fit s.d from FLUENT decreased from 0.038m to 0.035m and the best fit s.d from the real deposits decreased from 0.038m to 0.028m as the spray distance decreased from 0.25m to 0.2m.

#### 6.4.5.3 Effect of Melt Flow Rate

Fig 6.34 shows the s.d of the best fit Gaussian curve to (a) the normalised  $M_{ar/a}$  against radial distance from FLUENT and (b) the normalised deposit height against radial distance as a function of MFR. Overall, the s.d's from FLUENT and the real deposits were of similar order. The s.d from the real deposits decreased as the MFR decreased while the s.d's from FLUENT were more variable, although there was a slight decrease in s.d as the MFR increased. For example, at a spray distance and gas pressure of 0.2m and 0.503MPa respectively the best fit s.d from FLUENT decreased from 0.040m to 0.034m and the best fit s.d from the real deposits decreased from 0.029m to 0.023m as the MFR decreased from  $59\text{gs}^{-1}$  to  $38\text{gs}^{-1}$ .

### 6.5 Summary

A commercial computational fluid dynamic package, FLUENT, has been used to predict the dynamic and thermal behaviour of gas and droplets in spray forming under a range of process parameters. The atomisation process is not modelled directly and instead

droplets are injected into the gas flow field. The axial gas velocity decreased approximately exponentially with axial distance, but decreased more rapidly on impacting the droplets. Axial gas velocities were higher and radial gas velocities (divergence) were lower at: (i) greater gas pressure and (ii) lower MFR's.

The equilibrated spray temperature at the substrate increased as: (i) gas pressure decreased; (ii) spray distance decreased; and (iii) MFR increased. The temperature of individual droplets at the substrate was strongly dependent on droplet size with smaller droplets cooling much faster with axial distance. The temperature of individual 228 $\mu\text{m}$ , 155 $\mu\text{m}$  and 83 $\mu\text{m}$  droplets were not very sensitive to changes in gas pressure or MFR, and changes in spray temperature at the substrate were mainly due to changes in the overall droplet size distribution with process parameters and spray distance. The droplet radial distance (divergence) at the substrate increased and the maximum droplet axial velocity decreased as: (i) gas pressure decreased; (ii) spray distance increased; and (iii) MFR increased.

The normalised  $M_{\text{ar/a}}$  against radial distance from FLUENT and normalised deposit height against radial distance from the real deposits both fitted well to a Gaussian curve. In both cases the standard deviation of the best fit Gaussian curve decreased as the: (i) gas pressure increased; (ii) spray distance decreased; and (iii) MFR decreased. However, the prediction of deposit shape from FLUENT did not show good agreement with measured values in all cases.

## **CHAPTER 7-DISCUSSION AND COMPARISON OF EXPERIMENTAL AND COMPUTER MODELLING RESULTS**

This chapter discusses and compares the experimental spray forming results with the computer modelling predictions obtained using FLUENT. The effect of the independent variables: gas flow rate, spray distance and melt flow rate, and the dependent variable, deposition rate on deposit temperature as well as microstructural evolution are discussed.

### **7.1 Atomisation of the Melt**

The atomisation of the melt stream to form a spray of droplets, always with a range of sizes and therefore trajectories and temperatures is a critical aspect of the spray forming process. However, it is still poorly understood. Experimentally, the size distribution of gas atomised powders, produced under a range of conditions, is often found to obey a log-normal distribution (Lubanska 1970, See and Johnston 1978, Rao and Mehrota 1980) as shown below:

$$f(d) = \left(\frac{k}{s}\right) \exp\left(-\frac{(\ln(d)-\ln(m))^2}{2s^2}\right) \quad (7.1)$$

where  $d$  is the droplet diameter,  $m$  is the mean droplet diameter,  $s$  is the standard deviation and  $k$  is a normalising constant. However, distinct secondary or tertiary peaks have been reported for data plotted in this form for gas atomised Al and Mg powders (Unal 1989A and 1989B) and electric arc atomised Ni aluminides (Newbery 1993). Although as pointed out by Bewlay and Cantor (1990) bimodality is sometimes obscured when droplet size data is plotted cumulatively.



In this study overspray powder distributions from the modified atomiser generally approximate well to a log-normal distribution as shown in figs 5.3(a)-(e) and figs 5.39(a), (c) and (f). Although, figs 5.3(a)-(e) show the appearance of a secondary peak at 63µm-90µm as the MFR:GFR is decreased, i.e. there are more smaller droplets in the spray which indicates that atomisation was slightly more efficient, although droplet sizes are still quite large (mass median diameter=115-157µm).

The variation of the droplet mass median diameter ( $d_m$ ) is often found to follow the semi-empirical correlation proposed by Lubanska (1970) from experiments on iron and steel alloys using a spray ring atomiser:

$$\frac{d_m}{D} = k \left[ \left( 1 + \frac{M_l}{M_g} \right) \frac{w_l}{w_g W} \right]^{0.5} \quad (7.2)$$

where  $D$  is the diameter of the liquid metal stream,  $k$  is an experimentally determined constant for particular conditions of the atomiser and liquid stream and is in the range 40-50 (Lubanska 1970),  $w_l$  and  $w_g$  are the kinematic viscosities of the metal and gas respectively,  $M_l$  and  $M_g$  are the metal and gas mass flow rates respectively and  $W$  is the Weber number defined as  $W = \rho_l V_g^2 D / \gamma_l$ , where  $\rho_l$  is the metal density,  $V_g$  is the gas velocity and  $\gamma_l$  is the metal surface tension. This correlation shows that  $d_m$  is proportional to MFR:GFR (the ratio of  $M_l$  to  $M_g$ ).

Only overspray powder was measured in this study as the spray chamber was not long enough to collect fully solidified powder without the substrate. Overspray powder is not representative of the powder landing on the substrate because of the tendency for smaller droplets to be preferentially entrained in the gas flow (Grant *et al* 1993A). Newbery (1993) compared overspray powder with powder collected without a substrate

for electric arc sprayed Ni aluminides and found that the mass median size of overspray powder was up to 50% smaller than the powder collected without a substrate. Therefore, the mass median size of droplets landing on the substrate could be 200 $\mu$ m-250 $\mu$ m which is comparatively large in comparison with reported means of 100-150 $\mu$ m (Mathur *et al* 1989, Grant 1991, Bewlay 1988, Lavernia 1989) indicating that atomisation was relatively inefficient.

Two different atomisers were used for spray forming UDIMET 720. The first atomiser was used for runs 0-4 and the modified atomiser shown in fig 3.4 was used for all subsequent spray forming runs. Fig 7.1 shows  $\log(d_m/D)$  against the function  $\log(\Phi)$ , where  $\Phi$  is the bracketed function on the right hand side of equation 7.2. Only 2 data points are available for the original atomiser and  $k=38.8$  gives the best fit to these points.  $k=56.8$  gives a good fit for the modified atomiser, excluding runs 19 and 20. For runs 19 and 20 spray formed with the modified atomiser the nozzle failed completely causing a substantial change in the atomisation conditions leading to a best fit  $k=43.9$  for these runs.

## **7.2 Accuracy of Embedded Thermocouple Measurements**

Errors in embedded thermocouple measurements arise from: (i) the accuracy of thermocouples at high temperatures; (ii) the response time of the thermocouples and (iii) the build up of droplets prior to embedding on the thermocouples which further reduces the response time. The experimental reproducibility of spray forming is also a problem when making embedded thermocouple or thermal imaging measurements.

The accuracy of thermocouples operating near the limit of their range ( $\approx 1300^\circ\text{C}$ ) is  $\approx \pm 7^\circ\text{C}$ . From comparison of runs 33 and 35 and 19 and 20 in table 5.3, under

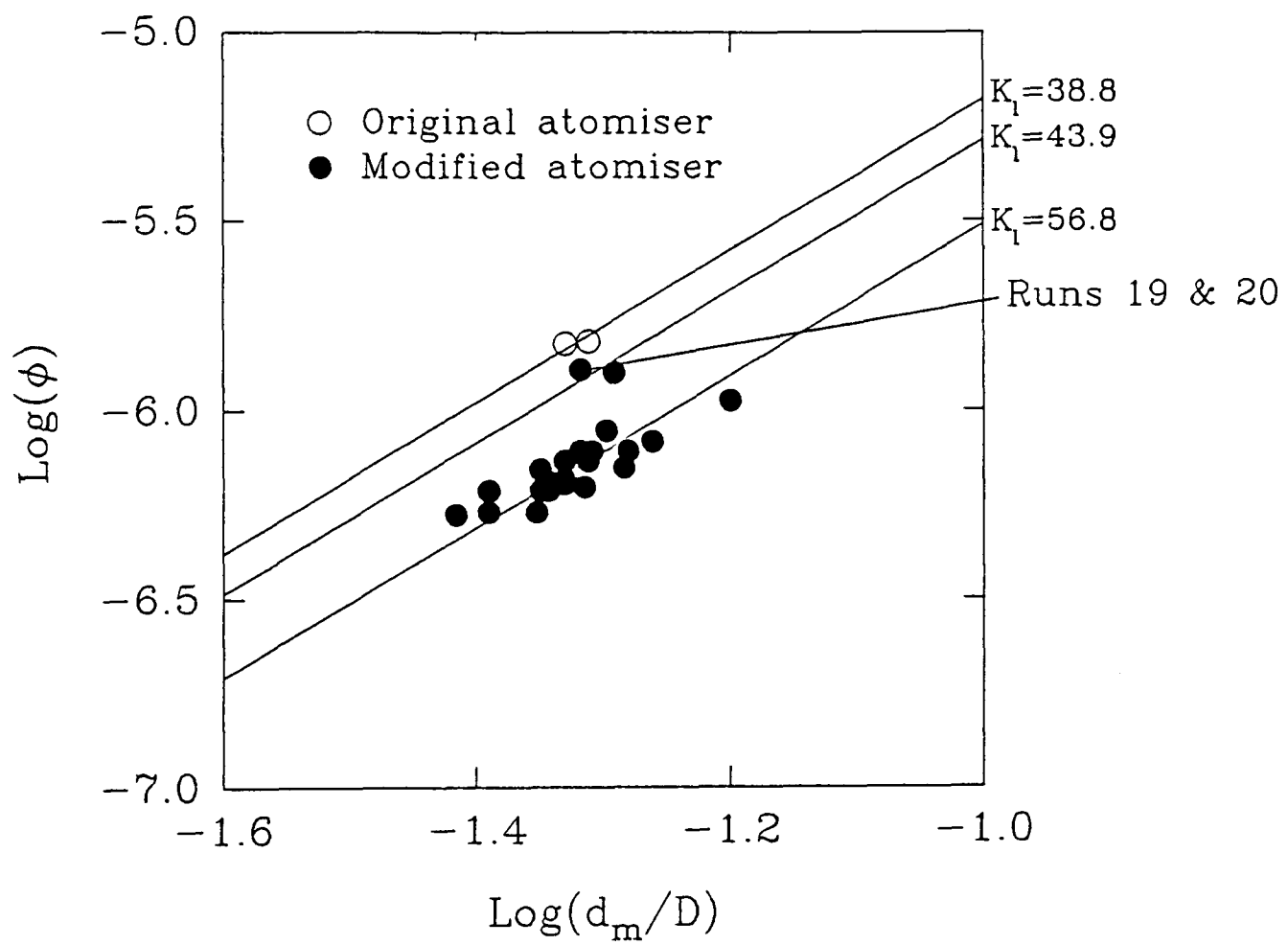


Fig 7.1  $\text{Log}(\phi)$  against  $\text{log}(d_m/D)$  showing the Lubanska correlation for overspray powder from the original and modified atomisers.

nominally similar process parameters the experimental reproducibility is within the above error for thermocouple accuracy.

The quoted manufacturer's thermocouple response time of  $\approx 0.4\text{ms}$  is small compared with the data collection rate of  $1.5\text{Hz}$ . However, the build up of droplets which form a mini deposit on the thermocouple tip during spray forming can significantly reduce the response time and affect the embedding of the thermocouple in the growing deposit by causing porosity to develop around the tip of the thermocouple so it is no longer in intimate contact with the deposit. The measurement of temperatures close to the solidus ( $T_{\text{sol}}$ ) also present similar problems of thermocouples embedding fully in the deposit because there is less liquid present. Therefore accurate temperatures are only likely when thermocouples are fully embedded in the deposit a few seconds after the deposit top surface passes by, and where measured temperatures indicate there is a reasonable liquid fraction present (i.e.  $>3\%$ ).

### **7.3 Accuracy of Thermal Imaging Measurements**

Errors in thermal imaging measurements of the deposit top surface temperature arise from: (i) the accuracy of the thermal imaging camera; and (ii) the calibration of thermal intensities into temperatures using embedded thermocouple measurements. The accuracy of the thermal imaging camera is  $\approx 1\%$ . From comparison of runs 32 and 34 in fig 5.12, under nominally similar process parameters the experimental reproducibility is quite good  $\approx 5\%$ , excluding the beginning and end of the runs. However, Grant (1991) from similar experiments on Al alloys determined the experimental reproducibility to be only  $\approx 10\%$ .

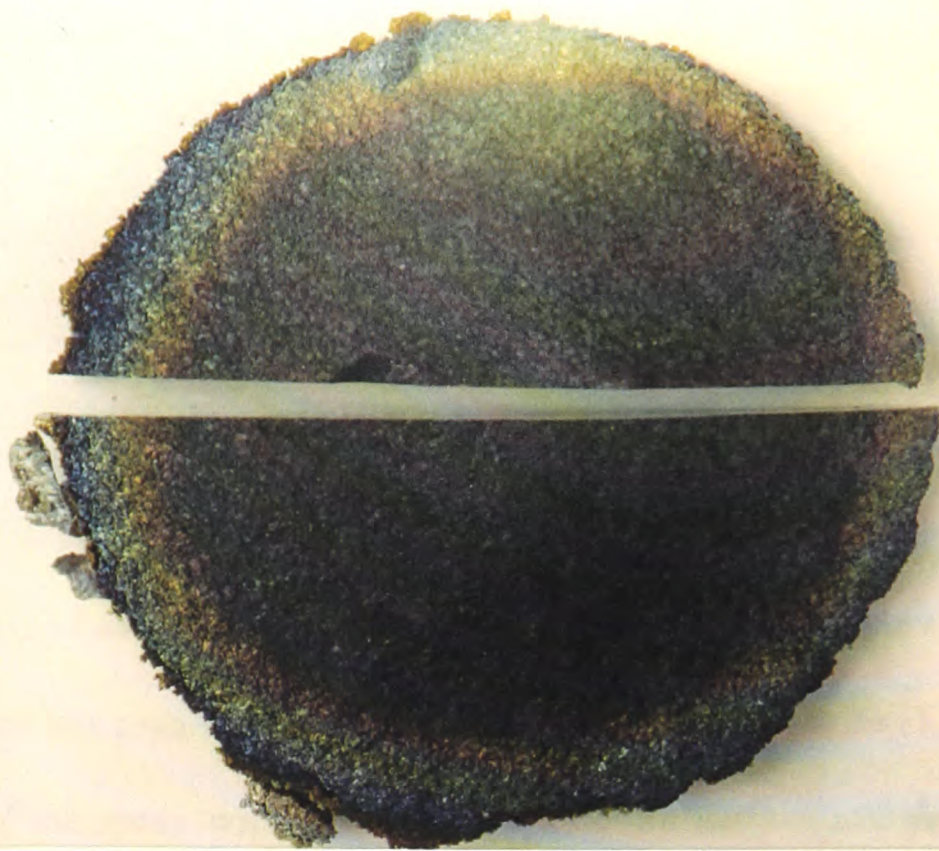


The largest source of error is likely to be from the calibration procedure since embedded thermocouples are not a true measure of surface temperature. This calibration technique also assumes a constant emissivity at all temperatures. This can be a particular problem if the deposit top surface is fully solid and oxidised or excessive liquid is present, both of which cause large changes in emissivity (Grant 1991). Fig 7.2 shows the deposit top surface after spraying for (a) run 27; (b) run 32; and (c) run 37. The extensive blackening of the top surface in the middle of the deposit and the concentric rings of different colours around the edge of the deposit indicates that substantial oxidation occurred. Therefore, the large increase in thermal intensity in the centre of the deposit shown in figs 5.11(c) and 5.12(c) just after the end of spraying is probably due to oxidation.

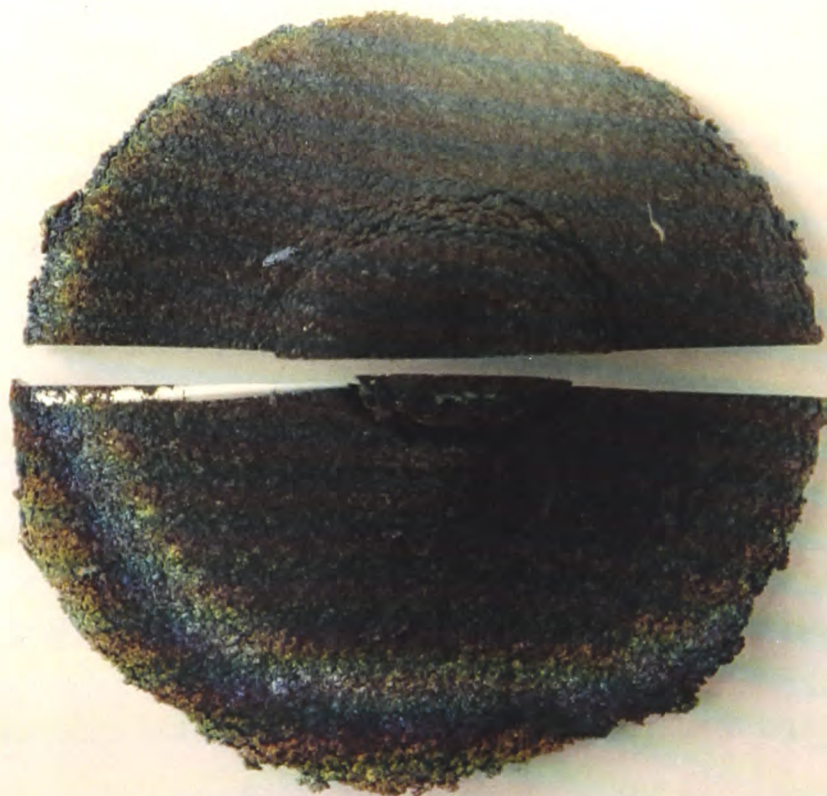
An alternative technique for calibration of thermal intensities used by Grant (1991) and Newbery (1993) involves re-heating small spray formed samples under an Ar atmosphere, through the solid/liquid region. The real top surface temperature as measured by a contact thermocouple on the deposit top surface is then compared with the top surface temperature measured by thermal imaging assuming the experimental emissivity. However, this technique is problematical for high melting point alloys where oxidation occurs (Newbery 1993).

## **7.4 Comparison of Experimental and Modelling Results**

The time spent by the solidifying deposit at high temperatures is very important in determining the final deposit microstructure (Grant 1995, Annavarapu and Doherty 1993 and 1995). This temperature is determined by: (i) the enthalpy content of the spray



(b)



(c)

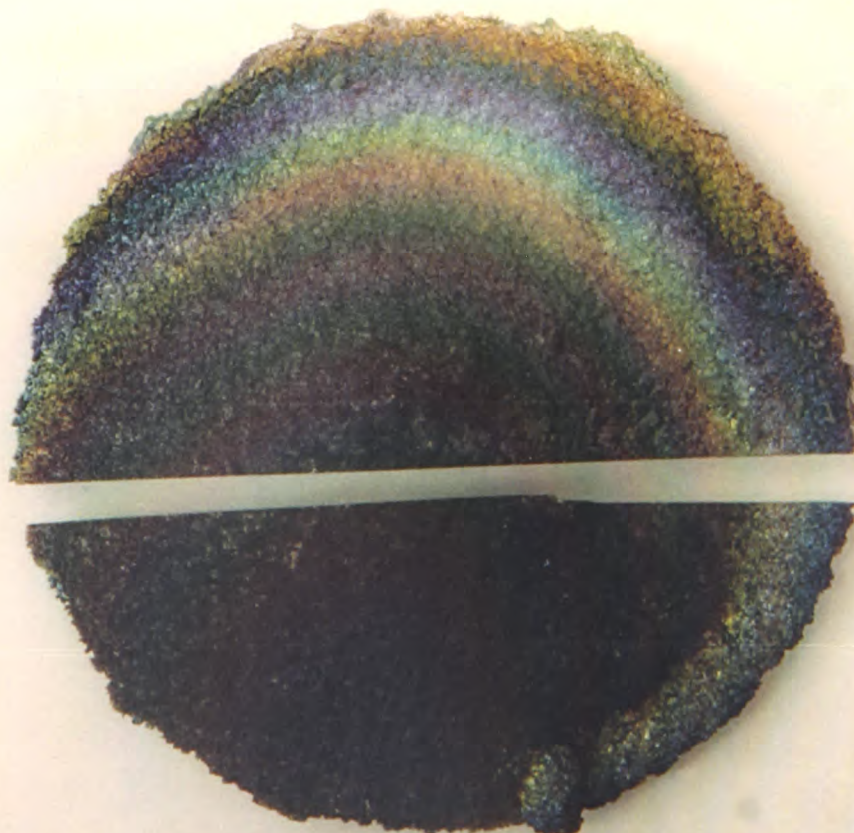


Fig 7.2 The deposit top surface after spray forming for (a) run 27, (b) run 32 and (c) run 37.

arriving at the substrate; (ii) the rate of mass arrival on the substrate; and (iii) the rate of removal of heat by conduction through the substrate and solidifying deposit and convection from the atomising gas on the deposit top surface. Fig 6.19 shows that predicted equilibrated spray temperatures ( $T_{sp}$ ) have consistent variations with (i) gas pressure, (ii) spray distance and (iii) MFR as the measured deposit temperatures. However, predicted  $T_{sp}$  are always less than measured maximum deposit temperature ( $T_{max}$ ) because of: (i) equilibration of the spray with the cooler deposit top surface immediately after deposition and (ii) deposit temperatures are measured just below the top surface once the thermocouple is fully embedded.

Figs 5.15 and 6.19 show the comparison of the measured deposit  $T_{max}$  and predicted  $T_{sp}$  from FLUENT respectively for variations in GFR/gas pressure, spray distance and MFR. Fig 5.15(a) shows the variation of  $T_{max}$  as a function of GFR while fig 6.19(a) shows the variation of  $T_{sp}$  as a function of gas pressure because FLUENT requires gas pressure as an input at the inlet boundary and not GFR. Fig 7.3 shows the experimentally measured relationship between GFR and gas pressure. Because there is an approximate linear relationship between GFR and gas pressure in the range investigated, the trends predicted in figs 6.19(a)-(c) can be directly compared with the measured trends in figs 5.15(a)-(c).

#### **7.4.1 Effect of Gas Flow Rate**

Comparison of figs 5.15(a) and 6.19(a) shows that both the equilibrated spray temperature ( $T_{sp}$ ) and maximum deposit temperature ( $T_{max}$ ) increase as the gas pressure decreases.

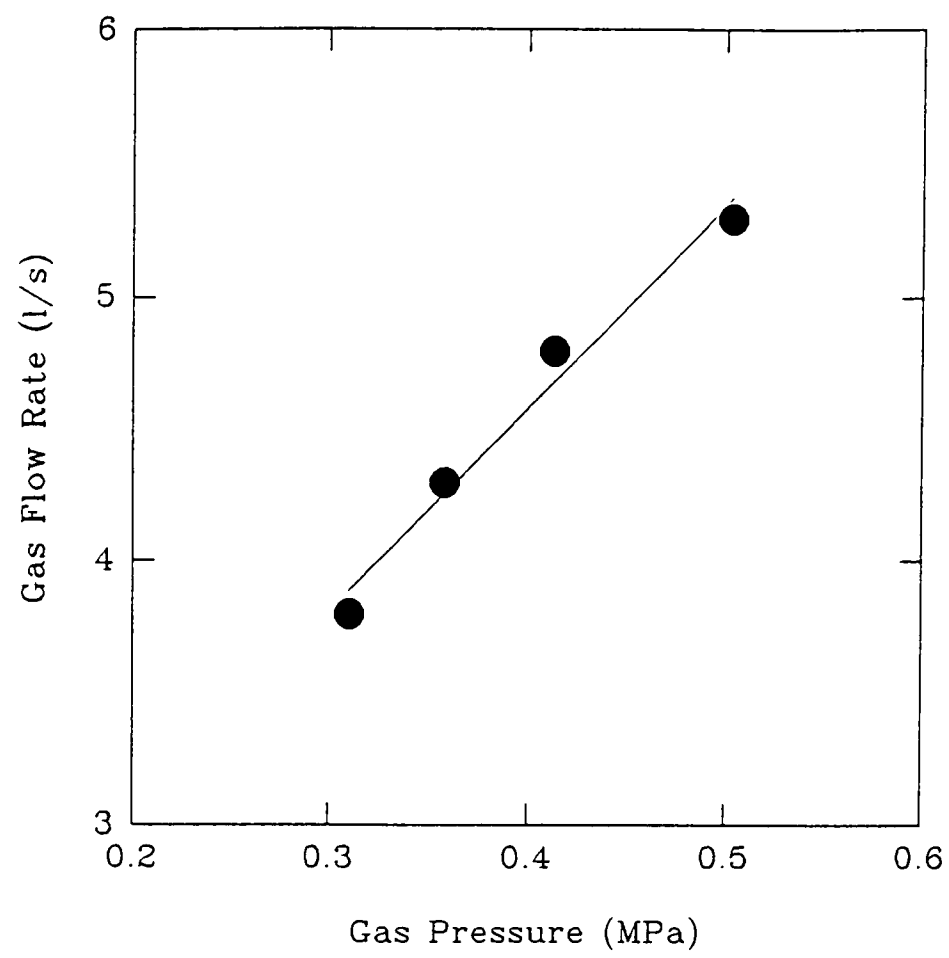


Fig 7.3 Gas flow rate (GFR) against gas pressure measured in the flow meter.



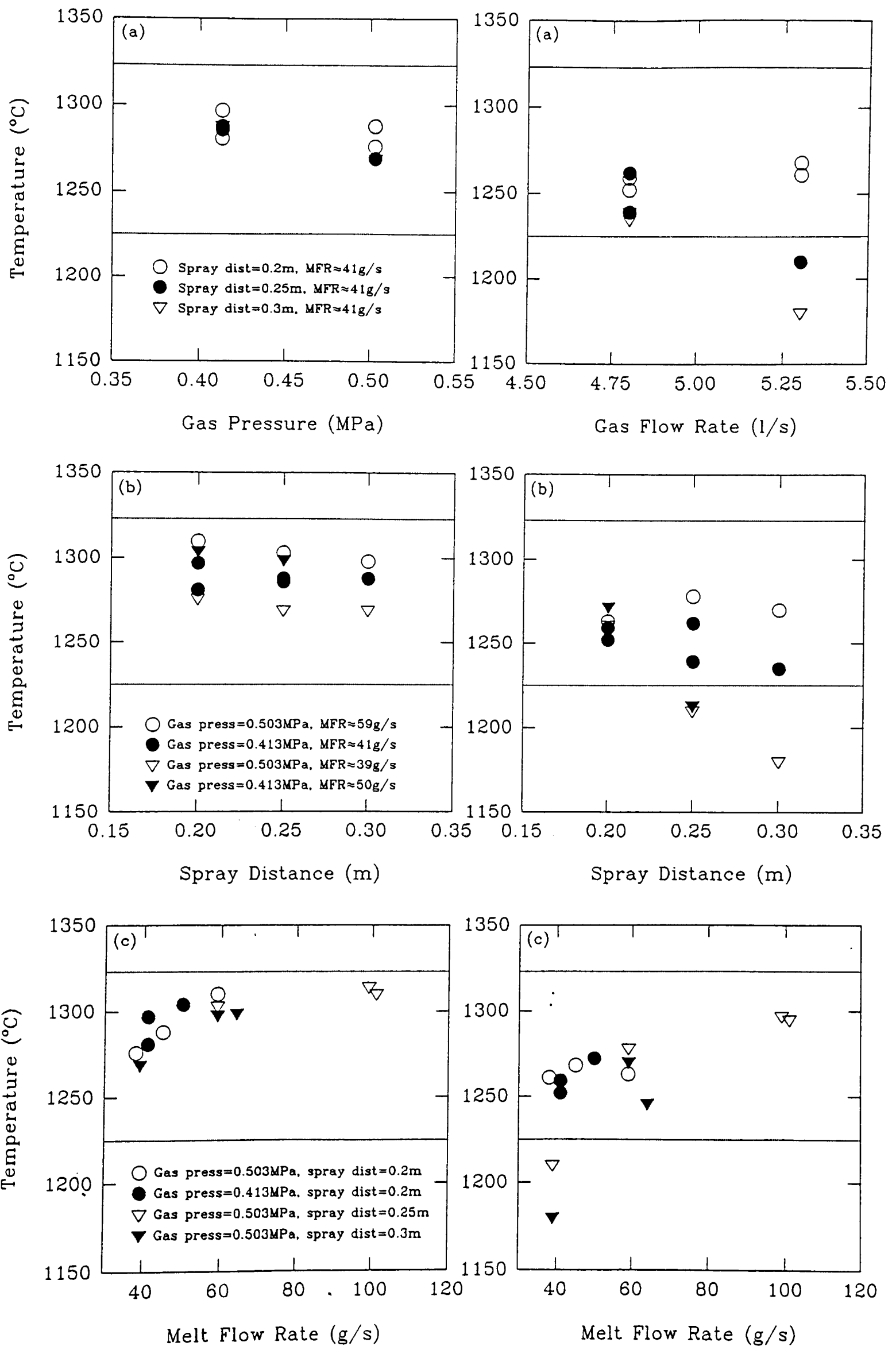


Fig 6.19 Predicted equilibrated spray temperature against: (a) gas pressure, (b) spray distance and (c) melt flow rate.

Fig 5.15 Measured maximum deposit temperature against: (a) gas pressure, (b) spray distance and (c) melt flow rate.

In both figures the dashed horizontal lines indicate the liquidus and solidus temperatures.

An increase in atomising gas pressure leads to: (i) a decrease in the mean droplet size because of more efficient atomisation (equation 7.2), although for the atomiser used in this study this effect is quite small; (ii) the gas velocities generally increase so that individual droplet flight times from the atomiser to the substrate decrease i.e. there is less time for droplet cooling despite higher forced convective heat transfer; and (iii) increased flow of gas over the top surface increases deposit convective cooling. However, for the predicted  $T_{sp}$  it is the relatively small decrease in mean droplet size which is the dominant effect (Grant *et al* 1993A and 1993B). Therefore, it is the shift to smaller, faster cooling droplets which causes the decrease in  $T_{sp}$  as shown in fig 6.19(a). The measured  $T_{max}$  also decreases with increasing gas pressure as shown in fig 5.15(a), but the effect is more marked because of the additional convective cooling of the deposit top surface at higher gas pressures.

Higher gas pressures also reduce the divergence of the gas flow field and droplets as shown in figs 6.15 and 6.22 so that more droplets are collected closer to the centre of the substrate. Therefore, there is a reduction in the standard deviation of the best fit Gaussian curve to the calculated normalised  $M_{ar/a}$  against radial distance from FLUENT and the measured normalised deposit height against radial distance from the real deposits as the gas pressures increases as shown in figs 6.32(a) and (b).

#### **7.4.2 Effect of Spray Distance**

Comparison of figs 5.15(b) and 6.19(b) shows that both  $T_{sp}$  and  $T_{max}$  increase as the spray distance decreases.

An increase in spray distance leads to: (i) an increase in droplet flight times; and

(ii) increased divergence of the spray cone so that the deposition rate at the substrate decreases. Longer droplet flight times at increased spray distances reduce the predicted  $T_{sp}$  as shown in fig 6.19(b) and combine with the progressively lower deposition rates to give a more dramatic decrease in measured  $T_{max}$  as the spray distance increases as shown in fig 5.15(b).

Also, at shorter spray distances more droplets are collected closer to the centre of the substrate due to the decreased divergence of droplets as shown in fig 6.25. Therefore, there is a reduction in the standard deviation of the best fit Gaussian curve to the calculated normalised  $M_{ar/a}$  against radial distance from FLUENT and the measured normalised deposit height against radial distance from the real deposits as the spray distance decreases as shown in figs 6.33(a) and (b).

### **7.4.3 Effect of Melt Flow Rate**

Comparison of figs 5.15(c) and 6.19(c) shows that both  $T_{sp}$  and  $T_{max}$  increase as the MFR increases.

An increase in MFR leads to: (i) an increase in the mean droplet size because of less efficient atomisation (equation 7.2); and (ii) an increase in the deposition rate. The shift to larger, slower cooling droplets causes an increase in  $T_{sp}$  with increasing MFR as shown in fig 6.19(c). The predicted  $T_{sp}$  does not show as great an increase at the highest MFR's ( $\approx 100\text{gs}^{-1}$ ) probably because there is not the expected increase in measured mass median overspray droplet size as shown in fig 5.40, which as suggested above has a major effect on  $T_{sp}$ .  $T_{max}$  continues to increase at the highest MFR ( $\approx 100\text{gs}^{-1}$ ) due to the continued increase in measured deposition rate.

Also, at lower MFR's more droplets are collected closer to the centre of the substrate due to the decreased divergence of droplets as shown in figs 6.18 and 6.28. Therefore, there is a reduction in the standard deviation of the best fit Gaussian curve to the calculated normalised  $M_{ar/a}$  against radial distance from FLUENT and the measured normalised deposit height against radial distance from the real deposits as the MFR decreases as shown in figs 6.34(a) and (b).

#### **7.4.4 Summary of the Effect of Process Parameters**

From an analysis of the complex equations governing droplet dynamic and thermal behaviour during spray forming using a 1-d model of Al-4wt%Cu droplet dynamic and thermal behaviour under the action of a exponentially decaying axial gas velocity (Grant *et al* 1993A and 1993B), Grant and Cantor (1995) proposed the following equation 7.3 below as an approximate expression for the spray solid fraction ( $f_s$ ) as a function of process parameters:

$$f_s = a_1 + a_2 \Delta T + a_3 \frac{v_{gi} z^{2/3}}{M_l^{1/2}} \quad (7.3)$$

where  $f_s$  is the spray solid fraction,  $\Delta T$  is the alloy superheat above the liquidus,  $v_{gi}$  is the initial gas velocity at atomisation,  $z$  is the spray distance,  $M_l$  is the metal mass flow rate (MFR) and  $a_1$ ,  $a_2$  and  $a_3$  are constants related to the physical properties of the alloy and the computer model used to obtain equation 7.3. For Al-4wt%Cu droplets Grant and Cantor (1995) showed best fit  $a_1$ ,  $a_2$  and  $a_3$  values of 0.019,  $-1.4 \times 10^{-3}$  and  $1.3 \times 10^{-3}$  respectively.

Fig 7.4 shows  $f_s$  against  $v_{gi} z^{2/3} / M_l^{1/2}$  for the calculated equilibrated spray solid



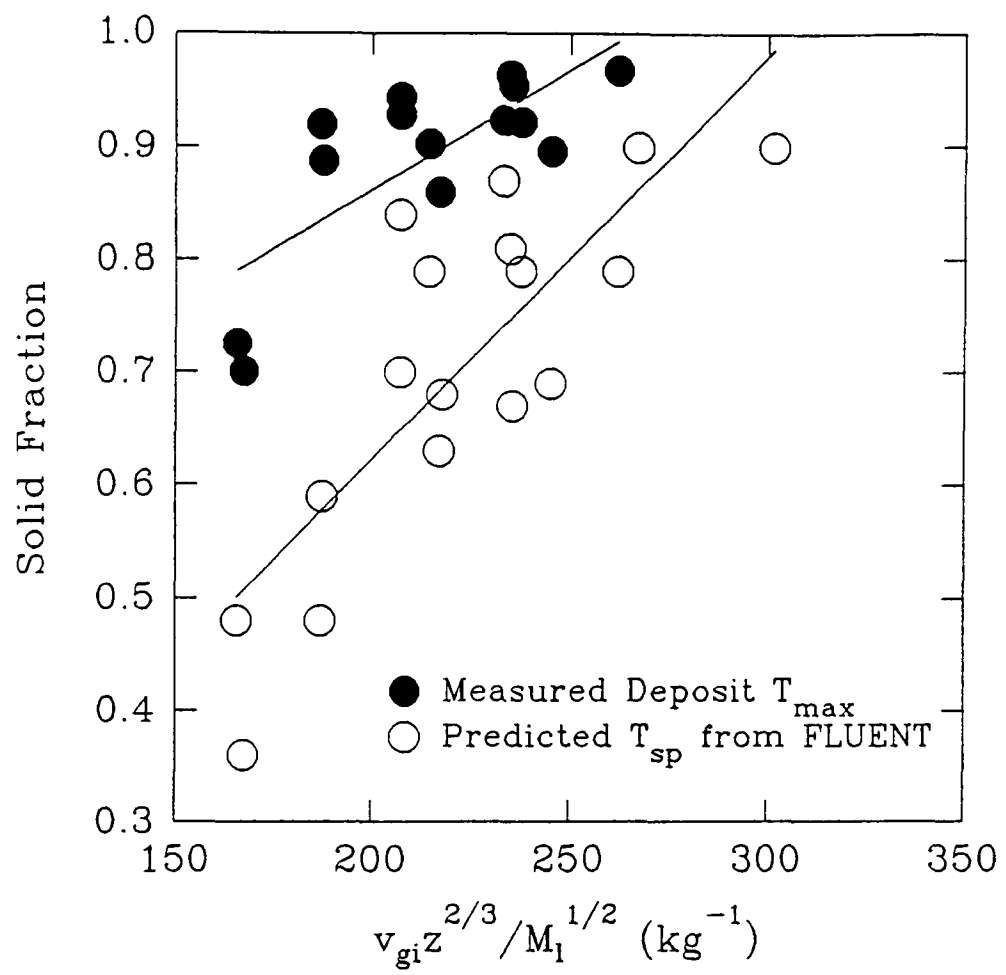


Fig 7.4 Solid fraction against  $v_{gi} z^{2/3} / M_1^{1/2}$  for (a) deposit  $T_{\max}$  temperatures and (b)  $T_{sp}$

fraction from FLUENT and the measured maximum deposit solid fraction from the experimental spray forming runs. In both cases  $v_{gi}$  was obtained from FLUENT because no experimental measurements of gas velocity were available. The solid lines are the best fit straight lines where  $a_1$ ,  $a_2$  and  $a_3$  are 0.12,  $-1.7 \times 10^{-3}$  and  $3.6 \times 10^{-3}$  respectively for FLUENT and 0.38,  $4.6 \times 10^{-4}$  and  $2.1 \times 10^{-3}$  respectively for the spray formed deposits. In both cases the data fit reasonably well to equation 7.3 and the slopes ( $a_3$ ) are similar, although as expected the calculated  $f_s$  from FLUENT is  $\approx 0.15$  lower than the measured maximum deposit solid fraction. Although equation 7.3 appears to be followed, more detailed investigation over a wider range of solid fractions and process parameters is necessary to confirm its validity.

## **7.5 Microstructural Evolution**

The final microstructure of spray formed deposits is determined by a heat balance between (i) the enthalpy of droplets arriving at the substrate, determined by the heat content of the droplets and deposition rate at the substrate and (ii) the heat removal rate, determined by heat conduction through the substrate and deposit and gas cooling of the deposit top surface. This heat balance leads to an equilibrated top surface which persists for between 20s and 45s. The different microstructural regions seen in the spray formed deposits are characterised by either high deposit solid fractions ( $>0.95$ ) throughout deposition or the persistence of lower deposit solid fractions ( $<0.95$ ) for substantial times during deposition.

### **7.5.1 Splat Structures**

When the heat balance leads is such that the heat removal rate is greater than the heat input rate into the deposit a high deposit solid fraction ( $>0.95$ ) is obtained. Droplets impact onto the deposit, spread out and solidify completely before other droplets arrive as shown in fig 5.4. The size and shape of the splats is determined mainly by the size, velocity and temperature of the original droplet. Pre-solidified droplets are not deformed much on impact and retain their spherical shape in the deposit. The absence of liquid in the deposit means that the interstices between the splats are not filled and a relatively high level of intersplat porosity is present in the deposit.

Two types of splat microstructure are possible:

- (i) the splats have a dendritic microstructure as shown in fig 5.4(a). The splats retain some of the dendritic microstructure of the original droplet and solidify quickly in the deposit. The porosity is quite high because there is very little liquid present.
- (ii) the thicker splats have a more equiaxed grain structure while a columnar grain structure develops in thinner splats with the grain boundaries parallel to the splat thickness and the heat flow out of the splat, as shown in figs 5.27 and 5.28. There is slightly more liquid present therefore the porosity is slightly lower. The columnar grains are influenced by the splats below them and the grain boundaries often cross the splats. As the solid fraction reduces, the outline of the splats becomes less well defined and regions of equiaxed grains form as shown in fig 5.29(k).

### **7.5.2 Equiaxed Structures**

When the deposit solid fraction is  $<0.95$  an equiaxed grain structure develops and

the porosity is reduced to very low levels of <1%. A solid/liquid region develops on the deposit top surface with a high nucleation density arising from pre-solidified droplets and broken dendrite arms. The small solid nuclei are surrounded by liquid and grow rapidly. The final grain size ( $d$ ) is determined by the initial grain size ( $d_0$ ), time ( $t$ ) spent in the solid/liquid region and coarsening rate constant ( $K$ ) according to equation 4.1:

$$d^3 = d_0^3 + Kt \quad (4.1)$$

Annavarapu and Doherty (1995) suggested that  $d_0 \approx 5\mu\text{m}$ , although small variations in  $d_0$ , for example, from the droplet size distribution, do not have much effect on the final grain size due to the cubic relationship. Therefore, the final grain size ( $d$ ) is mainly determined by the time ( $t$ ) spent in the solid/liquid region and the coarsening rate constant ( $K$ ) which is temperature and alloy dependent. The time spent in the solid/liquid region is also temperature dependent, since generally the time increases as the temperature increases. Experimental agreement with equation 4.1 using measured values of  $K$  from chapter 4 and  $d_0 = 5\mu\text{m}$  is quite good as shown in fig 5.36, although predicted grain sizes are  $\approx 15\mu\text{m}$  lower than measured grain sizes. This may be because the measured coarsening rate constants ( $K$ ) from the re-heating experiments in chapter 4 are lower than on the deposit top surface during spray forming, because as shown in fig 5.36 an assumed constant value of  $K = 10^{-15} \text{m}^3/\text{s}$  and  $d_0 = 5\mu\text{m}$  gives excellent agreement with measured grain sizes.

Annavarapu and Doherty (1995) suggested that, in general, coarsening rates are lower than expected from studies of conventional processes (Flemings 1974) or LPS (liquid phase sintering) (Kang and Yoon 1982 and Niemi and Courtney 1981) because of inhibition provided by second phase particles such as carbide particles formed late in solidification or gas filled pores present in all spray formed deposits. For several spray



formed alloys, the range of solid fractions for successful spraying of equiaxed grain deposits is 0.7-0.95 (Annavarapu and Doherty 1995, Grant 1991). As shown in fig 4.13 this gives coarsening rate constants (K) ranging from  $10 \times 10^{-18}$  -  $400 \times 10^{-18} \text{m}^3 \text{s}^{-1}$  for several different alloys. Numerical modelling calculations and experimental measurements indicate that the time spent in the solid/liquid region is of the order of 1-2mins (Mathur *et al* 1989, Grant *et al* 1993A and 1993B and Grant *et al* 1989), although slightly lower solidification times of 20-45s were measured in this study. Examination of the possible range of combinations of K and t indicate that there is only a small range of obtainable as-sprayed grain sizes (10-15 $\mu\text{m}$ ) for a particular alloy, in agreement with the observed limited range of spray formed grain sizes (Mathur *et al* 1989, Grant 1991) and general agreement within the spray forming community that it is difficult to change the as-sprayed grain size (Proceedings of ICSF2 1993).

The porosity of the UDIMET 720 deposits in this study remained low (<1%) at temperatures up to 1297°C. However, at higher temperatures the porosity is likely to increase again (Grant 1991, Underhill 1992) due to the whipping up of excess liquid on the deposit top surface creating entrapped gas porosity.

Fig 7.5 is a schematic diagram of the change in deposit microstructure from dendritic splat to equiaxed splat to equiaxed grain structure as the solid fraction decreases.

### **7.5.3 Through Thickness Microstructure**

The microstructure in regions II, III and IV described in 5.3.5 can be rationalised on the same basis as the splat and equiaxed grain structures described above. The microstructure in region II is always of the equiaxed splat type. The first droplets to splat

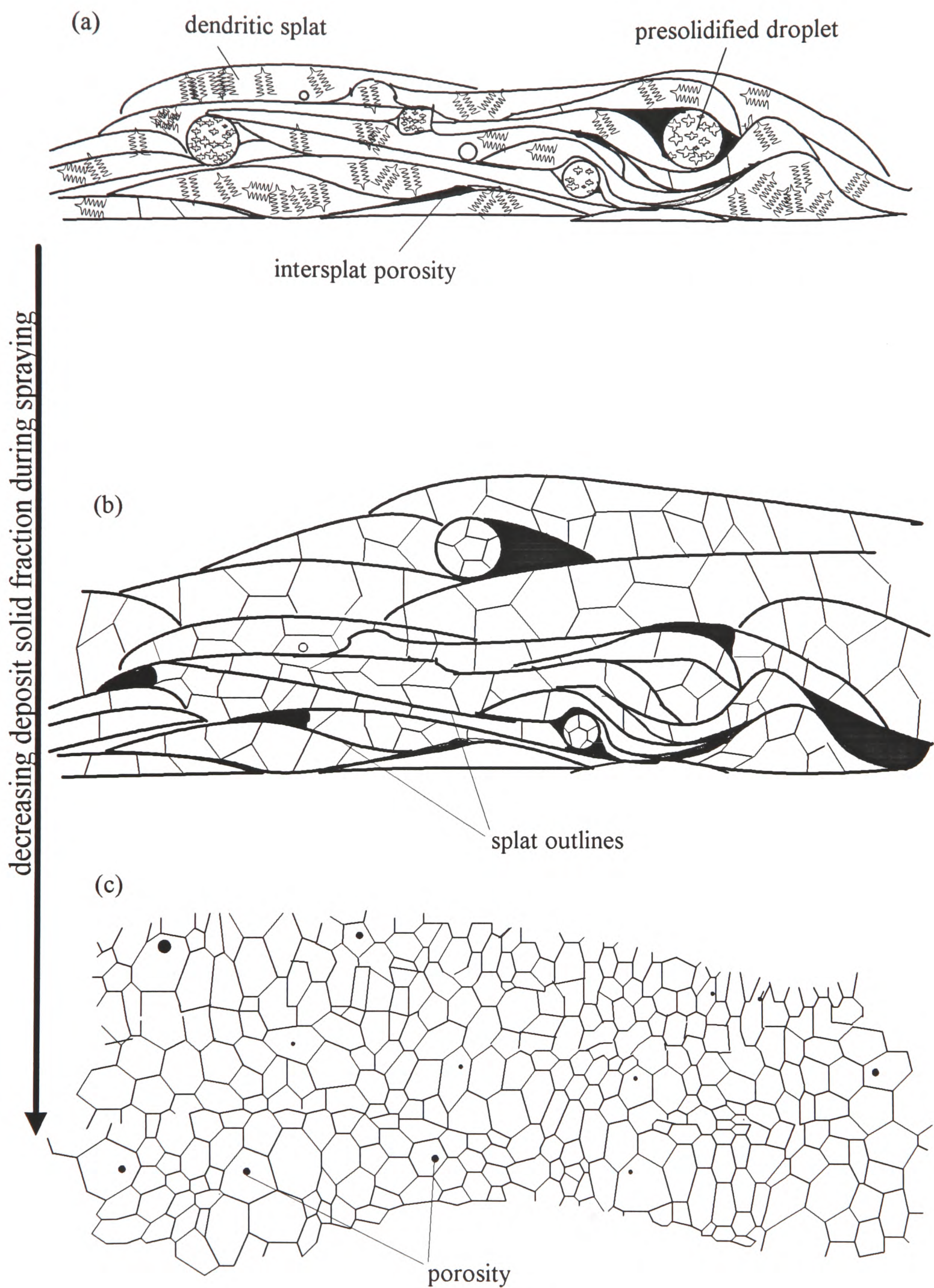


Fig 7.5 Schematic diagram of the change from (a) dendritic splat to (b) equiaxed splat to (c) equiaxed grain structure as the deposit solid fraction decreases.

are cooled efficiently by the substrate and are therefore fully solid before the next droplets arrive. As the deposit builds up the temperature increases, steady state conditions are approached and the solid fraction is either  $<0.95$ , to give an equiaxed grain structure or  $>0.95$  to give a splat structure and region III starts. In region III the microstructure is either a splat or equiaxed structure depending on the solid fraction. In this region the grain size is approximately constant for half or three quarters of the way through the deposit thickness and then decreases near the top surface. This decrease is probably due to more efficient gas cooling of the deposit top surface near the end of the run, which reduces the temperature and time spent in the solid/liquid region and so inhibits grain growth. In region IV, the greater gas cooling can increase the heat removal rate so that a splat structure may again form as shown in fig 5.24(b).

#### **7.5.4 Contraction Cracks**

Solidification defects such as contraction cracks are commonly observed in conventional castings under certain conditions, although they are not usually found in spray formed deposits. However, figs 5.38(a)-(c) clearly show that contraction cracks were present in spray formed deposits that had a liquid fraction  $>\approx 5\%$  ( $T_{\max} > \approx 1250^{\circ}\text{C}$ ). UDIMET 720 has a long freezing range of  $\approx 100^{\circ}\text{C}$  which makes it more susceptible to contraction cracking when the atomising gas efficiently cools the deposit top surface which then solidifies before the interior regions are fully solid. This leaves isolated liquid regions in the deposit which are subjected to hydrostatic shrinkage stresses on cooling and cause cracking.

## **7.6 Summary**

The overspray powder distributions fit reasonably well to the Lubanska correlation with  $k=56.8$ . Predicted spray temperatures at the substrate are consistent with measured maximum deposit temperatures under similar conditions and decrease with: (i) increasing gas pressure; (ii) increasing spray distance; and (iii) decreasing MFR. Measured maximum deposit temperatures were always below predicted spray temperatures because of spray equilibration with the cooler deposit top surface and delays for full thermocouple embedding. The UDIMET 720 deposit microstructure consists of either droplet splats or equiaxed grains, depending on the deposit solid fraction determined by a heat balance between the heat arrival rate from the droplet spray and the heat removal rate through the substrate and the atomising gas. If the deposit solid fraction is  $>0.95$  then the microstructure consists of droplet splats and if the deposit solid fraction is  $<0.95$  then the microstructure consists of equiaxed grains. The coarsening of grains in solidifying spray formed deposits follows a cubic rate law as given in equation 4.1.



## **CHAPTER 8 - CONCLUSIONS**

Samples of spray formed MAR-M-002 and UDIMET 720 have been heat treated in the solid/liquid region at solid fractions ( $f$ ) from 0.78-0.98 and in the fully solid region for times up to  $10^4$ s. MAR-M-002 shows virtually no increase in grain size when heat treated in the fully solid region. However, UDIMET 720 shows a steady increase in grain size when heat treated in the fully solid region above the  $\gamma'$  solvus temperature. When heat treated in the solid/liquid region there is an initial rapid increase in the grain size for both MAR-M-002 and UDIMET 720. Equation 4.1 below is a good fit to the grain growth data for MAR-M-002 and UDIMET 720 in the solid/liquid region:

$$d^3 = d_0^3 + Kt \quad (4.1)$$

where  $d$  is the grain size at time,  $t$ ,  $d_0$  is the initial grain size and  $K$  is a coarsening rate constant. The coarsening rate constants ( $K$ ) for MAR-M-002 and UDIMET 720 increase with decreasing solid fraction which is against the trend for LPS (liquid phase sintering) and lower than coarsening rate constants calculated from conventional processes. The presence of fine grain boundary carbide particles and/or gas filled pores may account for this discrepancy (Annavarapu and Doherty 1995).

Spray formed deposits with a range of microstructures from relatively high porosity (2-4%) splat structures to low porosity (<1%) equiaxed grain structures have been manufactured. Embedded thermocouple and infra-red thermal imaging measurements have been used to correlate the microstructure with the deposit temperature during spray forming. The transition from high porosity splat to low porosity equiaxed grain structures is observed to occur above a temperature of  $\approx 1250^\circ\text{C}$  ( $f_s > 0.95$ ) and deposition rate of

$\approx 20\text{g/s}$ . The transition in terms of temperature is more reliable. However, temperatures are difficult to obtain when spray forming real components and deposition rate is a reasonable indicator of relative deposit temperature. Grain size increases with increasing temperature and solidification time. There is reasonable agreement between measured and predicted grain size from equation (4.1) using previously measured values of the coarsening rate constant ( $K$ ), an assumed  $d_0 = 5\mu\text{m}$  and the measured solidification time in the solid/liquid region from embedded thermocouples. However, excellent agreement between measured and predicted grain sizes are obtained if  $K$  is assumed constant at  $10^{-15}\text{m}^3/\text{s}$  which indicates that the measured  $K$  values may be less than they actually were during solidification of the deposit. The measured maximum deposit temperature from embedded thermocouple measurements increases with: (i) decreasing gas flow rate; (ii) decreasing spray distance; (iii) increasing melt flow rate; and (iv) increasing deposition rate. The mass median size of overspray powder increases with increasing MFR:GFR and fits reasonably well to the Lubanska correlation with a best fit  $k=56.8$ . Contraction cracks similar to those observed in conventional castings were observed in deposits with a maximum deposit temperature  $>\approx 1250^\circ\text{C}$  i.e.  $>\approx 5\%$  liquid fraction.

A commercial fluid dynamics software package, FLUENT has been used to analyse the dynamic and thermal behaviour of gas and droplets in spray forming under a range of process conditions. An important limitation of the model is that it does not directly model the atomisation process, so droplets must be injected into the gas flow and model results are dependent on the details of the droplet injection. Nonetheless predicted spray temperatures at the substrate are consistent with measured deposit temperatures

under similar conditions and increase with: (i) decreasing gas pressure; (ii) decreasing spray distance; and (iii) increasing MFR.

The predicted maximum axial gas velocity of the single phase gas flow field from FLUENT increases with increasing gas pressure and the axial gas velocity decreases approximately exponentially with axial distance. The predicted axial gas velocity from FLUENT decreases rapidly on impacting the droplets. The axial gas velocity is higher at: (i) greater gas pressure and (ii) lower MFR's. The radial gas velocity (divergence) of the two phase gas flow field increases as the: (i) gas pressure decreases and (ii) MFR increases.

The mass median droplet sizes for the atomiser employed are quite large (115-157 $\mu\text{m}$ ), which cool slowly and predicted droplet temperatures at the substrate are not very dependent on gas pressure or melt flow rate and differences in spray temperature at the substrate are mainly due to relatively small changes in the overall droplet size distribution.

The predicted normalised mass arrival rate/area against radial distance from FLUENT agree reasonably well with the measured normalised deposit height against radial distance. In both cases profiles fit reasonably well to a Gaussian distribution and the best fit standard deviation decreases with: (i) increasing gas pressure; (ii) decreasing spray distance; and (iii) decreasing MFR, although the predicted standard deviations from FLUENT are more variable.

The predicted equilibrated spray solid fraction from FLUENT and measured maximum deposit solid fraction agree reasonably well with equation 7.3 developed by Grant and Cantor (1995) for the variation of spray solid fraction with process parameters

from an analysis of the complex equations governing droplet dynamic and thermal behaviour during spray forming:

$$f_s = a_1 + a_2 \Delta T + a_3 \frac{v_{gi} z^{2/3}}{M_l^{1/2}} \quad (7.3)$$

where  $f_s$  is the spray solid fraction  $\Delta T$  is the melt superheat,  $v_{gi}$  is the initial gas velocity,  $z$  is the spray distance,  $M_l$  is the melt mass flow rate and  $a_1, a_2$  and  $a_3$  are constants.



## **CHAPTER 9 - SUGGESTIONS FOR FURTHER WORK**

There is still a need for more detailed investigation into the process of grain growth in the solid/liquid region for a range of different alloys. In particular transmission electron microscopy (TEM) and electron probe microanalysis (EPMA) should yield valuable information about the role of second phase particles and the segregation of elements.

Improvements are needed in the melting arrangement to include a vacuum melting system and a professionally designed nozzle and atomiser. This should improve the reliability, reduce the MFR:GFR ratio and therefore the droplet mass median diameter to approach values used in industry.

The use of embedded thermocouples has provided valuable information of the temperature and solidification times of spray formed deposits. This information would be very useful for other spray formed alloys and would help validate equations (4.1) and (7.3).

There is a need to reduce the sensitivity of the FLUENT model to droplet injection conditions and to be able to use different solidification models.

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## APPENDIX A

The generalised conservation equation 6.1 can be expressed in 2-dimensional axisymmetric form as:

$$\frac{\partial}{\partial x}(\rho_g u_g \Phi) + \frac{1}{r} \frac{\partial}{\partial r}(\rho_g r v_g \Phi) - \frac{\partial}{\partial x} \left( \Gamma \frac{\partial \Phi}{\partial x} \right) - \frac{1}{r} \frac{\partial}{\partial r} \left( r \Gamma \frac{\partial \Phi}{\partial r} \right) = S_\Phi - S_{\Phi d} \quad (\text{A.1})$$

the source terms  $S_\Phi$ ,  $S_{\Phi d}$  and effective viscosity or thermal diffusivity terms ( $\Gamma$ ) for the dependent variables ( $\Phi$ ) of axial gas velocity ( $u_g$ ), radial gas velocity ( $v_g$ ), enthalpy ( $H$ ), turbulent kinetic energy ( $k$ ) and it's dissipation ( $\epsilon$ ) are given in table A.1 below (Fritsching *et al* 1992):

| $\Phi$     | $S_\Phi$                                                                                                                                                                                                                                              | $S_{\Phi d}$ | $\Gamma$                           |
|------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------|------------------------------------|
| 1          | -                                                                                                                                                                                                                                                     | -            | -                                  |
| $u_g$      | $\frac{\partial}{\partial x} \left( \Gamma \frac{\partial u_g}{\partial x} \right) + \frac{1}{r} \frac{\partial}{\partial r} \left( r \Gamma \frac{\partial v_g}{\partial x} \right) - \frac{\partial \rho_g}{\partial x}$                            | $S_{x,d}$    | $\mu_{\text{eff}}$                 |
| $v_g$      | $\frac{\partial}{\partial x} \left( \Gamma \frac{\partial u_g}{\partial r} \right) + \frac{1}{r} \frac{\partial}{\partial r} \left( r \Gamma \frac{\partial v_g}{\partial x} \right) - \frac{\partial \rho_g}{\partial x} - 2 \Gamma \frac{v_g}{r^2}$ | $S_{r,d}$    | $\mu_{\text{eff}}$                 |
| $H$        | -                                                                                                                                                                                                                                                     | $S_H$        | $\mu_{\text{eff}}/\sigma_H$        |
| $k$        | $G_k - \rho_g \epsilon$                                                                                                                                                                                                                               | -            | $\mu_{\text{eff}}/\sigma_k$        |
| $\epsilon$ | $\epsilon/k(C_1 G_k - C_2 \rho_g \epsilon)$                                                                                                                                                                                                           | -            | $\mu_{\text{eff}}/\sigma_\epsilon$ |

Table A.1 Source terms  $S_\Phi$ ,  $S_{\Phi d}$  and effective viscosity ( $\Gamma$ ) for the dependent variables ( $\Phi$ ) of axial gas velocity ( $u_g$ ), radial gas velocity ( $v_g$ ), enthalpy ( $H$ ), turbulent kinetic energy ( $k$ ) and it's dissipation ( $\epsilon$ ) where  $u_g$  is the axial gas velocity,  $v_g$  is the radial gas velocity,  $\rho_g$  is the gas density and:

$$G_k = \mu_{\text{eff}} \left\{ 2 \left[ \left( \frac{\partial u_g}{\partial x} \right)^2 + \left( \frac{\partial v_g}{\partial x} \right)^2 + \left( \frac{v_g}{r} \right)^2 \right] + \left( \frac{\partial u_g}{\partial r} + \frac{\partial v_g}{\partial r} \right)^2 \right\} \quad (\text{A.2})$$

and



$$\mu_{\text{eff}} = C_{\mu} \rho_g \frac{k^2}{\varepsilon} \quad (\text{A.3})$$

the coefficients  $C_1$ ,  $C_2$ ,  $C_{\mu}$ ,  $\sigma_k$ ,  $\sigma_{\varepsilon}$ , and  $\sigma_H$  are empirical constants in the  $k$ - $\varepsilon$  model of turbulence (Launder and Spalding 1974) which have the following empirical derived values:

$$C_1=1.44 \quad C_2=1.92 \quad C_{\mu}=0.09 \quad \sigma_k=1.0 \quad \sigma_{\varepsilon}=1.3 \quad \sigma_H=0.7$$

The source terms for momentum coupling to the dispersed phase  $S_{x,d}$  and  $S_{r,d}$  include the gravitational acceleration and external body forces from interaction with the dispersed phase. Using a force balance to equate the droplet inertia with the forces acting on the particle for the axial direction:

$$\frac{du_d}{dt} = F_D(u_g - u_d) + \frac{g_x(\rho_d - \rho_g)}{\rho_d} \quad (\text{A.4})$$

where  $u_d$  is the droplet axial velocity,  $g_x$  is the gravitational force in the axial direction,  $\rho_d$  is the droplet density and  $F_D(u_g - u_d)$  is the drag force per unit particle mass where:

$$F_D = \frac{18\mu}{\rho_d D_d^2} \frac{C_D \text{Re}}{24} \quad (\text{A.5})$$

where  $\mu$  is the molecular viscosity of the gas,  $D_d$  is the droplet diameter,  $\text{Re}$  is the relative Reynolds number, defined as:

$$\text{Re} = \frac{\rho D_d |u_d - u_g|}{\mu} \quad (\text{A.6})$$

and the drag coefficient  $C_D$  is a function of the relative Reynolds number of the general form:

$$C_D = a_1 + a_2/\text{Re} + a_3/\text{Re}^2 \quad (\text{A.7})$$

where  $a_1$ ,  $a_2$  and  $a_3$  are constants which apply over several ranges of  $\text{Re}$  given by Morsi and Alexander (1972). Integration of equation A.4 gives the droplet velocity at each point along the trajectory and the trajectory itself from:

$$dx/dt = u_d \quad (\text{A.8})$$

The source term  $S_H$  for enthalpy coupling with the dispersed phase includes the exchange of heat with the dispersed phase. Again a simple heat balance relates the droplet temperature ( $T_d$ ) to the convective heat transfer at the droplet surface (Fluent manual 1993):

$$m_d c_{eq} \frac{dT_d}{dt} = h SA (T_g - T_d) \quad (\text{A.9})$$

where  $m_d$  is the droplet mass,  $c_{eq}$  is the equivalent droplet heat capacity, modified as shown in equation 6.2 to include the droplet latent heat,  $SA$  is the droplet surface area,  $T_g$  is the local gas temperature and  $h$  is the convective heat transfer coefficient. Equation A.9 assumes Newtonian conditions within the droplet i.e. uniform temperature. The heat transfer coefficient ( $h$ ) is evaluated using the Ranz-Marshall correlation (1952A and 1952B):

$$Nu = \frac{hD_d}{k_g} = 2 + 0.6Re^{\frac{1}{2}}Pr^{\frac{1}{3}} \quad (\text{A.10})$$

where  $D_d$  is the droplet diameter,  $k_g$  is the thermal conductivity of the gas phase,  $Pr$  is the Prandtl number of the gas and  $Re$  is the Reynolds number based on the droplet diameter and relative velocity given by equation A.6. The heat lost by the droplet as it traverses each computational cell appears as a source of heat ( $S_H$ ) in subsequent calculations of the energy conservation equation.

## APPENDIX B

FORTRAN program written by Dr. PS Grant to create particle history files for post processing analysis of droplet dynamic and thermal behaviour in FLUENT.

**program bat**

character file\*8,ctho\*1,chun\*1,cten\*1,cone\*1,com\*20

open (unit=1,file='track\_ni.bat',status='unknown')

open (unit=2,file='del.bat',status='unknown')

n = 0

write (1,\*) 'rcf'

write (1,\*) 'rdf'

write (1,\*) 'op wh'

do i = 1,420

! total number of droplets

do j = 1,3

! number of stochastic tracks

n = n + 1

ntho = int(n/1000)

nhun = int(n/100)-(ntho\*10)

nten = int(n/10)-(ntho\*100)-(nhun\*10)

none = int(n)-(ntho\*1000)-(nhun\*100)-(nten\*10)

ctho = char(48+ntho)

chun = char(48+nhun)

cten = char(48+nten)

cone = char(48+none)

if (ntho.gt.0) then

file = ctho//chun//cten//cone//'.PAR'

else if (nhun.gt.0) then

file = chun//cten//cone//'.PAR'

else if (nten.gt.0) then

file = cten//cone//'.PAR'

else

file = cone//'.PAR'

end if

write (1,\*) 'pa'

! particle

write (1,\*) 'y'

! yes

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        write (1,10) i           ! injection number
        write (1,*) '3'         ! number of stochastic tracks
        write (1,10) j         ! which one to save
        write (1,*) '1'         ! timesteps between saves
        write (1,*) file        ! filename

        com  = 'rm '//file
        write (2,*) com

    end do
end do

write (1,*) 'q'
write (1,*) 'q'
write (1,*) 'q'
write (1,*) 'y'

10    format (i4)

stop
end

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FORTTRAN program written by Dr. PS Grant to read the above particle history files and calculate how much mass and enthalpy passes through a radial slice at a range of axial distances. The program has been modified to calculate the equilibrated spray solid fraction at the substrate.

#### **program impact**

- c Takes particles history files created by FLUENT and reads them in to
- c sort how much mass, heat and solid fraction is passing a radial slice
- c through the trajectories at a range of axial distances
- c
- c Injection particle size distributions must be the same at every injection
- c position
- c
- c Maximum number of droplets = 9999

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real s(3000),x(3000),y(3000),u(3000),
+   v(3000),T(3000),Md(0:1260),d(1260),
+   Pd(1260),d1(1260),P1(1260),H(3000),fs(3000),
+   TK(20),Cp(20),f(20),E(20,10),W(20,10),G(20,10),
+   xb(10),lb(20),ub(20),yb(20),a(3000,20),

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+    ff(20,10),TT(20,10),Ht(20),cum(0:1260),
+    sumEW(10),sumW(10),avE(10),avff(10)

real mass,dm,dmin,dmax,p,flow,xmin,xmax,ymin,ymax

integer np,nt,nx,nb,ni,ns,nz,n

character input*8,ctho*1,chun*1,cten*1,cone*1,str*14

data E,W,Ht,ff,G,sumEW,sumW,avff,avE/860*0.0/

np      = 100          ! no. particles per injection set
nt      = 3            ! no. stochastic tracks
ns      = 1            ! no. injection sets
nx      = 6            ! no. axial distance
nb      = 4            ! no. radial bins
xmin    = 0.05         ! min axial distance (m)
xmax    = 0.24         ! max axial distance (m)
ymin    = 0.0          ! min radial distance (m)
ymax    = 0.055        ! max radial distance (m)
p       = 1.52         ! spread parameter
dm      = 183.0e-6     ! mean diameter (m)
dmin    = 10.0e-6      ! min diameter (m)
dmax    = 700.0e-6     ! max diameter (m)
flow    = 0.00939      ! total melt mass flow rate (kg/s/radians)

open (unit=1,file='specific.txt',status='old') ! Cp=f(T)
  read (1,*) (TK(i),Cp(i),f(i),i=1,20)
close (1)

do i = 2,20
  Ht(i) = Ht(i-1) + (Cp(i)*(TK(i)-TK(i-1)))
end do

open (unit=7,file='specific.out',status='unknown')
  write (7,'(4(1x,e10.4))') (TK(i),Cp(i),f(i),Ht(i),i=1,20)
close (7)

xinc    = (xmax-xmin)/(nx-1)
yinc    = (ymax-ymin)/nb
dinc    = (dmax-dmin)/np

do k    = 1,nx          ! axial bin distances
  xb(k) = xmin + (xinc*(k-1))
end do

do j    = 1,nb
  yb(j) = (yinc/2.0) + (yinc*(j-1))          ! radial bin midpoints

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    lb(j) = yb(j)-(yinc/2.0)           ! bin lower bound
    ub(j) = yb(j)+(yinc/2.0)           ! bin upper bound
end do

do i = 1,np                           ! calculate the
    d(i) = dmin + (dinc/2.0) + (dinc*(i-1)) ! Rosin-Rammler distribution
    Md(i) = 1.0-exp(-((d(i)/dm)**p))
end do

Md(0) = 0.0

do i = 1,np                           ! normalise total area of
    Md(i) = (Md(i)/Md(np))             ! Rosin-Rammler to unity
    Pd(i) = (Md(i)-Md(i-1))
end do

do j = 1,ns                           ! extrapolate particle size to
    do i = 1,np*nt                     ! cover np*nt*ns particles
        ni = (int((i-1)/nt)) + 1
        nz = np*nt*(j-1)
        P1(nz+i) = Pd(ni)/(nt*ns)
        dl(nz+i) = d(ni)
    end do
end do

cum (0) = 0.0

do i = 1,np*nt*ns
    cum(i) = cum(i-1) + P1(i)
end do

if (abs(cum(np*nt*ns)-1.0).gt.1e-3) write (*,500) cum(np*nt*ns)
500 format (2x,'WARNING. Total probability = ',e10.4)

do i = 1,np*nt*ns

    ntho = int(i/1000)
    nhun = int(i/100)-(ntho*10)
    nten = int(i/10)-(ntho*100)-(nhun*10)
    none = int(i)-(ntho*1000)-(nhun*100)-(nten*10)

    ctho = char(48+ntho)
    chun = char(48+nhun)
    cten = char(48+nten)
    cone = char(48+none)

    if (ntho.gt.0) then
        input = ctho//chun//cten//cone//'.PAR'
    end if
end do

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else if (nhun.gt.0) then
  input = chun//cten//cone//'.PAR'
else if (nten.gt.0) then
  input = cten//cone//'.PAR'
else
  input = cone//'.PAR'
end if

open (unit=2,file=input,status='old')

read (2,10) str

n      = 1
k      = 1

100  read (2,*,err=110) s(n),x(n),y(n),u(n),v(n),T(n)

if (x(n).ge.xb(k)) then
  a(i,k)      = y(n)          ! is axial distance just > xb
  do j        = 1,nb
    if (y(n).ge.lb(j).and.y(n).lt.ub(j)) then ! which radial bin
      W(j,k)   = W(j,k) + (P1(i)*flow)      ! kg/radian/s
      call fraction (T,TK,f,fs,n)
      call enthalpy (T,Tk,Ht,H,n)
      E(j,k)   = E(j,k) + (H(n)*P1(i)*flow) !H=J/kg, flow=kg/rad.s
      !E=J/rad.s
    end if
  end do
  if (k.lt.nx) then          ! next axial distance
    k = k + 1
  else if (k.ge.nx) then
    goto 110
  end if
end if

n      = n + 1

goto 100

110  close (2)

end do

do j    = 1,nb
  do k  = 1,nx
    if (W(j,k).gt.0.0) then
      G(j,k)   = (W(j,k)*2*3.14)/(3.14*(ub(j)**2.0-lb(j)**2.0)) !G=kg/s.m^2
      E(j,k)   = E(j,k)/W(j,k)                                !E=J/kg.rad.s
      call average (E,ff,TT,Ht,TK,f,j,k)
    end if
  end do
end do

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        end if
    end do
end do

do k =1,nx
    do j =1,nb
sumEW(k)=sumEW(k) + E(j,k)*W(j,k)
    end do
end do
do k =1,nx
    do j =1,nb
sumW(k)=sumW(k) + W(j,k)
    end do
end do
do k =1,nx
avE(k) =sumEW(k)/sumW(k)
end do

do k      = 1,nx
do i      = 2,20
if (avE(k).gt.Ht(i-1).and.avE(k).le.Ht(i)) then
    avff(k)      = (((f(i)-f(i-1))/(Ht(i)-Ht(i-1))))*
+                (avE(k)-Ht(i-1))) + f(i-1)
end if
end do
end do

open      (unit=3,file='imp3-1 a.25.dat',status='unknown')
do i = 1,np
    write (3,50) d(i),Pd(i),Md(i)
end do
close      (3)

open      (unit=4,file='imp4-1 a.25.dat',status='unknown')
do i      = 1,np*nt*ns
    write (4,70) dl(i),(a(i,k),k=1,nx)
end do
close      (4)

open      (unit=5,file='imp5-1 a.25.dat',status='unknown')
do j      = 1,nb,1
    write (5,70) yb(j),(W(j,k),k=1,nx)
end do
write      (5,80)
do j      = 1,nb,1
    write (5,70) yb(j),(E(j,k),k=1,nx)
end do
write      (5,80)

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write (5,75) avE(1),avE(2),avE(3),avE(4),avE(5),avE(6)
write (5,80)
do j = 1,nb,1
  write (5,70) yb(j),(ff(j,k),k=1,nx)
end do
write (5,80)
write (5,78) avff(1),avff(2),avff(3),avff(4),avff(5),avff(6)
write (5,80)
do j = 1,nb,1
  write (5,70) yb(j),(TT(j,k),k=1,nx)
end do
write (5,80)
do j = 1,nb,1
  write (5,70) yb(j),(G(j,k),k=1,nx)
end do
close (5)

10  format (//////////,a14)
20  format (7(1x,e13.7))
30  format (4(1x,e10.4))
40  format (2(1x,e10.4))
50  format (3(1x,e13.7))
70  format (7(1x,e10.4))
75  format (1x,'avE',8x,e10.4,1x,e10.4,1x,e10.4,1x,e10.4,1x,e10.4,
clx,e10.4)
78  format (1x,'avff',7x,e10.4,1x,e10.4,1x,e10.4,1x,e10.4,1x,e10.4,
clx,e10.4)
80  format (1x,'  --  ',1x,'  --  ',1x,'  --  ',
+      1x,'  --  ',1x,'  --  ',1x,'  --  ',
+      1x,'  --  ',)

stop
end

subroutine fraction (T,TK,f,fs,n)

real T(1300),TK(20),f(20),fs(1300)

do j = 2,20
  if (T(n).gt.TK(j-1).and.T(n).le.TK(j)) then
    fs(n) = (((f(j)-f(j-1))/(TK(j)-TK(j-1)))*
+      (T(n)-TK(j-1))) + f(j-1)
    goto 100
  end if
end do

100  return
end

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subroutine enthalpy (T,Tk,Ht,H,n)

real T(1300),TK(20),Ht(20),H(1300)

do j      = 2,20
  if (T(n).gt.TK(j-1).and.T(n).le.TK(j)) then
    H(n) = (((Ht(j)-Ht(j-1))/(TK(j)-TK(j-1)))*
+          (T(n)-TK(j-1))) + Ht(j-1)
    goto 100
  end if
end do

100  return
end

subroutine average (E,ff,TT,Ht,TK,f,j,k)

real E(20,10),ff(20,10),TT(20,10),Ht(20),TK(20),f(20)

do i      = 2,20
  if (E(j,k).gt.Ht(i-1).and.E(j,k).le.Ht(i)) then
    ff(j,k) = (((f(i)-f(i-1))/(Ht(i)-Ht(i-1)))*
+              (E(j,k)-Ht(i-1))) + f(i-1)
    TT(j,k) = (((TK(i)-TK(i-1))/(Ht(i)-Ht(i-1)))*
+              (E(j,k)-Ht(i-1))) + Tk(i-1)
    goto 100
  end if
end do

100  return
end

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