

On the Shock Response of Single Crystal Tin



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“They say that the Celtic tin melts much more quickly than lead. A proof of its fusibility is that it is believed to melt even in water: at any rate, it seems, it stains quickly. Now it melts in the cold also, when the weather is frosty, because, as they say, the hot substance inherent in it is by reason of its weakness shut up and compressed within.” [1]

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Abstract

With its anisotropic crystal structure, tin exhibits a strong orientation dependence of its single crystal properties. How this anisotropy affects the behaviour under the one-dimensional compression of shock loading has not been examined. With implications for the understanding of other, higher symmetry materials, a better understanding of the role directionality plays on this material will also help design and validate computational models, most notably crystal plasticity and molecular dynamics techniques.

In this work, the behaviour of single crystal tin is examined through the lens of several key shock phenomena, and compared to the polycrystalline behaviour. The Hugoniot states at the elastic limit and $\beta \rightarrow \gamma$ phase transition were measured using photon Doppler velocimetry to track the surface velocity histories and shock wave speeds. While the former shows an unexpectedly strong orientation dependence, with an order of magnitude difference in the yield point between orientations, above the elastic limit only qualitative variations are observed, with no measurable differences. The spall behaviour also shows no anisotropy. Instead, the strain rate is the primary governing factor of spall strength, and suggests that the spall failure is more similar to a viscous flow than brittle failure, the latter being common in many other metals.

Recovery experiments are conducted to examine the resultant microstructure. All samples show a refinement of grain sizes, especially around surfaces, suggesting recrystallisation forms one of the key pathways of plasticity. Examination of these recovered crystals through EBSD techniques shows strong textures, and great similarity between samples independent of crystal orientation or whether a sample has undergone the phase transition. The primary mechanism of plasticity aligns with an axis flip in the γ phase, rather than the slip or twinning phenomena observed statically.

Considering these results together, the anisotropy of the ambient single crystal is lost when plastic mechanisms activate. With similarities between recovered textures, the bulk behaviours may occur in similar microstructural environments, and with similar plastic mechanisms, resulting in the lack of observed differences between samples. With such similar mechanisms acting in a drag controlled plasticity regime, the HEL and spall strain rate behaviours may also be explained.

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List of Abbreviations

bcc		Body centred cubic
bco		Body centred orthogonal
bct		Body centred tetragonal
CPFE		Crystal plasticity finite element
EBS		Electron Backscatter Diffraction
EOS		Equation of state
hcp		Hexagonal close-packed
HEL		Hugoniot elastic limit
MD		Molecular dynamics
PDV		Photon Doppler velocimetry

1

Introduction

The dynamic behaviour of materials is an important area to study within the context of aeronautics, astronautics, automotive, defence and planetary science applications. Wherever systems or parts of systems are moving at high velocities, large and high-rate deformation can occur. With non-hydrostatic stresses present under such loading, and significant energies to be dissipated, the processes that underpin high-rate conditions can show significant differences to more conventional loading scenarios. Understanding the response of materials to these loading conditions is vital when designing systems for these applications, with the goals of improved safety, efficiency and economy.

The phenomena induced under such a scenario have been widely studied and categorised [2–8]. With high pressure generated experimentally on the surface of a sample using techniques such as explosives, laser ablation or a physical impactor, see Figure 1.1, a compressive wave is generated in the material. This pressure pulse can propagate as an elastic wave at pressures below the yield point, also known as the Hugoniot elastic limit (HEL). Above this HEL the compression is propagated as a plastic wave, inducing significant entropy changes, elevated temperatures, and in some materials, a change of phase. In specific loading conditions, several such waves can be induced in a material, when the mechanism of each subsequent wave is slower. Similarly, when meeting a free surface, release waves can be reflected

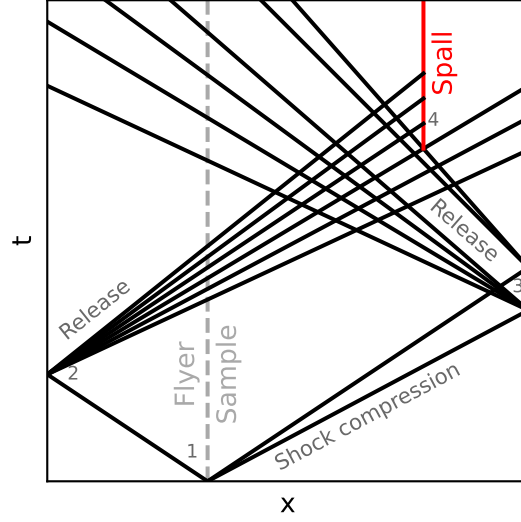


Figure 1.1: A displacement (x) - time (t) diagram representation of the waves generated in a plate impact experiment. During the impact, the inertia of the flyer plate causes compression at the impact surface (marked 1). The degree of compression is determined by the speed of the flyer, and the properties of the materials. This compression propagates in space and time as waves through the flyer and sample, until it reaches a free surface (points 2 and 3). Where the compression induces a change in the material, such as the transition from elastic to plastic behaviour, or a phase transition, the shock wave can split into distinct wave fronts. These wave-fronts propagate with a characteristic speed associated with the process and the local material state, with elastic waves typically travelling faster than plastic or transformation waves, as shown in the sample. The free surfaces are unable to support any stress, meaning the compressed material is able to relax, resulting in a release fan propagating back into the material. Where two such fans interact, tensile stresses are produced. If such stresses are sufficiently large, tensile failure can occur, (point 4), in a phenomenon known as spall.

back into the sample, inducing internal tensile failure, also known as spall. The challenges presented in predicting these dynamic behaviours bridges length-scales, with bulk scale phenomena rooted in lattice level changes.

Understanding the role that crystal structure plays in microstructural phenomenon is therefore key to understanding the bulk properties. On the atomic scale, relocations such as dislocation-glide underpin plastic mechanisms such as slip and twinning, which in turn influence bulk scale properties such as the transition from elastic to plastic behaviour, or even phase transformations. By studying single crystals, the crystal structure, and therefore the available mechanisms, can be controlled, enabling an in-depth understanding of the properties of materials.

For instance, the orientation of the single crystal can determine which plastic mechanisms are able to activate, thereby allowing a deeper study of the routes through which materials deform. In addition, studying single crystals reduces microstructural defects such as grain boundaries, whose presence can both seed and thwart plasticity and phase transformations. Studying single-crystals therefore gives insights into microstructural, and even crystal-lattice level changes in a material through bulk scale measurements.

One candidate to study is tin. From a low pressure diamond structure, to high pressure body-centred-tetragonal (bct - here after referred to as γ), body-centred-cubic (bcc) and hexagonal close-packed (hcp) structures [9], tin is noteworthy for the range, diversity and accessibility of its solid phases, see Chapter 2.2. The ambient phase of tin, β -tin, is noted for its asymmetric structure, shown in Figure 2.1a. This asymmetry gives opportunities to understand how directionality within materials affects their properties. For instance, static properties of tin, such as the compressibility and electrical resistivity of tin show significant orientation dependences [10], as does the thermal expansion [10–15], yield strength and post yield properties [16]. Static tests have also been used to study the wide range of deformation mechanisms displayed by tin, such as twinning and slip [16–19]. However, study of orientation has not yet been extended into the shock domain.

Indeed, shock provides a number of opportunities to study the behaviour of tin. Beyond the HEL and spall, which have been found to vary between orientations in other single crystals such as copper [20, 21] and aluminium [22] but are not yet studied in tin, the $\beta \rightarrow \gamma$ phase transition is readily accessible under shock.

A hysteresis between the forward ($\beta \rightarrow \gamma$) and reverse ($\gamma \rightarrow \beta$) transitions under shock has been found in polycrystals, with Chauvin *et al.* [23], Briggs *et al.* [24] and Mabire and Héréil [25] all mapping the hysteresis under shock. The result of Briggs *et al.* is particularly noteworthy, as their use of X-ray diffraction found a single crystal turned to polycrystal during the $\beta \rightarrow \gamma$ phase transition, a result that has been confirmed by Bober *et al.* [26]. The mechanisms of this behaviour, and the phase change, are not yet known. Another gap in the literature

is the lack of single crystal data. The only published data regarding the phase transition in single crystals is the molecular dynamics (MD) study of Soulard and Durand [27]. This study suggests a variation in the transition pressure depending on both the original orientation and the peak shock pressure. These results do not, however, fit the other experimental data well, especially the simulated (111), (110) and (001) orientations. It remains to be seen therefore whether these results are representative of the true behaviour of tin, or are instead due to an inaccurate potential, or other aspects of the model and its analysis.

Single crystal tests of the phase transition are therefore necessary to understand whether there is indeed an orientation dependence of the transition. By conducting these experiments in such a way so as to measure the transition in single crystals, the HEL, which has not yet been studied in single crystal tin, can also be measured. The characteristic pull-back of spall can also be measured in the results. In a single crystal, only certain mechanism such as twinning and slip can operate for a given orientation, so measuring the HEL can give an insight into the plastic behaviour. Similarly, the spall strength, a measure of the tensile failure when the release waves from the opposite free surfaces meet, is dependent on the microstructure of the sample. Consequently, a single set of experiments can provide data about a wide range of phenomenon, from which the behaviour of tin can be established. Additional diagnostics to study the crystal structure under or after shock are also necessary to better understand the previously mentioned phenomena. Two key methods can be used to achieve this goal: *in situ* diffraction, and shock recovery. With the former giving access to states that are only temporally stable for microsecond durations or less, while the latter able to give much richer data of the resultant post-shock microstructure.

1.1 Research Objectives

This work will therefore aim to better understand the properties of tin through a shock study of single crystal tin. Studying the HEL, $\beta \rightarrow \gamma$ transition and spall will all offer a better understanding of the dynamic behaviour of tin, and more generally

anisotropic materials. High-quality, quantitative measurements of these Hugoniot states, and the spall strength in single crystals will build a broader database of experimental measurements from which to build and train future models of tin.

Identifying independent variables beyond crystal orientation is key to understanding the observations in tin. Strain-rate and peak-pressure are key variables when predicting the HEL, $\beta \rightarrow \gamma$ transition and spall. Examining these can offer additional insights into the underlying mechanisms. Paired with diagnostics aimed at understanding the microstructural changes, an enhanced understanding of the high-rate properties of tin can be achieved.

Understanding the microstructural level changes of tin that underpin the bulk scale phenomena is another key objective. Fielding diffraction based techniques may be able to identify the phase transition pathway, or mechanisms of plasticity.

Through this enhanced understanding, and expanding the range of literature experimental measurements, this work aims to build a strong foundation for future theoretical work to understand the role of anisotropy in dynamic material behaviour. The lessons learnt in modelling tin are likely to have broader applicability to materials that do not show such anisotropy. For instance, other carbon group metals such as silicon and germanium shown similar phase transformations, including β tin phases, at high pressures [28, 29], meaning the lessons learnt from tin may help understanding of those materials. More generally, structural materials such as iron and titanium undergo solid-solid phase transformations, meaning improving the understanding of phase transformations through the study of tin may have a broader range of applications.

2

Background

Contents

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A substantial body of research has focused on understanding the behaviour of tin. Early studies concentrated on characterising the ambient phase and the low-temperature phase transition, while later work extended to high-pressure regimes. Advances in experimental techniques have enabled increasingly sophisticated in-

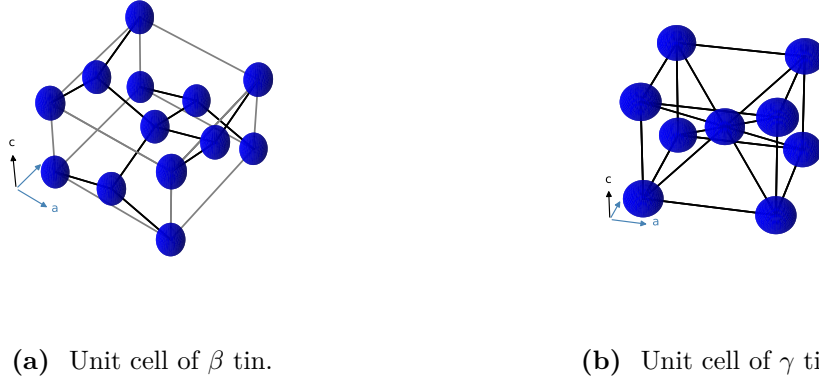


Figure 2.1: Unit cells for ambient (β) tin, and a high pressure (γ) phase.

vestigations, expanding the accessible regions of the phase diagram and enriching the dataset for well-studied phenomena. In parallel, complementary modelling approaches have emerged, providing deeper insights into these behaviours. The following review summarises this progression, loosely following this chronological path of discovery while grouping studies by their focal areas.

2.1 β -Tin

2.1.1 Identification of the Ambient Crystal Structure

The tetragonal nature of tin has long been known [30, 31]. However, only with the development of X-ray crystallography was the exact location of the atoms within the unit cell determined [32, 33]. This unusual crystal structure takes the appearance of a diamond lattice compressed along one of the diagonals, see Figure 2.1a. With a c/a ratio of 0.54543 [34], the lattice exhibits comparatively low symmetry relative to typical metallic crystal structures such as body centred cubic (bcc) and hexagonal-closed-packed (hcp).

2.1.2 Static Properties of the Single Crystal

As a result of the tetragonal microstructure, the properties of the lattice are known to depend on the crystal axis. For instance, the electrical resistance is known to

Table 2.1: Elastic constants of single crystal tin from literature. All values are in units of GPa.

Source	C_{11}	C_{12}	C_{13}	C_{33}	C_{44}	C_{66}
Bridgman [10]	83.9	48.7	28.1	96.7	17.5	7.4
Prasad and Wooster [36]	86	35	30	133	49	53
Mason and Bömmel [37]	73.5	23.9	24.9	87.6	22.0	22.6
House and Vernon [38]	76	62	44	97	22	23
Rayne and Chandrasekhar [39]	72.30	59.40	35.78	88.40	22.03	24.00
Cardinal [40]	72.46	58.9	36.0	88.00	22.08	24.20
Kammer <i>et al.</i> [41]	72.0	58.5	37.4	88.0	21.9	24.0
Mathew <i>et al.</i> [42] ¹	73.5	44.2	40.7	103.0	38.3	42.8

¹ Theoretical prediction using deformation theory.

depend on the orientation [10, 35], as does the thermal expansion [10–15]. Notably these parameters are different by around 50 % for the two principle axis of the crystal.

The compressibility of the crystal is similarly anisotropic [10, 36–41]. While the earlier studies report a broader and more variable range of values [10, 36], later investigations [39–41] converge towards a consistent set of values, see Table 2.1. Discussion regarding the disparity of these earlier works is presented in Rayne and Chandrasekhar [39] and Kammer *et al.* [41].

Third-order stiffness parameters have also been determined through sound velocity measurements [43], and calculated through deformation theory [42], see Table 2.2. The method used to calculate the parameters through deformation theory only considers the proximity of the nearest neighbours, rather than any directional changes to the bonds. The bonds in tin however are directional, see Figure 2.1a, potentially explaining why the second and third order parameters calculated by Mathew *et al.* [42] do not match well the experimental data of Swartz *et al.* [43]. Fourth order parameters have been predicted for tin using finite strain theory [44].

2.1.3 Crystal Plasticity

Single crystal tin is known to exhibit orientation-dependent yielding [16]. Due to the tetragonal crystal lattice, various slip and twin systems can operate, with the crystal rotation and system activation threshold determining which systems are likely to operate.

Table 2.2: Third-order elastic constants of single crystal tin from literature. All values are in units of GPa.

Constant	Swartz <i>et al.</i> [43]	Mathew <i>et al.</i> [42] ¹
C_{111}	−410	−410.0
C_{112}	−583	−246.9
C_{113}	−467	−202.3
C_{123}	128	−100.2
C_{133}	−156	−259.4
C_{144}	−162	−76.0
C_{155}	−177	−208.1
C_{166}	−191	−242.3
C_{333}	−1427	−929.5
C_{344}	−212	−200.4
C_{366}	−78	−72.2
C_{456}	−52	−72.2

¹ Theoretical prediction using deformation theory.

A number of slip and twin systems have been proposed for tin. The earliest observations of deformation mechanisms are of the $\{301\}$ twin system [17, 19, 30, 31, 45, 46]. Early sources, however, use a larger unit cell, and therefore a different reference system [47]. A second twin system, $\{101\}$ has also been observed [18, 46]. Notably twinning is observed during impact loading along, or close to, the $[100]$ axis [17, 19]. Twinning is also hypothesised to be the dominant mechanism of plastic deformation at high pressure [48, 49].

The slip behaviour of tin is less well defined, with a number of systems proposed by many different authors. The most commonly observed systems are [50, 51]: $\{100\}\langle 001\rangle$, $\{100\}\langle 010\rangle$, $\{100\}\langle 011\rangle$, $\{110\}\langle 001\rangle$, $\{110\}\langle \bar{1}11\rangle$, $\{101\}\langle \bar{1}01\rangle$, and $\{121\}\langle \bar{1}01\rangle$. Literature observations and predictions of the systems are presented in Table 2.3

Other systems have been predicted theoretically, such as $\{001\}\langle 100\rangle$, $\{101\}\langle 11\bar{1}\rangle$, $\{121\}\langle 11\bar{1}\rangle$ and $\{\bar{1}01\}\langle 010\rangle$ [59, 60, 73], although observations remain sparse. Honda [55, 76] reports $\{101\}\langle 010\rangle$ has been observed, but does not provide a reference. In Düzgün and Aytaş [68], two systems, $\{121\}\langle 11\bar{1}\rangle$ and $\{001\}\langle 110\rangle$, are reported to have been observed in Aytaş’s “Thesis of Associate Professorship”, although this author has been unable to find that source. Later work by Düzgün [69, 70] does

Table 2.3: Summary of slip systems observed and predicted in tin.

System	Experimental observations	Theoretical predictions
$\{100\}\langle 001\rangle$	[16, 32, 52–58]	[59, 60]
$\{100\}\langle 010\rangle$	[16, 53, 55, 61–72]	[59, 60, 73]
$\{100\}\langle 011\rangle$	[32, 56–58, 62, 63, 67–70]	[59, 60]
$\{110\}\langle 001\rangle$	[16, 32, 52–54, 56, 57, 71, 74, 75]	[59, 60]
$\{110\}\langle \bar{1}11\rangle$	[16, 53, 54, 56–58, 61, 62, 67, 71, 74, 76–78]	[59, 60, 73]
$\{101\}\langle \bar{1}01\rangle$	[16, 52, 54, 57, 62, 68, 69, 72]	[59, 60, 73]
$\{121\}\langle \bar{1}01\rangle$	[16, 52, 57, 63, 68–70, 74, 79]	[59, 60, 73]
$\{001\}\langle 100\rangle$	[57]	[59]
$\{101\}\langle 11\bar{1}\rangle$	[58]	[59, 60]
$\{121\}\langle 11\bar{1}\rangle$	[58, 68]	[59, 60, 73]
$\{\bar{1}01\}\langle 010\rangle$		[59, 60, 73]
$\{001\}\langle 110\rangle$	[57, 68]	
$\{110\}\langle \bar{1}\bar{1}0\rangle$		[80]

not repeat these systems. In Düzgün *et al.* [69], $\{100\}\langle 101\rangle$ is given, although this is not orthogonal, and presumed to be a typographic mistake for another system.

Zhou *et al.* [57] report observations of $\{001\}\langle 110\rangle$ and $\{001\}\langle 100\rangle$, and lists a further possible system for tin: $\{110\}\langle \bar{1}\bar{1}0\rangle$. This additional system is also used in crystal plasticity finite element (CPFE) modelling by Darbandi [81], where it is credited to Düzgün and Aytaş [68], although it does not occur in that source. CPFE modelling by Pokharel *et al.* [80] uses the $\{110\}\langle \bar{1}\bar{1}0\rangle$ system instead of $\{101\}\langle \bar{1}01\rangle$ in their simulations, due to “better agreement” between their modelling and experimental results. Kaira *et al.* [58] report observations of $\{101\}\langle 11\bar{1}\rangle$ and $\{121\}\langle 11\bar{1}\rangle$ based on transmission electron microscopy inspection of twin boundaries in micropillars.

Later CPFE modelling studies use similar sets of systems, sometimes with $\{101\}\langle 11\bar{1}\rangle$ and $\{001\}\langle 100\rangle$ [82, 83], or the hypothetical system $\{110\}\langle \bar{1}\bar{1}0\rangle$ [80, 82–84]. While the existence of these additional systems cannot be ruled out, the lack of experimental observation, especially relative to the seven listed systems, and the inconsistency between the sources, leaves questions as to their presence. They may operate under specific temperatures, loading conditions or alloys, thereby contributing to the difficulty in repeated observation.

The critical shear stresses for the systems are even more uncertain. Depending on a range of factors, including temperature and purity, the critical shear stresses are in the range of tenths to tens of megapascals [16, 19, 52, 53, 74]. Some of the systems have wide ranges of published critical shear stresses, others lack any experimentally measured values at all [74].

2.2 Phase Boundaries

Tin exhibits a range of solid phases, from the diamond α phase at low temperatures, up to the high-pressure bcc and hcp phases. A summary of the literature is presented in Figure 2.2, with detail of the $\beta \rightarrow \gamma$ transition in Figure 2.3.

2.2.1 α Tin

Beyond ancient remarks about tin “melting” in the cold [1], a significant body of work has examined the low-temperature transformation from β tin to the diamond-cubic α phase: far more than can be discussed here. Periodic reviews highlight key trends in the study of α tin, reflecting evolving motivations over time.

The earliest investigations focused on the disintegration of solid tin during the $\beta \rightarrow \alpha$ transition, a phenomenon commonly referred to as tin pest [103]. This transformation, being a relatively accessible solid-solid phase change, has since attracted attention as a model system for studying phase kinetics and nucleation [101, 104, 105].

Interest in α tin intensified following the discovery of its semiconducting properties [106–108], and more recently, in the context of low-temperature performance of lead-free solders [109]. Notably, the transformation rate is sufficiently slow that metastable β tin can be tested at low temperatures [16, 110, 111]. For this reason, the boundaries of stability are somewhat difficult to determine, especially at low temperatures.

To the author’s knowledge, no shock study has yet been conducted on low-temperature tin beyond spall strength measurements [111]. While investigating the Hugoniot elastic limit (HEL) or further mapping phase transitions under shock

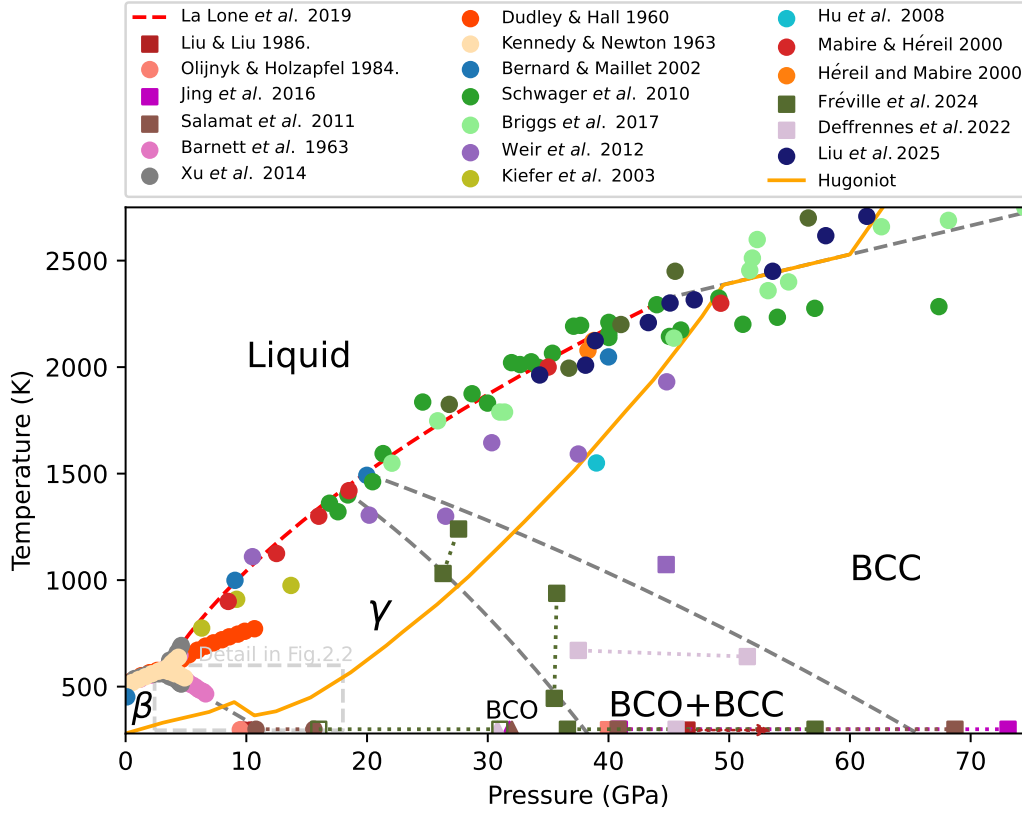


Figure 2.2: Phase diagram of tin showing literature boundaries [9, 25, 29, 85–102]. Room temperature taken as 25 °C unless specified by source. Squares $\square$ denote the boundary of the bcc region, with filled squares marking increases in pressure or temperature, while hollow squares mark decreasing pressure. Triangles mark $\triangle$ the transition from γ (bct) to bco. Dotted lines between points indicate a coexistence region, where both phases have been observed. Guide lines (dashed, grey) have been added with approximate locations of the transitions. For simplicity, α tin has been omitted from this plot due to its low-temperature and low-pressure stability domain.

at low temperatures could be of interest, the practical value remains unclear. Shock studies of α tin would be particularly challenging due to the difficulty in synthesising suitably sized, non-powder samples [112].

2.2.2 β - γ -Liquid Triple Point

A number of studies have examined the melting of tin under pressure. These are conducted using static cells and furnaces, with the phase diagnosed using electrical resistance measurements [90, 113, 114], by measuring the latent heat [91, 115–117], analysing volume changes [118], using X-rays [88, 119] or measuring sound speeds

[89]. The exact location of the triple point varies subtly between studies, likely due to the different press design, pressure medium and diagnostic technique used between studies [115, 120], although overall they are in good agreement. It has been noted that recalculating the results of Dudley and Hall [90] using an improved pressure scale yields better agreement between this dataset and the other determinations [101, 115, 121]. The original values are presented in Figure 2.2. For other work examining the phase boundary, the data density is unable to resolve the triple point [117, 118]. These early studies [88, 90, 91] form some of the first observations of the $\beta \rightarrow \gamma$ phase transition in tin through the inflection in the melt curve.

2.2.3 Static Studies of the β - γ Boundary

After the discovery of the phase change through the mapping of the melt line, a number of studies sought to examine the transition pressure at room temperature [29, 34, 86, 100, 113, 122–124, 128]. Again these studies use pressure cells, diagnosing the phase change through X-ray diffraction [29, 34, 86, 100, 123, 124, 128], or through the change in electrical resistance [113, 122]. Later work indicates pressure regions where both β and γ coexist [34, 86, 100]. This could be as low as 6 GPa [129], although most studies find a significantly smaller coexistence region [34, 86, 100]. Studies of the transition in single crystals indicate the strain-history influences the pressure at which the transformation occurs [100], and with the transition from $\gamma \rightarrow \beta$ occurring at lower pressures than the transition from $\beta \rightarrow \gamma$.

2.2.4 High-Rate Studies of the β - γ Boundary

A number of high-rate studies examine the phase transition. Following the static probing of the transition, early plate-impact shock experiments sought to determine the pressure of the stepped profile characteristic of the phase transition under shock loading [130–133]. Mappings of the characteristic two-wave profile indicative of the phase transition under shock were later conducted [134, 135]. Thermodynamic equation of state (EOS) models have been developed from these experiments [25, 99, 126, 134, 136, 137], further discussion in Section 2.4.1, to predict the shock

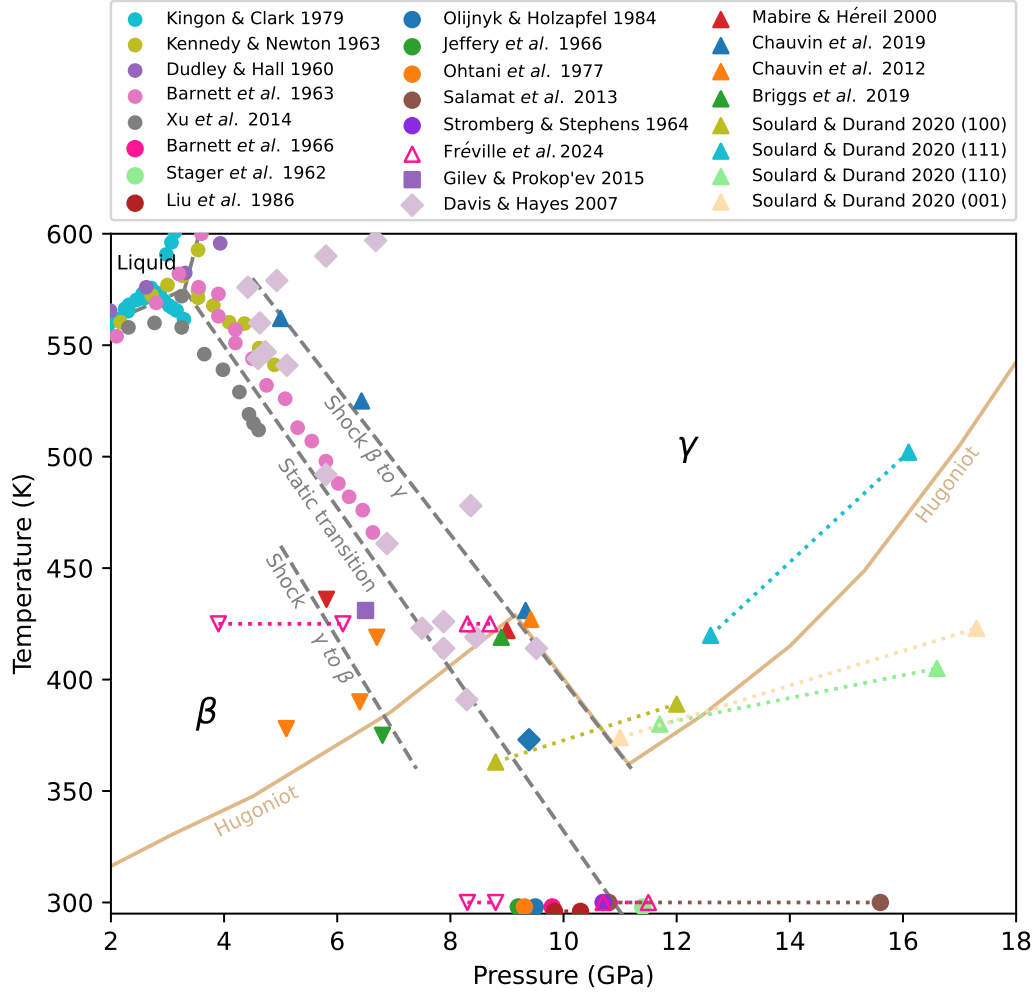


Figure 2.3: Phase diagram of tin showing the boundary between β and γ regions [23, 25, 27, 29, 34, 86, 88–91, 95, 100, 114, 115, 122–127]. Room temperature taken as 25 °C unless specified by source. Circles $\circ$ denote static tests, diamonds $\diamond$ denote isentropic loading, squares $\square$ denote ramp loading, upward $\triangle$ and downward ∇ triangles denote the forward and reverse transition, where filled triangles denote shock loading and unfilled triangles denote static loading. Dotted lines between points indicate a coexistence region, where both phases have been observed. Guide lines (dashed, grey) have been added as guides to the approximate locations of the transition.

temperature. With the development of pyrometry, the temperature of the transition has been directly measured [23, 127, 138].

Due to the low time duration of the transition under shock loading, attempts to directly observe the phase change under shock compression using X-ray imaging have largely been limited to the release state [24, 139]. Direct attempts to measure the phase change have struggled to detect sufficient numbers of Bragg peaks to determine the crystal structure and transition pathway [140, 141]. Notably, X-ray studies suggest that the $\beta \rightarrow \gamma$ transformation is associated with a significant refinement in the grain size under shock loading [24, 26, 139, 142]. Notably, these studies compare as-received samples to the peak shock state after the transformation, meaning refinement may occur prior to the transition. Indeed, the grain refinement may be a feature of the high temperatures and strains, with Taylor tests of β tin showing significant refinement despite impact speeds far below the threshold required for the phase transition [49].

A clear hysteresis in the transition is evident in the shock data [23, 24, 132, 134], similar to that observed statically [100], suggesting an energy barrier to the transition. The magnitude of this transition appears to be similar between static [100] and dynamic measurements on unloading, but greater for shock on loading, see Figure 2.3. With only a few data points available, further investigation may shed more light on any potential rate-dependence to the threshold. Notably, only the ramp loading of Davis and Hayes [126] consider the transition in single crystals, finding no orientation dependence, meaning the shock study of single crystals may offer additional insights into the transition and its mechanisms.

2.2.5 Mechanisms of the β to γ Phase Transition

The earliest mechanism of the $\beta \rightarrow \gamma$ phase transition was proposed before the full refinement of the γ phase [143] (γ crystal structure shown in Figure 2.1b), although it was later refined to the bct lattice [144]. Having been reproduced without reference to the original sources by Katzke and Tolédano [145], it is commonly referred to as the Katzke mechanism in recent literature. In this work it is attributed to

the earlier author, and referred to as the Musgrave mechanism. This mechanism maintains the same c axis in the parent and child lattices through the relationship $(110)_\beta || (010)_\gamma$ and $[001]_\beta || [001]_\gamma$. This mechanism would fit the observations under shock by Chauvin *et al.* [140] (see also [142]). Through X-ray diffraction, the latter highlights a delay between the shock-wave arrival and the initiation of the $\beta \rightarrow \gamma$ transformation, as well as an extended retention of the high pressure phase after release. This mechanism has also been observed under static loading [146], forming twins along the $\{101\}$ plane, effectively flipping the c - and a -axis. Such an axis flip has also been predicted in MD simulations, where loading along the $[001]$ direction induces this twin [147]. A (101) twin only results in a 90° rotation if the c/a ratio is unity, i.e. the lattice is bcc. If $c \neq a$, the $\{101\}$ plane is not at 45 degrees from the axes and the mapping is not exact, meaning in tetragonal γ , this is not a true axis flip.

A second mechanism has also been observed by Fréville [146] in a single crystal sample undergoing the transformation for a second time. This mechanism has the relation $(101)_\beta || (110)_\gamma$ and $[10\bar{1}]_\beta || [1\bar{1}1]_\gamma$. The possibility of several mechanisms and their twins could offer an explanation of the refinement of the grain structure under the phase transition. Under shock loading, where the transition is severely overdriven, local lattice disturbances, such as dislocations, could nucleate γ with different parent-child relationships through the different mechanisms. If β grains were to nucleate γ through the different mechanisms, further perturbed by the compaction due to the density change, a new microstructure with a broad distribution of grains could be created. In single crystals with a uniform starting orientation, the γ grains may therefore be modally distributed around the various pathways.

2.2.6 Higher Pressure Phase Changes

The examination of the crystal phase was extended to increased pressure, searching for high pressure metallic phases similar to those found in other carbon group elements [29, 86]. Through these X-ray studies, the transition from the γ phase into a bcc phase was observed. These studies suggest that the bct phase coexists with the bcc phase, an observation not reproduced by Desgreniers *et al.* [148]. This

discrepancy was hypothesised to be due to non-hydrostatic stresses, both within the sample and the pressure medium. Notably, modern synchrotron based X-ray studies suggest the existence of a body centred orthogonal (bco) phase, and an extended coexistence region of bco and bcc [34, 87, 100, 101]. At higher temperatures a coexistence has also been observed [100, 101]. The hysteresis of this transformation is significant, with coexistence observed in the range 31 GPa to 16 GPa on unloading and 36.6 GPa to 57.1 GPa during loading. As a result, a sample can be entirely one phase, or a mixture, at a given pressure and temperature [100] within the coexistence range. This implies a lattice with a low energy difference between phases, resulting in the stable phases forming dependent on the local stress field. The exact stability boundaries of the intermediate phases is not yet fully defined, with variations between sources [34, 86, 87, 101, 146].

At yet higher pressures, an hcp phase is observed [9, 100, 149]. At pressures greater than 1.3 TPa tin is predicted to return to bcc [9]. The stability region of this hcp phase is thought to be limited by temperature, with laser-driven ramp-compression up to 1.2 TPa only observing bcc [150]. At lower pressures between 50 GPa to 100 GPa, laser-driven ramp-compression experiments by Floyd *et al.* [151] observe a low anisotropy of strain, suggesting a low material strength.

The melt curve above the β - γ -liquid triple point has been the focus of a number of studies. Probing of the melt curve above the triple point has been conducted statically using diffraction [95, 97, 100, 152], laser speckle motion [94] and electrical resistance measurements [96]. The melt curve has also predicted by first principle calculations [92]. High-rate loading has also been used to study the melt curve at high pressures and temperatures. Through the use of an EOS, the state information of melt on release was calculated under shock [25, 134] and ramp loading [153]. Pyrometry [85, 99, 102] and X-rays [139, 154, 155] have been used to determine the phase on release. Together, these show a steep initial rise in melt temperature above the β - γ -liquid triple point, and a much more gradual rise when transforming from bcc to liquid. The lack of a discontinuity in gradient of the melt curve highlights the more gradual change in phase between bct and bcc.

Measurements of the wave speeds have also been used by Hu *et al.* [98, 156] to determine the onset of melt on the Hugoniot. However, the data presented are open to other conclusions, which could place the data closer to the other literature data, with the melt point on the Hugoniot around 60 GPa. In particular, Hu *et al.* prescribe the bcc region to two data points, taking further higher pressure points to be solid-liquid coexistence. However, the derived quantities do not show any significant difference in properties between these two regions. Furthermore, the fit Hu *et al.* attribute to be the solid-liquid coexistence region could be extended to include the points described as bcc.

When placed on a plot of pressure and temperature, the interpretation taken by Hu *et al.* aligns with a series of low pressure data that Hu *et al.* cite as Kiefer *et al.* [97]. However, Kiefer only presents three points, all above the $\beta \rightarrow \gamma$ transition, while the data presented by Hu *et al.* show a total of around twenty points both sides of the triple point. It may be that the dataset belongs to Dudley and Hall [90], which Hu *et al.* plots as mapping the $\beta \rightarrow \gamma$ transition, despite Dudley and Hall only studying the melt curve. Noting that Dudley and Hall's pressure data has been corrected [101, 115, 121], it may be suggested that Hu *et al.* have interpreted their results to place the uncorrected data of Dudley and Hall [90] alongside other high pressure melt measurements by hypothesising an extended region of solid-liquid coexistence.

Further experiments to measure the sound speed by Song *et al.* [157], expanding the data of Hu *et al.* [98, 156], better resolve the drop in shear modulus around 60 GPa, supporting the conclusion that this is indeed the melt point on the Hugoniot. Sound velocity measurements of porous tin in this region yield a shear modulus more similar to that of γ tin determined by Hu *et al.* [98, 156]. This suggests that porosity may thwart the phase change from γ to bcc, although extending the domain of the tests would strengthen this hypothesis. A further observation made by Song *et al.* [157] is that the porous samples show a decrease in shear modulus attributed to melting at lower pressures with increasing porosity, as might be expected where pore collapse results in local heating.

The predictions by Song *et al.* [157] of a “new unknown phase” between bcc and melt on the Hugoniot are largely based on a single point of Hu *et al.* [98, 156] in the bcc region. It is this point that underpins Hu *et al.*’s hypothesised solid-liquid coexistence region, leaving questions to its accuracy: removing it from the dataset would yield a single region between γ and melt where shear strength is constant. Without any method to diagnose the phase, and considering the uncertainty of the phase stability in static data, it may be that this point has not transformed, or transformed to bco rather than bcc. Song *et al.* [157] calculate shock temperatures based on extending model parameters of the γ phase EOS up through the bcc region and melt curve, highlighting the need for greater understanding of this regime, and emphasising the intricacies of the transition between γ and bcc phases.

Considering also the differences between location of the boundaries of the γ , bco and bcc phases [100, 101, 150] and melt curves between sources [94, 95, 100, 102, 152, 157], it is suggested that the specific experimental conditions may affect the stability regions for each phase. Effects such as sample purity and load history, and in particular any non-hydrostatic effects, may alter the energetic preferability of these phases, and the onset of melt [101]. Beyond the discrepancies between investigations, other factors such as the structural similarities between the solid phases, the gradual transition as the c/a ratio approaches unity, and the extended region of phase coexistence all support the hypothesis that pressure alone does not dictate phase stability. However, further examination of this phenomenon remains a challenge, given the difficulty in achieving a purely hydrostatic load under high pressures in cells, or measuring the specific loading a sample experiences [158].

2.3 Other Shock Studies

A number of studies have examined the behaviour of tin under shock loading beyond the phase diagram. This includes the Hugoniot states, the HEL and the spall behaviour.

2.3.1 Hugoniot Elastic Limit

While no study has directly examined the HEL of tin, a number of values of the peak elastic pressure under shock have been proposed, or can be deduced from published experimental traces. For instance, in shock experiments examining the phase transitions, Hugoniot and release states, Mabire [134] reports a value of around 0.2 GPa. However, the use of VISAR systems tuned to the higher-pressure states may exhibit reduced sensitivity to the relatively low surface velocities involved. Similarly, Steinberg [159] quotes a value of 0.24 GPa without providing the provenance, while Chong *et al.* determine a value of 0.124 GPa in ramp tests [160]. At high strain-rates under laser induced shock, the pressure history traces of Moshe *et al.* indicate a precursor state in excess of 1 GPa [161], consistent with the 100 m s^{-1} free surface velocities of de Ressaiguier *et al.* [162]. Finally, the free surface velocity traces of Anderson *et al.* [135] gives a step at around 50 m s^{-1} .

A range of magnitudes of HEL are therefore found for tin. Comparing the static yield strengths (see Section 2.1.3) to the HEL values, it is clear that the threshold for plasticity is significantly higher under shock loading. Strain-rate effects may well be behind this phenomenon, with Schill *et al.* [48] finding a discontinuity of gradient when comparing yield stress to strain rate. Attributed to “twinning anelastic behavior,” twinning is predicted to be the dominant high-rate mechanism of plasticity. It has also been noted that increasing the temperature under high-rate loading increases the yield stress, a behaviour consistent with drag-controlled plasticity [163]. Ultimately the HEL behaviour is relatively uncertain, meaning further work to map out the influence of variables such as strain rate, purity, microstructure and precursor decay would be valuable to better understand the elastic-plastic transition in tin. Single crystals also show a significant deviation in yield behaviour statically [16], so studying the single crystal shock HEL could also provide insights into the properties of tin.

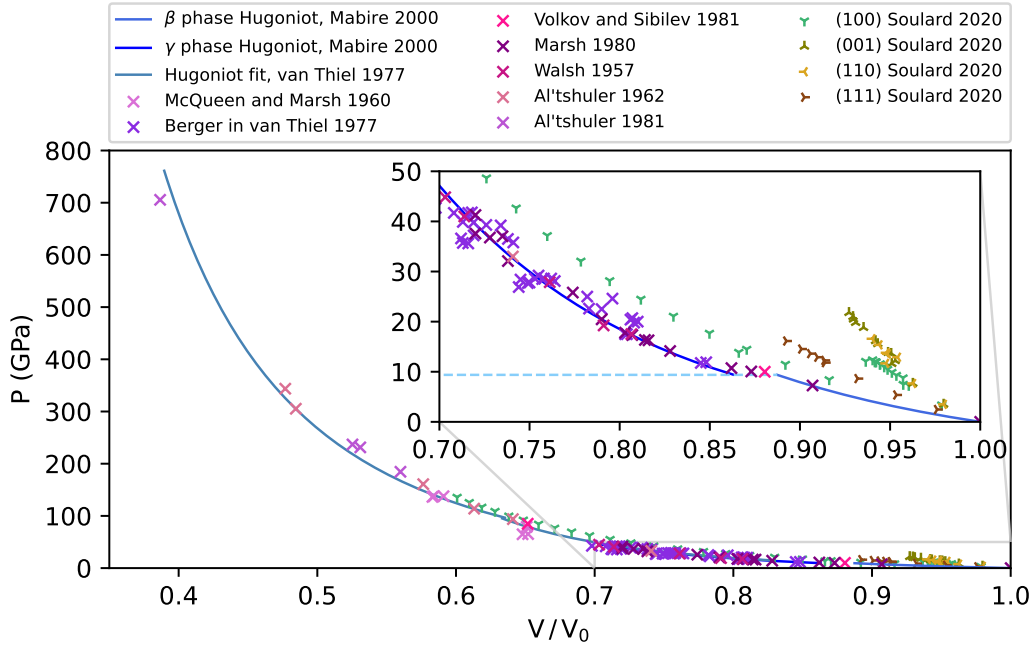


Figure 2.4: Literature Hugoniot compression curve for tin. Data [27, 164–167, 169, 170, 172] are plotted alongside modelled curves, [25, 165].

2.3.2 Hugoniot Data

The Hugoniot of tin has been extensively studied, with compression data presented in Figure 2.4. Perhaps the most widely used dataset is by Marsh *et al.* [164], presenting data up to 165 GPa. A second large dataset is presented by Berger and Fauquignon in van Thiel [165], in the range 12 GPa to 43 GPa. Smaller datasets are presented by Walsh *et al.* between 17 and 43 GPa [166], Al'tshuler *et al.* between 33 and 350 GPa [167] (some data in this source are corrected from [168]) and between 185 and 705 GPa [169], and McQueen and Marsh between 79 and 138 GPa [170]. As mentioned previously, a number of studies have also aimed to determine the Hugoniot states of the phase transitions [25, 130–132, 134]. The Hugoniot of porous tin has also been studied [171], as have the Hugoniots of pre-heated liquid tin [172, 173]. Overall, the focus of much of this work, beyond categorising the Hugoniot curve, is to study the melting behaviour on the Hugoniot. As a result, much of the data are focused on this region, with a scarcity of points in the β phase, or around the $\beta \rightarrow \gamma$ transition.

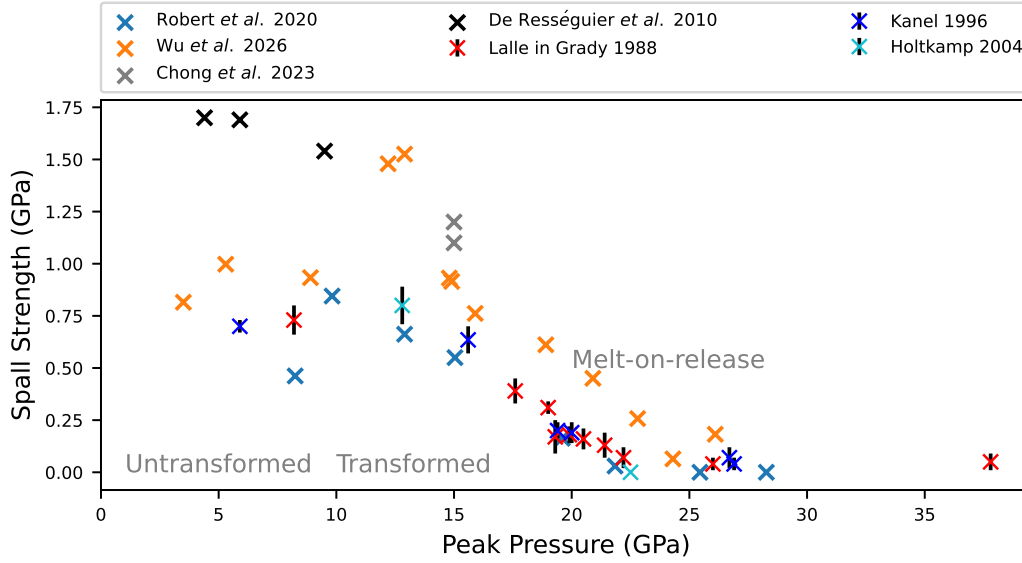


Figure 2.5: Summary of spall literature [93, 133, 160, 174, 176]. With increasing peak pressure, spall strength rapidly drops as the material melts on release.

2.3.3 Spall and Dynamic Tensile Failure

While spallation failure has been studied in tin, the major focus has been on studying the onset of melting on release [93, 133, 162, 174–178], see Figure 2.5. This phenomenon was first investigated by Lalle in Grady [174] through explosive driven shock, and expanded upon through gas-gun experiments by Kanel *et al.* [133], Robert *et al.* [93] and Wu *et al.* [178]. Together, these show a sharp drop-off in spall strength for shock pressures between 15 GPa to 20 GPa. Laser driven spall follows this trend [162, 175–177], although the threshold required for tin to melt on release may be lower than at these higher strain-rates. Examination of the recovered samples show that the spall failure in these laser driven spall experiments is ductile fracture, mostly transgranular [176]. The spall planes in two states, one well above the threshold to melt on release, and one below, were examined by Holtkamp *et al.* [179] through proton-radiography, highlighting the differences between the failure modes. The drop in spall strength corresponds with melt on release detected through surface reflectivity in gas-gun driven experiments, where melt is induced for release from shock states above 23.5 GPa [180].

With relatively few points below 15 GPa, the spall behaviour of solid tin is poorly understood. For instance, the lowest point of Robert *et al.* has “a noise in data measurements prevent to properly define [the spall strength]”. Estimating the spall strength from the velocity traces presented, the spall strength of this point appears to be much lower than the work of Kanel *et al.* [133] and Lalle [174]. Wu *et al.* [178] find the spall strength to be higher under triangular wave shocks compared to flat-topped, whilst also hypothesising an increase in the spall strength of material that has undergone the transformation to γ tin. Ramp-wave data, meanwhile, shows a somewhat higher spall strength [160], as does laser shock [161, 162, 175–177]. While Moshe *et al.* seek to investigate the strain-rate behaviour [161], the data do not seek to explain the differences in observed spall strength between studies. These experimental methods are associated with significant changes in the strain rate, meaning a more complete investigation of the strain rate may be able to better explain the diversity in these literature results.

Beyond the influence of spall strain rate, the $\beta \rightarrow \gamma$ phase transition is associated with a significant refinement of the crystal structure [24, 26, 139], and the properties of the β single crystal are highly anisotropic. Considering that the spall strength of other metals has been found to depend on the microstructure [181–183] and single crystal orientation in materials with greater crystal symmetry than tin [20–22, 181, 184], tin offers a rich domain to study.

The spall strength of tin has also been studied in preheated or pre-cooled samples [111, 185, 186]. As initial temperature is decreased below ambient, spall strength is observed to very weakly decrease, and as the initial temperature is increased towards melt, the spall strength very rapidly drops-off [111]. Kanel *et al.* [185] and Ashitkov *et al.* [186] pre-heat their material to test the spall strength of liquid tin.

Release failure in the form of jetting has also been studied in tin [26, 155, 177, 187–193]. The accessibility of liquid, solid, and mixed states means tin ejecta provides a rich domain to study. Together, these phenomena, alongside the previously mentioned solid-solid phase transition and possible strain-rate

dependence mean that tin offers a significant opportunity for probing complex material behaviour under dynamic loading.

2.4 Modelling Tin

2.4.1 Equation of State Models

EOS models have been developed for tin, taking two main approaches. The first uses a Birch-Murnaghan isotherm alongside a Mie-Grüneisen model [25, 126, 134–136, 153, 194, 195], the second a Helmholtz free-energy approach [196–200]. Strength models have also been proposed [49, 159].

The Birch-Murnaghan formulation is noteworthy given phase transition kinetics between β and γ phases can be represented through the use of rate-dependent laws [134, 137, 153]. Using a rule of mixtures, the Gibbs free energy difference between the metastable and stable phases can be used to calculate a transformation rate through empirical equations. Thus, the wave profiles with increasing pressure can be more accurately matched than simply assuming thermodynamic equilibrium [137], and the hysteresis captured [134].

2.4.2 Molecular Dynamics

MD simulations offer a powerful method to examine the properties of tin from the atomic scale. A number of potentials have been proposed for tin [201–208]. Those of Baskes and Ravelo [203], Ko *et al.* [202], and Nitol *et al.* [201] are perhaps the most significant, due to their ability to represent the directionality of the bonds within tin. Baskes and Ravelo [203, 204] and Ko *et al.* [202] take a conventional embedded atomic model, modifying it to model this directionality. These potentials are fitted to the α and β phases, meaning they perform best when modelling the low pressure behaviour. The work of Nitol *et al.* [201], meanwhile, fits the potential to the properties of tin using a machine learning approach for a much broader range of phases.

MD simulations have been used to study many of the properties of tin mentioned previously in this literature review. For instance, many potentials are used to

predict the phase diagram and the isothermal and Hugoniot compression curves, comparing to experimental results [201, 206, 207]. However, the small scale of atomic simulations, with typical length scales in the tens or hundreds of nanometres and times in the picosecond domain, makes direct comparison to experimental measurements challenging. However, continued advances in computational power allow for increasingly advanced simulations.

Studies of shock loaded tin using MD are therefore a relatively recent phenomenon. In this regard, the phase transition from β to γ has been examined for single crystals under shock loading [27]. This study shows a strong orientation dependence for the Hugoniots below the transition, and significant differences to the threshold pressures. The only orientation with tabulated data (001) shows a pressure dependence of the transition threshold, with higher peak pressures remaining as β to higher pressures. This is not unexpected behaviour, however, as can be seen in Figure 2.3, the predicted transition does not match well the existing experimental data. Experiments to directly study the phase transition in single crystals are therefore required to verify these predictions.

Dynamic tensile failure, has been modelled, including jetting [209, 210] and spall [211–214]. These offer potential insight into the formation of damage in tin. However, some key areas of uncertainty remain. For instance, the lack of experimental studies in this area, such as post-mortem recovery to examine incipient spall, means the simulation results are difficult to validate. Building a broader experimental dataset, with pressure-volume data, elastic properties and spall would therefore be valuable to aid the development of MD simulations that can well model directional materials such as tin.

2.5 Summary of the Literature

With a highly anisotropic crystal lattice, the effects of crystal orientation on the single crystal properties have been examined in a number of static studies. The mechanisms of plasticity are not well understood. While a number of slip systems have been documented, others have been proposed with little experimental observation. The

operating regimes, especially critical stresses are not well documented, and worthy of further study to better understand and predict plasticity in tin.

The solid phases at room temperature have been extensively probed, with a large body of data focused on mapping the transitions at lower pressures and temperatures. However, the mechanisms of the $\beta \rightarrow \gamma$ transition are still relatively uncertain, with little experimental evidence. Experimental and theoretical work to examine this phenomenon, especially under high-rate loading would be valuable. Shock experiments on single crystals may highlight any orientation dependence of this transition, and therefore the influence of shear, or orientation on the mechanisms. In addition, the specific conditions for the transition from the γ phase to the bcc phase have not been well mapped at high temperature, although a few studies have examined this region statically. The boundaries of stability for these phases, as well as the intermediate bco phase, are still unclear, and require further investigation.

Under shock loading, dynamic phenomena, including the HEL and spall are under-developed fields. The data available are often incomplete, and do not fully explore the variables that affect these phenomena. Measurements are often by-products of experiments examining other phenomena, such as the $\beta \rightarrow \gamma$ phase transition, or the onset of melt on release. Consequently data sets are often standalone, with little to combine and understand the different experimental conditions between studies, especially strain rate. Further study of the HEL and spall, with consideration of the existing literature data, is required to better understand the behaviour of polycrystal tin under high-rate loading. In addition, given the large anisotropy in the single-crystal properties, the effect of crystal orientation have similarly not been considered. This is of particular interest given that the HEL and spall are both phenomena that are influenced by sample microstructure and crystal orientation in other materials, but have not yet been studied in tin.

Beyond developing an understanding of the role of microstructure on the properties of tin, further experiments are required to help challenge, validate and develop MD models. With scarce experimental data to verify the models, the

full potential of MD to understand the atomic-level processes that dominate the macroscopic response of materials cannot be truly unveiled. The rich properties of tin offers a diverse domain in which to improve the capabilities of atomistic simulations, from which the understanding of other materials can be developed.

Experiments to study the properties of single crystal tin are therefore of great interest. Through the one-dimensional compression of shock loading, the directional response of tin can be examined. Properties such as the HEL and spall strength can be measured, alongside the solid-solid phase transition, to better understand the role the asymmetry of the crystal lattice plays on the behaviour of tin.

3

Methods

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A number of systems are available to generate the shock loading required to induce the $\beta \rightarrow \gamma$ phase transition in tin. Most notably, laser ablation and gun driven impacts offer two of the most commonly used techniques in modern scientific environments. However, when studying millimetre order length scales, only gun-driven shocks are able to provide a sufficiently sized 1D shock.

The experimental results discussed in Chapters 4, 5 and 6 are all conducted using the gas-gun system in the Department of Engineering Science at the University of Oxford. Fundamentally, this system uses compressed gas to propel a sabot, typically made from polycarbonate, fronted with a flyer plate. The design of this system has been discussed elsewhere, see for instance refs. [215, 216].

With such high velocities as found under shock, the diagnostic techniques available are similarly limited. With quantitative descriptions of the loading history required, the most common methods to make measurements are the interferometric techniques, VISAR and photon Doppler velocimetry (PDV). Other diagnostics, such as shorting pins, pressure transducers and streak cameras have all been used historically, and can be found in many of the literature shock studies in Chapter 2. However, for its ease of use, non-invasive nature, and simplicity of analysis, PDV is used here.

In the plate impact experiments presented in Chapters 4 and 5, PDV is used as the primary diagnostic. This laser based technique relies on the Doppler shift of light reflected from a moving surface to determine that surface's velocity. With a sample rate of 40 GS^{-1} as used in this work, typical time resolutions are in the ns domain. The velocity resolution is dependent on both the experimental conditions, such as the signal-to-noise ratio of the laser return path, and the analysis method, such as the time window. Despite this, velocity uncertainties for the measurements made here are usually well below 10 m s^{-1} . The details of the lasers and instrumentation used in this work have been extensively described elsewhere, see for instance ref. [215].

In a theoretically ideal shock experiment, the shock wave is perfectly parallel to the surfaces. However, due to misalignment between the flyer and the impact surface, and between the surfaces of the samples, this is not achievable in practice. As a result, the shock wave can be slightly misaligned, resulting in deviations from the theoretical one dimensional compression with the addition of a shear strain-component. In addition, the shock wave will not break out uniformly across any surface. This is especially important for single crystal samples, where the crystal properties are anisotropic. Indeed, single crystal orientation can be measured

using diffraction techniques, in this case Laue back diffraction, thereby enabling the orientation of the shock to be tied directly to the crystal lattice directions. It is therefore vital to determine the impact wave tilt and arrival time on the driver-sample interface as well as the free-surface profile of the sample, which can only be achieved through fielding multiple probes. The details of the mathematical analysis to achieve this is presented in Section 3.1.

In order to economically field this system of probes a relay was designed, using a double telecentric design. A further advantage of this system is that the array can be magnified, meaning that the PDV points can be more closely packed on the sample surface than using bare-probes alone. The following proceedings paper, titled “Demonstration of a Telecentric Lens Relay System for PDV in Plate-Impact Experiments,” was published in the Proceedings of the 23rd Biennial Conference of the APS Topical Group on Shock Compression of Condensed Matter, describing this array and its use.

3.1 Computing Shock Parameters on the Driver-Sample Interface

The PDV array provides not only the arrival times at various locations of the shock wave, but imaging allows the specific locations of the probes on the surface, such as that in Figure 3.4b. Taking the central probe as the origin, the x - and z -coordinates of the radial probes can be taken, from which a more rigorous mathematical derivation of shock tilt and shock-wave arrival time can be determined.

3.1.1 Shock Tilt

To calculate the shock tilt θ , the y -coordinate, given by the time of arrival multiplied by the impact speed, gives a three dimensional location of the shock-wave [216], with a 2D representation shown in Figure 3.1. With the x - and z -coordinates taken from the imaged point locations, the three radial probes thus define a plane with normal vector $\hat{\mathbf{n}}$. The shock tilt, the angle θ between the normal of the shock plane and the surface normal can then be determined by

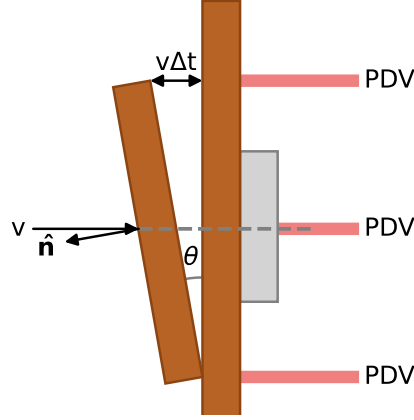


Figure 3.1: Schematic 2D representation of the tilt measurement. The flyer impacts the surface of the driver plate with a small angle (exaggerated in this plot to aid visualisation). This results in the shock wave arriving on the measured surface, upon which PDV is fielded, at different times. Where the spacing of the PDV points is known, the plane of the impact $\hat{\mathbf{n}}$, and therefore the tilt angle θ can be calculated.

$$\theta = \cos^{-1} \left(\hat{\mathbf{n}} \cdot \begin{pmatrix} 0 \\ 1 \\ 0 \end{pmatrix} \right) \quad (3.1)$$

which simplifies to

$$\theta = \cos^{-1} (\hat{n}_y) \quad (3.2)$$

The rotation ϕ can also be calculated using a similar technique, giving the angle of the projection of $\hat{\mathbf{n}}$ in the x - z plane through

$$\phi = \cos^{-1} \left(\frac{\hat{n}_x}{\sqrt{\hat{n}_x^2 + \hat{n}_z^2}} \right), \quad (3.3)$$

correcting for the quadrant, although for polycrystal samples this does not hold any meaning. The reference angle, the line of symmetry of the driver plate (see image inset in Figure 3.4b and Figure 4.3b), can be found by mounting a wire between the holes drilled on the driver plate to give the image rotation. The shock-wave can thereby be indexed relative to the driver plate, from which the sample's crystallographic rotation is also taken.

3.1.2 Shock-Wave Arrival

The three radial points can also be used to estimate the arrival time of the shock-wave into the sample, thus allowing the shock wave speed to be calculated. To determine this arrival time, a plane can be determined in a similar fashion to the shock tilt, although unlike the shock tilt, the y dimension is the time rather than displacement. These three points then form a plane, the y -coordinate of which can be evaluated at $x = 0$ and $z = 0$, the location of the central probe as defined previously. Thus a propagation time can be defined between this calculated arrival on the sample-driver interface and the sample free-surface.

Assuming the geometric centre of the three points radial is located on this origin, (most typically because the points are located rotationally symmetrically on a constant radius), this operation is equivalent to taking the mean arrival time of the three points. Generally, the points do not significantly diverge from the theoretical ideal locations, meaning the difference between the planar arrival time and the mean is smaller than the time resolution of the PDV used. However, the imaging of the PDV points allows for a more accurate calculation.

3.2 Demonstration of a Telecentric Lens Relay System for PDV in Plate-Impact Experiments

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Abstract

Photon Doppler velocimetry (PDV) has become a standard diagnostic in impact and shock experiments, due to its relative flexibility and ease in alignment compared to other optical diagnostics. The conventional approach to fielding PDV in plate-impact experiments is to place probes directly down-range from the projectile and target. In experiments such as these, probes are almost certainly destroyed, resulting in significant replacement cost particularly when multiple probes are used. To overcome these shortcomings, a free-space optical relay system has been developed to focus multiple channels of PDV from outside an experimental tank onto a target of reduced size than the probe array. This system has been successfully tested on plate-impact experiments measuring a Hugoniot state of polycrystal tin, and the peak elastic stress and spall strength in 6082 aluminium alloy, in both cases adding information about the shock tilt to the experimental data generated.

3.2.1 Introduction

First proposed by Strand *et al.* [217, 218], photon Doppler velocimetry (PDV) has become a standard diagnostic fielded in high-rate and shock experiments to measure the motion of a surface in a time-resolved fashion. Based on the Doppler shift of laser light reflected from a moving surface, PDV has been shown to provide a robust and flexible method of measuring velocity in the direction of the probe on a range of surface types, with tailorable velocity sensitivities, and across various timescales depending upon the limiting sampling rate of recording systems [219, 220].

Despite a wide range of configurations, PDV is typically fielded using optical probes or bare fibres to convey laser light to, and collect said light from, the measurement surface over relatively short stand-off distance [219]. While this is a convenient and reliable method of fielding PDV, it can prove costly when fielding multiple probes that are often destroyed in impact tests. Additionally, the size of the probe body used can place physical limits on the spatial arrangement of measurement points on the diagnosed surface.

Optical methods can be used to direct and focus multiple channels of PDV, improving the capabilities of the system at the expense of simplicity. For instance, a significant body of literature [221–226] has been devoted to developing fish-eye lenses to focus multiple channels from a single fibre bundle onto the inner surface of a curved bowl. As the surface implodes under an explosive driver, the collapse can then be measured at a number of points on the surface. In other examples, lens pair systems have been used to extend the distance between probes and the target. In work by Kaufman *et al.* [227], a 1:1 magnification double lens relay is used to remove the probes from the impact axis in gas-gun driven experiments by reflecting the lasers onto the measured surface using a pellicle beamsplitter. This helped avoid a secondary impact with the probes and their mounts, thereby facilitating clean recovery of the target for post-shock analysis. A lens pair system has also recently been used by Yang *et al.* [228] to focus eight channels of PDV onto a line along a target. This system scales-up (magnifies) the array meaning the points on the target are less tightly-packed than at the originating probes. Thus the probes are aligned as a group bundle.

In this paper, we describe an optical relay to focus the output for multiple PDV probes from outside an experimental tank onto a target smaller than the probe array. This means not only are the optical components reused in multiple experiments, but also that the probes can be kinematically mounted and fine-tuned with relative ease compared to an array of the same size as the target. By exploiting a simple 4f double-telecentric design to focus the array of probes, the cost of fielding PDV is reduced, with only a single mirror lost in each experiment. The use of

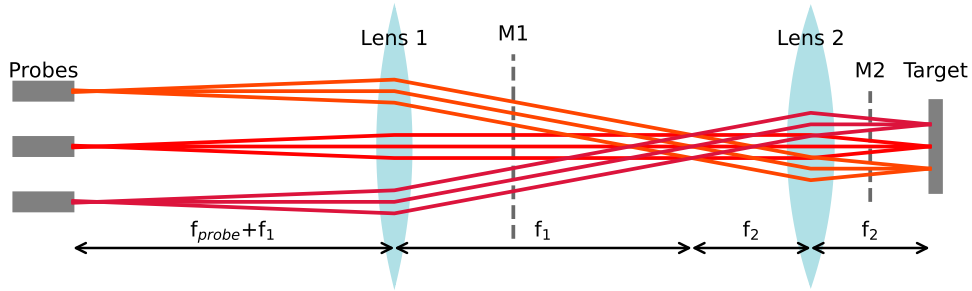


Figure 3.2: Schematic representation of rays in the double-lens PDV relay. By placing the components on their focal lengths, a central region with parallel rays is created between two regions with parallel beams. Symmetry can then be exploited to focus a large array of probes onto a small target. In the real design, mirrors are placed at the marked locations M1 and M2 to remove the components from the path of debris created in the gas-gun experiment.

the double-telecentric design also ensures the magnification is unchanged with target stand-off or motion. This array has been demonstrated on two plate-impact experiments, to gather surface velocities from an array of target points of interest, and to simultaneously diagnose impact tilt from differences in shock break-out time.

3.2.2 Double Telecentric Lens Design

The principle behind a double telecentric lens is relatively simple. By placing lenses and other optical elements spaced by their respective focal lengths (f), a robust relay is produced, shown in Figure 3.2. Note the parallel beams between the probes and lens 1 and between lens 2 and the target, while between the two lenses the rays in each beam are parallel and unfocused. Due to the symmetry of this central region, using lenses of different focal lengths results in a magnification (target PDV point spacing to probe spacing ratio) given by the ratio of the focal lengths of the two lenses. This design is relatively robust, with an image plane formed reliably in the desired location. The optical axes of the parallel beams are also perpendicular to the target meaning the system is optimally configured to collect sufficient return signal with only minor adjustments. Importantly, the parallelism of probe optical axes minimise relative shifts in measurement location throughout rear surface motion, an issue often associated with non telecentric imaging relays.

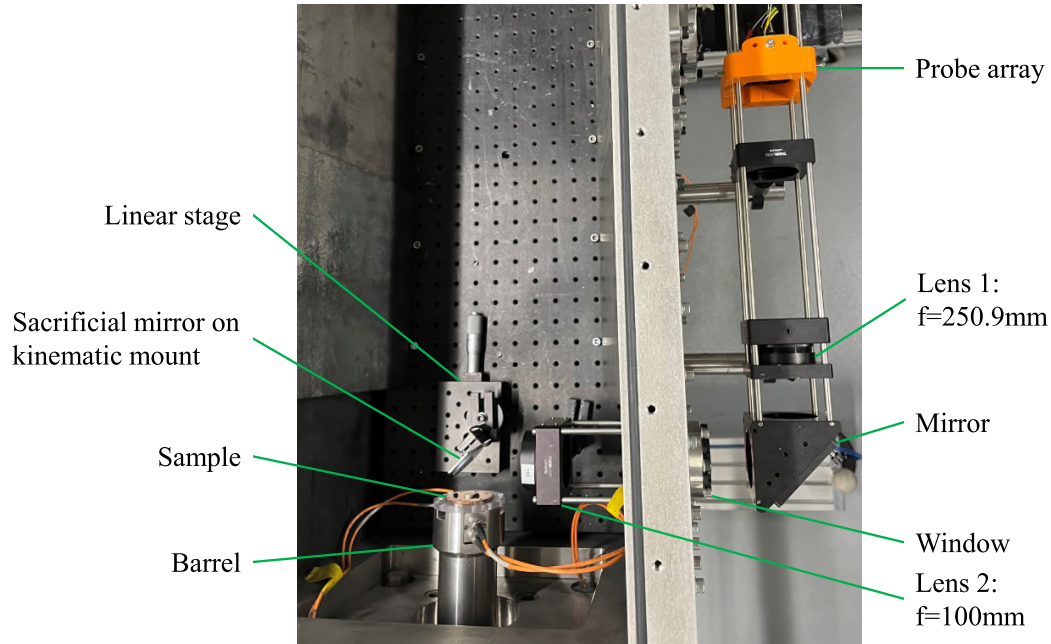


Figure 3.3: Picture of practical setup mounted onto a single-stage gas gun target tank.

3.2.2.1 Practical Setup

The practical setup of this telecentric lens system is shown in Figure 3.3 prepared for use in a plate-impact experiment on a single-stage gas gun. As shown, a mirror is used behind the target to remove most of the costly components from the path of the gas gun (M2 position in Figure 3.2). This mirror is mounted on a kinematic mount and a manual linear stage to align the beam path to the target. Due to the dimensions of the array, the second lens and probes can therefore be mounted outside the target tank, requiring a wedged window into the tank to remove back-reflections from the primary beam path. The wedged window and both lenses are all anti-reflective coated for the 1550 nm laser used by the PDV system. A second mirror (M1 position in Figure 3.2) is used outside the tank so the external components run parallel to the wall of the tank. Cage systems are used to ensure the lenses and probes can easily be moved parallel to the beam path. The probes are mounted in kinematic mounts on a 3D-printed block designed to fit the cage system. This gives a fine degree of angular and spatial positioning. To improve the economy of the design, pigtailed collimators are used instead of focusing probes, with half their ideal coupling length used as the focal length of the probe in Figure 3.2.

3.2.2.2 Aligning and Visualising the Array

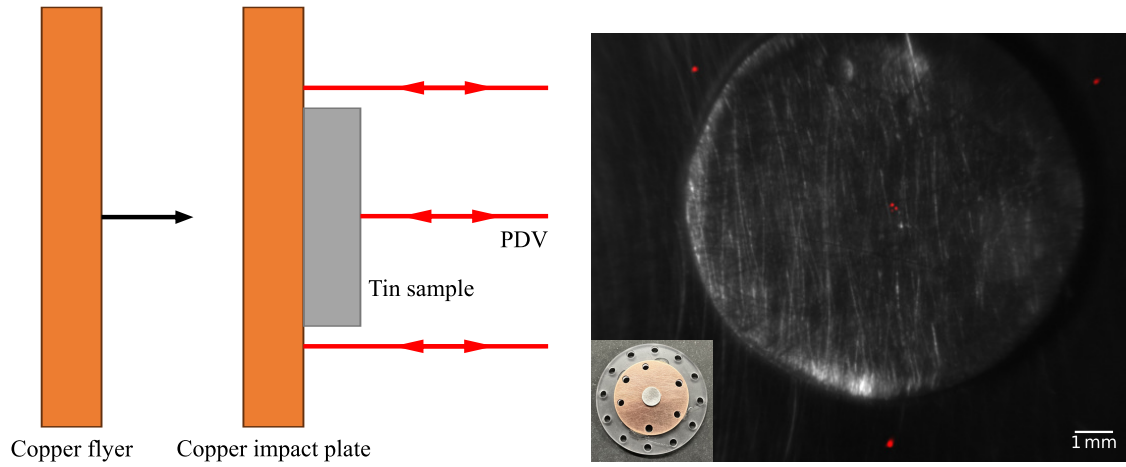
Due to the requirement for the optical path to be formed perpendicular and central to the target, aligning the system requires two main steps. Firstly, the cages and mirrors are aligned to a laser shone centrally down the barrel from the breech end. The spacing of the lenses and probes are taken along this laser path. After this the lenses and target are mounted, and the radial probes fine-tuned to ensure a return path.

By following these steps, the probes are reliably aligned to the centre of the target. However to ensure the PDV probes are correctly positioned, and in order to measure the spacing for the tilt analysis, the position can be visualised. To do this, two methods can be used. The simplest method to visualise the laser points is to use a visible-light laser, rather than the 1550 nm wavelength of standard PDV. However, the refractive index of the fused silica lenses varies with wavelength, meaning the paths are different for each wavelength. Alternatively, a pellicle beamsplitter can be inserted into the beam path, and a camera sensitive to both visible and infrared light used. While the beamsplitter will perturb the beam path slightly, this system allows for head-on visualisation as shown in Figure 3.4b. From this image the pitch circle diameter of the radial probes can be measured, with the result of 13.5 mm in the example shown, corresponding to the probe pitch circle diameter multiplied by the magnification.

3.2.3 Experimental Application

3.2.3.1 Plate Impact Experiment to Measure a Hugoniot State of Tin

The telecentric PDV imaging system has been demonstrated on two separate plate impact experiments showcasing its utility in measuring impact tilt and shock state, as well as detecting the onset of radial release. A “top hat” geometry with a C101 copper driver and 5N tin sample is shown in Figure 3.4a. The three radial probes have been used to quantify the arrival time in the tin, as well as the arrival tilt, while the central probe was used on the rear surface of the tin to measure break out, with the resulting velocity histories calculated using the short-time Fourier transform approach and plotted in Figure 3.5a. From this, the shock wave speed and

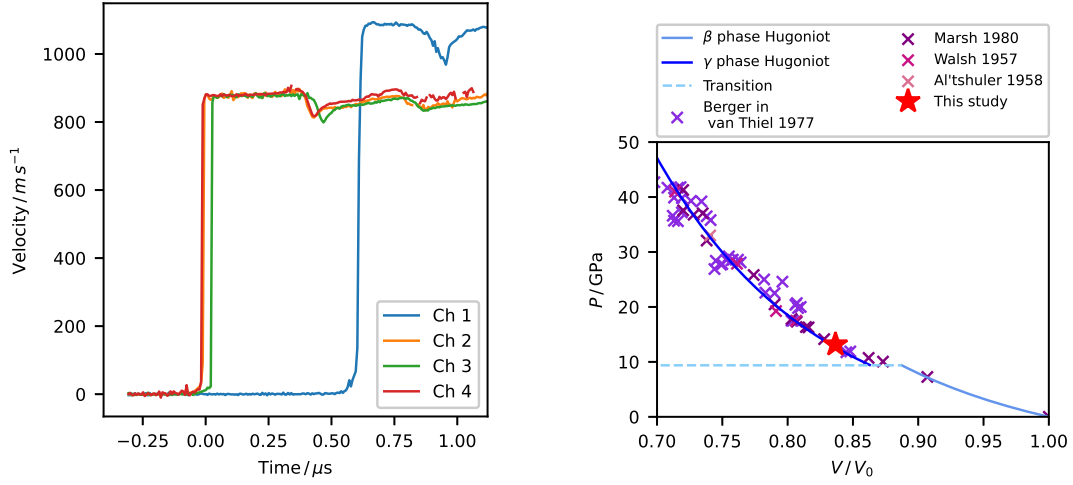


(a) Schematic geometry of the “top-hat” experiments, showing the copper flyer striking a copper impact plate to enable a Hugoniot state measurement in the tin sample.

(b) A composite image visualising the PDV points (recoloured red) on a “top-hat” sample using a camera sensitive to visible light and 1550 nm. The central probe on the sample (11.00 mm) and three radial probes focused on the driver plate can be seen. An image of the target on the barrel-interface plate is inset.

Figure 3.4: A “top-hat” experiment can be used to measure the shock wave speed and magnitude in order to determine Hugoniot state information. Knowing the spacing of the PDV points is key to measuring the impact tilt.

particle velocities can be determined, enabling other state information including the density and pressure to be calculated using the Rankine-Hugoniot jump conditions [6]. Assuming the isentrope does not significantly deviate from the Hugoniot, the internal particle velocity is half the peak release velocity on the free surface, with Figure 3.5a giving an internal particle velocity of 543 m s^{-1} . Taking arrival times from the 50% threshold of each wave, and taking the mean arrival time of the radial copper probes gives a shock wave speed of 3330 m s^{-1} . An initial density of 7.29 g cm^{-3} results in a pressure of 13.2 GPa and a relative volume of 0.837. This pressure-volume pair is plotted alongside literature values in Figure 3.5b. Similarly, the shock arrival times can be found on the copper driver using the radial probes. These can be directly used to determine the tilt of the shock wave as it enters the sample [216], which in this case is found to be 3.1 mrad.



(a) PDV velocity traces, with Ch1, the central channel, showing the breakout on rear surface of the tin sample, and channels 2-4 showing the radial breakout on a copper transmission plate. The impact tilt can be determined from the relative timing between these radial channels.

(b) Pressure vs. relative volume data determined experimentally using the PDV relay compared to literature values [164–166, 168]. The Hugoniot shown is calculated from Birch-Murnaghan parameters published by Mabire and H  reil [25].

Figure 3.5: Experimental data from a “top-hat” plate-impact experiment of a polycrystal tin sample.

3.2.3.2 Plate Impact Experiment to Measure the Shock Properties of an Aluminium Alloy

In order to further demonstrate the viability of the telecentric PDV image relay, we also present velocity histories obtained during a typical spall experiment. The relative dimensions were chosen to cause radial release of the outer radial array of 3 probes and hence challenge the optical efficiency. The spall experiment employed a symmetric impact of Al 6082-T6 aluminium alloy, and the impact speed is 353 m s^{-1} . From the PDV data, the peak elastic stress σ_E , initial plastic strain rate of shock $\dot{\epsilon}_p$ [229], spall strength σ_{spall} and spall pull-back strain rate $\dot{\epsilon}_s$ can be calculated based on the rear surface velocity [230–232].

The rear surface velocity is shown in Figure 3.6. Due to the tilt of the impact, the four probes detect the arrival time in sequence, with the tilt found to be 0.75 mrad . Taking a literature value of density for Al6082 alloy of 2.70 g cm^{-3} and longitudinal sound speed of 6400 m s^{-1} [236], gives a Hugoniot elastic limit of 0.68 GPa . The time taken for the change of particle velocity from $u_{E, \text{Min}}$ to $2u_{E, \text{Min}}$ during the main shock

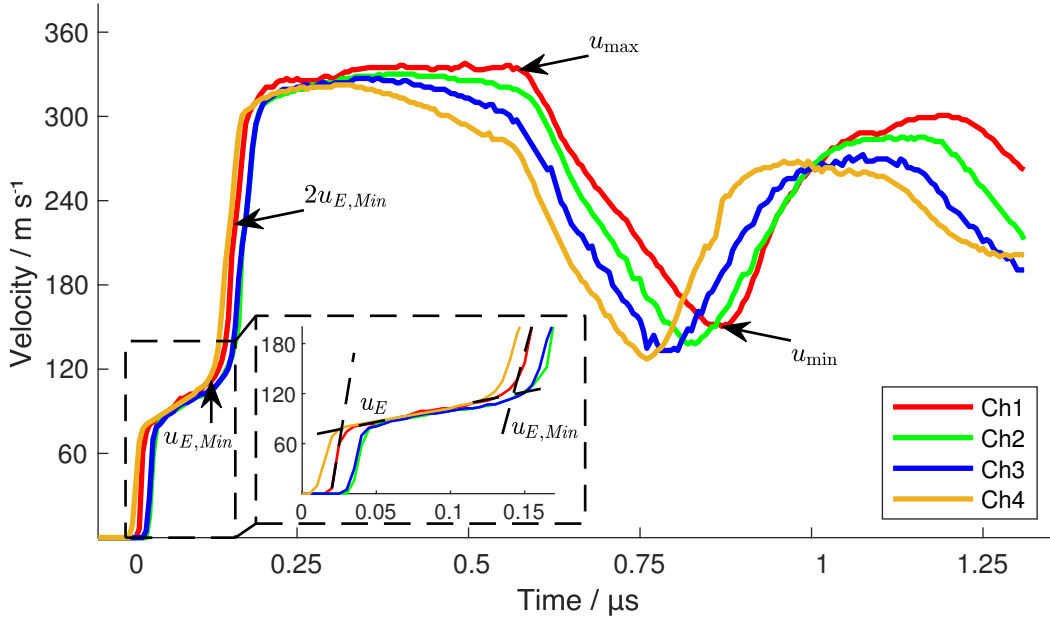
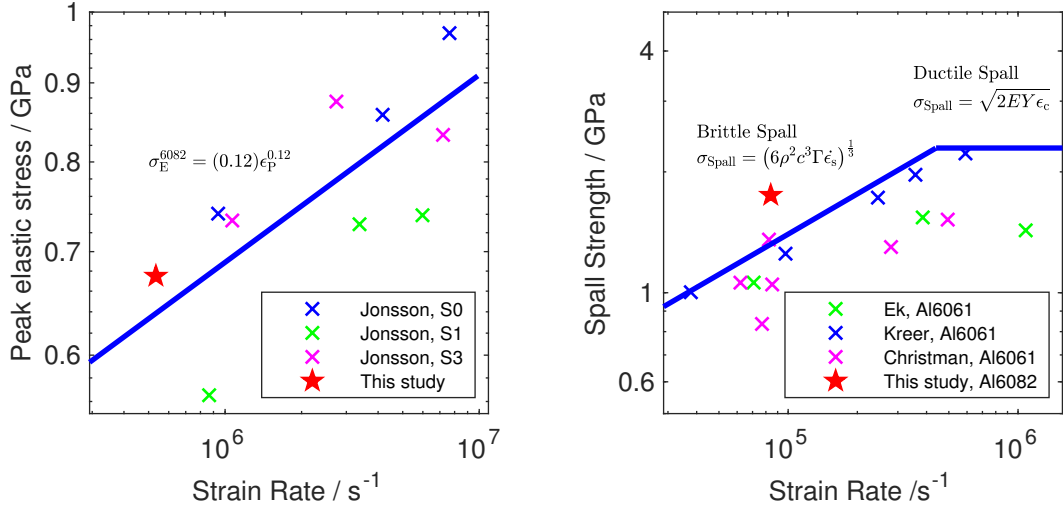


Figure 3.6: PDV velocity traces, with Ch1-4 showing the velocity of rear surface of Al6082-T6. The Ch1 shows the central channel. Due to lateral release from the edge of the sample, the three radial probes, Ch2-4, detect release earlier than the center signal Ch1.

wave gives an initial plastic strain rate of $4.98 \times 10^5 \text{ s}^{-1}$ following the approach of Smith *et al.* [229]. These results are plotted against literature values in Figure 3.7a.

In addition to the differences in the arrival time of the elastic precursor, it is also noted that the release from the peak shock state is different between all four probes, due to the three radial probes detecting the lateral release at different times. This serves to demonstrate the robustness of the optical relay to non-planar motion, where despite the lengthened optical path, adequate signal collection was maintained. The central channel, channel one, was in the centre of the sample where lateral release would arrive the latest at $\sim 1.81 \mu\text{s}$, long after the signature of spall following longitudinal release has arrived at the free surface. Analysing the spall pull back, the initial rear-surface velocity is 336 m s^{-1} , dropping to a minimum of 150 m s^{-1} , giving a spall strength [7, 237] of 1.75 GPa and a spall pull-back strain rate [231, 232] of $4.56 \times 10^4 \text{ s}^{-1}$.¹ Due to a lack of literature data of Al6082-T6

¹Consistent with the literature values, spall strength σ is calculated using $\sigma = \rho_0 c_l \Delta u_{fs} \frac{1}{1+c_l/c_b} + \Delta\sigma$, where $\Delta\sigma$ is a correction for spall depth $\Delta\sigma = 0.5 \frac{d\sigma}{dt} h (1/c_b - 1/c_l)$. Here, c_{extl} and c_{extb} are the longitudinal and bulk sound speeds, ρ_0 is the initial density, h the spall depth and Δu_{fs} the spall pull-back magnitude. Spall strain rate is calculated using Equation 5.6.



(a) Peak Hugoniot elastic stress in Al 6082 plotted against strain rate. S0, S1 and S3 refer to specimens that have been rotary swaged zero, once and three times respectively, as measured by Jonsson [216].

(b) Spall strength for Al6082-T6 as a function of strain rate from Al6061-T6 literature [233–235] presented in [174]. Spall strength fits based on the results of Grady [174].

Figure 3.7: Data from plate-impact spall experiments with an aluminium alloy sample.

spall, these results are compared to spall data for Al6061-T6, an aluminium alloy with a very similar composition and therefore mechanical properties to Al6082-T6, calculated by Grady [174]. The definition of strain rate used by Grady is determined by the approximate pulse duration at the flyer-sample interface, which in the case of the experimental results presented here, is found to be $8.4 \times 10^4 \text{ s}^{-1}$, shown in Figure 3.7b. It is found that the Al6082-T6 material has a higher spall strength than Al6061-T6 at the same strain rate.

3.2.4 Conclusion

A powerful but economical method of fielding multiple channels of PDV on small targets in gas gun-driven experiments has been demonstrated. This system uses a pair of lenses in a double-telecentric style design to relay and focus an array of probes onto the target, enabling multiple probes to be kinematically mounted outside the experimental tank. This enables the measurement of surface velocity on multiple points on the target, giving a wealth of data, including impact tilt, surface and shock speed velocities. By demonstrating these capabilities on a “top-hat” plate impact, a Hugoniot state measurement was made, while spall experiments using

this system measured the peak elastic stress and spall strength. As a result, this approach has the capacity to be a beneficial development for conventional PDV systems in a wide range high-velocity experiments beyond the plate impact equation of state measurement and spall strength determination demonstrated here.

3.2.5 Acknowledgements

This work would not have been possible without the assistance of Stuart Carter and the ISML workshop for help adapting the target tank to mount the array. The authors also gratefully acknowledge AWE for their continued support. UK Ministry of Defence © Crown Owned Copyright 2023/AWE.

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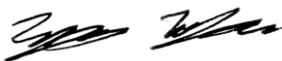
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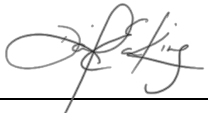
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Student Confirmation

Student Name:	Jasper George Threadingham		
Contribution to the Paper	Conceptualisation (lead); Data curation (lead); Formal analysis (lead); Investigation (lead); Methodology (lead); Writing – original draft (lead); Writing – review & editing (lead). Designed and built the methodology showcased, and demonstrated its use on the measurement of a Hugoniot state of tin.		
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Supervisor Confirmation

By signing the Statement of Authorship, you are certifying that the candidate made a substantial contribution to the publication, and that the description described above is accurate.

Supervisor name and title: Professor Daniel Eakins			
Supervisor comments I agree with the contributions as stated above.			
Signature		Date	10/02/26

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4

Shock Hugoniot Measurements of Single Crystal Tin

Contents

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The influence of the crystal orientation on the shock properties of tin has not yet been fully explored. Given the lack of single crystal HEL data, see Section 2.3.1, single crystal shock studies of tin are imperative to understand the elastic shock-properties of tin, and the transition from elastic to plastic behaviour. Furthermore, studying this region will help reduce the data scarcity of pressure-volume data, see

Section 2.3.2. Studying the $\beta \rightarrow \gamma$ phase transition in single crystals will also verify the molecular dynamics studies of Soulard and Durand [27], whose simulations suggest a strong orientation dependence of the transition. Given the large anisotropy of single crystals, an orientation dependence would not be unexpected, but remains to be experimentally determined. The following paper, titled “Effects of crystal orientation on the shock properties of single crystal tin,” published in the Journal of Applied Physics, presents a series of experiments undertaken to examine this topic.

After preparation and submission of this paper, further experiments were conducted. While the primary focus of this second series of experiments was to examine the spall strength of tin below the $\beta \rightarrow \gamma$ phase transition, the HEL and Hugoniot states can be analysed to further strengthen the depth of these observations. As a result, the paper below has been updated to include this additional data, with Tables 4.1, 4.2, 4.3 and Figures 4.5, 4.6, 4.7, and 4.8 updated to include the full dataset. The inclusion of the new data does not change the conclusions, but is presented in this format to concisely include the complete data without duplicating any plots and tables.

4.1 Influence of Sample Thickness on the Transition

In the paper, the influence of sample thickness on the $\beta \rightarrow \gamma$ is discussed, concluding that no quantitative differences are determined. With this conclusion, examining the PDV traces, aligned by shock break-out time to account for sample thickness, does not significantly contribute to the key messages of the paper. However, further elaboration of this data may still be useful, especially to compare phase transition models, and is therefore presented here.

As mentioned, no quantitative differences can be determined, with no divergence visible in Figure 4.9, for example. However, in Figure 4.1, it can be seen that the phase transition profiles show some changes beyond simple linear dispersion with thickness. In particular, the transition appears to show two segments with approximately linear gradients, the second segment having steeper gradient than

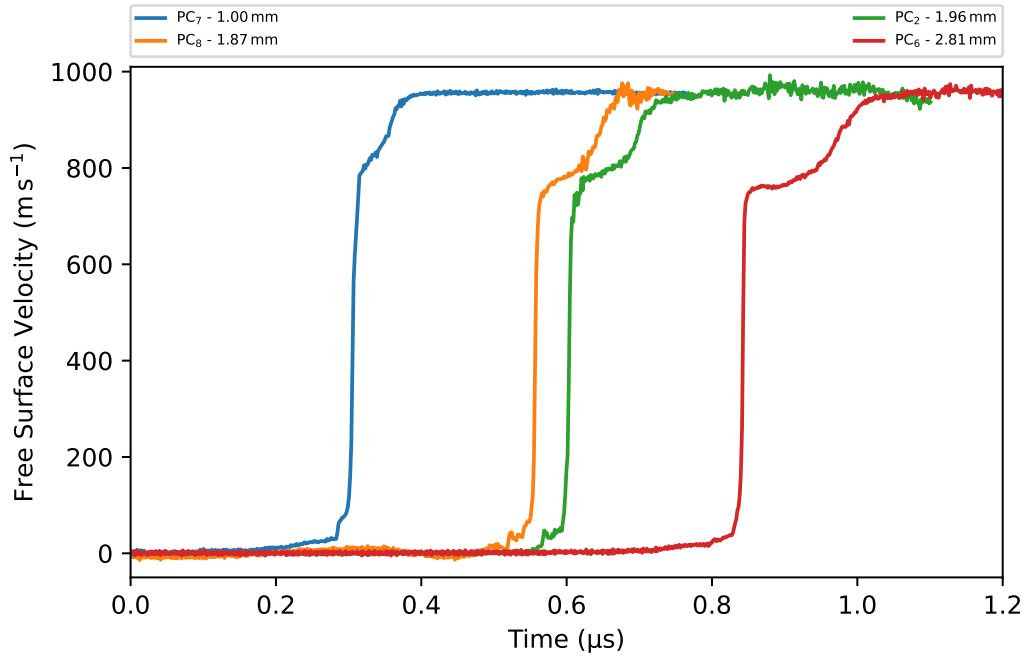


Figure 4.1: Free surface velocities for tin samples with different thicknesses. The reference time is taken as the arrival of the shock wave on the copper-tin interface. Sample thicknesses are listed.

the first. This is consistent between all the samples, although the gradients and magnitude change subtly between samples. Most noteworthy however, is the flat dwell period before the gradient rises in the thickest sample.

While the specific transition dynamics that result in this effect cannot be established, these plots suggest the transition rate, and possibly the mechanisms, develop with the sample depth. Similar observations are present in zirconium modelled with an differential-energy-dependent kinetic model [238], meaning transition dynamics models in tin, such as those of Cox and Robinson [137], may be well positioned to reproduce the observations made here.

4.2 Effects of Crystal Orientation on the Shock Properties of Single Crystal Tin

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Abstract

Tin is known for its asymmetric crystal structure and numerous solid phase transitions, with molecular dynamics studies suggesting the beta to gamma phase transition exhibits a strong orientation dependence. In this study shock compression experiments are conducted on tin single crystals and polycrystals to probe the effects of the crystal orientation on this phase transition through Hugoniot measurements, with peak pressures between 9 GPa and 13 GPa. A strong order-of-magnitude orientation dependence of the elastic limit is found, however, the transition and post-transition behaviour show at best only qualitative differences to the velocimetry profiles, with no quantitative variation. A dependence of the transition on the peak pressure is also observed. Explanations of these results based on potential transformation pathways identified through prior static high pressure work are discussed.

4.2.1 Introduction

Sitting between the semiconductors silicon and germanium, and metallic lead in the carbon group of the periodic table, tin shares some of the complexity of the former's phase diagrams with the simplicity of the latter [28, 239]. At low temperatures, tin transforms to a diamond α -phase [240], while, at increasing pressures, tin exhibits body centred phases (tetragonal (γ), orthorhombic, and cubic) and hexagonal close-packed phases [9, 34, 148, 150]. At ambient pressures and temperatures, tin is stable in the tetragonal phase β -tin [32, 33].

The asymmetry of this tetragonal crystal structure gives rise to differences in the properties of single crystals relative to the axis tested. For instance, the compressibility [10, 36–41] and electrical resistivity [10] of tin show significant orientation dependences, as does the thermal expansion [10–15], yield strength and post-yield behaviour [16]. Tin also exhibits a wide range of deformation mechanisms, such as twinning and slip [16–19, 32, 52, 53, 55, 56, 61–70, 76, 241], depending on the crystal orientation and deformation mode. The combination of these phenomena makes tin an ideal candidate material for investigating and understanding the effect directionality plays on the properties of materials.

A further property of tin is the relative accessibility of the change from its ambient tetragonal β phase to a high pressure body-centred-tetragonal γ phase. This $\beta \rightarrow \gamma$ phase transition has been mapped statically, at room temperature [29, 34, 86, 114, 122–124, 128], at elevated temperatures [88, 89, 91, 115], and under shock [130–132] and ramp [160] wave loading, as seen in Figure 4.2. The ramp wave data by Chong *et al.* are noted for being around 10 % to 20 % lower than those from conventional shock experiments [160], although the reason for this is unclear. Isentropic compression experiments have been used to probe the transition in single crystals of orientation (100) and polycrystal samples [126], showing no significant difference. Different orientations are not, however, compared. While these studies focus on determining the conditions to induce the phase transition, more recent static tests [34, 86] indicate that the phases co-exist over a range of pressures. Indeed, the static high pressure work of Fréville *et al.* [100] indicates that different crystals undergo the phase transition at different pressures during both the forward and reverse transitions. Hydrostatic pressure and temperature are, therefore, not the sole variables affecting the phase change. Similarly, hysteresis between forward and reverse loading has been observed in shocked polycrystals [23–25], including at elevated temperatures [127]. The double shock wave created by the transition has been experimentally measured for increasing peak pressures [135] and modelled using a mixture model where the phase-transformation rate is dependent on the free energy difference between the two phases [137]. A similar model [25, 134] has also

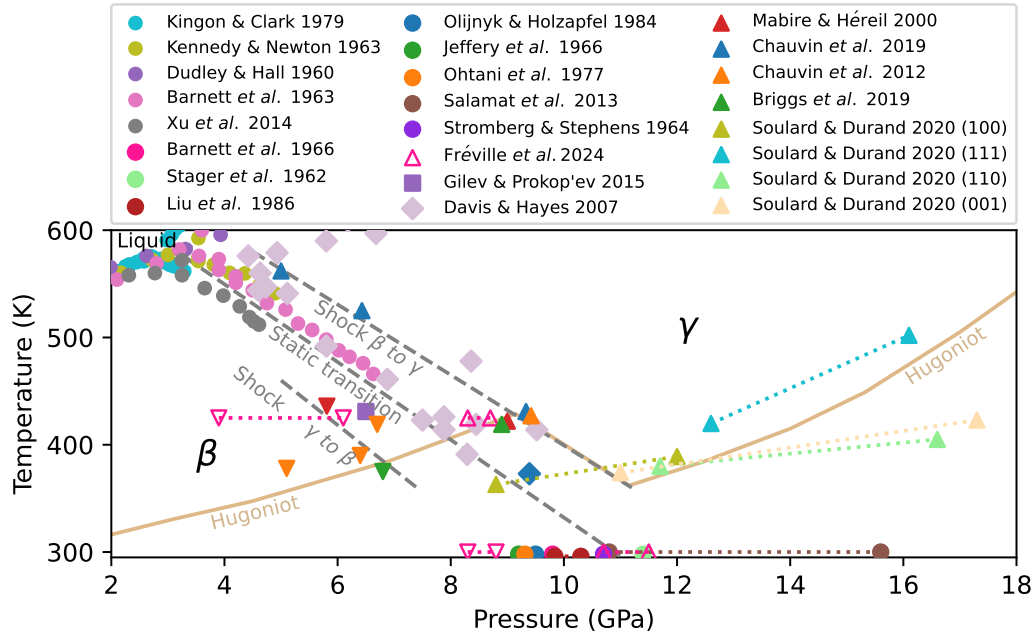


Figure 4.2: Literature summary of the $\beta \rightarrow \gamma$ phase transition in tin. Circles $\circ$ denote static tests, squares $\square$ denote ramp loading, diamonds $\diamond$ denote isentropic loading, upward $\triangle$ and downward ∇ triangles denote the forward and reverse transition, where filled triangles denote shock loading and unfilled triangles denote static loading. Grey dashed lines have been added as guides to the approximate location of the transition. Dotted lines denote a pressure range between which the transition takes place [23–25, 27, 29, 34, 86, 88–91, 100, 114, 115, 122–128].

been applied to the previously mentioned elevated temperature shock experiments [23, 127]. While this modelling approach is well able to match the shock wave surface velocity traces, further developments are required to replicate the static findings, particularly the mixed phase region.

Tin has been studied with X-ray diffraction under shock compression [24, 26, 140, 155]. This technique allows comparison between microstructure, with both Briggs *et al.* [24] and Bober *et al.* [26] finding a substantial grain size refinement across the phase transition. The cause of this phenomenon is unclear; Briggs *et al.* focus on the transition on release, while Bober *et al.* fields diffraction on jetted material, forming part of a larger body of literature examining jetting [26, 177, 187–193]. X-ray diffraction also allows for the orientation relationships between the phases to be studied. The single crystal of orientation (100) shocked by Chauvin *et al.* [140] hints

at the proposed mechanism of Musgrave [143, 145], although the mechanisms or post-transition micro-structure cannot be conclusively established. Notably, recent diamond anvil cell work by Fréville [146] suggests two transformation pathways between the β and γ phases of tin, including the mechanism of Musgrave [143, 145], depending on loading conditions, along with their respective symmetry twins.

Molecular dynamics simulations offer a potential approach to understand the behaviour of tin from the perspective of the interatomic interactions, with a number of potentials having been proposed for tin [201–208]. Perhaps the most significant, due to their ability to represent the directional nature of the bonds in tin, are the potentials of Nitol *et al.* [201], Ko *et al.* [202], and Baskes and Ravelo [203, 204]. The former uses machine learning to optimise the potential to best represent the behaviour of tin across all phases. The potentials of Ko *et al.*, and Baskes and Ravelo, however, take a conventional embedded atom model, with modifications to better represent the directionality of the bonds in tin. These two models perform best in the α and β region, to which the potentials have been fitted. However, the Baskes and Ravelo potential has been used by Soulard and Durand [27] to model single crystals under shock loading, from the ambient β phase, through the γ phase, and up to liquid. A strong orientation dependence of the $\beta \rightarrow \gamma$ phase transition and the compressibility of the orientations below the transition is found. Given the asymmetry of the crystal properties discussed above, and the one-dimensional compression generated under shock loading, this orientation dependence is not surprising, but experimental verification is required.

Experiments to directly study the orientation dependence of the transition in tin are, therefore, vital to confirm whether the $\beta \rightarrow \gamma$ transition is indeed sensitive to the crystal axis along which the shock propagates. An improved understanding of this phase transition can then be used as the foundation for improved modelling, from which the understanding of other materials can be developed.

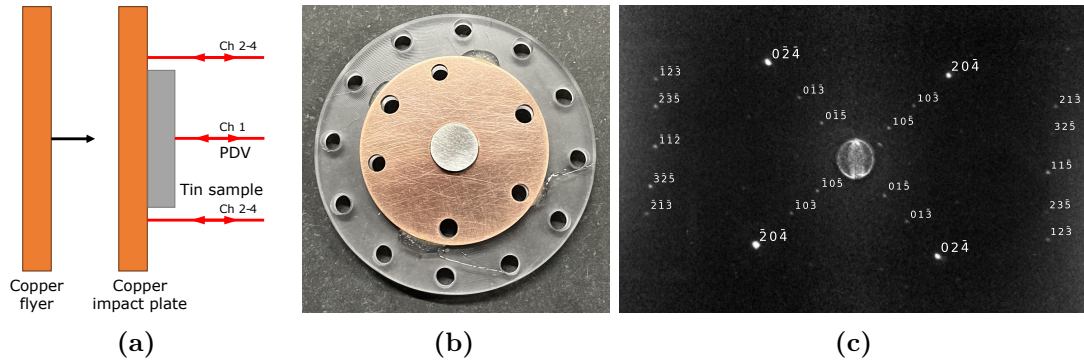
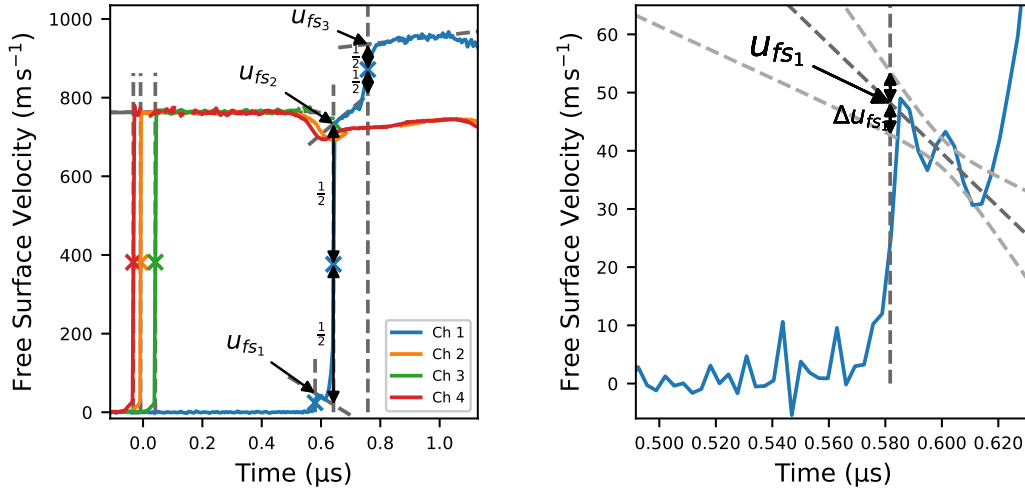


Figure 4.3: Experimental geometry and target used in the plate-impact experiments. By fielding PDV on the driver plate and sample, schematic in (a), the transmission speed of shock waves can be measured as well as the free surface velocities. Samples are indexed relative to the copper plate using Laue diffraction, with the indexed diffraction pattern of sample 001₁ shown (c). Similarly the impact tilt is also determined using the radial probes as the shock breaks-out from the copper. The pattern of holes with no rotational symmetry shown in (b) is used to reference the axial rotation of both the shock tilt and the crystal.

4.2.2 Methods

To probe the phase transition in single crystals of tin, a series of plate-impact experiments have been conducted. A ‘top-hat’ geometry, Figure 4.3, using a C101 copper driver plate allows the shock wave to be measured as it reaches the free surfaces of both the plate and the tin sample using a double telecentric photon Doppler velocimetry (PDV) relay (more information about the experimental methods can be found in our previous work, see Chapter 3). The PDV was sampled at a rate of 40 GS s^{-1} and analysed using standard short-time Fourier transform techniques [217, 218] with a time window length in the range 64 to 256 depending on signal to noise ratio. Using a 50 % overlap, the time resolution for the PDV measurements is therefore ranged from 0.8 ns to 3.2 ns. The most common setting used was a 128 sample window yielding a 1.6 ns resolution.

The output PDV traces are segmented, as shown in Figure 4.4, to give the arrival times t_n and free surface velocities u_{fs_n} for each wave, following an approach similar to Barker and Hollenbach [242]. This approach divides the profile into ideal trapezoidal sections, based on fitting straight-lines to the plateaus, with vertical jumps for the arrival of each successive wave. This approach has significant



(a) Segmentation to determine wave arrival times and surface velocities u_{fs} . (b) Uncertainty of the free surface velocity u_{fs} is determined by the 95 % confidence interval (light grey dashed lines) of the fit (dark grey dashed line).

Figure 4.4: Segmentation of the velocity trace for a single crystal 110₁ to determine the free surface velocities, wave speeds and their uncertainties. The central channel (Ch 1) measures the rear surface of the tin target, while three channels (Ch 2-4) measure the surface of a copper driver plate at points distributed radially around the sample.

advantages over other methods, such as gradient or velocity based thresholds as it can be used where the PDV signal has large noise or even drops out, as well as giving statistical uncertainties that well represent the measurements made.

The vertical jump is calculated by finding the midpoint between the adjacent plateaus, as shown in Figure 4.4a, thereby giving the arrival time of each wave. For almost all of the waves, especially the precursors and pre-transition waves, the time resolution of the PDV is unable to meaningfully resolve this jump: as a result the time resolution is used as the uncertainty for most arrival times. However, where the arrival is more diffuse, uncertainty is defined by the time difference between the trace reaching the 50 % and 75 % threshold. Higher upper thresholds, or thresholds set below 50 %, would often lie in the roll-off regions. This is typically only used for the post-transition wave, such as PC₄ and PC₅ in Figure 4.5. The uncertainty of the arrival time is, therefore, given by either time difference between the 50 % and 75 % threshold, or the time resolution of the PDV analysis, whichever is greater.

The free surface velocity of each wave is extrapolated from the fit to the specified arrival time. A 95 % confidence interval computed at the arrival time is used to determine the free-surface velocity uncertainty, an example of which is shown in Figure 4.4b. The signal noise, linearity, and degree of extrapolation are thus encapsulated within this uncertainty.

The regions for which the plateau segments are taken, from which the arrival times and free-surface velocities are calculated, are determined through a bounded brute-force optimisation where the free surface velocity uncertainty is minimised. For each possible fitting region, the fit is calculated, the arrival time of the wave determined, and the uncertainty evaluated, with the minimum uncertainty used to determine the best fit. Thus the number of fitted points, extent of extrapolation and quality of fit are all considered to analytically and repeatably segment the profiles into the various waves. Exact values and their uncertainties for the times and velocities are propagated for derived variables.

Three probes are fielded radially around the cylindrical target (Ch 2-4 in Figure 4.4a), from which the shock wave tilt relative to the single crystal normal can be calculated (data tabulated in Table 4.1). The arrival time between these three channels is taken alongside the measured spot positions imaged on the target to give the entry time t_{Cu} of the shock wave into the centre of the tin sample. The Eulerian shock velocity U_{s_n} can then be deduced using

$$U_{s_n} = \frac{y_n}{t_n - t_{Cu}}. \quad (4.1)$$

Given the free surface is moving, the distance the waves travel from the driving surface to the measured free surface y_n is only the measured initial thickness y_0 for the first wave. This displacement of the free-surface can be determined by integrating the free surface velocity. Assuming the free-surface trace does not deviate significantly from the linear fits, a trapezium rule approach can be used, meaning the surface displacement for each subsequent wave n is given by

$$y_n = y_0 + \sum_{i=1}^n \frac{(u_{fs_{i-1}} + u_{fs_{i-1}}^*)}{2} (t_i - t_{i-1}), \quad (4.2)$$

where $u_{\text{fs}_{n-1}}^*$ denotes the free surface velocity immediately before the following wave's arrival, thereby taking into account any decay or rise in the free surface velocity [243]. This correction is typically small where the waves arrive in close proximity, with a change in thickness of less than 0.5 % for the pre-transition wave and around 2 % to 3 % for the post-transition wave in the majority of samples, but can be up to 10 % for the post-transition waves in PC₄ and PC₅.

The approximation

$$u_p = \frac{u_{\text{fs}}}{2} \quad (4.3)$$

is used to determine the internal particle velocity u_p . This relies on the assumption that the release isentrope matches the loading Hugoniot. While this is not strictly true, the error introduced is small, usually less than 1 %, and systematic [243, 244]. The pressure P and specific volume V for the n th wave are, therefore, given by the Rankine-Hugoniot conditions:

$$P_n - P_{n-1} = \rho_0 \frac{V_0}{V_{n-1}} (U_{s_n} - u_{p_{n-1}}) (u_{p_n} - u_{p_{n-1}}) \quad (4.4)$$

and

$$\frac{V_n}{V_0} = \frac{U_{s_n} - u_{p_n}}{U_{s_n} - u_{p_{n-1}}} \frac{V_{n-1}}{V_0}, \quad (4.5)$$

where the subscript 0 denotes the initial conditions [243, 245].

Alongside polycrystal samples, three single-crystal orientations were tested: (100), (001) and (110). Polycrystal samples were cut from a stock bar with 5N purity and 11 mm diameter except sample PC₇ which was reduced to a 9 mm diameter. Single-crystal tin samples had a diameter of 10 mm. To determine the crystal normal relative to the driver plate, and, therefore, the tilt of the shock wave measured by the radial probes, the single-crystal samples were indexed using X-ray Laue diffraction, Figure 4.3c (rotation of crystal normal to specified direction presented in Table 4.1). The density of the crystals was also measured. By measuring the thickness of the samples as well as the distance between the rear surface of the sample and driver plate, an indication of the glue thickness between the copper and the tin is determined. In this case, the glue layer was not resolvable by the depth gauge used, and is therefore less than the resolution of 0.01 mm.

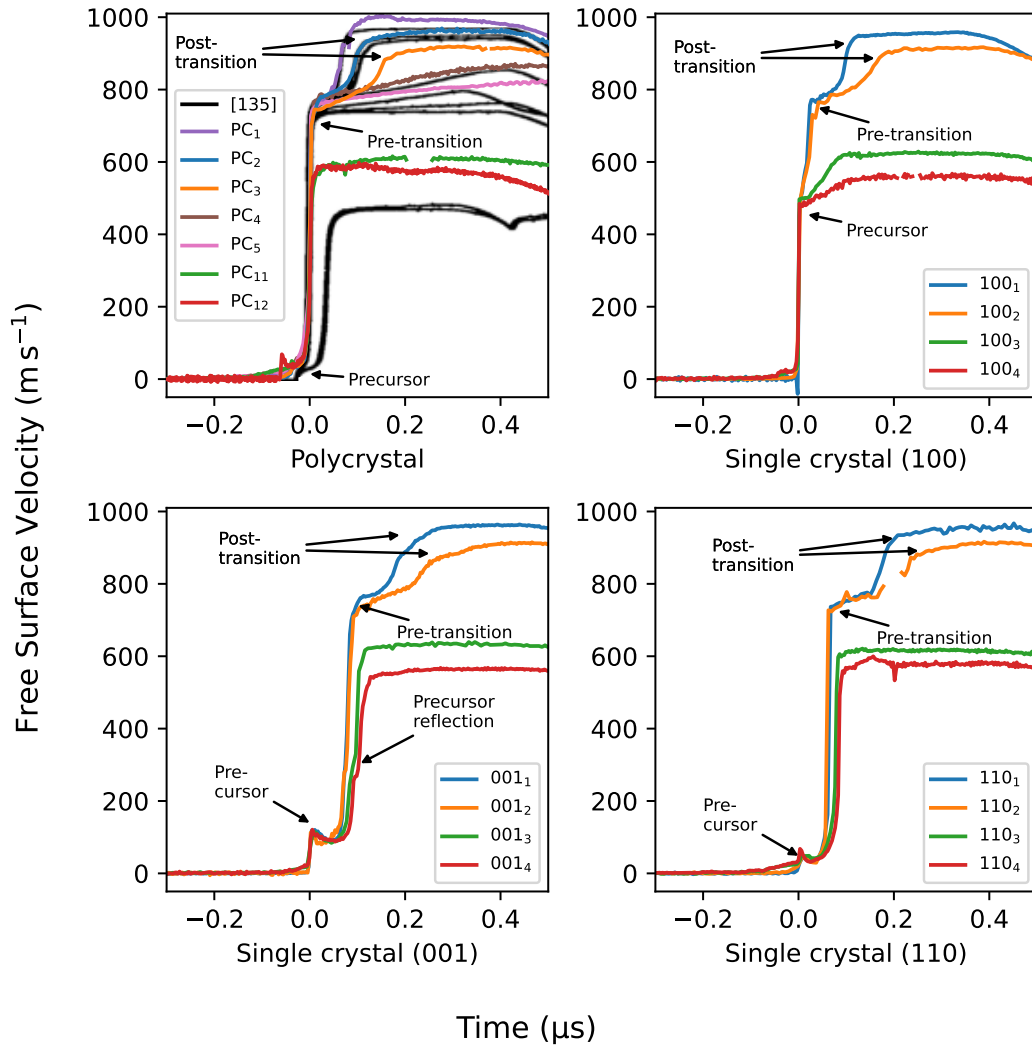


Figure 4.5: Rear surface velocity traces for plate impact experiments on a range of tin samples. Key features are indicated. Polycrystal data is in good agreement with the free surface velocity traces of Anderson *et al.* [135] (black lines), reproduced from W. W. Anderson *et al.*, “Phase transition and spall behavior in β -tin,” in *Shock Compression of Condensed Matter - 1999* (AIP Publishing, 2000) pp. 443–446, with the permission of AIP Publishing. Times referenced to pre-transition wave.

4.2.3 Results

The resulting free surface velocity traces are shown in Figure 4.5. The polycrystal traces are in good agreement with the literature results of Anderson *et al.* [135]. It is noted that these literature traces have been time adjusted, assuming linear dispersion rates, to account for the different thickness samples used. The pressure-volume states and shock experimental data are tabulated in Table 4.1 and plotted in Figure 4.6.

Table 4.1: Table of experimental and sample data.

Sample	Impact Velocity (m s ⁻¹)	Thickness <i>y</i> ₀ (mm)	Shock Tilt ^a		Crystal Rotation ^b			Precursor			
			Angle (mrad)	Rotation (rad)	<i>R</i> _x (mrad)	<i>R</i> _y (mrad)	<i>R</i> _z (mrad)	<i>U</i> _s (m s ⁻¹)	<i>u</i> _p (m s ⁻¹)	σ (GPa)	<i>V/V</i> ₀
100 ₁	775 ± 2	2.08 ± 0.01	5.0 ± 0.3	2.60 ± 0.07	-3 ± 2	871 ± 2	-3 ± 2	3.31 ± 0.04	225 ± 11	5.4 ± 0.4	0.932 ± 0.005
100 ₂	742 ± 1	2.08 ± 0.01	6.4 ± 0.5	-2.66 ± 0.07	3 ± 2	-1216 ± 2	-2 ± 2	3.31 ± 0.04	217 ± 8	5.2 ± 0.3	0.934 ± 0.003
100 ₃	519 ± 1	2.19 ± 0.01	2.5 ± 0.2	-0.57 ± 0.07	4 ± 2	865 ± 2	15 ± 2	3.30 ± 0.03	248 ± 3	6.0 ± 0.2	0.925 ± 0.002
100 ₄	470 ± 1	2.12 ± 0.01	5.1 ± 0.2	2.31 ± 0.03	4 ± 2	192 ± 2	2 ± 2	3.29 ± 0.03	236 ± 7	5.7 ± 0.3	0.928 ± 0.003
001 ₁	777 ± 1	2.10 ± 0.01	0.3 ± 0.2	-0.84 ± 0.67	5 ± 2	717 ± 2	-5 ± 2	3.71 ± 0.04	61 ± 4	1.7 ± 0.2	0.984 ± 0.002
001 ₂	743 ± 3	2.06 ± 0.01	4.5 ± 0.3	-3.06 ± 0.04	-9 ± 2	-236 ± 2	-7 ± 2	3.74 ± 0.05	58 ± 6	1.6 ± 0.2	0.984 ± 0.002
001 ₃	529 ± 1	2.09 ± 0.01	5.5 ± 0.2	-0.43 ± 0.04	1 ± 2	438 ± 2	9 ± 2	3.75 ± 0.07	62 ± 2	1.7 ± 0.1	0.983 ± 0.001
001 ₄	472 ± 1	2.04 ± 0.01	0.4 ± 0.2	-2.36 ± 0.35	8 ± 2	406 ± 2	9 ± 2	3.72 ± 0.03	56 ± 2	1.5 ± 0.1	0.985 ± 0.001
110 ₁	765 ± 2	2.15 ± 0.01	5.2 ± 0.4	1.31 ± 0.05	0 ± 2	665 ± 2	0 ± 2	3.70 ± 0.06	24 ± 6	0.7 ± 0.2	0.993 ± 0.002
110 ₂	742 ± 2	2.12 ± 0.01	8.2 ± 0.5	-2.77 ± 0.06	3 ± 2	-1466 ± 2	-2 ± 2	3.63 ± 0.08	26 ± 7	0.7 ± 0.2	0.993 ± 0.002
110 ₃	513 ± 1	2.04 ± 0.01	5.8 ± 0.2	2.90 ± 0.04	7 ± 2	-1388 ± 2	4 ± 2	3.60 ± 0.03	26 ± 2	0.7 ± 0.1	0.993 ± 0.001
110 ₄	485 ± 2	2.04 ± 0.01	7.4 ± 0.3	-2.82 ± 0.09	-1 ± 2	-861 ± 2	6 ± 2	3.66 ± 0.04	37 ± 4	1.0 ± 0.2	0.990 ± 0.002
PC ₁	809 ± 2	1.92 ± 0.01	2.2 ± 0.2	-	-	-	-	3.45 ± 0.03	19 ± 2	0.5 ± 0.1	0.994 ± 0.001
PC ₂	776 ± 1	1.96 ± 0.01	7.0 ± 0.5	-	-	-	-	3.46 ± 0.03	24 ± 5	0.6 ± 0.2	0.993 ± 0.002
PC ₃	746 ± 2	1.85 ± 0.01	6.5 ± 0.3	-	-	-	-	3.50 ± 0.07	20 ± 5	0.5 ± 0.2	0.994 ± 0.002
PC ₄	711 ± 2	1.75 ± 0.01	2.1 ± 0.3	-	-	-	-	3.44 ± 0.04	31 ± 6	0.8 ± 0.2	0.991 ± 0.002
PC ₅	646 ± 2	1.84 ± 0.01	1.1 ± 0.4	-	-	-	-	3.65 ± 0.11	17 ± 5	0.5 ± 0.2	0.995 ± 0.002
PC ₆	785 ± 3	2.81 ± 0.01	3.1 ± 0.3	-	-	-	-	3.52 ± 0.03	15 ± 4	0.4 ± 0.1	0.996 ± 0.002
PC ₇ ^c	782 ± 1	1.00 ± 0.01	5.0 ± 0.4	-	-	-	-	3.52 ± 0.06	37 ± 2	0.9 ± 0.1	0.990 ± 0.001
PC ₈	786 ± 1	1.87 ± 0.01	5.5 ± 0.4	-	-	-	-	3.61 ± 0.05	20 ± 3	0.5 ± 0.1	0.995 ± 0.001
PC ₉	770 ± 5	1.96 ± 0.01	6.0 ± 0.5	-	-	-	-	3.64 ± 0.05	23 ± 10	0.6 ± 0.3	0.994 ± 0.003
PC ₁₀ ^d	878 ± 9	2.02 ± 0.01	3.3 ± 0.4	-	-	-	-	3.56 ± 0.06	26 ± 2	0.7 ± 0.1	0.993 ± 0.001
PC ₁₁	504 ± 1	1.75 ± 0.01	1.4 ± 0.2	-	-	-	-	3.48 ± 0.04	25 ± 6	0.6 ± 0.2	0.993 ± 0.002
PC ₁₂	477 ± 3	1.78 ± 0.01	7.1 ± 0.3	-	-	-	-	3.58 ± 0.04	35 ± 5	0.9 ± 0.2	0.990 ± 0.002

^aShock tilt angle is given by the angle between the normal of the shock plane and the normal of the driver plate (y axis). The rotation is given as the rotation of the normal of the shock plane projected onto the driver plate, positive taken as clockwise from the vertical (z axis), defined by the mirror symmetry of the target (see Figure 4.3b)

^bCrystal rotation is given as the angle of the specified orientation relative to the normal to the driver plate (y axis). The x axis is horizontal, while the z axis is the vertical axis formed by the mirror symmetry (see Figure 5.6b).

^cWith a 2 mm thick flyer and 1 mm thick samples, typical spall behaviour is not observed in this sample.

^dRe-analysis of the data presented in Chapter 3. The pre-transition step is only measurable with high time resolution analysis, resulting in low statistical confidence for this measurement.

Table 4.1: (continued)

Sample	Density	Pre-transition				Post-transition			
	ρ_0 (g cm ⁻³)	U_s (km s ⁻¹)	u_p (m s ⁻¹)	P (GPa)	V/V_0	C_1 (km s ⁻¹)	u_p (m s ⁻¹)	P (GPa)	V/V_0
100 ₁	7.28 ± 0.02	3.22 ± 0.04	380 ± 3	9.0 ± 0.2	0.884 ± 0.003	2.97 ± 0.05	474 ± 1	11.0 ± 0.2	0.852 ± 0.003
100 ₂	7.28 ± 0.02	3.18 ± 0.04	376 ± 6	8.9 ± 0.3	0.884 ± 0.003	2.79 ± 0.06	453 ± 2	10.4 ± 0.3	0.856 ± 0.003
100 ₃	7.29 ± 0.02	3.06 ± 0.07	310 ± 1	7.3 ± 0.2	0.905 ± 0.002	-	-	-	-
100 ₄	7.29 ± 0.02	3.02 ± 0.09	279 ± 3	6.6 ± 0.2	0.914 ± 0.002	-	-	-	-
001 ₁	7.28 ± 0.02	3.26 ± 0.04	377 ± 2	9.1 ± 0.2	0.886 ± 0.002	2.91 ± 0.11	477 ± 1	11.2 ± 0.3	0.851 ± 0.003
001 ₂	7.28 ± 0.02	3.26 ± 0.05	361 ± 2	8.8 ± 0.3	0.892 ± 0.003	2.74 ± 0.20	454 ± 2	10.6 ± 0.4	0.857 ± 0.005
001 ₃	7.29 ± 0.02	3.21 ± 0.05	317 ± 2	7.6 ± 0.2	0.904 ± 0.002	-	-	-	-
001 ₄	7.29 ± 0.02	3.14 ± 0.04	281 ± 1	6.7 ± 0.2	0.913 ± 0.002	-	-	-	-
110 ₁	7.28 ± 0.02	3.35 ± 0.06	367 ± 2	9.0 ± 0.3	0.891 ± 0.003	2.97 ± 0.05	463 ± 2	11.1 ± 0.3	0.858 ± 0.003
110 ₂	7.29 ± 0.02	3.30 ± 0.07	361 ± 5	8.8 ± 0.4	0.891 ± 0.004	2.77 ± 0.11	454 ± 3	10.6 ± 0.4	0.857 ± 0.005
110 ₃	7.29 ± 0.02	3.18 ± 0.03	309 ± 1	7.2 ± 0.2	0.904 ± 0.002	-	-	-	-
110 ₄	7.29 ± 0.02	3.19 ± 0.04	287 ± 2	6.8 ± 0.2	0.911 ± 0.002	-	-	-	-
PC ₁	7.29 ± 0.02	3.24 ± 0.03	369 ± 4	8.8 ± 0.2	0.886 ± 0.003	2.99 ± 0.08	496 ± 1	11.5 ± 0.3	0.844 ± 0.003
PC ₂	7.29 ± 0.02	3.26 ± 0.03	378 ± 3	9.0 ± 0.2	0.884 ± 0.002	2.92 ± 0.06	473 ± 2	11.0 ± 0.2	0.851 ± 0.003
PC ₃	7.28 ± 0.02	3.27 ± 0.06	364 ± 2	8.7 ± 0.3	0.889 ± 0.003	2.72 ± 0.07	456 ± 2	10.5 ± 0.3	0.854 ± 0.004
PC ₄	7.28 ± 0.02	3.25 ± 0.04	387 ± 1	9.2 ± 0.2	0.881 ± 0.002	2.39 ± 0.05	429 ± 1	9.9 ± 0.2	0.863 ± 0.003
PC ₅ ^a	7.29 ± 0.02	3.26 ± 0.04	380 ± 1	9.1 ± 0.2	0.884 ± 0.002	2.15 ± 0.09	411 ± 1	9.5 ± 0.2	0.868 ± 0.003
PC ₆	7.29 ± 0.01	3.34 ± 0.02	378 ± 2	9.2 ± 0.1	0.887 ± 0.001	3.00 ± 0.06	478 ± 1	11.4 ± 0.2	0.853 ± 0.002
PC ₇ ^b	7.29 ± 0.02	3.28 ± 0.06	384 ± 2	9.2 ± 0.3	0.884 ± 0.003	2.89 ± 0.08	478 ± 1	11.2 ± 0.3	0.850 ± 0.004
PC ₈	7.29 ± 0.02	3.36 ± 0.04	374 ± 2	9.2 ± 0.2	0.889 ± 0.002	3.00 ± 0.07	483 ± 2	11.6 ± 0.3	0.852 ± 0.003
PC ₉	7.29 ± 0.02	3.36 ± 0.04	385 ± 3	9.5 ± 0.3	0.886 ± 0.003	2.98 ± 0.06	466 ± 2	11.2 ± 0.3	0.858 ± 0.003
PC ₁₀ ^c	7.29 ± 0.02	3.32 ± 0.04	390 ± 99	9.5 ± 2.6	0.883 ± 0.031	3.29 ± 0.06	536 ± 7	13.0 ± 0.4	0.839 ± 0.005
PC ₁₁	7.28 ± 0.02	3.23 ± 0.04	304 ± 1	7.2 ± 0.2	0.906 ± 0.002	-	-	-	-
PC ₁₂	7.28 ± 0.02	3.19 ± 0.04	295 ± 1	7.0 ± 0.2	0.909 ± 0.002	-	-	-	-

^aPhase transition incomplete. Post transition state taken as highest state reached before spall.^bWith a 2 mm thick flyer and 1 mm thick samples, typical spall behaviour is not observed in this sample.^cData initially presented in Chapter 3, re-analysed in Chapter 4. The pre-transition step is only measurable with high time resolution analysis, resulting in low statistical confidence for this measurement.

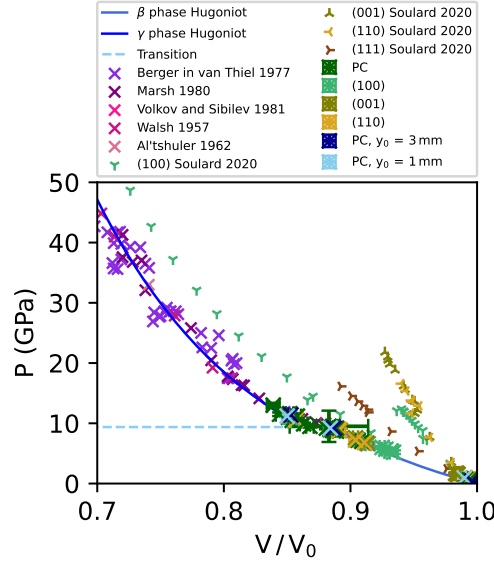


Figure 4.6: Pressure-volume data determined in this work compared to literature values [27, 164–168, 172]. Hugoniot and transition calculated from the Birch-Murnaghan model determined by Mabire and H  reil [25].

4.2.3.1 Precursor

The precursor waves in Figure 4.5 show considerable differences between orientations. Orientation (100), for instance, shows a precursor wave that is an order of magnitude higher than the other orientations, and significantly slower transmission speed.

The polycrystal precursor pressure is found to be somewhat larger than other Hugoniot elastic limit (HEL) literature values. For example, Mabire reports around 0.2 GPa [134], although the use of VISAR tuned to the phase transition may be less sensitive to the HEL. Similarly, Steinberg [159] reports a value of 0.24 GPa. The provenance of this is unclear, however, this value is similar to the value of 0.194 GPa found in ramp-wave tests [160]. Indeed, laser shock experiments at a much higher strain-rate indicate a value in excess of 1 GPa [161] and free surface velocities around 100 m s^{-1} [162]. The strain rates for these various measurements cannot be numerically compared, although the strain rate of the shock experiments conducted here would be expected to sit towards the centre of the range of literature strain rates, with the measured HEL reflecting this intermediate position. Notably the

Table 4.2: Elastic longitudinal wave speeds in single crystal tin measured at ambient conditions and under shock.

Orientation	Ambient conditions [43]	Shock - this study
(001)	3.446 ± 0.015 3.430 ± 0.010	3.71 ± 0.04
		3.74 ± 0.05
		3.75 ± 0.07
		3.72 ± 0.03
(100)	3.112 ± 0.015	3.31 ± 0.04
		3.31 ± 0.04
		3.30 ± 0.03
		3.33 ± 0.04
(110)	3.481 ± 0.018	3.69 ± 0.04
		3.63 ± 0.08
		3.60 ± 0.03
		3.66 ± 0.04

results presented here are good agreement with the particle velocity measurements in the plate-impact experiments of Anderson *et al.* [135].

The wave speed of this polycrystal precursor is a little faster than the ambient longitudinal sound speed of 3.43 km s^{-1} given by Marsh [164]. Notably the polycrystal sound speed is consistent with the sound speed of 3.54 km s^{-1} measured at 300 K and 0.75 GPa [89]. Similarly, the single-crystal wave speeds are faster than ambient ultrasonic velocity measurements determined using the pulse-echo technique [43], see Table 4.2. Orientation (100) exhibits the slowest sound speed under both shock and ambient conditions. However, while (110) and (001) have similar ambient longitudinal wave speeds, under shock loading, where (001) exhibits a significantly greater compression, the wave speed in (001) is larger. This observation implies, given the elastic longitudinal wave speed correlates with square root of the elastic modulus, the material stiffens with increasing stress.

Indeed, the apparent stiffness C can be calculated using the wave speed through

$$C = \rho_0 U_s^2. \quad (4.6)$$

The resulting apparent stiffness is presented in Table 4.3, compared to literature second order coefficients [39]. The second order coefficients clearly underestimate the stiffness, suggesting a third order model, where the material stiffness increases

as the material is compressed, should be used. For each orientation, the principal stress σ in the direction of the wave is given by [246]

$$\sigma_{100} = -\frac{V}{V_0}\eta \left(C_{11} + \frac{\eta}{2}C_{111} \right), \quad (4.7a)$$

$$\sigma_{001} = -\frac{V}{V_0}\eta \left(C_{33} + \frac{\eta}{2}C_{333} \right), \quad (4.7b)$$

and

$$\sigma_{110} = -\frac{\eta}{2}\frac{V}{V_0} \left(C_{11} + C_{12} + 2C_{66} + \frac{\eta}{4}(C_{111} + 3C_{112} + 12C_{166}) \right) \quad (4.7c)$$

where

$$\eta = \frac{1}{2} \left(\left(\frac{V}{V_0} \right)^2 - 1 \right). \quad (4.8)$$

Plotting these third order approximations in Figure 4.7 with second order coefficients from Rayne and Chandrasekhar [39] and third order coefficients from Swartz *et al.* [43] fits the measured data well. Thus, the strain-rate dependence on the elastic properties is likely indirect, with increasing strain rate increasing the threshold for plasticity, resulting in a larger magnitude, and, therefore, faster, elastic wave propagating in the material.

The agreement with the elastic model and statically determined wave speeds lends confidence to the conclusion that the precursor waves are indeed elastic. However, this does not explain the magnitude of the waves, especially in the (100) orientation. An unusually high precursor was reported by Davis and Hayes [126] in isentropic compression tests of (100) orientation single crystals, although “bond-layer thermal distortions” was the hypothesised cause. It is noted that applying compressive low-energy impact loads along the [100] direction drives plastic deformation through twinning [19]. The high pressure plastic behaviour of tin has been suggested to be dominated by twinning in split-Hopkinson pressure bar, pulsed power driven ramp, hole closure, Richtmyer-Meshkov instability and Taylor cylinder tests [48]. At cryogenic temperatures, this orientation is also found

Table 4.3: Table of elastic data.

Sample	Stiffness $\rho_0 U_s^2$ (GPa)	Second order	
		Coefficient	Value [39] (GPa)
100 ₁	79.8 ± 1.9	C_{11}	72.30
100 ₂	79.6 ± 1.6		
100 ₃	79.2 ± 1.1		
100 ₄	78.8 ± 1.2		
001 ₁	100.4 ± 2.1	C_{33}	88.4
001 ₂	101.9 ± 2.7		
001 ₃	102.3 ± 3.4		
001 ₄	101.0 ± 1.6		
110 ₁	99.9 ± 3.2	$\frac{1}{2}(C_{11} + C_{12} + 2C_{44})$	88.25
110 ₂	96.0 ± 3.8		
110 ₃	94.3 ± 1.5		
110 ₄	97.5 ± 2.1		

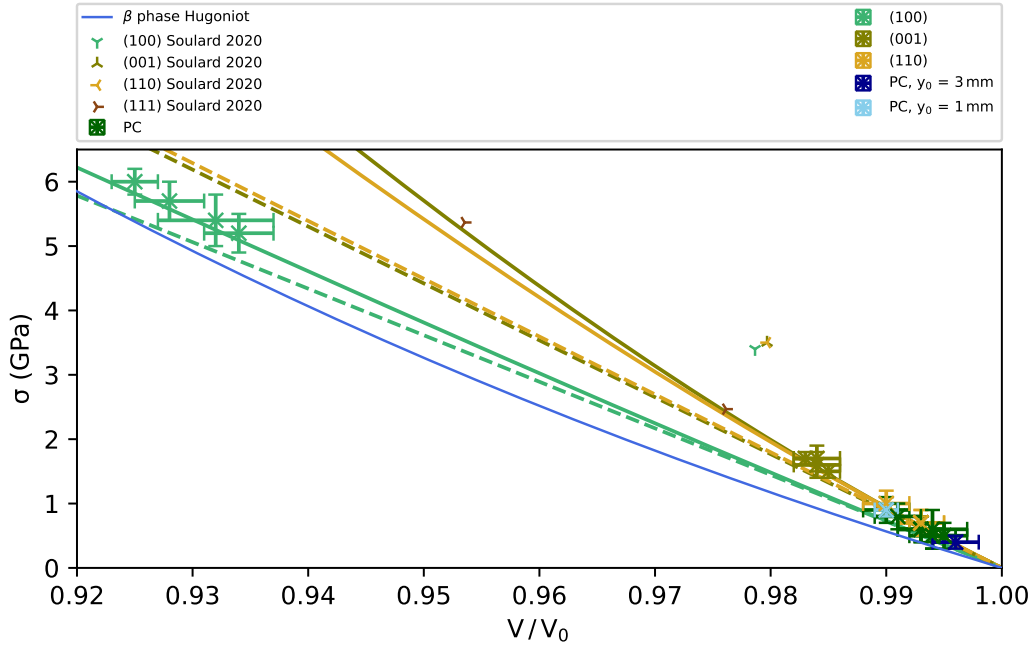


Figure 4.7: Precursor data plotted against a second order (dashed lines) and third order elastic stress-strain model (solid lines, same colour as experimental data of the same orientation) [246]. Second order parameters from Rayne and Chandrasekhar [39] and third order from Swartz *et al.* [43]. Hugoniot for the β phase calculated from the Birch-Murnaghan model of Mabire and H  reil [25]. Single-crystal molecular dynamics simulation states by Soulard and Durand [27] are included for comparison.

to fail without plasticity at a significantly higher pressure than other orientations [16], suggesting that twinning may require large stresses to initiate in time- or energy-constrained conditions in this orientation.

An estimation of the internal stresses in the samples at the precursor state, the state where the properties diverge from the predicted single-crystal elastic compression curve, can be determined using the previously mentioned third order elastic model. By taking the mean of the uniaxial strain at the precursor for the two tests per orientation, the stress field can be calculated, from which the resolved shear stress for the potential slip and twinning systems in tin can be estimated (detail in the Appendix). Seven slip systems have been experimentally observed in tin [16, 32, 52, 53, 55, 56, 61–70, 76, 241], alongside two twin systems [17–19], tabulated in Table 4.4. Critical shear stresses are somewhat uncertain and incomplete for these slip systems. Typical values are around or slightly below 1 MPa [16, 19, 52, 53]. Other systems have been proposed [55, 59, 68, 81], however measurements are scarce and observations inconsistent between sources. While it may be possible that these additional systems occur under specific loading and temperature conditions, only the core seven systems that are repeatedly observed are used for this analysis [16, 50, 247]. The results of this analysis are presented in Table 4.4.

It can be seen that more slip systems experience shear in the (110) orientation than the other orientations. The magnitude of the shear stresses for all systems is also lower in (110) than for the other tested orientations, while still being an order of magnitude larger than the critically resolved shear stresses determined for quasi-static conditions [16, 52, 53]. While rate effects may explain the rise in critically resolved shear stress, an order-of-magnitude rise is perhaps unexpected. Orientation (100) shows the highest resolved shear stresses, especially in the twinning systems. Notably, all the systems show a higher stress than in the (001). Assuming a simple Schmid model where the slip or twinning system activates when a critical stress is reached, any system that activates in (001) should activate well before reaching the HEL state measured in (100). A single critical value of resolved shear stress cannot therefore predict the onset of plasticity, and given the elastic anisotropy,

Table 4.4: Shear stress in GPa resolved for each slip system and crystal direction at the precursor state.

Slip system	Shock Orientation		
	(100)	(001)	(110)
(100)[001]	0	0	0
(010)[001]	0	0	0
(110)[001]	0	0	0
(1 $\bar{1}$ 0)[001]	0	0	0
(110)[1 $\bar{1}$ 1]	0.20	0	0
(110)[$\bar{1}$ 11]	0.20	0	0
(1 $\bar{1}$ 0)[111]	0.20	0	0
(1 $\bar{1}$ 0)[$\bar{1}$ 11]	0.20	0	0
(101)[10 $\bar{1}$]	0.91	0.39	0.09
($\bar{1}$ 01)[101]	0.91	0.39	0.09
(011)[0 $\bar{1}$ 1]	0.69	0.39	0.09
(0 $\bar{1}$ 1)[011]	0.69	0.39	0.09
(121)[10 $\bar{1}$]	0.66	0.28	0.17
($\bar{1}$ 21)[101]	0.66	0.28	0.04
(1 $\bar{2}$ 1)[10 $\bar{1}$]	0.66	0.28	0.04
($\bar{1}$ 21)[101]	0.66	0.28	0.17
(211)[0 $\bar{1}$ 1]	0.48	0.28	0.17
($\bar{2}$ 11)[0 $\bar{1}$ 1]	0.48	0.28	0.04
(2 $\bar{1}$ 1)[011]	0.48	0.28	0.04
($\bar{2}$ 11)[011]	0.48	0.28	0.17
(100)[010]	0	0	0.18
(010)[100]	0	0	0.18
(100)[011]	0	0	0.15
(100)[0 $\bar{1}$ 1]	0	0	0.15
(010)[101]	0	0	0.15
(010)[10 $\bar{1}$]	0	0	0.15
(301)[$\bar{1}$ 03]	0.90	0.42	0.10
($\bar{3}$ 01)[103]	0.90	0.42	0.10
(031)[0 $\bar{1}$ 3]	0.74	0.42	0.10
(0 $\bar{3}$ 1)[013]	0.74	0.42	0.10

factors such as strain rate and elastic potential energy may give better insights.

Behaviour more complicated than a classical activation stress model is, therefore,

observed, and warrants further study.

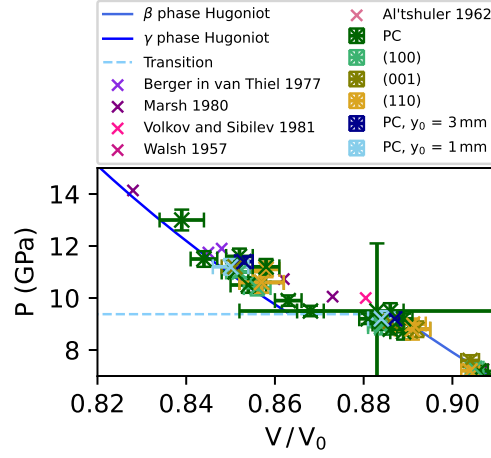


Figure 4.8: Pressure-volume data for the pre- and post-transition states plotted alongside literature values [164, 165, 172]. Hugoniot and transition calculated from the Birch-Murnaghan model determined by Mabire and Héréil [25].

4.2.3.2 Phase Transition

While these precursor waves show significant orientation dependence, the pre- and post-transition states show only qualitative differences. For instance, the transition waves of (001) appear to roll-off more than the other orientations, as shown in Figure 4.5, while (110) shows a much more angular profile. The profile of the post-transition wave in (001) could be indicative of a phase interface reflection, similar to that seen in iron [242]. Why this effect is orientation dependent is unclear, but could be tied to the elastic properties. However, the pressures and volumetric compressions appear to be clustered between the different orientations in Figure 4.8, without exceeding the experimental uncertainty. Consequently, any orientation dependences are likely to be small, if present at all.

The spread of impact velocities gives an insight into the possible pressure relationship of the transition. As plotted in Figure 4.9 alongside literature data, a linear relationship of the form

$$P_{\text{transition}} = mP_{\text{max}} + c \quad (4.9)$$

between the peak pressure and the pre-transition pressure is proposed.

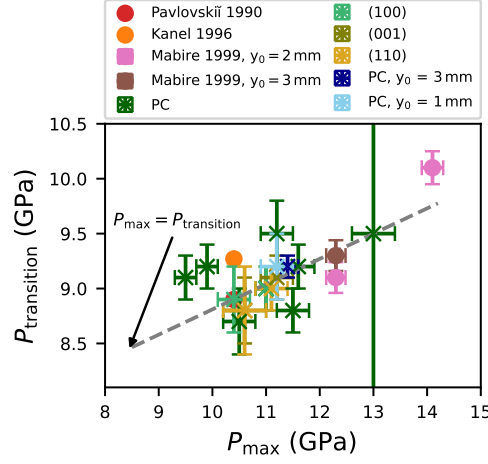


Figure 4.9: Comparison between the pre-transition wave pressure to the peak pressure state, including literature values [132–134]. A linear fit is taken (grey dashed line).

To test this hypothesis, a reduced χ^2 statistical test is conducted on the data presented in Table 4.1. Given the experimental uncertainties,

$$\chi^2 = \sum \frac{(P_{\text{transition}} - (mP_{\text{max}} + c))^2}{\Delta P_{\text{transition}}^2 + (m\Delta P_{\text{max}})^2} \quad (4.10)$$

estimates of the quality of the fit by taking the ratio of the distance of the data points from the line of best fit to their measured uncertainties. As a result, after dividing by the degrees of freedom (dof; the number of data points minus the number of fitting parameters), a good fit should reach a value close to unity. If the data deviate significantly from the proposed fit, the experimental uncertainties will be smaller than the deviation from the fit, meaning χ^2/dof would be much larger than one, and similarly if the uncertainties are larger than the deviation, χ^2/dof would be much smaller than one.

To determine the optimal fit, Equation (4.10) is minimised. Data from Mabire [134] are included in the fit, though it is noted that their uncertainties are blanket percentages and may not, therefore, represent the individual experimental measurement. The resulting relationship is found to be

$$P_{\text{transition}} = 0.23P_{\text{max}} + 6.55, \quad (4.11)$$

where both the gradient m and the intercept c are quoted to two decimal places, and in units of GPa for the intercept. An implication of this equation is that the minimum transition pressure, when the maximum pressure is equal to the transition pressure, is 8.5 GPa. This is between the values for an unstrained (8.7 GPa) and strained (8.3 GPa) crystal determined statically at a temperature corresponding with the approximate Hugoniot temperature by Fréville *et al.* [100]. The upper bound can be found by graphically applying the Rayleigh condition, yielding around 15 GPa. The value of χ^2/dof is found to be 1.3. This suggests the uncertainties are slightly underestimated, should the proposed fit be valid, especially given the lower uncertainties presented by Mabire and the large uncertainty of the transition state due to the small step in experiment PC₁₀. Further shock tests are required to better explore this relationship, especially to probe the minimum threshold pressure as well as measure the influence of other variables, such as initial sample temperature.

Modelling the transition using a transformation rate dependent on the Gibbs energy difference between the two phases, such as that by Cox and Robinson [137], has replicated the velocity profiles presented in Anderson *et al.* [135] that are indicative of the pressure dependence of the transition. A similar approach by Chong *et al.* [160], supported by ramp wave experiments, shows a decay in the pre-transition wave with the thickness of the sample. The data analysed here do not replicate this effect. From the thickest samples, 6 mm and 9.15 mm from Pavlovskii and Komissarov [132], to the thinnest, no significant deviations from the fit are visible.

4.2.4 Discussion and Conclusion

Through plate-impact shock experiments, the complex behaviour of tin has been unveiled. The precursor waves map well to elastic compressibility, but the plastic transition shows an unexpected order-of-magnitude difference between single-crystal orientations. Resolving the state at the peak of this precursor indicates that a simple Schmid model of critical shear stress does not well describe this behaviour. The causes of this phenomenon are unclear; however, it is noted that (100) has the highest compressibility, and, therefore, it is possible that the strain rate is

significantly higher in this orientation than in the other orientations. Although the strain rate is too fast to be accurately measured with the current PDV system, increasing the strain rate would be expected to increase the threshold required to slip or twin, especially if the change in strain rate were to move into the phonon-drag regime. Higher time resolution diagnostics to measure the corresponding strain rate of these waves may shed some light on any link between the yield and strain rate.

These experiments also cannot diagnose the exact mechanisms of plasticity. To confirm the plastic mechanisms that act at these high-rates, further experiments are required, such as shock recovery experiments. Further experiments and modelling are, therefore, required to better understand the phenomenon underpinning the elastic-plastic transition in tin.

While some qualitative differences are evident between the phase transitions in the different orientations, the experimental uncertainties are such that no statistically significant differences can be established. Consequently, it is concluded that there is a large orientation dependence of the HEL, above which there is not a significant orientation dependence of the properties. This is in contrast to previous molecular dynamics simulations [27], Figure 4.6, although it is acknowledged that the strain rates and sample volumes differ by many orders of magnitude. Indeed, considering Figure 4.7, some orientations follow the elastic compressibility curves, suggesting further refinements to the potentials are needed to better represent tin under shock loading.

Given that multiple transition pathways have been detected statically [146], it is hypothesised that multiple transformations of different pathways are initiated under shock loading. A variety of mechanisms may explain the qualitative differences between free surface velocity profiles (Figure 4.5), with different mechanisms being associated with different transformation rates. In addition, due to the reduction in density across the phase change, residual stresses will be generated at the interaction boundaries of these regions. These residual stresses will then further perturb the original post-transition orientation, leading to a broad distribution of polycrystalline orientations, with modes centred around an unperturbed post-transition orientation.

Thus single-crystal samples form a much finer microstructure across the transition; the results of Morgan *et al.* [139], Briggs *et al.* [24] and Bober *et al.* [26].

Alternatively, the lack of orientation dependence may lie with shock-induced recrystallisation prior to the phase transition. The proximity to melt may allow recrystallisation to occur after the HEL is reached [49], thereby losing any initial orientation relationship. Whilst verifying these hypotheses remains a challenge, *in situ* X-ray diffraction and soft recovery of shocked samples offer potential methods to study this phenomenon. These methods could help build a better understanding of the pre-transition plastic behaviour.

A plate-impact study has been performed on single-crystal tin to investigate the role of crystal orientation on the shock response. The HEL was observed to show a significant anisotropy of wave speed, compressibility and magnitude. Above this limit, the phase transition shows some qualitative differences but no quantitative variation between orientations. This behaviour points towards microstructural changes, notably plastic phenomenon such as recrystallisation as well as phase-transformation kinetics, eradicating any sample orientation effects. Further experimentation and modelling, such as sub-transition plate-impact tests, shock-recovery or *in situ* high-rate X-ray diffraction experiments, as well as molecular dynamics simulations, are, therefore, required to better understand these phenomena. Through this enhanced understanding and modelling capability, a better understanding of other materials may be developed.

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4.2.6 Author Declarations

4.2.6.1 Author Contributions

Jasper G. Threadingham: Conceptualisation (lead); Data curation (lead); Formal analysis (lead); Investigation (lead); Methodology (lead); Writing – original draft (lead); Writing – review & editing (lead). **Xuefei Liang:** Investigation (equal). **Edward Leggett:** Investigation (equal); Writing – review & editing (equal). **Liam C. Smith:** Investigation (equal); Writing – review & editing (equal). **Jeremy C. F. Millett:** Conceptualisation (supporting); Funding acquisition (equal) Resources (equal); Supervision (supporting). **Glenn Whiteman:** Conceptualisation (supporting); Funding acquisition (equal); Resources (equal); Supervision (supporting). **Viviane Peçanha-Antonio:** Methodology (supporting); Resources (supporting). **Andrew T. Boothroyd:** Methodology (supporting); Resources (supporting); Writing – review & editing (supporting). **David J. Chapman:** Conceptualisation (equal); Funding acquisition (equal); Investigation (equal); Supervision (equal); Writing – review & editing (equal). **Daniel E. Eakins:** Conceptualisation (equal); Funding acquisition (lead); Investigation (equal); Supervision (lead); Writing – review & editing (equal).

4.2.7 Appendix

4.2.7.1 Calculating Resolved Shear Stress

The shear on the slip plane is given by

$$\tau = \hat{\mathbf{d}} \cdot (\boldsymbol{\sigma} \cdot \hat{\mathbf{m}}). \quad (4.12)$$

The stress field $\boldsymbol{\sigma}$ can be determined using the aforementioned third order model, assuming one-dimensional strain. The principal components for each orientation are given by

$$\sigma_1 = -\frac{v}{v_0}\eta \left(C_{11} + \frac{\eta}{2}C_{111} \right), \quad (4.13a)$$

$$\sigma_2 = -\frac{v}{v_0}\eta \left(C_{12} + \frac{\eta}{2}C_{112} \right), \quad (4.13b)$$

$$\sigma_3 = -\frac{v}{v_0}\eta \left(C_{13} + \frac{\eta}{2}C_{113} \right) \quad (4.13c)$$

for orientation (100);

$$\sigma_1 = -\frac{v}{v_0}\eta \left(C_{13} + \frac{\eta}{2}C_{133} \right), \quad (4.14a)$$

$$\sigma_2 = -\frac{v}{v_0}\eta \left(C_{13} + \frac{\eta}{2}C_{133} \right), \quad (4.14b)$$

$$\sigma_3 = -\frac{v}{v_0}\eta \left(C_{33} + \frac{\eta}{2}C_{333} \right) \quad (4.14c)$$

for orientation (001); and

$$\sigma_1 = \sigma_2 = -\frac{v}{v_0}\frac{\eta}{2} \left((C_{11} + C_{12}) + \frac{\eta}{4}(C_{111} + 3C_{112}) + \eta C_{166} \right), \quad (4.15a)$$

$$\sigma_6 = -\frac{v}{v_0}\eta (C_{66} + \eta C_{166}), \quad (4.15b)$$

$$\sigma_3 = -\frac{v}{v_0}\frac{\eta}{2} \left(2C_{13} + \frac{\eta}{4}(2C_{113} + 3C_{123}) + \eta C_{366} \right) \quad (4.15c)$$

for (110), where all other terms are zero and η is defined in Equation 4.8. Using the mean volumetric strain for each orientation from Table 4.1 ($(v/v_0)_{(100)} = 0.933$, $(v/v_0)_{(001)} = 0.984$, $(v/v_0)_{(110)} = 0.993$), the second order parameters from Rayne and Chandrasekhar [39] and the third order parameters from Swartz *et al.* [43] yields

$$\boldsymbol{\sigma}_{100} = \begin{bmatrix} -5.17 & 0 & 0 \\ 0 & -4.73 & 0 \\ 0 & 0 & -3.08 \end{bmatrix} \text{ GPa}, \quad (4.16a)$$

$$\boldsymbol{\sigma}_{001} = \begin{bmatrix} -0.62 & 0 & 0 \\ 0 & -0.62 & 0 \\ 0 & 0 & -1.56 \end{bmatrix} \text{ GPa}, \quad (4.16b)$$

and

$$\boldsymbol{\sigma}_{110} = \begin{bmatrix} -0.47 & -0.18 & 0 \\ -0.18 & -0.47 & 0 \\ 0 & 0 & -0.25 \end{bmatrix} \text{ GPa.} \quad (4.16c)$$

The slip plane normal and slip direction are then given by

$$\hat{\mathbf{d}} = \frac{1}{\sqrt{(ua_1)^2 + (va_2)^2 + (wc_3)^2}} \begin{bmatrix} ua_1 \\ va_2 \\ wc_3 \end{bmatrix} \quad (4.17a)$$

and

$$\hat{\mathbf{m}} = \frac{1}{\sqrt{\left(\frac{h}{a_1}\right)^2 + \left(\frac{k}{a_2}\right)^2 + \left(\frac{l}{c_3}\right)^2}} \begin{bmatrix} \frac{h}{a_1} \\ \frac{k}{a_2} \\ \frac{l}{c_3} \end{bmatrix}, \quad (4.17b)$$

where (h, k, l) and $[u, v, w]$ have their usual crystallographic meaning and are tabulated in Table 4.4. Due to the large strains, especially in the (100) orientation, the strained lattice parameters are used. Assuming one-dimensional strain, for (100) $a_1 = a \frac{v}{v_0}$, $a_2 = a$ and $c_3 = c$, while for (001) $c_3 = c \frac{v}{v_0}$ and $a_1 = a_2 = a$. The loading case for (110) is more complicated, with the stretch field

$$\mathbf{F}' = \begin{bmatrix} \frac{v}{v_0} & 0 & 0 \\ 0 & 1 & 0 \\ 0 & 0 & 1 \end{bmatrix} \quad (4.18)$$

transformed into Cartesian coordinates using

$$\mathbf{F} = \mathbf{R}^T \mathbf{F}' \mathbf{R} \quad (4.19)$$

where

$$\mathbf{R} = \begin{bmatrix} \cos(45) & \sin(45) & 0 \\ -\sin(45) & \cos(45) & 0 \\ 0 & 0 & 1 \end{bmatrix} \quad (4.20)$$

to give unit cell parameters

$$\begin{bmatrix} a_1 \\ a_2 \\ c_3 \end{bmatrix} = \mathbf{F} \begin{bmatrix} a \\ a \\ c \end{bmatrix}. \quad (4.21)$$

Unstrained lattice parameters $a = 5.8318\text{\AA}$ and $c = 3.1819\text{\AA}$ are taken from Deshpande and Sirdeshmukh [11].

4.2.8 References

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5

Spall and Release Measurements of Single Crystal Tin

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While the shock experiments were not optimised to measure the release behaviour,

spall traces are present in the results, and offer exploration of the tensile failure of single- and polycrystal tin samples. Spall behaviour is not simply a property of the material, but also the microstructure and loading history, meaning a wide domain can be explored. In the following article “Influence of strain rate and phase history on the spall failure of single and polycrystal tin”, the spall behaviour of single crystal tin is measured and discussed. This article has been published in the *Journal of Applied Physics*.

Included in this chapter is discussion of sample PC₇. With a flyer thickness of 2 mm, and a sample thickness of 1 mm, non-standard spall behaviour is observed in this sample. With reflected release waves from the sample-driver interface, a highly complex loading scenario is observed. The complexity of the loading case in this sample requires significant extra explanation, and still does not fully account for the differences between this sample and others where more standard spall behaviour is observed. If included within the paper, the analysis of this single sample would account for nearly a fifth of the total written text, and yet does not contribute to the conclusions. This test is still interesting to examine, and is included here for completeness.

Similarly, release wave measurements are included and discussed here. The geometry of the experiments, intended to measure the Hugoniot, is stretched to evaluate the release wave speed. The incomplete nature of this data set, with only around a quarter of the data exhibiting measurable traces, represents a further weakness. The resulting data are presented here, with the aim of justifying the assumptions made within the main paper, and stimulating further investigation of the properties of single crystal tin through release wave measurements.

5.1 Release Wave Speed Measurements

In a similar manner to the direct wave speeds in Chapter 4, the release wave speed can be determined. Using the inflection at the start of the spall pull-back in both the copper driver and the sample, see Figure 5.1, an estimate of the transmission time Δt can be determined.

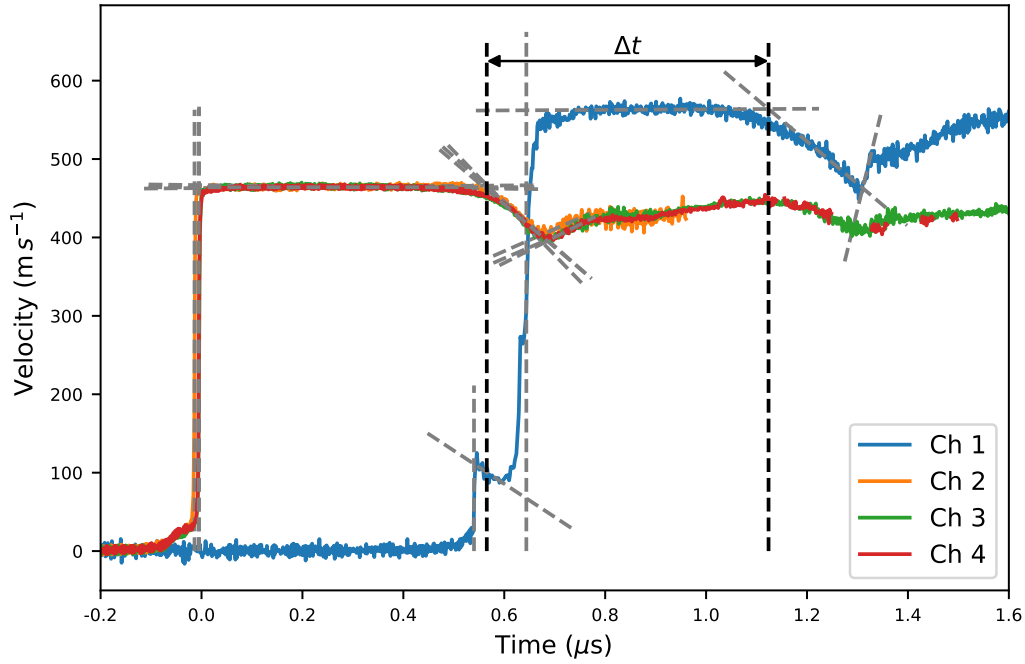


Figure 5.1: Free surface velocity profiles, showing release pull-backs on both the copper and tin free-surfaces, from which a release wave speed can be determined.

Applying Equation 4.1, allows for an estimation of the release wave speed. However, the loading case offers significant complexity, as shown in Figure 5.2. As a result, a thickness change correction similar to Equation 4.2 cannot be applied to the copper-tin interface due to the different impedance of that interface and the measured free surfaces, see Figure 5.2a. The measured free surface displaces faster than the interface, meaning the two are not directly comparable. Similarly, as shown in Figure 5.2b, the forward shock will be fully reflected from the free-surface, but only partially reflected from the copper-tin interface, see Figure 5.2c. The release from the rear surface of the flyer thereby propagates through different environments, resulting in different arrival times on the copper-tin interface and copper free-surface. As a result, only the Lagrangian sound speed, the velocity measured in the reference conditions, can be determined, with the original sample thickness used.

The complex interactions between release waves may act to cancel these two phenomena, meaning this measurement approximates the Eulerian sound speed (the sound speed defined in the compressed material) measured [157, 164, 248, 249] and

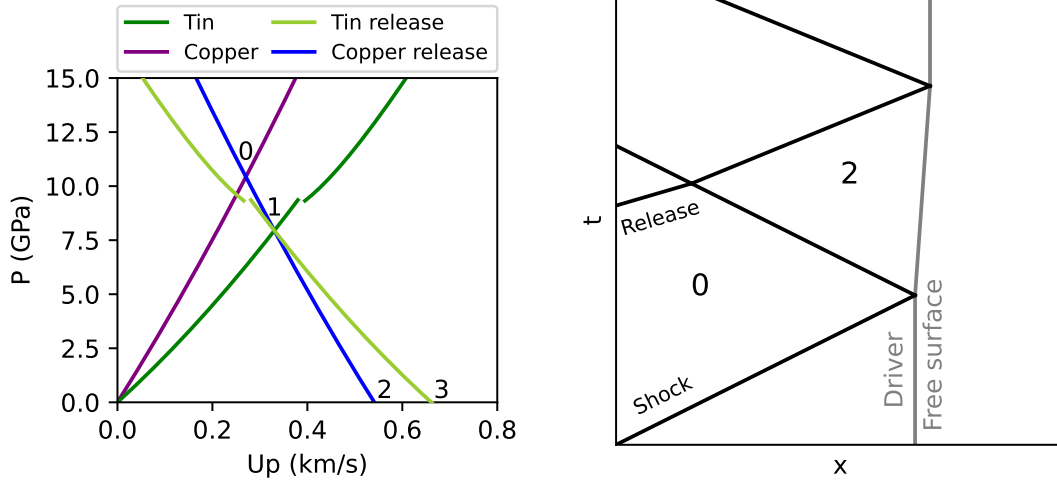
Table 5.1: Release wave velocities.

Sample	Transmission Time (μs)	Velocity (km s^{-1})
100 ₃	0.59 ± 0.02	3.72 ± 0.09
110 ₃	0.56 ± 0.02	3.65 ± 0.11
110 ₄	0.54 ± 0.03	3.76 ± 0.21
001 ₃	0.58 ± 0.02	3.60 ± 0.13
001 ₄	0.56 ± 0.02	3.65 ± 0.12
PC ₁₀	0.46 ± 0.02	4.37 ± 0.13

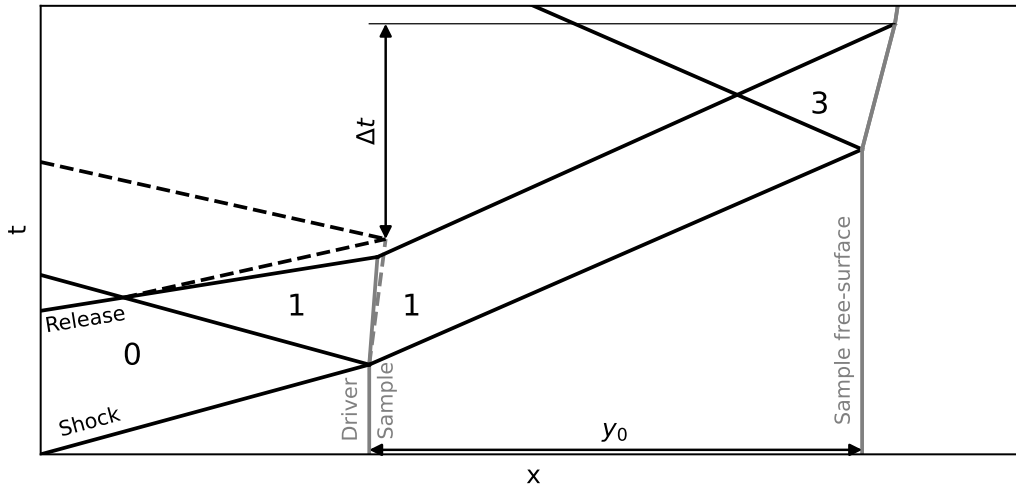
modelled [136] in polycrystals, but no direct comparison can be made. However, given the already established similarity in Hugoniot states between orientations, the behaviour of the interface is likely to be similar between samples shocked to the same pressure. As a result, the error induced by this assumption is likely to be systematic, and independent of the sample, allowing comparison of the release wave speeds to be made for samples shocked to the same pressure.

The location of the radial probes means these data cannot be determined for all the samples. Given all three channels are required to accurately determine the release wave breakout, a lack of signal in just one channel means no measurement can be made. The cause of the dropout could be for a number of reasons, such as jetting or lateral waves from the copper-tin interface, or simply the surface moving out of focus of the PDV probes. However, a number of samples retain sufficient information to be able to compare release wave speeds.

The results for the samples where the measurement can be made are tabulated in Table 5.1 and presented in Figure 5.3. The lower pressure tests roughly correspond in magnitude to existing literature Lagrangian wave speeds, while the higher pressure test PC₁₀ sits significantly higher than would be expected. The split-wave domain found in sample PC₁₀ may be behind this, with the release wave from the flyer passing through two release waves from the free surface. Similarly the two wave structure and larger shock pressures may cause significant deviation from the assumptions regarding sample thickness. In the other tests, below the phase transition, only one plastic wave is present, meaning these effects may be less pronounced.



(a) Example pressure (P) - particle velocity (u_p) (b) Displacement (x) - time (t) diagram for plot for states in the driver plate and sample shock and release in the flyer and driver plate during the first impact and release. where the latter does not about the tin sample.



(c) Displacement (x) - time (t) diagram for shock and release in the flyer, driver and sample. The case shown in b is shown here using dashed lines, from which the measured transmission time for the release wave through the sample Δt is taken.

Figure 5.2: Diagrams showing the internal states and wave positions during the release wave measurement. During the initial impact between the copper flyer and driver plate, a shock wave with state 0 is created. This wave propagates through the driver until it reaches an interface. In regions of the driver that do not abut the sample, the driver releases to zero pressure, state 2. Otherwise, the shock wave partially transmits into the tin, state 1. In both cases a leftwards moving release wave is created, however, the magnitude is different, meaning the rightwards moving release from the rear surface of the flyer will propagate differently, and over a different distance. As a result, it arrives on the measured surface of the driver at a different time to the arrival on the driver-sample interface. The differences in arrival times of the release wave on the driver and sample can therefore only approximate the release wave speed in the sample. In these plots, wave interactions are simplified, surface motion is exaggerated to aid visualisation and tensile failure is omitted.

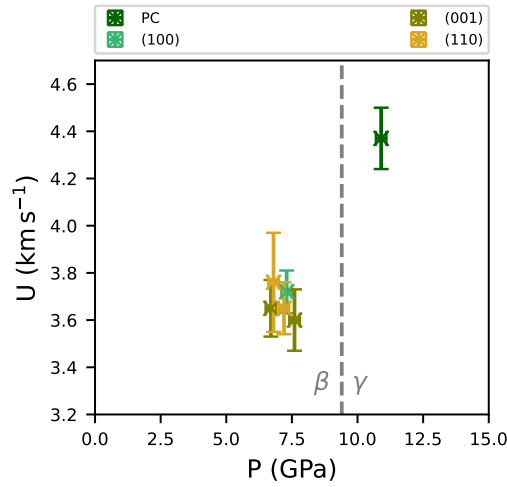


Figure 5.3: Release wave velocity U versus peak pressure P .

The lower pressure tests suggest that the sound velocities are very similar between orientations. Possibly (001) is slightly lower than the other orientations, although this is within the measured uncertainty. In literature static ambient measurements and in the shock Hugoniot, orientation (100) shows the lowest wave speed, around 10% smaller than (110) and (001), which are roughly similar, see Table 4.2. This supports the assumptions made in Section 5.3.2.1, that the sound speeds are no longer orientation dependent, and reinforces the conclusion that, above the elastic-plastic transition, no orientation dependence is observed.

A series of tests to directly measure the sound speed of shocked tin in single crystal samples would provide a better dataset. Traditional high-pressure sound speed measurements impact a thick window with the tin sample resulting in a single wave and its reflection [98, 156, 157], or calculate the speed from the release-shock overtake [248, 249]. Through these methods, a more robust dataset would be determined, which could strengthen the hypothesis that there is no longer any orientation dependence above the HEL. The uncertainty regarding the speed measured in PC₁₀ would also be resolved, and further data regarding the properties of γ tin determined.

5.2 Driver-Sample Interface Release Reflection

In sample PC₇, a 2 mm flyer is used to impact a 1 mm sample. This sample was prepared from the same stock material as the other samples, although the outside diameter was reduced to 9 mm. As a result of this experimental geometry, the release wave from the sample's free surface reaches the interface between the driver plate and the sample before the release from the rear surface of the flyer. The difference in shock impedance between the copper and the tin then causes the release wave from the sample to be partially reflected from the interface, as shown in the $x - t$ and $P - u_p$ diagrams in Figure 5.5. The release transition from γ to β is transmitted in this reflection back to the measured surface of the sample, before the reflected wave spalls the sample, prior to the arrival of the release from the flyer. The release transition thus appears as a step in the spall pull-back, see Figure 5.4.

Examining the release wave speed using the methods in Section 5.1, highlights the implausibility of the flyer's release wave reaching the measured surface. Noting that only channel 3 has a complete spall pull-back and rebound, but that the other radial channels begin to roll-off at approximately the same time, the time between roll-off on the driver plate and the phenomena in the velocity history of the sample can be taken. A velocity of 5.2 km s^{-1} would be required for the release wave from the flyer to cause the step midway through the pull-back, and 16.2 km s^{-1} to cause the initial drop from the peak shock state, both of which are far higher than are reasonably possible at these pressures (see Figure 5.3).

Noting that Figure 5.4 shows a pull-back and rebound, spall analysis offer some insights into the behaviour. From Figure 5.5a, by treating the release fan as a series of discrete wavelets, the release wavelets in-bound on the copper interface generate a smaller release travelling back to the free surface. Considering one such discrete wavelet, shown as a red arrow, it can be seen that the reflection generated when such a wave reaches the interface, shown as a green arrow, is significantly smaller in magnitude. Thus, in Figure 5.5b, crossing a primary release wave on the red arrow results in a greater pressure drop than crossing the reflected wave (green arrow). From the dimensions of the triangle formed in Figure 5.5a by states 2,4 and

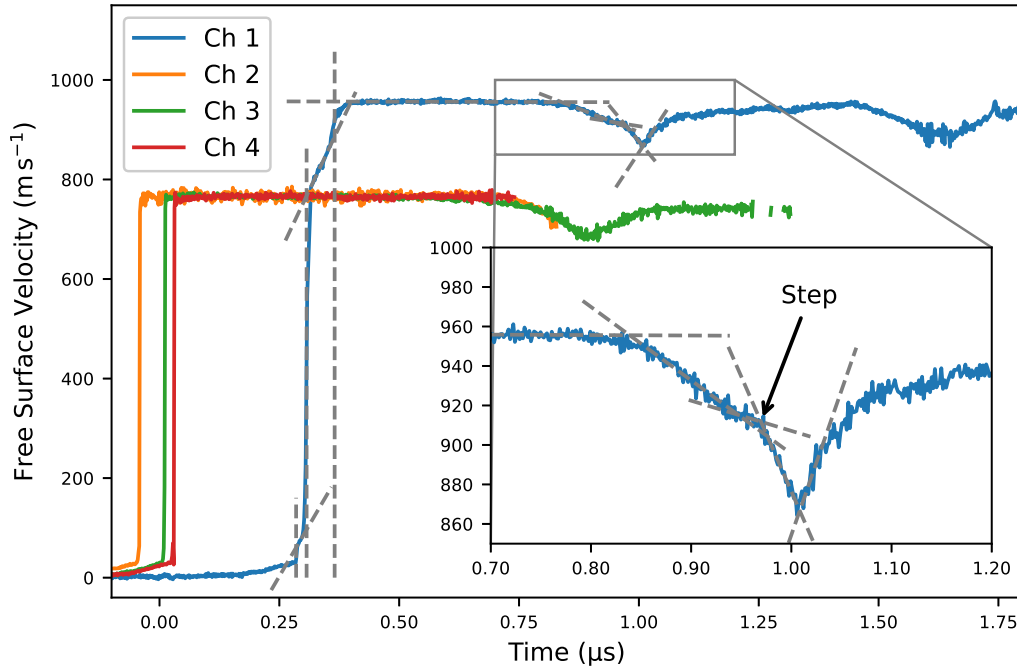
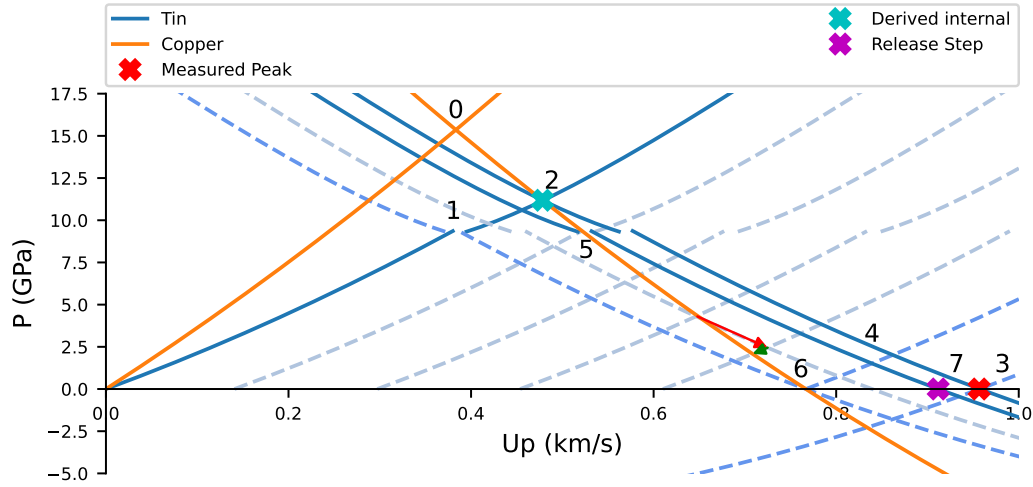


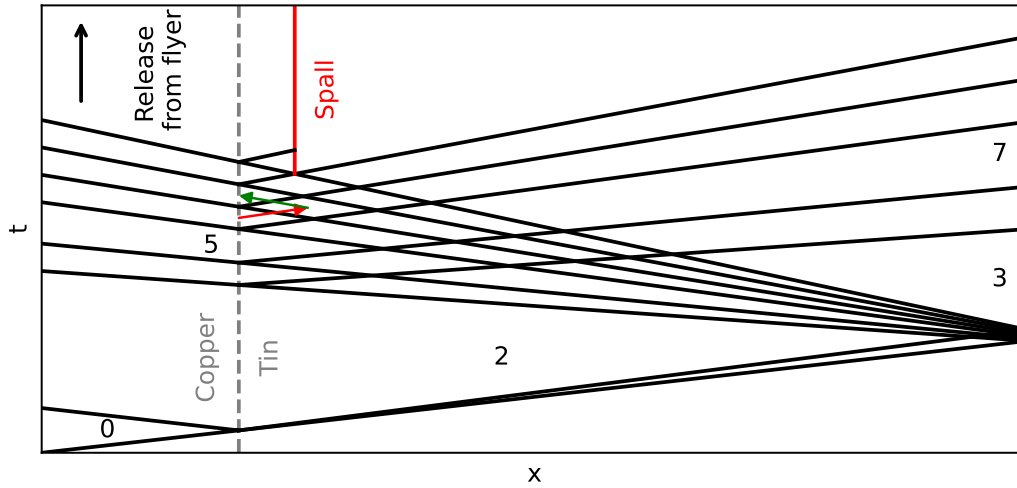
Figure 5.4: Rear surface velocity measurement for sample PC₇, with the spall pull-back detail shown inset. Time is referenced to the arrival time of the shock as it exits the driver plate into the sample.

6, the ratio in magnitude is approximately a factor of four different. Consequently, in Figure 5.5b, the trailing wavelet from the free surface corresponds with the lowest pressure for any given time step, and is therefore presumed to fail first. The spall trace in this dataset can therefore be analysed following conventional techniques, with data in Table 5.2. However, the pull-back magnitude is still lower than in similar samples that spall conventionally.

The proposed spall corrections cannot be readily applied to this geometry. Calculating the thickness from the primary reflection (Equation 5.4), for instance, yields a spall depth of 1.08 mm, exceeding the initial thickness of the material. This suggests that the sound speed approximations overestimate the speed of the waves travelling within the material. Notably, using c_b for both the release from the free surface and the re-compression wave yields a depth of 0.98 mm. From Figure 5.5, the wavelet reflected from the driver-sample interface that induces spall must travel slightly faster than the rearmost release, which is assumed to travel at the bulk sound speed. A depth of 0.98 mm would imply that the reflected



(a) Pressure - particle velocity plot showing the states reached during the shock experiment in a thin tin sample alongside measured free surface velocities.



(b) Displacement - time plot showing the evolution and interaction between the various waves. Note plot is exaggerated to highlight details.

Figure 5.5: During the shock experiment, a series of states are reached in different regions of the target. The first state reached is the initial shock in the copper, state 0. The copper then releases into the tin sample, creating a split wave due to the forward phase transition, states 1 and 2. When the corresponding waves reach the free surface, a release fan is generated, between 2 and 3. This release, plotted here as a series of discrete wavelets further interacts with the copper driver. Due to the higher impedance of the driver, a series of secondary release waves are reflected back into the tin sample. The pause in wavelet corresponding to the reverse transition (state 5) reaches the free surface as state 6. The interaction between the copper and tin eventually reaches a state of negative pressure, state 6. Depending on the strength of the glue layer, the sample and driver will separate. The arrival of the remaining release (4 to 3) then result in tensile stresses, causing spall to form.

Table 5.2: Spall data for sample PC₇.

Sample	Pull-back magnitude m s^{-1}	Pull-back gradient $\times 10^8 \text{m/s}^2$	Spall strength GPa	Spall strain rate $\times 10^8/\text{s}$
PC ₇	87 ± 4	10.4 ± 1.0	0.89 ± 0.04	1.86 ± 0.18
Primary mm	Spall depth Secondary mm	Corrected spall strength GPa	Corrected Spall strain rate $\times 10^8/\text{s}$	
1.08	1.07	1.33	9.51 ± 0.93	

wavelet travels 1.02 mm, and is therefore around 4% faster than the direct wavelet. A value of 1.07 mm is found by calculating the spall depth from the post-spall reverberation (Equation 5.5). It is therefore concluded that the accuracy of the sound speeds used are insufficient to precisely define the spall depth. Whether this observation effects the other spall tests is unclear: the spall behaviour in this test does not well agree with the other tests.

As mentioned previously, the interface between the copper driver plate and the tin sample may have an influence, especially on the corrected spall strain rate. It might be convenient to assume that the dispersion occurs over twice the sample thickness $y_0 = 1 \text{ mm}$, accounting for the initial release from the free surface travelling across the thickness, then the reflection from the driver. This would yield $\dot{\epsilon}_{\text{corrected}} = 16.26 \text{ s}^{-1}$, suggesting the sample conditions should be well into the ductile region. However, this neglects the change in magnitude of the release wave: the volumetric expansion corresponding with this reflected wave is much less than the initial release. In Figure 5.5a, the wave $2 \rightarrow 4$ is reflected from the copper as $4 \rightarrow 6$, with $u_{2 \rightarrow 4}$ approximately four times larger than $u_{4 \rightarrow 6}$. Treating the initial release as a continuous fan, then a scaling factor acting as the reflection, followed by a continuous fan back to the free surface, implies a larger equivalent thickness. Assuming the particle velocities of the initial release fan are scaled by the factor of four by approximating the relative magnitudes of the release wave and reflection in Figure 5.5, equivalent to a four-fold increase in dispersion distance, the equivalent

thickness is therefore $5y_0$, yielding a strain rate of $\dot{\epsilon}_{\text{corrected}} = 9.52 \times 10^{-5} \text{ s}^{-1}$. This is much closer to the viscous fit, but still somewhat below.

It is possible that this altered release history causes the unusually low spall strength, with changes to the level of plastic-work-induced heating resulting in the pre-spall state sitting closer to the melt-line. In addition, the proximity of the spall site to the interface possibly acts to alter the spall behaviour. In the previous analysis, it is assumed that the glue layer on the interface is negligible, with zero thickness and strength. While not anticipated to be as strong as the spall of tin, (typical values for epoxy are 300 MPa to 400 MPa [250–252]), the interface is likely to play an impact in the spall. As a result, a reliable value of the spall strength and strain rate cannot be determined.

Similarly, the exact pressure of the reflected wave cannot be established by $P-u_p$ curve matching, Figure 5.5a. The technique shows that the kink corresponds with the transition in the Birch-Murnaghan model of Mabire and H  reil [25], but cannot resolve the transition pressure. The assumptions made during $P-u_p$ matching, most notably that the release states do not diverge from the Hugoniot, paired with the low convergence of the copper and tin Hugoniots around point 5, leaves a high degree of uncertainty regarding the specific intersection. When paired with the experimental uncertainties, no specific value can be prescribed for the release transition.

More advanced modelling, such as that conducted with a 1D hydrocode, may be better positioned to be able to replicate and understand the behaviour exhibited in this test. This geometry, while providing an interesting opportunity to observe the phase transition on release, is suitable only as a test for modelling, to reproduce the velocity trace. It cannot, for instance, be used to measure any hysteresis of the transition, due to the dependence on modelling to diagnose the internal state. Further tests using this geometry may therefore be of limited use to provide a stronger validation dataset, with wider domain of pressures and thicknesses.

5.3 Influence of Strain Rate and Phase History on the Spall Failure of Single and Polycrystal Tin

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Abstract

Spall occurs when materials are subjected to shock impacts; under this loading, the material properties can be modified through microstructural changes and phase transitions. The effect of these changes on subsequent spall has been under-explored. The anisotropy of tin's ambient crystal structure and the accessibility of the $\beta \rightarrow \gamma$ solid-solid phase transition under shock loading means that tin offers a rich domain in which to study spall failure. Through testing single-crystal and polycrystal samples shocked above and below this transition, the effects of these variables on the deformation behaviour of tin can be determined. Although no orientation dependent spall behaviour is observed, unusual strain-rate-dependent behaviour is observed, indicating likely mechanisms for the high-rate behaviour of tin.

5.3.1 Introduction

When compressive shock waves reflect from surfaces, tensile waves can be created. The interaction of such waves can result in the creation of new surfaces through internal tensile failure, commonly known as spall. The phenomenon of spall, the creation of new surfaces through internal tensile failure, is not a fixed material property, but rather a dynamic response shaped by a number of factors of both the experiment and the sample. In particular, the role of microstructure [181–183, 253], crystal orientation [20–22, 181, 184], release strain-rate [161, 174, 254],

phase transformations [255] and peak shock pressure [93, 111, 133, 174] can all influence the mechanisms of spall.

One particularly interesting feature of tin is the asymmetry of its ambient phase, β -tin[32, 33]. This phase takes the form of a diamond lattice compressed along the diagonal, resulting in properties such as the electrical resistivity [10], yield strength [16], thermal expansion [10–15] and the compressibility [10, 36–41] all showing a clear dependence on the orientation of the crystal. Tin also undergoes a phase transition to a tetragonal γ phase at approximately 9 GPa under shock loading (see Section 2.2 and Chapter 4). This phase transformation is readily accessible under shock, and is associated with a significant refinement of the grain size [24, 26, 139]. Together these phenomena mean tin offers a rich domain of states in which to test spall failure. Through testing spall in a range of single- and polycrystal samples, both above and below this phase transition, a better understanding of the underlying behaviours of tin can also be reached, complementing the authors' previous study of the Hugoniot states of single crystal tin (Chapter 4).

5.3.1.1 Background

The earliest spall measurements of tin by Lalle in Grady [174] map the drop in spall strength corresponding to melt-on-release, with the dataset later expanded by Kanel *et al.* [133]. Between these two sources, measurements are mainly between a peak pressure of 15 and 30 GPa. These data were later augmented with additional experiments and modelling by Robert *et al.* [93] and most recently by Wu *et al.* [178]. The damage induced in these two key loading situations, namely solid and melt on release, has also been studied using proton radiography [179].

Together, these show a strong drop in spall strength as the peak shock pressure increases, see Figure 5.9. However, some uncertainty remains regarding the strength of the solid phases. Some authors have suggested that the spall strength may increase slightly with peak pressure [178], although there is considerable variation between sources.

While these studies all use peak shock pressure to heat the sample, a number of studies preheat or cool the sample to investigate the spall strength. In one such example by Zaretsky and Kanel [111], the sample's initial temperature is varied, showing a very weak decrease in spall strength for temperatures below ambient, and a very rapid drop off approaching the melt point. With peak free-surface velocities around 400 m s^{-1} , the material is unlikely to reach stresses high enough to induce the transition to the γ phase and the time at low temperatures insufficient for significant α to form. Similarly Kanel *et al.* [185] and Ashitkov *et al.* [186] preheat the sample prior to spall to test the strength of the liquid phase.

Spall has also been observed using ramp wave loading, with Chong *et al.* [160] using this loading to measure the spall strength in samples that have reached the γ phase. Laser driven shocks have also been used to measure the spall strength of solid tin. Moshe *et al.* [161] investigate the spall strength and its relationship with the spall strain-rate. The quoted particle velocities for these experiments are in the range 0.29 km s^{-1} to 0.8 km s^{-1} , indicating a range of peak pressures spanning the β and γ phases. However, neither the particle velocity nor pressure is published, meaning the pressure history cannot be estimated.

Combined with recovery, laser driven shocks have been used to investigate the onset of melting upon release [162, 175–177]. Fielding velocimetry at low pressures shows a clear pull-back corresponding to spall, while at higher pressures no such clear pull-back is visible. Indeed the increased noise levels are perhaps a better indication of the change in phase than the numerical values attributed to the pull-back for the high-pressure tests. Considering also the large decay in peak shock-pressure between the driven and free surfaces, these results perhaps bridge between the domain of spall and the domain of dynamic fragmentation. The use of X-rays to diagnose the phase and temperature of the fragments released, and modelling, by Yang *et al.* [256] adds additional data underlining the complexity of this loading case.

Spall in tin has been modelled, most notably using molecular dynamics in single crystals [211, 213, 214, 257]. With molecular dynamics being able to model the underlying phenomenon, such as the initiation, growth and coalescence of voids,

these computational studies offer insights beyond that which can be achieved by free-surface measurements, or even X-ray imaging [258]. However, it remains to be seen how closely these simulations match experimental values. For instance the simulations by Soulard and Durand [27] do not match well the existing single-crystal experimental Hugoniot (see Chapter 4).

As such, reliable and accurate measurements are required to isolate the influence of strain-rate, peak-pressure and phase on the spall behaviour. The influence of single crystal orientation is also an important consideration. Given anisotropic spall behaviour has been found in materials with greater crystal symmetry than tin such as molybdenum [184], copper [20, 21] and aluminium [22, 181], studying the spall in single crystal tin is imperative to better understand the orientation dependent behaviour of the material. Spall is also dependent on the microstructure [181–183], so studying spall above and below the β to γ phase transition may indicate any changes in microstructure that occur.

5.3.2 Methods

Continuing the experiments the authors presented in Chapter 4, a ‘top-hat’ geometry, Figure 4.3, is used. This design, using a C101 copper driver plate upon which PDV is fielded using the double telecentric array (see Chapter 3), allows for the shock wave speeds to be measured, and also provides a datum from which the crystal orientation can be quantified. With a scope sampling rate of 40 GS s^{-1} , a typical window size of 128 samples and a 50 % overlap are used, giving a time resolution of 1.6 ns. Where the PDV signal is weaker, the window size is increased to 256 samples, corresponding to a resolution of 3.2 ns. While this geometry is optimised for Hugoniot measurements, characteristic pull-backs are present in the free-surface traces, allowing analysis of spall in single-crystal tin above and below the phase transition.

Tests using this setup are conducted at four impact velocities, on three single-crystal orientations (001), (100), and (110), and polycrystal samples. The single crystals are all of diameter 10 mm and were indexed relative to the driver plate in order to compare the shock tilt to the crystal axis; see Figure 4.3. Polycrystal

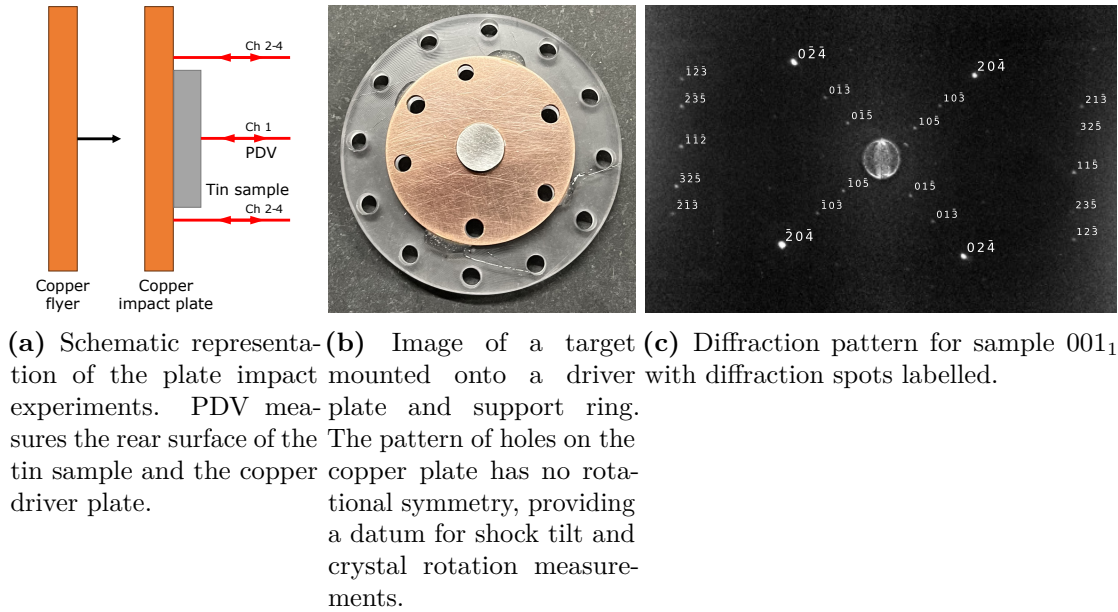


Figure 5.6: Target geometry used in plate-impact experiments. A driver disk with no rotational symmetry is used to index the crystal and measure the shock front tilt. PDV is fielded to measure the surface velocities and transmission speeds.

samples were cut from a stock bar with a diameter of 11 mm. All sample densities were determined using the Archimedes method. The sample thickness was measured before and after mounting, although the depth gauge used was unable to resolve the difference in thickness, meaning the glue layer is assumed to be less than the resolution of 0.01 mm. Sample measurements, including Hugoniot state measurements are presented in Appendix 5.3.8.1.

Two tests are conducted with peak pressures above the threshold for the β to γ phase transition and two below. Thus, comparison between the spall strength of material that has undergone a transition and that which has not in single-crystal and polycrystal samples can be made. In addition, further polycrystal tests are conducted using different thickness samples and flyers to study the strain-rate dependence of spall. Experimental data are tabulated in Table 5.4, and selected rear surface traces are presented in Figure 5.7.

Experimental traces are linearly segmented to analytically determine the pull-back height; see Figure 5.8. The velocity prior to pull-back $u_{\max}$ and the lowest velocity reached $u_{\min}$ are then calculated from the intersections between the fits. Not

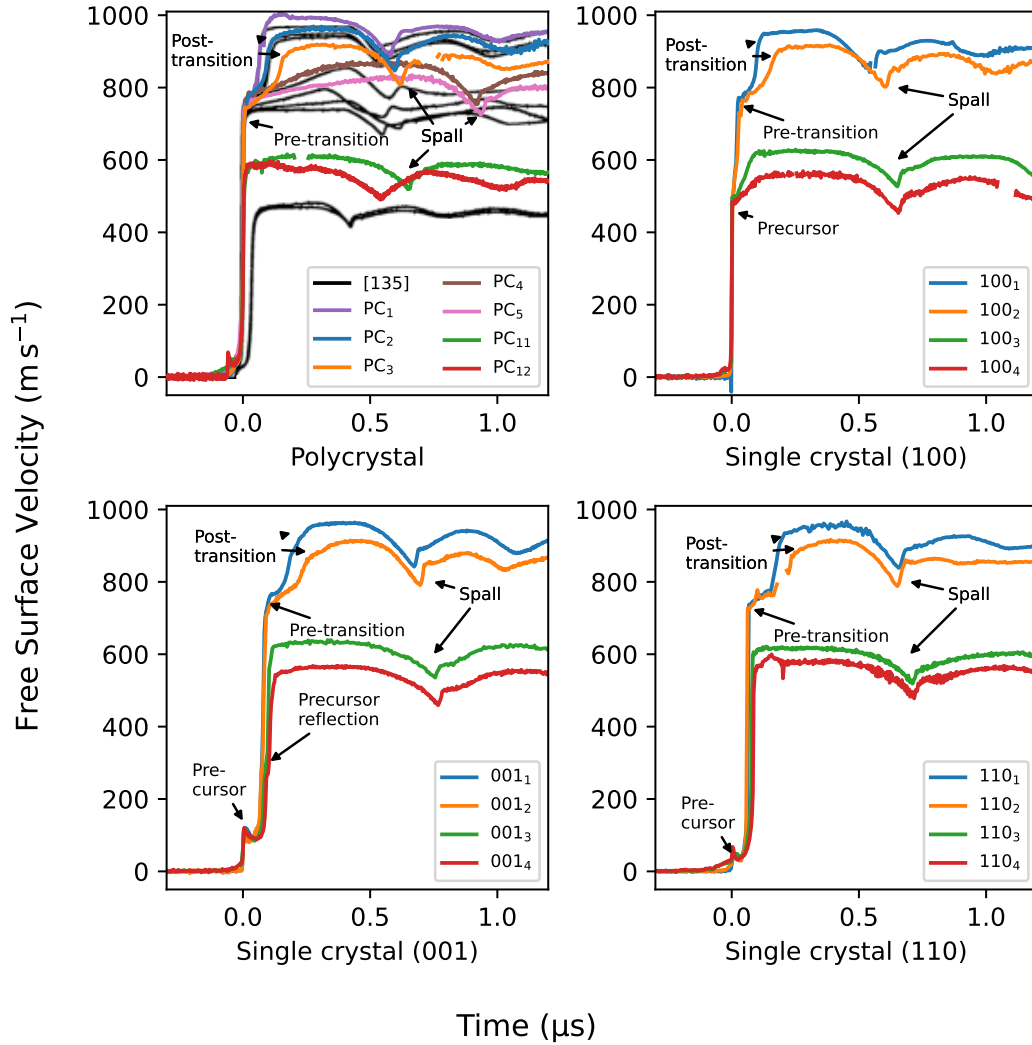


Figure 5.7: Surface velocities traces indicate spall failure in the tin samples. The polycrystal data of Anderson *et al.* [135] (black lines), reproduced from W. W. Anderson *et al.*, “Phase transition and spall behavior in β -tin,” in Shock Compression of Condensed Matter - 1999 (AIP Publishing, 2000) pp. 443–446, with the permission of AIP Publishing, is plotted for comparison, applying a linear dispersion correction to account for the different sample thickness.

only does this approach simplify the inherently noisy PDV signals, but it also allows bridging of the regions where the signal drops out. Given the peak state is assumed to be constant, the uncertainty of u_{max} is estimated using the 95% confidence interval of the fit to the peak state. Similarly, the uncertainty of u_{min} is estimated to be the 95% confidence interval of the pull-back fit evaluated at the minimum point.

To determine the optimum fit, a brute force optimisation is applied. The peak state is determined through minimising the free-surface uncertainty (see Chapter

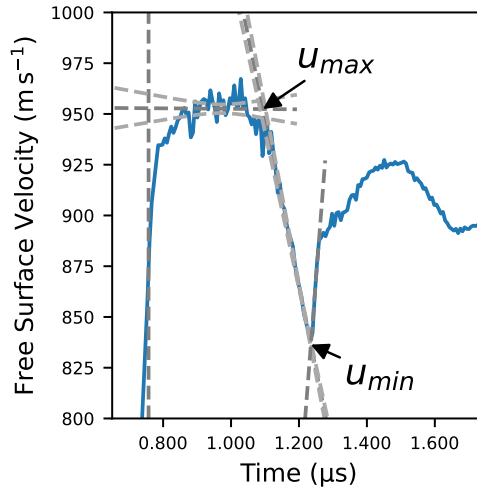


Figure 5.8: Linear fits (dark grey lines) are taken to determine the peak state, the pull-back and the rebound. Intersections between these lines are then calculated to determine the maximum and minimum velocities. Light grey lines show 95 % confidence intervals.

4 for additional details), while the pull-back is determined through minimising the uncertainty of both $u_{\max}$ and $u_{\min}$.

5.3.2.1 Spall Strength

The simplest method of determining the spall strength uses the drop in the free-surface velocity (Δu , the difference between $u_{\max}$ and $u_{\min}$), to estimate the corresponding tensile stress at failure σ using

$$\sigma = \frac{1}{2} \rho_0 c \Delta u. \quad (5.1)$$

Here, ρ_0 denotes the initial density of the tin sample and c the wave speed. This wave speed is typically taken as the bulk sound speed, representing a wave speed slower than the initial release wave from the rear free surface, and the ambient elastic wave speed; but above the shear wave speed.

This equation is used extensively in the polycrystal tin spall literature. For instance, Robert *et al.* [93] use Equation 5.1 with a sound speed of 2.8 km s^{-1} , corresponding with the bulk sound speed calculated from the polycrystal wave speeds of Marsh [164]. The same equation is used by Kanel *et al.* [133], although

the wave speed used is not specified. De Rességuier *et al.* also use the bulk sound speed to evaluate Equation 5.1 without giving a value [162, 175–177]. Wu *et al.* [178] use a value of 2.61 km s^{-1} , in contrast to the other studies. This corresponds with the value of C used by that source to fit the Hugoniot of tin using a linear $U_s - u_p$ relationship, Equation 5.7. Due to the $\beta \rightarrow \gamma$ phase transition, the equation does not well model the low pressure phase, acting to average the discontinuity in properties across the transition. Accounting for the transition using the multiphase Birch-Murnaghan model proposed by Mabire [25, 134] yields an ambient $C = 2.75 \text{ km s}^{-1}$. Considering the consistent use throughout the literature, and proximity to the zero pressure wave speed from this multi-phase model, a polycrystal bulk sound speed of 2.8 km s^{-1} is used in this study. The data from Wu *et al.* [178] have been adjusted in Figures 5.9 and 5.10 to account for this methodological difference. Spall studies in liquid tin, meanwhile, use a liquid sound speed typically between 2.2 km s^{-1} to 2.5 km s^{-1} [185, 186, 259].

In single-crystal samples with orientation dependent properties, the wave speed used to calculate spall strength requires careful consideration. In different orientations, single crystals often exhibit different properties, particularly compressibilities and wave speeds. A consensus has not yet emerged regarding the sound speed to use for single-crystal spall, with, for example, Chen *et al.* [181] using the polycrystal sound speed to determine the spall strength of single-crystal aluminium, while Owen *et al.* [22] calculate the bulk sound speed for the specific orientations of aluminium single-crystals tested, going as far as to calculate two strengths for orientations where two shear waves can propagate.

In β tin, with its tetragonal structure, the wave speeds vary more than in higher symmetry materials, and two shear sound speeds can be determined for the principal directions. Ambient single-crystal wave speeds are tabulated in Table 5.3, with estimations of bulk sound speeds for each mode. While the bulk sound speeds for (001) align well with the literature polycrystal value, (100) shows a significantly slower longitudinal sound speed and corresponding bulk sound speeds. Orientation (110) shows the most unusual behaviour, with one shear sound speed

almost a factor of two lower than for other systems. As a result, applying Equation 5.1 with the most divergent speeds for each orientation would result in spall strengths that vary by up to 30 %.

However, under shock loading, a number of processes act to change the crystal properties, meaning the ambient wave speeds may not be the most appropriate. It has been noted, for instance, that grain size is significantly refined when tin is shocked, [24, 26, 139], suggesting that single-crystals likely devolve into polycrystals. The polycrystal sound speeds [164], $c_b = 2.8 \text{ km s}^{-1}$ and $c_l = 3.43 \text{ km s}^{-1}$ are used in this work for polycrystal and single-crystal samples, with further discussion of the appropriateness of these values presented in Section 5.3.3.

5.3.2.2 Correction for Spall Plate Thickness

During spall, the wave-front of the recompression wave typically over-takes part of the tensile wave, resulting in a reduced pull-back magnitude on the free surface. This can be corrected by adapting Equation 5.1 using

$$\sigma = \frac{1}{2} \rho_0 c_b (\Delta u_{fs} + \delta). \quad (5.2)$$

All symbols have their usual meanings, and δ , the correction can be found through

$$\delta = \left(\frac{h}{c_b} - \frac{h}{c_l} \right) \frac{|\dot{u}_1 \dot{u}_2|}{|\dot{u}_1| + \dot{u}_2}, \quad (5.3)$$

where $\dot{u}_1$ and $\dot{u}_2$ are the slope of the pull-back and rebound respectively, c_l the longitudinal wave speed, c_b the bulk wave speed and h is the depth of the spall plane [260–262].

Given the lack of symmetry in the impact geometry, the spall depth thickness must be estimated from the velocimetry traces, for which there are two principle methods [260]. Firstly, the primary release can be used. Assuming the release from the measured free surface travels to the spall plane with a velocity c_b , while the re-compression wave travels with velocity c_l , the spall depth h is given by

$$h = t_{\text{spall}} \frac{c_b c_l}{c_b + c_l}. \quad (5.4)$$

Table 5.3: Table of sound speeds for each single-crystal orientation. All speeds are in units of km s^{-1} .

	Primary Orientation	Secondary direction 1			Secondary direction 2		
		Longitudinal sound speed [43]	(110)	Shear sound speed [43]	(010)	Shear sound speed [43]	Bulk sound speed
(001)	3.446 ± 0.015			1.727 ± 0.008		1.718 ± 0.007	2.819 ± 0.025
	3.430 ± 0.010						2.799 ± 0.019
(100)	3.112 ± 0.015		(001)	1.717 ± 0.008	(010)	1.795 ± 0.008	2.321 ± 0.029
(110)	3.481 ± 0.018		(001)	1.739 ± 0.010	(110)	0.955 ± 0.005	3.302 ± 0.029

The time t_{spall} is taken from the difference between the incident wave's arrival on the free-surface and the arrival time of the minimum (u_{min}). For (100), the precursor is the largest forward wave and is therefore used to define t_{spall} , Figure 5.7, while for all other orientations, the pre-transition wave is used.

Alternatively, the secondary reflections of the release and compression from the spall surface can be used. This approach uses the period of the ringing after the shock re-compression t_{ringing} to estimate the spall depth. This assumes that the trapped reverberation waves travel with the longitudinal wave-speed c_1 , giving

$$h = \frac{1}{2}t_{\text{ringing}}(c_1). \quad (5.5)$$

In these experiments, Equation 5.4 offers the most robust method, with spall reverberations often showing dispersion, noisy velocimetry signals and dropouts. Fitting a sinusoid to estimate either the second minimum or period of the reverberations offers a method to overcome some of these shortcomings, however, the fit is often poorly conditioned. In addition, the period of the reverberations tends to decrease with further periods, where available, suggesting a degree of damping. As a result, only Equation 5.4 is used when calculating the spall strength in Equation 5.3, although both are presented in Table 5.4.

5.3.2.3 Spall Strain-Rate

Typically, the strain rate of spall ($\dot{\epsilon}$) is given as

$$\dot{\epsilon} \approx \frac{\Delta u}{\Delta t} \frac{1}{2c_b}. \quad (5.6)$$

Here, $\frac{\Delta u}{\Delta t}$ is taken as the gradient of the linear fit to the pull-back (the line between u_{max} and u_{min} in Figure 5.8). This is fitted to the developed release wave immediately prior to the inflection and therefore corresponds to the local conditions at the nucleation of damage. Defining Δt from the start of roll-off would, therefore, underestimate the strain-rate for the specific conditions where spall first occurs. To the author's knowledge, this equation first occurs in English speaking literature in Kanel *et al.* [261]. Prior to this, the pressure-rate was used in a similar

manner, with pressure measured using a transducer on the rear surface instead of velocity measurements [232, 263, 264]. Earlier sources use only the pulse width [233, 265] to assess the onset of spall failure. Intermediate derivations may lie in Russian literature sources.

This equation relies on several key assumptions. Firstly, it is assumed that spall initiates immediately after the final release from the sample free surface, meaning only the release wave from the flyer drives the nucleation and growth of spall. Secondly, the release wave is assumed to disperse minimally, with the release strain-rate being constant throughout the sample. Finally, the material is assumed to display a linear pressure-volume relationship, meaning wave interactions are additive. As a consequence of these assumptions, the free-surface strain-rate calculated through Equation 5.6 can be taken as a proxy for the strain rate on the spall plane. This provides a useful estimate for the strain rate, and has been used to determine the threshold between ductile and brittle behaviour [161, 174] (see also Chapter 3).

It has been noted that the spall strain rate calculated by this equation systematically underestimates the strain rate when compared to computational simulations [266]. Indeed, it is suggested by Utkin [267] that the strain rate on the measured surface calculated through Equation 5.6 is one quarter the strain rate at the spall. An alternative analysis by Antoun *et al.* [260] yields the same conclusion.

The assumption of negligible dispersion of the release wave is also possibly unreliable, with plate impact experiments typically represented with a release fan, the release from the flyer dispersing approximately linearly. Many material's Hugoniot are modelled using the relationship

$$U_s = C + Su_p, \quad (5.7)$$

where U_s is the wave velocity, C and S constants and u_p the particle velocity [159, 164]. While the release follows an isentrope, for low pressures, the release follows the Hugoniot to within a few percent, and Equation 5.7 provides a reasonable estimate, [166]. From this, the pull-back, itself a decrease in u_p with time, would correspond with a linear decrease in wave the velocity as u_p decreases, creating a release fan

originating temporally from a single time on the flyer free-surface. This of course neglects any disturbance caused by the interaction of the two releases from opposing surfaces. However, in this case, the strain rates vary through the sample by a factor α relative to the strain-rate at the measured-surface, where

$$\alpha = \frac{y_0}{y_0 - y}. \quad (5.8)$$

Here, y is the distance from the measured free-surface, and y_0 is the thickness of the sample and flyer, with a full derivation in Appendix 5.3.8.2. If y is taken as h , the depth of the spall plane from the measured surface, then the spall strain rate is given by

$$\dot{\epsilon}_{\text{corrected}} \approx 4\alpha \frac{\Delta u}{\Delta t} \frac{1}{2c_b} = \frac{y_0}{y_0 - h} \frac{\Delta u}{\Delta t} \frac{2}{c_b}. \quad (5.9)$$

This equation remains valid where spall first occurs on the final release wavelet from the measured free surface. Thus the initiation of spall is driven by release from the flyer. For this to be true, h must be less than half y_0 .

For classical spall experiments, where the impactor is thinner than, and of the same material as, the sample, this simple approximation may significantly improve the accuracy of the spall strain rate measurement. For instance, in the case of Turley *et al.* [266], experimental measurements of strain rate determined by Equation 5.6 differed from strain rates calculated in simulations by a factor of six. In this case the samples are twice as thick as the flyer ($y_0 = 3, h = 1$) meaning a correcting factor of $\alpha = 3/2$ should be applied. Through the application of Equation 5.9, the reported factor of six difference between simulated strain rates and measured values is thereby accounted for.

5.3.3 Results

A qualitative analysis of these traces indicates that the spall pull-back profiles are very similar for the various orientations. The samples all show a similar sharp rise, followed by a plateau leading to a more gradual rise indicative of a transition from a faster rate of damage to a slower rate, typically assumed to be the transition from

brittle to ductile behaviour [268]. This effect is also present in the traces of Anderson *et al.* [135]. It is noted that Anderson *et al.* use thicker samples. To compare the traces with the work conducted here in Figure 5.7, a linear scaling factor is applied to the time axis to account for the different sample thickness. Despite this, the thicker samples have the effect of softening and dispersing the sharp kick.

The similarity between orientations is borne out in the measured spall strengths. Using Equation 5.1 with the bulk sound speed of 2.8 km s^{-1} gives the results presented in Figure 5.9. The use of a single sound speed for all orientations allows for a comparison of the magnitude of the pull-back between samples, and between the literature and these experiments. Preliminary data, based on the release waves arrival on the copper and tin free-surfaces, indicate that the release wave speeds are similar between samples. Some clear trends are visible from this analysis.

Firstly, no clear distinction between single crystals and polycrystals can be made. This is somewhat unexpected: typically, a grain structure is expected to reduce the spall strength. For example, molybdenum and copper single-crystals show spall strengths roughly twice as high as polycrystals [20, 21, 184], while aluminium offers a roughly 20 % difference [22, 181]. Given also that the experimental traces show an almost identical pull-back profile, with similar rebound characteristics, orientation does not appear to influence spall. As a result, the use of a single wave speed is perhaps the more appropriate approach.

A further observation is made that samples that have undergone the γ phase change appear to show a somewhat higher pull-back magnitude than those that have not, although the explanation of this behaviour is unclear.

5.3.3.1 Strain Rate

The spall strengths presented here sit somewhat higher than the gas-gun and explosively driven experiments of Lalle in Grady [174], Kanel *et al.* [133] and Robert *et al.* [93], but below the laser driven measurements of de Rességuier *et al.* [176] in Figure 5.9. Given the associated strain-rate of these experimental methods, it is suggested that the strain rate may strongly influence the spall strength of tin.

Table 5.4: Table of spall data.

Sample	Peak state phase	Pull-back magnitude m s^{-1}	Pull-back gradient $\times 10^8 \text{m/s}^2$	Spall strength GPa	Spall strain rate $\times 10^5/\text{s}$	Primary ^a mm	Spall depth Secondary ^b mm	Corrected spall strength GPa	Corrected strain rate $\times 10^5/\text{s}$
100 ₁	γ	117 $\pm$ 2	6.9 $\pm$ 0.2	1.19 $\pm$ 0.03	1.24 $\pm$ 0.03	0.82	0.76	1.47	5.81 $\pm$ 0.15
100 ₂	γ	116 $\pm$ 8	7.4 $\pm$ 0.7	1.18 $\pm$ 0.09	1.32 $\pm$ 0.12	0.89	0.79	1.50	6.26 $\pm$ 0.57
100 ₃	β	94 $\pm$ 4	5.7 $\pm$ 0.3	0.96 $\pm$ 0.05	1.02 $\pm$ 0.05	0.92	1.09	1.25	4.87 $\pm$ 0.24
100 ₄	β	103 $\pm$ 10	6.8 $\pm$ 0.7	1.05 $\pm$ 0.10	1.21 $\pm$ 0.12	0.91	0.94	1.34	5.79 $\pm$ 0.58
001 ₁	γ	128 $\pm$ 3	8.6 $\pm$ 0.3	1.31 $\pm$ 0.03	1.53 $\pm$ 0.05	0.94	0.70	1.75	7.34 $\pm$ 0.24
001 ₂	γ	128 $\pm$ 3	8.4 $\pm$ 0.2	1.30 $\pm$ 0.04	1.51 $\pm$ 0.04	0.98	0.59	1.78	7.31 $\pm$ 0.20
001 ₃	β	94 $\pm$ 5	5.3 $\pm$ 0.4	0.96 $\pm$ 0.05	0.94 $\pm$ 0.06	1.01	1.11	1.21	4.60 $\pm$ 0.30
001 ₄	β	101 $\pm$ 5	5.4 $\pm$ 0.4	1.03 $\pm$ 0.05	0.97 $\pm$ 0.07	1.03	1.10	1.34	4.74 $\pm$ 0.35
110 ₁	γ	116 $\pm$ 6	8.2 $\pm$ 0.6	1.18 $\pm$ 0.07	1.47 $\pm$ 0.10	0.92	0.75	1.55	7.01 $\pm$ 0.48
110 ₂	γ	130 $\pm$ 5	9.7 $\pm$ 0.5	1.33 $\pm$ 0.06	1.74 $\pm$ 0.08	0.92	0.42	1.78	8.31 $\pm$ 0.39
110 ₃	β	87 $\pm$ 5	5.4 $\pm$ 0.5	0.88 $\pm$ 0.06	0.96 $\pm$ 0.08	0.98	1.13	1.17	4.67 $\pm$ 0.39
110 ₄	β	99 $\pm$ 9	6.3 $\pm$ 1.2	1.01 $\pm$ 0.09	1.12 $\pm$ 0.20	0.98	1.09	1.31	5.45 $\pm$ 0.98
PC ₁	γ	111 $\pm$ 28	8.3 $\pm$ 0.4	1.13 $\pm$ 0.29	1.48 $\pm$ 0.06	0.90	0.72	1.49	7.09 $\pm$ 0.29
PC ₂	γ	119 $\pm$ 8	7.6 $\pm$ 0.6	1.21 $\pm$ 0.08	1.36 $\pm$ 0.11	0.93	0.70	1.61	6.58 $\pm$ 0.54
PC ₃	γ	116 $\pm$ 9	10.8 $\pm$ 1.1	1.19 $\pm$ 0.09	1.93 $\pm$ 0.20	0.96	0.74	1.68	9.47 $\pm$ 0.99
PC ₄	γ	120 $\pm$ 4	8.0 $\pm$ 0.4	1.23 $\pm$ 0.05	1.43 $\pm$ 0.07	1.42	1.10	1.79	7.59 $\pm$ 0.38
PC ₅	$\beta - \gamma$ mix	107 $\pm$ 5	6.5 $\pm$ 0.4	1.09 $\pm$ 0.05	1.16 $\pm$ 0.06	1.45	0.87	1.60	6.19 $\pm$ 0.33
PC ₆	γ	113 $\pm$ 4	9.1 $\pm$ 0.5	1.15 $\pm$ 0.04	1.62 $\pm$ 0.08	1.25	1.01	1.66	8.11 $\pm$ 0.41
PC ₈	γ	122 $\pm$ 3	8.8 $\pm$ 0.2	1.24 $\pm$ 0.03	1.57 $\pm$ 0.02	0.58	0.46	1.53	7.14 $\pm$ 0.10
PC ₉	γ	113 $\pm$ 6	8.7 $\pm$ 0.7	1.16 $\pm$ 0.07	1.56 $\pm$ 0.12	1.34	1.02	1.77	8.06 $\pm$ 0.62
PC ₁₀	γ	109 $\pm$ 12	11.3 $\pm$ 1.5	1.11 $\pm$ 0.12	2.01 $\pm$ 0.26	0.54	0.49	1.43	8.90 $\pm$ 1.16
PC ₁₁	β	86 $\pm$ 4	5.4 $\pm$ 0.4	0.88 $\pm$ 0.05	0.96 $\pm$ 0.06	1.01	0.88	1.14	4.75 $\pm$ 0.30
PC ₁₂	β	95 $\pm$ 9	4.6 $\pm$ 0.2	0.96 $\pm$ 0.10	0.82 $\pm$ 0.04	0.84	0.82	1.10	3.91 $\pm$ 0.20

^a Spall depth calculated from the reflection of the first release wave from the free surface, equation 5.4.^b Spall depth calculated from the secondary reflection of the release, i.e. the first reverberation after spall, equation 5.5.

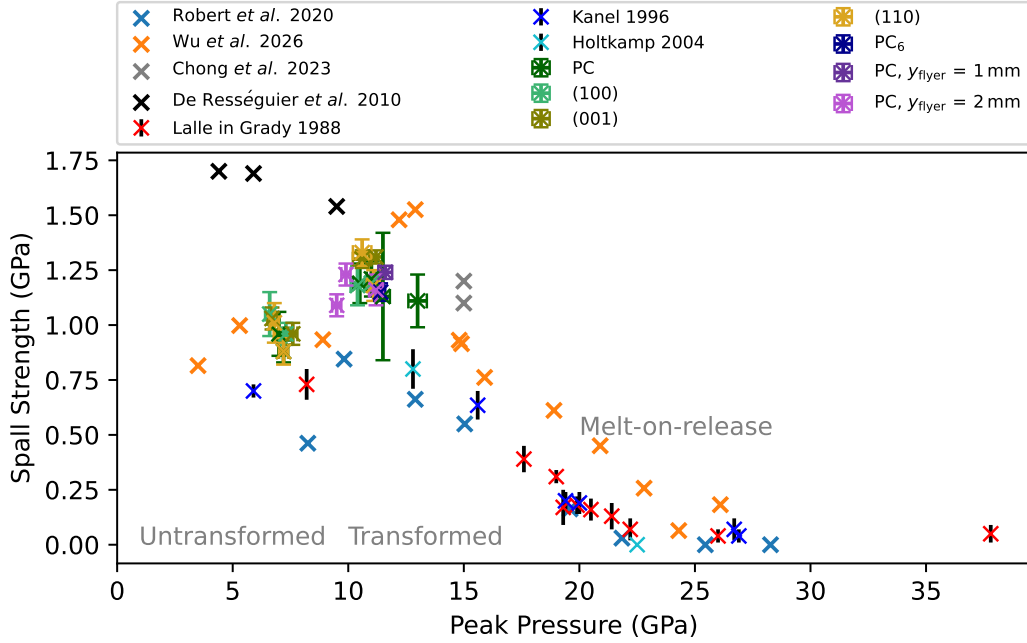


Figure 5.9: Uncorrected spall strengths presented for a range of single and polycrystal samples of tin. Using a bulk sound speed of 2.8 km s^{-1} for all samples allows for comparison of the pull-back magnitude between the literature [93, 133, 160, 174, 176, 178, 179] and the various samples. All samples use a 1.5 mm thick copper flyer (y_{flyer}) and are nominally 2 mm thick unless otherwise stated, except for sample PC_6 , ($y_{\text{flyer}} = 2 \text{ mm}$, $y_0 = 3 \text{ mm}$). Labels used to indicate approximate regions where the samples remain β (untransformed), undergo the forward and reverse transition to γ (transformed) and melt prior to spall failure.

In literature spall strain rate measurements, Equation 5.6 is used [161]. For other literature studies, the strain rate can be estimated from the presented velocity traces. In most studies, experimental geometries are not documented, meaning Equation 5.9 cannot be applied. In order to compare results, Equation 5.6 must be used, with results in Figure 5.10.

Estimating the spall strain rate confirms that the data presented here sit at higher strain-rates than other literature gas-gun data. The experimental geometry for the literature data is not published, meaning variations in sample and flyer thickness may explain the difference between these data and the literature.

Inspecting Figure 5.10, the data follow a straight line, with the exception of the two highest polycrystal data points. As a result, it may be that the jump in spall strength observed in Figure 5.9 for material that has cycled $\beta \rightarrow \gamma \rightarrow \beta$ is an effect

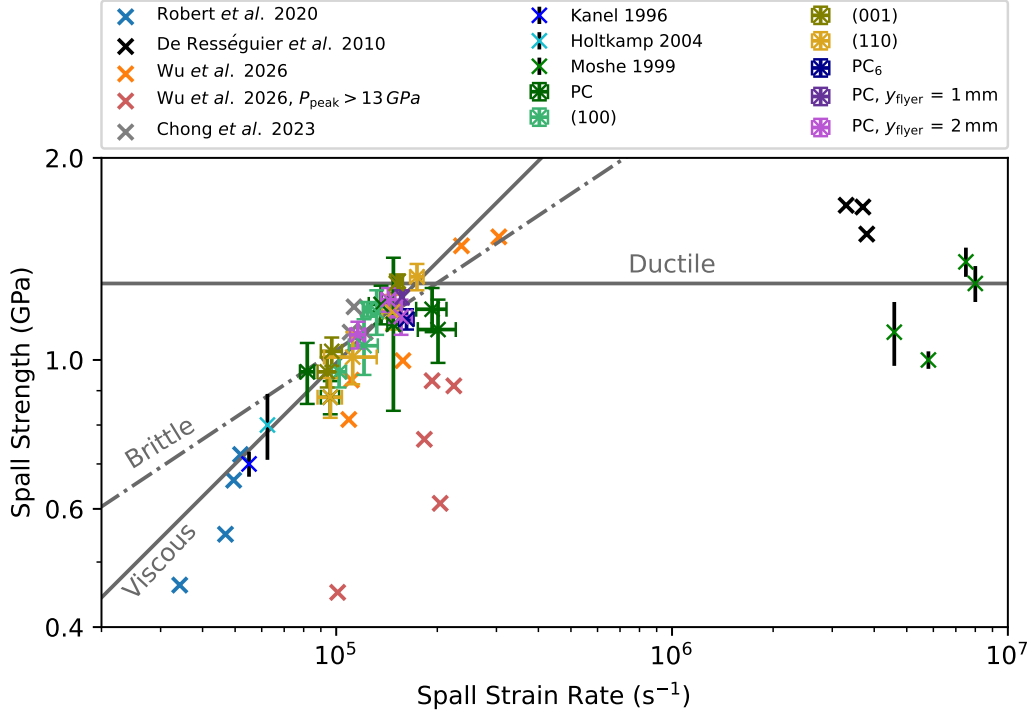


Figure 5.10: Spall strain-rate and strength for a range of tin samples and peak pressures compared to literature [93, 133, 160, 161, 176, 178, 179]. A bulk sound speed of 2.8 km s^{-1} is used to estimate the spall-strength and strain-rate. Grey dotted line used as a guide between samples in this study that transform to the γ phase at the peak state. All samples use a 1.5 mm thick copper flyer (y_{flyer}) and are nominally 2 mm thick unless otherwise indicated, except for sample PC₆, ($y_{\text{flyer}} = 2 \text{ mm}$, $y_0 = 3 \text{ mm}$).

of the strain rate rather than any change in properties resulting from the phase changes. A change in spall strength due to the phase history would be expected to result in different trends for samples that had undergone the transition and those that had not. These phenomena are not observed in the data with all data agreeing with the straight line fits. The higher rate experiments meanwhile do not show any increase in spall strength. This behaviour is typically assumed to be indicative of a brittle to ductile transition, following the theory proposed by Grady [174].

The ductile spall strength, where fracture is dominated by energy dissipated through plastic yielding, can be determined using

$$\sigma = (2\rho c^2 Y \varepsilon_c)^{\frac{1}{2}} \quad (5.10)$$

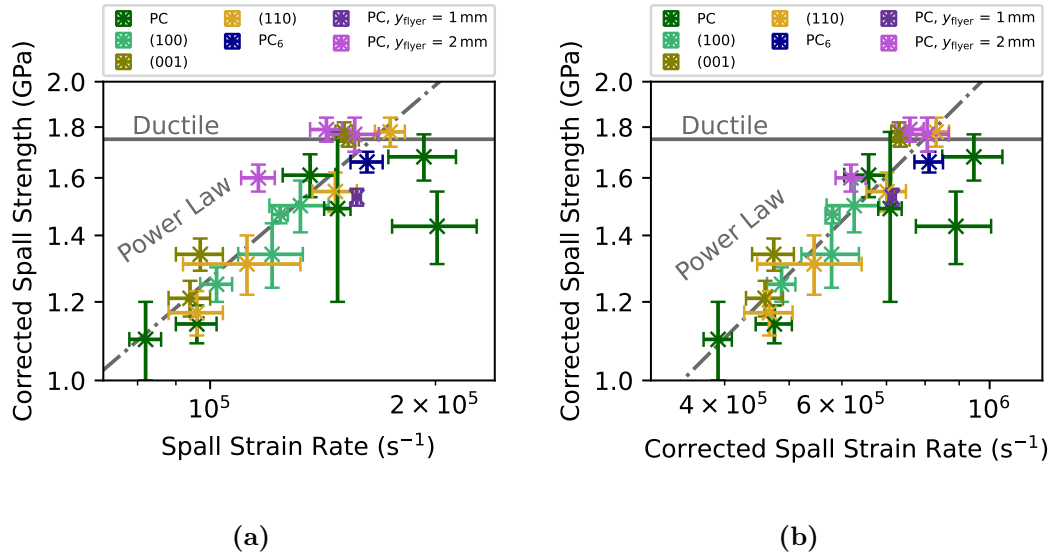


Figure 5.11: Spall strengths, corrected for re-compression overtake (equations 5.2 and 5.3), compared to the uncorrected strain-rate (equation 5.6, left) and strain rate corrected for dispersion (equation 5.9, right). Samples are impacted with a 1.5 mm flyer (y_{flyer}) and are 2 mm thick, unless otherwise noted, except for PC₆, which uses a 2 mm flyer and 3 mm sample.

where Y is the flow stress in simple tension, given by Grady as a range between 0.05 and 0.12 GPa for tin, while ε_c is the critical void volume fraction, taken as 0.15. Taking the middle of the range of Y as 0.085 GPa, the density as 7290 kg m^{-3} and the bulk sound speed c as 2.8 km s^{-1} yields a value of 1.2 GPa. This is in good agreement with the highest spall strengths measured in these experiments, as well as the laser shocks of Moshe *et al.* [161] and de Rességuier *et al.* [162, 175–177]. The post-spall recovered samples of de Rességuier *et al.* confirm that ductile fracture, predominantly transgranular, is the mechanism of spall in this domain [162].

After applying the spall strength and strain-rate corrections, it can be seen by visually inspecting Figure 5.11b that the highest strain-rate tests, assumed to fail through ductile spall, show a spall strength around 1.75 GPa. This is in better agreement with the data from de Rességuier *et al.* [162, 175–177] than Moshe *et al.* [161], see Figure 5.10, although neither apply the correction for spall depth (Equation 5.3). However, with pulse durations of approximately 10 ns, more than an order of magnitude shorter than the pulse here, the correction would likely be a similar magnitude smaller, and therefore not significantly change the values.

Below this, the rate-dependent region is assumed to be governed by rate-dependent cracking. For many structural materials, such as aluminium, uranium, molybdenum, beryllium and steel [174, 184, 237, 254, 269], this phenomenon can be modelled assuming energy dissipation through the creation of new surfaces. In this case

$$\sigma = (3\rho c K_c^2 \dot{\epsilon})^{\frac{1}{3}} \quad (5.11)$$

gives the spall strength, where K_c is the fracture toughness of the material [174]. However, for many other materials, applying a more general fit of the form

$$\sigma = a\dot{\epsilon}^n \quad (5.12)$$

better maps the spall strength. Many common metals exhibit a power n to be less than $\frac{1}{3}$, with stainless steel 35Kh3NM, titanium, copper and magnesium Mg95, exhibiting n to be in the range of 0.05 to 0.25 [181, 260]. This may be due to the variation of K_c with strain rate: fracture toughness typically decreases with strain rate as the reduced time duration decreases energy dissipation through plastic work [270].

A least squares fit conducted on the data for tin measured in this study, excluding the two highest polycrystal data points (samples PC₃ and PC₁₀), yields an unusually high power of $n = 0.509$, (three decimal places, variance of 0.004). Including the literature data from Robert *et al.* [93], Chong *et al.* [160], Kanel *et al.* [133] Holtkamp *et al.* [179], and the data for Wu *et al.* [178] with a peak pressure below 13 GPa, into the fit yields $n = 0.481$ (variance of 0.002). Correcting the strain rate and the spall strength only increases the power (Figure 5.11b), further deviating from the brittle model.

Limited evidence in tin-based solders suggests that fracture toughness increases with strain rate [271], although the transferability to pure tin and the strain rates measured under shock is unclear. The authors are aware of no measurements of the fracture toughness of pure tin beyond soldered joints [272]. However, if K_c were a function of strain rate, Equation 5.11 would still hold, and could map to

a higher power than one third. Indeed, any strain rate relationship would have to approximate $K_c \propto \dot{\epsilon}^{0.25}$ for Equation 5.11 to map to a power of a half. This would imply that tin had no resistance to cracking when the strain rate is equal to zero. Any offset of the form $K_c = a\dot{\epsilon}^{\frac{1}{4}} + b$, where a and b are constants would result in a non-linearity in $\log \dot{\epsilon}$ - $\log \sigma$ space (Figure 5.10). However, no curvature is visible in the data presented, although further testing in the lower strain-rate domain would improve the confidence in this assessment.

Taken together, this behaviour suggests that tin may not conform to traditional brittle fracture models, but instead exhibits characteristics more consistent with a material governed by viscous dissipation mechanisms. Notably a power law relationship of

$$\sigma = (2\rho c^2 \eta \dot{\epsilon})^{\frac{1}{2}} \quad (5.13)$$

is predicted for a material where viscous dissipation dominates the spall strength [174].

Given the spall strength is around three orders of magnitude larger than the statically observed critically resolved shear stresses [16, 19, 52, 53], it is supposed that plastic deformation is readily facilitated, forming the dominant deformation mechanism during the nucleation and growth of voids. Therefore, the spall behaviour could be governed by the energy dissipation during this plastic flow, rather than through surface energy in brittle fracture. Tin is also known for exhibiting creep deformation [61, 239, 273–278] at low strain rates. With forces used to induce creep in the mega-pascal domain and quasi-static strain rates, extrapolation to the shock domain is unable to offer any meaningful results. Nonetheless, the observed power-law rate-dependence of spall could point to similar underlying mechanisms, meaning creep behaviour could offer a window to interpreting the viscous-like response of tin at high strain rates.

A viscous-like flow could well explain other observations of the high-pressure behaviour of tin such as the lack of observed anisotropy above the elastic-plastic transition under shock (see Chapter 4). Once the viscous flow regime is activated,

initial orientation and texture may no longer be preserved to any great extent. Similarly, this flow like structure would be unable to preserve any changes induced by the phase transition cycle, meaning spall is limited by the flow regardless of the phase history. However, the order-of-magnitude difference of the elastic limit between the orientations remains a puzzle, especially given the lack of a clear Schmidt law response, see Chapter 4.

Further recovery and *in situ* X-ray experiments are, therefore, required to better understand the mechanism of spall in tin. Additionally, molecular dynamics studies may help develop understanding on the phenomenon through predictions of the atomic scale processes involved.

5.3.4 Further observations

Examining Figure 5.11a highlights the need to apply the correction for the spall strain rate, Equation 5.9. Samples impacted with a thicker flyer appear to show a higher spall strength for the same strain rate, while those impacted with a thinner flyer show lower spall strength. No reason beyond analytical inaccuracies is thought to be behind this effect. Indeed, after applying the correction (equation 5.9 with y_0 taken as the total thickness of the flyer, driver plate and sample to produce Figure 5.11b), this spread is no longer observed. The samples impacted with 2 mm and 1 mm flyers now sit much closer to the trend-line. The proposed correction can, therefore, reduce at least some of the error inherent in the assumptions used to derive Equation 5.6, despite the different materials used in the flyer and driver plate.

However, the data points still sit towards the edges of the trend-line. For the experiments presented here, the linear approximation gives a much more empirical estimate of the strain rate, given Equation 5.9 assumes that dispersion is linear. The dispersion rates in the copper driver and tin sample will be different, while the impedance mismatch between the two adds additional complexity. Two approaches could be taken to account for this. Following an approach similar to the derivation of the spall strength equation, the dispersion rates could be approximated using the ratio c_1 to c_b for the two materials. The coupling between the two could

then be estimated using the elastic moduli to convert the strain rate on one side of the interface to the other, based on stress and displacement continuity. An alternative approach, assuming predominantly plastic deformation, would be to use pressure-particle-velocity matching. Assuming a relationship given by Equation 5.7, the gradient S could be used to give a measure of the dispersion in the two materials, with the shock jump conditions used to estimate the coupling. However, it is unclear without further analysis and modelling, such as through hydrocode simulations, whether either of these two approaches would yield a better result for the experimental geometry tested here. In particular, neither approach would model well the distortion in the profile caused by the γ to β phase transition, nor the interactions between the various waves.

The use of these additional corrections relies heavily on the validity of the underlying assumptions regarding material behaviour. Since each assumption may deviate from a more accurate representation, such as one obtained through hydrocode modelling, reducing the number of assumptions may lead to a more robust model. Applying additional corrections based on dispersion rates without additional modelling would, therefore, complicate the simplicity of Equation 5.9, without necessarily improving the accuracy. As such, applying Equation 5.9 in Figure 5.11 with the uncertainty from Equation 5.6 scaled appropriately appears to significantly reduce scatter from the expected trend. For a classical spall experiment, where the flyer and sample are of the material, Equation 5.9 is expected to have the best results.

Applying the corrections highlights that the highest pressure polycrystal (PC₁₀) sits detached from the main trend. It may be the case that sample is starting to weaken with the onset of melt. In Figure 5.9, the transition between solid spall and liquid spall shows a decrease in spall strength. The lower end of this transition region is not clearly defined, but appears to be around 12 GPa to 15 GPa, in which region this sample is shocked.

5.3.5 Conclusions

In this study, the effects of spall strain-rate, single crystal orientation and peak phase on the spall strength of tin have been examined. Any anticipated orientation or microstructural dependence of the spall strength, based on the static properties and comparison to other materials, is not found in tin. Instead, the spall strength of solid tin appears to be best predicted using only the strain rate. The nature of this strain-rate dependence appears to correlate with a viscous model, with spall strength proportional to the square root of strain-rate. This response, it is hypothesised, may be due to the low threshold for plastic deformation resulting in fluid-like flow. This hypothesis may help the understanding of other shock observations, such as the lack of any observed orientation dependence of the Hugoniot states above the Hugoniot elastic limit, with the plastic flow overcoming any pre-shock properties. An improvement to the calculation of the spall strain rate for plate-impact experiments is also proposed. To better verify these observations, further experimental measurements are suggested alongside additional modelling. Key areas of focus could include molecular dynamics simulations of shock and spall in tin, alongside *in situ* X-ray diagnostics of phase and microstructure, or shock recovery.

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5.3.7 Author Declarations

5.3.7.1 Conflicts of Interest

The authors have no conflicts to disclose.

5.3.7.2 Author Contributions

Jasper G. Threadingham: Conceptualisation (lead); Data curation (lead); Formal analysis (lead); Investigation (lead); Methodology (lead); Writing – original draft (lead); Writing – review & editing (lead). **Xuefei Liang:** Investigation (equal). **Edward Leggett:** Investigation (equal); Writing – review & editing (equal). **Liam C. Smith:** Investigation (equal); Writing – review & editing (equal). **Jeremy C. F. Millett:** Conceptualisation (supporting); Funding acquisition (equal) Resources (equal); Supervision (supporting). **Glenn Whiteman:** Conceptualisation (supporting); Funding acquisition (equal); Resources (equal); Supervision (supporting). **Viviane Peçanha-Antonio:** Methodology (supporting); Resources (supporting). **Andrew T. Boothroyd:** Methodology (supporting); Resources (supporting); Writing – review & editing (supporting). **David J. Chapman:** Conceptualisation (equal); Funding acquisition (equal); Investigation (equal); Supervision (equal); Writing – review & editing (equal). **Daniel E. Eakins:** Conceptualisation (equal); Funding acquisition (lead); Investigation (equal); Supervision (lead); Writing – review & editing (equal).

5.3.8 Appendices

5.3.8.1 Shock wave data

Measured pressures and shock wave speeds are presented in Table 4.1 alongside the sample data.

5.3.8.2 Derivation of Equation 5.8

Assuming a simple wave with momentum and mass conservation, wave propagation is given by

$$\frac{\partial u_p}{\partial t} + U_s(u_p) \frac{\partial u_p}{\partial y} = 0. \quad (5.14)$$

If U_s can be estimated given u_p by Equation 5.7, a step change from u_{p0} to u_{p1} at $t = 0$ and $y = 0$ results in u_p varying in space and time by

$$u_p(y, t) = \begin{cases} u_{p0}, & y < (C + Su_{p0})t \\ \frac{y - Ct}{St}, & (C + Su_{p1})t < y < (C + Su_{p0})t \\ u_{p1}, & y > (C + Su_{p1})t \end{cases} \quad (5.15)$$

In the decreasing region

$$\frac{\partial u_p}{\partial t} = \frac{-y}{St^2}. \quad (5.16)$$

Evaluated along a line of constant u_p ,

$$\left. \frac{\partial u_p}{\partial t}(y) \right|_{t=y/(C+Su_p)} = \frac{-(C + Su_p)^2}{Sy} \propto \frac{1}{y}. \quad (5.17)$$

Notably, this neglects any change in u_p due to the opposing release fans interacting.

However, the ratio at a given point y to the surface is given by

$$\alpha = \frac{\frac{\partial u_p}{\partial t}(y_0 - y)}{\frac{\partial u_p}{\partial t}(y_0)} = \frac{y_0}{y_0 - y}. \quad (5.18)$$

5.3.9 References

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6

Shock Recovery of Single Crystal Tin

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In Chapter 4, above the elastic limit, no quantitative differences in the shock states due to initial sample crystallinity or orientation are observed. An unusually large anisotropy of yields was also observed. In Chapter 5, no orientation or shock-induced-microstructural dependence is observed, in contrast to many other

metals. Through observations using *in situ* X-ray diffraction under shock, tin is also known to recrystallise under shock loading, [24, 26, 139], hypothesised to correspond with the $\beta \rightarrow \gamma$ phase transition, although all observations compare states above the transition to as-received material. Indeed, The pathway *in situ* X-ray diffraction has been unable to determine the causes of this recrystallisation, nor the $\beta \rightarrow \gamma$ transition pathway, although the Musgrave mechanism [143] would agree with the results of Chauvin *et al.* [140].

To better understand these observations, and study the underlying microstructural changes, shock-recovery is a powerful tool. While it does not directly measure the state *in situ* under shock, it can be used to determine phase transition pathways, for instance in iron [279], and detect any retained high pressure phases, such as high-pressure ω -titanium [280]. In addition, recovery can be used to examine slip bands and twins to determine the mechanisms underlying high-rate plastic deformation [280, 281]. Recovery can also be applied to spall, especially incipient spall, to better understand the failure mechanisms [176, 178].

In tin, recovery has only been applied in laser shock experiments, and shows the mechanism of spall to be ductile failure, predominantly transgranular [162]. The advantages offered by laser shock, notably the absence of a physical impactor or sabot, offer significant convenience to recover samples. However, the small drive diameter, limited by the focus of the laser, restricts the size and thickness of the sample that can be studied. The size of the sample thus limits the length-scale of the possible processes, especially any recrystallised microstructure. In addition, the small length scales correspond with higher strain rates, further altering the mechanisms from the larger samples tested in this work. As a result, gas-gun driven recovery experiments may offer a better route to understand the previously observed phenomena in tin.

6.1 Methods

The geometries of gas-driven shock experiments have benefited from significant historical optimisation. The use of trapezoidal profile lateral rings to prevent lateral release waves, as well as plates along the axis of the shock to contain spall damage

is well established [281]. While the theory is therefore well established, designing, constructing and fielding a recovery experiment requires careful consideration given the unique nature of every material and launcher.

Recovery of tin presents a number of significant additional challenges to overcome compared to more conventional materials. Firstly the low strength of tin means it is extremely easy to damage. Secondary impacts are a particular danger, especially with velocities up to and in excess of 1 km s^{-1} , meaning the projectile is a significant hazard to consider when designing the experiment. The low strength also limits the ability to precisely machine samples, with close fits between plates required to prevent local stress concentrations and unwanted releases and shocks from pore collapse. Secondly, the proximity of the released state to melt [25, 93, 133, 174], adds additional complexity. With the recovered tin likely in the range 350 K to 400 K [23, 25], compared to a melt temperature of 505 K [90, 115, 117, 118, 282], tin is liable to recrystallise even after coming to rest. As a result, the experimental design is required to isolate the sample from the projectile, and slow the sample gradually to minimise large stresses that could alter the microstructure or induce plastic heating. These factors are all on top of the challenges posed when recovering a more conventional material, such as limiting the influence of lateral waves, and reducing spall damage.

6.1.1 Sample Design

To achieve these goals, the system represented in Figure 6.1 was developed, with the sample geometry shown in Figure 6.1b. Lateral rings prevent release waves from the edge surfaces from reaching the sample. As the release waves from the sample free surface reaches the angled interface, the interface opens, preventing lateral effects from influencing the sample. A spall plate is used to conduct momentum from the impact, and reduce tensile damage to the sample. A cover plate is also used on the interface with the driver plate. These rings and plates are made from polycrystal tin to economically match impedances as closely as possible to prevent waves from being reflected from the various interfaces. This assembly is pressed together by hand, with friction sufficient to hold the rings together. This arrangement is then

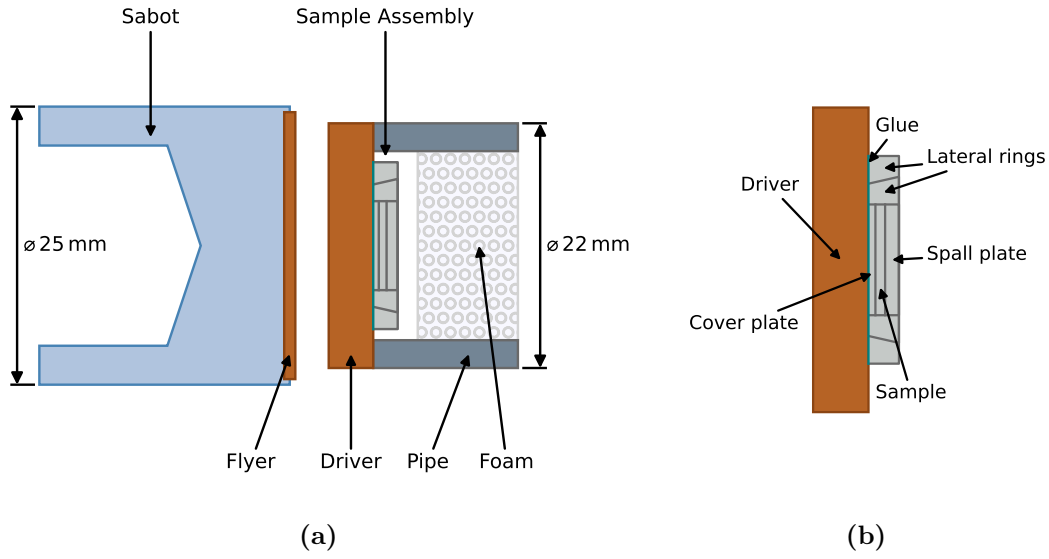


Figure 6.1: Schematic representation of the recovery experiment geometry, left, with sample detail right. Sabot body and pipe have been shortened to aid visualisation; other components retain their correct proportions.

glued to the copper driver plate. While the geometries, especially the thicknesses, are different from the target design used in the previous chapters, the design is intended to be as similar as possible so the loading conditions are comparable.

6.1.2 Experimental Design

The sample assembly is placed within a steel tube, this tube having several key purposes. Firstly, the tube has a smaller outside diameter than that of the sabot. The body of the sabot is therefore carried by its momentum outside the tube, reducing the likelihood of secondary impacts, and removing the need for a dedicated sabot stripper. The use of a copper driver plate also acts to seal the tube, further protecting the sample. Secondly, by placing the driver plate over the end of the tube, significant energy is dissipated into the tube and into shearing the driver plate and sabot. Thus some momentum is carried by the tube, reducing the kinetic energy of the fragments of the impactor. To catch the sample, a low-density foam, Rohacell 31 IG-F, is used to fill the tube. In the highest velocity shots, the stopping distance is around 70 cm. This foam is vacuum compatible, making it suitable for plate-impact experiments.

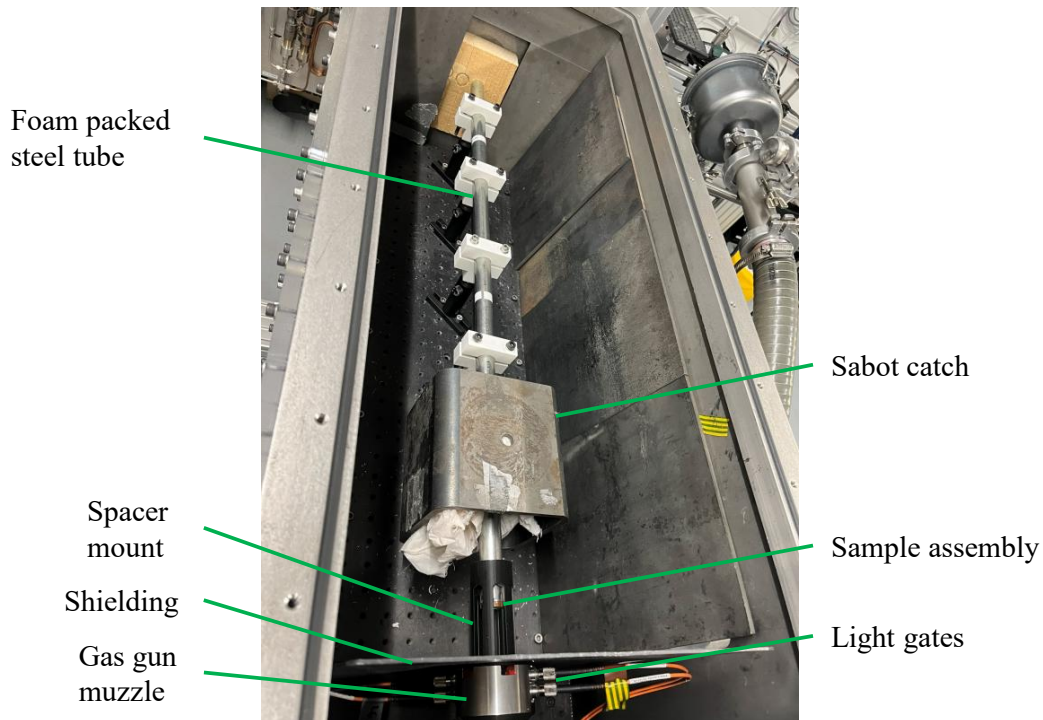


Figure 6.2: Picture of the recovery experiment with the target chamber of the 25 mm bore single stage gas gun. Key features of the experimental design are indicated.

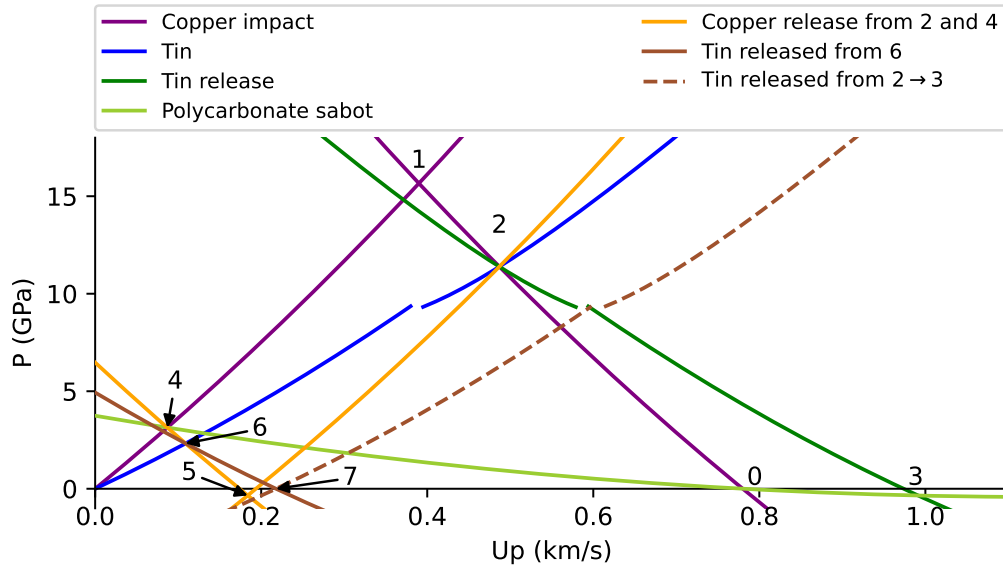
To enable the radial release of the sabot, the tube is spaced from the muzzle of the gun, see Figure 6.2. To ensure concentricity, recovery tubes are aligned to a laser shone down the barrel prior to each experiment. A 3D-printed mount is used to directly support the tube on the barrel, further ensuring good alignment. Through these precautions the shock tilt is minimised. Placing a mirror or probe behind the target is impractical due to this experimental design, and would likely damage the sample during recovery, meaning velocimetry cannot be fielded to directly measure the shock tilt. However, it is presumed it is not significantly different from the other experiments in Chapters 4 and 5.

A further feature of this design is the use of the sabot to not fully release the copper flyer and driver. By backing the flyer with polycarbonate, the release from the rear surface of the flyer is incomplete, state 4 in Figure 6.3, resulting in some retained momentum in the sample. Thus the sample is propelled, reducing the relative velocity between itself and the flyer and driver plate, reducing the likelihood and severity of secondary impacts. The alternative, to place a void behind the copper

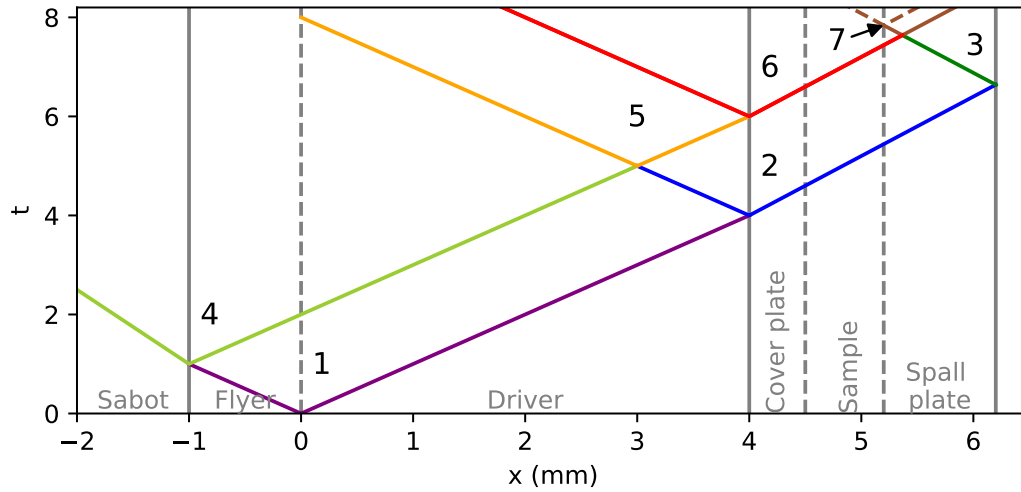
would mean the sample would not retain any momentum. While this would appear to aid recovery, the rear surface of the void, required to seal the propellant gas, would then impact the slowly moving flyer and driver, which then impact the sample. The "reverse-cup" design, pictured in Figure 6.1a, thus propels the sample, is sufficiently long to minimise the impact tilt, and reduces the likelihood of secondary impact.

This design was optimised and validated using Ansys Autodyn software. Experimental geometries were simulated to verify that the samples did not experience significant forces from lateral release waves or secondary impacts. The simulation geometry is shown in Figure 6.4a. More details about the simulation methods and parameters can be found in Appendix 6.5.1. While this approach represents a simplified version of the problem, the simulation is able to aid understanding of the motion of the sample prior to interaction with the foam, thereby improving confidence in the design. In particular, the relative speeds of the flyer and driver plate is a key takeaway, Figure 6.5. These velocities determined from the simulations agree with the 1D $P - U_p$ curve matching shown in Figure 6.3. As can be seen in Figure 6.4 and Figure 6.5, the sample is shown to retain sufficient velocity to slightly out pace the driver plate, reducing the likelihood and severity of secondary impacts.

While these experiments aim to closely reproduce the loading in the previous chapters, pre-cooling the sample could reduce the temperature of the post-shock state, and reduce any post-recovery recrystallisation. The use of the capped tube offers the possibility to pre-cool the sample by fully sealing the tube and flooding with liquid nitrogen or helium. However, in the current experimental work, no cooling is applied. Given the possibility of recrystallisation forming part of the mechanism of plasticity in shocked tin, pre-cooling may alter the mechanisms of shock. Future investigations may wish to investigate this phenomenon, especially given tin is known to exhibit different plastic behaviour when cooled [16], and the design presented here can easily be adapted to achieve this goal.



(a) Pressure-particle velocity diagram of states predicted in the recovery experiment. Hugoniot taken from literature [25, 134, 164, 283]



(b) Displacement-time diagram of states predicted in the recovery experiment. Release waves, wave interactions and wave speeds have been simplified to aid visualisation.

Figure 6.3: Diagram of states predicted in a recovery experiment using a 1D analysis. After the initial impact (1), a compressive pulse is generated in the copper driver and flyer. The driver then partially releases, driving compression in tin (2). This compressive wave then reflects from the free surface of the spall plate (3). The initial compressive wave also propagates through the flyer until it partially releases into the polycarbonate sabot (4). This release wave then meets the release from tin, forming state 5 in the driver plate. This state then reaches the tin components, recompressing to state 6. Finally state 3 and state 6 meet, creating a tensile region in the spall plate. The interface between the spall plate and sample, having no resistance to tension, opens when the stress reaches zero. With the geometry ensuring wave $2 \rightarrow 6$ traverses the interface first, zero pressure occurs when the release wave $2 \rightarrow 3$ traverses the interface, reaching state 7.

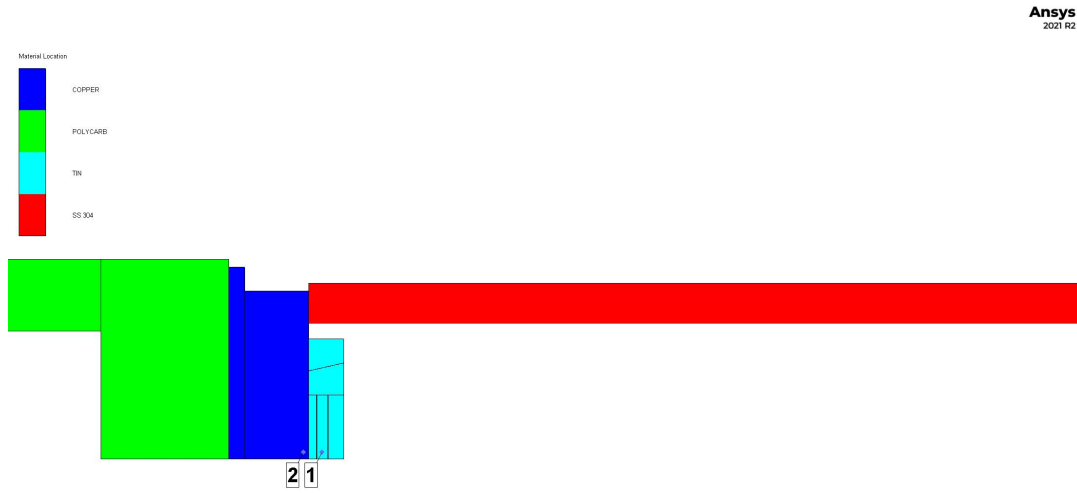
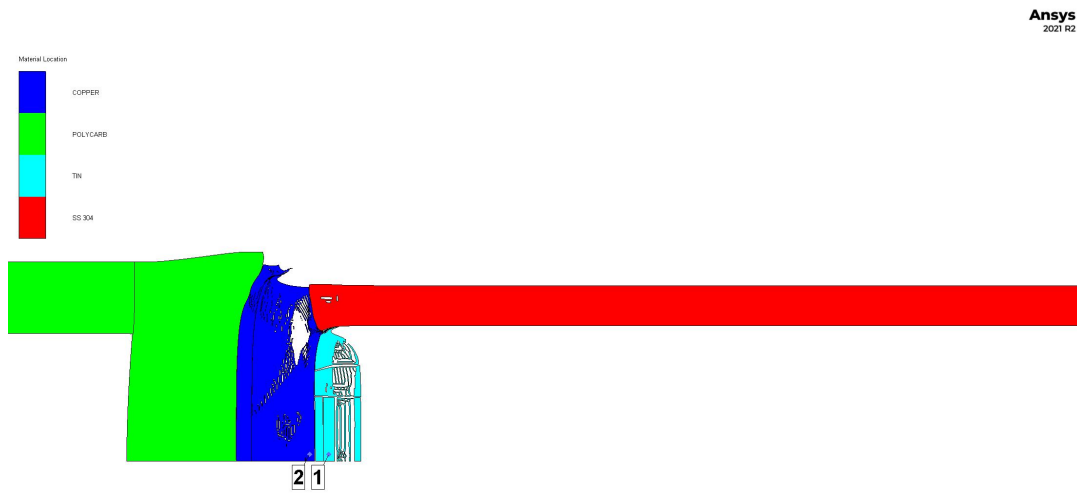
(a) $t = 0 \mu s$ (b) $t = 2.71 \mu s$

Figure 6.4: Collage of images showing progression of the recovery experiment. Points 1 and 2 mark the tracked locations whose history is shown in Figure 6.5, with point 1 on the sample and point 2 on the driver plate.

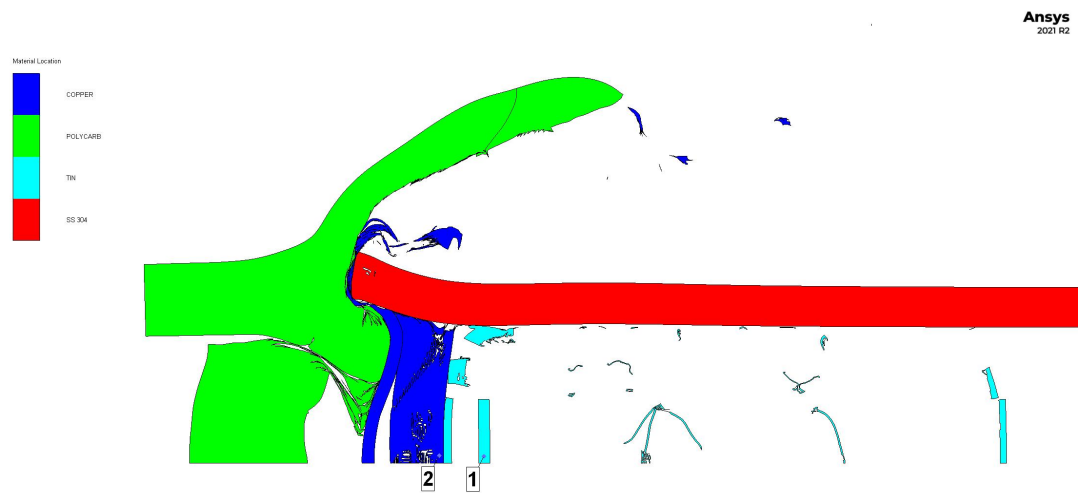
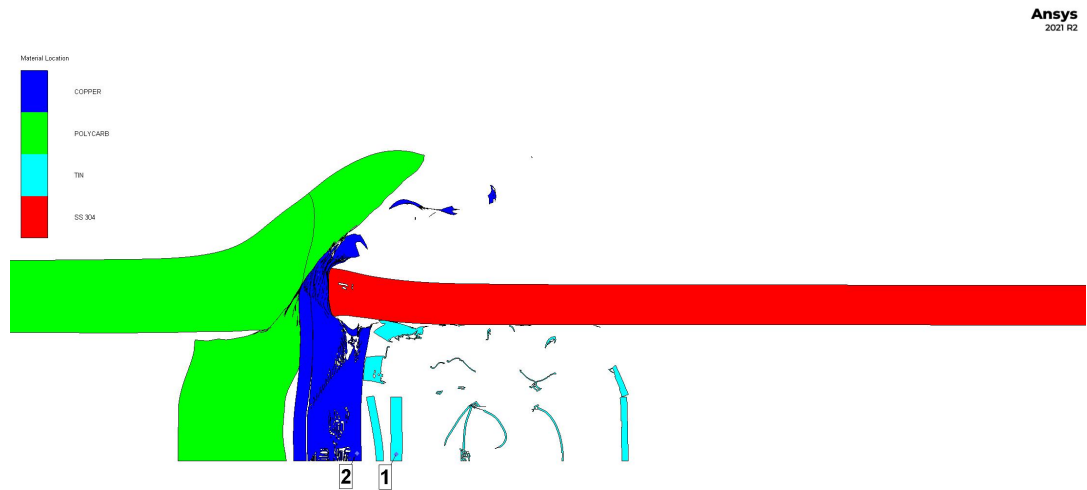


Figure 6.4 (continued): Collage of images showing progression of the recovery experiment .

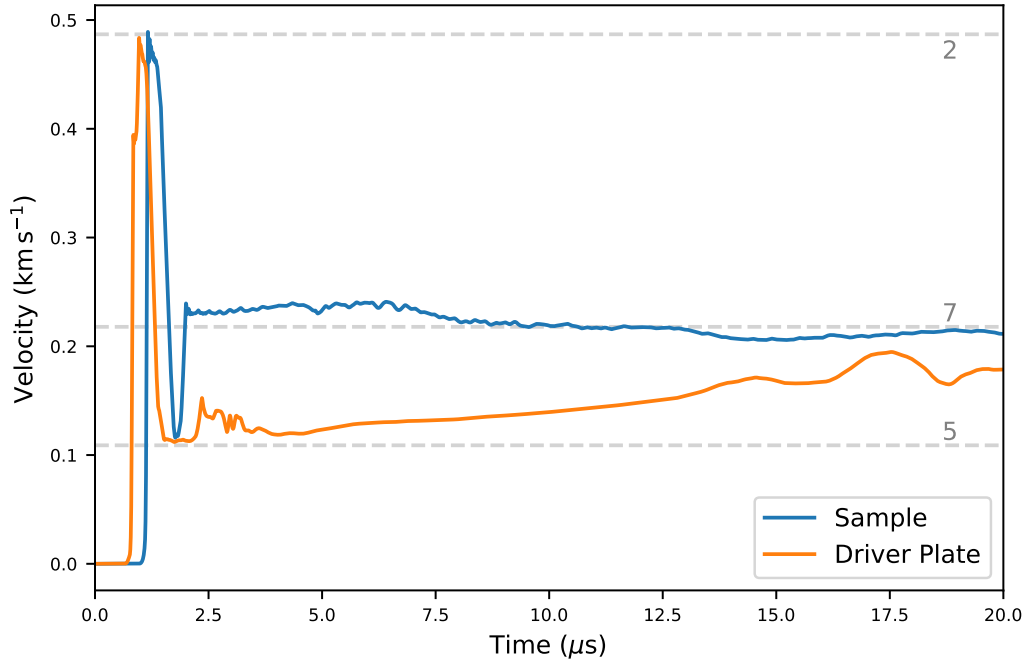


Figure 6.5: Velocity history of the flyer and the driver plate. The particle velocities of internal states predicted using the 1D theory in Figure 6.3 are plotted as dashed lines.

6.1.3 Samples

Experimental data are tabulated in Table 6.1. Impact velocities are determined from light-gates situated on the muzzle of the gas gun, while peak pressures are estimated using impedance matching techniques. For copper, the linear shock-particle velocity relationship given by Marsh [164] is used, while for tin, the multiphase Birch-Murnaghan model of Mabire is used [25, 134]. Three polycrystal samples are tested, alongside three each of orientation (001) and (100). Two polycrystals are shocked to a state above the $\beta \rightarrow \gamma$ phase transition, and one below. For (001), one shock is conducted above the transition, one below, and one such that the transition is expected to be incomplete before release to ambient conditions.

Orientation (100) is noted for its particularly large precursor, see Chapter 4. Using a 1.25 mm Al6061 aluminium flyer to reduce the peak shock pressure, one recovery experiment sits below the HEL values observed in Chapter 4. A further shot exceeds these values, but remains below the $\beta \rightarrow \gamma$ transition, while the last is above the transition. Following the analysis conducted in Chapter 4, the pressure

Table 6.1: Impact conditions for recovered samples.

Sample	Impact Velocity (m s ⁻¹)	Estimate Peak Pressure (GPa)	Predicted Peak State
PC _{R-1}	783 ± 2	11.4	γ
PC _{R-2}	532 ± 2	7.8	β
PC _{R-3}	735 ± 1	10.7	γ
100 _{R-1}	538 ± 2	7.9	β
100 _{R-2}	738 ± 3	10.7	γ
100 _{R-3} ¹	573 ± 1	5.0	Elastic
001 _{R-1}	541 ± 1	7.9	β
001 _{R-2}	759 ± 3	11.0	γ
001 _{R-3}	652 ± 2	9.5	$\beta - \gamma$ mix

¹ This experiment uses a 1.25 mm thick Al6061 flyer (for all other samples, a 1 mm C101 copper flyer is used).

for the shot below the HEL is estimated using the linear velocity relationships for Al6061 and copper given by Marsh [164], alongside a third order elastic model for tin using the second order parameters of Rayne and Chandrasekhar [39] and the third order from Swartz *et al.* [43].

As-received polycrystal samples are also scanned. These, alongside the recovered polycrystal samples, are cut from the same stock bar as the samples in Chapters 4 and 5. The as-received polycrystals are of two thicknesses: 1 mm and 2 mm, the latter corresponding with the thickness of the samples used in Chapters 4 and 5. These samples are denoted by AR, with AR1 used for the former, while AR2 is used for the latter.

6.1.4 EBSD and Sample Preparation.

Recovered samples are bisected, polished and an EBSD scan performed. Of the two parts, one is scanned along the cut, longitudinal to the shock direction, and one on the sample face transverse to the shock direction, shown in Figure 6.6b. Thus, any sample texture can be highlighted and examined from these two key angles. Due to the softness of tin, samples are embedded in a resin prior to polishing and scanning to prevent deformation. For this purpose, the cold mounting resin EpoFix is used to prevent recrystallisation through elevated temperatures during curing.

Samples are polished in the following steps. First, silicon carbide grinding discs are used to remove any resin on the sample surface, and ensure that the surface is flat, parallel, and retains no residual surface texture from machining. Grit numbers #800, #1200 and #4000 are used sequentially for this purpose. After this, the samples are polished, first using a 3 μm diamond solution, and ultimately using a fumed silica suspension.

Scans were performed using the JOEL IT-800 fielding a EDAX/Gatan Velocity EBSD Camera at the David Cockayne Centre for Electron Microscopy within the University of Oxford. Size limits for EBSD scans depend on factors of the equipment, such as beam current and energy, sensor size and efficiency, factors of the sample, most importantly efficacy of the conductive path, and the user's tolerance to low signal to noise ratio and drift. The scans taken here push the upper envelope of the equipment used, with dimensions approximately 3 mm by 2.5 mm common. Increasing the size of the scan would require stitching multiple scans together. Bisected samples are semi-circles with radii of 4 mm, and scans already cover much of the body of the sample, meaning stitching scans would exhibit lower yields relative to the increased duration and computational cost. Raw Kikuchi patterns were indexed to library data using APEX software, with a spherical indexing routine determining orientation through comparison to a simulated pattern.

6.2 Results

After data collection and indexing, an uncertainty threshold is applied to the raw data to remove poorly indexed regions. A grain boundary analysis is then conducted, with misorientations greater than 1° flagged. The grains are relatively homogeneous, with almost no internal misorientation, and distinct from their neighbours, meaning the choice of angle within reasonable bounds has almost no effect on the resulting plots. Consequently, a conservative value is taken, to avoid missing any boundaries, and highlight the smoothness of the grains. Grain boundaries within 3° of the (301) twin relationship (see Section 2.1.3) are marked with red lines.

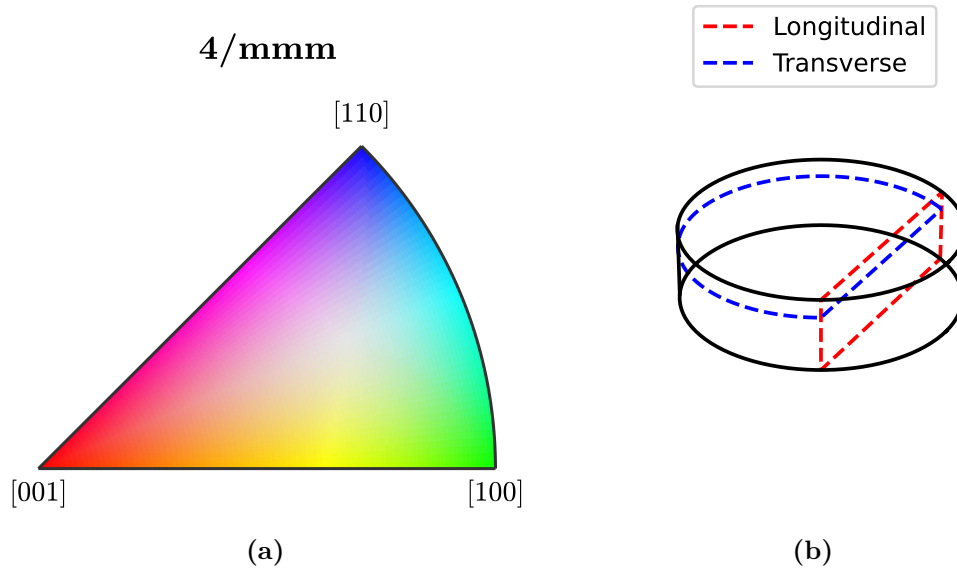


Figure 6.6: Key for grain orientations in EBSD maps, left, alongside a diagram showing approximate planes of scans within the cylindrical sample, right.

Small grains are excluded from the analysis, with pixels that form grains with fewer than 10 total pixels removed. These pixels would otherwise introduce noise into the plots, occasionally internal to grains, but most commonly forming as bands, single pixels thick, along the edges of the samples, or small clusters outside the sample. Notably, these pixels are often found in clusters, each pixel highly misoriented with respect to their neighbours. With most grains containing hundreds of pixels, some even thousands, these small grains are assumed to be scanning artifacts. EBSD plots shown in this Chapter are otherwise raw data, with no filling or smoothing applied. The key for grain orientations is shown in Figure 6.6a. Full pole figures can be found in Appendix 6.5.2.1, while grain size distributions and grain boundary misorientations are found in Appendix 6.5.2.2.

6.2.1 As-Received Material

The raw scans for the as-received polycrystalline material are presented in Figure 6.7 for 2 mm samples and Figure 6.8 for 1 mm samples. These show large grains, with millimetre order lengths scales. A slight aversion of (100) with the transverse normal is observed. Twinning is also prevalent in several grains. Due to the destructive nature of the sample preparation and inability to safely remove samples

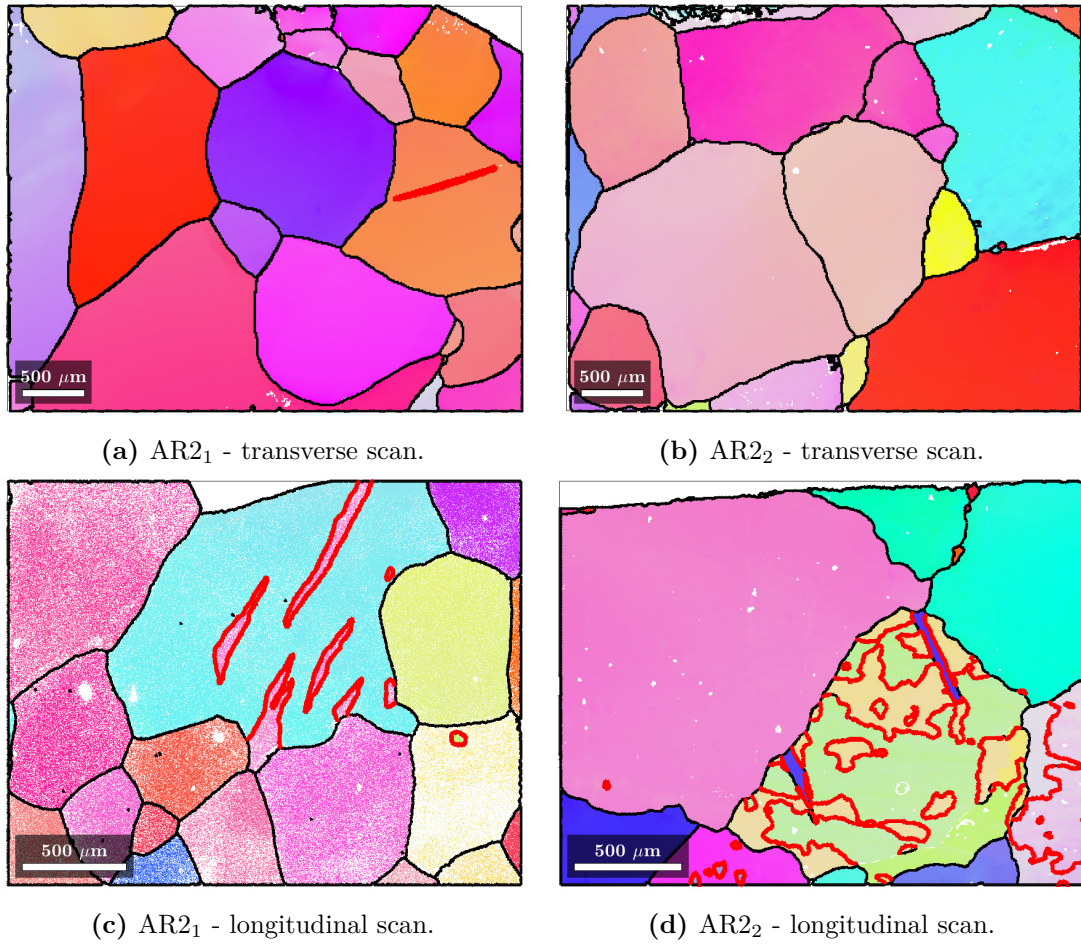


Figure 6.7: EBSD maps for two as-received samples with a thickness of 2 mm. Grain boundaries are shown as black lines, while twin boundaries are marked with red lines. For the orientation map key, see Figure 6.6a.

from mounting resin, no scans were taken of as-received single crystals. However, given they are provided from the manufacturer as previous chapters, it can be assumed that the properties are similar to the data tabulated in Table 4.1, and the Laue diffraction in Figures 4.3c and 5.6c.

6.2.2 Shock Recovered Samples

Recovered sample scans are shown in Figures 6.9, 6.14 and 6.11, for samples (001), (100) and polycrystal samples, respectively. Very little twinning is observed, and grains show very little internal misorientation. Compared to the as-received material, these generally show much smaller grain sizes, and an avoidance of (001)

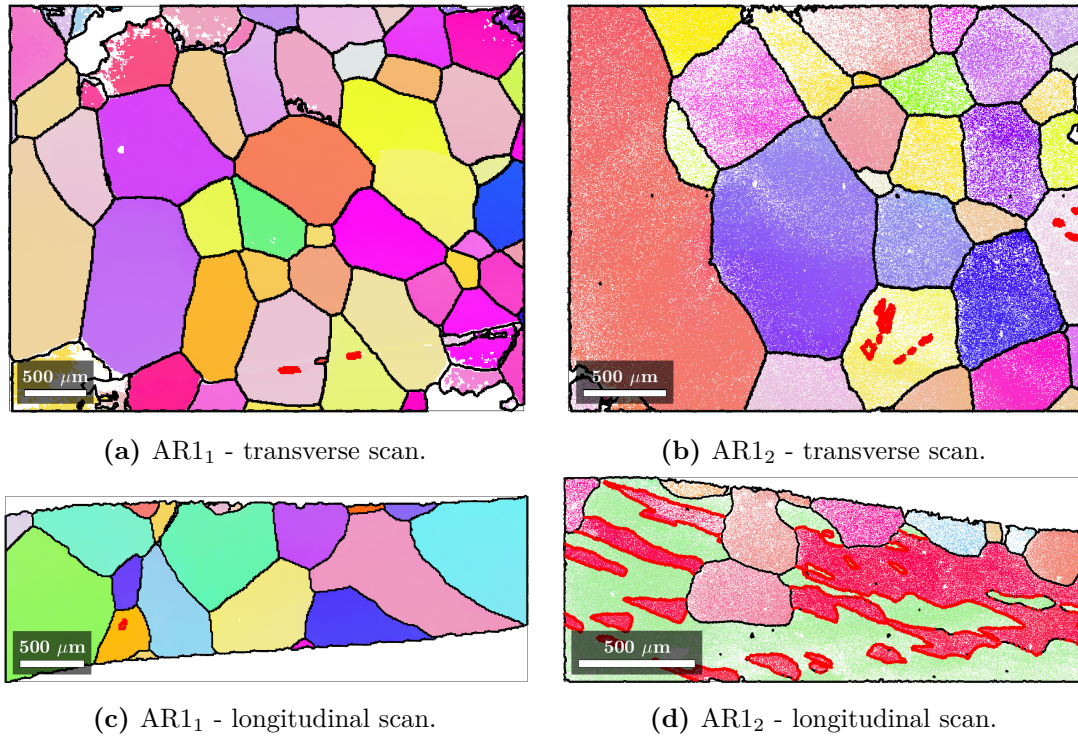


Figure 6.8: EBSD maps for two as-received samples with a thickness of 1 mm. Grain boundaries are shown as black lines, while twin boundaries are marked with red lines. For the orientation map key, see Figure 6.6a.

being oriented along the shock direction. The small grain size is in contrast to typical re-crystallisation in heated tin, where grains typically grow larger [284, 285]. However, smaller grain sizes and less twinning is consistent with the high-rate recrystallisation observed in Taylor tests [49].

6.2.2.1 Recovered (001) Single Crystals

In a number of grains in samples that started as (001), it appears that the c and a axis have roughly swapped. In an ideal (001) crystal, (100) poles would be oriented at 90° angles from the normal, and from each other, see Figure 6.10. However, in recovered samples, a significant reorientation of grains to align (100) with the shock direction is observed. In the highest pressure sample, 001_{R-2}, this occurs in almost all grains.

A full 90° rotation is unexpected; this behaviour does not match the existing known twins in tin, these being (301) and (101). At ambient conditions, the plane (102) is perhaps the closest, with an angle of 42.8° from the (100) direction

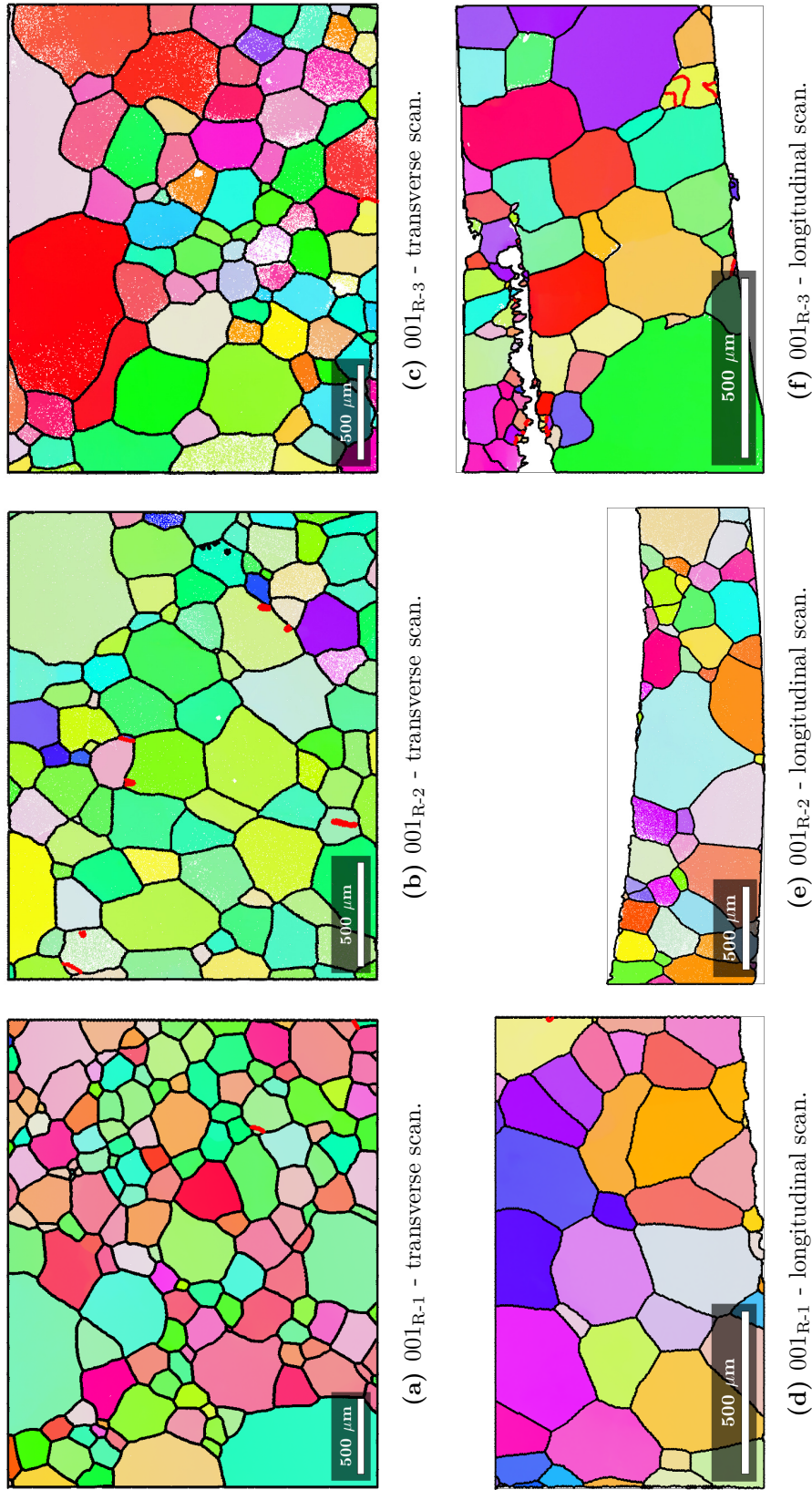


Figure 6.9: EBSD maps for shock recovered single crystals with orientation (001). For the orientation map key, see Figure 6.6a. Sample 001_{R-2} is predicted to transform to the γ phase, while 001_{R-1} is predicted to remain β . Sample 001_{R-3} is expected to partially transform to γ , with release occurring prior to reaching a constant peak state.

at ambient conditions. Even assuming this is a possible twinning operation, it would still require additional plastic rotation to match the observed results. This behaviour may be explained through repeated operation of other systems, although a pairing of any of the 301 and/or 101 cannot achieve this rotation, with each resulting in a roughly 60° misorientation.

Instead, the rotation might be associated with the γ phase. In MD simulations, a flip transform of the c and a axis in γ is observed when the c axis is compressed [147]. When β tin transforms to γ , this axis flip has also been observed, hypothesised to occur through a twin about the (101) plane [146]. However, this twin plane would not correspond to a direct axis flip (a 90° rotation directly transplanting [001] onto [100] or [010]), unless the c/a ratio is unity.

A transformation occurring in γ tin explains why 001_{R-2} (predicted to be fully γ) has almost completely transformed, and why 001_{R-3} (predicted to be $\beta - \gamma$ mix) has partially transformed. However, sample 001_{R-1} (predicted to remain β) corresponds with pressures far below the split wave profile indicative of the phase transition (see Chapter 4). Notably, in static measurements, γ has been observed to start to form at pressures as low as 6 GPa [129], while β has been observed as high as 13 GPa. Considering this, and the similarities between 001_{R-1} and 001_{R-3}, it is therefore likely that the transformation has at least partially occurred in 001_{R-1}, allowing some grains to flip.

6.2.2.2 Polycrystals

Polycrystal samples show the most diverse microstructure of any of the recovered samples. Damage is found in PC_{R-1} and PC_{R-3}, and these regions are bisected in the transverse scans. The grains in these damaged regions are more randomly oriented, smaller, with analysis of these areas discussed in Chapter 6.2.3. The undamaged regions however, show a tendency to avoid aligning the [001] direction with the z -axis of the sample (the shock direction), with a greater prevalence of grains oriented between [110] and [100]. This would match with randomly oriented grains re-orienting with the axis flip observed in the single crystal samples.

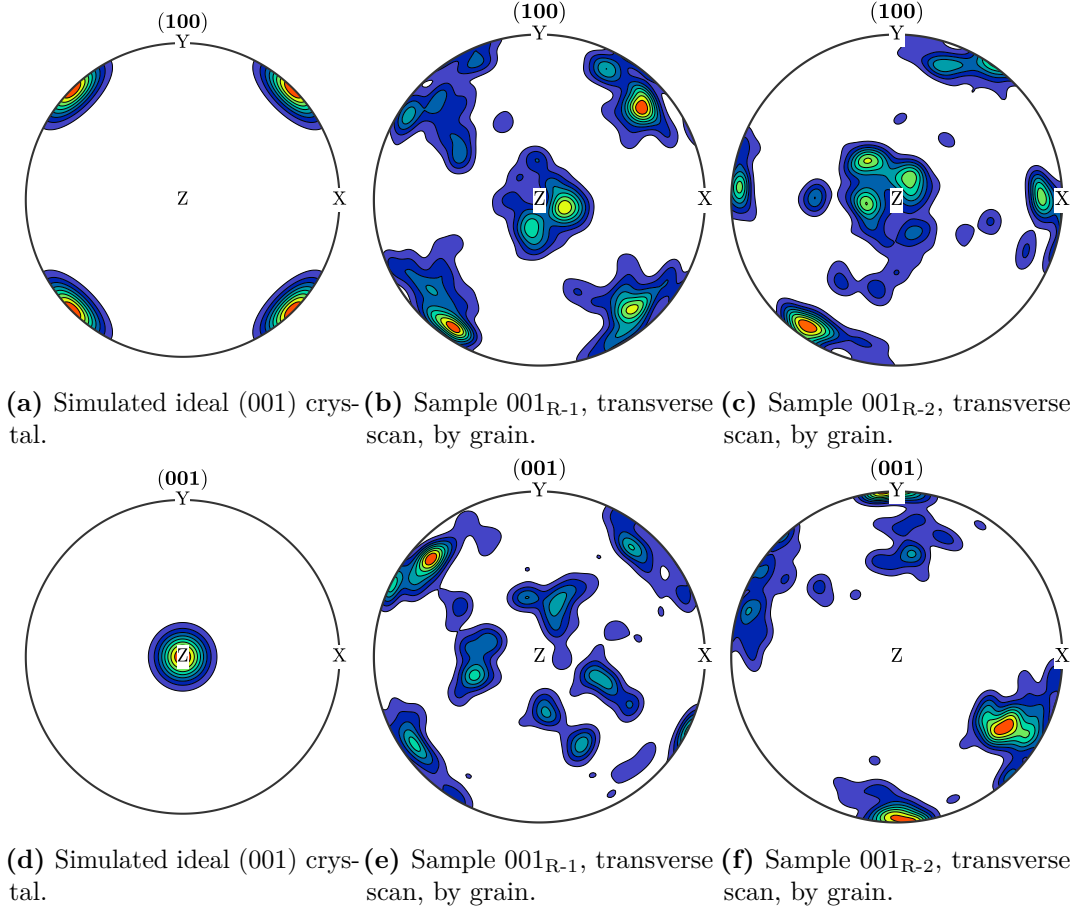


Figure 6.10: Selected pole figures for (001) samples. Sample 001_{R-1} is predicted to transform to the γ phase, while 001_{R-2} is predicted to remain β .

Supposing that $[001]$ would swap with the $[100]$ or $[010]$ direction if those directions were closer to perpendicular to the z -axis, any crystal where $[001]$ was within 45° would be expected to transform. Crystals where $[001]$ was between 45° to 54.7° would have one of $[100]$ or $[010]$ closer to the z -axis than $[001]$, but could still interchange the other with $[001]$. In this case the angle difference would be less, reducing the effects of the transformation. For angles greater than 54.7° , $[001]$ can be the axis closest to perpendicular from the z -axis. In some grains in this range, one of $[100]$ or $[010]$ could still be closer to perpendicular, and therefore could still swap, although the angle change would likely be small.

Indeed, comparing the inverse pole figures (Figure 6.12) highlights this trend. Comparatively fewer grains where $[001]$ is aligned to within the previously mentioned angles to the z -axis are observed in the polycrystal samples. In sample PC_{R-2}, the

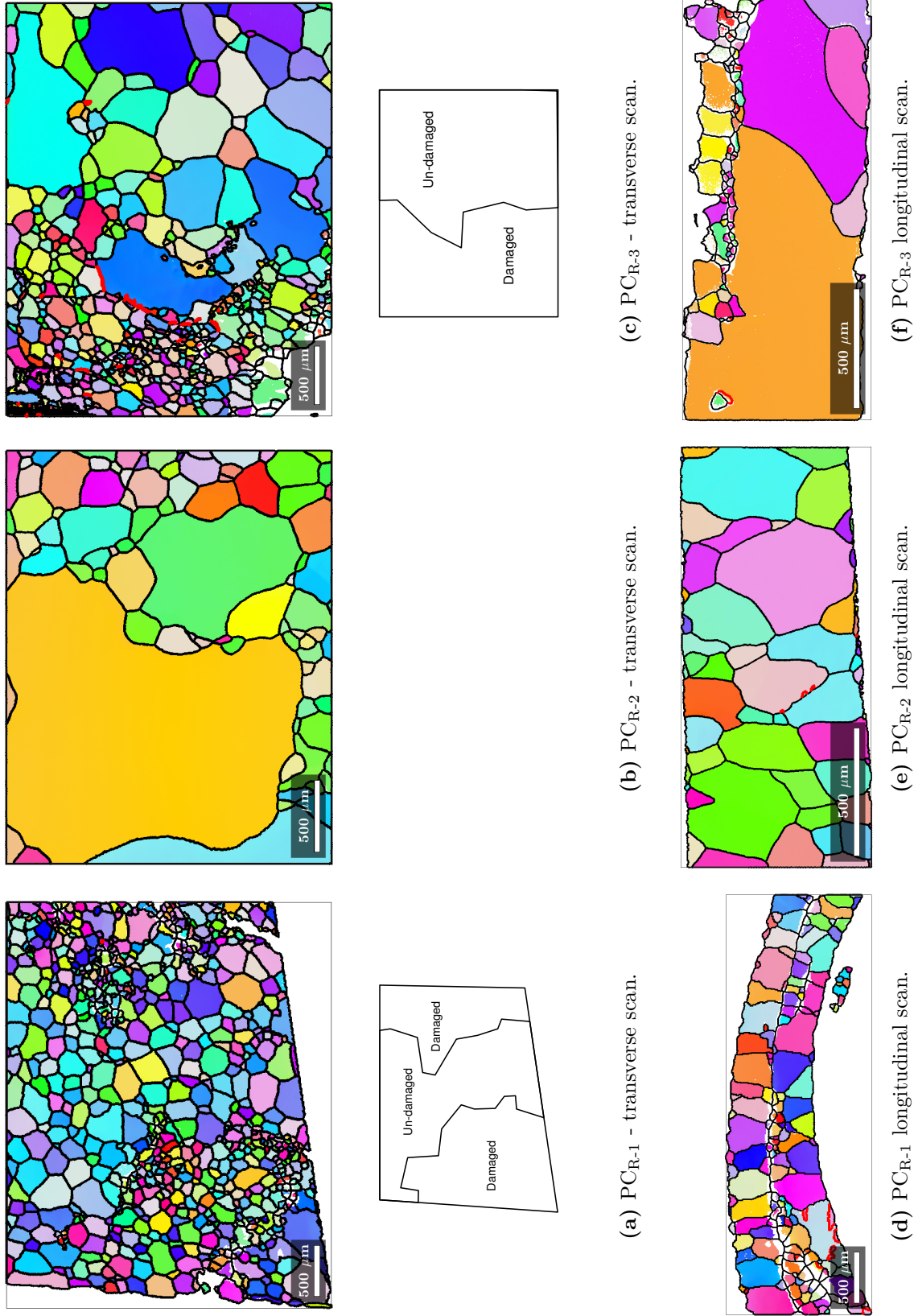
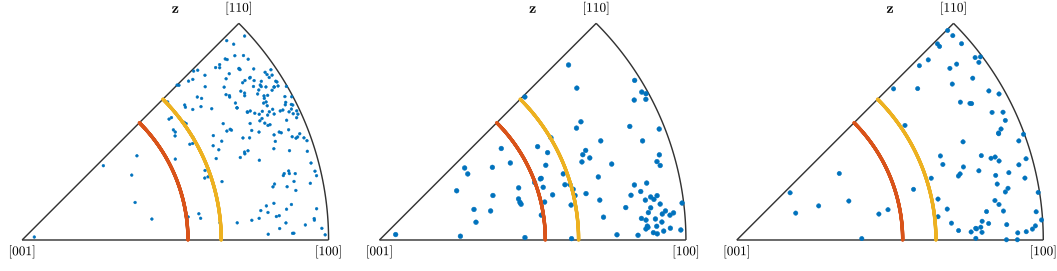
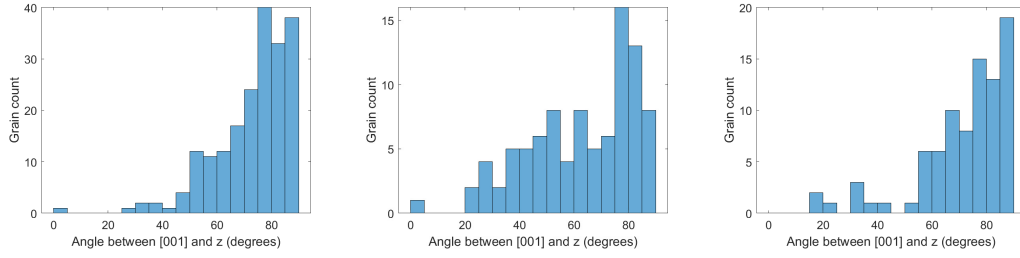


Figure 6.11: EBSD maps for shock recovered polycrystal samples. PC_{R-1} and PC_{R-3} are both hypothesised to transform completely to γ at the peak state, while PC_{R-2} is predicted to remain β .



(a) PC_{R-1} , transverse scan, undamaged region. (b) PC_{R-2} , transverse scan. (c) PC_{R-3} , transverse scan, undamaged region.

Figure 6.12: Inverse pole plots for recovered samples. Red line denotes where the [001] direction is 45° from the z -axis, below which it would be the closest principle direction to the shock axis, while the yellow line denotes 54.7° , the smallest angle where no direction could be further from the shock axis. PC_{R-1} and PC_{R-3} are both hypothesised to transform completely to γ at the peak state, while PC_{R-2} is predicted to remain β .



(a) PC_{R-1} , transverse scan, undamaged region. (b) PC_{R-2} , transverse scan. (c) PC_{R-3} , transverse scan, undamaged region.

Figure 6.13: Histogram of angle between the [001] axis in each grain and the z -axis for each grain in polycrystal samples.

only polycrystal sample that has not reached the phase transition step, the flip is less prevalent, with a greater number of grains oriented with [001] closer to the loading axis than in the samples that have completed the transition.

However, very few grains are oriented with [001] within 20° of the axis, see Figure 6.13. This suggests that the grains with [001] closest to the axis have transformed to γ , before swapping their axis, resulting in the spike around 80° . Meanwhile, samples which have reached the theoretical phase transformation pressure, and been allowed enough time to transform, have almost completely flipped axis wherever possible, resulting in most very few grains below 55° , and most grains with angles above 75° .

6.2.2.3 Recovered (100) Single Crystals

Samples that started as (100) show unusual behaviour compared to the other samples. The distribution of orientations are much more tightly clustered, possibly due to the presence of fewer grains. Sample 100_{R-2} shows two particularly large grains. Notably the grain size decreases noticeably towards the edge of the sample. This shot is the second highest impact velocity, and therefore the largest peak and residual temperature, which may explain the unusually large grains through post-recovery recrystallisation.

A striking feature of the pole plots of these samples, Figure 6.15, is the symmetry. In sample 100_{R-3}, below the HEL, a clear two-fold symmetry is observed, while 100_{R-1} (predicted to remain β) and 100_{R-2} (predicted to completely transform to γ), show a four-fold symmetry. The well defined nature of the poles, with limited spread and no intermediate points suggest that the rotation is completed in one step. A slip driven rotation is possible, but the explanation behind the limited resultant angles is not clear.

Examining 100_{R-3}, it can be seen that operation is roughly equivalent to a $\pm 20^\circ$ rotation about $[100]$ and a $\pm 20^\circ$ rotation about the $[010]$ direction. This could correspond to the double operation of a twinning system of the form $(x\ 0\ 1)$ followed by $(0\ x\ 1)$, of which two, $\{3\ 0\ 1\}$ and $\{1\ 0\ 1\}$ are established to operate in tin. However, neither of these two systems would produce the measured rotations, with each corresponding to approximately a 60° rotation. A higher magnitude x , around $x = 9$, yields a better estimate. A single twin of the form $(9\ 9\ 2)$ could also result in a similar pole figure. However, neither of these systems are known or hypothesised to operate in tin, nor represent symmetry planes in the crystal lattice. These transformations also do not fully reproduce the measured pole plots. Some deviation could be explained through additional plastic rotation from slip, post-recovery recrystallisation, and from the limits of analysing the relatively small number of grains. Despite this, the behaviour is unexpected, and does not agree with the existing understanding of the plastic behaviour of tin.

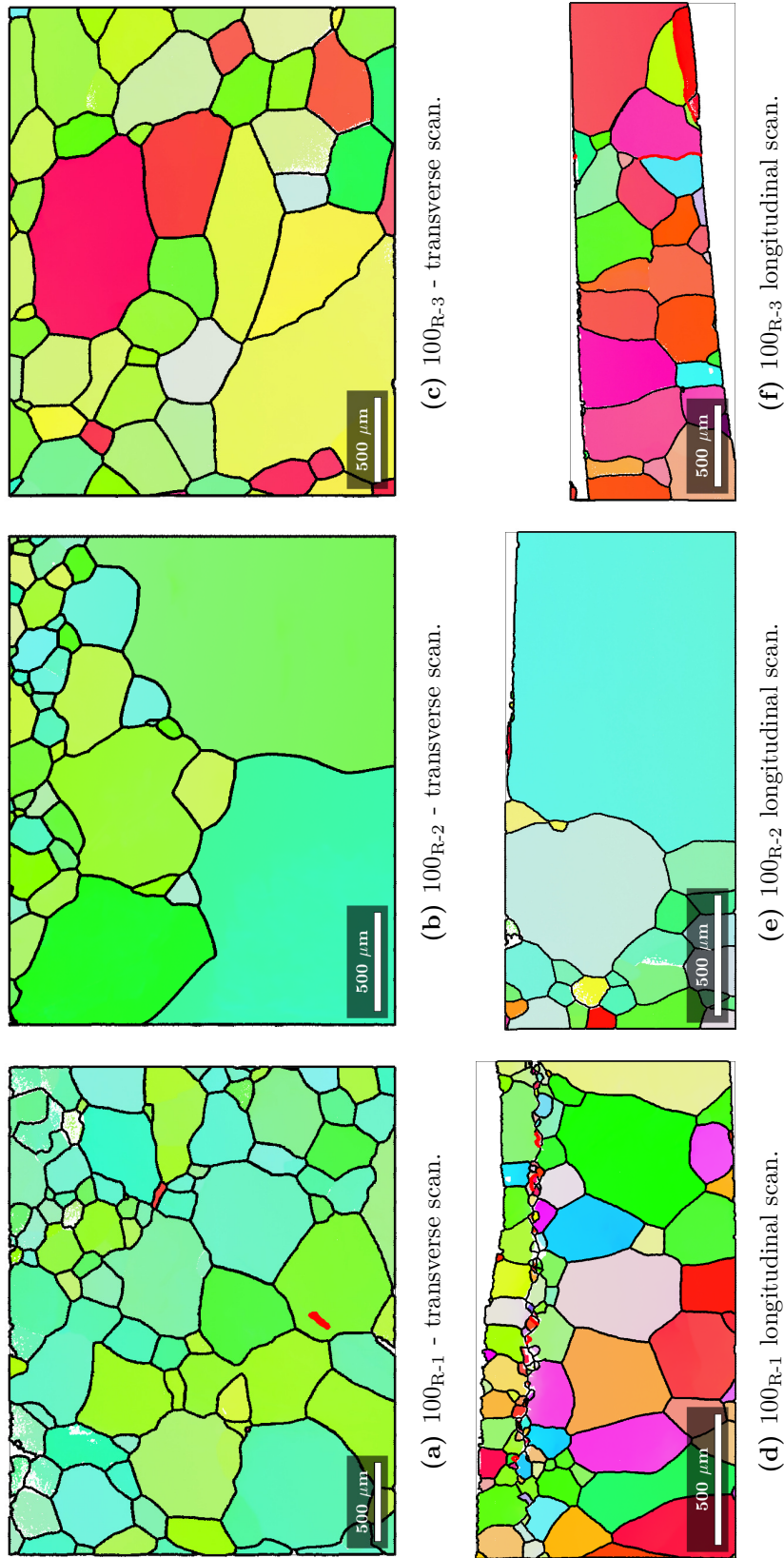


Figure 6.14: EBSD maps for shock recovered single crystals with orientation (100). For the orientation map key, see Figure 6.6a. Sample 100_{R-1} is predicted to remain γ , 100_{R-2} to completely transform to γ and 100_{R-3} to remain elastic at the peak state.

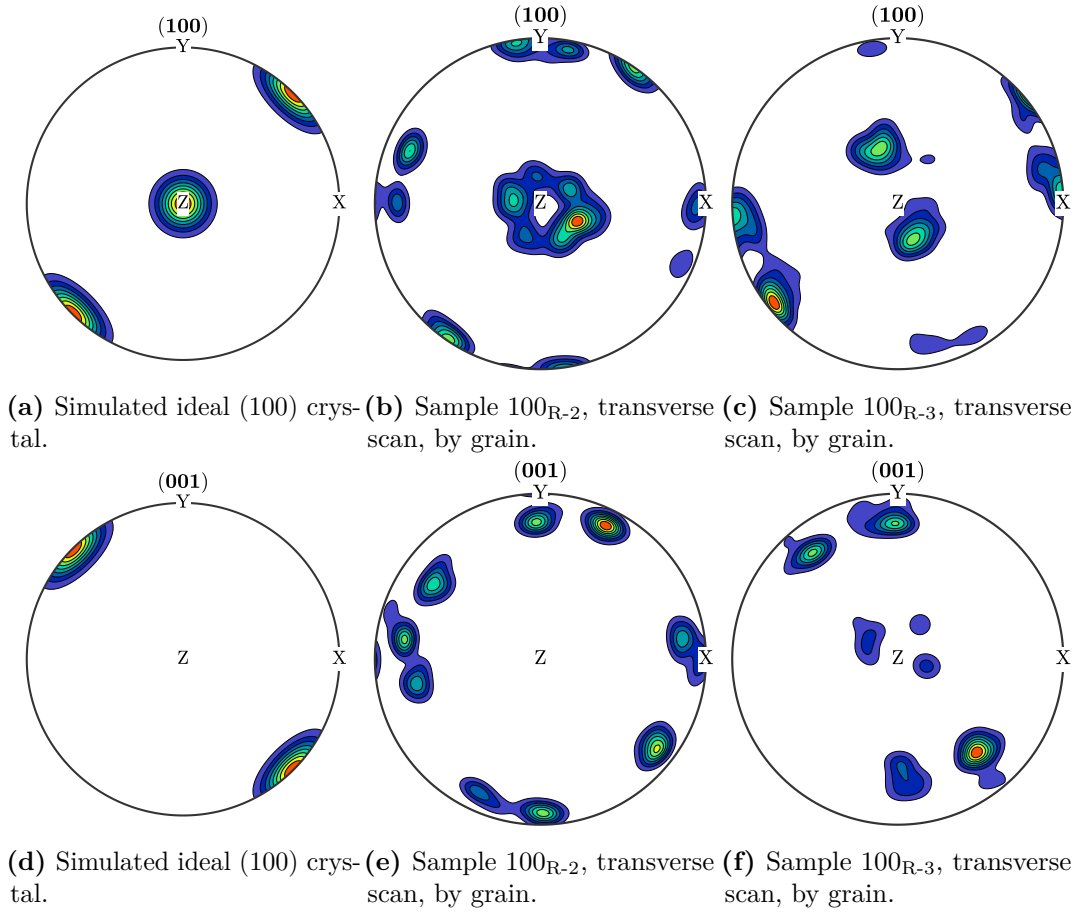


Figure 6.15: Selected pole figures for (100) samples. Sample 100_{R-2} is predicted to completely transform to γ and 100_{R-3} to remain elastic at the peak state.

Samples 100_{R-1} and 100_{R-2} show a similar trend, but with a four-fold symmetry. Considering the symmetry of 100_{R-3} and the apparent $c-a$ flip mentioned previously, it may be that, in some grains, a $c-a$ swap occurs. If this were to occur partially, creating new grains rotated around the z -axis by 90° , the pathways hypothesised in 100_{R-3} could reproduce the observed final microstructures.

If this was the potential pathway, the four central peaks would be oriented with the pairs along the centreline: a direct 90° rotation of the pole plots of 100_{R-3}. This would take the shape of a cross pattée (⌄), with each central pair aligned along the line of symmetry of the rotated outer pairs. This behaviour is more difficult to establish, possibly due to the spread of grains from an ideal structure. Several potential explanations are possible which may account for this, such as plastic slip and recrystallisation. Furthermore, several of the possible twins

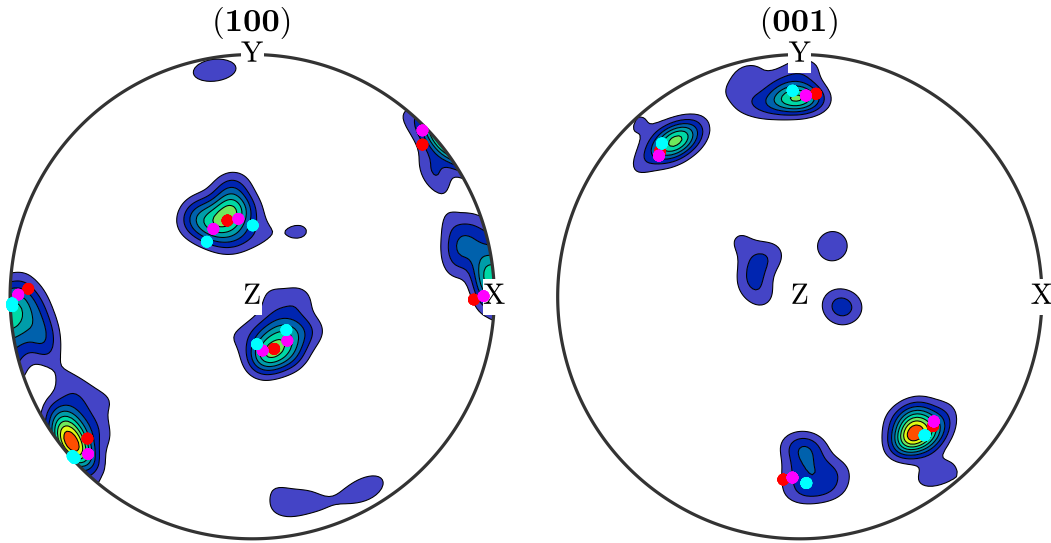


Figure 6.16: Pole plots for the transverse scan of sample 100_{R-1}, predicted to remain elastic at the peak state, by grain. Potential poles from an ideal (100) crystal are marked (cyan - a rotation of $\pm 20^\circ$ [100] and [010], red - a (0, 9, 1) twin followed by a (9, 0, 1) twin, magenta - a (9, 9, 2) twin.)

and rotations discussed for 100_{R-3} show split central peaks, which may further obscure the central pattern.

6.2.3 Spall and Damage

While many of the spall plates are highly deformed, as well as being contaminated with foam, the plate of 100_{R-1} survived the impact, and was sufficiently undamaged to enable dissection and scanning. Due to the proximity of the spall site to the interface, only partial spall has occurred, with resulting cross-sectional EBSD scans of this plate presented in Figure 6.17a and 6.17c. In several recovered samples, damage is also present. While this may be due to a secondary impact, partial transmission of release waves may also be a factor. Details of these regions are also shown in Figure 6.17.

Together these show significant grain refinement around the damage sites. This is most obvious in the primary spall observed in Figure 6.17a and 6.17c. Many smaller grains can be seen around the spall region, with cracks running at angles to the primary damage plane. The opening of these cracks, is perhaps unusual, with

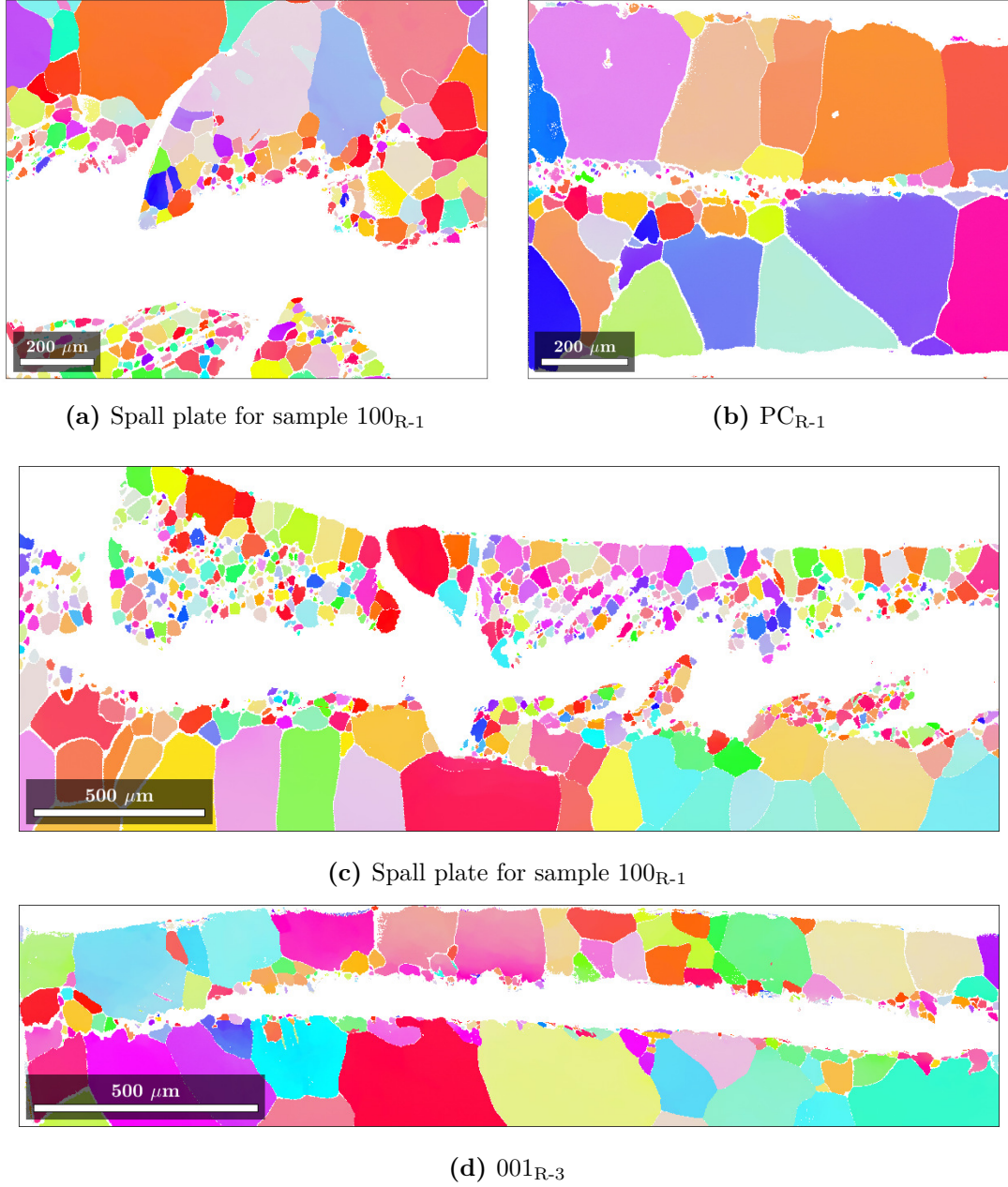


Figure 6.17: EBSD maps for areas of spall damage. Two scans are of the recovered spall plate of experiment 100_{R-1} (a and c), while others show detail from samples PC_{R-1} (b) and 001_{R-3} (d). For the orientation map key, see Figure 6.6a.

the primary tensile forces expected to act perpendicular to the damage plane.

The presence of these cracks appears in two primary forms. The first is in smaller, finger like protrusions, containing many smaller grains, and angles typically in the range 30° to 60° to the primary damage plane. This suggests perhaps that these cracks are related to shear flow. With flow expected to occur at these approximate angles from the primary loading, it may be that localisation of plasticity along the edge of these fingers forms voids, or grains so small they cannot be detected in the EBSD scans. However, the out of plane component cannot be examined through these 2D scans, meaning 3D techniques, such as tomography may be better placed to understand this phenomenon.

The second form of tangential cracks are at much steeper angles, with sharper boundaries, extending into the larger grains away from the primary damage region. These are possibly due to Poisson effects acting to open existing grain-boundaries. This is most obvious in Figure 6.17a.

In the secondary damage, for instance in PC_{R-1} , Figure 6.17b, some smaller grains are present, but fewer, and closer to the damage site than in the spall plate. In samples PC_{R-1} , Figure 6.11d, the longitudinal scans show similar small pockets of damage, close to the sample's rear surface, while the transverse scan, Figure 6.11a, shows regions of small voids surrounding small grains.

Whether these grains form as part of the damage process, or recrystallisation occurs after, nucleating around the higher levels of plasticity present in these area, is unclear. In either case, grain sizes appear to have expanded sufficiently to fill the available space between voids. However, both outcomes would rely on high degrees of plastic flow, this reinforces the lack of brittle fracture hypothesised in Chapter 5. In addition, these scans show several potential stages of damage, starting with initial void nucleation, then coalescence to create the jagged fracture surfaces shown.

6.3 Discussion and Conclusion

Several key observations are made of the recovered samples. Firstly, the samples have recrystallised, typically with a finer grain structure, though in a few samples

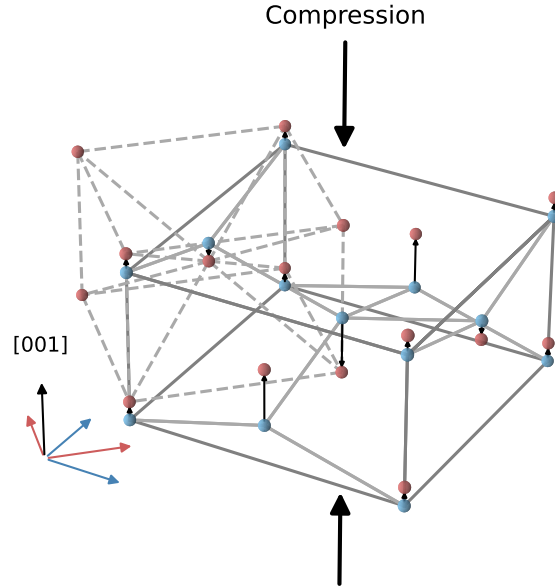
large grains have been formed. Secondly, this new microstructure is textured, with the [001] direction avoiding aligning with the shock direction. This appears to correlate roughly with a rotation of the crystal by 90° such that the [001] direction aligns with either the [100] or [010] (those being the same through symmetry). In samples that started as [100] single crystals, the recrystallisation texture shows additional symmetry.

The avoidance of [001] with the shock axis corresponds with a flip between the c and a -axis, and the phase transformation to γ tin. In polycrystal samples and (001) single crystals shocked above the phase transition, very few grains are left with [001] close to the shock axis, with the texture below the phase transition showing less of a change. Some flipping is observed to occur in lower pressure tests suggesting some nucleation of γ below the step observed in the wave profiles in Chapter 4. This appears to occur based on the proximity of the angle of the parent grain to the shock. Grains with [001] closest to the shock angle are absent in sample PC_{R-2}, indicating that these grains have transformed first, at pressures far below the predicted phase transition value.

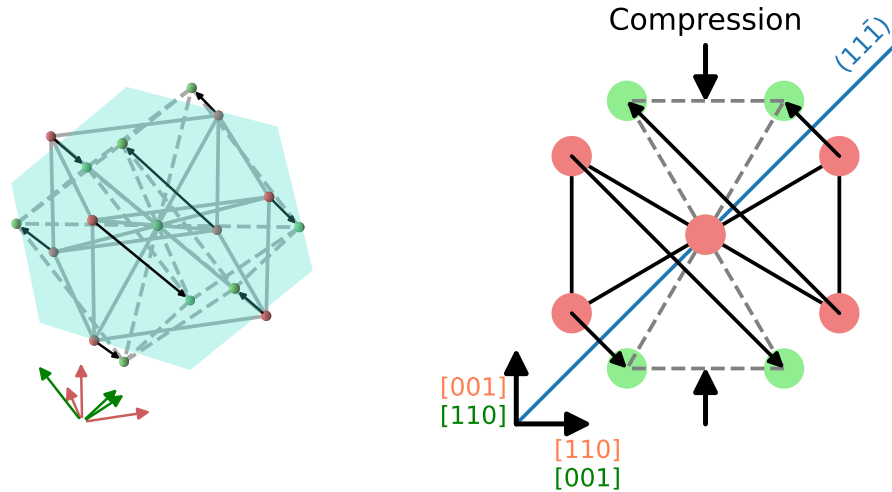
Two pathways for the transition are known with the relationship $(110)_\beta || (010)_\gamma$, $[001]_\beta || [001]_\gamma$ and $(101)_\beta || (110)_\gamma$, $[10\bar{1}]_\beta || [1\bar{1}1]_\gamma$ [146]. The former is particularly well positioned for the observed axis flip. With the [001] direction preserved the shock axis is therefore maintained in the γ phase. Indeed examining a starting and final β unit cell in Figure 6.18 highlights the rotation of the γ phase either side of the transition for this mechanism.

This rotation of the γ lattice between Figure 6.18a and Figure 6.18d corresponds with a $\{111\}$ twin, shown in Figure 6.18b. This maps the [001] direction onto the [110] direction in the new grain, resulting in the observed lattice rotation. This also acts to flip the direction of the isosceles triangles formed by neighbours in the $\{110\}$ plane of this phase, see Figure 6.18c.

While this operation cannot be directly observed in these experiments, the mechanisms would agree well with the experimental results. The proposed pathway would fit with the observed swap of the c and a axis in β tin. With all grains

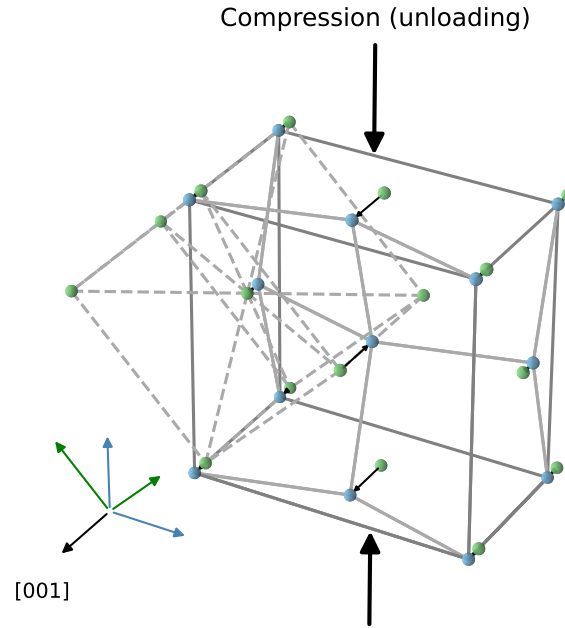


(a) Configurations of the β and γ lattices for the Musgrave [143] mechanism of the $\beta \rightarrow \gamma$ transition for a (001) β single crystal prior to shock. Grey dashed lines indicate the new γ unit cell and bonds, solid dark grey lines the β unit cell, and pale grey lines the bonds in β .



(b) Relative locations of the two γ lattices in (c) Mapping the rearrangement of atoms on Subfigures a and d, with the $(11\bar{1})$ plane shown the $(\bar{1}10)$ plane, corresponding with (010) in in blue and arrows indicating the symmetric the β unit cell, highlights the rotations made positions of the lattices. by nearest neighbour bonds.

Figure 6.18: Proposed pathway to the c/a flip observed in the recovered samples.



(d) Configurations of the β and γ lattices for the Musgrave [143] mechanism of the $\gamma \rightarrow \beta$ transition after undergoing the $\{111\}$ twin in γ tin. Grey dashed lines indicate the γ unit cell and bonds, solid dark grey lines the β unit cell, and pale grey lines the bonds in β . The β unit cell is now oriented with the $[100]$ direction where previously the $[001]$ direction pointed.

Figure 6.18 (continued): Proposed pathway to the c/a flip observed in the recovered samples.

swapping above the theoretical transition, this twin in γ tin would match the observed behaviour well. Below the theoretical transition, the pathway appears to only occur partially, with some grains retaining their initial orientation, as in 001_{R-1} and 001_{R-3} . Indeed, the $(110)_{\beta} || (010)_{\gamma}$, $[001]_{\beta} || [001]_{\gamma}$ pathway would conserve the $[001]$ direction, meaning the $(111)_{\gamma}$ twin operation would experience the greatest shear stresses in the grains when $[001]$ is close to the shock direction. In PC_{R-2} , the observation that grains with $[001]$ closest to the shock direction have transformed, would thus be reproduced.

An unusual aspect of this pathway is the apparent one-way nature of the twin. The direction $[001]$ maps to $[110]$, but the opposite does not occur, or if it does, to a much lesser degree. This may arise from the tetragonal nature of γ tin. When γ tin is compressed along $[001]$, it would become more tetragonal, with c/a diverging

from unity. Meanwhile, a 1-D compression along one of $\langle 100 \rangle$ would result in an orthogonal lattice, which has already previously been observed [34, 87, 101]. It may therefore be preferable to transform the lattice to become orthogonal under strain, to maintain the ratios of side lengths to be close to unity.

The nature of the recrystallisation is not clear. Under high temperatures, larger grains tend to form in tin [284, 285]. This may have occurred in several samples, most notably two of the higher pressure tests, 100_{R-2} and PC_{R-3}, but for most of the others, grain sizes are substantially smaller than in as-received material. However, the proposed mechanisms may offer an explanation as the cause of this behaviour. Considering that the γ phase likely nucleates at pressures far below the step in split-wave profiles in Chapter 4, new γ grains are seeded in the β grains. The nature of this nucleation and any preceding changes that facilitate the transformation cannot be established here, however, conventional plastic relocations may precede the transition. In single crystal samples, multiple nucleation sites clearly occur in a single grain. With the possible twins for this pathway, a refinement has already occurred, without needing to fully transform the material. Further twins are possible in the axis flip, but only likely if the two a -axes are located at similar angles from the shock direction, as is the case in (001) single crystals.

However, a clear caveat is that the temporal nature of recrystallisation cannot be definitively established through recovery experiments alone. Indeed, sample 100_{R-3} is assumed to remain elastic during the compression wave, based on the results of Chapter 4, and yet has a similar recovered grain sizes to the other samples. Similarly damaged regions show recrystallisation. This might imply that the recovered microstructures have recrystallised during release.

Post recovery recrystallisation is also a further possibility, due to the high temperatures relative to tin's melt point likely inducing some recrystallisation while the sample cools to ambient conditions after the experiment. This last point in particular may explain the smooth grains, with low internal misorientations, with the high relative temperatures allowing for relief of internal strains.

The observed microstructures are therefore unlikely to be those present during the shock-wave, or indeed immediately prior to release. Considering literature *in situ* X-ray observations under shock, where the peak state shows significant grain refinement when compared to the initial sample [24, 26, 139], and the similarities between samples that have and have not undergone the transition, recrystallisation likely occur at all stages, not just the phase transition, or shock compression. And yet examining sample 100_{R-3} shows some key differences that may be due to the lack of a plastic shock wave, meaning the resulting microstructure is a function of the full loading history, rather than one step obscuring all those before it.

Damage induced recrystallisation adds additional complexity. The pressure reached during spall failure does not reach 2 GPa, see Chapter 5. At 425 K, the approximate temperature of the shock phase transition, γ is observed as low as 4 GPa [100]. The phase transition is likely occurring at lower pressures in the forward shock than hypothesised, so it may be possible that small regions of γ are nucleating at high temperatures and low stresses. Thus, with the highly directional stress fields around void and damage formation, grains may be transforming, rotating and reverting repeatedly, to create the fine grains observed. Indeed early measurements of the heat capacity indicated a phase transition at around 440 K, [286]. While this was revised by later work [101], it may be the case that such measurements encountered the γ phase at ambient pressures through inadvertently straining their crystals.

However, given the high temperatures, lower pressures and length scales, conventional plastic processes may be the dominant mechanism of damage. With the recrystallisation forming as part of damage, or immediately after, the more random texture may be formed. The mechanisms of damage cannot therefore be established through this work.

Together, these changes may explain the lack of observed orientation dependence in previous chapters. With similar mechanisms likely activating in all samples, including recrystallisation, the resulting microstructures shows significant convergence. Especially by the time spall occurs, the crystals are likely similar in size

and similarly oriented, resulting in spall occurring in very similar microstructures independent of the initial state.

6.4 References

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6.5 Appendix

6.5.1 Simulation Parameters

Further details of the Autodyn simulations used to design and demonstrate the recovery parameters are included here. The simulations were performed in 2D axial symmetry. Detail of the mesh is shown in Figure 6.19. In the tin and copper components, the mesh density is approximately 100 elements per mm, while in the pipe and sabot the element density is 50 elements per mm. In trapezoidal parts, namely the lateral rings, the density is specified along the middle of the component, with the narrower end exhibiting thinner elements, and the wider end thicker elements.

A simplified version of the sabot is used in the simulation, due to differences between manufacturing techniques and numerical modelling approach. A further

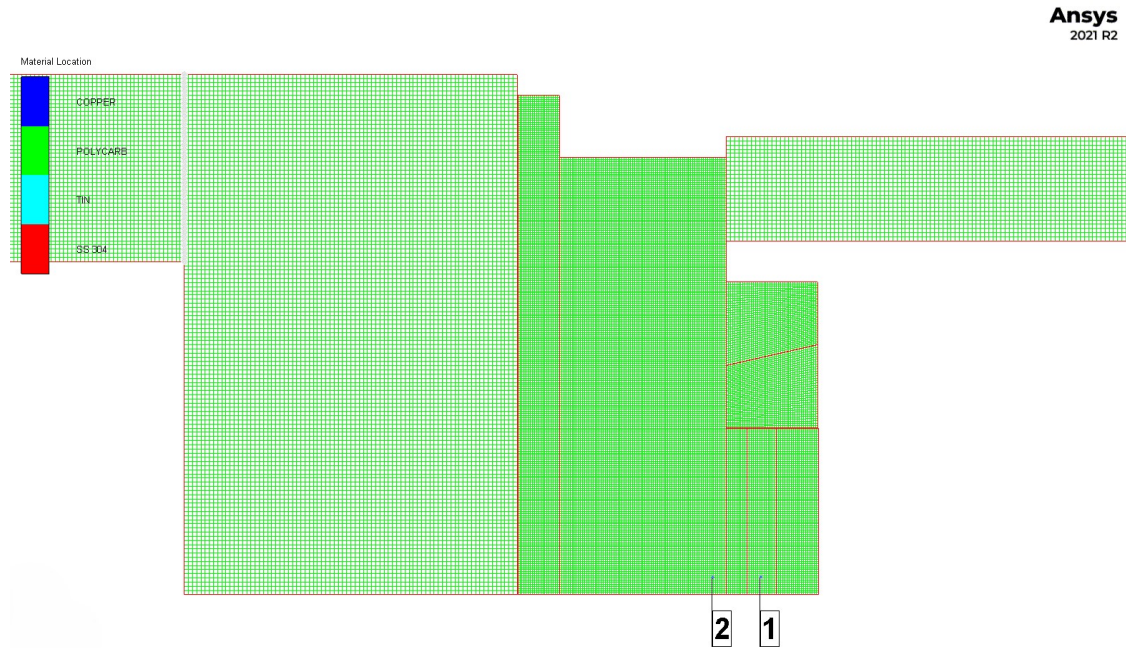


Figure 6.19: Detailed view of the mesh used in shock recovery design simulation. The location of gauge points is marked, with gauge one near the centre of the sample and gauge two near the front of the copper driver. The sabot is made from two rectangular sections, with joined nodes marked using grey diamonds.

limitation of this simulation technique is that no foam is included in the simulation. The properties of the foam used have not been studied under shock, meaning a valid model is not available. In addition, the simulation failure mechanisms of penetration, and the large deformations exhibited by the foam mean a stable and accurate representation of the recovery cannot be achieved.

In Table 6.2 the EOS parameters are listed, while in Tables 6.4 and 6.3 the strength model parameters are listed for materials that are modelled by a piecewise Johnson-Cook and a Steinberg-Guinan model respectively. Table 6.5 gives tensile failure conditions for the various materials, with the values for tin estimated from Chapter 5. This failure condition is used to erode elements, simulating spall. In addition, degenerate elements are eroded. For polycarbonate, an geometric strain condition is used for erosion, with the maximum permissible instantaneous strain set to 4.00.

Table 6.2: Table of material data for the shock model used in Autodyn simulations. Parameters are given the same name as in Autodyn. If a symbol is given in this work, it is listed. Parameters are zero unless listed.

	Parameter	Unit	Copper [287]	Tin [159]	Stainless steel [159]	Poly- carbonate [283]
ρ	Density	g cm^{-3}	8.90	7.287	7.90	1.20
	Grüneisen coefficient		2.00	2.27	1.93	0.61
C	Parameter C1	m ms^{-1}	3.958 001	2.59	4.57	1.933
S	Parameter S1		1.497	1.49	1.49	2.65
	Reference Temperature	Kelvin	300	300	300	300
	Specific Heat	$\text{J g}^{-1} \text{K}$	0	0.222	0.423	0

Table 6.3: Table of material data for the Steinberg-Guinan strength model used in Autodyn simulations. Parameters are given the same name as in Autodyn. Parameters are default (zero, one, or 1×10^{20} - effectively infinite) unless listed.

Parameter	Unit	Tin [159]	Stainless Steel [159]
Shear Modulus	GPa	17.9	77.0
Yield Stress	GPa	0.16	0.34
Maximum Yield Stress	GPa	0.22	2.5
Hardening Constant		2000	43
Hardening Exponent		0.06	0.35
Derivative dG/dP		1.55	1.74
Derivative dG/dT	GPa K^{-1}	-0.037 950	-0.035 040
Derivative dY/dP		0.013 860	0.007 684
Melting Temperature	K	656.599 976	2380

Table 6.4: Table of material data for the piecewise Johnson-Cook strength model used in Autodyn simulations. Parameters are given the same name as in Autodyn. Parameters are zero unless listed.

Parameter	Unit	Copper [287]	Polycarbonate [283]
Shear Modulus	GPa	46.399 998	1.00
Yield Stress (zero plastic strain)	GPa	0.12	0.0806
Eff. Plastic Strain #1		0.3	0.1
Eff. Plastic Strain #2		1×10^{20}	0.5
Eff. Plastic Strain #3		0	0.6
Eff. Plastic Strain #4		0	0.7
Yield Stress #1	GPa	0.45	0.088
Yield Stress #2	GPa	0.45	0.1425
Yield Stress #3	GPa	0	0.168
Yield Stress #4	GPa	0	0.187

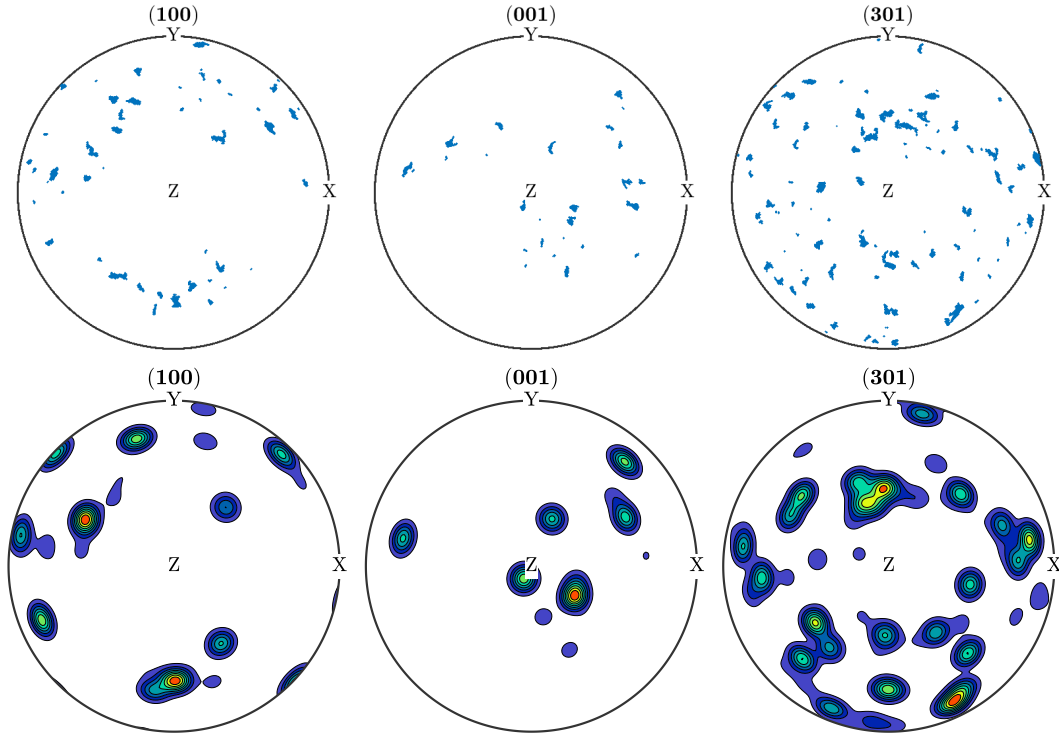
Table 6.5: Table of material data for the Hydro (Pmin) failure model used in Autodyn simulations. Parameters are given the same name as in Autodyn. Approximate values of the spall strength of the materials are taken, with elements eroded at this condition to represent spall failure. For all, reheal is selected, while crack softening and stochastic failure are not.

Parameter	Unit	Copper	Tin	Stainless Steel
Hydro Tensile Limit	GPa	−1.0	−1.5	−2.0

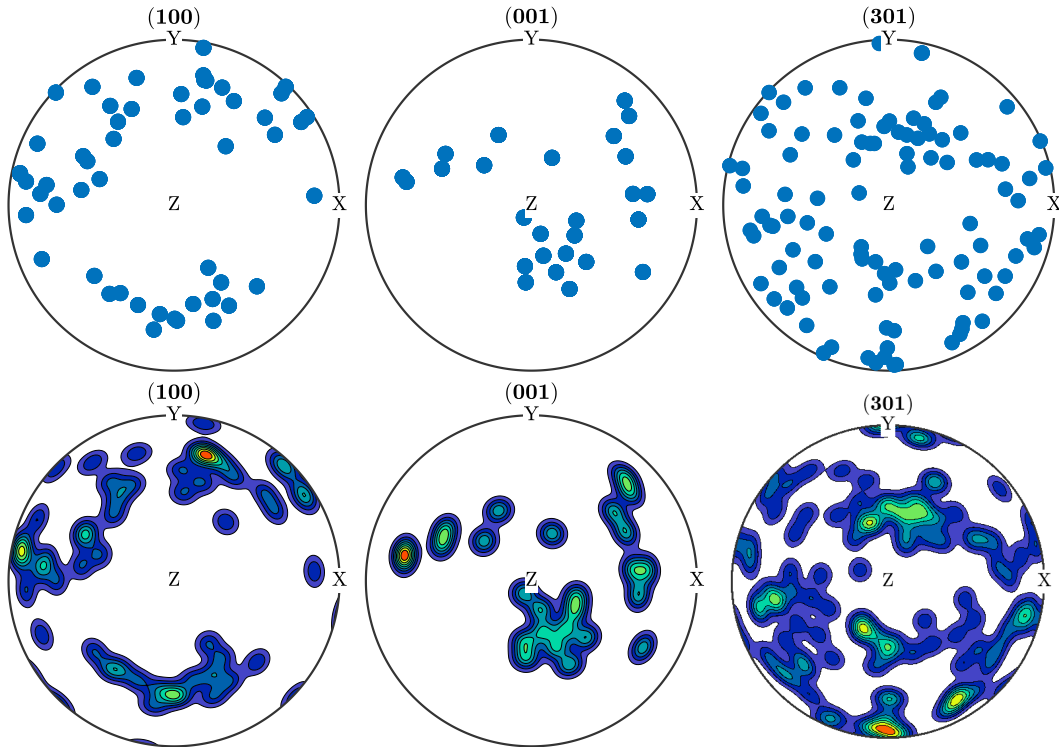
6.5.2 Additional EBSD Plots

A full set of pole plots, grain-size distributions and grain-boundary misorientations are presented here. Chapter 7 can be found on page 198.

6.5.2.1 Pole Figures

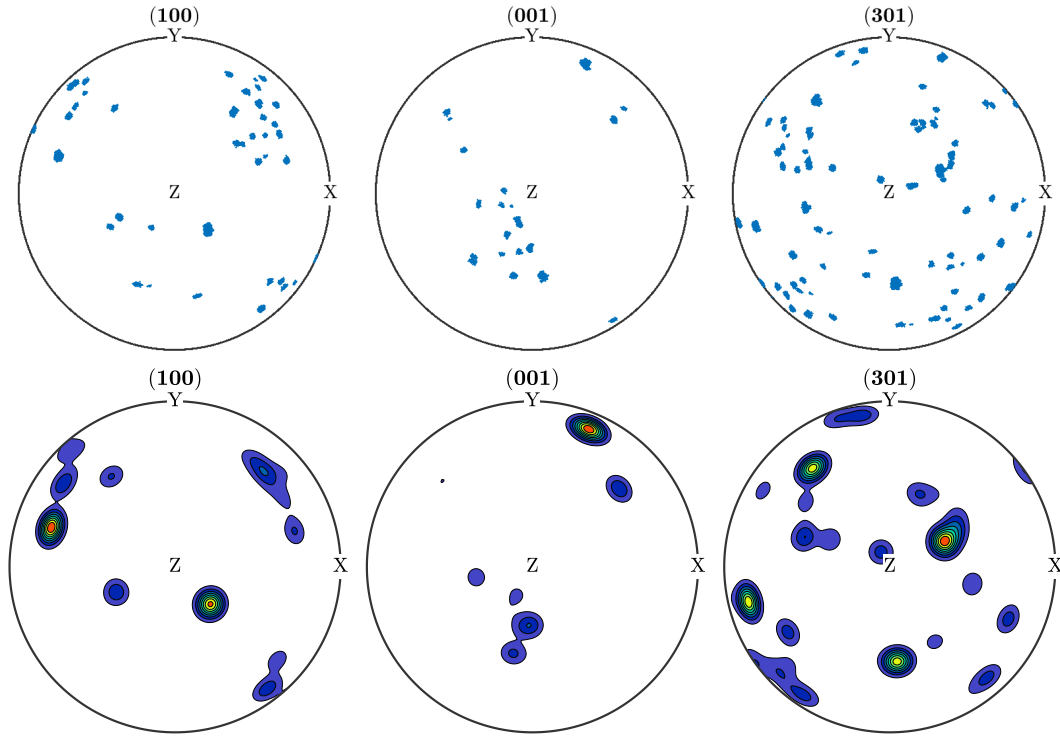


(a) Pole figures for the transverse scan of sample AR2₁ by point.

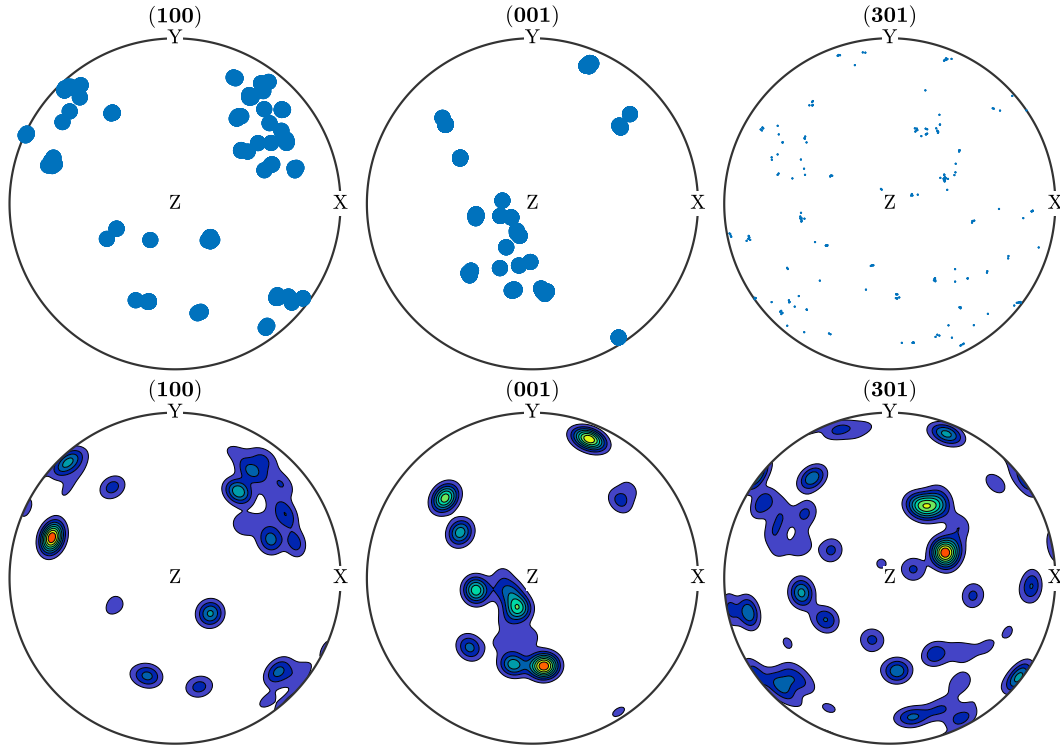


(b) Pole figures for the transverse scan of sample AR2₁ by grain.

Figure 6.20: Pole figures for the transverse scan of sample AR2₁.

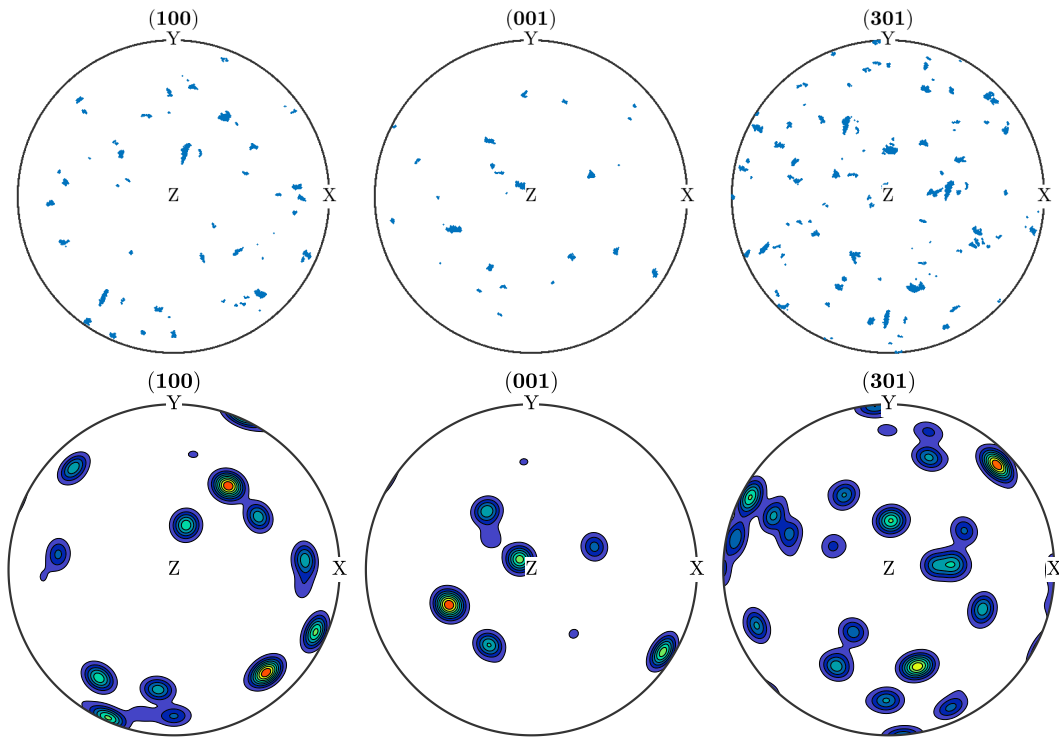


(a) Pole figures for the longitudinal scan of sample AR2₁ by point.

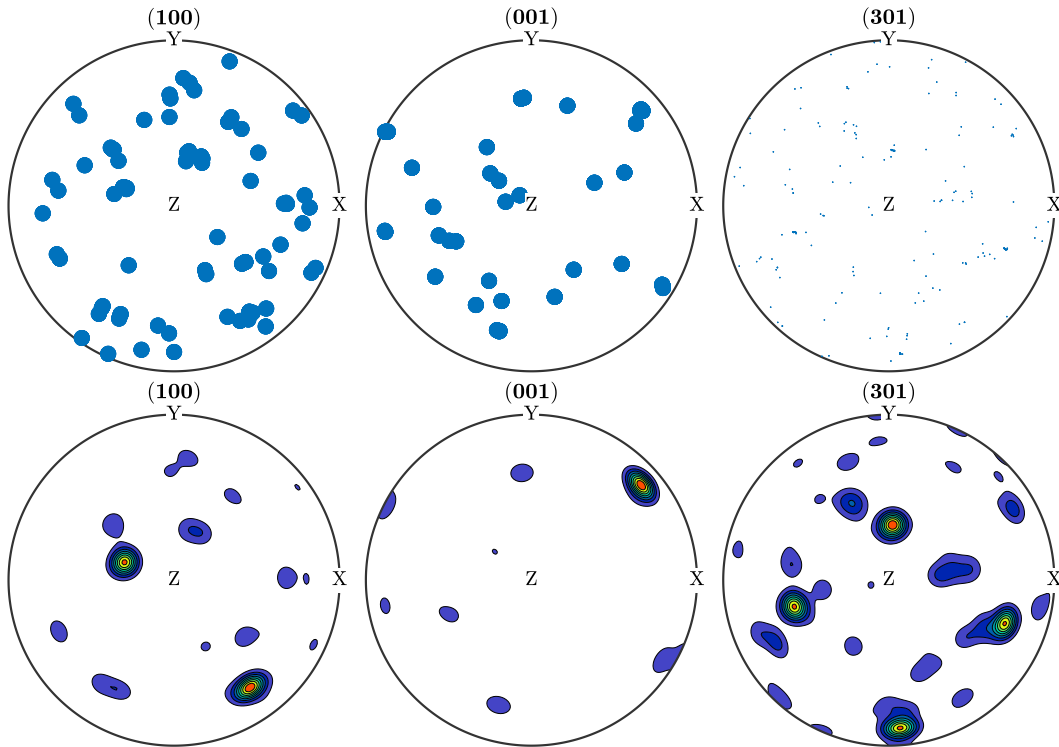


(b) Pole figures for the longitudinal scan of sample AR2₁ by grain.

Figure 6.21: Pole figures for the longitudinal scan of sample AR2₁.

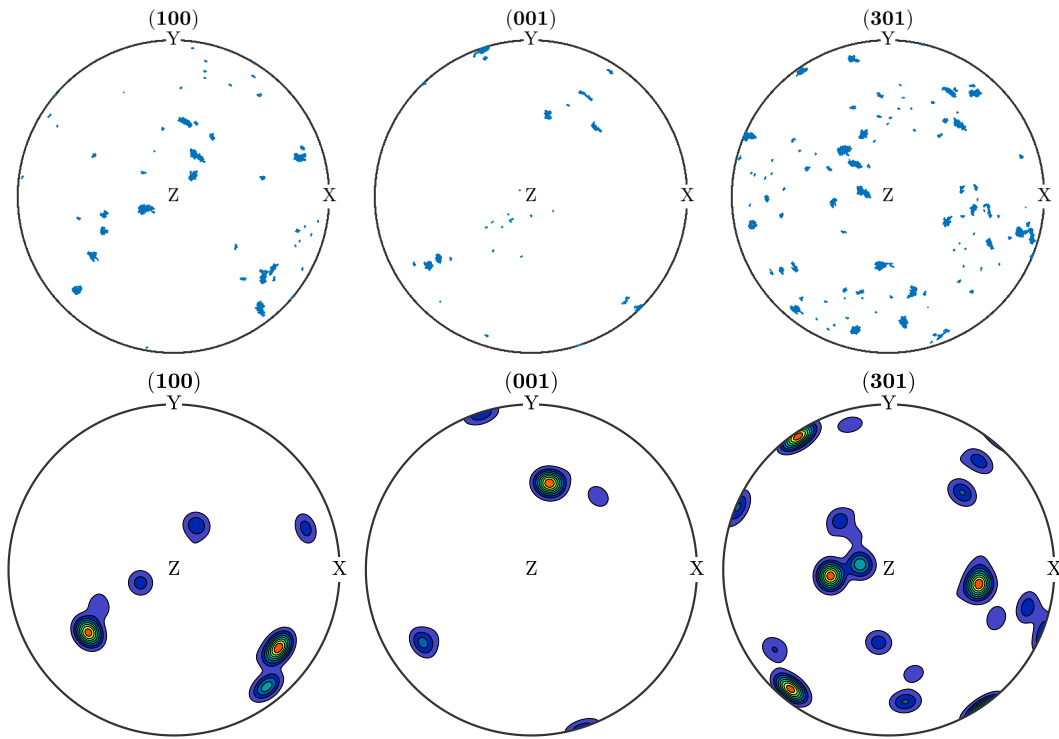


(a) Pole figures for the transverse scan of sample AR2₂ by point.

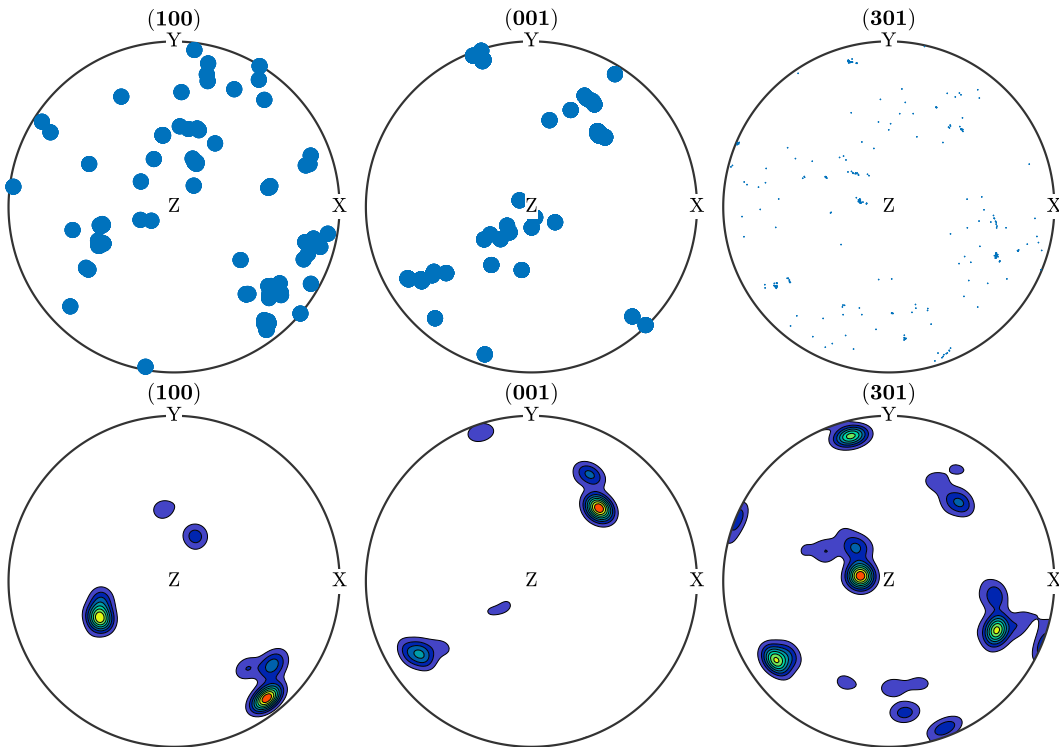


(b) Pole figures for the transverse scan of sample AR2₂ by grain.

Figure 6.22: Pole figures for the transverse scan of sample AR2₂.

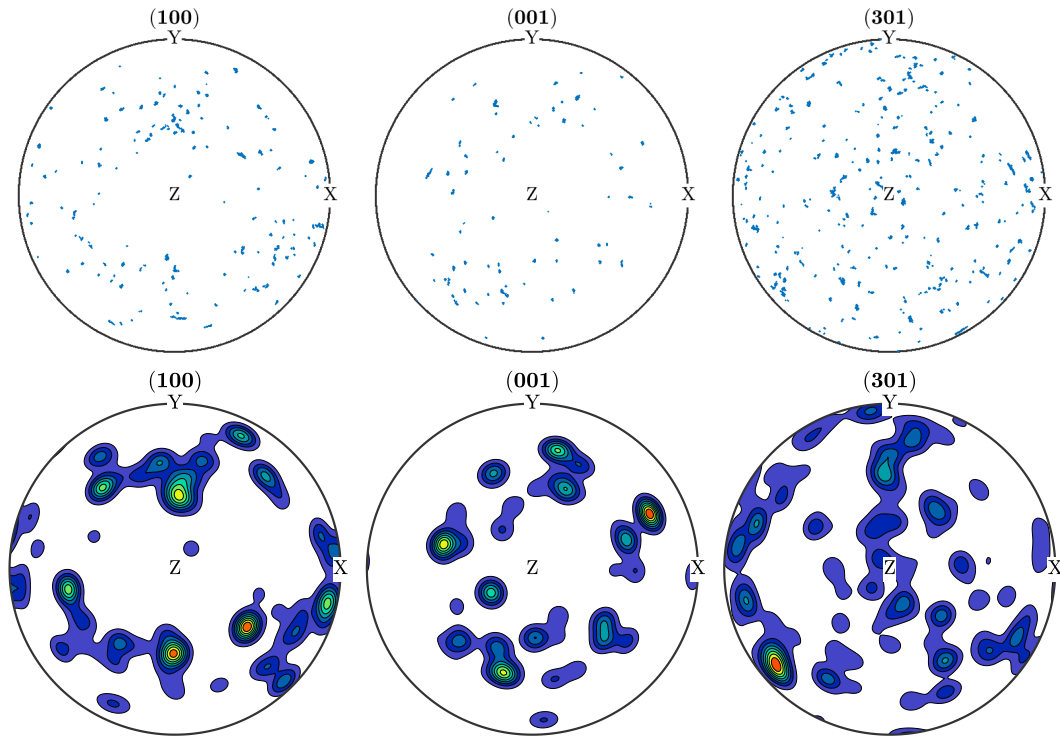


(a) Pole figures for the longitudinal scan of sample AR2₂ by point.

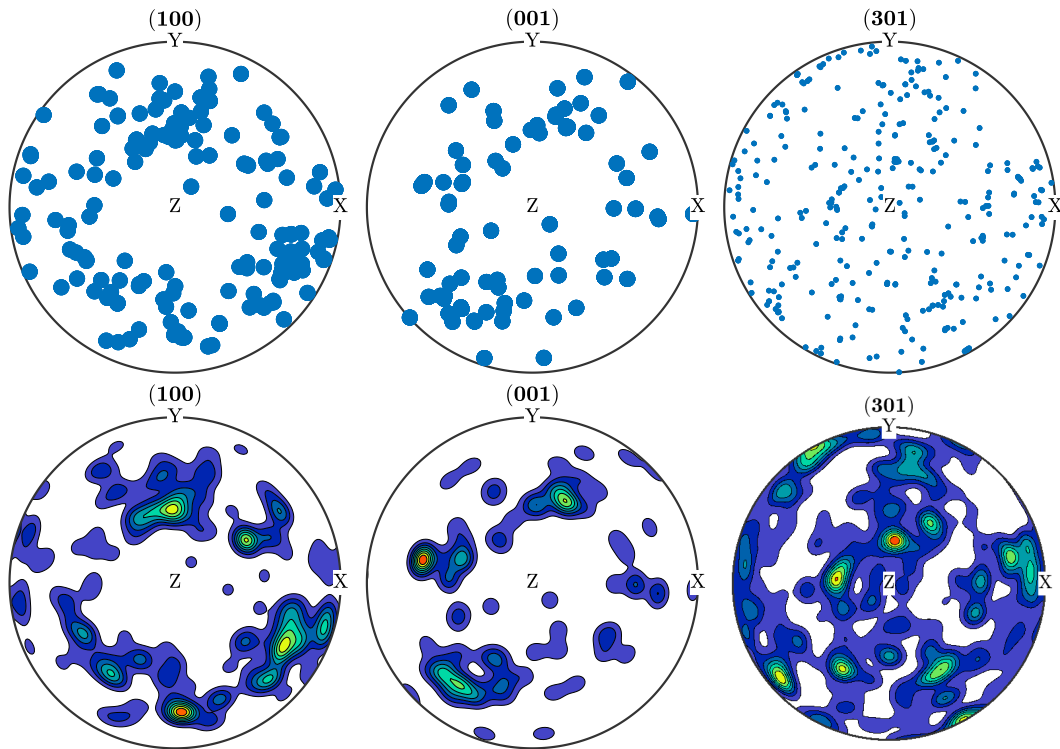


(b) Pole figures for the longitudinal scan of sample AR2₂ by grain.

Figure 6.23: Pole figures for the longitudinal scan of sample AR2₂.

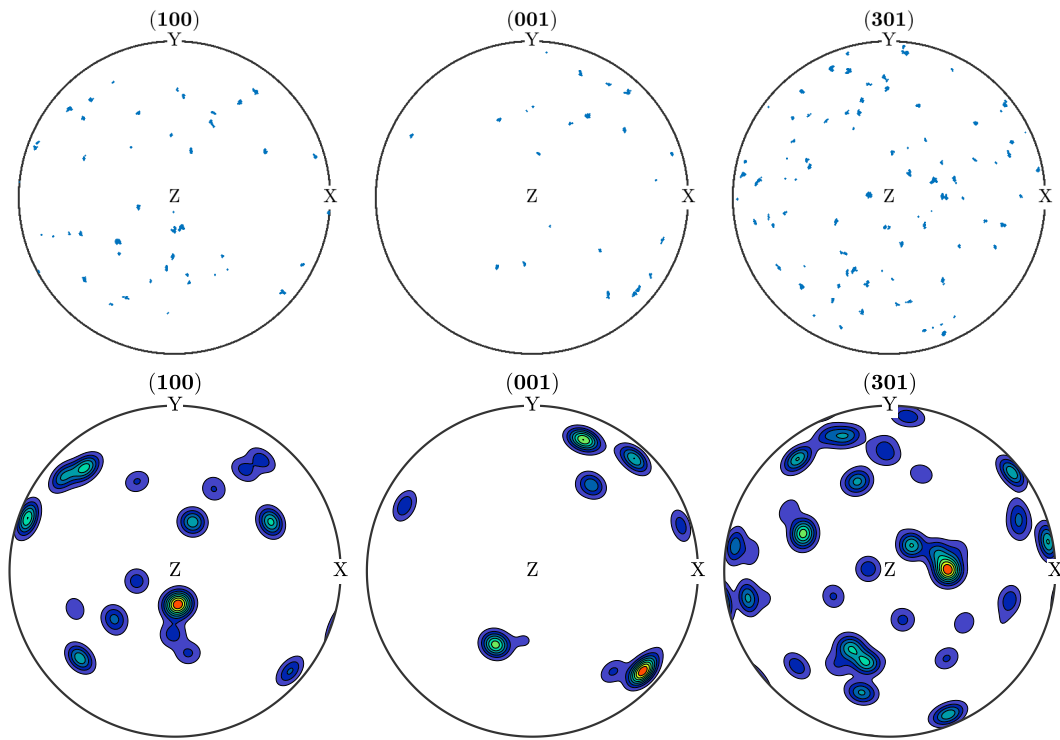


(a) Pole figures for the transverse scan of sample AR1₁ by point.

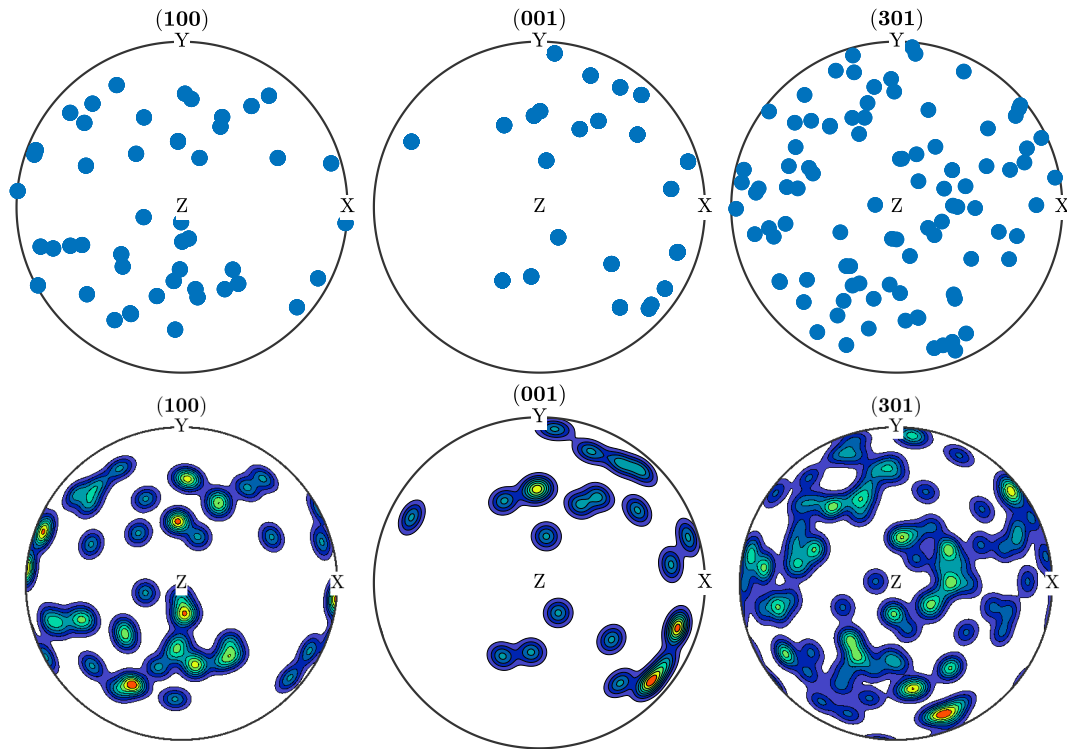


(b) Pole figures for the transverse scan of sample AR1₁ by grain.

Figure 6.24: Pole figures for the transverse scan of sample AR1₁.

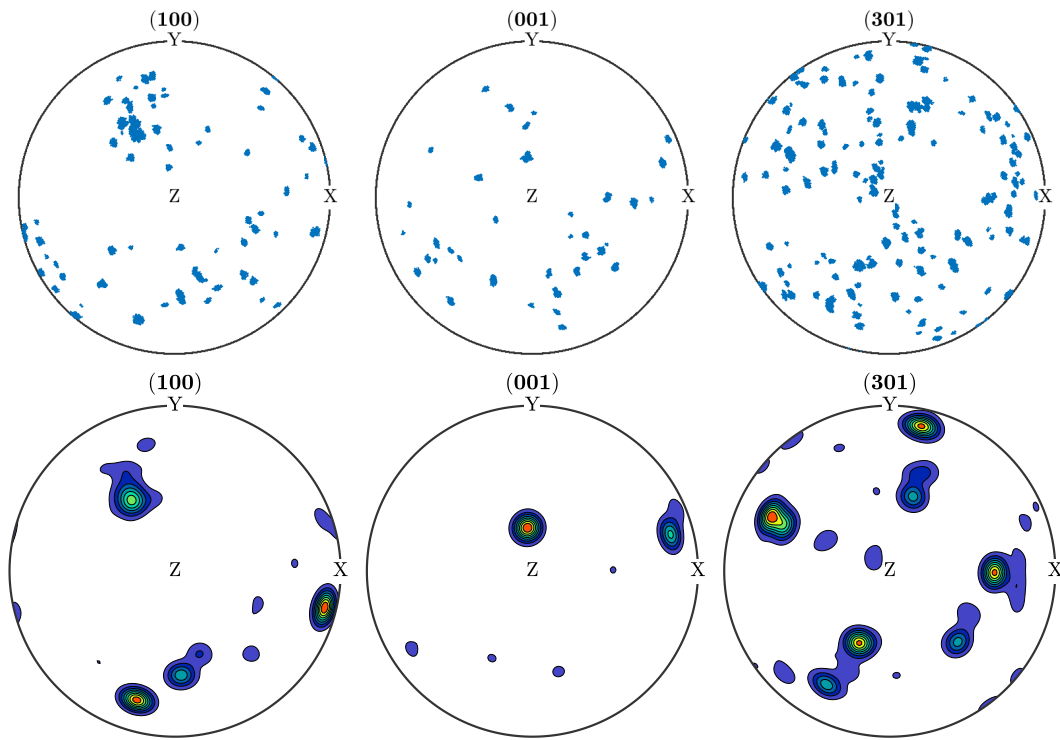


(a) Pole figures for the longitudinal scan of sample AR1₁ by point.

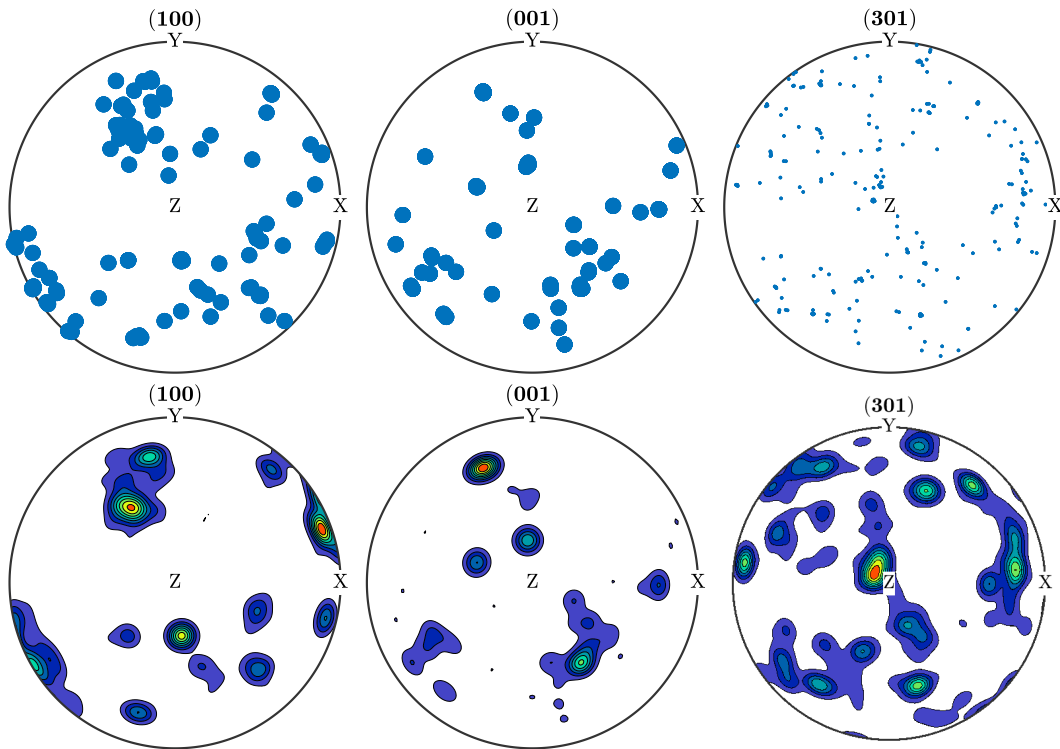


(b) Pole figures for the longitudinal scan of sample AR1₁ by grain.

Figure 6.25: Pole figures for the longitudinal scan of sample AR1₁.

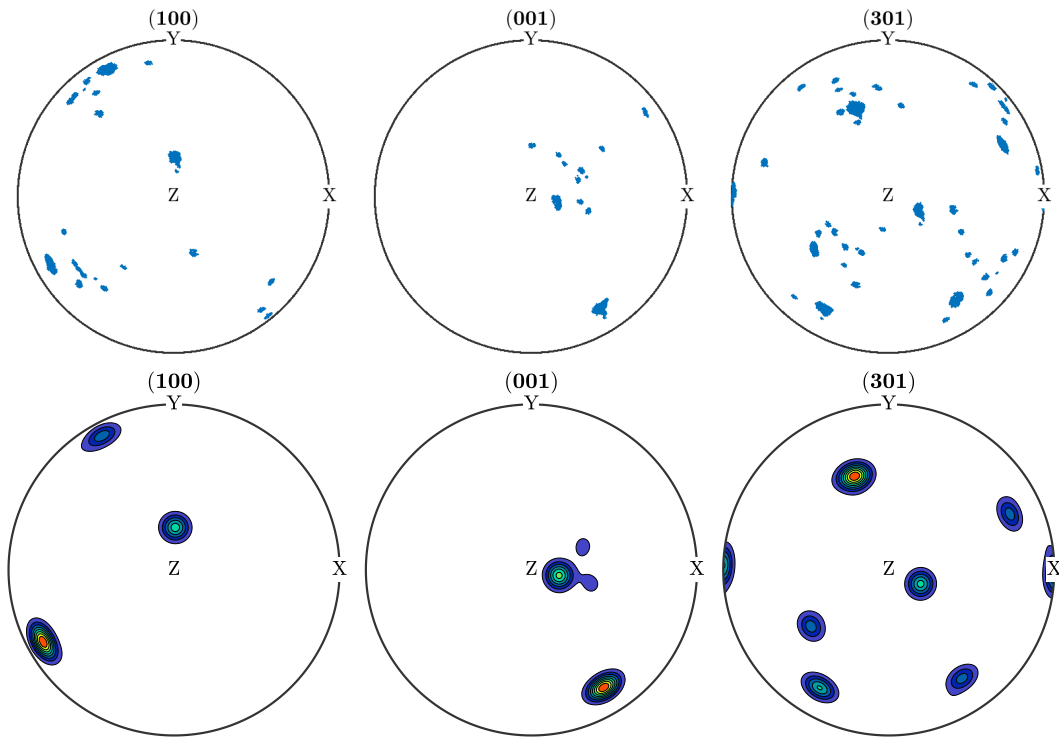


(a) Pole figures for the transverse scan of sample AR1₂ by point.

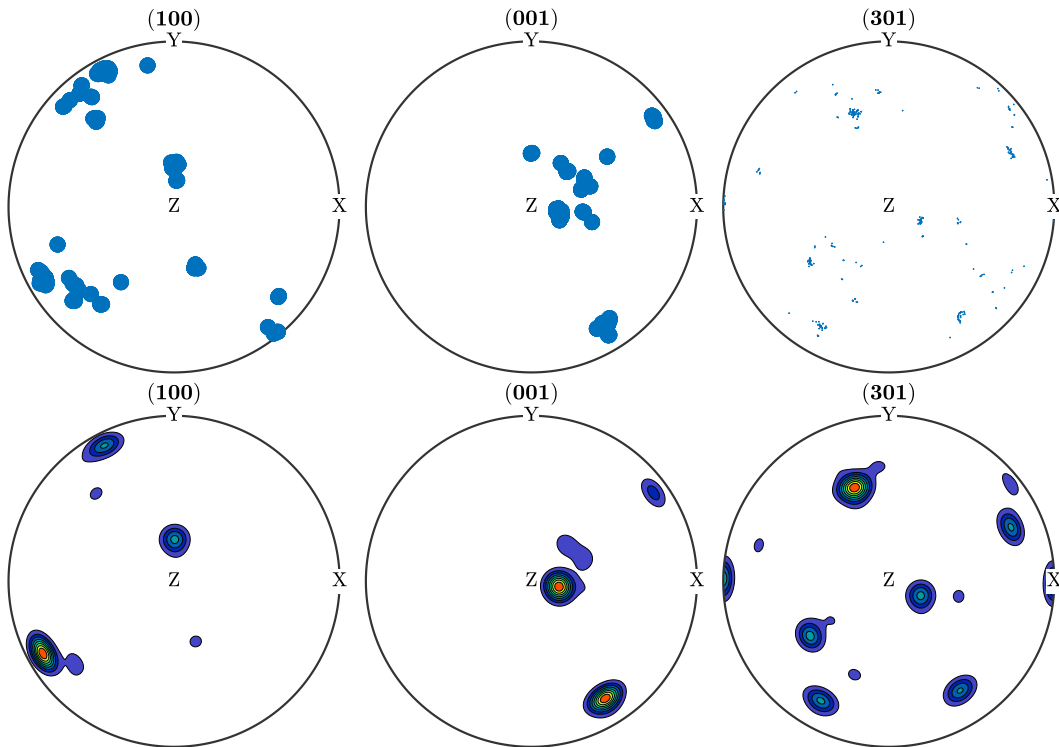


(b) Pole figures for the transverse scan of sample AR1₂ by grain.

Figure 6.26: Pole figures for the transverse scan of sample AR1₂.

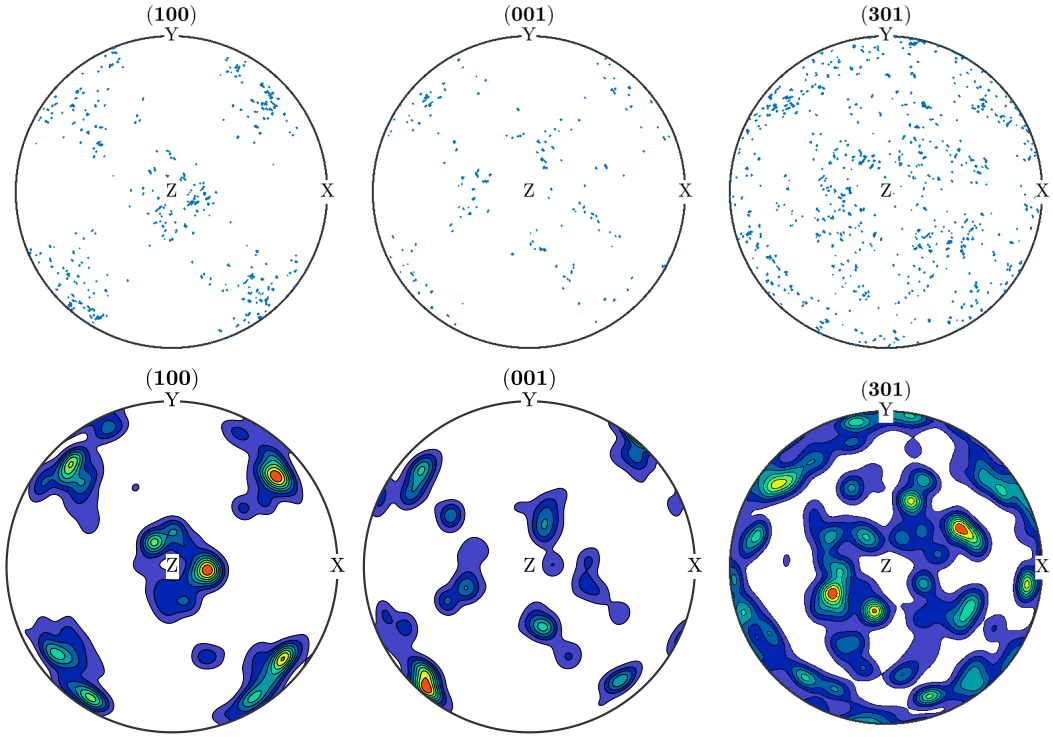


(a) Pole figures for the longitudinal scan of sample AR1₂ by point.

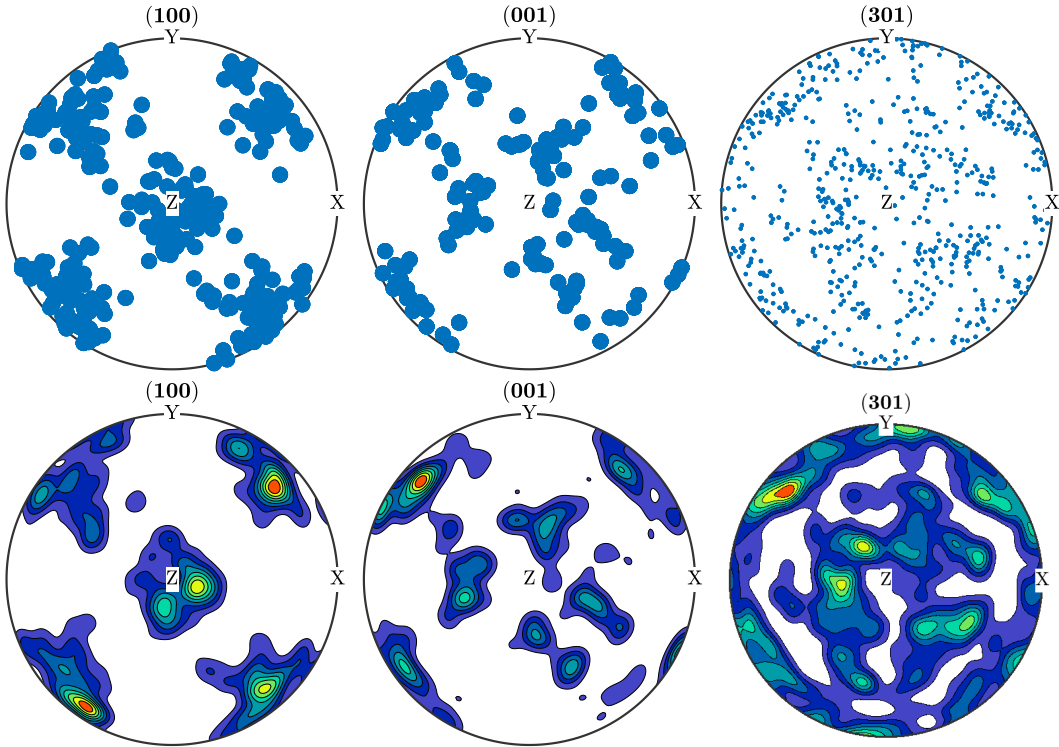


(b) Pole figures for the longitudinal scan of sample AR1₂ by grain.

Figure 6.27: Pole figures for the longitudinal scan of sample AR1₂.

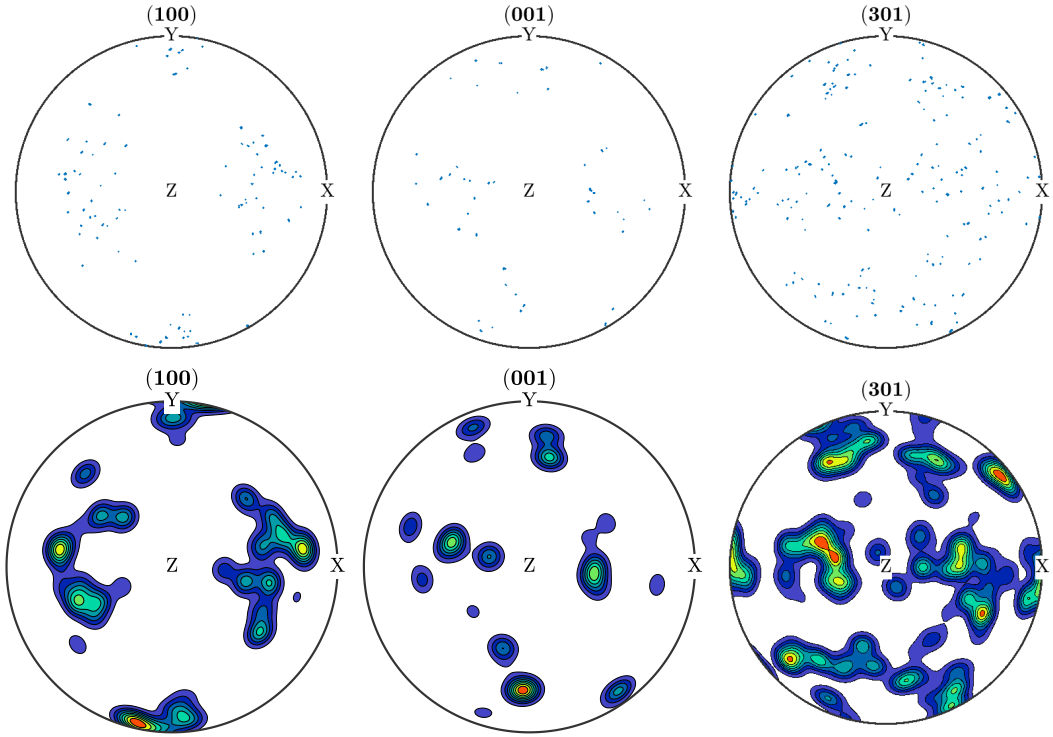


(a) Pole figures for the transverse scan of sample 001_{R-1} by point.

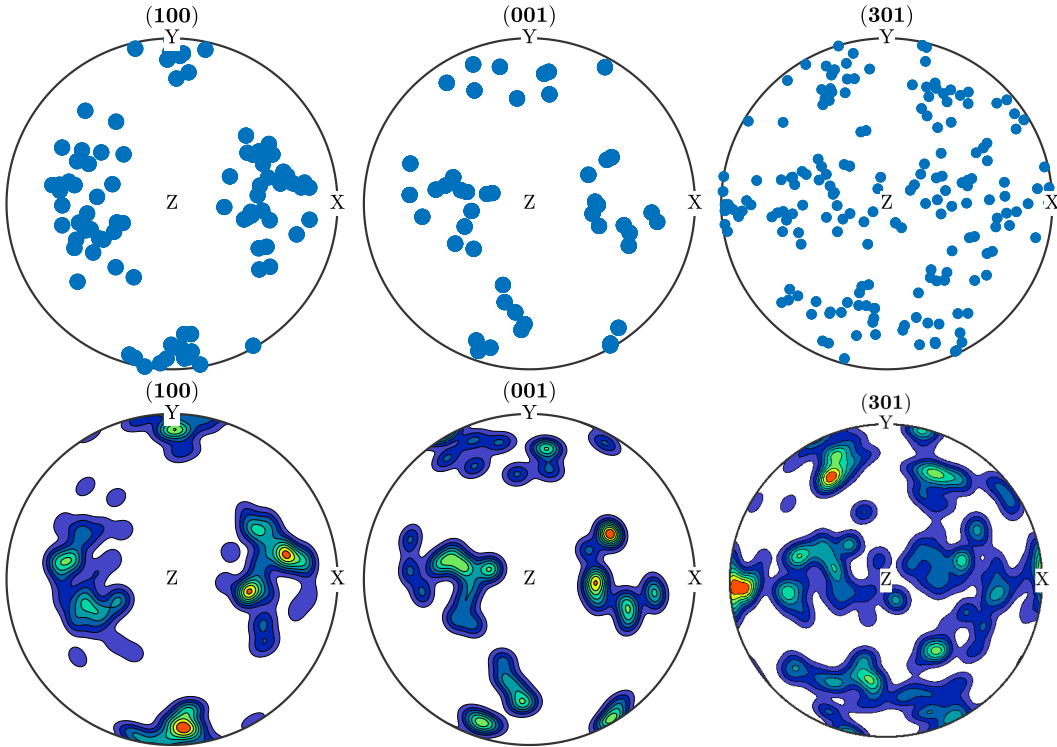


(b) Pole figures for the transverse scan of sample 001_{R-1} by grain.

Figure 6.28: Pole figures for the transverse scan of sample 001_{R-1}.

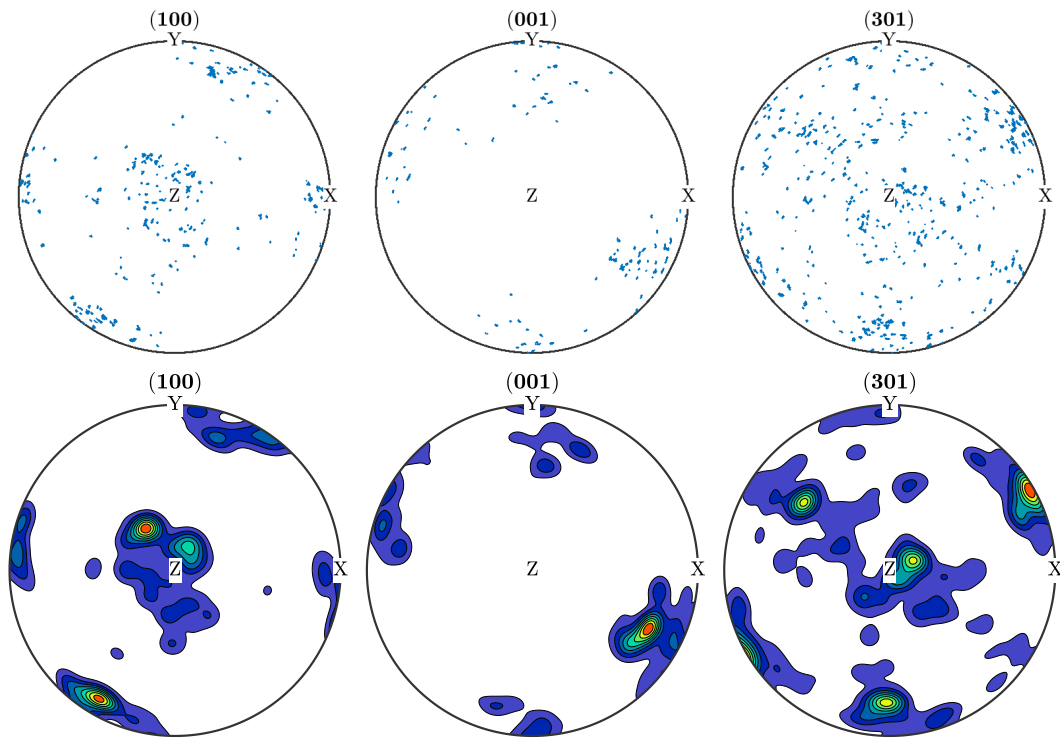


(a) Pole figures for the longitudinal scan of sample 001_{R-1} by point.

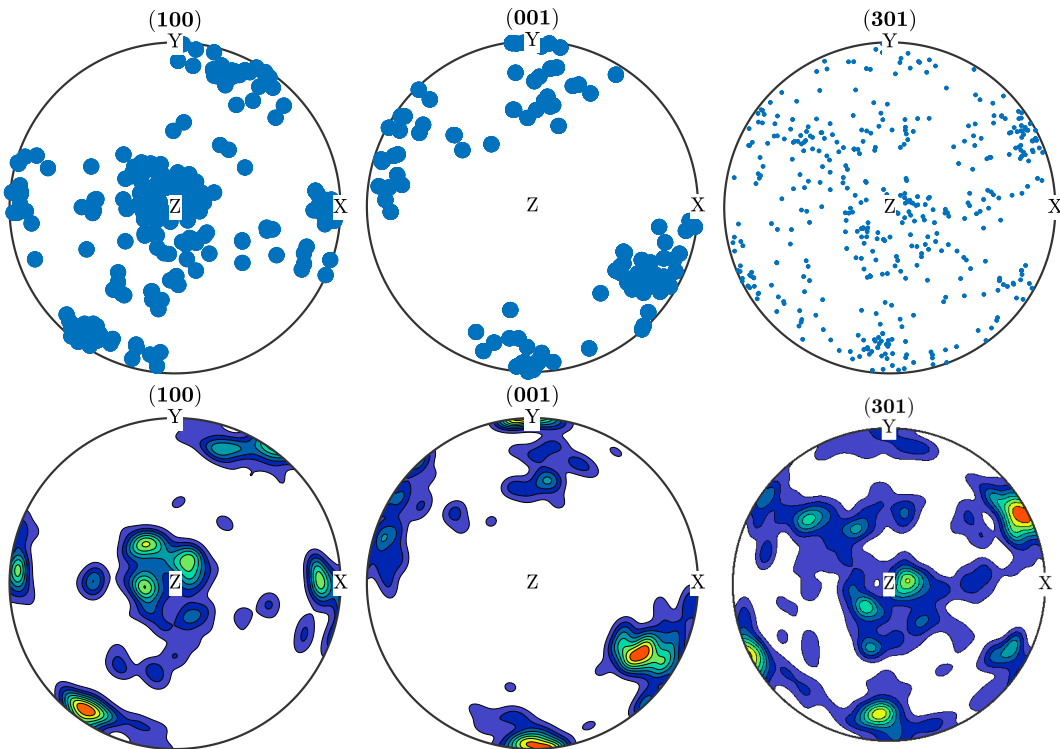


(b) Pole figures for the longitudinal scan of sample 001_{R-1} by grain.

Figure 6.29: Pole figures for the longitudinal scan of sample 001_{R-1}.

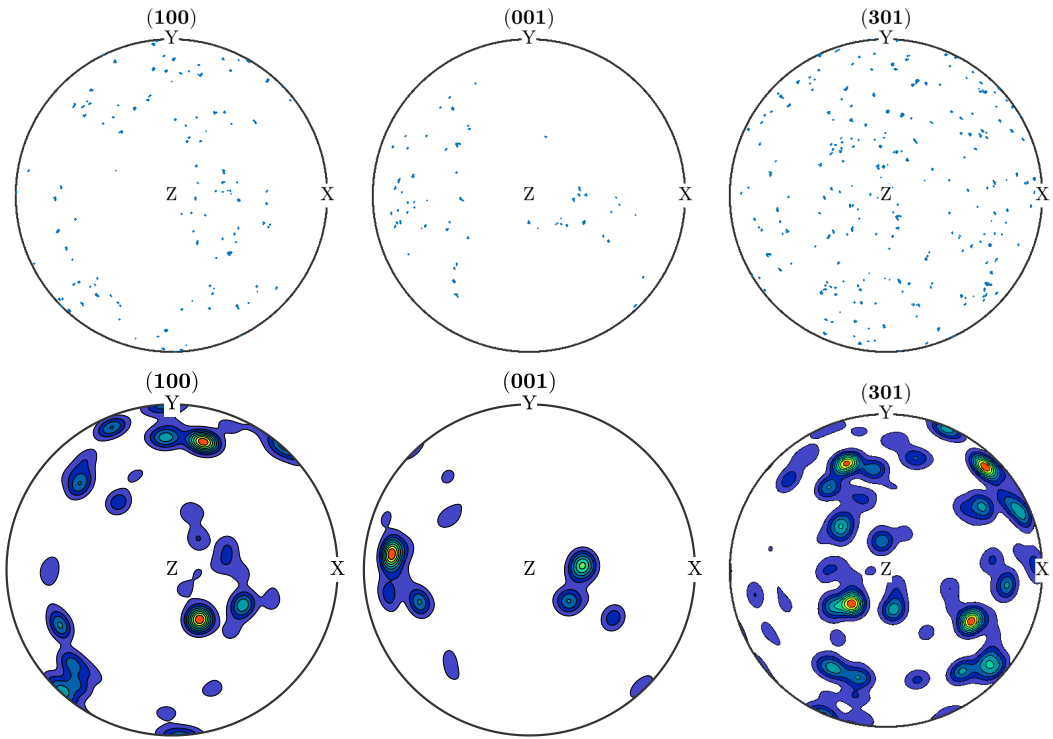


(a) Pole figures for the transverse scan of sample 001_{R-2} by point.

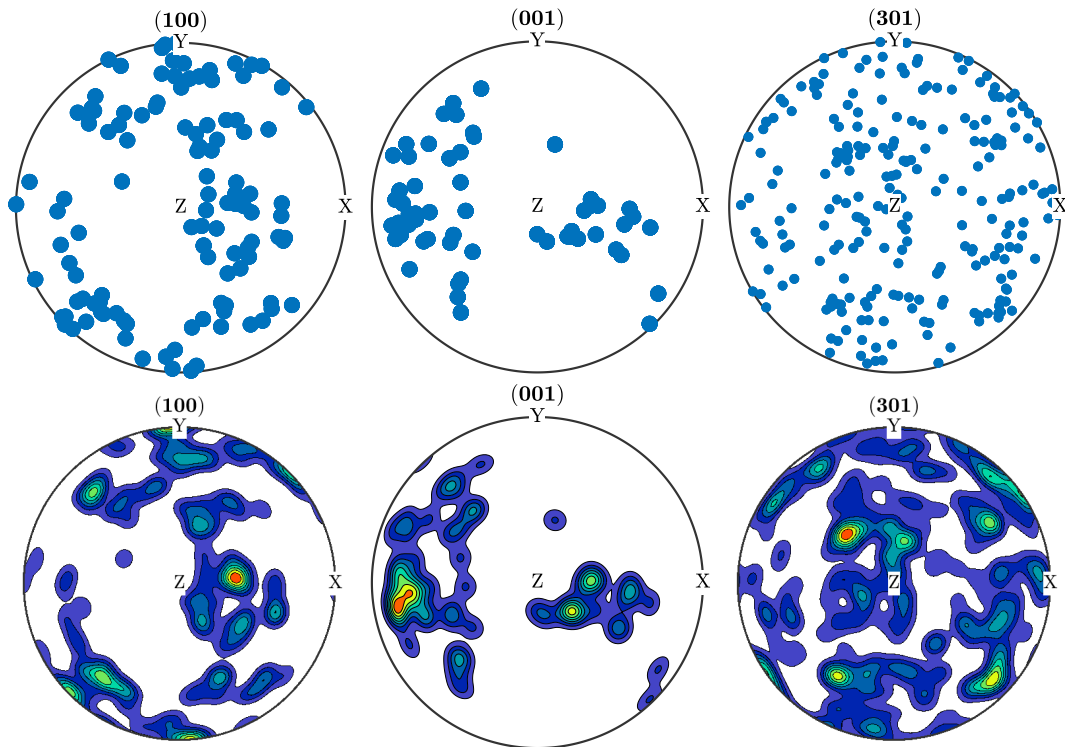


(b) Pole figures for the transverse scan of sample 001_{R-2} by grain.

Figure 6.30: Pole figures for the transverse scan of sample 001_{R-2}.

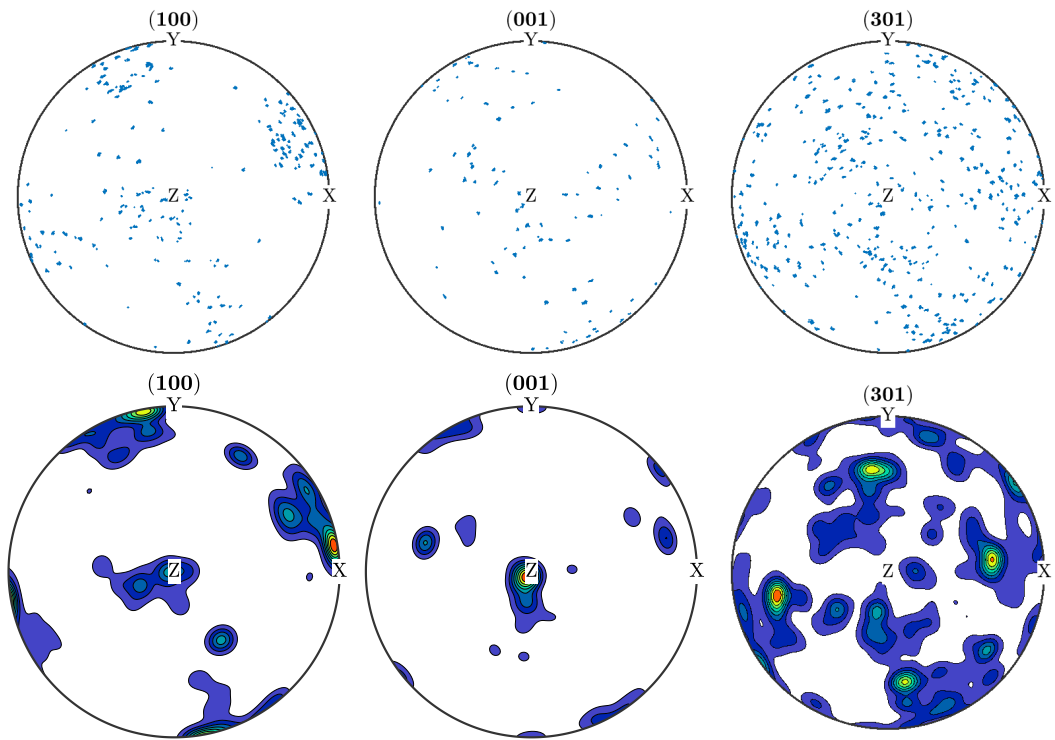


(a) Pole figures for the longitudinal scan of sample 001_{R-2} by point.

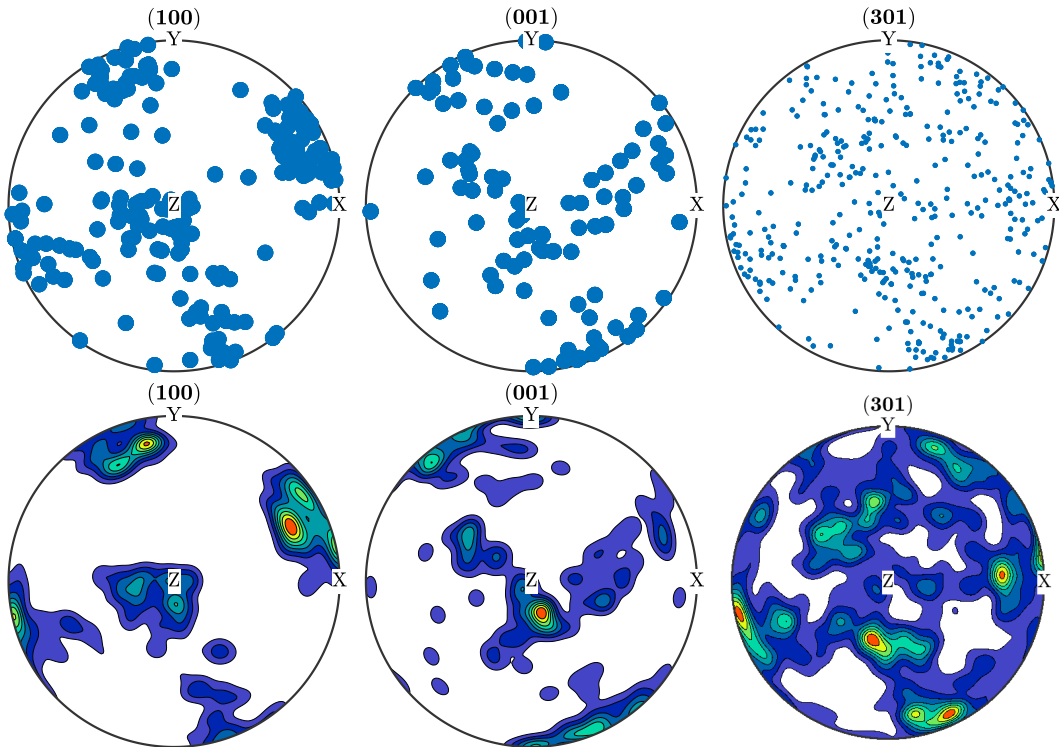


(b) Pole figures for the longitudinal scan of sample 001_{R-2} by grain.

Figure 6.31: Pole figures for the longitudinal scan of sample 001_{R-2}.

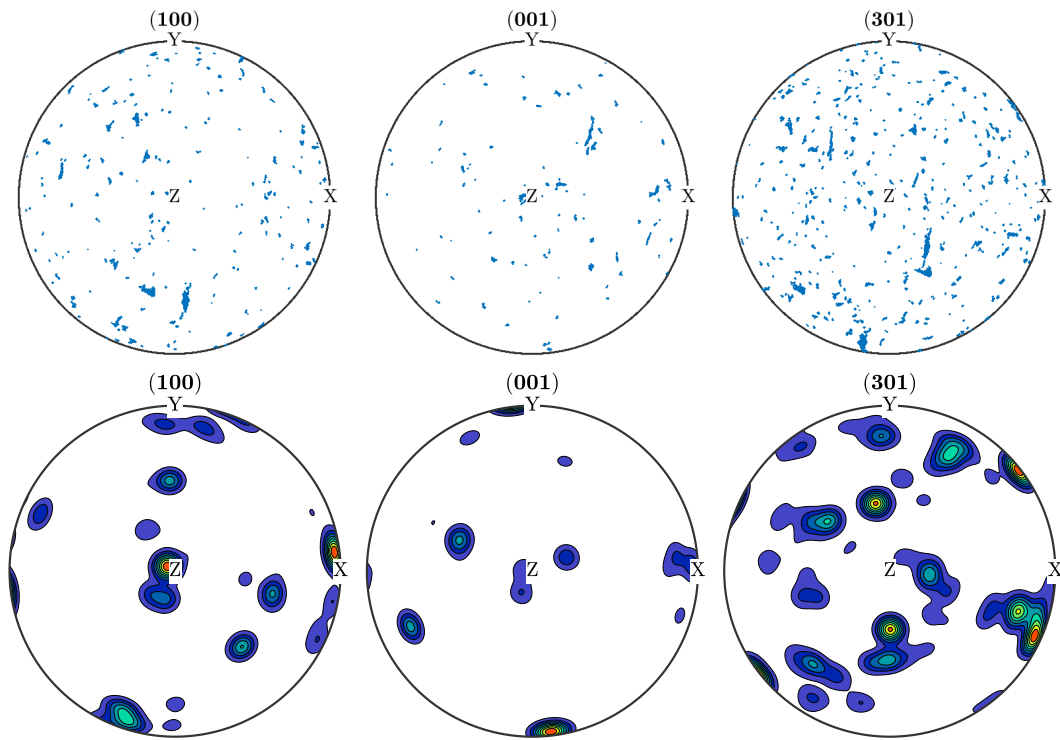


(a) Pole figures for the transverse scan of sample 001_{R-3} by point.

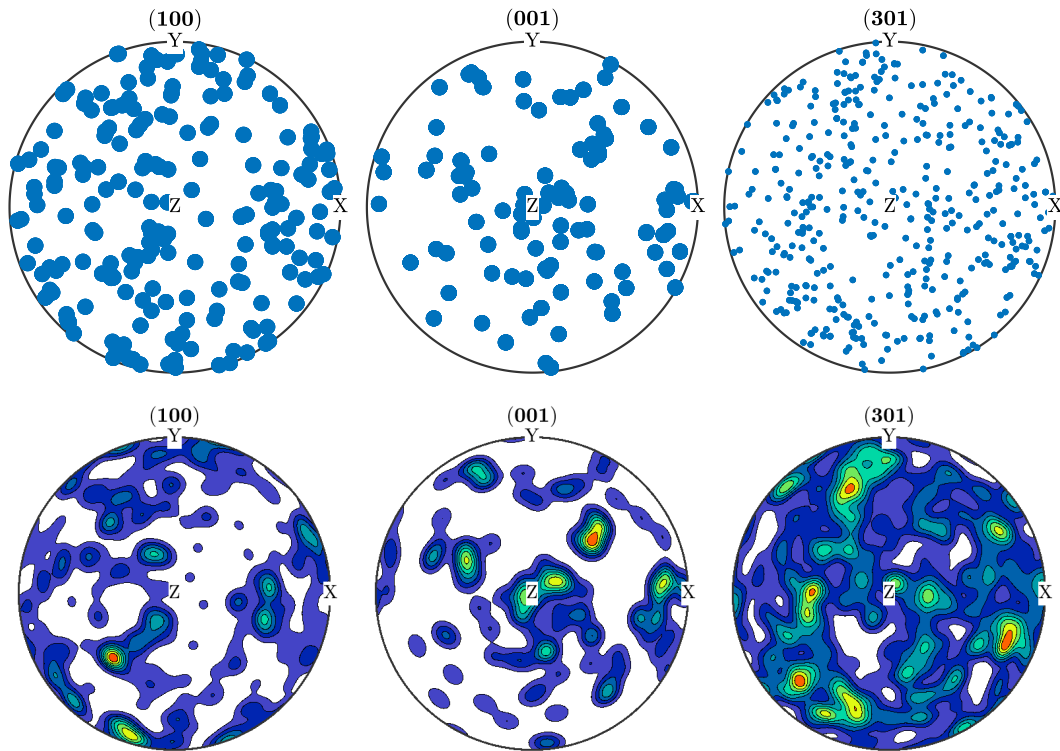


(b) Pole figures for the transverse scan of sample 001_{R-3} by grain.

Figure 6.32: Pole figures for the transverse scan of sample 001_{R-3}.

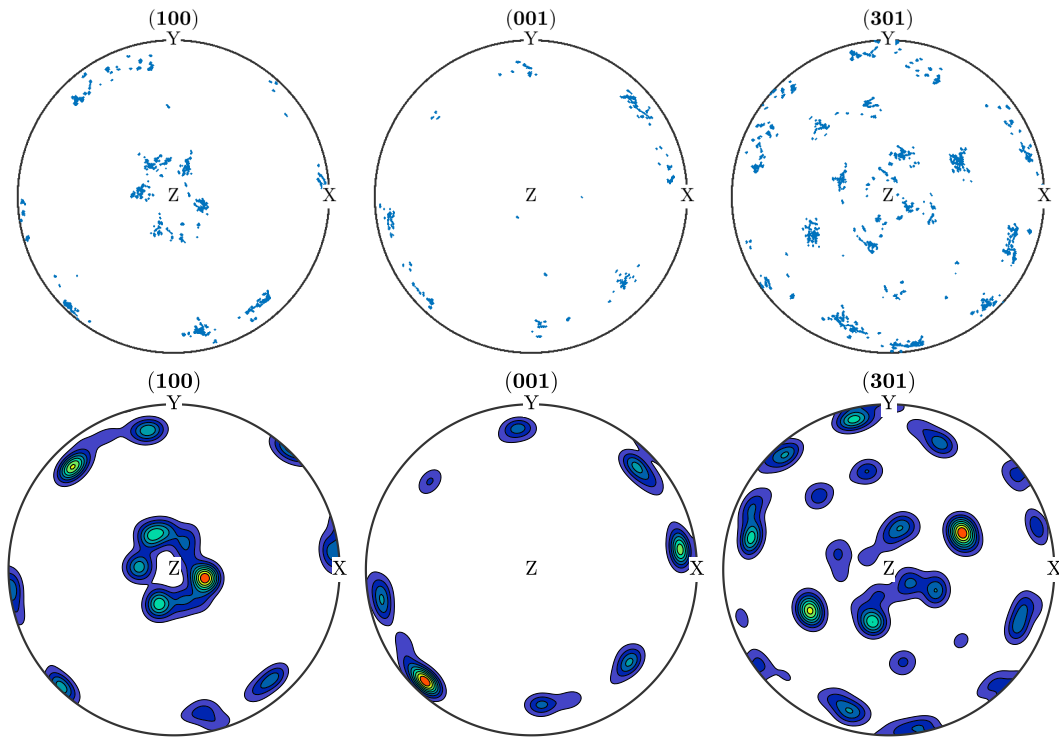


(a) Pole figures for the longitudinal scan of sample 001_{R-3} by point.

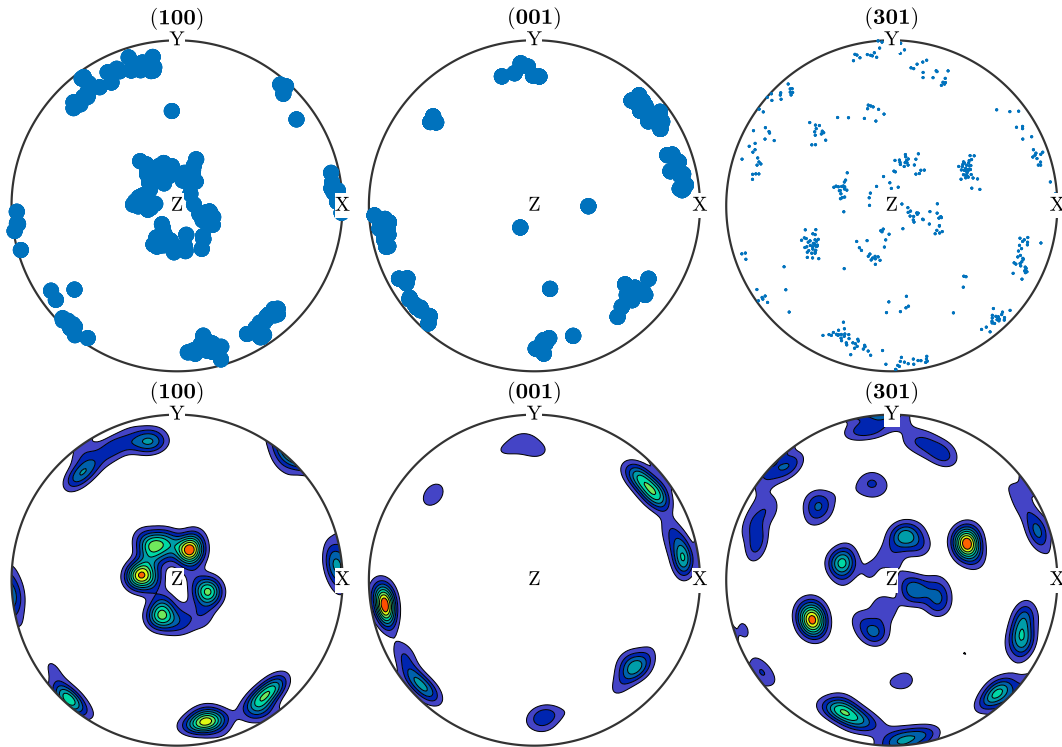


(b) Pole figures for the longitudinal scan of sample 001_{R-3} by grain.

Figure 6.33: Pole figures for the longitudinal scan of sample 001_{R-3}.

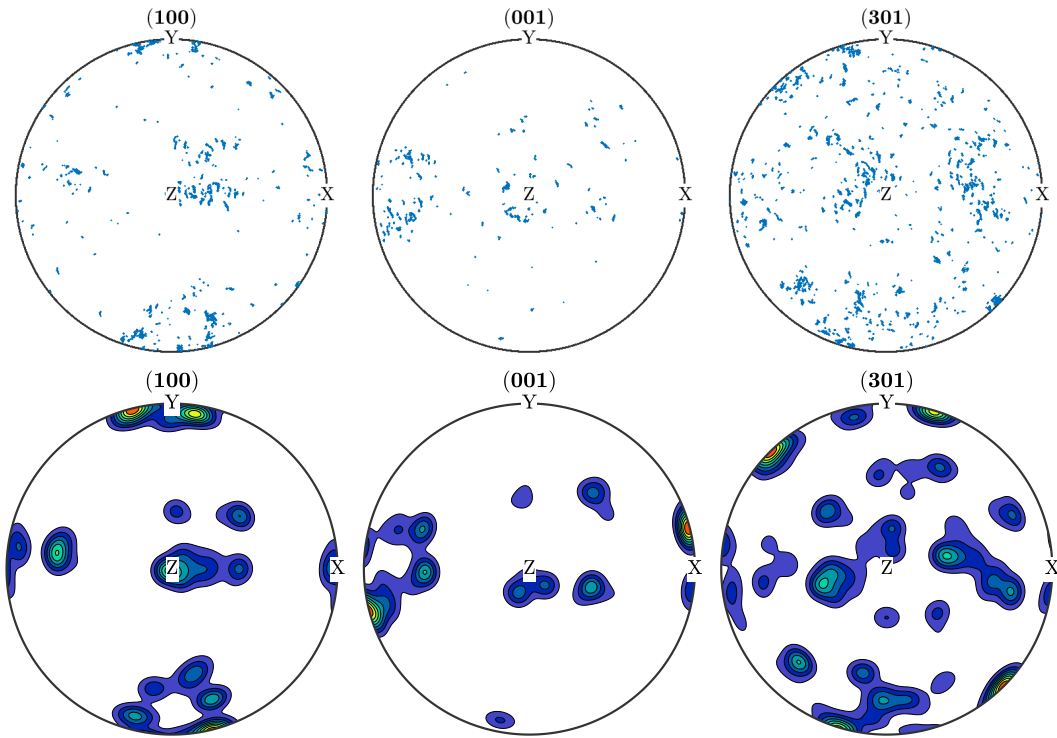


(a) Pole figures for the transverse scan of sample 100_{R-1} by point.

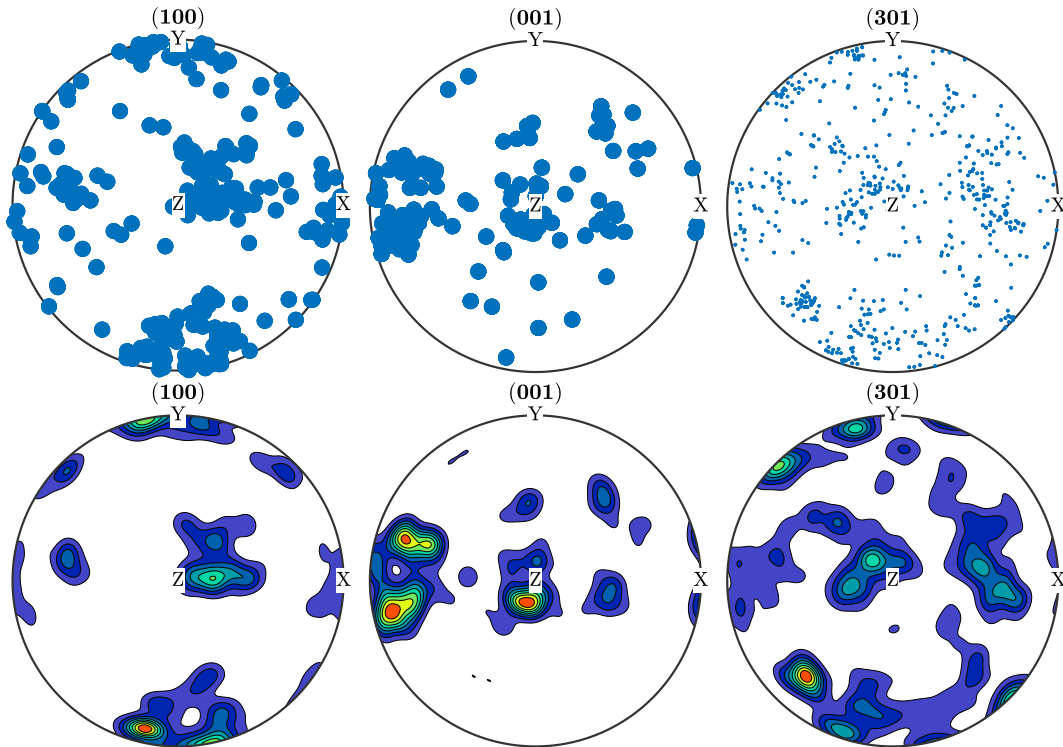


(b) Pole figures for the transverse scan of sample 100_{R-1} by grain.

Figure 6.34: Pole figures for the transverse scan of sample 100_{R-1}.

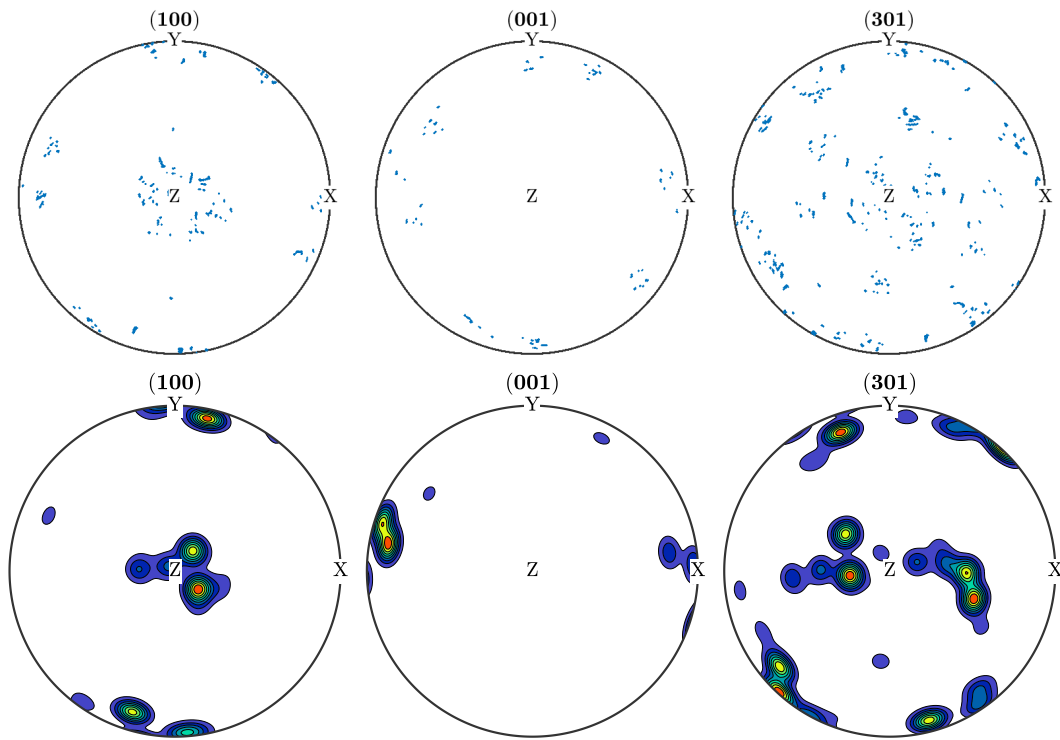


(a) Pole figures for the longitudinal scan of sample 100_{R-1} by point.

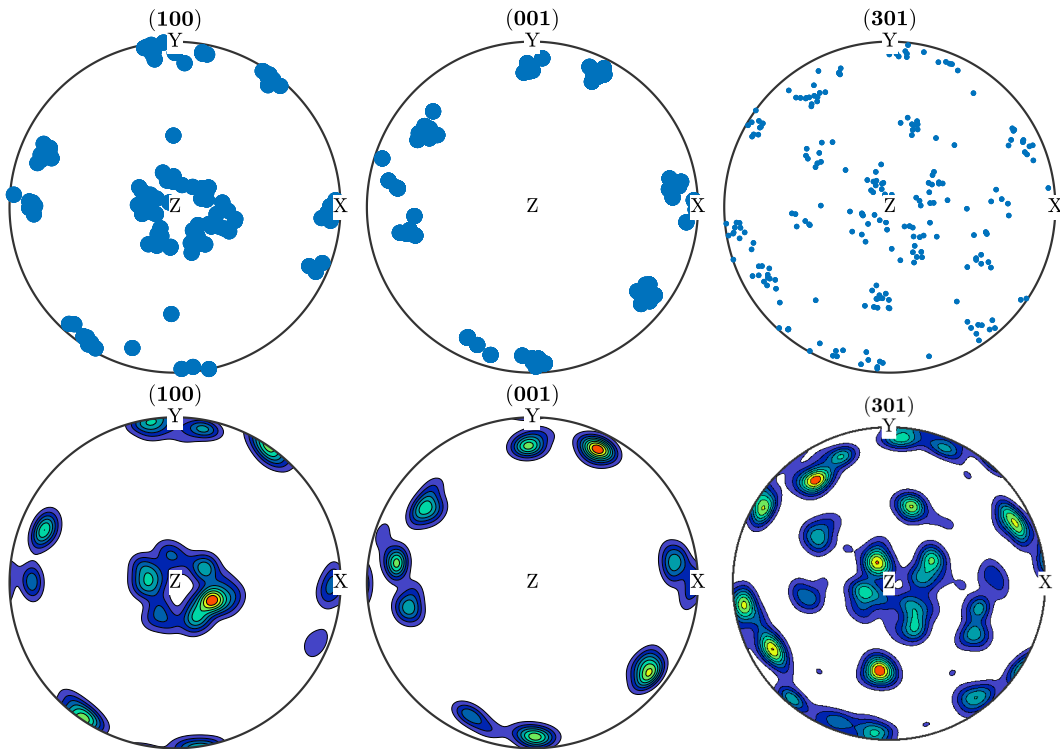


(b) Pole figures for the longitudinal scan of sample 100_{R-1} by grain.

Figure 6.35: Pole figures for the longitudinal scan of sample 100_{R-1}.

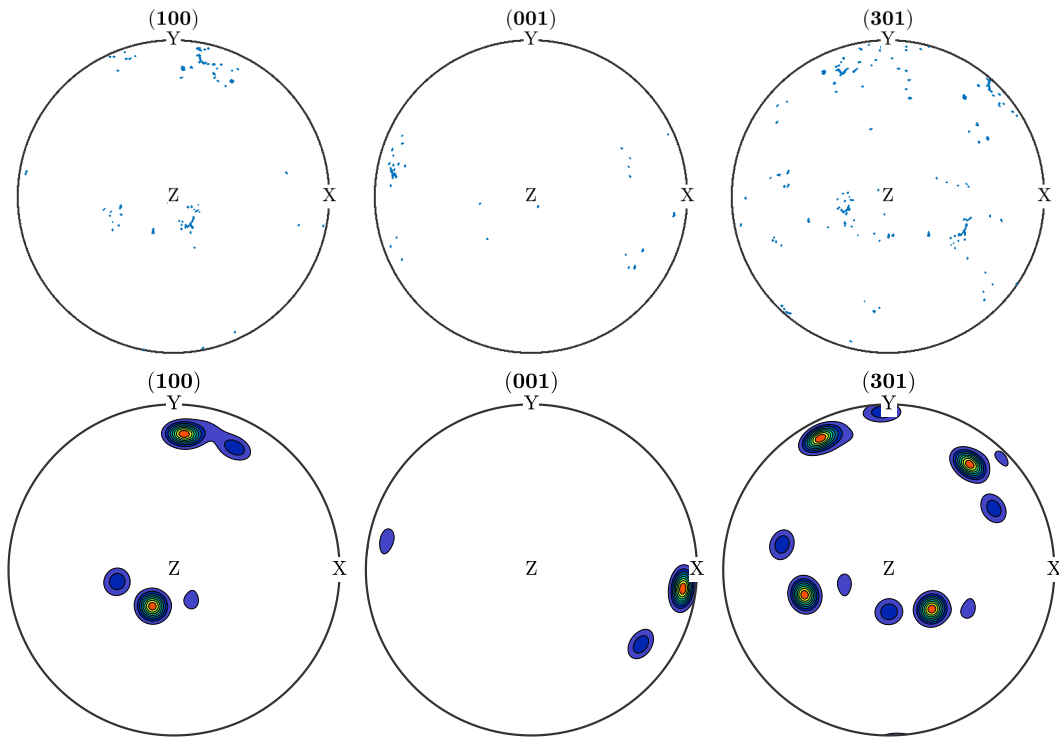


(a) Pole figures for the transverse scan of sample 100_{R-2} by point.

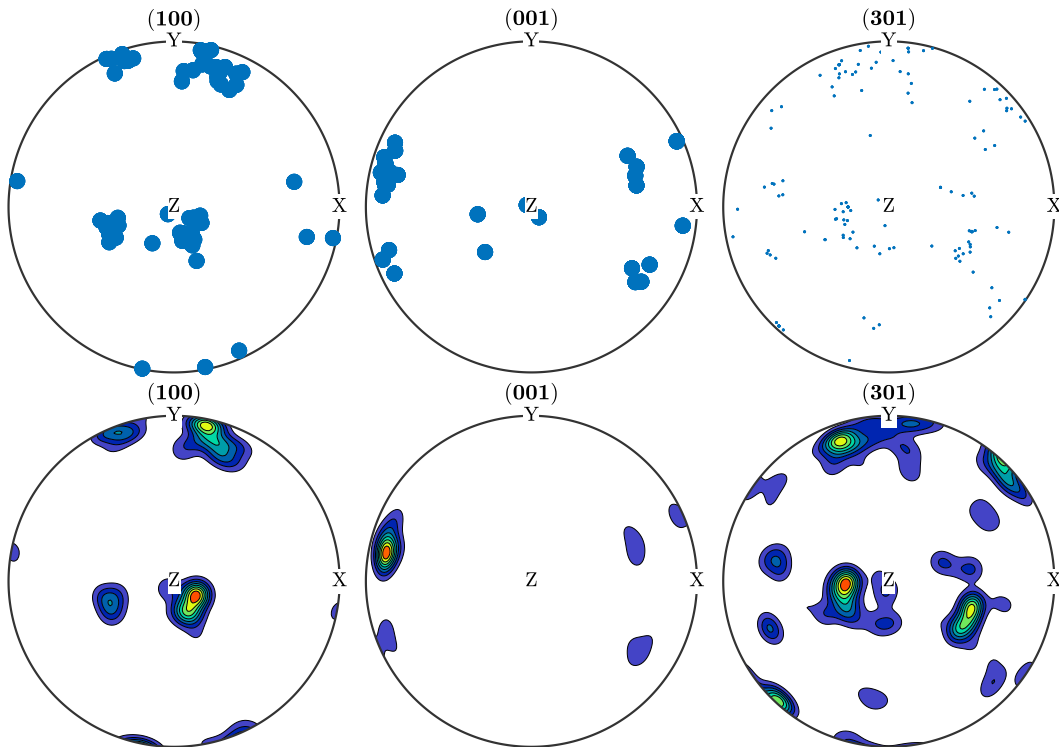


(b) Pole figures for the transverse scan of sample 100_{R-2} by grain.

Figure 6.36: Pole figures for the transverse scan of sample 100_{R-2}.

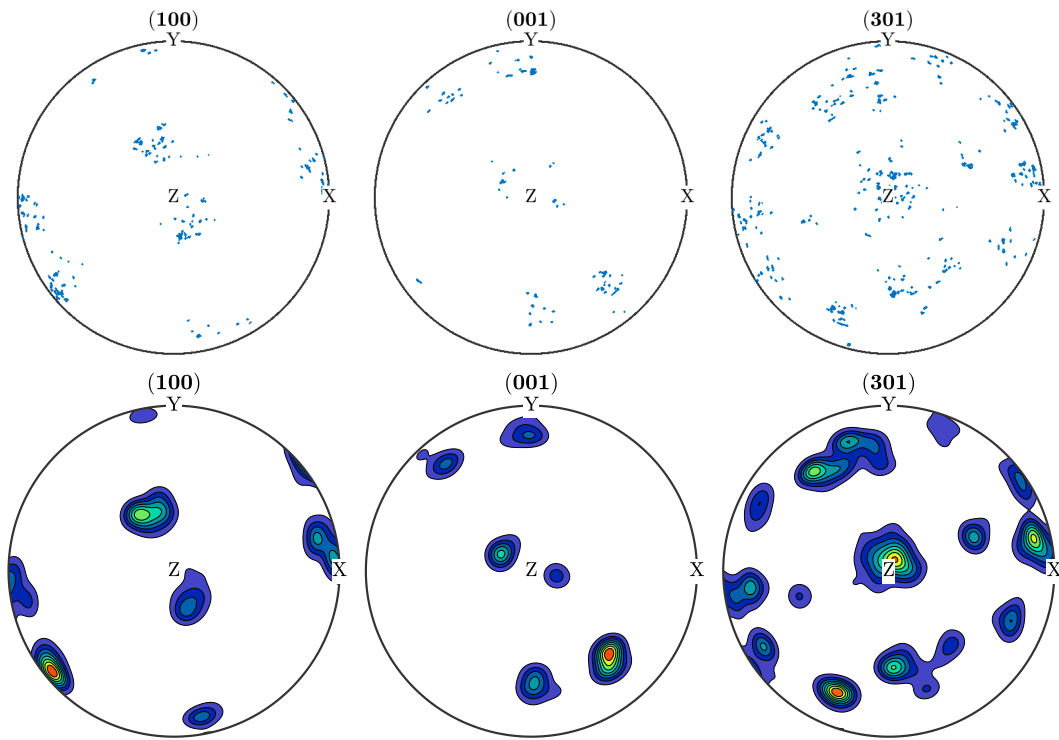


(a) Pole figures for the longitudinal scan of sample 100_{R-2} by point.

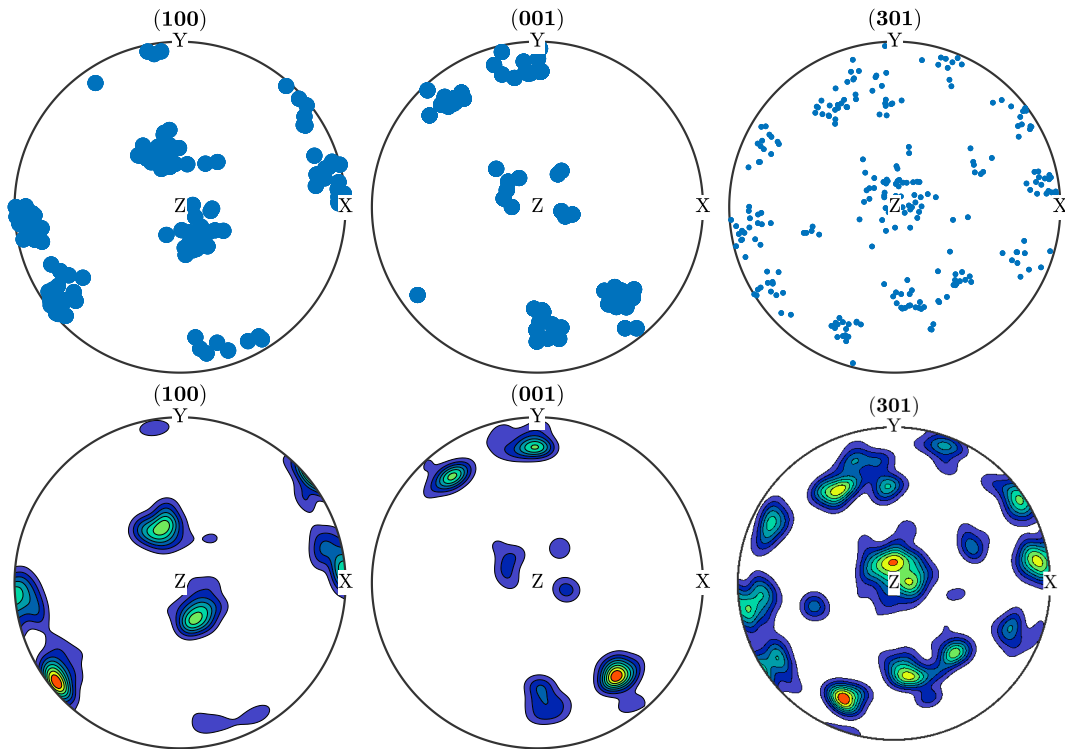


(b) Pole figures for the longitudinal scan of sample 100_{R-2} by grain.

Figure 6.37: Pole figures for the longitudinal scan of sample 100_{R-2}.

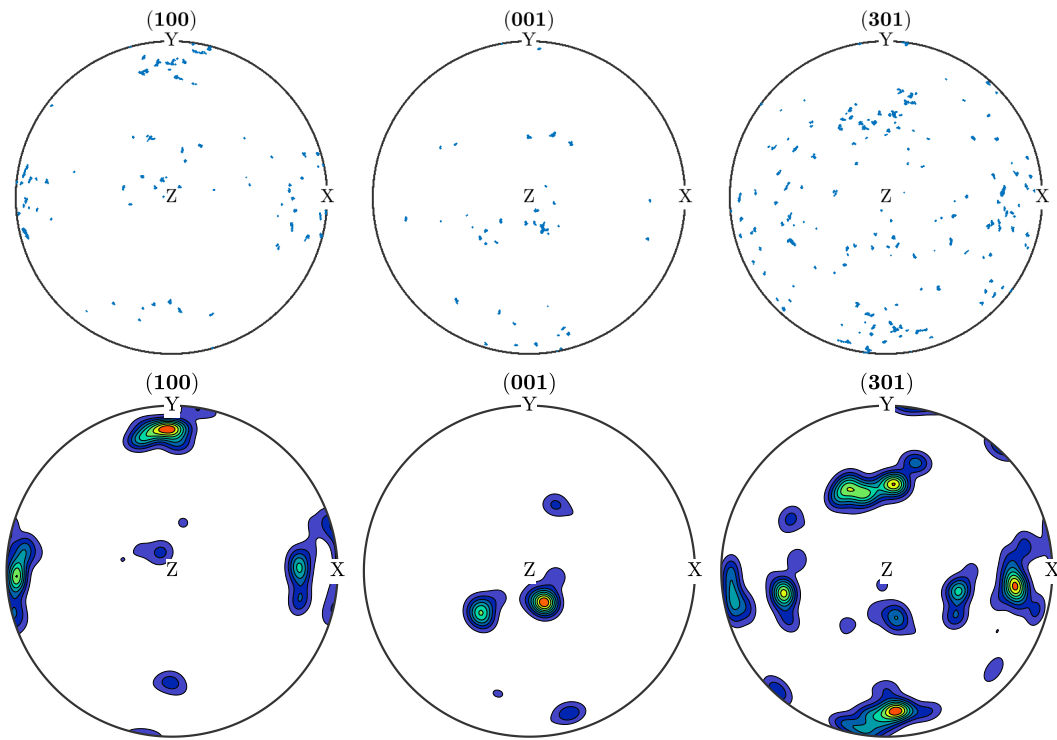


(a) Pole figures for the transverse scan of sample 100_{R-3} by point.

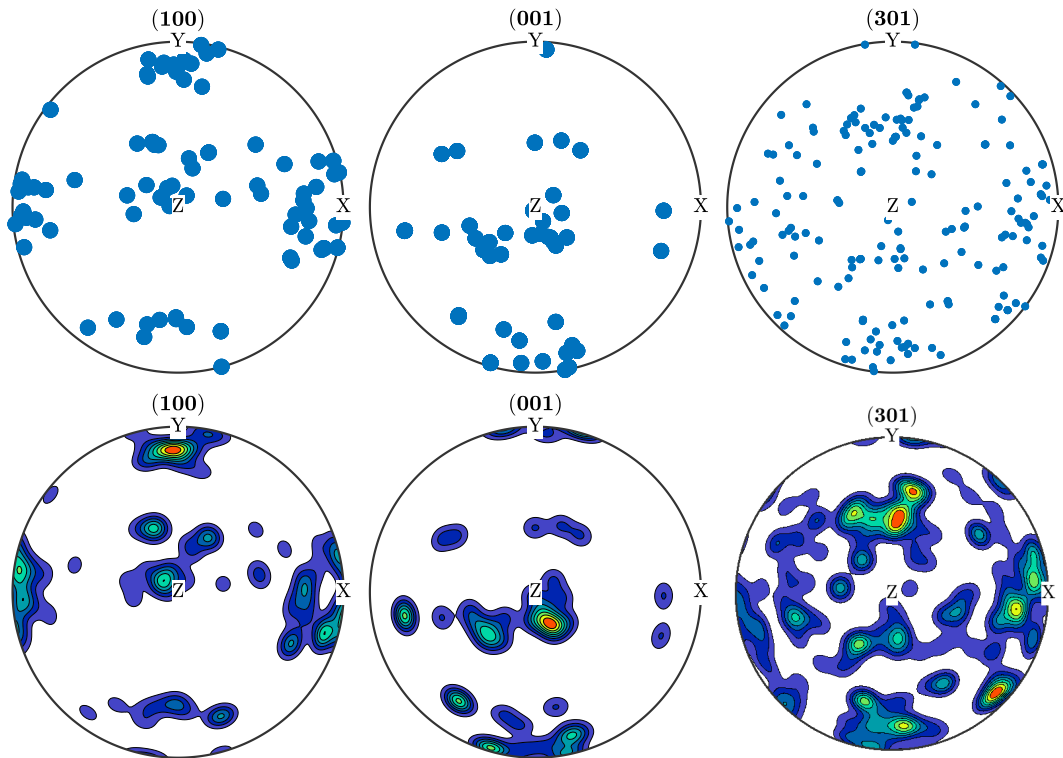


(b) Pole figures for the transverse scan of sample 100_{R-3} by grain.

Figure 6.38: Pole figures for the transverse scan of sample 100_{R-3}.

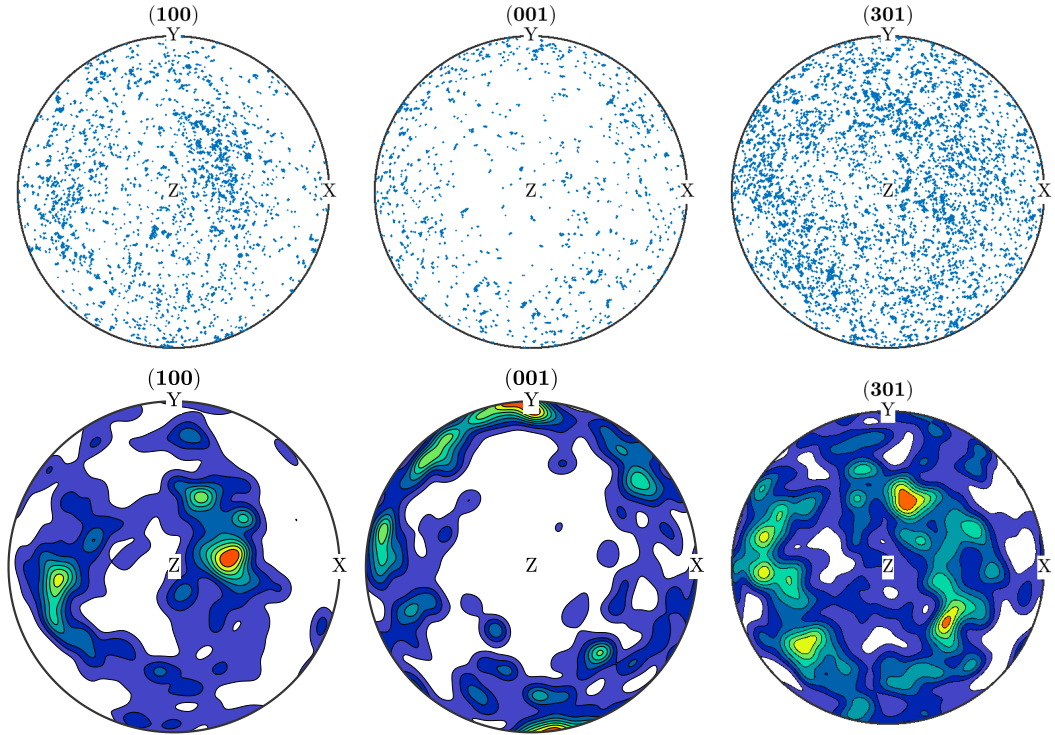


(a) Pole figures for the longitudinal scan of sample 100_{R-3} by point.

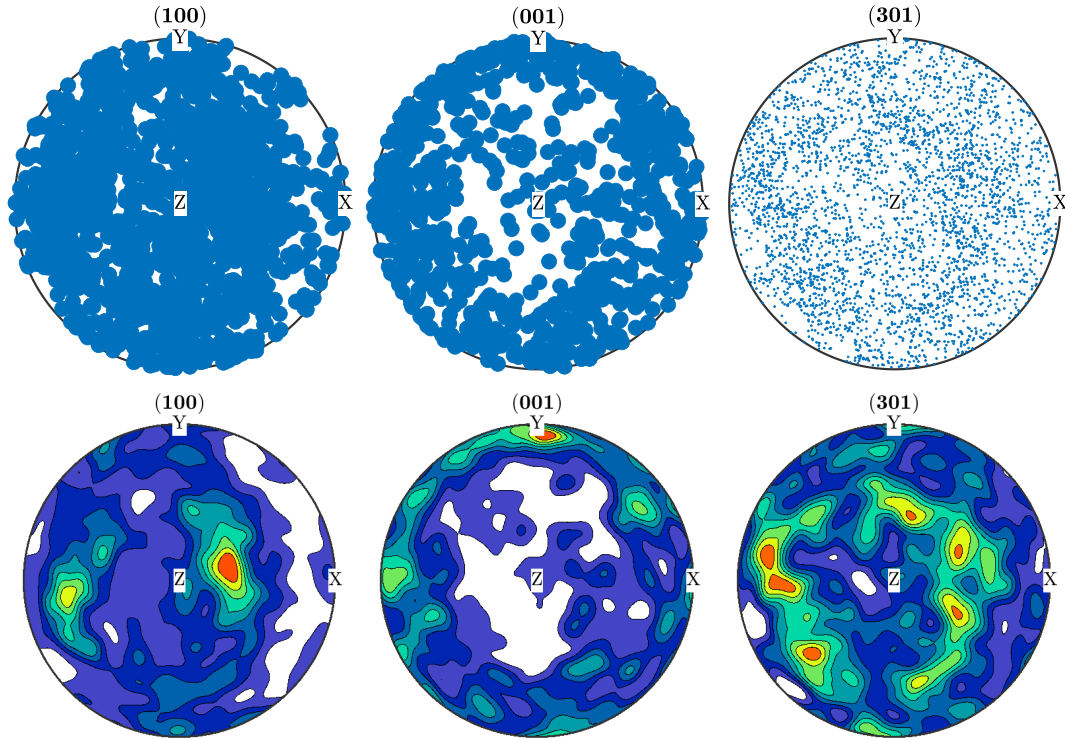


(b) Pole figures for the longitudinal scan of sample 100_{R-3} by grain.

Figure 6.39: Pole figures for the longitudinal scan of sample 100_{R-3}.

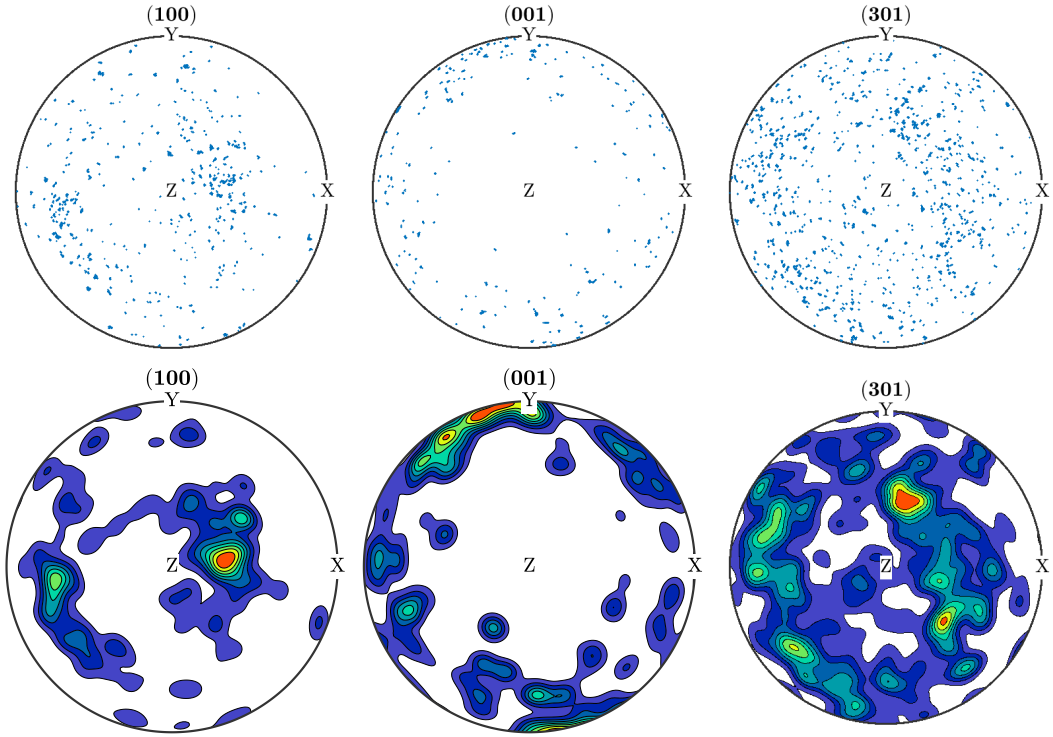


(a) Pole figures for the transverse scan of sample PC_{R-1} by point.

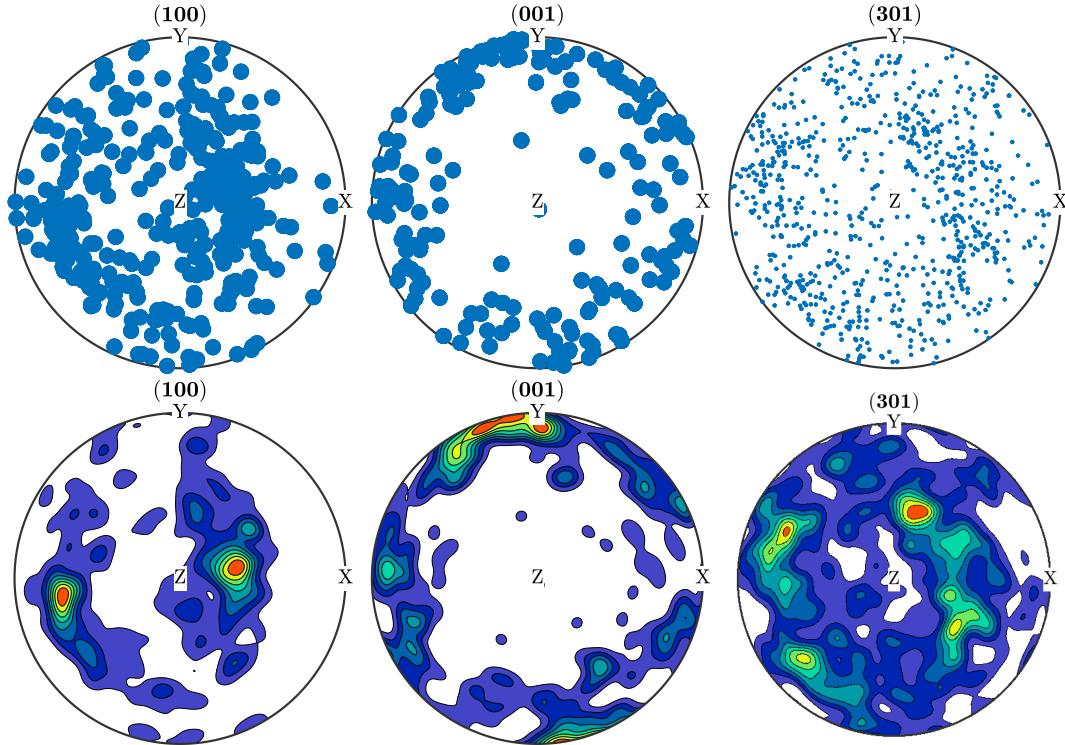


(b) Pole figures for the transverse scan of sample PC_{R-1} by grain.

Figure 6.40: Pole figures for the transverse scan of sample PC_{R-1} .

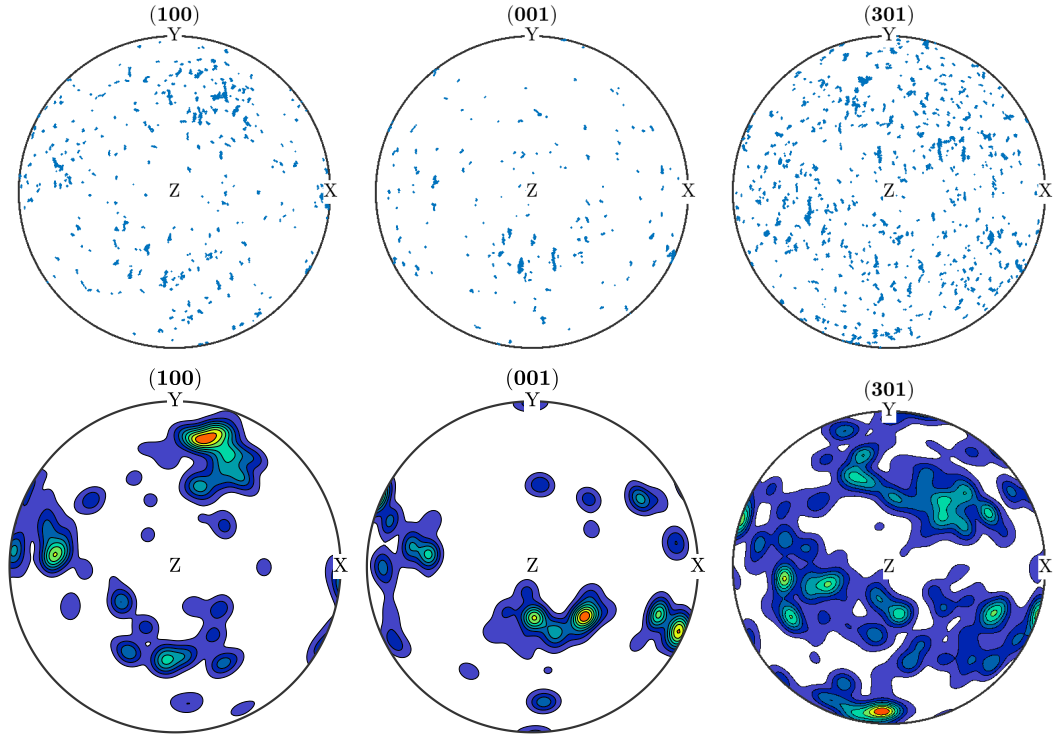


(a) Pole figures for the undamaged region of the transverse scan of sample PC_{R-1} by point.

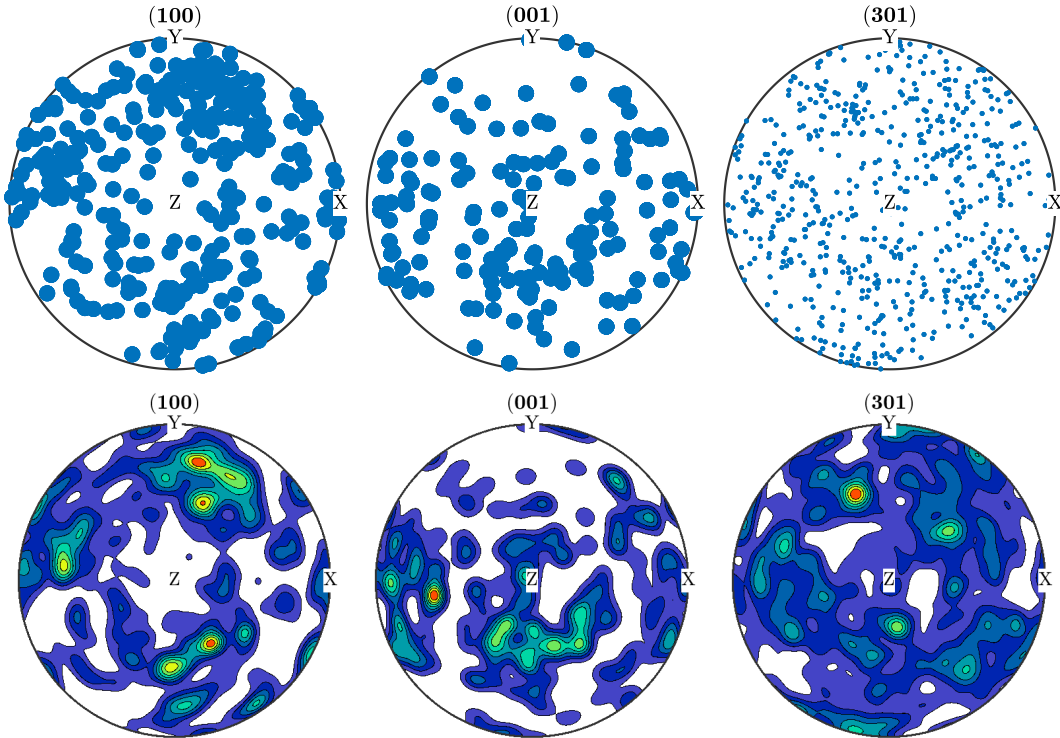


(b) Pole figures for the undamaged region of the transverse scan of sample PC_{R-1} by grain.

Figure 6.41: Pole figures for the undamaged region of the transverse scan of sample PC_{R-1} .

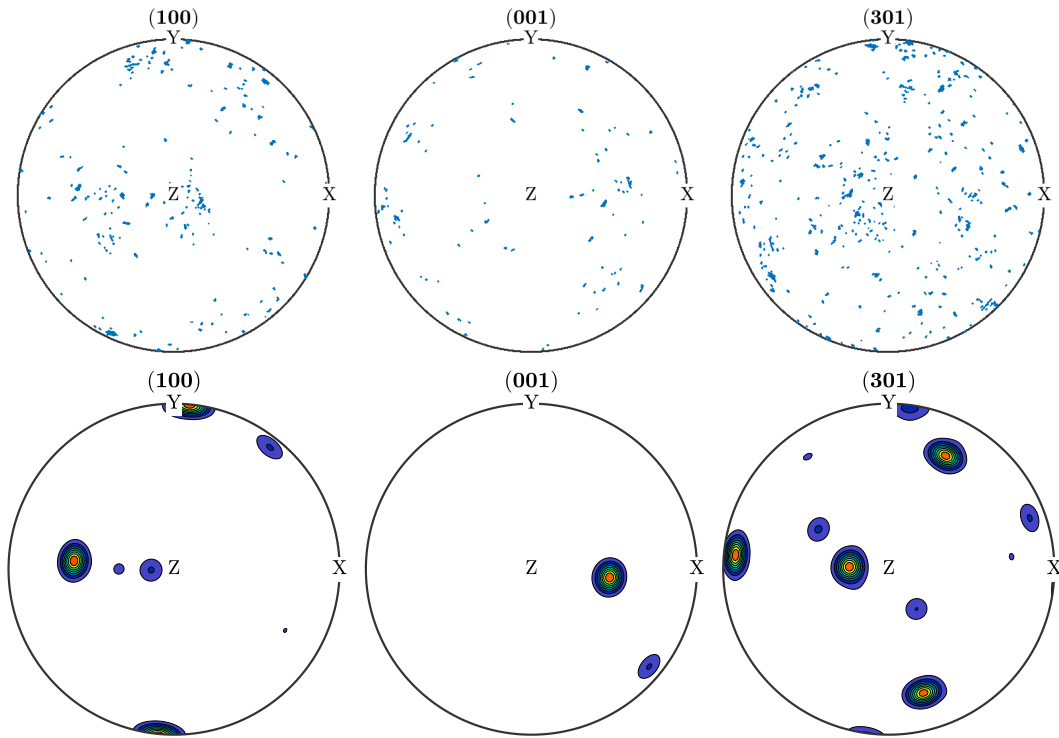


(a) Pole figures for the longitudinal scan of sample PC_{R-1} by point.

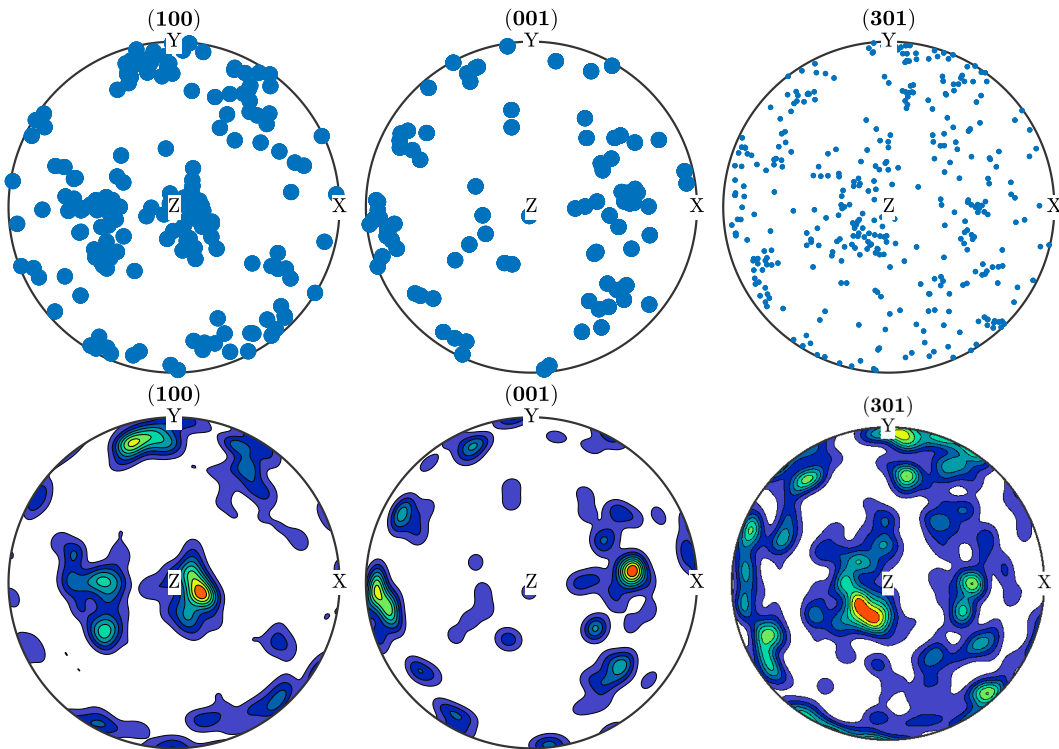


(b) Pole figures for the longitudinal scan of sample PC_{R-1} by grain.

Figure 6.42: Pole figures for the longitudinal scan of sample PC_{R-1} .

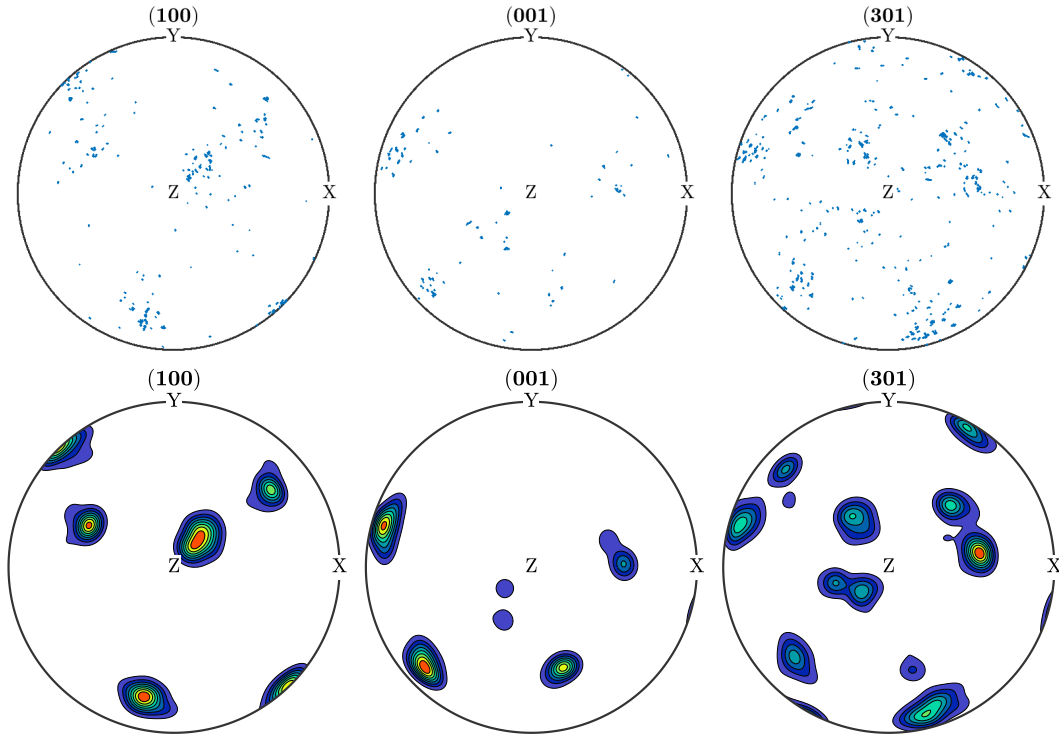


(a) Pole figures for the transverse scan of sample PC_{R-2} by point.

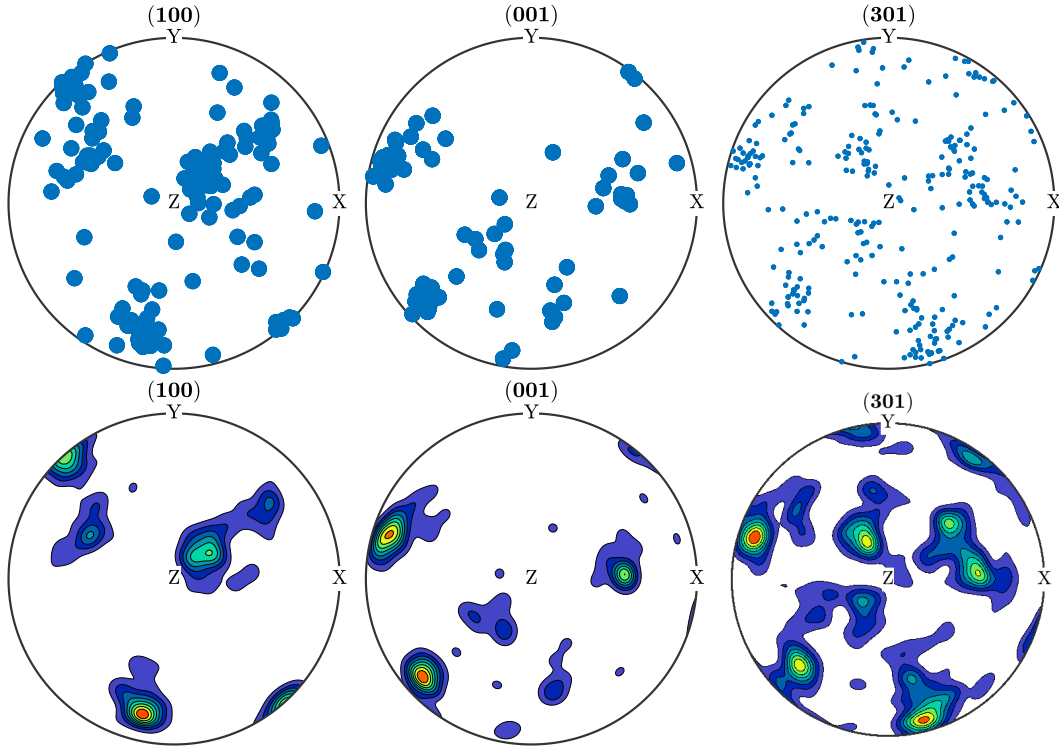


(b) Pole figures for the transverse scan of sample PC_{R-2} by grain.

Figure 6.43: Pole figures for the transverse scan of sample PC_{R-2} .

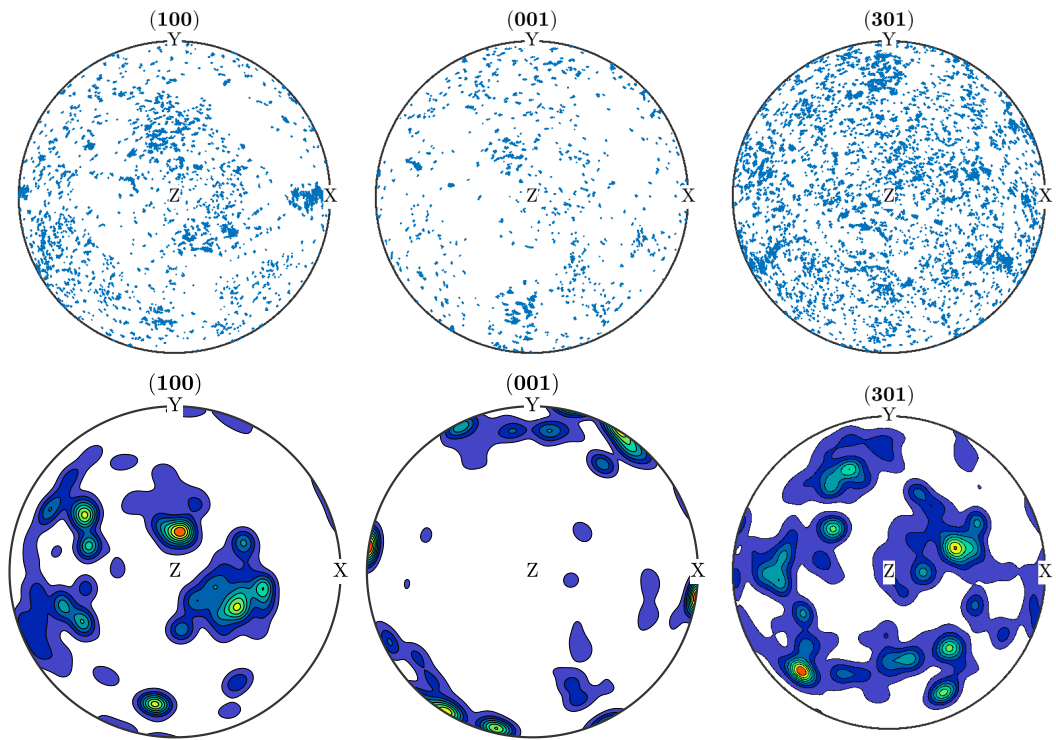


(a) Pole figures for the longitudinal scan of sample PC_{R-2} by point.

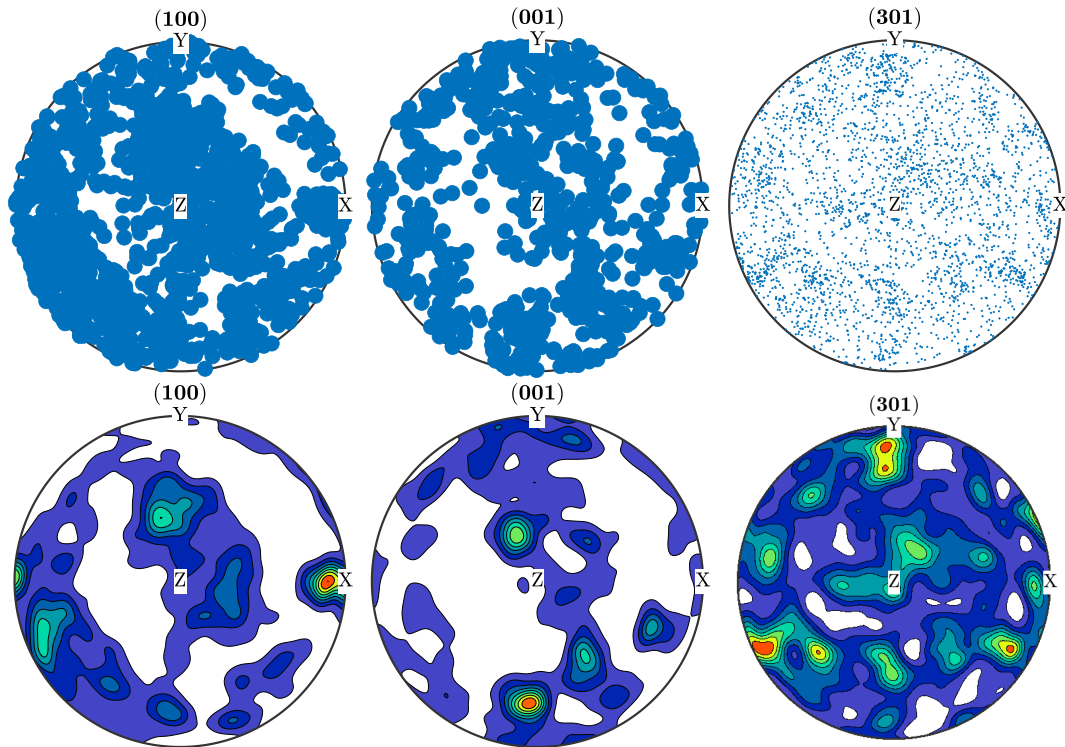


(b) Pole figures for the longitudinal scan of sample PC_{R-2} by grain.

Figure 6.44: Pole figures for the longitudinal scan of sample PC_{R-2} .

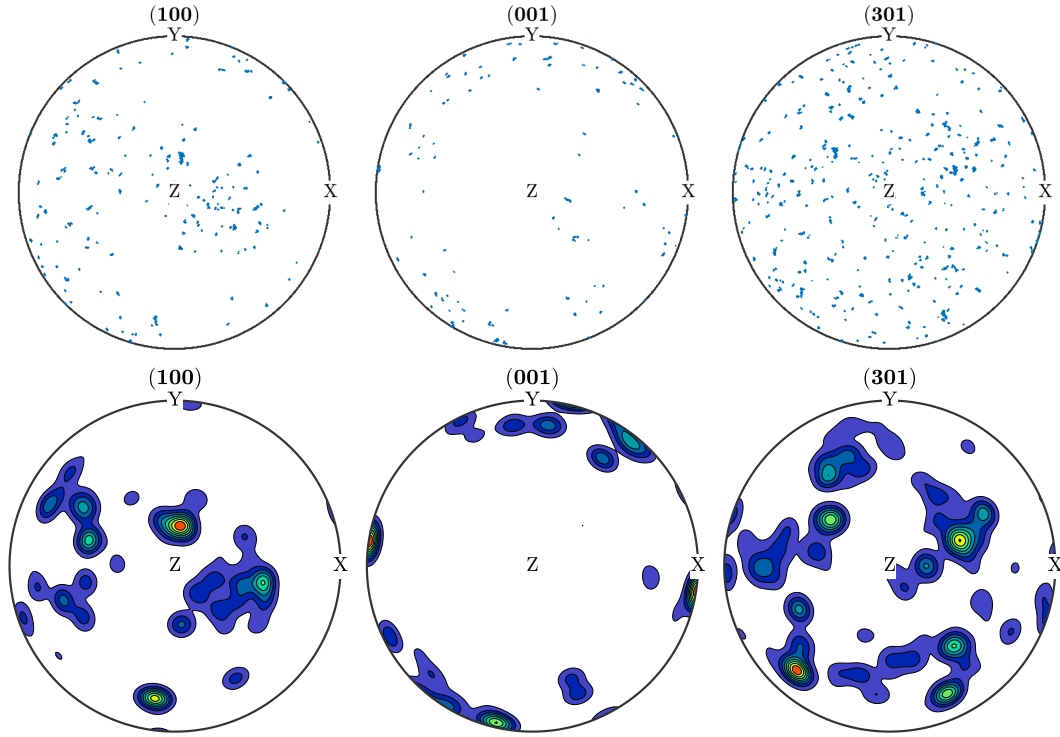


(a) Pole figures for the transverse scan of sample PC_{R-3} by point.

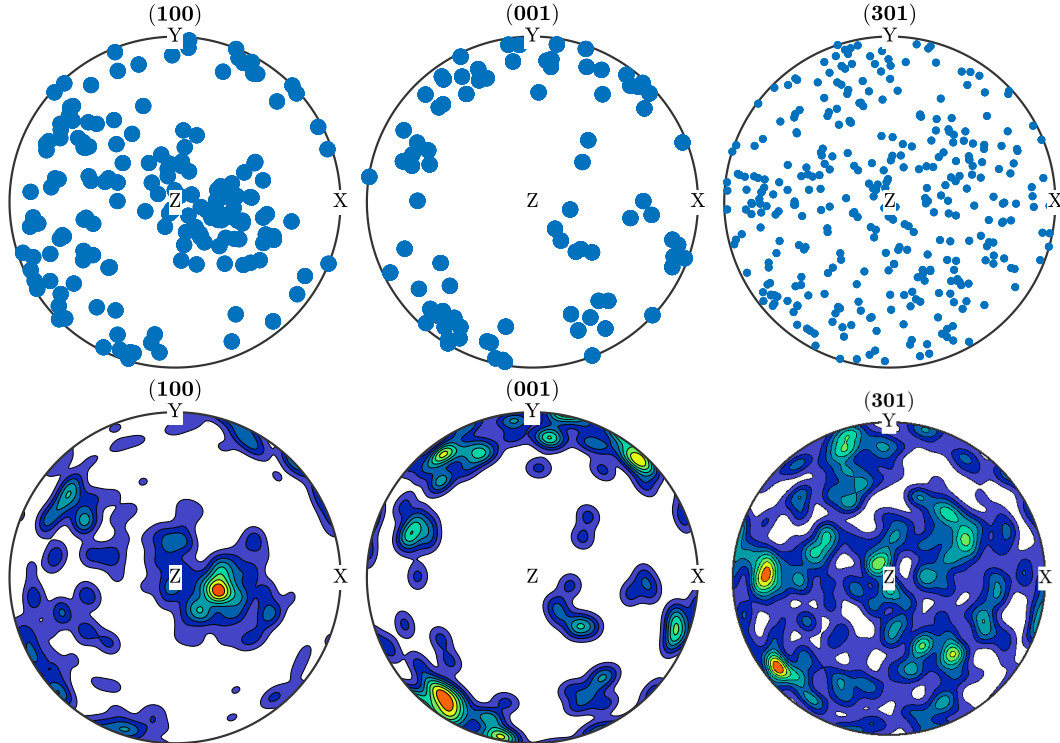


(b) Pole figures for the transverse scan of sample PC_{R-3} by grain.

Figure 6.45: Pole figures for the transverse scan of sample PC_{R-3} .

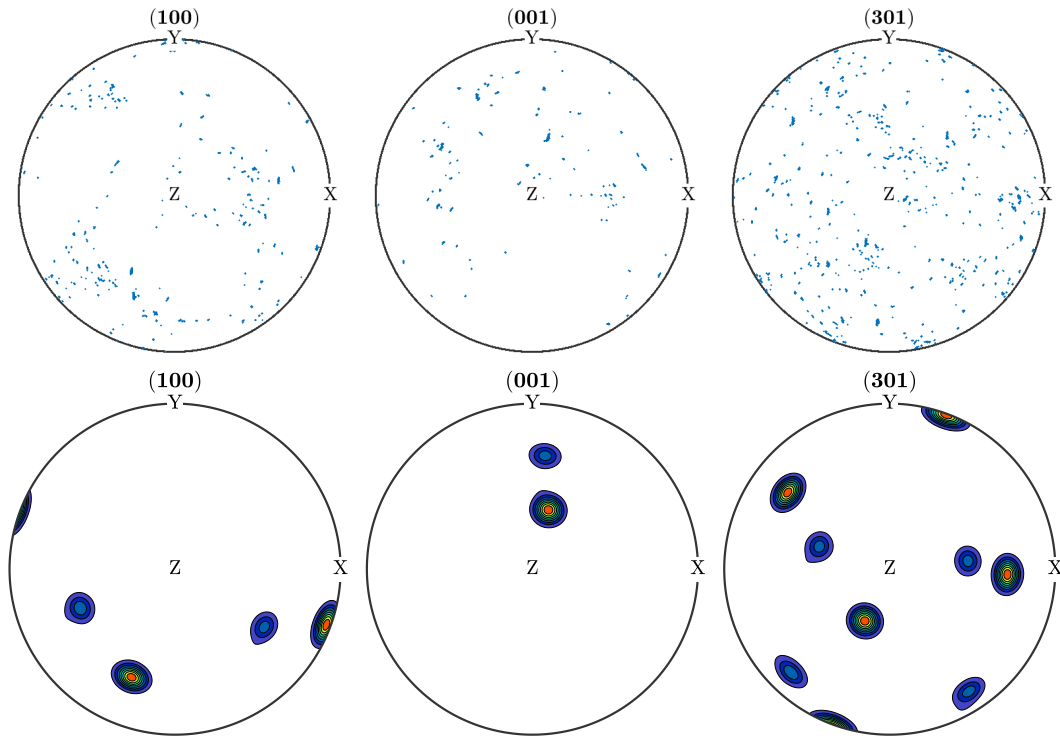


(a) Pole figures for the undamaged region of the transverse scan of sample PC_{R-3} by point.

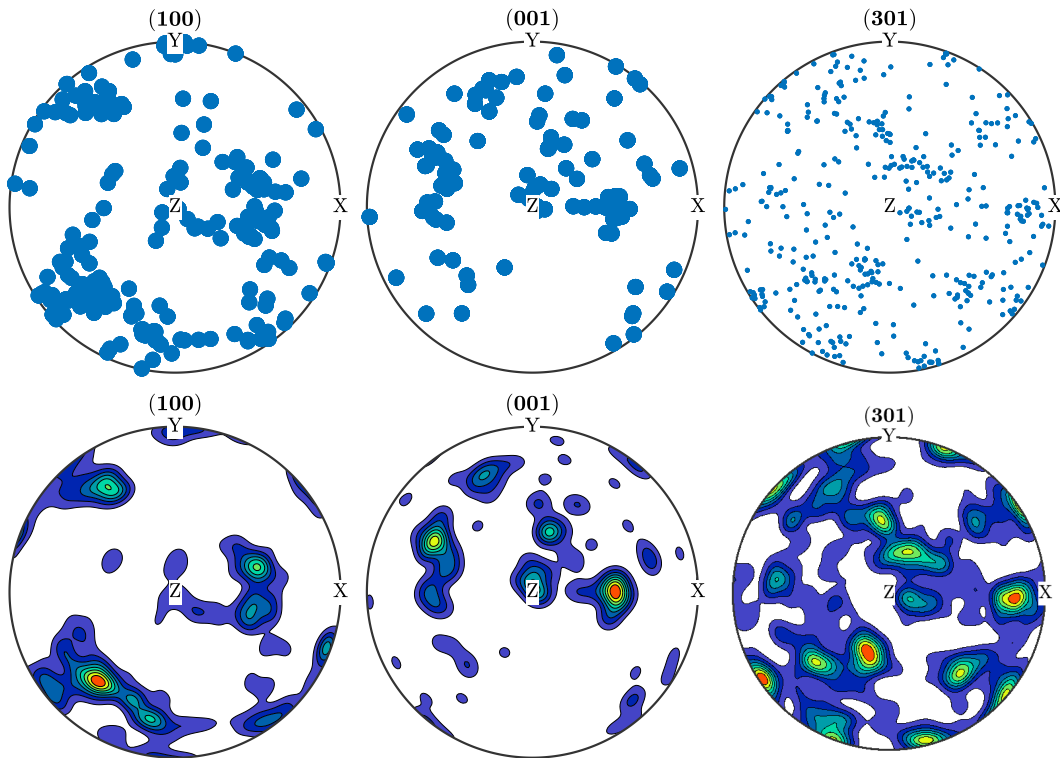


(b) Pole figures for the undamaged region of the transverse scan of sample PC_{R-3} by grain.

Figure 6.46: Pole figures for the undamaged region of the transverse scan of sample PC_{R-3} .



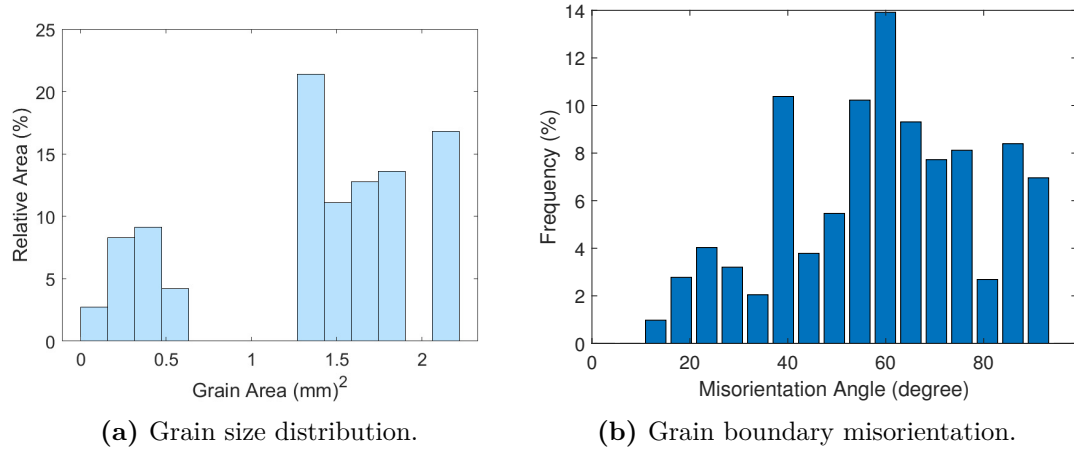
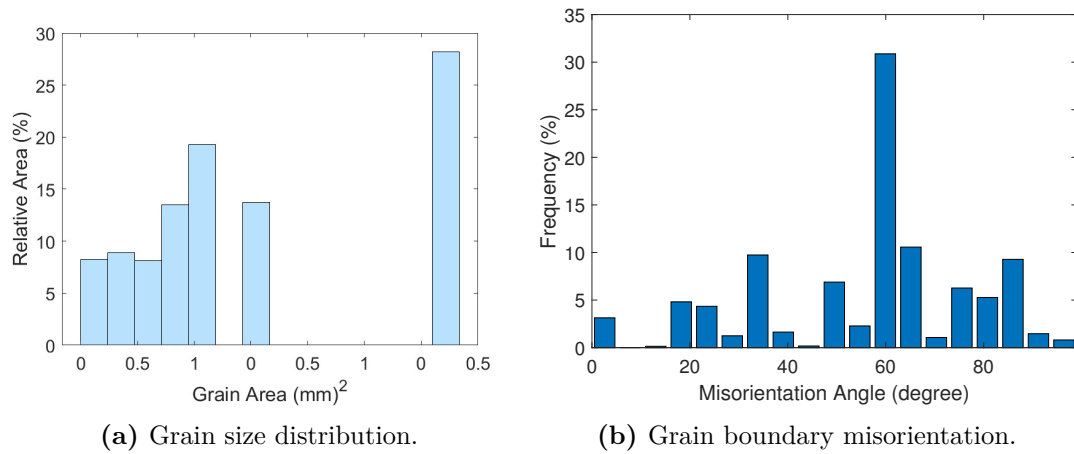
(a) Pole figures for the longitudinal scan of sample PC_{R-3} by point.



(b) Pole figures for the longitudinal scan of sample PC_{R-3} by grain.

Figure 6.47: Pole figures for the longitudinal scan of sample PC_{R-3} .

6.5.2.2 Grain Statistics

**Figure 6.48:** Plots of grain statistics for the transverse scan of sample AR2₁.**Figure 6.49:** Plots of grain statistics for the longitudinal scan of sample AR2₁.

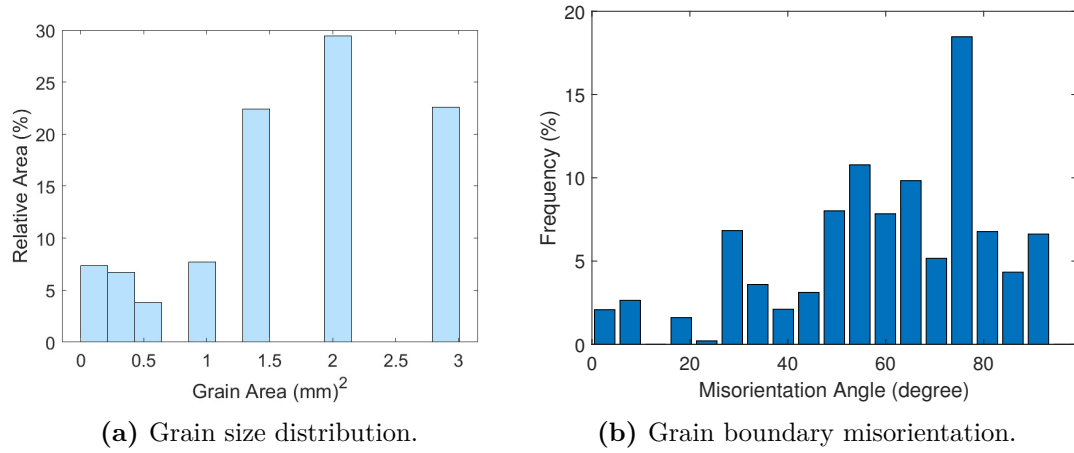


Figure 6.50: Plots of grain statistics for the transverse scan of sample AR2₂.

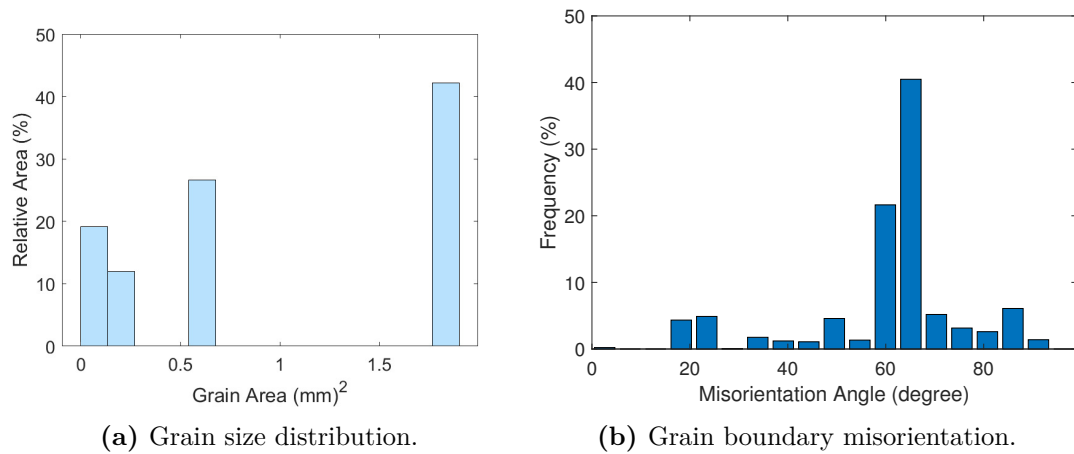


Figure 6.51: Plots of grain statistics for the longitudinal scan of sample AR2₂.

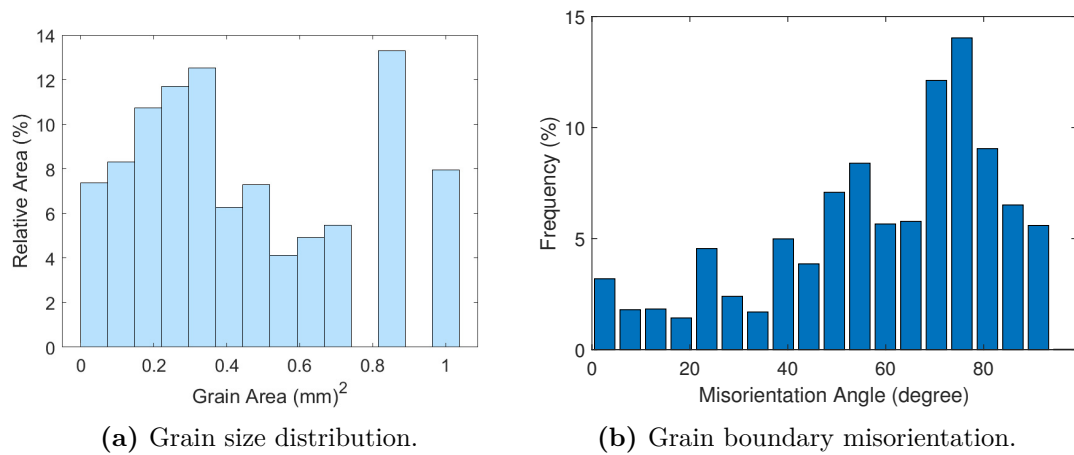


Figure 6.52: Plots of grain statistics for the transverse scan of sample AR1₁.

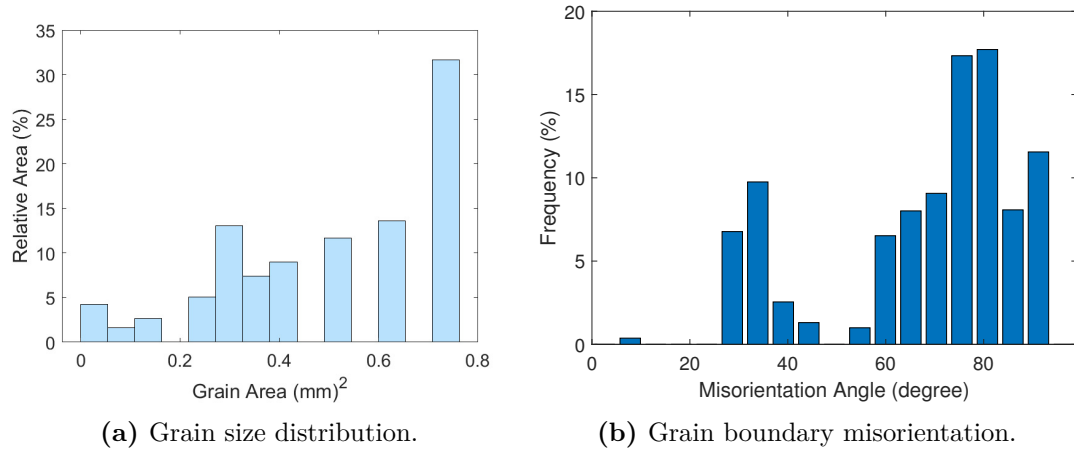


Figure 6.53: Plots of grain statistics for the longitudinal scan of sample AR1₁.

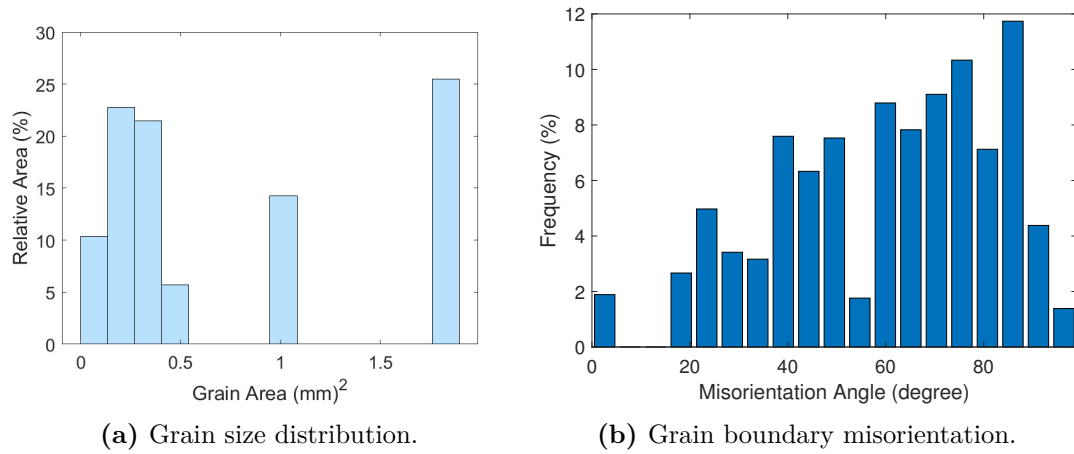


Figure 6.54: Plots of grain statistics for the transverse scan of sample AR1₂.

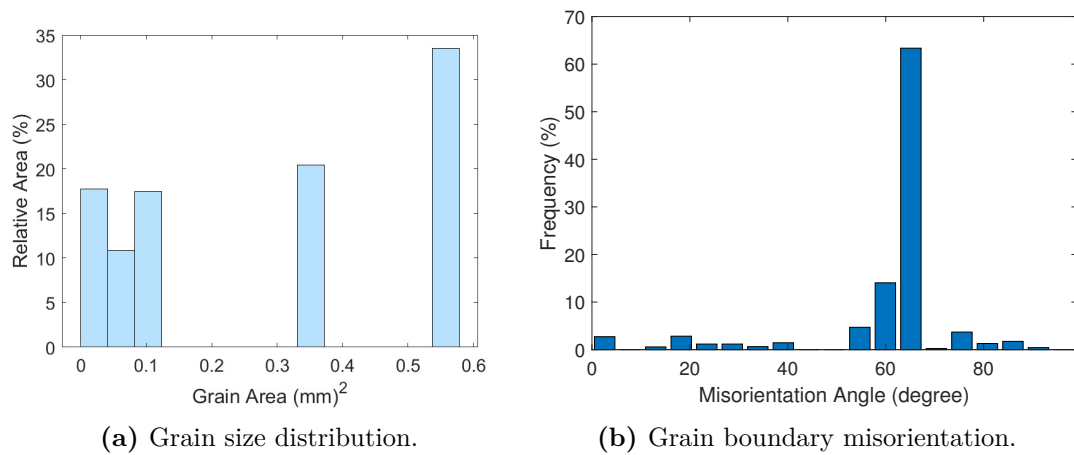


Figure 6.55: Plots of grain statistics for the longitudinal scan of sample AR1₂.

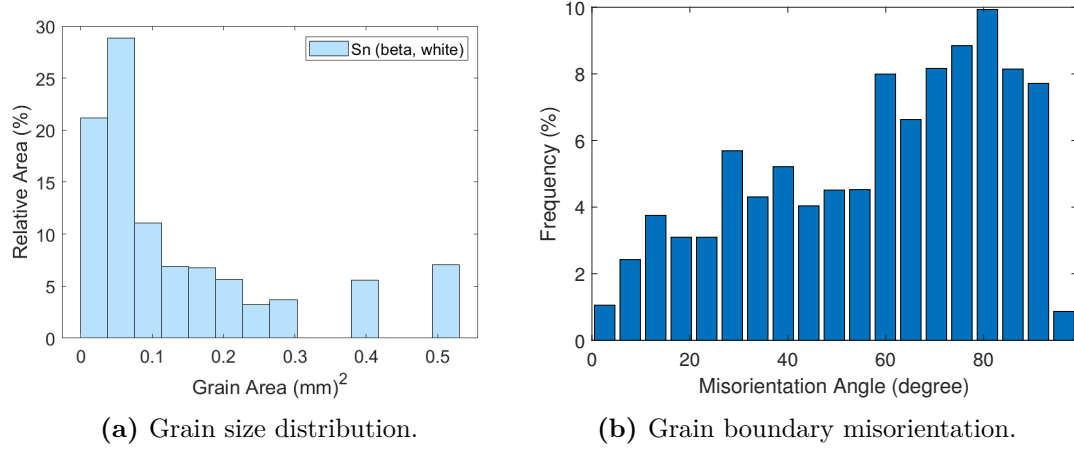


Figure 6.56: Plots of grain statistics for the transverse scan of sample 001_{R-1}.

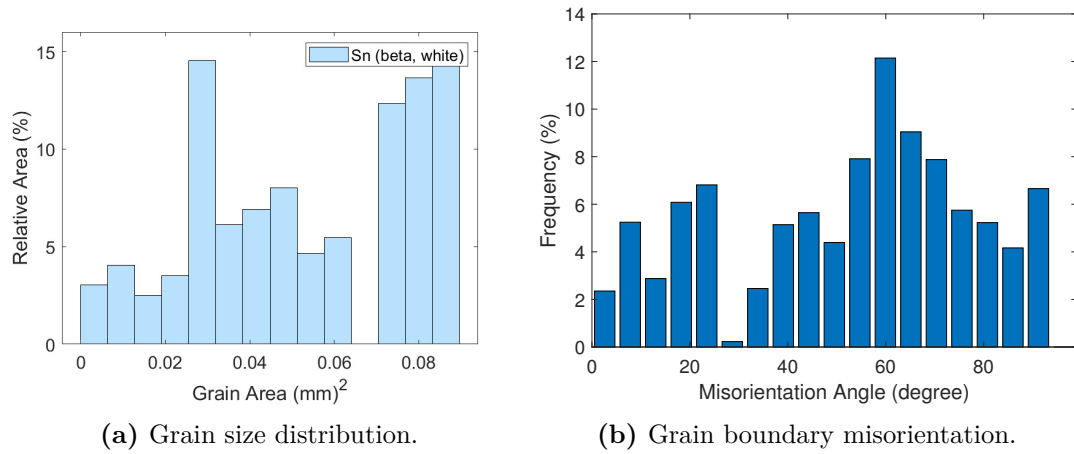


Figure 6.57: Plots of grain statistics for the longitudinal scan of sample 001_{R-1}.

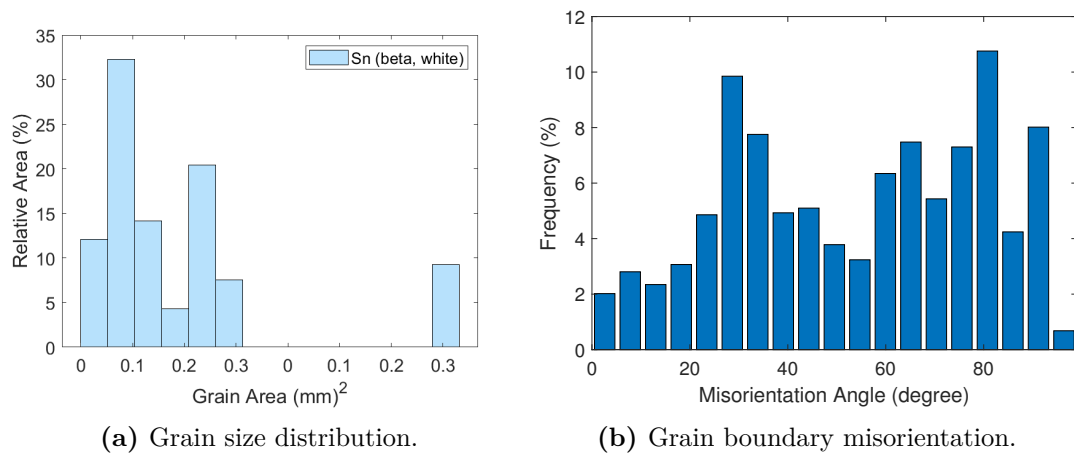


Figure 6.58: Plots of grain statistics for the transverse scan of sample 001_{R-2}.

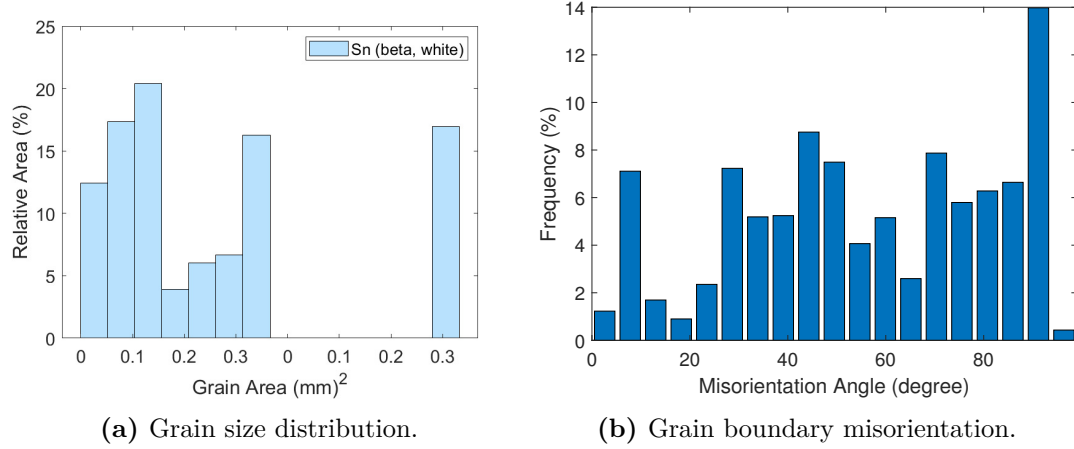


Figure 6.59: Plots of grain statistics for the longitudinal scan of sample 001_{R-2}.

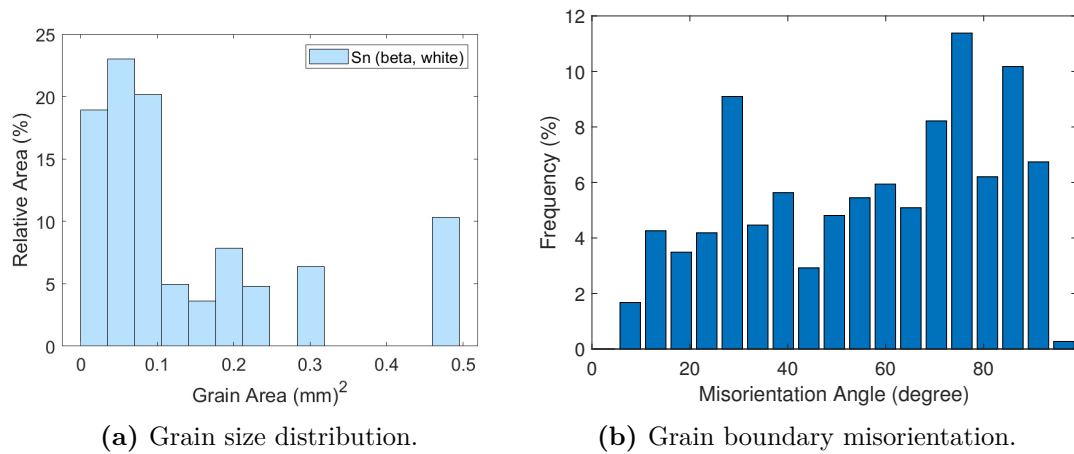


Figure 6.60: Plots of grain statistics for the transverse scan of sample 001_{R-3}.

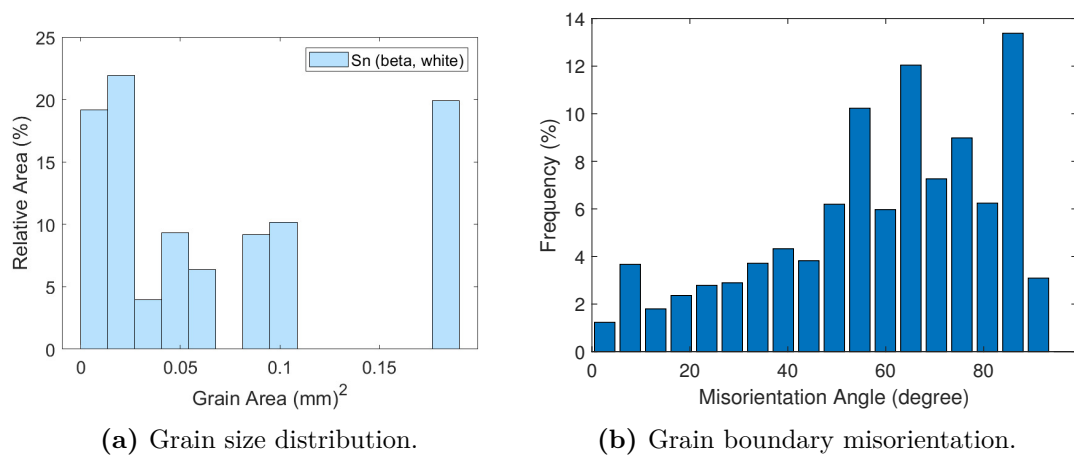


Figure 6.61: Plots of grain statistics for the longitudinal scan of sample 001_{R-3}.

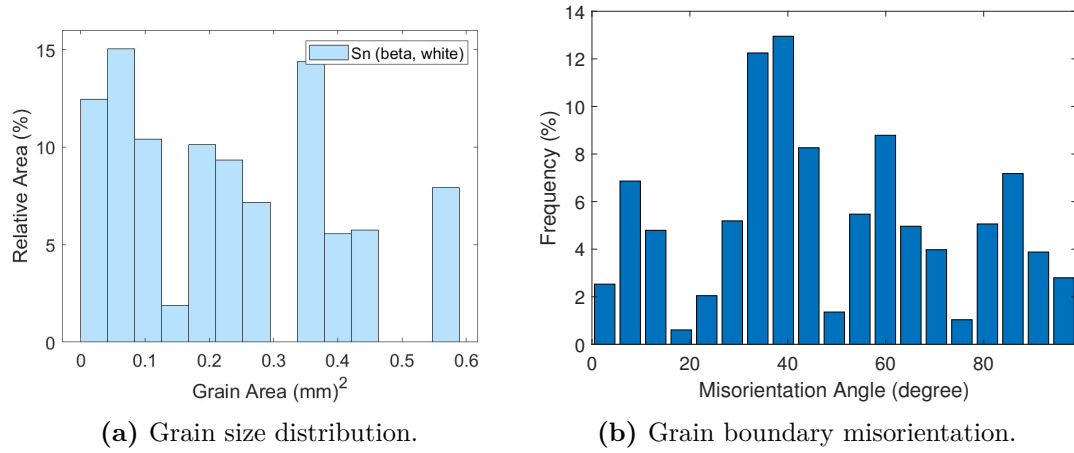


Figure 6.62: Plots of grain statistics for the transverse scan of sample 100_{R-1}.

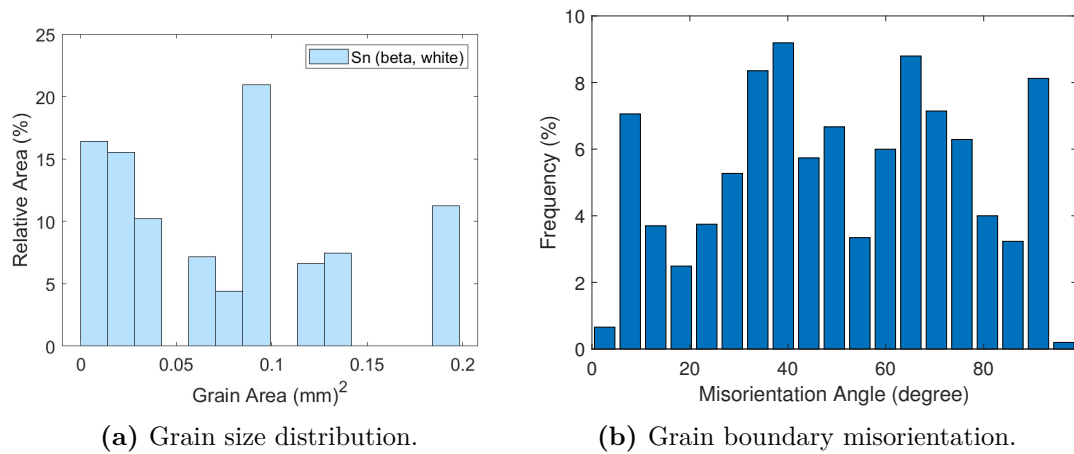


Figure 6.63: Plots of grain statistics for the longitudinal scan of sample 100_{R-1}.

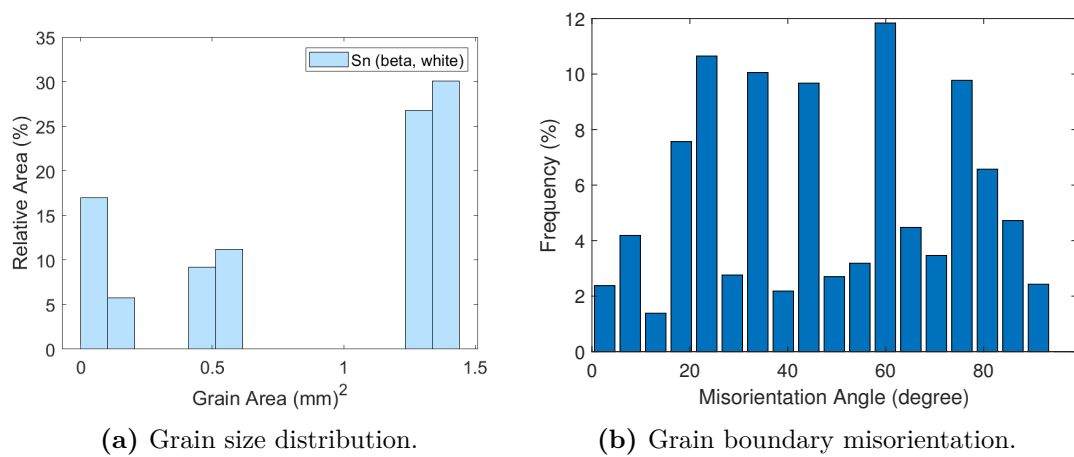


Figure 6.64: Plots of grain statistics for the transverse scan of sample 100_{R-2}.

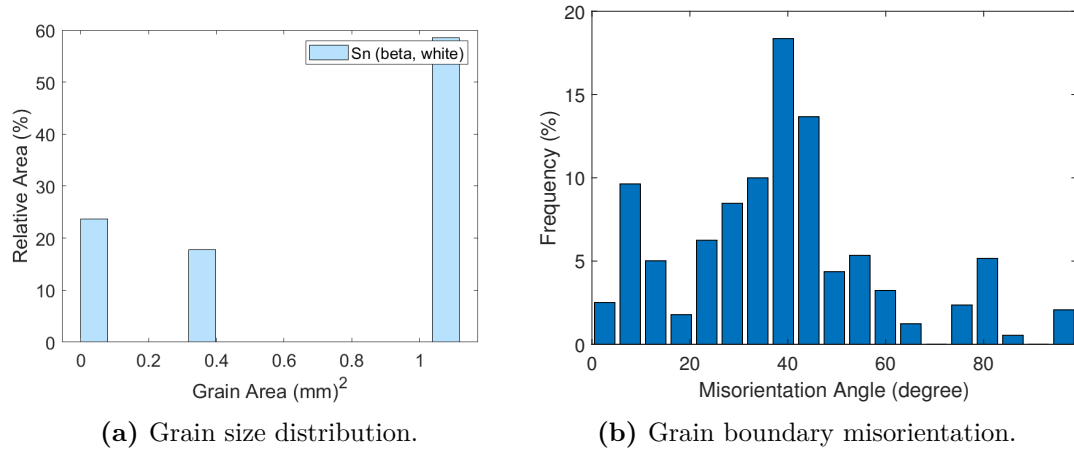


Figure 6.65: Plots of grain statistics for the longitudinal scan of sample 100_{R-2}.

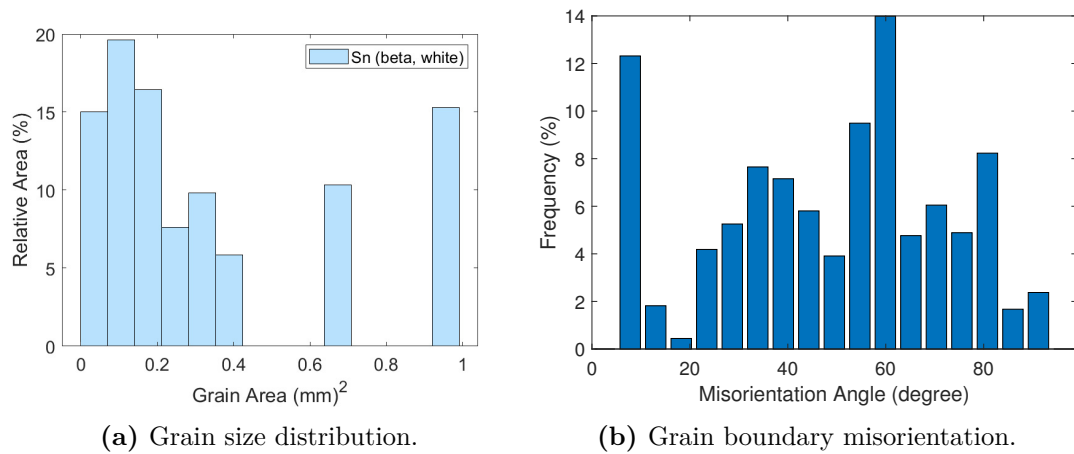


Figure 6.66: Plots of grain statistics for the transverse scan of sample 100_{R-3}.

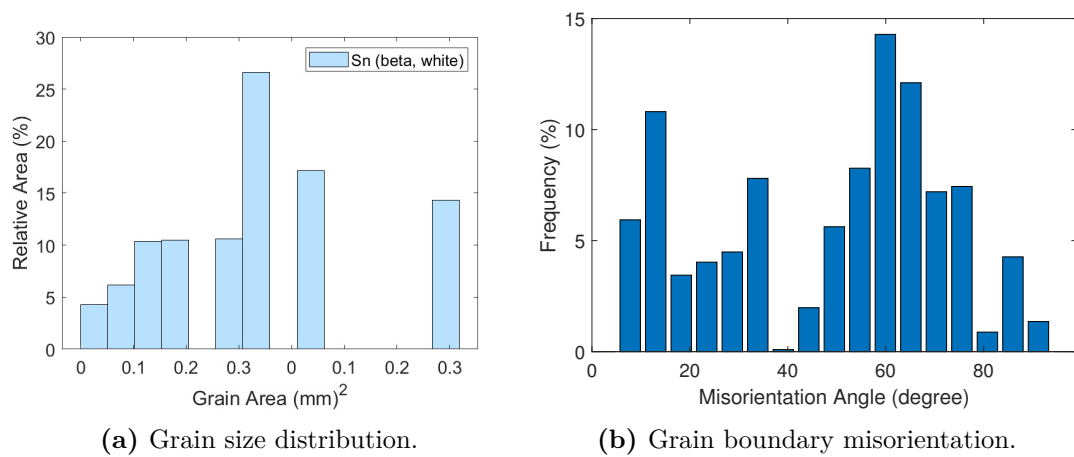


Figure 6.67: Plots of grain statistics for the longitudinal scan of sample 100_{R-3}.

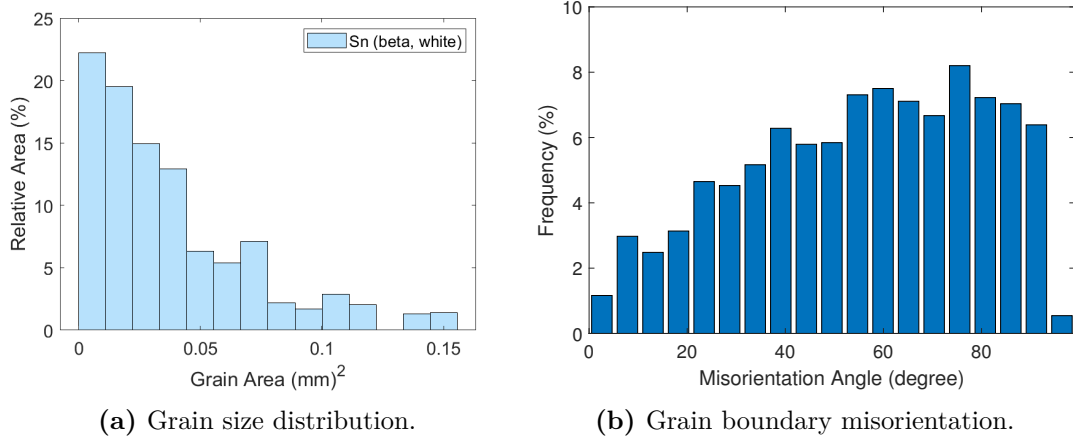


Figure 6.68: Plots of grain statistics for the transverse scan of sample PC_{R-1}.

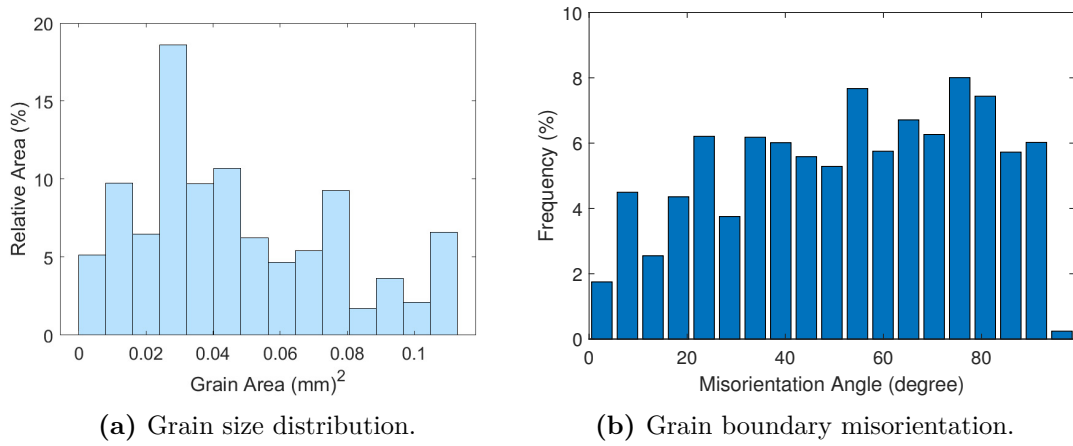


Figure 6.69: Plots of grain statistics for the undamaged region of the transverse scan of sample PC_{R-1}.

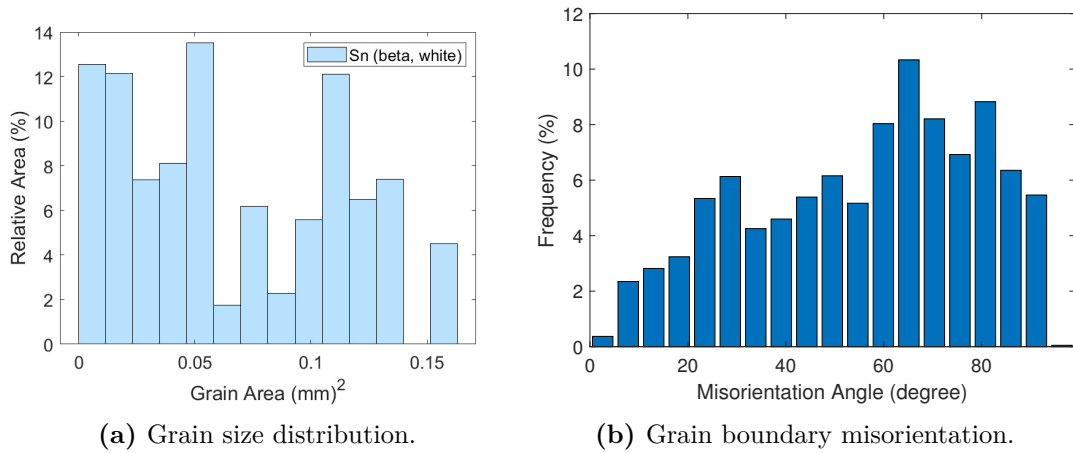


Figure 6.70: Plots of grain statistics for the longitudinal scan of sample PC_{R-1}.

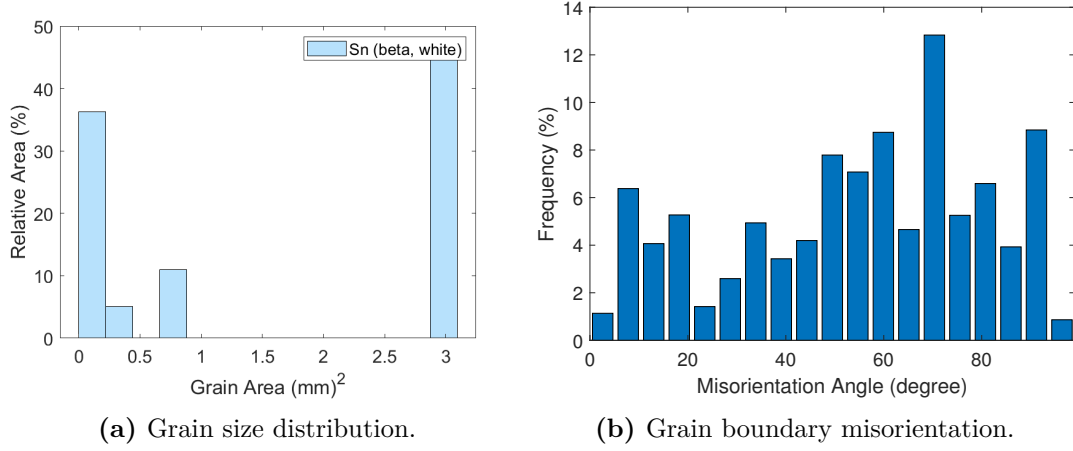


Figure 6.71: Plots of grain statistics for the transverse scan of sample PC_{R-2}.

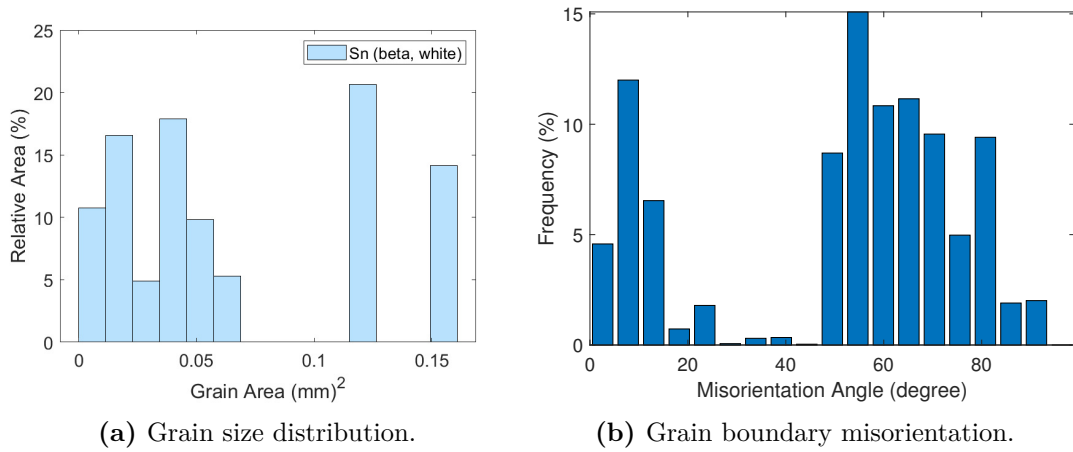


Figure 6.72: Plots of grain statistics for the longitudinal scan of sample PC_{R-2}.

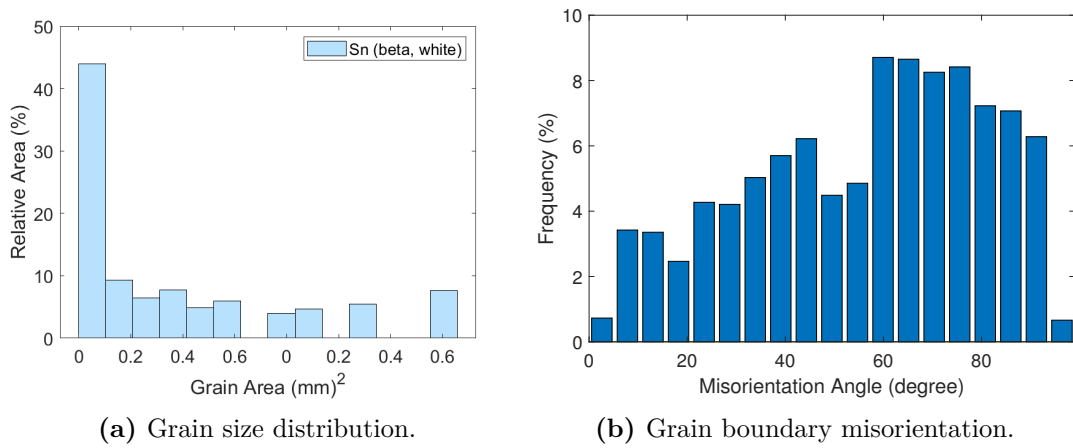


Figure 6.73: Plots of grain statistics for the transverse scan of sample PC_{R-3}.

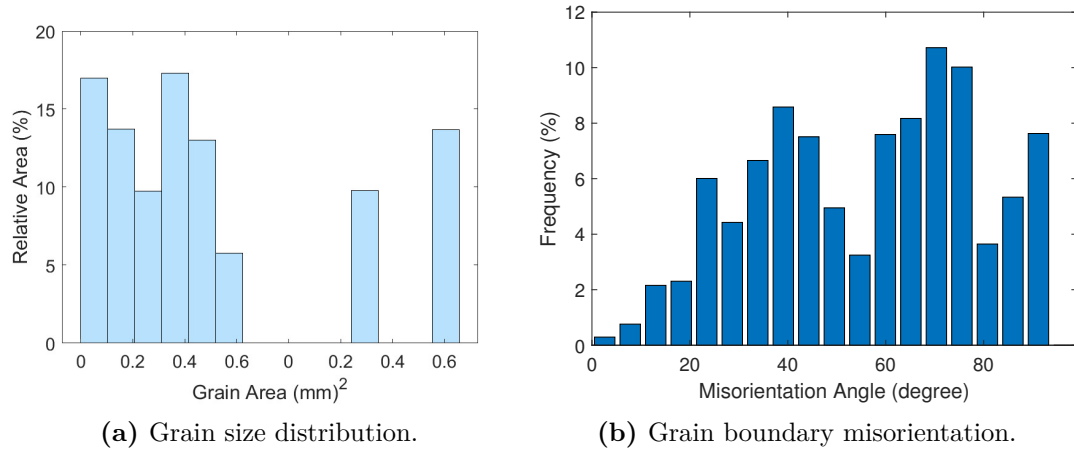


Figure 6.74: Plots of grain statistics for the undamaged region of the transverse scan of sample PC_{R-3}.

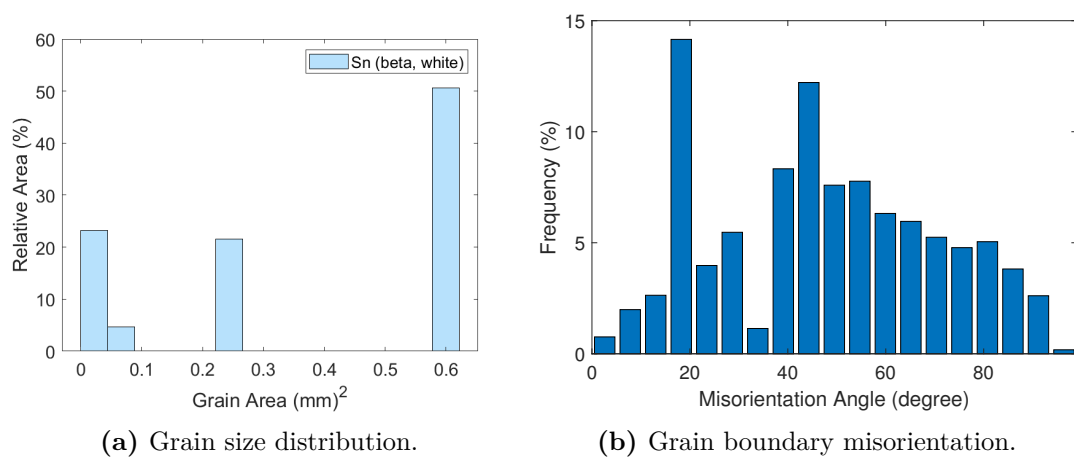


Figure 6.75: Plots of grain statistics for the longitudinal scan of sample PC_{R-3}.

7

Discussion and Conclusions

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7.1 Discussion

Through the study of single crystal tin, a number of key observations have been made. Firstly, the HEL shows anisotropic compressibilities and waves speeds. The magnitude of HEL itself is also highly anisotropic, with an order of magnitude difference between orientations. Above this limit however, the phase transition shows only qualitative differences to the profile, but no quantitative differences to the states. Similarly, both spall and the post shock microstructure show no dependence on either the initial orientation, or the phase reached at the peak state.

By recovering the samples, the resulting microstructures have been examined. This shows significant recrystallisation, especially where damage has formed, and recovered textures that cannot be easily explained using the slip and twin systems observed at ambient pressures in tin.

7.1.1 Plastic Deformation Regime

The anisotropic compressibilities and wave speeds agree well with existing elastic models of tin. However, the magnitude of the HEL is perhaps more unexpected. The HELs in the (100) orientation are around ten times larger than those in polycrystal samples, and the polycrystal HELs also of the same order larger than the static polycrystal yield strength. With a single critical yield stress on a single slip or twinning system unable to predict the yield stress, it is possible that strain rate has a significant effect on the ability of slip systems to activate. Orientation (100) has the lowest elastic compressibility, meaning it may deform faster than other orientations. As a result, plasticity may occur at a higher strain rate, requiring a higher activation stress. With different mechanisms likely active in different orientations, as well as different shear stress fields due to the influence of off-diagonal stiffness constants, this effect may be further compounded. Unfortunately, however, the time resolution of the PDV used is unable to measure the strain rate, meaning that this hypothesis cannot be further examined.

However, a large strain rate dependence is observed in the spall data. Together this might imply a drag-controlled plasticity regime, where the speed of plastic deformation is limited by the scattering of phonons. This would also correlate with the preliminary assessment that spall strength drops at lower pressures when strain rate is increased. As the strain rate increases, so does the resistance to plasticity, inducing additional heating. In micro-ballistic impacts, at a given temperature, increasing the strain rate can sufficiently heat the sample for the response to start to soften [163], an observation that would correlate with the cross-platform spall literature.

Together, the unusual HEL data and spall behaviour, alongside the literature data [163] suggest that the high strain-rate plastic deformation behaviour of tin is governed by drag-controlled plasticity. The data plotting yield stress to strain rate of Schill *et al.* [48] implies a change of regime around $1 \times 10^5 \text{ s}^{-1}$, although the mechanism is proposed to be a “Bauschinger twinning anelastic behavior.”

The mechanism of high-rate deformation proposed in Chapter 6, being a phase transformation and twinning in γ tin may fit the drag behaviour, given the $\beta \rightarrow \gamma$ transition is hypothesised to occur through two phonon modes [143]. Alternatively, conventional plasticity may be thwarted by the drag regime, resulting in this mechanism taking over.

The transition to a drag-controlled regime may also explain unusual texture of the recovered microstructures. In this domain, the barriers to many of the plastic mechanisms observed at low pressures may be much higher, while systems that cannot activate statically may be able to occur more readily.

7.1.2 Recrystallisation

Above the HEL, the anisotropy is lost, with only qualitative differences to the phase transition. The explanation for the qualitative differences may lie in the mechanisms of the phase transition. With multiple transition pathways proposed [93], the multiple paths could activate dependent on the initial orientation, and plastic history. Thus, the dynamics of nucleation and growth of the new phase may occur at different rates and pressures for the various orientations, resulting in the different profiles, while overall threshold remaining broadly the same. These competing mechanisms may well result in the significant grain refinement associated with the transition [24, 26, 139].

Alternatively, the qualitative differences in the phase transition may be due to residual reverberations of the elastic precursors causing the sharper or more rounded features. Given the large differences in elastic behaviour, this might not be unexpected. The recovered samples examined in Chapter 6 all show a similar microstructure, independent of reaching the phase transition, suggesting the

recrystallisation may occur prior to the phase change. If this were the case, and the microstructure had already converged, then the phase transformation would be expected to occur at the same pressure between all the samples. Thus, the reverberations of the elastic waves would therefore be the key cause of the qualitative differences, with the underlying properties remaining the same.

Indeed, the recovered microstructures under shock appear to be similar in samples that have undergone the phase transition, and those that have not. All samples recovered from pressures above their HEL show evidence of having undergone the $\beta \rightarrow \gamma$ phase transition, and a flip of the c and a axis. Not only does this hint at similar mechanisms acting in the samples, but also that the phase transition may not form the primary driver of recrystallisation, but part of a chain of steps each with multiple symmetries. Instead recrystallisation likely occurs at pressures below the transition step.

This latter theory, where the microstructure is dynamic, and recrystallisation occurs readily, would also explain the lack of anisotropy in the spall behaviour. Having recrystallised during the shock loading, and further recrystallised during release, any trace of the samples original state are therefore eradicated. As a result, spall occurs in a similarly structured local environment, resulting in the independence from both peak state and initial microstructure.

The temporal nature of the recrystallisation cannot be definitively determined in the existing literature work. The lack of *in situ* X-ray observations of the shocked β phase in particular mean the specific nature of the recrystallisation cannot be specified. Only γ is compared to ambient material. The recovered samples presented here similarly show that recrystallisation has occurred between the start and the end of the experiment. Indeed, examining the spall planes shows that the grains are forming alongside, or even after damage. The grains are found to be of the same length scales to the spacing between pores. Whether therefore recrystallisation is an intrinsic part of the deformation behaviour, or occurs during the quasi-stable dwell periods, with grains expanding to reduce the stored deformation energy, is therefore unclear.

7.1.3 Deformation Behaviour

The c/a flip observed in Chapter 6 appears to dictate the behaviour of β tin at elevated pressures. Likely underpinned by a $\{111\}$ in the γ phase, this may act to prevent the lattice becoming more tetragonal. This may explain why (100) remains elastic for so long, due to the small reduction in internal energy that such a twin could achieve when the [001] and [010] axis are very close to perpendicular to the shock axis.

However, one would expect (001) with the [001] direction the closest to the axis to yield at the lowest pressures, consistent with the observations in polycrystal samples. However, this is not the case, with (110) exhibiting the lowest yield point, closer to the polycrystal value determined in Chapter 4. Activation of another process, likely slip or twinning, at lower pressures may better explain HEL magnitudes.

Assessment of energies associated with the various loading conditions, microstructures, and mechanisms may also help develop understanding of how they evolve. Orientation (100) for instance offers the lowest compressibility, and such the lowest strain-energy for a given 1D strain between orientations. After the HEL, the measured pressure and volume for all the orientations and the polycrystal converge onto a single Hugoniot curve. Thus, for a given volumetric compression, and the minimum strain-energy for a given volumetric strain being the hydrostatic loading case, plasticity could be expected to rotate and deform the lattice from the prescribed shock-induced 1D strain towards a local hydrostatic stress field. With [100] offering the lowest 1D compressibility, the corresponding strain energy for a set strain will be closer to the hydrostatic condition than other orientations, leaving less energy available for plasticity to occur. This is similarly reflected in the stress field (see Chapter 5.3.8.2), which shows comparatively smaller deviatoric stresses than other tested orientations.

While this may explain part of why the HEL in (100) is particularly large, such an approach still does not explain the magnitude of the difference between the orientations. With $C_{11} = 72.30 \text{ GPa}$, $C_{33} = 88.40 \text{ GPa}$, $C_{110} = 88.25 \text{ GPa}$ and $C_{\text{Bulk}} = 54.6 \text{ GPa}$, calculated from the parameters of Rayne and Chandrasekhar [39], the energy difference does not match the difference in the magnitude of HEL,

even considering a third order model (see Figure 4.7). However, it may be able to better explain the textures observed in the recovered material. While the theoretical hydrostatic case would have no preference with regards to sample axis, orienting the stiffer [001] axis away from the shock direction would accommodate small 1D strains better. Indeed, from the stiffness matrix, it can be seen that the low coupling between [001] and [100] means that a 1D strain applied along [001] would more significantly change the deviatoric component, further decreasing the desirability of [001]. This remains speculative, and it is highly likely that the plastic processes involved in the recrystallisation shape the possible microstructure.

7.1.4 Discussion Summary

Together, these results imply several key phenomena underpinning the behaviour of tin. Firstly, drag controlled plasticity likely dictates the mechanisms of deformation. At high strain-rates, the plastic behaviour appears to be significantly different to the ambient behaviour, with drag controlled plasticity able to provide an explanation of many of the key observations made here. Secondly, dynamic recrystallisation likely shapes the bulk behaviour of the material. This may be driven by a c/a flip, corresponding to twinning in γ tin, but likely occurs at all steps in the loading history. Notably recrystallisation may erase any orientation dependent properties above the elastic limit, especially if the deformation mechanisms are similar between orientations. Drag-controlled plasticity meanwhile may form a good predictor of the activation thresholds for phenomena such as spall and plasticity.

7.2 Future Work

Further work is clearly required to further understand the plasticity, recrystallisation and $\beta \rightarrow \gamma$ phase transition in tin. A number of potential avenues are available to do this, each offering distinct approaches, goals, and likely outcomes.

7.2.1 Theoretical Work

One of the key aims of this work has been to challenge and verify existing models of tin. Towards this, seeking to reproduce and understand the results of the experiments in this work represents a goal for future work. Two techniques in particular, MD simulations and CPFE modelling are especially relevant. This relationship is not just one way, with these techniques also able to offer a better understanding of this work's key observations.

MD, with its focus on modelling the forces between atoms, offers a technique to understand the properties of tin from the bottom up. This can offer insights into the changes to the crystalline structure, and how they influence the bulk properties. In particular, using the technique to predict the onset and mechanisms of plasticity and the $\beta \rightarrow \gamma$ phase transition could prove fruitful endeavours, further increasing understanding of these phenomena. The rapid proliferation of machine learning based potentials has significantly enhanced the capabilities of this technique. With its physics based approach, if molecular dynamics is able to reproduce the key results of the experimental work, it inherently provides a window into the atomic rearrangements that underpin the larger scale phenomena in a way that cannot be easily reproduced through other techniques, experimental or theoretical.

Similarly CPFE can be used to predict and model the bulk properties. The experiments presented in this work have shown that the plastic mechanisms are one of the key influences on the overall behaviour. This may prove more challenging compared to MD simulations, due to the uncertainty regarding the specific conditions under which the individual slip mechanisms operate. For instance, this work has highlighted that a simple Schmid model is insufficient to describe the HEL. Speculation on a more appropriate activation modelling may yield valuable results, but given that even static activation stresses for the slip systems in tin are not well defined, this may prove challenging. As a result, using MD to predict the deformation and transformation behaviour, coupled with CPFE to bridge the length and time scales to reproduce the bulk measurements here would combine the strengths of the two techniques. Ultimately, a versatile CPFE model could be used to model more

than just the behaviours discussed in this work, but could be used to predict other lengths, time scales and loading conditions that cannot be easily studied.

Together, these theoretical techniques offer powerful routes to further investigate the measurements made here. Testing hypotheses within their frameworks can offer unique insights into the key observations of this work. Particular areas of focus, beyond simply reproduction of results, may be to focus on the mechanisms of the previously mentioned phenomena, notably the c/a flip, plastic-drag and recrystallisation. The two techniques are each better positioned to focus on one of the two key mechanisms. MD, with its focus on the atomic interactions in the lattice, may be better able to examine the plastic mechanisms, and how they are effected by the strain rate. However, the comparatively small lengths scales compared to the typical grain sizes examined here make studying recrystallisation phenomena more challenging. The problem for CPFE is reversed, with models of slip required to determine grain scale processes such as recrystallisation. The two processes therefore compliment each other well, and their implementation will build a more detailed understanding of the high-strain properties of tin.

7.2.2 Experimental Work

Further experimental work can also build upon, and strengthen the results presented here. For instance, the release wave measurements presented in Chapter 5.1 are incomplete, and further measurements of single crystals above and below the phase transition would better validate the conclusions reached. The initial results suggest that the release wave, unlike the HEL, has the same speed regardless of orientation, although a more complete dataset is required to improve confidence in this assessment. Release wave measurements of (100) around the shock HEL may be useful, given the different strain rates on release.

Similarly, additional experiments to better map out any potential relationship between peak pressure and the phase transition may resolve the uncertainties regarding the trend in Figure 4.9. Complementing this data with hydrocode modelling, similar to that by Cox and Robinson [137] or Mabire [25, 134] would

also be mutually beneficial, helping develop the model's capabilities, as well as the understanding of the underlying phenomena.

Investigation of the threshold for melt-on-release during spall may also be valuable to explore. Considering the strain rate relationship in Figure 5.10, samples approaching melt-on-release rapidly diverge from the trend-line. It can be seen that the lowest strain rate tests, by Robert *et al.* [93], do not diverge significantly from the trend, despite the peak stress reaching 15 GPa. The intermediate tests by Wu *et al.* [178] start to diverge around 13 GPa. Meanwhile the laser shocks by de Ressaiguier [162, 175–177] in Figure 2.5 appear to start rolling-off at the lowest pressures of any experimental measurements, perhaps as low as 10 GPa. A multi-platform approach and analysis may be able to better map out this hypothesis, expanding the datasets already generated and yielding greater confidence to this assessment.

Further investigation of the HELs would offer additional insight into the onset of plasticity. Examining other orientations and strain rates would offer a broader dataset. While this may not directly identify the mechanisms of plastic deformation, a broader dataset would provide a more robust foundation to test theoretical yield criteria. The limited data presented here can only offer a snapshot into this behaviour. Perhaps more significant, fielding faster diagnostics, and modifying experimental geometries to change the strain rate is perhaps a higher priority experimental focus. This is likely to shine additional light on the causes of the unexpectedly high (100) precursor, and the underlying possibility of drag-controlled plasticity. Both the forward loading and unloading (release wave) could integrate well in this regard.

A broader dataset of recovered samples would also benefit understanding. Expanding the domain of the current orientations and studying additional orientations such as (110) will likely strengthen the conclusions made here. In addition, cooling the sample pre-shot may reduce the recrystallisation after the experiment has taken place, giving results that are closer to the shocked state. Although this may alter the mechanisms of deformation, the additional data may give further insight into the higher pressure microstructure. Pairing the low temperature recovery with

conventional shock experiments in a manner similar to the work presented here would be vital to confirm the effects cooling has on the behaviour of tin.

Finally, direct *in situ* X-ray diffraction could be used to study tin under shock loading. With literature X-ray shock studies primarily limited to comparing the γ phase to as-received material, examining the shocked β phase could determine whether the observed microstructural changes in these literature studies occur before or after the phase transition. It may also be able to identify the plastic mechanism at play, especially if twinning occurs. Pump-probe laser shock experiments may offer the best route to achieve this due to the relatively high control over timing. A foil target with through-thickness grains could offer a broad distribution of quasi-single crystals.

Another possible aim of X-ray shock studies may be to examine the $\beta \rightarrow \gamma$ phase transition in order to identify the possible rearrangement mechanisms, and to examine the proposed twin relationship in γ tin. Starting from a single crystal, predictions of the possible diffraction patterns can be made and compared to experimental mechanisms to verify and evaluate the proposed mechanisms.

7.3 Concluding remarks

One of the key objectives of this work has been to better understand the shock properties tin through studying single crystals, and this work has highlighted the unusual high strain rate behaviour of tin. Below the HEL, the behaviour is highly anisotropic, with wave speeds, compressibilities and yield strengths all dependent on the single crystal orientation. The yield strengths in particular are, unusually, an order of magnitude different between the largest and smallest, themselves an order of magnitude larger than ambient measurements. Above this point, the phase transition shows only qualitative differences, while spall strength shows no dependence on either the initial sample state, or even whether the sample has undergone the phase transition.

A further objective has been to identify the microstructural changes that underpin the bulk scale measurements. Towards this, recovered samples have

been found to show a significant recrystallisation, with similar microstructures present. This has enabled a deformation pathway to be proposed, based on a twin in the high pressure phase.

Together, these point to several key mechanisms at play, governing the high-strain-rate behaviour of tin. Firstly, drag-controlled plasticity fits well the key observations regarding the onset of plasticity at the HEL and during spall. Finally, recrystallisation would explain the lack of orientation based behaviour after plasticity has activated. However, while both these mechanisms would fit well the observed behaviour, further research is required to better understand the full behaviour of each.

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