

Composite Materials for Microwave Frequency Agile Planar Devices

by

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*A thesis submitted in partial fulfilment of the requirements
for the degree of Doctor of Philosophy,
University of Oxford,
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Abstract

The potential of Calcium-Vanadium garnet loaded binary composites for use in the production of planar frequency agile microwave devices has been investigated. A WR90 rectangular waveguide system using the transmission / reflection technique has been used to compare effective medium theory predicted permittivities and permeabilities for unmagnetised and transversely magnetised composites with actual measured composite properties. Use of the Bruggeman effective medium theory with manufacturer supplied garnet permittivity and values of garnet permeability calculated using simple empirical models were demonstrated to be as accurate as predictions made using the measured properties of the composites' individual constituents. Errors in predicted material properties for unmagnetised and transversely magnetised samples relative to measured data were less than 5% across almost half of the 8.2 – 12.4GHz measurement band and within a worst-case error of 15% across the whole band. A series of end-coupled linear microstrip resonators using garnet-loaded composite substrates has been fabricated and tested. Tunabilities in resonance of up to 3.9% at 9.2GHz have been demonstrated for DC magnetic bias fields applied transverse to the microwave magnetic field component. An entirely new and previously unpublished broadband microstrip technique for the measurement of the effective permittivity and permeability of bulk gyromagnetic and gyromagnetic material loaded composite substrates subject to transverse DC magnetic bias fields is presented. This will have wide application in the design of frequency agile microwave integrated circuits.

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Chapter 1: Introduction and Background

Applications such as phased-array RADAR, frequency hopping military communications systems, local oscillators, synthesisers, filters and reconfigurable cellular communications transceivers can all benefit from using so-called frequency agile or tuneable microwave devices. As with all aspects of microwave engineering the design and realisation of frequency agile devices is inextricably linked with Maxwell's equations with the electromagnetic propagation constant being key. To produce a frequency agile microwave device control has to be exercised over the phase component of the propagation constant making it independent of frequency. This preserves the desired relationship between operating wavelength and the physical dimensions of the device. Leaving aside physically changing a device's dimensions the two principle ways to produce frequency agility are by varying either the electric permittivity or magnetic permeability.

1.1 Ferroelectric Materials

Changing the permittivity to produce a tuning action was first considered theoretically by Morgenthaler (1958) in his analysis of electromagnetic waves propagating through dielectric materials whose properties were not time independent. Morgenthaler calculated frequency modulation and phase change, demonstrating linear phase modulation in the process, when a monochromatic electromagnetic wave passed through what he termed a velocity modulator dielectric. The specific material he considered was a bulk ceramic formed from 27% Strontium Titanate (SrTiO_3) and 73% Barium Titanate (BaTiO_3) whose dielectric properties had been characterised previously by Rubin and Davis (1953). Both SrTiO_3 and BaTiO_3 belong to a class of materials known as the ferroelectrics the first example of which, Rochelle Salt, was

identified in the 1920s. As described by, amongst many, Fiedziszko et al (2002) and Balanis (1989) the positive and negative ions in a ferroelectric material can occupy two different equilibrium positions. Each of these positions is associated with a different net electrical polarisation. Applying an electric field to the material causes the ions to move to their most energetically favourable, lowest energy, position. Reversing the direction of applied electric field to return the ions to their original equilibrium positions requires in excess of a specific material dependent minimum electric field strength known as the coercive field. This results in the net material polarisation lagging behind the changing electric field strength and giving a nonlinear hysteresis curve of polarisation, hence permittivity, as a function of applied electric field strength. This behaviour occurs in the ferroelectrics' polar phase and takes place at temperatures below a material's ferroelectric Curie temperature. Above the Curie temperature the ferroelectric is provided with sufficient thermal energy from its environment so that any organised spontaneous ionic polarisation is disrupted and the material operates in the so-called paraelectric state. In this condition the ferroelectrics' relative permittivity varies with applied electric field strength according to the Curie-Weiss law. According to Gevorgian and Kollberg's (2001) review article all current and past microwave devices using ferroelectrics for tunability have done so with the material in its paraelectric state. This is to avoid the piezoelectric behaviour often encountered in polar state ferroelectrics which according to Gevorgian and Kollberg results in large losses at relatively low, less than 10GHz, microwave frequencies.

The use of paraelectric mode ferroelectrics for tuneable microwave devices has seen two relatively short periods of research activity. The first period of interest in ferroelectrics was in the 1960s following on from Morgenthaler's (1958) theoretical

investigation. Amongst the earliest of the microwave ferroelectric devices was DiDomenico and Pantell's (1962) X-band (8.2 – 12.4GHz) rectangular waveguide reciprocal phaseshifter. This was formed from a slab of bulk polycrystalline ferroelectric, 73% BaTiO₃ and 27% SrTiO₃, completely filling the transverse dimension of a section of reduced height waveguide. Impedance matching to adjoining sections of full height air-filled waveguide being achieved using dielectric slabs placed symmetrically about the ferroelectric section. The lengths of the matching sections and ferroelectric region were chosen by the authors to give a bandpass filter response from 8.7 to 9.3GHz. This was done assuming only fundamental TE₁₀ mode propagation in the waveguide enabling the whole device to be considered as a series of cascaded section of different impedance transmission-lines. The filter response could therefore be simply designed using wave cascading matrices – see Kerns and Beatty (1967) or Marks and Williams (1992). By using reduced height waveguide and splitting the ferroelectric in half using the wire supplying the DC bias voltage it proved possible to minimise electrical arcing in the device. A further precaution taken, common to all of the rectangular waveguide and cavity based ferroelectric devices, was to replace any air in the waveguide with Sulphur Hexafluoride gas due to its higher dielectric breakdown voltage. A heating coil, heatsink and thermal switch were used to try and maintain the ferroelectric section at a constant 52°C to avoid the significant problem of thermally induced permittivity and phaseshift changes.

Measurements of the device's response showed it capable of 40 - 50° of phaseshift with around 5dB of insertion loss for DC bias fields up to 20kVcm⁻¹. This performance was similar to predictions made by the authors using an analysis they developed for an arbitrary geometry ferroelectric phaseshifter. By way of

comparison the authors discussed the performance of the industry standard reciprocal ferrite phaseshifter developed by Reggia and Spencer (1957). This could provide several hundred degrees of phaseshift with 0.5dB insertion loss at input power levels up to 25W. DiDomenico and Pantell (1962) stated that while burdened by high material loss tangents, around 0.1, and limited phaseshift their ferroelectric design had similar performance to diode based designs albeit with superior power handling. The one advantage over the ferrite phaseshifter was the need for much lower power levels to achieve a tuning action. This wasn't however enough of an advantage to lead to any attempts at commercial or academic uptake of the approach for decades to come.

Amoss et al (1965) produced encapsulated 31.5% Lead Strontium Titanate variable capacitors capable of a factor of two change in capacitance from zero bias to 1kV of applied DC bias. The material was selected as the best compromise between tunability and dielectric loss tangent ($\tan \delta_e$) of 0.06 at 1.2GHz. The variable ferroelectric capacitors were used to produce two and three shunt-stub reflective switches using stripline waveguide. Comparison between measured and computer predicted switch performance, used in the design process, confirmed that 40dB of isolation and less than 1dB of insertion loss was feasible over a 10% fractional bandwidth centred at 1.2GHz. Switching speeds of 100ns and the ability to maintain switch performance without microwave E-field induced changes in material permittivity for input powers up to 0.5W with no irreversible damage at higher power levels were also demonstrated.

The change in a ferroelectric's permittivity when subject to high power microwave E-fields as well as constant DC bias conditions was used by Cohn and Eikenberg (1965)

to produce a high power limiter. Their device consisted of an adjustable short-circuit tuned coaxial cavity resonator loaded with a bulk polycrystalline 45% Lead Titanate 55% SrTiO₃ cylinder. A small heater-coil wrapped around the sample end of the cavity was used to maintain the ferroelectric in the vicinity of its Curie point. The adjustable short-circuit was used to tune the cavity resonance to 218MHz with power coupled into and out of the cavity used loops. At low input power levels the cavity was designed to work in transmission mode. For high input power levels the E-field intensity would cause a change in the ferroelectric's permittivity resulting in detuning of the cavity and the reflection of much of the input signal. Measured data showed the cavity capable of responding to peaks in input power within 10 μ s and able to deal with peak power levels in excess of 25kW. Saturated output power from the cavity for the particular sized ferroelectric cylinder used was 300W with 0.5dB of insertion loss. Cohn and Eikenberg (1965) determined the dielectric loss tangent of their ferroelectric to be 3.7×10^{-3} from measured cavity Q. A theoretical analysis of their design was said to suggest that larger ferroelectric samples would be capable of handling several megawatts of input power. Despite this the commercial microwave power limiters encountered use gases or plasmas, ferrites or PIN or Schottky diodes.

Despite these early designs confirming that it was possible to produce ferroelectric analogs of existing tuneable ferrite and garnet devices by the end of the 1960s ferroelectric devices had all but entirely disappeared from the microwave engineering research literature. Lower bias supply power consumption requirements relative to those of the magnetic devices could not make up for disadvantages of poor temperature sensitivity and high losses. The absence of published microwave ferroelectrics research, other than investigations of substituted BaTiO₃ for compact dielectric resonators, continued for almost thirty years.

The resumption of research into frequency agile ferroelectric based devices followed the discovery and development of high temperature superconductors (HTS). HTS materials, complex mixed oxide ceramics, such as Yttrium Barium Copper Oxide (YBCO) and the various stoichiometries of Thallium Barium Calcium Copper Oxide (TBCCO) can have values of microwave surface resistance up to two orders of magnitude lower than cryogenically cooled Copper. This low-loss advantage is capable of being sustained up to frequencies as high as 100GHz – see Pambianchi et al (1993), Mueller et al (1995), Jenkins et al (1997), O'Connor et al (1999). Achieving such low losses requires a good crystallographic match between the HTS and the substrate material it is grown on. This requirement and the realisation of the very similar so-called Perovskite crystal structure of both HTS and ferroelectric materials prompted a renewal of interest in the use of ferroelectrics. The start of this second period of microwave ferroelectric device research is credited by Fiedziszko et al (2002) to the work of Beall, Ono and Price (1993) on tuneable microstrip resonators. They achieved tuning of their one-port resonators' odd-order modes by varying the capacitance of a SrTiO_3 thin-film grown in the $5\mu\text{m}$ coupling gap between their microstrip lines. Up to 50V of DC bias was applied to the microstrip lines via RF blocking inductors. The resulting electric field generated in the SrTiO_3 thin-film gave 300MHz shift in the 5.6 and 11.6GHz measured resonances. Beall et al (1993) reported that the devices' tunability was independent of temperature from 4K up to 80K. Based upon measured resonator Q they set an upper limit of 0.07 on the dielectric loss tangent of their SrTiO_3 thin-films at 4K. Similar work has subsequently been reported worldwide by numerous groups using both conventional and HTS conductors including Carroll et al (1993), Chrisey et al (1993), Chakalov et al (1998) and Lee et al (1999). All four of these groups making use of various forms

of the ferroelectric Barium Strontium Titanate (BST). They all also used epitaxial thin-films of BST grown on either monocrystalline Magnesium Oxide (MgO) or Lanthanum Aluminate (LaAlO_3) substrates. By using thin-film ferroelectrics of up to $1\mu\text{m}$ thickness the DC voltages necessary to achieve the kVcm^{-1} field strengths needed for tuning were reduced down to values of 8V or lower. The exception to this being Lee et al (1999) who used up to 400V to tune their microstrip resonators. The same high bias voltages were applied in a bipolar fashion, alternate waveguide sections at $+400\text{V}$ and -400V respectively, by Subramanyam et al (2000) and Miranda et al (2000) to tune a variety of YBCO on SrTiO_3 thin-film microstrip devices including edge-coupled bandpass filters, ring resonators, a complete local oscillator and phaseshifters at frequencies from 16 to 20GHz. These devices proved capable of up to 9%, 12% and 6% tuning in centre frequency. The YBCO phaseshifter demonstrated over 360° of phaseshift at temperatures below 77K. As noted by Lancaster et al's (1998) review paper the most common transmission-line geometry used to investigate the tunability, losses and dielectric constant of thin-film ferroelectrics is the coplanar waveguide (CPW). There are several reasons for this as follows. Firstly, conventional CPW requires only a single layer of conventional of HTS conductors which simplifies fabrication. Secondly, by using very narrow gaps between the central signal carrying line and the two groundplanes running parallel to it there is a high microwave E-field across the gap. This will interact strongly with the underlying ferroelectric thin-film giving greater tuning sensitivity and additionally requiring lower DC voltages to be applied for a given DC electric bias field. This was demonstrated by Chakalov et al (1998) who measured up to 38% decrease in CPW effective BST permittivity with 40V of bias and Kim et al (2002) who demonstrated a 9% increase in the centre frequency of a two-pole CPW bandpass filter at 35V of bias.

All of the ferroelectric microwave devices regardless of waveguide type require the application of bias voltages via some form of conducting i.e. lossy set of contacts. This is far from ideal in a microwave device due to interactions with signal fringing fields and the potential for coupling of microwave signals between distant points in the device through the bias network. Achieving a uniform electric field bias across a ferroelectric is therefore extremely difficult and results in uneven and unpredictable changes in the material's complex permittivity and hence device operation. As noted by Subramanyam et al (2000) this complicates the design of tuneable microwave ferroelectric devices quite markedly as the decline in bias field with distance from the signal carrying lines is not known a priori. Additionally, current electromagnetic design tools neither calculate this static electric field or are able to take account of its impact upon the ferroelectric's permittivity assuming instead a single spatially uniform value. This is not however the most serious factor precluding ferroelectrics from being used in those applications that most critically require tunability namely very low-loss narrowband filters with steep skirts and high Q resonators for low phase noise local oscillators. As acknowledged by Gevorgian and Kollberg (2001) there has been no significant reduction in the ferroelectrics' high microwave values of $\tan \delta_e$ since they were first investigated back in the 1960s. This is confirmed by the values reported in recent years in the research literature. Lee et al (1999) measured values of $\tan \delta_e$ at 6GHz for their BST thin-films of 0.07 under zero bias and 0.015 at 100V DC bias. Chakalov et al (1998) reported 20GHz values of BST $\tan \delta_e$ of between 0.01 and 0.1 while Delprat et al (2003) measured an average value of 0.05 from 3 to 16GHz. Even at Tombak et al's (2003) 0.5GHz measurements of thin-film parallel plate capacitors gave values of $\tan \delta_e$ from 3×10^{-3} to 9×10^{-3} . By way of comparison monocrystalline LaAlO_3 , often used as the substrate for the trilayer HTS-

ferroelectric-Au / HTS devices, has a 10GHz $\tan \delta_\epsilon$ of less than 1×10^{-5} – see O'Connor et al (1999).

Attempts have been made to reduce the dielectric loss of the thin-film ferroelectrics by doping with materials such as Magnesium or Manganese – see Cole et al (2000) and Jain et al (2003). This has so far only met with limited success. Cole et al (2000) managed to produce BST doped with 5% Magnesium with a $\tan \delta_\epsilon$ of 7×10^{-3} but found that higher substitution levels merely lead to increasing losses and reduced tunability due to the formation of a mixture of different materials. Jain et al (2003) managed to slightly lower losses using a multilayer gradation in Manganese doping of BST however their $\tan \delta_\epsilon$ value of 5×10^{-3} was only measured at 1MHz.

1.2 Magnetic Materials

As already stated the other main approach to achieving frequency agility in a microwave device is to control its magnetic permeability. This is possible using the magnetic analogs of the ferroelectric materials just discussed. The two principle groups of magnetic materials of interest to the microwave engineer are the ferromagnets and ferrimagnets. In the ferromagnets on-axis spinning of unpaired electrons orbiting the materials' atoms or ions gives rise to magnetic dipole moments. These are sufficiently close to one another to cause alignment of their nearest neighbours. This process of near neighbour interaction and resulting near parallel alignment of the unpaired electron spins' magnetic moments causes regions of the material (domains) to have a spontaneous magnetisation in the absence of an applied magnetic field – see Lax and Button (1962), Chikazumi (1978), Aharoni (1998). This spontaneous magnetisation is only present at temperature below the materials' Curie temperature where thermal energy from the environment is insufficient to disrupt the electron spin coupling. A similar situation underlies the properties of the ferrimagnetic materials. The phenomenological theory developed by Néel (1932, 1936, 1948) considers the ferrimagnets as composed of two sublattices of neighbouring ions whose unpaired electron spins, and associated magnetic dipole moments, are aligned alternately antiparallel. The coupling of the dipole moments results in full (antiferromagnetic materials) or partial cancellation (ferrimagnetic materials) and consequently spontaneous magnetisations that are lower than those of the ferromagnets. As with the ferromagnets this behaviour only occurs at temperatures below a critical value called the Néel or Ferrimagnetic Curie temperature.

Applying a DC magnetic field to a ferro- or ferrimagnetic material causes the material's domains to try and align themselves with the direction of the applied field.

This gives a rise in the material's magnetisation, hence permeability, with increasing applied magnetic field strength. As with the ferroelectrics this is a nonlinear relationship with the material magnetisation obtained dependent upon the magnetic history of the sample i.e. there is a material specific hysteresis curve linking magnetisation to field strength – see Chikazumi (1978) and Aharoni (1998).

Both the ferro- and ferrimagnetic materials have anisotropic magnetic permeabilities that are expressed in the form of tensors. These anisotropic properties give rise to different phase terms and propagation constants for right and left-handed circularly polarised electromagnetic wave propagating through DC magnetised materials in the direction of the applied magnetic field. The established phenomenological analysis of ferro- / ferrimagnetic magnetisation used to derive the tensor permeability values, essentially the basis of the design of all of the magnetic microwave devices, was formulated by Polder (1949) – see chapter 2 for details. As described by Adam et al (2002) there are five key characteristics of microwave ferrites and garnets that are used to explain the operation of magnetic microwave devices. These are Faraday rotation which is the previously mentioned rotation in the polarisation of a TEM wave, expressible as the sum of two counter rotating circularly polarised waves, as it propagates through a magnetised ferro- / ferrimagnetic material. Secondly, there is the ferromagnetic or gyromagnetic resonance of a material which is the peak in the material's power absorption when an elliptically polarised microwave H-field is applied perpendicular to the direction of a DC magnetic bias field. Thirdly, there is the concept of field displacement wherein the microwave E and H-fields are displaced in the material transversely to the direction of wave propagation. This can result in higher or lower field concentrations within the ferro- / ferrimagnet and can often be manipulated using pieces of dielectric material to increase the effect of the tensor

permeability components on the microwave signal. At high microwave power levels the ferro- / ferrimagnets display a series of nonlinear properties such as the ability to amplify signals and be used as frequency doublers due to the generation of harmonic distortion. Finally, the microwave magnetic materials have an additional mode of wave propagation known as the spin wave. These are short wavelength waves of magnetisation, similar to surface acoustic waves, capable of propagating at any angle to the direction of applied DC magnetic bias field. Numerous references exist dealing with these five characteristics and their uses for example Lax and Button (1962), Morgenthaler (1988) and Adam (1988).

Much of the early systematic work on developing and characterising synthetic ferrite materials for use in, initially RF and subsequently microwave devices, was carried out by Snoek (1936, 1946, 1947, 1949) and his group at the Philips Research Laboratories in Eindhoven. The first and perhaps most influential work on devices and application for the new group of materials was carried out by Hogan (1952, 1953) at the Bell Telephone Laboratories. He produced the first microwave gyrator using a length of rectangular waveguide axially loaded with a ferrite rod and subject to a DC magnetic field directed along the direction of wave propagation. This was the first nonreciprocal microwave device and made use of Faraday rotation to give zero phaseshift for signals propagating in one direction through the waveguide and a 180° relative phaseshift for signals propagating in the reverse direction – see Collin (1992). Based upon this one device Hogan (1952, 1953) proposed a whole new class of magnetic microwave components. These were the circulator, Faraday-resonance isolator, amplitude modulator or switch and a frequency modulator all of which were based around the microwave gyrator – see Hogan (1952, 1953), LeCraw (1954), Brown et al (1958), Rizzi (1958) and Lax and Button (1962).

Hogan's work opened up a whole new field of microwave device and materials science research that was predominantly driven by the cold war military's requirements for low-loss, high power tuneable devices for use in phase-array RADAR systems. Within four years of Hogan's first paper microwave ferrites were though significant enough to warrant an entire issue of the Proceedings of the Institute of Radio Engineers to be devoted to their materials science, theory, device design and applications potential. The view was expressed that their impact upon the field of microwave engineering would be as significant as that of another recent and rapidly developing field – semiconductors. The vast number of new materials, devices and the broad frequency span of application, from 300MHz to > 300 GHz, almost fifty years on would seem to support this.

As with the ferroelectrics the ferromagnets and ferrimagnets are multi-element ceramics containing Iron and typically some other divalent element such as Manganese or Magnesium. The magnetic materials tend to have dielectric permittivities from ten to fifteen and low dielectric loss tangents – see Trans-Tech (1999) and Edoceramics (1999). Research on the mechanisms of dielectric loss in ferrites established the primary cause to be the presence of multivalent Iron ions, Fe^{2+} and Fe^{3+} , which permit excess electrons in the material to jump from one valence state to another. This provides a means of electrical conduction and hence poor dielectric losses. By careful selection of ferrite precursors and the substitution of nonmagnetic trivalent ions it is possible to obtain materials with a stoichiometry such that Iron only exists in a single Fe^{3+} state. This has resulted in commercial ferrites and garnets having dielectric loss tangents of less than 2×10^{-4} at 10GHz – see Nicolas (1980). Such values are a minimum of an order of magnitude lower than the values of $\tan \delta_{\epsilon}$

reported for ferroelectrics. Depending upon the choice of ferro- / ferrimagnetic material and its mode of operation it is possible to keep the imaginary (lossy) components of the permeability tensor sufficiently small such that the overall magnetic loss tangent is on the order of 10^{-5} – see Lax and Button (1962), Hook et al (1968), Patton (1969), Nicolas (1980), Rodrigue (1988), Adam et al (2002). The key to achieving this is use of materials with a narrow ferromagnetic resonance which over the years has meant the magnetic garnets and particularly Yttrium Iron Garnet (YIG) first discovered by Bertaut and Forrat (1956).

This combination of low dielectric and magnetic losses has lead to the development and use of YIG in many tuneable microwave devices. As described by Tanbakuchi et al (1989) small, highly polished spheres of single crystal YIG can be used to form bandpass and notch response filters operating with centre frequencies from 0.5 to 26.5GHz and capable of decade bandwidth tuning and good linearity. The basic device structure places the YIG sphere at the centre of two orthogonally arranged coupling loops formed from the inner conductors of coaxial transmission-lines. In the absence of a DC magnetic bias field there is no coupling of microwave power between the loops. Applying a DC bias field along the YIG sphere's vertical axis causes the magnetic dipoles of the material's unpaired electrons to align themselves with the direction of the applied field. By using an appropriate bias field the material's gyromagnetic resonance can be tuned to the same frequency as the filter's input signal. As described by Polder's (1949) analysis the magnetic dipoles will precess about the DC magnetisation at the microwave input frequency. The resulting circularly polarised microwave H-field couples to the output loop with a 90° phaseshift and hence transmission occurs. By having a very low-loss YIG sphere the material's gyromagnetic resonance is very narrow and so input signals differing from

the dipole precession frequency are not coupled from one port to the other and a filtering action of achieved. Tanbakuchi (1987) described how three such YIG sphere filters were used to construct the input stage of a 23GHz bandwidth automated spectrum analyser. Greene (1968) described how the same tuneable nonreciprocal filter response was achievable using microstrip line to generate the circularly polarised microwave field needed to couple to and from a magnetised YIG sphere. He achieved this by locating the sphere at the junction of an open-circuit stub connected to a single straight through microstrip line. Use of a stub an odd integer multiple of an eighth of a wavelength long enable the production of X-band (8.2 to 12.4GHz) filters. These were capable of in excess of 40dB of isolation at the design frequency and over 20dB of isolation and less than 1dB of insertion loss when tuned over a 3GHz bandwidth.

The sharp gyromagnetic resonance, Q values of 1000 to 5000, of YIG spheres has also been used to produce tuneable oscillators – see Tanbakuchi et al (1989). Papp and Koyano (1980) used a loop coupled YIG sphere as the resonator element in a FET oscillator capable of being tuned from 7.9 to 18.5GHz and with an output power of 11.8dBm. Tuneable oscillators have also been produced using YIG thin-films and bulk polycrystalline substrates. Mizunuma et al (1988) using the former approach with a Gallium-substituted YIG disc biased by a permanent magnet to give a 13GHz resonance. This was used as the resonating element in a GaAs FET common gate oscillator circuit – see Gonzalez (1997). A small wire coil was used to magnetically tune the oscillator by up to 0.5GHz with an output power of 11 ± 0.1 dBm from 13 to 13.5GHz and a sufficiently low phase noise, -93dBc at 10KHz offset, for use as the local oscillator in a Very Small Aperture Terminal (VSAT) communications system. Glance (1973) used a ferrite substrate to tune the output of an IMPATT diode

oscillator with a microstrip resonator. Changing a DC magnetic bias field applied transversely to the microwave H-field i.e. axially along the microstrip caused the ferrite substrate's effective permeability to be reduced. This enable the output signal of $25 \pm 0.5\text{dBm}$ to be tuned from 9.4 to 10.5GHz by varying the applied magnetic field from 0 to 600 Oe (47.76 kAm^{-1}). In principle, application of a sufficiently strong DC magnetic bias field to a YIG sphere filter or oscillator should enable it to be tuned well above 20GHz and into the millimetre wave bands. In practice this is not feasible due to the power levels needed to generate the required magnetic flux densities which are in excess of 15kOe (1.194 MAm^{-1}). The solution to the problem is to make use of spheres of one of the magnetoplumbite ferrimagnets, normally Barium (Hexa)Ferrite – see Went et al (1951). These have a very high internal magnetic anisotropy field which serves to self-bias the material with the equivalent of an external field strength from 0 to in excess of 30kOe (2.388 MAm^{-1}). Tuneable Hexaferrite sphere filters and mixers covering the waveguide frequency bands from 26.5 to 110GHz inclusive for high performance spectrum analysers have been produced commercially using this approach – see Nicholson (1990) and Nicholson et al (1990).

The disadvantage of the YIG and Hexaferrite sphere filters is their limited power handling ability, input powers typically less than 200mW, due to the potential for the generation of spurious higher order harmonic distortion components. Waveguide based filters containing magnetised ferrite slabs are capable of producing multi-pole bandpass filter responses with sharp skirts and low insertion losses for input powers from tens to hundreds of Watts. Snyder (1981) describes a five pole bandpass filter using sections of resonated waveguide operating below their cutoff frequency, so-called evanescent mode filters, tuned by stepped ferrite slabs placed symmetrically

against the waveguide sidewalls. His design had a centre frequency tuneable from 8.4 to 9.2GHz with reasonably constant passband width. Uher and Hoerfer's (1991) review of technologies for tuneable microwave and millimetre wave bandpass filters describes E-plane ferrite loaded rectangular waveguide designs with an average of 1.4dB of insertion loss and with centre frequency tuneable from 12.3 to 13.1GHz. Using ferrite slabs tuning speed was said to be equivalent to that of YIG sphere designs on the order of milliseconds. Using ferrite toroids to produce flux confinement and more efficient magnetisation enabled the tuning speed to be reduced to the order of microseconds.

Unlike the ferroelectric materials making use of the low losses and broadband tunability of microwave ferrites and garnets with HTS materials has not been straightforward. The reasons for this are the chemical and crystallographic incompatibility of ferrite and the HTS compounds. These problems have only been successfully overcome relatively recently using pulsed laser deposition grown thin-film buffer layers between the ferrite substrate and HTS circuit. This approach was successfully demonstrated by Piqué et al (1995) for monocrystalline YIG substrates and by Jia et al (1998) for polycrystalline material. Additional adaptations also need to be made to the device design as the DC magnetic bias fields required for tunability have a negative impact upon the low values of surface resistance of the HTS conductors – see Pambianchi et al (1993). Failure to take account of this problem and modify designs accordingly can result in no improvement in performance over cryogenically cooled Copper designs – see Denlinger et al (1992).

A number of different tuneable HTS devices have been successfully produced by researchers from MIT's Lincoln Research Laboratories using thin-film buffered

ferrite substrates. Oates et al (1997)¹ produced a tuneable YBCO stripline resonator on a monocrystalline YIG substrate with a 12GHz resonance tuneable by up to 1GHz using a DC bias field of 2kOe (159.1 kAm⁻¹) applied parallel to the direction of wave propagation. This direction of applied magnetic bias gave the greatest tunability but with the most significant deterioration in device Q as well. This problem can be overcome using the idea of DC magnetic flux confinement developed by Dionne et al (1994). They used a separate coil wound ferrite toroid pressed into contact with a Niobium low temperature superconductor (LTS) microstrip line. The magnetic bias fields were confined totally within the ferrite toroid enabling tunability without Q degradation. This idea was subsequently extended to nonreciprocal LTS and HTS microstrip meanderline phaseshifters - see Dionne et al (1996). One such LTS phaseshifter proved capable of 200° of phaseshift at 9GHz and 500° at 12GHz with less than 0.3dB of insertion loss. A YBCO design was said to give similar results. The performance of these devices was quantified in terms of the number of degrees of phaseshift per decibel of insertion loss. The LTS devices produced therefore had figures-of-merit of 1000° dB⁻¹ while Oates et al's (1997) ² YBCO meanderline grown on a LaAlO₃ substrate and placed in contact with a polycrystalline ferrite substrate achieved a peak value of 5000° dB⁻¹ in its 6 to 12GHz operating bandwidth. This compares with 130° dB⁻¹ over the same frequency span for a room temperature device produced by Hansson et al (1981). According to both Dionne et al (1996) and Oates et al (1997)² even a cryogenically cooled Copper device would be an order of magnitude poorer than their superconducting circuits. Oates and Dionne (1999) also produced tuneable YBCO half-wavelength microstrip resonators and filters. These demonstrated centre frequency tunabilities of 3% at 7.25GHz and 13% at 10GHz respectively.

The preceding reviews have demonstrated the means by which a variety of ferroelectric and ferro- / ferrimagnetic tuneable microwave devices can be produced. Aside from greater complications when used with HTS materials and higher power supply requirements to achieve tunability the magnetic devices are the obvious choice for high performance (low-loss) frequency agile microwave devices. Unfortunately, neither they nor the ferroelectrics offer the microwave engineer much flexibility. Having chosen the method and material for tunability the engineer then has to fit his or her design to its properties and ‘making do’ with whatever physical dimensions it imposes upon the device. This is obviously the reverse of the ideal situation wherein a tuneable low-loss i.e. magnetic material is required whose properties are specified by the microwave engineer to fit the application. The work detailed in the following chapters seeks to try and implement this idea by modifying the approach of Huynen et al (1999). They produced frequency agile microstrip bandstop filters using a nanoporous polymer substrate containing electrochemically deposited ferromagnetic wires of Iron, Nickel, Cobalt and an Iron-Nickel alloy. The approach adopted here is to produce a solid composite material formed from a dielectric host loaded with low-loss microwave garnet powder and use it as a substrate for the fabrication of tuneable planar microwave devices.

To this end **Chapter 2** examines the development of low-loss microwave garnet materials and the rationale behind the selection of the materials used in this research. Also discussed are the mathematical models for predicting the magnetic permeability of the selected garnets at microwave frequencies when subject to DC magnetic bias fields as well as the field of effective medium theories which will be used to predict the properties of the fabricated composites. **Chapter 3** examines the various techniques for measuring the complex permittivity and permeability of microwave

materials. **Chapter 4** discusses the microwave materials measurement system constructed and used to compare effective medium theory predictions of permittivity and permeability with the actual measured properties of the fabricated composites. **Chapter 5** uses the composites materials as substrates for half-wavelength microstrip resonators and examines their response to DC magnetic bias fields applied parallel to the direction of wave propagation. **Chapter 6** draws conclusions based on the previous chapter's experimental results and presents an entirely new and hitherto unpublished broadband microstrip measurement technique with quantifiable uncertainties for the measurement of the effective permittivity and permeability of transversely magnetised bulk gyromagnetic and gyromagnetically loaded materials.

Chapter 2: Microwave Garnets and Effective Medium Theory of Composite Materials

Development of frequency agile microwave devices requires control to be exercised over a waveguiding structure's electromagnetic propagation constant such that it remains independent of frequency. To successfully do this using magnetic material loaded composites the modelling issues and intrinsic properties of the loading material, low-loss Rare Earth Iron Garnets, are presented along with the effective medium theories that can be used to predict the complex microwave permittivity and permeability of composite materials.

2.1 Microwave Garnet Materials

Low-loss Rare Earth Iron Garnets materials belong to a group of ceramic-like mixed oxides of generic formulation $M_3Fe_2(FeO_4)_3$. M can be a nonmagnetic trivalent element such as Yttrium or Lutetium or alternatively one of the magnetic Rare-Earth elements of the periodic table from Lanthanum to Gadolinium inclusive. The most widely used of the microwave garnets is Yttrium Iron Oxide (YIG) discovered by Bertaut and Forrat (1956). This has found widespread use in tuneable devices due to its very low values of $\tan \delta_e$ typically less than 2×10^{-4} at 10GHz. This desirable property stems from the fact that the magnetic Iron ions responsible for its tunability exist solely in a trivalent form. This gives an extremely low electrical conductivity for the material as there is no possibility for electrons to be between exchanged between di- and trivalent Iron ions.

To expand the area of the electromagnetic spectrum available to ferrite devices much work over the decades since YIG's discovery has been concerned with developing low-loss ferrimagnetic garnets with similar Curie temperatures but lower saturation magnetisations than pure YIG. All of this work has examined the substitution of trivalent Iron in the garnet structure with nonmagnetic species or through the use of magnetic ions whose dipole moments partially cancel those of the Fe^{3+} ions. One of the most widely used forms of substituted YIG are the Calcium-Vanadium substituted garnets first successfully explored by Geller et al (1964). Using X-ray diffraction to determine material composition they were able to locate iteratively the exact sintering temperatures required to produce single phase polycrystalline garnets of a variety of $Ca^{2+} - V^{5+}$ substitution levels. Sensitivity to the exact sintering temperature required for a particular substitution was shown to be very high with less than ten degrees of variation about an $1165^\circ C$ optimum required to produce fully substituted

$\text{Ca}_3[\text{Fe}_2][\text{Fe}_{1.5}\text{V}_{1.5}]\text{O}_{12}$ garnet. Measurement of the various substitutions' saturation magnetisations and Curie temperature curves were said to magnetisation versus temperature curves not very different from pure YIG. Curie temperatures of Ca-V garnets surprised Geller et al (1964) by slightly exceeding or being a few tens of degrees lower than those of pure YIG. This was contrary to expectations based upon Silicon and Germanium substituted garnets. This extremely useful combination of low saturation magnetisations and high Curie temperatures lead to work to develop extremely low-loss, characterised by narrow ferromagnetic resonances, Ca-V substitutions.

Ferromagnetic resonance losses have been extensively studied over the years - see for example Schloemann (1956, 1958, 1971), Winkler et al (1972) and Nicholas (1980). According to Winkler et al (1972), ferromagnetic resonance losses can be attributed to a number of factors. These are the intrinsic loss of the material; losses due to porosity - see for example Patton (1969, 1972) and Schloemann (1998); losses due to surface roughness; losses due to conductive or polarisable impurities and losses due to the magnetic anisotropy energy of the material. This final loss term is the difference between the energy required to saturate a material along its preferential crystal plane of magnetisation relative to the energy required for saturation along its non-preferential plane see Lax and Button (1962). In a polycrystalline material with randomly oriented crystallites the anisotropy energy gives rise to a whole series of slightly different saturation magnetisations for each constituent crystallite. The net result of this is a perturbational broadening of the ferromagnetic resonance peak i.e. increased losses. Following on from successes of earlier researchers in reducing the anisotropy energy of YIG by substituting Scandium and Indium ions for Iron van Hook et al (1968) investigated the effects of variable Indium substitution levels on

Ca-V substituted YIG. They demonstrated reductions in the anisotropy energy of mono- and polycrystalline materials leading to an overall reduction in losses along with what was at the time the lowest resonance losses of a material operated at two thirds of its Curie temperature. Four years later Winkler et al (1972) carried out extensive studies on the ferromagnetic resonance losses of substituted polycrystalline YIG. To isolate the contribution of the anisotropy energy to total observed losses their samples were polished to an optical finish followed by annealing to remove grinding induced stresses which, as noted by Green et al (1968), can increase measured by losses by up to an order of magnitude. High purity precursor powders were used to prepare the samples to minimise the presence of lossy fast relaxing impurities to a minimum. Care in the ceramic processing of the powders enabled Winkler et al (1972) to keep sample porosity to negligible levels. The minimum resonance losses obtained for Indium, Zirconium and Tin substituted Ca-V garnets were demonstrated to occur approximately with the doping levels extrapolated from experiments on monocrystalline samples as giving near-zero anisotropy energy.

Calculation of the tensor components of μ_r in magnetically saturated ferrimagnets at microwave frequencies makes use of the established phenomenological model developed by Polder (1949) for lossless ferromagnets. He considered the spinning electrons orbiting the materials' atoms as spinning mechanical gyroscopes subject to the earth's gravitational force. For the case of an RF magnetic field applied perpendicularly to a DC magnetic bias Polder (1949) calculated the electrons' rate of change of angular momentum and by relating this to the torque determined expressions for the natural precession frequency (gyromagnetic resonance frequency) of a magnetic dipole subject to a constant magnetic field. Expressions for the complex tensor components were developed by Landau and Lifshitz (1935) and

Bloembergen (1950). Lax and Button (1962) describe these two phenomenological loss models as applicable to different circumstances of ferrite usage. The Landau-Lifshitz (1935) model was said to be easily incorporated into Polder's (1949) permeability expressions and represented overall magnetic losses well enough to evaluate different microwave device structures. Bloembergen's (1950) model was described as being better suited to the analysis and description of the individual factors contributing to the overall observed losses in a ferrite sample.

Polder's (1949) permeability tensor expressions while useful for predicting the performance of saturated nonreciprocal devices are inapplicable to partially magnetised ferrite devices. This is a particular shortcoming when trying to design and evaluate the performance of partially magnetised resonators as well as reciprocal phase-shifters which can be employed in their thousands in phased-array RADAR systems. It was with this application in mind that Green et al (1968) carried out research for the US airforce on the microwave properties of a number of commercially available ferrites operated in partially magnetised states. They measured the real and imaginary parts of the permeability tensor components κ and μ using cylindrical four-port TM_{110} cavity resonators. These contained a rod of the material under test that protruded from the cavity endwalls to finish just short of the poles of a large electromagnet. Under DC magnetic bias the cavities' two degenerate resonant modes split into right and left-hand circularly polarised modes which could be excited individually through the appropriate pair of ports. Measuring the two modes' resonant frequencies and Q values enable Green et al (1968) to fully characterise κ^* and μ^* using an exact solution of the wave equation for the cavity geometry - see Lax and Button (1962). Confirmation of the exact solution's results was obtained at the lower measurement frequencies using perturbation theory.

Measurements on materials with very low μ'' values, on the order of 10^{-4} or smaller, were made using two-port rectangular TE_{102} cavities. Following a measurement of the empty cavities' resonant frequency to determine their length the samples were inserted, completely filling one endwall, and the new resonant frequency and Q measured when subject to a large DC magnetic field parallel to the microwave H-field component such that μ was approximately unity. Solution of the wave equation for the cavity geometry enabled the calculation of ϵ_r^* , having first corrected for the presence of any airgaps. With the DC magnetic field removed, the sample demagnetised and the new resonant frequency and Q could be used to calculate μ^* . Green et al (1968) noted that account had to be taken in the calculations for the change in cavity wall losses and energy storage distribution brought about by the changes in μ^* . Error analysis for different ferrite slab thicknesses was said to give uncertainty in μ' of 0.1 to 0.01 which for the very low loss materials under test was said by Sandy et al (1968) to give insignificant error in μ'' .

Measurements of the real and imaginary components of μ_z were made using spherical samples centrally located in one-port rectangular TE_{102} cavities. Perturbational analysis applied to the change in reflection coefficient and resonant frequency relative to a reference DC magnetisation of 9600 Oe gave both components of μ_z^* . All three of the complex tensor components were measured as functions of DC magnetisation, saturation magnetisation (changed by sample heating) and frequency, beginning just above gyromagnetic resonance and continuing upwards to far beyond it.

Green et al (1968) demonstrated a reasonable agreement between Rado's (1953) expression for κ' of partially magnetised ferrites and their own experimental data. They then proceeded to examine the assumptions made by Rado (1953) in developing his theory and the reasons for its failure to correctly predict the frequency and magnetisation dependencies of μ and μ_z . From their experimental data Green et al (1968) presented two different empirical expressions for μ' and one for μ_z' , stressing that no physical basis for them existed. Using these expressions it was felt that reciprocal phase-shifter design using digital computers had become feasible with reasonable correlation between observed and predicted responses. Key amongst their observations was the much smaller magnitude of the loss term κ'' relative to μ'' for ratios of the gyromagnetic resonance frequency to operating frequency of 0.8 and smaller. In this region of operation Green et al (1968) stated that a single loss term, that of μ'' measured in a fully demagnetised state, was sufficient to characterise microwave materials as this was the condition for maximum loss with μ_z'' reaching a maximum value equal to μ'' over this frequency span as well. Writing one year later and based upon this statement and their experimental results Green et al (1969) presented a simple power law expression for μ'' with coefficients specific to the materials measured.

Green et al's (1968, 1969) empirical relations were employed by Massé and Pucel (1972) to predict the effective permeability, Z_0 and total losses of ferrite and garnet microstrip ring resonators measured in both demagnetised and in remanent magnetisation. To do this, use was made of Pucel and Massé's (1972) companion paper on the calculation of effective permeability for microstrip lines fabricated on magnetic substrates. Their expressions made use of a duality relationship between

the analytical expressions for effective permittivity of purely dielectric substrate microstrip produced by Wheeler (1965) and the effective permeability encountered with magnetic substrates. Pucel and Massé's (1972) justification for the duality principle began with the assumptions common to all microstrip analysis namely TEM mode propagation, ideal lossless conductors and an isotropic, homogeneous and nongyromagnetic substrate. Considering their microstrip line enclosed within a cylindrical conducting boundary, the authors pointed out that, having exchanged the normal and tangential requirements, the electric and magnetic potential function solutions to the wave equation satisfied identical sets of boundary conditions. The forms of the magnetic potential solutions were said to be identical to the electric potential solutions if the reciprocal of the magnetic permeability was substituted for all instances of the electric permittivity on condition that the surface charge and current densities were proportional. Pucel and Massé (1972) showed that having made this duality assumption, it followed that the electric and magnetic field components of the propagating wave were spatially orthogonal as expected. Considering the question of proportionality between the surface charge and current densities, it was stated that strictly speaking for an inhomogeneous system such as microstrip the assumption was invalid. The reasoning for this was that with proportionality assumed a change in substrate permittivity would change the charge distribution on line which would in turn change the surface current density and magnetic field distribution. Such changes are, however, ruled out by the other assumption of the authors' analysis, that of TEM mode propagation, whereby a dielectric material can have no influence on magnetic field distribution. From experimental work Pucel and Massé (1972) pointed out that the inductance per unit line length did not change in a noticeable way when substrate permittivity changed. It was therefore argued that the surface charge and current density were essentially

proportional and that it was the physical dimensions of the line and its substrate rather than the substrate properties that dictated the resulting capacitance and inductance per unit length.

In order for this analysis to be of practical use with gyromagnetic ferrite and garnet substrates Massé and Pucel (1972) considered microstrip lines with the DC magnetic bias field applied along the direction of wave propagation. For an assumed TEM propagation they stated that the magnetic permeability took the form of a two-dimensional tensor composed of the terms κ^* and μ^* . The empirically derived expressions of Green et al (1968, 1969) were used to calculate values of κ^* and μ^* for the two cases of demagnetised and partially magnetised, in the remanent state, substrates. The scalar permeability of the substrate, required to calculate the line's properties, was then determined from numerically curve fitted graphs of inductance per unit length for microstrip on gyromagnetic materials developed by Sandy (1971) using an extension of his work on finite difference approximations - see Sandy and Sage (1971).

Massé and Pucel (1972) measured microstrip ring resonators fabricated on a number of commercially available ferrite and garnet substrates. Measured resonant frequencies and Q were used with the average resonator circumferences to determine guide wavelengths. From the guide wavelengths the product of effective permittivity and permeability was graphed as a function of frequency, along with substrate effective permittivity which was assumed to be dispersive with a linear frequency dependence. The permittivity line was made to intersect with the product of effective permittivity and permeability at the upper measurement frequency where permeability was assumed to be approximately unity. At zero frequency the intersection of the

permittivity line with the axis was fixed to coincide with the value predicted using Wheeler's (1965) formula for microstrip on purely dielectric substrates. Using this graph the effective permeability of the microstrip lines was predicted and said to show a satisfactory match with theoretical predictions. Z_0 of the resonators was subsequently calculated from the substrate properties apparently giving values consistent with data obtained from VSWR measurements on straight microstrip line sections measured with carefully designed coaxial-microstrip transitions.

Total substrate losses were calculated by the authors from the resonators' measured Q_0 . The magnetic loss contribution was isolated from the total by subtracting the calculated metallisation losses, using Pucel et al's (1968) formulae and manufacturer supplied dielectric losses. The value obtained was itself compared with a theoretical prediction using Green et al's (1969) simple power-law model in conjunction with the Massé and Pucel (1972) microstrip theory. Good agreement between the two values was subsequently demonstrated, including the rapid rise in total losses as gyromagnetic resonance was approached due to the combination of rising magnetic losses and the increase in metallisation losses brought about by the decreasing Z_0 which is inversely proportional to skin depth. In conclusion Pucel and Massé (1972) noted the generally very good agreement between their theory and experiment. From a practical viewpoint their key recommendations were that, as the rapid rise in signal attenuation at low frequencies was unavoidable for a given material, care should be taken to choose a substrate with the lowest intrinsic loss operated as far as possible above its gyromagnetic resonant frequency.

2.2 Effective Medium Theory of Composite Materials

As described by Sihvola (1999) the development of mathematical models to predict the properties of composite materials has been an area of active research since the middle of the nineteenth century. The most widely encountered of the effective medium theories is that of Maxwell-Garnett (1904). He developed an expression to explain and predict the permittivity of glasses containing spherical particles and films of Silver, Gold, Potassium and Sodium. His model considered a single spherical dielectric inclusion subject to a uniform electric field. Assuming the absence of free charges inside or outside the inclusion, solution of Laplace's equation in spherical co-ordinates gave the relationship between the fields within the inclusion and outside in the host material - see Jackson (1999). The relationship derived can be used to calculate an effective permittivity for the composite using the volume averaged electric flux density and field strength this latter term being divided into contributions from within the inclusions and outside them in the host material. The effective permittivity of the binary composite derived by Maxwell-Garnett therefore took the following form:-

$$\epsilon_{\text{effective}} = \epsilon_{\text{host}}^* + 3V\epsilon_{\text{host}}^* \frac{\epsilon_{\text{inclusion}}^* - \epsilon_{\text{host}}^*}{\epsilon_{\text{inclusion}}^* (1 - V) + 2\epsilon_{\text{host}}^* (1 + V)} \quad (1)$$

where V is the volume fraction of inclusions determined from their density and mass as follows:-

$$V = \frac{\frac{\text{inclusion mass}}{\text{inclusion density}}}{\frac{\text{inclusion mass}}{\text{inclusion density}} + \frac{\text{host mass}}{\text{host density}}} \quad (2)$$

Using similar analysis an expression of identical form can be written for the complex permeability of a binary composite in terms of the volume weighted properties of the host and inclusions. The Maxwell-Garnett formula is numerically correct for the limit cases of no inclusions (V = 0) and no host (V = 1). The model itself though

is asymmetric giving different values of effective permittivity / permeability for fifty-percent volume fractions when the rôles of host and inclusion are exchanged see Sihvola (1999).

Bober et al (1997) investigated the applicability of the Maxwell-Garnet model to composites formed from epoxy resin loaded with either Manganese Zinc Ferrite, Strontium Ferrite, Nickel Zinc Ferrite, Barium Tetratitanate or Graphite powders. Of particular interest to them was the behaviour of the model as a function of inclusion volume fraction for cases of large differences in the host and inclusions' real permittivities and permeabilities. They postulated a limit expression for the Maxwell-Garnett model for cases of large contrast in host and inclusion properties and predicted a growing discrepancy between predicted and measured data as the limit was met. Toroidal samples of the resulting composites were machined to fit 7mm coaxial airline for measurement from 45MHz to 18GHz using the transmission / reflection technique described - see chapter 3. Transformation of the measured values of S_{11} and S_{21} into complex permittivity and permeability used either the Nicolson-Ross-Weir algorithm or the iterative approach of Baker-Jarvis et al (1990)^{1,2}. Measurements of the complex permittivity and permeability of the bulk material components of the various composites were used to provide the input data for the Maxwell-Garnett model.

Measurements of bulk sintered samples of Nickel Zinc Ferrite and Barium Tetratitanate were said to confirm manufacturer supplied permittivity data while values for the other candidate loading materials were immeasurable due to the combined problems of high real components of permittivity and airgaps between sample and coaxial conductors – see chapter 3. The composites fabricated were said

to have real components of permittivity from twelve to in excess of one hundred. Comparison of measured and predicted real permittivity data for the Nickel Zinc Ferrite for volume fractions up to 0.4 showed less than four percent difference while 0.38 volume fraction Barium Tetratitanate composites differed from predictions by ten percent. Bober et al (1997) stated that for the Barium Tetratitanate composites their limit form of the Maxwell-Garnet model was beginning to be reached. To give guidelines on the limiting relationship between host and inclusion permittivity Bober et al (1997) referred to the measured data of Dionne et al (1976) for Paraffin Wax loaded with two different forms of Titanium Dioxide – Anatase and Rutile. Measured data on both composites were said to give real components of permittivity greater than those predicted using the Maxwell-Garnett model but only the Rutile, over twice the real permittivity of Anatase, gave values larger than Bober et al's (1997) limit expression. Based on this data Bober et al (1997) stated that the limiting form of the Maxwell-Garnett model was applicable when the ratio of host to inclusion permittivity was approximately equal to 0.04. Ratios smaller than this, inclusions of higher real permittivity, were said to give no change in predicted effective permittivity using the Maxwell-Garnett model.

For the imaginary component of composite permittivity Bober et al's (1997) measurements on the Nickel Zinc Ferrite and Barium Tertratitanate composites were all lower than the predicted values independent of inclusion volume fraction. This was said to be the result of assumptions used in the derivation of the Maxwell-Garnett model namely that each inclusion experienced an identical electric field i.e. the presence of neighbouring particles was ignored. The effect of this was more visibly evident, as expected, with rising inclusion volume fraction as inclusion field interactions became more significant. The Maxwell-Garnett model predicted values of effective complex permittivity higher than that measured for the pure epoxy resin

host material a physical impossibility given that the loading materials were of lower loss than the host.

Following measurements of the real part of permeability for bulk sintered Nickel Zinc Ferrite Bober et al (1997) examined the ability of the Maxwell-Garnett model to predict composite permeability. For a ratio of host to inclusion permeability of around 0.33, below the limit established for permittivity data, the measured data followed the trend of the model's prediction with actual values lying both above and below the curve. As measurement frequency was increased towards the ferrite's gyromagnetic resonance, increasing the ratio of host to inclusion permeability, the measured data fell increasingly below the predicted values. This behaviour was contrary to that encountered with permittivity and was said by the authors to indicate the inability of the Maxwell-Garnett model to predict composite permeability. Bober et al (1997) attributed this failure to the model's assumption of identical permeability for both powdered and bulk material. To support this argument they compared the measured components of real permeability for bulk sintered ferrite and composites at two different frequencies. The frequencies selected corresponded to bulk ferrite real components of permeability less than one. Assuming identical behaviour from the powdered material would therefore have implied that increasing volume fractions of it in the composite would cause the measured permeability to decrease from the unity value of the nonmagnetic epoxy host. Such behaviour was not observed in Bober et al's (1997) measurement data. To extend the investigation to volume fractions of 0.68 the authors included Ogasawara et al's (1970) measurements on Nickel Zinc Ferrite loaded Isoprene rubber. The measured permeability was shown to continue to rise with increasing volume fraction. This was attributed by Bober et al (1997) to the difference in magnetic domain structure, see Lax and Button (1962) and Aharoni

(1998), between bulk sintered and powdered material. In the case of powdered ferrite it was demonstrated that a nonpredictable volumetric loading needed to be exceeded in order for the individual ferrite granules to agglomerate and take on a similar domain structure to the bulk material. A priori prediction of the magnetic properties of composites using the Maxwell-Garnet model was therefore said by the authors to be impossible. To try to overcome this problem Bober et al (1997) produced second order polynomial curve fits of experimental results of composites' complex permittivity and permeability. The resulting 'models' could subsequently be used to predict the properties of volumetric loadings other than those measured, as well as overcoming the permittivity ratio limit of the Maxwell-Garnett model.

Seemingly unknown to Bober et al (1997) was the modified Maxwell-Garnett model specifically for ferrite loaded composites developed and experimentally verified by Geyer et al (1994). They considered equally spaced spherical inclusions, penetrable or nonpenetrable by electric and magnetic fields, in a host material where both components of the binary composite could have complex permittivities and permeabilities. Considering an electromagnetic plane wave incident upon the composite Geyer et al (1994) developed coupled integral equations for the total electric and magnetic fields within the composite. They assumed that field components were negligibly small at the inclusion centres and that the sphere dimensions were sufficiently small for the fields to be constant across their volume. Using Lewin's (1947) analysis they considered the ferrite loaded composite as a series of excited Hertzian electric and magnetic dipole sources. The sum of the incident electric and magnetic field components as well as those terms due to scattering by all of the inclusions in a lattice, thereby departing from the Maxwell-Garnett (1904) inclusion in isolation approach, was calculated. This was subsequently used to

determine the composite's effective propagation constant, characteristic impedance and hence effective permittivity and permeability. The resulting expressions for the general case are shown below where V is once again the volume filling factor of inclusions.

$$\epsilon_{effective}^* = \epsilon_{host}^* + 3V\epsilon_{host}^* \frac{\epsilon_{inclusion,p}^* - \epsilon_{host}^*}{\epsilon_{inclusion,p}^* (1 - V) + \epsilon_{host}^* (2 + V)} \quad (v)$$

$$\mu_{effective}^* = \mu_{host}^* + 3V\mu_{host}^* \frac{\mu_{inclusion,p}^* - \mu_{host}^*}{\mu_{inclusion,p}^* (1 - V) + \mu_{host}^* (2 + V)} \quad (vi)$$

The key difference here with the conventional Maxwell-Garnett (1904) model is the complex amplitude and phase wavenumber function modified inclusion properties $\epsilon_{inclusion,p}^*$ and $\mu_{inclusion,p}^*$.

$$\epsilon_{inclusion,p}^* = \epsilon_{inclusion}^* G(k_p a) \quad (5)$$

$$\mu_{inclusion,p}^* = \mu_{inclusion}^* G(k_p a) \quad (6)$$

where k_p is equivalent to $\omega(\epsilon_{inclusion}\mu_{inclusion})^{-0.5}$. The modifying function $G(k_p, a)$ quantifies the effect of the ratio of the inclusion radius 'a' to operating wavelength on the degree of field penetration into the inclusion:-

$$G(k_p a) = \frac{2 \sin(k_p a) - k_p a \cos(k_p a)}{k_p a \cos(k_p a) + (k_p^2 a^2 - 1) \sin(k_p a)} \quad (7)$$

For the case of inclusions that are physically small relative to the operating wavelength i.e. the inclusions are totally

field penetrable then $G(|k_p a|) \cong 1$. As with the conventional Maxwell-Garnett (1904) model, the Geyer et al (1994) expressions are mathematically consistent for the cases of vanishing inclusions ($V = 0$) and vanishing host material ($V = 1$). They also share the same asymmetry of effective properties for fifty-percent volumetric loading when the host and inclusion rôles are exchanged.

Geyer et al (1994) compared the suitability of their new model with the effective medium theory of Bruggeman (1935) in predicting the properties of various compositions of Barium Tetratitanate and Magnesium Copper Zinc Ferrite. In total contrast to the Maxwell-Garnett effective medium theory the Bruggeman model for predicting composite properties has no concept of host and inclusion(s) instead treating each component identically. As describe by Sihvola (1999) the polarisations of the individual components of the composite are calculated assuming they are embedded in a homogeneous background medium with the electrical properties of the composite. Determining the effective permittivity and permeability of the composite is then a case of finding the appropriate volume fraction weightings such that the sum of the polarisations of the components of the composite is the same as the average polarisation of the effective medium. Making the same assumption of spherical components materials as the Maxwell-Garnett theory this can be expressed as equations 8 and 9 for the complex permittivity and permeability respectively of an N component composite:

$$\sum_{j=1}^N V_j \frac{\epsilon_j^* - \epsilon_{\text{effective}}^*}{\epsilon_j^* + 2\epsilon_{\text{effective}}^*} = 0$$

where V is the volume fraction of each component and $\epsilon_{\text{effective}}^*$ and $\mu_{\text{effective}}^*$ are the complex effective permittivity and permeability of the

composite. For binary composites such as those examined by Geyer et al (1994) the symmetric effective medium formulae for the complex permittivity and permeability of the composite take the forms of equations 10 and 11 respectively:

$$V_1 \frac{\epsilon_1^* - \epsilon_{effective}^*}{\epsilon_1^* + 2 \epsilon_{effective}^*} + V_2 \frac{\epsilon_2^* - \epsilon_{effective}^*}{\epsilon_2^* + 2 \epsilon_{effective}^*} = 0 \quad (10)$$

$$V_1 \frac{\mu_1^* - \mu_{effective}^*}{\mu_1^* + 2 \mu_{effective}^*} + V_2 \frac{\mu_2^* - \mu_{effective}^*}{\mu_2^* + 2 \mu_{effective}^*} = 0 \quad (11)$$

where V_1 and V_2 are the volume fractions of the composite's

constituents of known subscripted complex permittivities and permeabilities. Rearranging equations 10 and 11 the effective permittivity and permeability of the composite are given explicitly by the roots of quadratic equations 12 and 13 below:

$$\left(\epsilon_{effective}^*\right)^2 - \frac{\epsilon_{effective}^*}{2} \epsilon_1^* (2V_1 - V_2) + \epsilon_2^* (2V_2 - V_1) - \frac{\epsilon_1^* \epsilon_2^*}{2} = 0 \quad (12)$$

$$\left(\mu_{effective}^*\right)^2 - \frac{\mu_{effective}^*}{2} \mu_1^* (2V_1 - V_2) + \mu_2^* (2V_2 - V_1) - \frac{\mu_1^* \mu_2^*}{2} = 0 \quad (13)$$

As with Bober et al's (1997) work Geyer et al's (1994) input data to the Bruggeman model and their own modified Maxwell-Garnett model consisted of measured values of complex permittivity and permeability. These were obtained from two-port transmission / reflection measurements in 14mm coaxial lines from 1MHz to 2GHz - see chapter 3. Geyer et al (1994) stated that both the Bruggeman (1935) model and their own compared well with measurements on composites. For different volumetric loadings at a single measurement frequency of 100MHz the modified Maxwell-Garnet model proved closest to measured data for a composite with low Barium Tetratitanate loading when the ferrite was specified as model host. For high Barium Tetratitanate loading levels exchanging the host and inclusion rôles was found to give the best match to the measurements. For approximately equal volume filling

factors of Barium Tetratitanate and ferrite Geyer et al (1994) showed the Bruggeman (1935) model to be the closest match to experimental data. In general the authors advised that exchanging host and inclusion rôles for the two phases in the composite would give upper and lower bounds for the actual measured complex permittivity and permeability.

Another approach used to investigate the variation in a composite's complex material properties is percolation theory. As implied by the name this deals with flows and has been widely applied in disciplines as diverse as oil exploration, management of forest fires and disease control – see Stauffer and Aharoni (1994). In the context of the microwave properties of materials it is the flow of electric and magnetic flux that is of interest. In composites the formation of conjoined regions of inclusions can lead to preferential paths for flux transport. As stated by Sihvola (1999) this means that percolation is a highly nonlinear phenomenon. At a particular inclusion volume fraction, known as the percolation threshold, a very sudden and marked transition in material properties would be observed.

Youngs (2000) investigated the applicability of both effective medium and statistical percolation theories to the prediction of the complex permittivity and permeability of dielectric-conductor composites. The composites examined were formed from Paraffin Wax loaded with volume fractions, from 0 to 1 inclusive, Silver coated 15 μ m spheres. The complex permittivity of the samples was measured from 1Hz to 1MHz using a commercial dielectric spectrometer while both complex permittivity and permeability were measured from 1 - 18GHz with a VNA using the NRW equations to process two-port T / R data. Calculated material property uncertainties were said to be less than 2% for frequencies below 1MHz and less than 5% from 1MHz to

1GHz. The resulting data demonstrated large variations in permittivity and permeability with inclusion volume fraction with both real and imaginary components capable of exceeding 10^3 over the lower measurement frequencies. Comparison of 113MHz measured complex permittivity data with effective medium predictions, including both the Maxwell-Garnett and Bruggeman models, gave very marked discrepancies for inclusion volume fractions greater than 0.1. The measured data showing a very sudden and large increase in both real and imaginary components. This difference between actual and predicted behaviour was said not to be surprising owing to the assumption inherent in the effective medium theories of uncoupled inclusion polarisations and a lack of preferential paths for flux transport. For inclusion volume fractions greater than 0.18 Youngs (2000) described the composites as showing

“a frequency dependence in their imaginary permittivity which is characteristic of a material exhibiting a bulk DC conductivity mechanism.”

Comparison of the experimental data with three statistical percolation theory models, Bueche (1972), Kirkpatrick-Zallen (1983) and McLachlan (1987) gave a much better fit. Of the three models only that of Bueche (1972) attempted to calculate the composite's percolation threshold. While unable to do this correctly, underestimating the threshold volume fraction, the model gave almost exactly the same response as was measured. Both the Kirkpatrick-Zallen (1983) and McLachlan (1987) models fitted the experimental data well but needed to be supplied with the actual percolation threshold value. This inability to predict a composite's percolation threshold greatly limits the practicality of percolation theory especially to the garnet composites of this research where both host and inclusion are insulators and preferential flux paths are

unlikely to occur. For this reason no attempts were made to apply statistical percolation theory to the modelling of garnet loaded dielectric composites.

Chapter 3: Microwave Materials Characterisation

Advances in high frequency semiconductor devices and demands for more compact and heavily integrated microwave circuitry have driven research and development of planar and multilevel transmission line geometries. Coaxial and hollow metallic waveguides have consequently been replaced in many areas with stripline, microstrip or coplanar waveguide (CPW). All three technologies immerse the propagating electromagnetic fields completely or partially within a supporting substrate with dielectric and/or magnetic properties. Design and simulation of strip transmission lines of specific characteristic impedance therefore relies upon accurate complex permittivity (ϵ_r^*) and permeability (μ_r^*) data. Many techniques have been developed over the decades to supply such information each offering different combinations of accuracy, loss sensitivity, measurement bandwidth and material suitability. In essence though all of the methods fall into one of the three following groups:

Guided Wave techniques

Freespace techniques

Resonator techniques

The rest of this chapter will examine the principal methods of each of the three main approaches.

3.1 Guided Wave Techniques :-

Guided wave determination of ϵ_r^* and μ_r^* can itself be subdivided into three approaches the first of which is off-resonance two-port measurement of the S-Parameters of a filled section of coaxial line or metallic, usually rectangular, waveguide. Often referred to as the transmission/reflection (T / R) technique, the most common means of obtaining values of ϵ_r^* and μ_r^* from S-Parameter data was developed by Nicolson and Ross (1970). Their analysis of the reflected and transmitted waves assumed a single dominant propagating mode in an ideal length of waveguide filled with a perfect sample. This allowed them to combine expressions for measured S_{11} and S_{21} to give explicit solutions for both ϵ_r^* and μ_r^* . As shown in Appendix I, this requires the calculation of the natural logarithm of a complex variable. Choosing the correct solution from the infinity available requires either the measurement of two identical but different length samples or comparison of the calculated and measured group delays of a single sample see Weir (1974) and Baker-Jarvis et al (1993). Lederer (1990) worked on the assumption that starting at a sufficiently low measurement frequency, the number of half wavelengths in a physically short specimen could be predicted. As the measurement frequency increased Lederer (1990) stated that:

“it becomes easy to detect the point at which the results are no longer sensible – that is the point where the electrical length of the sample just exceeds one half-wavelength”.

When this occurs the solution of the complex natural logarithm is rotated by $2n\pi$ radians to obtain the correct solution. The so-called Nicolson-Ross-Weir (NRW) equations work well for off-resonance measurement data but completely break down for low-loss materials when there are an integer number of half-wavelengths within

the sample. This is due to an inability to accurately measure the phase of S_{11} for small values of S_{11} magnitude.

The simplest way to overcome this numerical instability is solely to use samples that are less than half a wavelength thick at the highest measurement frequency. This does lead to increased levels of uncertainty in the calculated material properties as demonstrated by an uncertainty analysis using partial differentials carried out by Baker-Jarvis et al (1990)^{1,2}. To avoid this problem they developed expressions for ϵ_r^* and μ_r^* independent of sample length and reference plane position plus a numerically stable iterative approach to solving for ϵ_r^* in nonmagnetic samples. The NRW equations are used as the basis for the start of the iterative solution with each previously calculated ϵ_r^* value used as the starting point for the next frequency point. At the time of publication a stable numerical technique for combined ϵ_r^* and μ_r^* determination was not available. Boughriet et al (1997) developed a numerically stable non-iterative approach to solving the scattering equations based on a detailed examination of the source of the numerical instability in the NRW approach. From a reformulation of the NRW equations Boughriet et al (1997) produced sample length invariant non-iterative solutions for ϵ_r^* and μ_r^* assuming a TEM mode of propagation. They also produced a general, mode independent, non-iterative solution for ϵ_r^* in non-magnetic samples. Comparison with the Baker-Jarvis et al (1990)^{1,2} iterative technique for non-magnetic samples gave comparable results. Use of Boughriet et al's (1997) non-iterative combined ϵ_r^* and μ_r^* solutions has subsequently been applied to quasi-TEM mode measurements on microstrip and CPW by Hinojosa (2001)^{1,2}. Wan and Hoorfar (1999) have reported a two-port method of explicitly determine the ϵ_r^* and μ_r^* of materials using a partial two-port calibration. Scattering parameter

measurements of the two VNA reference planes connected by a through, an empty waveguide sample holder and a filled waveguide sample holder were combined by the authors using wave-cascading matrix and error-box analysis, see Kerns and Beatty (1967) or Marks and Williams (1992), to produce explicit expressions for the complex propagation constant of the sample under test. In the case of purely dielectric materials this is equivalent to knowing the material's ϵ_r^* directly. The expressions used were shown to have none of the numerical instability of the NRW method for both real and imaginary components of ϵ_r^* even when faced with samples a half-wavelength in thickness. This was demonstrated by the authors' measurements of a nylon sample using the new technique alongside results obtained using the NRW equations. Using their technique to determine both ϵ_r^* and μ_r^* good agreement with values produced by the NRW solutions was shown, although the new technique shared the same half-wavelength instability problems. Despite this, the authors stressed the greater speed and simplicity of the new approach due its requirement for only a partial two-port calibration.

The most sophisticated solution to the need for a stable T / R numerical technique was developed by Baker Jarvis et al (1992, 1993). The NRW equations were used to obtain initial estimates of ϵ_r^* and μ_r^* for use in an orthogonal distance regression routine developed by Boggs et al (1989). This carried out an ordinary non-linear least-squares error minimisation between measured S-Parameters and values calculated using one of two pole-zero models for ϵ_r^* and μ_r^* . Using this approach systematic uncertainties in the measurement setup such as sample length, reference plane position and waveguide cutoff frequency could be detected and corrected over user specified ranges. Further advantages of the technique include usability for one-

port short-circuit line (SCL) measurements, the ability to obtain solutions using only some of the measured data for example just S_{21} or S_{11} or magnitude values only. Perhaps of greatest utility is that the material models used operate for a single fundamental mode of propagation so high ϵ_r^* samples, very likely to support evanescent higher-order modes, demonstrate none of the spurious resonant effects in ϵ_r^* and μ_r^* that are features of the NRW approach. Validation of the technique included comparisons with NRW measured samples in 7mm coaxial line as well as with data from a mode-filtered cylindrical TE_{01n} cavity. It was stated that in some of the measurements on high ϵ_r^* samples, such as Barium Titanate, the optimised least-squares approach obtained solutions where the NRW equations failed. For such materials it was noted that multiple minimal error solutions existed and choice of the correct one still relied upon a group delay comparison exactly as for the NRW method.

Fossion et al (1992) proposed a method of calibrating planar transmission lines and in the process determining wavelength and losses for lines fabricated on dispersive substrates. They achieved this by examining the Line-Reflect-Line (LRL) variant of Engen and Hoer's (1979) TRL calibration procedure. They noted that no use was made of the length difference between the two Line calibration standards and that assuming symmetrical error boxes, associated with the coaxial-planar transitions, this difference could be used to determine the error box coefficients and so fully characterise the transmission line. They also noted that for the assumption of lossless two-port transitions only a single Line standard need be used to calibrate the system. The practical implementation of Fossion et al's (1992) work involved an initial Open-Short-Load-Thru (OSLT) two-port coaxial calibration followed by S-Parameter measurements of two planar Line standards. From the Line standards data solutions

of four nonlinear equations enabled error coefficient determination and gave the attenuation coefficient and wavelength for the Line. In the case of lossless two-ports the technique involved measurement of S_{11} and S_{21} of the single Line standard. Fossion et al (1992) compared both two-port assumptions with a conventional LRL calibration using slotline standards. Differences between the conventional LRL and symmetric two-port calibration were said to be less than the actual measurement uncertainties. The assumption of lossless two-ports gave rise to a four times difference in amplitude values relative to the LRL calibration but identical phase difference to that obtained with the symmetrical two-port assumption. Comparison of the simulated performance of microstrip, slotline and CPW lines on a commercial microwave substrate with DC – 40GHz data measured using the symmetric two-port assumption showed substantial differences between manufacturer quoted ϵ_r^* and ϵ_r^* derived from measurements. With the substrate being the only common feature of the three transmission lines Fossion et al (1992) carried out an optimisation procedure to give a value of ϵ_r^* that fitted the simulated data to the experimental results. Fossion et al (1992) concluded that their symmetric two-port calibration technique was a valid alternative to the conventional LRL in non coaxial media and was capable of accurately extracting the losses and wavelength information of planar transmission lines from DC to 40GHz using two rather than four calibration standards.

Steer et al (1992) described a very similar approach to that of Fossion et al (1992) for characterising ϵ_r^* of materials used for planar transmission line substrates. Steer et al (1992) described another variant of Engen and Hoer's (1979) TRL calibration paper which was said to overcome the conventional TRL calibration's measurement uncertainties when the phase delay of the Line standard was an integer multiple of 2π radians or when there was uncertainty in the characteristic impedance of the Line

standard itself. As with Fossion et al's (1992) work the Thru-Line (TL) calibration of Steer et al (1992) began with an OSLT coaxial calibration up to the coaxial-microstrip transitions. Subsequently, it was assumed that the error boxes associated with the two transitions were symmetrical and that with a Thru connection linking them there was complete symmetry of response. Steer et al (1992) showed mathematically that in this case measurement of the Thru and Line standards enables the calculation of the measured S-Parameters of an ideal short-circuit located at the midpoint of the Thru connection. These calculated values for the known, ideal, reflective mathematical termination permitted the solution of Engen and Hoer's (1979) TRL equations essentially free from phase ambiguity. Determination of the Line standard's characteristic impedance and substrate ϵ_r^* involved the calculation of the transmission line capacitance for an air substrate using the existing Line geometry combined with its measured propagation constant. Neglecting conductor resistance and internal inductance an approximate value of effective ϵ_r^* was calculated. The high frequency asymptotic value of effective ϵ_r^* was then taken as the actual value at all measurement frequencies. Steer et al (1992) produced expressions for the frequency dependent effective ϵ_r^* and μ_r^* of nonmagnetic substrates and subsequently the characteristic impedance. Proof of the validity of their technique was demonstrated by measurements on a thin-film dielectric encapsulated microstrip line.

Janezic and Williams (1997) proposed three closely related approaches to determine ϵ_r^* of a nonmagnetic material using measurements on CPW transmission lines. In all three cases identical geometry lines were fabricated on the material under test as well as a monocrystalline Sapphire reference substrate. The propagation constants of the two sets of lines were measured using the enhanced accuracy multiple-Line TRL

calibration developed by Marks (1991). The frequency independent capacitance per unit length of the Sapphire Line standard was then calculated from measurements of its DC resistance using the approximate expression developed and experimentally verified by Williams and Marks (1991). From this capacitance value and the measured propagation constant Janezic and Williams (1997) were able to calculate the characteristic impedance of the Sapphire Lines using the technique described by Williams and Marks (1991). This involved the assumption that, due to the extremely low $\tan \delta_e$ of Sapphire, the conductance term in an RLGC lumped element model of a transmission line was negligible.

The first ϵ_r^* characterisation approach, described as the equivalent impedance method, expressed the ratio of measured propagation constants in terms of RLGC lumped elements. In addition to the assumption of zero Sapphire conductance the authors assumed that the CPW metallisations were absolutely identical. This enabled them to cancel out the resistance and inductance per unit length terms. Using the capacitance per unit length of the Sapphire Line and the ratio of propagation constants Janezic and Williams (1997) calculated the capacitance and conductance per unit length of the test material. Using Heinrich's (1993) quasi-TEM model of CPW these two values could be converted into ϵ_r^* of the test substrate. Comparison of results for a semi-insulating GaAs substrate with data at approximately 9GHz from a Kent (1988) resonator showed good ϵ_r' agreement but substantial differences in $\tan \delta_e$. At low frequencies, below about 3GHz, the equivalent impedance method gave an asymptotically decreasing value of ϵ_r' .

Janezic and Williams (1997) attributed the discrepancy in $\tan \delta_e$ and the anomalous low frequency behaviour of ϵ_r' to differences in the conductor thicknesses and geometry between the two sets of CPW. To try to overcome this flaw in their assumptions they calculated the actual resistance and inductance per unit length terms of the Sapphire CPW standards from the measured per unit length capacitance and propagation constant using the earlier work of Williams and Marks (1993). From DC resistance and geometrical measurements Janezic and Williams (1997) used Heinrich's (1993) CPW model to calculate the differences in the resistance and inductance per unit length terms between the Sapphire and test samples. Having done this the same method as previously described was used to calculate the test material's ϵ_r^* . This approach was termed the corrected equivalent impedance method and was experimentally demonstrated to be free of anomalous ϵ_r' behaviour and gave comparable $\tan \delta_e$ values to a Kent (1988) resonator for GaAs, Fused Quartz and LaAlO_3 substrates.

The third approach described made use of Williams and Marks (1992) calibration comparison technique which quantifies the differences between measured S-Parameters using any two two-port VNA calibrations. For the case of two-port TRL calibrations using identical geometry transitions and transmission lines Williams and Marks (1992) demonstrated that the difference in measured S-Parameters were due to differences in the Line standard characteristic impedance and reference plane location. In the case of CPW ϵ_r^* characterisation the difference in measured Line S-Parameters between the Sapphire and test cases results from the change in Line characteristic impedance brought about by the change in substrate ϵ_r^* . Having already calculated the characteristic impedance of their Sapphire reference Lines

Janezic and Williams (1997) used the calibration comparison expressions of Williams and Marks (1992) to calculate directly the characteristic impedance of the test material lines. From this value and the measured propagation constant Janezic and Williams (1997) could calculate the lines' equivalent capacitance and inductance per unit length and hence, via Heinrich's (1993) model, substrate ϵ_r^* . Comparison of calculated values of ϵ_r^* with data from a Kent (1988) resonator was said to show fair agreement. In conclusion Janezic and Williams (1997) highlighted the directness of this third approach and its lack of a requirement for either EM simulation or knowledge of the actual metallisation geometry. Relative to the corrected equivalent impedance method the calibration comparison method of determining ϵ_r^* was said to suffer from higher levels of random measurement uncertainty. Subsequent work by Janezic et al (2003) has applied the calibration comparison technique to multilayer thin-film microstrip transmission lines, of the sort found in RFICs, on both SiO₂ and Benzo-Cyclo-Butene (BCB) operating from 50MHz to 40GHz. Using the same techniques applied to CPW lines Janezic et al (2003) extracted the RLGC parameters of their thin-film microstrip lines. Using a commercial two-dimensional finite-difference-time-domain (FDTD) simulator they examined the relationship between substrate thin-film ϵ_r' and measured lumped element capacitance C. A polynomial curve fit was applied to the resulting data and was used to determine the substrates films' ϵ_r' from measured values of C versus frequency. Both the SiO₂ and BCB thin-films were said to show less than $\pm 5\%$ variation in ϵ_r' across the measurement band. Janezic et al (2003) reported that conductor loss was the dominant factor in the total measured losses and that ϵ_r'' of the thin-film substrates could not be isolated due to the unknown losses of the levelling / passivation SiO₂ and SiN layers involved in the multilayer structure.

The SCL technique is the longest established guided wave means of determining ϵ_r^* and μ_r^* having first been introduced by Roberts and von Hippel (1946). They used a slotted-line and diode detector to measure the VSWR of a sample containing length of short-circuited waveguide. From measured VSWR and sample length it is possible to write a transcendental equation for ϵ_r^* . This can be solved iteratively from an initial estimate of the material properties but as pointed out by Baker-Jarvis (1990) some prior knowledge of a feasible result is needed to avoid convergence at one of the infinity of other equally valid solutions. Current use of the SCL technique replaces the slotted-line with a vector network analyzer (VNA) measuring S_{11} . This enables faster, more accurate and repeatable broadband characterisation of a sample. Two approaches have been chiefly used to convert S_{11} to ϵ_r^* and μ_r^* . The first of these explicitly solves a field theory derived expression for S_{11} in terms of ϵ_r^* , see Baker-Jarvis (1990), using measurements on a single sample at two different positions relative to the short-circuit. The two sample locations that minimise uncertainty in ϵ_r^* and μ_r^* are flush with the short-circuit and a quarter of a wavelength from it. These locations coincide respectively with a region of low E-field and high H-field maximising μ_r^* sensitivity and high E-field and low H-field for maximum ϵ_r^* sensitivity. Having calculated ϵ_r^* the complex propagation constant of the sample-filled section of transmission line is used as an intermediate term to finding μ_r^* . One step in the calculation procedure does, however, involve a inverse hyperbolic tangent for which an infinite number of solutions exists. As with the NRW equations comparison of calculated and measured group delay can be used to resolve the uncertainty. A further similarity between the two methods is that the two position

SCL explicit solution for ϵ_r^* is also numerically unstable for samples an integer number of half wavelengths long.

The alternative, numerically stable, SCL approach involves measurements of S_{11} for two identical but different length samples located flush with the short-circuit. From the ratio of the measured data it is possible to solve iteratively an expression for the samples' reflection coefficient see - Baker-Jarvis (1990). Using this result the sample characteristic impedance, propagation constant and ultimately ϵ_r^* and μ_r^* can be calculated. As the analysis procedure involves a natural logarithm of a complex variable, group delay comparison is once again required to choose the correct solution. An explicit solution for ϵ_r^* and μ_r^* for two sample SCL measurements was developed by Szenrenyi et al (1988) for the special case where one sample was exactly twice the length of the other. The chief disadvantage of the two sample approach is being able to provide two identical samples of different length. A more recent approach described by Baker-Jarvis et al (1992, 1993) uses nonlinear least squares error minimisation to calculate ϵ_r^* and μ_r^* from S_{11} data. As with the two-port T / R usage this approach is numerically very stable even for high ϵ_r^* samples but in a comparison of SCL methods Baker-Jarvis et al (1993) point out the possibility of the data reduction algorithm arriving at an incorrect alternative minima solution. Group delay comparison is once again their recommended means of identifying and avoiding this eventuality.

The open-circuit coaxial probe technique has predominantly been used to measure liquid, semi-liquid and powder samples but can be applied to solid materials of interest to a microwave engineer. In all cases the open ended coaxial probe, outer

conductor flared outwards to produce a groundplane, is brought into contact with the sample and the complex reflection coefficient measured. Changing the sample backing from an open to a short-circuit places the sample in, respectively, a strong electric or magnetic field and enables both ϵ_r^* and μ_r^* to be measured. The technique has the advantages of being nondestructive, requiring no sample machining and provides broad bandwidth data to the TEM upper frequency limit of the coaxial line used. Disadvantages are several including minimal field interaction with the sample at low frequencies and the presence of both radial and axial E-field components at the probe head. This causes the calculated sample ϵ_r^* and μ_r^* to be a composite of the material's axial and radial properties which could lead to significant errors in the case of highly anisotropic materials. Secondly, and more seriously, the presence of the axial E-field makes the technique extremely sensitive to airgaps between the probe head and sample, hence the greater historical use of the approach for liquid or otherwise conformable materials.

Many reports of the technique, such as Jiang et al (1993)¹, chose to ignore the airgap problem with the result that they were able to use a very simple parallel capacitor model of the test setup to determine low-loss material properties. Jiang et al's (1993)¹ calibration of the probe head involved the determination of the parasitic fringing capacitances at the frequency of interest. This required either a graphical solution of the relationship between the measured reflection coefficient phase of a short-circuit and two reference materials, distilled water and a commercial microwave substrate, or a more involved analytic technique using an open-circuit and the two references. Following calibration, the complex reflection coefficient of several different thickness samples backed by a thick reference material sample were

measured. Unknown sample ϵ_r^* was then calculated by exponentially curve fitting the measured data to the parallel capacitor model. Jiang et al (1993)^{1,2} validated the technique and model by comparison with results obtained using Fan et al's (1990) more complicated spectral domain derived model. Jiang et al (1993)^{1,2} stated that the uncertainty of their calculated values of ϵ_r^* was $\pm 3\%$, half of which was attributable to neglect of airgap effects.

Reduction of measurement uncertainty to contributions solely from uncertainty in sample thickness and measured reflection coefficient was achieved by Baker-Jarvis et al (1994). They produced a full wave model of the probe-sample setup including airgaps for the cases of short-circuit, open-circuit and dielectric backed samples. The analysis was shown to have good agreement with coaxial probe measurements for both zero and nonzero airgaps and to give comparable results to a mode-filtered TE_{01n} cylindrical cavity. Additional benefits put forward by Baker-Jarvis et al (1994) were the ability to optimise probe geometry for a given measurement plus the calculation of both ϵ_r^* and μ_r^* from measurements on a single sample at two different airgap lengths. Later work by Baker-Jarvis et al (2001) showed the coaxial probe to give comparable ϵ_r^* for FR-4 material to both re-entrant cavity and full sheet resonance techniques but at the expense of greater measurement uncertainty.

3.2 Freespace Measurements:-

Freespace measurements of ϵ_r^* and μ_r^* offer a number of advantages over the guided wave techniques discussed. As pointed out by Lederer (1990) and Ghodgaonkar et al (1989) these include the ability to measure inhomogeneous samples free from the generation of evanescent higher order modes at air-sample interfaces, nondestructive contactless measurement and essentially no upper operating frequency limit. The two difficulties to be addressed with freespace measurements are beam diffraction at the sample edges and multiple reflections between the transmit-receive antennas and sample. A common approach to overcoming both problems is that of Ghodgaonkar et al (1989) – see figure 1. They described what is essentially a freespace variant of the SCL technique comprising measurements of S_{11} for a metal backed sample. Use of spot focussing dielectric lens horn antennas confines energy to a beam a third of the sample diameter minimising diffraction effects. The problem of multiple reflections from the sample front face was prevented using a freespace twelve term error correction, see Marks (1997), using the TRL calibration developed by Engen and Hoer (1979). This removed all system losses and impedance mismatches up to a reference plane located at the sample's front face. Ripple in the measured data due to residual source and load mismatches was reduced using time-domain gating. This resulted in S_{11} uncertainty of $\pm 0.0005\text{dB}$ and $\pm 2^\circ$ said to give a resolution in $\tan \delta_\epsilon$ down to 3×10^{-3} . Values of sample ϵ_r^* were calculated iteratively using the sample thickness and an initial estimate of ϵ_r^* by comparing measured and simulated values of S_{11} . For completely unknown materials comparison of data from two different sample thicknesses was used to determine ϵ_r^* . Ghodgaonkar et al (1989) presented data for PVC, PTFE and Fused Quartz samples showing good agreement with conventional SCL results over a 14.5 – 17.5GHz range. As with the guided wave

technique care was taken to avoid using samples an integer number of half wavelengths thick.

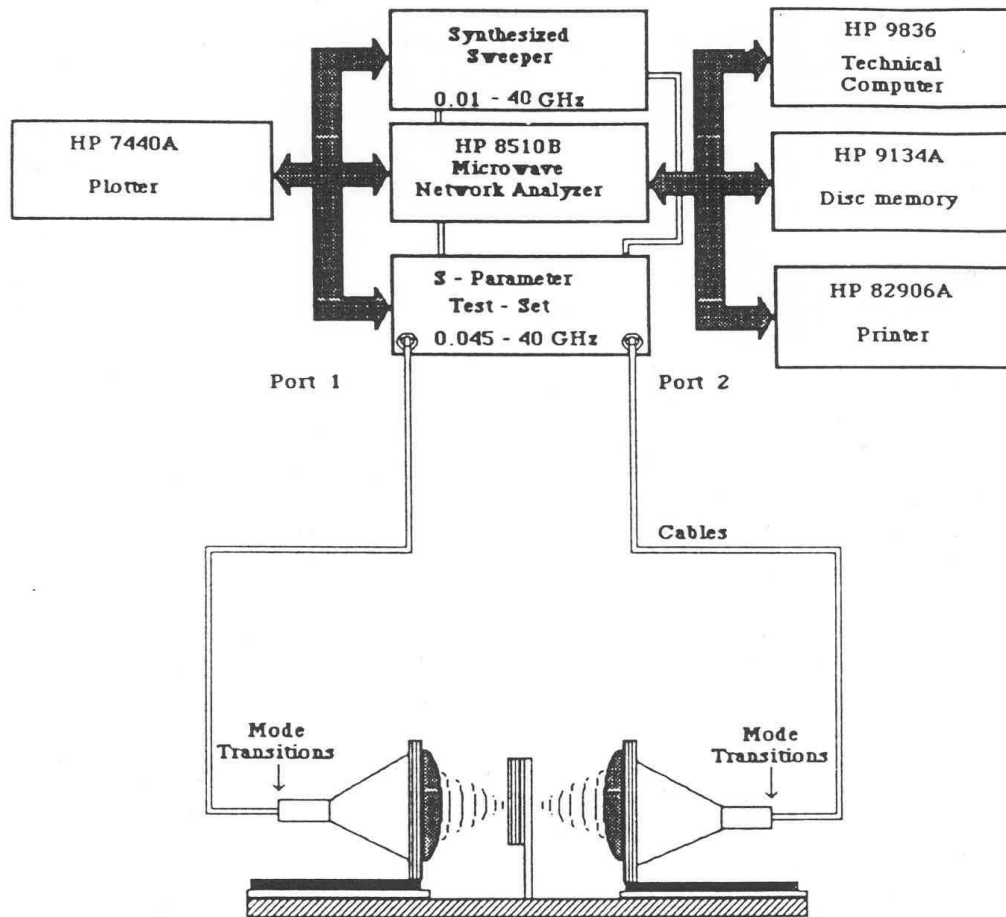


Figure 1:- Freespace Materials Measurement System - Ghodgaonkar et al(1990)

One year later Ghodgaonkar et al (1990) had modified their approach to perform freespace two-port T / R measurements of ϵ_r^* and μ_r^* . They used the same experimental setup and calibration as before to supply the S_{11} and S_{21} values for use in the NRW equations. Measurements on materials sufficiently soft and flexible as to sag under their own weight, an epoxy resin and two different commercial microwave absorbers, were carried out by sandwiching the samples between two Fused Quartz plates of half wavelength thickness at the measurement bandwidth mid-frequency.

Ghodgaonkar et al (1990) presented an analysis of the resulting multilayer structure showing how the S-Parameters of the unknown sample could be calculated from those measured for the whole composite structure. Comparison of ϵ_r^* and μ_r^* data for PTFE measured with and without the Fused Quartz plates was said to show less than $\pm 2\%$ difference. Good agreement between this new technique, their earlier freespace SCL results and data from other authors was presented. Ghodgaonkar et al (1990) pointed out the suitability of this two-port approach to high temperature measurements a fact confirmed by Varadan et al's (1991) freespace measurements of Fused Quartz and Boron Nitride from room temperature to 850°C.

A difficulty with the freespace techniques presented so far is the expense and limited measurement bandwidth available using spot focussing dielectric lens antennas. Matlacz and Palmer (2000) dealt with both issues by replacing the dielectric lens antennas with broad bandwidth ridged waveguide horns and two offset pairs of low cost parabolic satellite television antennas to provide beam focussing. The source horn antenna was then placed at the focal point of the first parabolic dish which reflected the energy via a second dish onto the sample. The sample was located at the focal point of a third parabolic dish antenna so that energy transmitted through the specimen could be focussed via the fourth parabolic dish into the mouth of the receiver ridged waveguide horn. The positioning of the transmit and receive horns was optimised as a function of the frequency response by monitoring the E-field strengths in the horn throats. Measurements of ϵ_r^* and μ_r^* for PTFE and PMMA from 2 – 18GHz confirmed a useable system bandwidth of 4 – 18GHz. The low frequency limit of the system was said to be set by the focussing ability of the parabolic dishes while the high frequency limit was a combination of effects from the ridged

waveguide horns and parabolic dishes. Matlacz and Palmer(2000) compared measurements of PTFE and PMMA samples using their new technique with a conventional dielectric lens antenna system from 11 – 17GHz and found less than 1% difference in ϵ_r^* and μ_r^* values. Comparison of measurements of 3mm and 6mm PTFE samples using the new system showed 0.43% and 1.97% differences in ϵ_r^* and μ_r^* respectively.

An alternative two-port freespace dielectric lens antenna method for measurement of ϵ_r^* is described by Campbell (1978). His approach placed the sample under test on a 360° rotating turntable between the two antennas. The sample was rotated until it was located at its Brewster angle relative to the source and the resulting reflected signal power was a minimum. From the measured angle the real component of ϵ_r^* could be calculated having first used the author's analysis of multiple reflections from a dielectric sheet at arbitrary angles of incidence to confirm that the minimum located was not due to multiple reflections within the sample destructively interfering. Sample $\tan \delta_e$ was calculated from the ratio of power transmitted with the sample aligned at its Brewster angle place relative to freespace propagation. Campbell (1978) reported measurements on PMMA, Bakelite and 96% Alumina at 35GHz with a stated uncertainty of $\pm 5\%$. The measurement uncertainties were attributed to sample roughness and warping, a spherical wavefront instead of the plane wave assumed in the reflection analysis and signal source drift and noise.

3.3 Resonator Techniques:-

As stated by Ni and Stumper (1985) in their opening remarks the cylindrical TE_{01n} cavity resonator is the reference technique for measuring ϵ_r^* at metrology laboratories throughout the world. Two variations on the approach exist – the length tuning (LTM) and the frequency tuning method (FTM) – see figure 2. Both determine ϵ_r' by iteratively solving the characteristic equation for the TE_{01n} resonant mode. The LTM does this using the measured resonant frequency for the empty cavity plus the change in cavity length needed to maintain the same frequency when the cylindrical sample is introduced. The FTM makes use of a fixed cavity length and substitutes the shift in resonant frequency between the empty and filled cavity cases for the length term see Ni and Stumper (1985) and Cook (1973). In both cases the cylindrical cavity used is typically operated in the TE_{01n} mode from 8.2 – 12.4GHz. Experimental and theoretical comparisons of the two techniques by Ni and Stumper(1985) revealed that the less commonly encountered FTM had comparable levels of loss sensitivity and uncertainty in ϵ_r' to the LTM. Uncertainty in ϵ_r' was said to be less than 7×10^{-4} based on measurements of PTFE, Polyethylene and Fused Quartz. Vanzura and Kissick's (1989) report on NIST's 60mm diameter TE_{01n} cavity gave estimated uncertainties in the ϵ_r' of six Fused Quartz samples of 0.2% with 6% uncertainty in $\tan \delta_\epsilon$ values from 9×10^{-5} to 2.7×10^{-4} . They also stated that a better knowledge of the cavity dimensions and error corrections should enable them to achieve an order of magnitude improvement in measurement uncertainties.

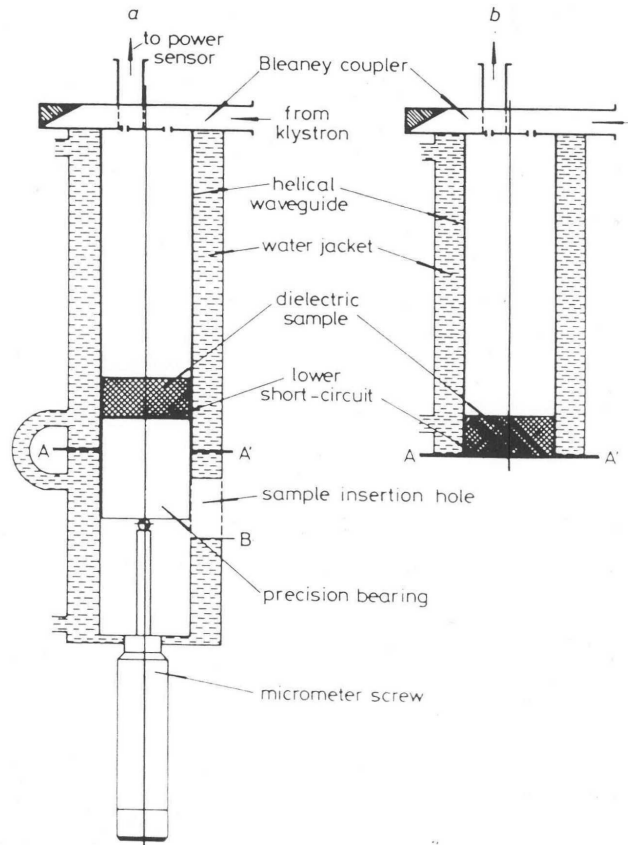


Figure 2:- LTM and FTM TE_{01n} Cavity Measurement - Ni & Stumper(1985)

Steps taken to minimise uncertainty in measured ϵ_r^* by both Ni and Stumper (1985) and Vanzura and Kissick (1989) included use of a temperature regulated water jacket around the cavity to prevent thermally induced dimensional changes in addition to the use of a precision micrometer controlled short-circuiting endplate for the LTM – see figure 3. Sensitivity to material losses was maximised by attenuating unwanted low Q resonant modes adjacent to the TE_{01n} mode by forming the cavity body from a length of helically wound circular waveguide which acts as a mode trap as described by Cook (1973). The TM_{11m} modes, degenerate with the TE_{01n} , can be suppressed by using the endplate coupling scheme developed by Bleaney, Loubser and Penrose (1947).

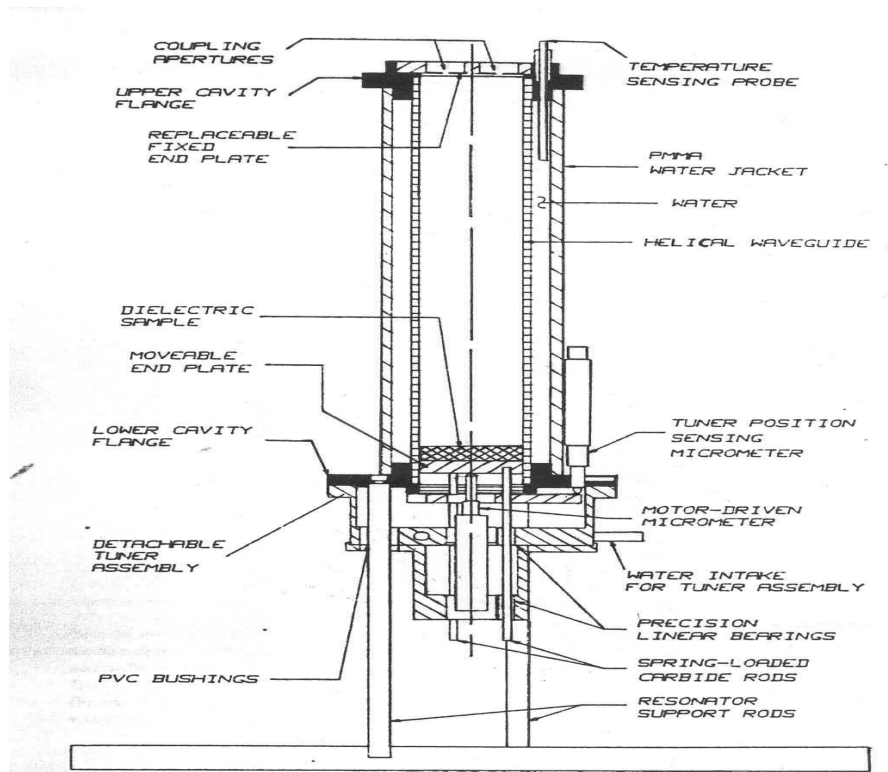


Figure 3:- NIST 60mm TE_{01n} Cavity Resonator – Vanzura & Kissick(1989)

The split-cylinder technique for ϵ_r^* measurement was developed by Kent (1988). Requiring no sample machining, this whole body resonator approach sandwiches the sample between two shorted sections of cylindrical waveguide with the sample extending out beyond the guide walls – see figure 4. Careful choice of guide dimensions enables the excitation of easily identifiable TE₀₁₁ resonances, see Snitzer (1961), in the sample over about an octave bandwidth as reported by Kent (1988, 1996¹). Measurements of the resonance centre frequency and Q enable ϵ_r' to be calculated. In so doing, the presence of radial fields within the sample, but outside the confines of the guides need to be accounted for. Axial fields outside the sample exist as rapidly decaying evanescent TE_{0n} modes below the cutoff frequency of the cylindrical waveguides.

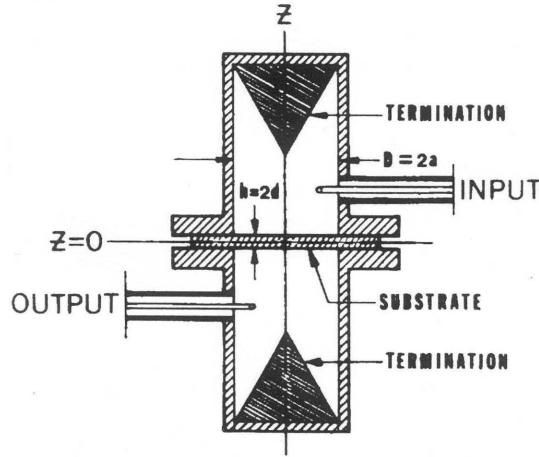


Figure 4:- Kent Dielectric Resonator – Kent (1988)

Treatment of the radial field components by perturbation analysis was performed by Kent (1988) and further refined by Kent and Bell (1996) with errors in ϵ_r' stated to be less than 1%. A full wave electromagnetic analysis of the resonator arrangement by Janezic and Baker-Jarvis (1999) further reduced the systematic errors in ϵ_r' . Their calculated values of ϵ_r' lay within the uncertainty bounds for repeat measurements of the samples using NIST's TE_{01n} mode-filtered cylindrical cavity. The contribution of guidewall losses to calculated values of $\tan \delta_e$ are negligibly small as demonstrated by Kent (1988) and Janezic and Baker-Jarvis (1999) in comparisons with cylindrical cavity measurements. This enables ϵ_r'' to be calculated simply from the reciprocal of measured Q . Kent (1988) claimed a $\tan \delta_e$ resolution of better than 1×10^{-5} at 12GHz and suitability of the technique for sample ϵ_r' from 1 – 154.

Hakki and Coleman (1960) first proposed the use of the dielectric post resonator as a high accuracy method for the measurement of ϵ_r^* and μ_r^* . The technique makes use

of a right-hand circular cylindrical rod placed between two large diameter short-circuiting Copper endplates – see figure 5.

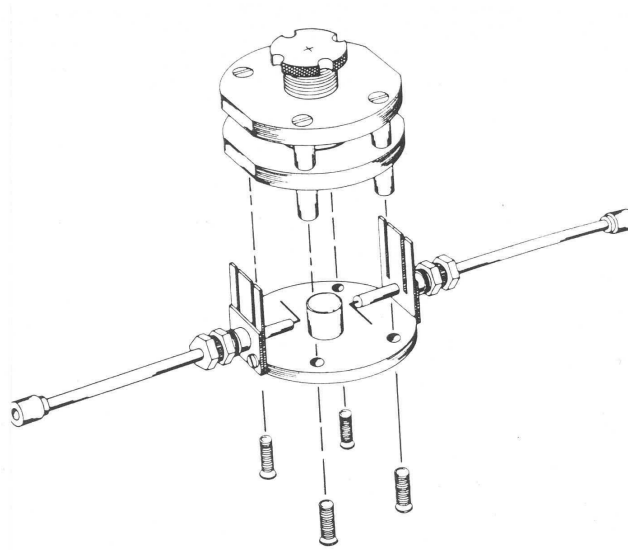


Figure 5:- Short-Circuited Dielectric Rod Resonator – Courtney (1970)

Determination of sample ϵ_r^* made use of two or more TE_{0nl} modes, the transcendental characteristic equations of which were solved graphically for a number of axial nodes in the field distribution on resonance. From these solutions a number of values of ϵ_r' were calculated and the single common value of the datasets taken as the actual sample value. Confirmation of the calculated ϵ_r' was obtained by using this value to design a resonator and comparing the measured and predicted resonant frequencies. Having done this Hakki and Coleman (1960) calculated $\tan \delta_e$ from expressions for the field distribution both inside and outside the sample, measured Q and an assumed value for the conductivity of the short-circuiting endplates. Determination of μ_r^* took an identical approach but made use of the solution of the characteristic equation for two or more TM_{0nl} modes. In either the dielectric or magnetic cases Hakki and Coleman (1960) gave an accuracy in the real components of $\pm 0.1\%$ limited by the

sensitivity of their wavemeter and the precision with which sample dimensions could be measured. Uncertainty in the values of $\tan \delta_e$ for a PTFE sample were quoted as $\pm 10\%$ for a value of 1×10^{-3} limited by their non heterodyne measurement arrangement.

Ten years later Courtney (1970) revisited the dielectric rod resonator technique determining likely accuracies and suggesting a means to improve the measurement of the μ_r^* of unmagnetised microwave ferrites operated far from their gyromagnetic resonance frequency. This two step procedure first operated the cylindrical sample in the TE_{0n1} mode, said to be more easily identifiable than the TM_{0n1} mode, by subjecting the sample to a sufficiently large DC magnetic field to cause saturation. This reduces the magnetic losses to all but zero and enables the calculation of sample ϵ_r^* exactly as for a purely dielectric sample. The second step involved demagnetising the sample and measuring the new resonant frequency and Q for the same TE_{0n1} mode as before. From the new data and the precisely calculated value of ϵ_r^* the μ_r^* of the sample could be calculated. Comparisons between Courtney's (1970) approach and the work other authors indicated an accuracy in ϵ_r' of better than 0.3% *"for tolerances of ± 0.0005 inch in D and L and the frequency measured to the nearest MHz"*. A theoretical comparison between Courtney's (1970) approach and the recommended cavity perturbation technique of the American Society for Testing Materials (ASTM) suggested a factor of fifty times the measurable change in resonant frequency between the sample and sample-free cases for Courtney's (1970) approach. For the case of an assumed $\tan \delta_e$ of 10^{-4} Courtney (1970) calculated that measuring the resonant frequency with less than 0.1MHz uncertainty combined with a 30% uncertainty in the assumed endplate conductivity contributed less than 0.5×10^{-5} total error in measured

$\tan \delta_e$. He speculated that ongoing work to develop very low-loss dielectrics would eventually provide a means to accurately measure the endwall conductivity. A similar analysis of the errors when measuring low-loss, $\tan \delta_\mu$ less than about 10^{-5} , garnet samples suggested better than 1% accuracy for both components of μ_r^* with sensitivity to loss components on the order of 10^{-6} . This higher sensitivity than for the pure dielectrics is a result of the endplate conductivity playing no part in the calculation of μ_r^* values.

As already mentioned, Courtney's (1970) analysis attributed the majority of the uncertainty in measured $\tan \delta_e$ for low-loss, $\tan \delta_e$ less than 10^{-4} , samples to uncertainty in the actual conductivity of the short-circuiting resonator endplates. By modifying the rod-resonator technique to use two samples Kobayashi and Katoh (1985) developed a means of measuring the actual endplate conductivity and so greatly improved the accuracy of $\tan \delta_e$ measurement. The dimensions of the two samples used were chosen, using expressions derived by Kobayashi and Tanaka (1980), so as to give TE_{011} and TE_{01n} ($n \geq 2$) resonances at the same nominal frequency and with good separation from surrounding lower Q modes. From the analytic expressions for a TE_{01n} mode, actual sample dimensions, measured Q and resonant frequencies simultaneous equations were produced from which actual endplate conductivity (σ), surface resistance (R_s) and sample $\tan \delta_e$ could be calculated independently of each other. Combining experimental data with first order approximations to the mean square errors in measured values of ϵ_r' , $\tan \delta_e$, R_s and σ Kobayashi and Katoh (1985) gave accuracies for their technique of $\epsilon_r' \pm 0.1\%$, $R_s \pm 1.5\%$, $\sigma \pm 3\%$, $\tan \delta_e \approx 10^{-4} \pm 1.2\%$ and $\tan \delta_e \approx 10^{-5} \pm 12\%$ with resolution in $\tan \delta_e$ of

10^{-6} . The validity of first order approximations for calculating the temperature dependence of ϵ_r^* was demonstrated by experimental results along with an analysis of the impact of using finite diameter endplates on measurement accuracy. Further improvements in $\tan \delta_e$ sensitivity to values of 7×10^{-6} and lower are feasible by measuring at cryogenic temperatures using high temperature superconducting endplates. Using this approach Geyer and Krupka (1995) measured $\tan \delta_e$ and ϵ_r' for anisotropic single crystal Quartz, PTFE and cross-linked Polystyrene at a temperature of 77K. To obtain the parallel ϵ_z tensor component they used the hybrid HE_{111} and HE_{211} modes they stated accuracies for the approach of 0.3% for ϵ_r and 1 – 2% for ϵ_z .

An alternative whole body resonator technique capable of measuring ϵ_r^* as well as scalar and tensor values of μ_r^* for low-loss ferrites, was developed by Krupka (1991). The approach, extended to work from 2 – 25GHz by Krupka and Geyer (1996), used a single cylindrical ferrite sample with two low-loss dielectric ring resonators – see figure 6.

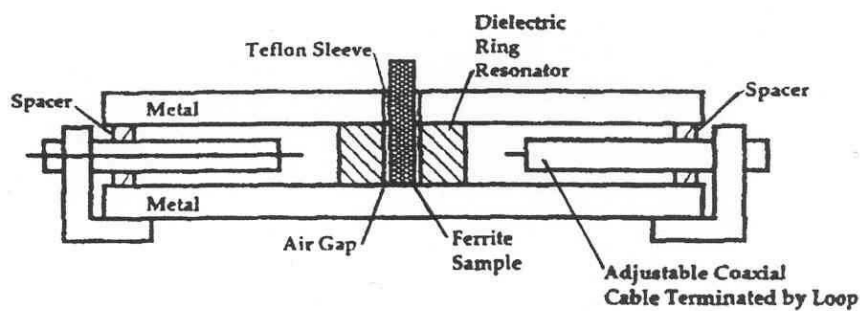


Figure 6:- Ring Resonator Measurement of Ferrites – Krupka & Geyer (1996)

The two rings shared a common internal diameter but different external dimensions so as to resonate in the TE_{011} and $HE_{111}^{\pm}$ modes - see Snitzer (1960), at the same nominal

frequency. Solving the characteristic equations for the modes for air-filled rings using measured Q s and frequencies gave the values of ϵ_r^* for the two rings. Using this information and the remeasured Q and frequencies for completely demagnetised ferrite-filled rings the characteristic mode equations could be resolved to give the values of ϵ_r^* and scalar μ_r^* for the ferrite. Using the scalar magnetic properties the tensor values could be determined by repeating the measurements with different levels of applied DC magnetic field strength.

This is feasible as a result of ferrite materials' different response to left and right circularly polarised microwave signals when subject to a DC magnetic bias field - see Lax and Button (1962). Practically this involved measurement of the two HE_{111} mode variants, left and right circular polarisations, in addition to the TE_{011} using different pairs of four orthogonally oriented coaxial probes. Differences in resonant frequency of the $HE_{111}^{\pm}$ and TE_{011} modes were accounted for in the calculated tensor values of μ_r^* using the empirically derived frequency dependencies of Sandy and Green (1974)^{1,2}. Actual corrections required were said by Krupka (1991) to be largely insignificant due to resonant frequency differences of no more than 10%.

Krupka (1991) confirmed the accuracy of the scalar value of μ_r' by measuring both saturated magnetic samples as well as purely dielectric materials. In both cases calculated values of μ_r' differed from the expected unity value by less than 0.15%. A more rigorous uncertainty analysis on Krupka and Geyer's (1996) extended frequency version of the technique gave the uncertainty in the scalar value of μ_r' as $\pm 0.8\%$ with a resolution in the imaginary term down to 2×10^{-5} with $\pm 1 \times 10^{-5}$ uncertainty. Comparisons of the tensor values with data obtained using Krupka's (1989) work with

solid cylindrical ferrite resonators showed differences of less than 1% in real components and less than 15% in imaginary terms. Both Krupka (1991) and Krupka and Geyer (1996) demonstrated the technique's suitability as an alternative to the IEC's proposed TM_{110} cavity perturbation technique on grounds of higher magnetic loss sensitivity for a given sample size, smaller actual sample requirements, and a high degree of measurement repeatability due to the lack of insertion holes in the short-circuiting endplates and subsequent modal insensitivity to the quality of contact made at these plates.

As measurement frequencies rise, so too do the inherent difficulties in performing accurate and low-loss sensitive measurements of ϵ_r^* and μ_r^* . This is a result of the diminishing cavity or sample dimensions required for a particular resonant frequency as well as increased sensitivity to dimensional inaccuracies and airgaps. Cavity resonators suffer the additional problem of falling Q values leading to greater uncertainty and poorer sensitivity to low values of $\tan \delta$. Two techniques commonly employed to overcome these problems as measurement frequencies rise into the millimetre and submillimetre bands are the Fabry-Perot open resonator and the Whispering Gallery Mode (WGM) resonator. Of these two the Fabry-Perot resonator is the longest established having been first used to determine values of ϵ_r^* by Culshaw and Anderson (1962). As with all variations of the technique, they used two opposed metallic mirrors with the dielectric sample located in-between. As a result of their choice of parallel plane mirrors Culshaw and Anderson (1962) were unable to take full advantage of the high Q values available from the sidewall-free open resonator due to diffraction losses. Subsequent work has always used either a semi-confocal or fully confocal arrangement of spherical mirrors to make the diffraction losses negligible – see figure 7. This has enabled the production of open

resonators with Q values of 10^5 at frequencies of 100GHz and more as described by Komiyama et al (1991).

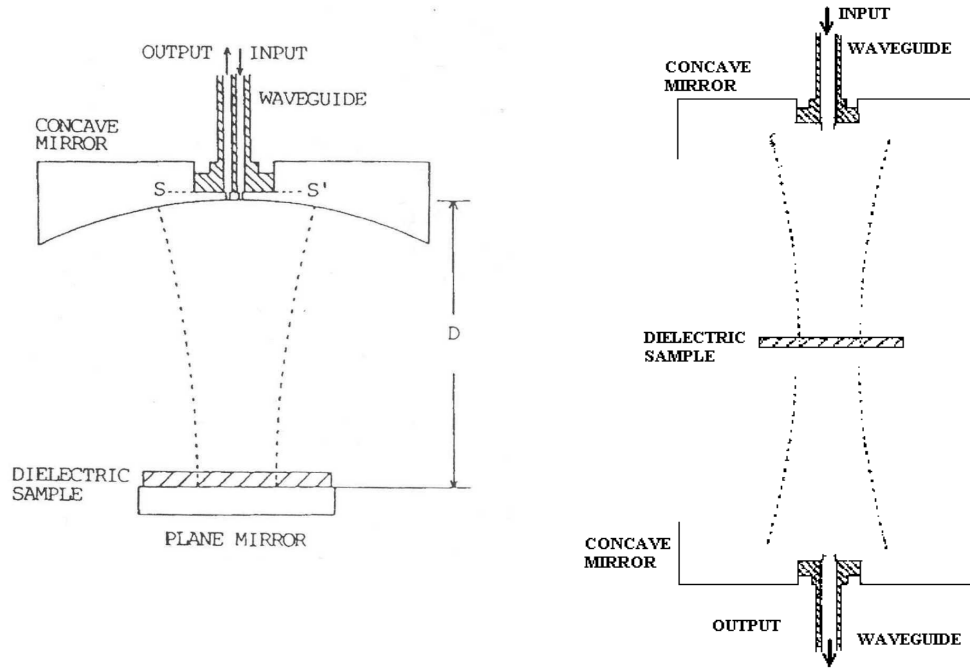


Figure 7:- Semi-Confocal and Confocal Fabry-Perot Resonators – Komiyama et al (1991)

In the introduction to their paper Cullen and Yu (1971) noted that the full and semi-confocal resonators avoid the difficulties in electromagnetic analysis of plane mirrors, instead permitting use of the much simpler Gaussian Beam theory of Kogelnik and Li (1966). Starting from this basis and making the simplifying assumption of a sample of identical spherical profile to the resonator mirrors Cullen and Yu (1971) produced expressions for the odd and even order TEM resonant modes supported. They applied a perturbation theory derived correction to measured resonant frequencies, accounting for the use of planar samples, before iteratively solving the appropriate transcendental mode equation for sample ϵ_r^* . Doing this they presented data for Polystyrene and PMMA measured at 10GHz in a fully confocal resonator. Following

detailed error analysis they gave uncertainty bounds of $\pm 0.25\%$ for ϵ_r' and $\pm 28\%$ and $\pm 6\%$ for the $\tan \delta_e$ of Polystyrene and PMMA respectively. At the time of writing an order of magnitude improvement in $\tan \delta_e$ accuracy was thought to be possible through a combination of modified signal coupling and use of high conductivity Copper rather than Duralumin mirrors. Writing eleven years later Yu and Cullen (1982) presented a more accurate analysis of the open resonator. Working from an exact solution to Maxwell's equations they produced expressions for the TEM resonant frequencies with easily quantifiable errors, the so-called vector theory. This vector theory analysis has replaced their previous, approximate wave equation, scalar theory. Experimental validation of the vector theory analysis was provided in a companion paper by Lynch (1982). In it he demonstrated ϵ_r^* uncertainty values of less than 1% for non optimal combinations of frequency, sample thickness and ϵ_r' .

Whichever mathematical analysis of the open resonator is used the experimental procedures can take two forms. These are the fixed frequency or fixed length approaches and operate on the same basis as the two approaches already mentioned in relation to the mode-filtered cylindrical cavity resonator. A third so called frequency-length hybrid is described by Afsar et al (1990). This uses the fixed frequency technique of a swept mirror separation to locate and identify the various modes present in the empty and sample-filled cavity. The modes of interest are then measured using the fixed frequency approach in order to calculate empty and sample-filled Q values. Iterative solution of the appropriate transcendental mode equation gives ϵ_r' while $\tan \delta_e$ is calculated from the change in Q between the lossy sample-filled case and that of the resonator filled with an identical ϵ_r' lossless sample. The three techniques all have advantages and disadvantages. The fixed frequency

approach does away with the need for stable, broadband, fine tuneable signal sources which are far from trivial to produce as operating frequency increases. This offers the potential for improved sensitivity to low values of $\tan \delta_e$ due to an improvement in SNR brought about by the absence of complicated electronic tuning systems. It does, however, come at the expense, literally, of requiring a means of repeatably adjusting the mirror separation in a finely stepped manner over a distance of several tens of millimetres. As the signal wavelength can be of the order of millimetres, so submicron variability in mirror separation is required. The fixed length technique advantages and disadvantages are exactly the converse of those just presented; i.e. mechanical robustness is traded for increased complexity hence cost of the signal source. On the face of it the hybrid approach would seem to offer the best and worst of both approaches. The results presented using any of the three approaches do, however, seem to be fairly comparable. This was demonstrated by Afsar et al's (1999) fixed frequency development of an earlier 60GHz fixed length / hybrid system see Afsar et al (1990). The more recent fixed frequency system used a 20nm resolution micrometer stage to vary the length of a semiconfocal mirror arrangement by up to 25mm. This was said to give improved SNR and better $\tan \delta_e$ resolution over the earlier work which was itself capable of resolving values of $\tan \delta_e$ of order 10^{-5} with $\pm 10\%$ uncertainty. Support in favour of the fixed length system is provided by Komiyama et al (1991) who achieved levels of ϵ_r' uncertainty of less than $\pm 0.1\%$ with $\pm 10\%$ uncertainty in $\tan \delta_e$ of order 10^{-5} at frequencies in excess of 100GHz.

The second resonator technique applicable to ϵ_r^* and μ_r^* determination at high (10^+GHz) frequencies is the already mentioned whole body WGM resonator. First examined as an acoustic phenomenon, Rayleigh (1914), WGMs take the form of

surface electromagnetic waves propagating around the concave surfaces of normally cylindrical samples. Field confinement is within a thin surface layer of the material due to reflections from the air-dielectric interface at the sample circumference. By suitable choice of resonant mode, one of high azimuthal wave number, field radiation from the sample can be made negligible. As noted by Vedrenne and Arnaud (1980) the resulting Q is independent of any enclosing metallic walls and is determined solely by the intrinsic material losses. This overcomes the decreasing Q and loss sensitivity with frequency of cavity techniques and makes the WGM resonator especially sensitive to low values of $\tan \delta$. This was confirmed by Braginsky et al's (1987) ability to resolve values of $\tan \delta_e$ of 10^{-9} for cryogenic monocrystalline Sapphire samples. As already mentioned, WGMs are based on radial field confinement and have only minimal axial dependence, hence resonant frequencies are functions of sample radius and material properties. For a given resonant frequency this makes WGM resonators physically larger than other whole body techniques enabling their use at frequencies up to 100GHz as described by Krupka et al (1994).

WGM mode resonators have been used for the determination of both ϵ_r^* and μ_r^* in isotropic and anisotropic materials. Due to the hybrid nature of the WGM, taking the form of either quasi-TE or quasi-TM field distributions, numerical simulations of the sample under test are an essential part of the measurement technique. Work in this area by Krupka et al (1989, 1994, 1996, 1999) has involved the use of the Rayleigh-Ritz, Finite Element and Radial Mode Matching approaches to analysing the electromagnetic field distributions. For anisotropic ϵ_r^* determination Krupka et al (1999) adopted a five step procedure. First, WGM resonant frequencies were calculated using ϵ_r^* data from literature and other measurement techniques by solving

the eigenvalue equations for the quasi-TM and TE modes. The resulting resonant frequencies were then compared with actual measured values from the sample under test to identify specific modes. A pair of quasi-TM and TE modes were then selected and the sample dimensions used to calculate the values of ϵ_r^* parallel and perpendicular to the anisotropy axis. The values of ϵ_r^* calculated were then used to recalculate the sample's WGM resonances by way of confirmation. Having done this, the field confinement of the modes could be determined and used to calculate sample $\tan \delta_\epsilon$ from sample Q. Krupka et al (1999) reported that calculated and measured resonant frequencies for a monocrystalline Sapphire sample differed by less than 0.01% with a total uncertainty in ϵ_r^* of 0.05% attributed largely to uncertainty in sample dimensions. The authors cautioned that the quasi-TM and TE modes are at nominally offset frequencies and that precision of sample machining is essential in order to preserve the mode splitting and make measurement possible at all.

In the same work Krupka et al (1999) also measured the isotropic ϵ_r^* of an unspecified low-loss ceramic. With no tensor values to determine the measurement data for a single WGM was sufficient to solve the eigenvalue equation for ϵ_r^* . Results obtained using this approach were said to compare favourably with data from a dielectric rod $TE_{01\delta}$ resonator. An additional benefit of the technique as a whole highlighted by Krupka et al (1999) is the ability to provide broadband ϵ_r^* data from a single sample by appropriate choice of mode. In the case of the low-loss ceramic used this was true over an octave bandwidth.

WGM resonators have also been applied to the measurement of the tensor μ_r^* of ferrites. Krupka et al (1996) performed both Rayleigh-Ritz and Finite Element

electromagnetic simulations of WGM modes for cylindrical ferrite samples under uniform axial DC magnetic bias. In doing so they demonstrated the much greater sensitivity of the quasi-TM modes to changes in the κ and μ tensor terms and the quasi-TE modes' sensitivity to changes in μ_z . They also made comparisons with measurements of the impact of variations in radial magnetisation upon the resonant frequencies of circularly polarised quasi-TM and TE modes. As a result the authors proposed a means of measuring κ and μ from measurements of right and left circularly polarised quasi-TM modes. The measured resonant frequencies and sample dimensions used to solve iteratively eigenvalue equations for κ and μ . Having obtained these two parameters μ_z could be found from the iterative solution of the eigenvalue equation for a quasi-TE mode using the average resonant frequency of right and left circularly polarised forms with the same azimuthal wave number. Comparison between the authors' results for a polycrystalline YIG sample at 21.5 and 25GHz with published data from lower frequency techniques was said to show similar trends in magnetic bias field dependence of the tensor components. Magnetic losses for a partially magnetised low saturation magnetisation material, such as YIG, were said to be unmeasurable. When biased into saturation Krupka et al (1996) reported a value of $\tan \delta_\mu$ for their YIG specimen of 2×10^{-4} at 25GHz. As with work on dielectric materials, Krupka et al's(1996) work highlighted the sensitivity to magnetic losses of the WGM approach, practical dimensions of test samples even at 100GHz and the ability to fully determine all tensor μ_r^* components at a number of frequencies from a single sample.

Chapter 4: Waveguide Measurements of Materials

Detailed below are the design, construction and usage of a WR90 rectangular waveguide system for the measurement of solid material samples at X-band. Verification of the system's correct implementation of two-port transmission / reflection (T / R) material measurement using the Nicolson-Ross-Weir (NRW) equations is demonstrated by comparison of measurement data for standard laboratory materials Polytetrafluoroethene, Polymethylmethacralate and Air with data from published literature. Qualitative confirmation of high levels of accuracy in the measurement of the real components of permittivity and permeability is given by comparison with rod-resonator measurements of TE_{01n} mode Polytetrafluoroethene cylinders. Shortcomings of the T / R system are discussed before the system is used to characterise the dielectric host and garnet materials to be used to form binary composites. Comparisons are made between the T / R measured garnet properties and values provided by the manufacturer and calculated using empirically derived models. The T / R setup is then used to investigate the ability of the Bruggeman and NIST-modified Maxwell-Garnett effective medium theories to predict the real components of permittivity and permeability of two different volume fractions of magnetised and unmagnetised microwave garnet loaded composites.

4.1 WR90 Waveguide Experimental Setup

Measurements of pure host dielectric, garnet and composite material samples' complex permittivity and permeability used a combination of techniques. Broadband, loss-insensitive, two-port T / R measurements using the NRW equations were combined with low-loss sensitive single frequency data obtained using Kobayashi and Katoh's (1985) rod-resonator method. The T / R measurements were made in precision WR90 rectangular waveguide from 8.2 – 12.4GHz (X-band). Each coaxial-waveguide transition was joined to the sample test-cell by a 77.0 ± 0.05 mm long section of guide – see figure 8.



Figure 8:- WR90 Transmission / Reflection test setup

These sections, two freespace wavelengths long at 7.7868GHz, acted as mode filters to remove any high order evanescent modes generated by the transition probes. This ensured that only the fundamental TE_{10} mode interacted with the material samples – see Baker-Jarvis et al (1993). Two 2mm diameter holes for ground Silver Steel alignment dowels were drilled into the flanges of the mode filters, calibration standards and test-cells using a purpose built jig. The alignment dowels were used when making connections to prevent flange misalignment or rotation. This improved

the overall calibration return loss and repeatability and went some way to reducing the performance gap between the quick-fit, threaded ring joined, NATO flanges available and the precision versions of IEC UBR100 flanges found in commercial calibration kits – see for example Flann (1998) and Maury (1976, 1978)

Engen and Hoer's (1979) TRL calibration technique was used to establish the mode filter flanges as the measurement reference planes carrying out a full twelve term error model correction of the VNA in the process – see Marks (1997) and Rytting (2001). A calibration kit comprising a WR90 quarter wavelength line section and two flush mounting short-circuits were machined from precision Brass waveguide and Brass stock respectively. The quarter wavelength line was designed for the geometric mean frequency of the WR90 band in accordance with the VNA manufacturer's guidelines – see Agilent(2000). Calibration and sample measurements were both made at 801 equally spaced points across the full WR90 band. Each recorded datapoint was the average of 512 measurements to reduce the effects of system noise. In order to ensure consistency between calibration and sample measurements the VNA was configured to phase-lock with its internal crystal oscillator reference at each measurement point rather than solely at the start and stop frequencies.

Attempts were made to employ both 7mm coaxial airline and WR90 rectangular waveguide for T / R measurements from 2.25 to 18GHz. The coaxial airline was fabricated according to Lederer's (1990) design taking the form shown in figure 9.

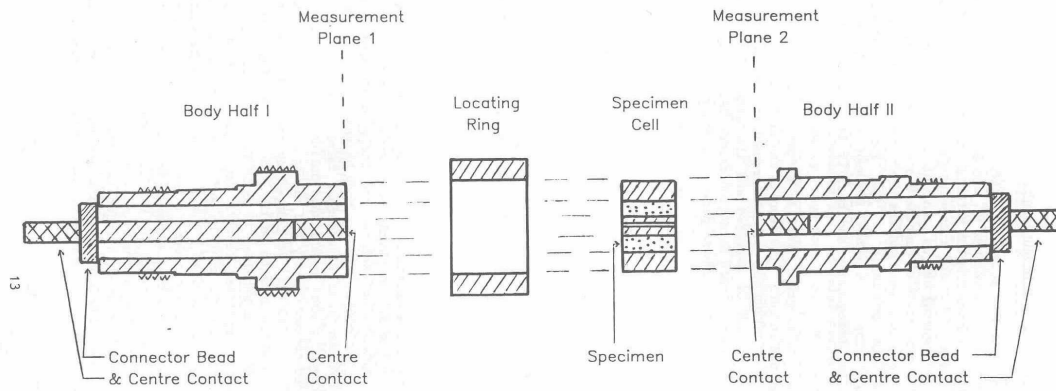


Figure 9:- 7mm Coaxial Airline Design

The beaded airline coaxial connectors available differed slightly from those employed by Lederer in that the centre conductor contact collets were fixed rather than spring loaded. The fixed contacts were removed and a new set of alignment pins, replacing Lederer's design, machined. Lacking the small degree of axial travel found in the spring-loaded collets any excess torque applied when joining the two halves of the airline, during calibration or sample measurement, could force the calibration reference planes apart to reveal part of the alignment pin. The resulting step change in centre conductor diameter manifested itself in poor quality calibrations, low return loss due to impedance mismatch, and a lack of connection repeatability. NRW processed S-Parameters from an air-filled test-cell demonstrate this as non unity frequency dependent real components of permittivity and permeability – see figures 10 and 11. For this reason the airline was not employed to measure the composites or their individual components.

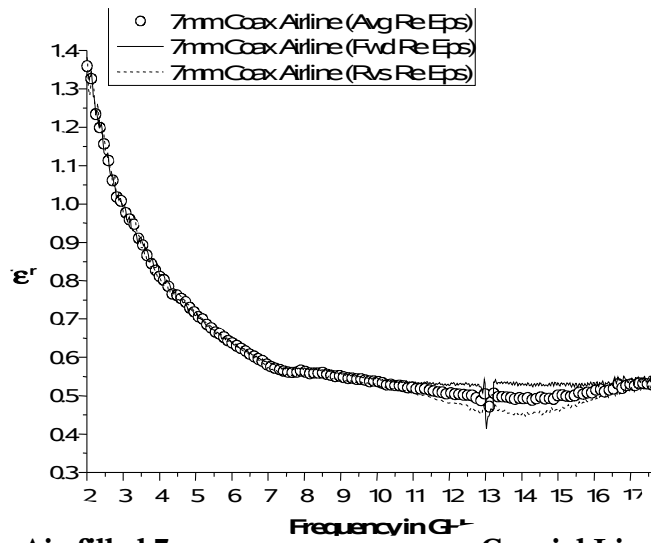


Figure 10:- Air-filled 7mm Coaxial Line Real Component of Permittivity

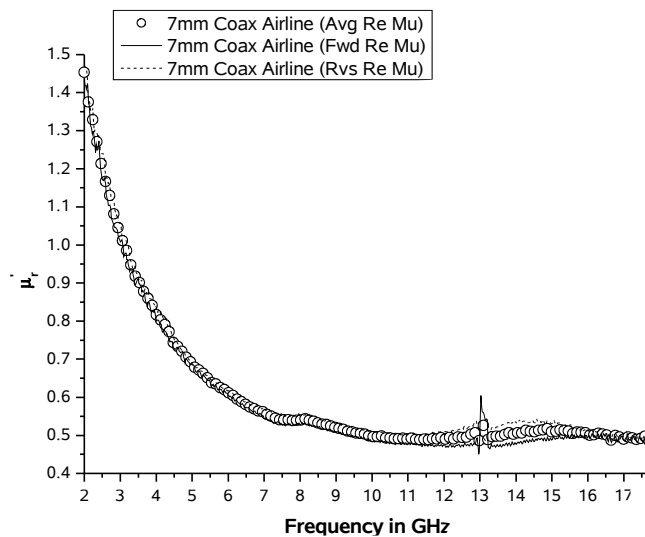


Figure 11:- Air-filled 7mm Coaxial Line Real Component of Permeability

Connection repeatability and low return loss were not an issue using WR90 waveguide. Confirmation of correct NRW equation implementation provided by measurements on three standard materials namely air, Polytetrafluoroethene (PTFE) and Polymethylmethacralate (PMMA). Figures 12 through 15 show the measured complex permittivity and permeability of three thicknesses of PTFE along with the average value.

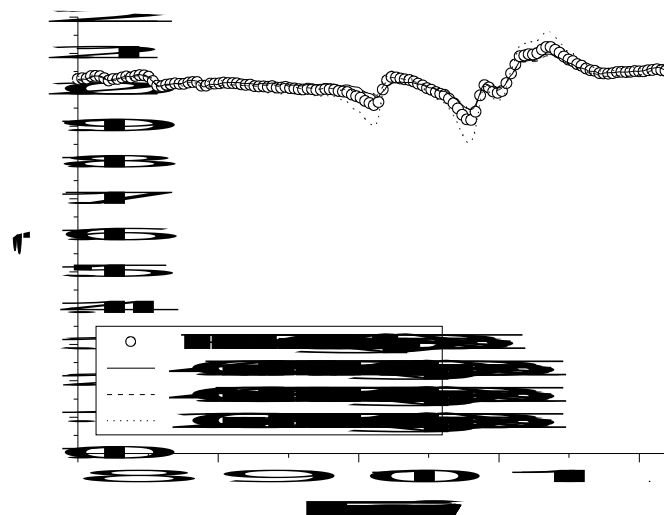


Figure 12:- WR90 Measured PTFE Real Component of Permittivity

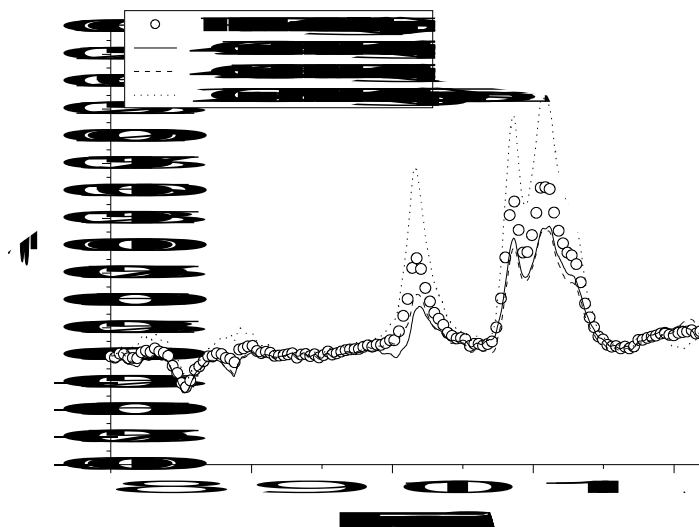


Figure 13:- WR90 Measured PTFE Imaginary Component of Permittivity

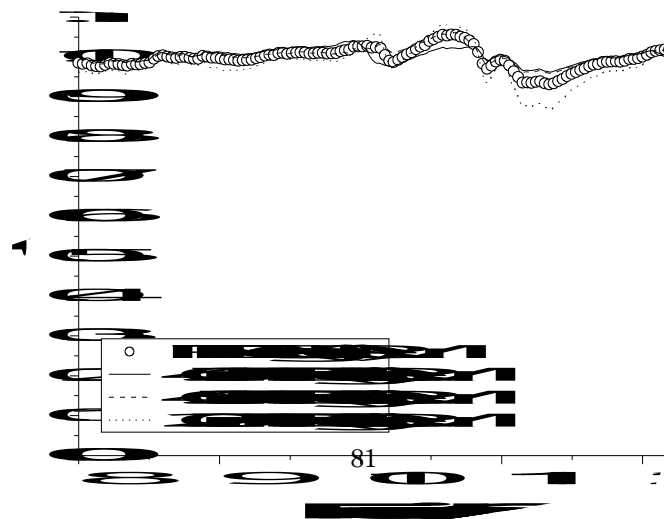


Figure 14: WR90 Measured PTFE Real Component of Permeability

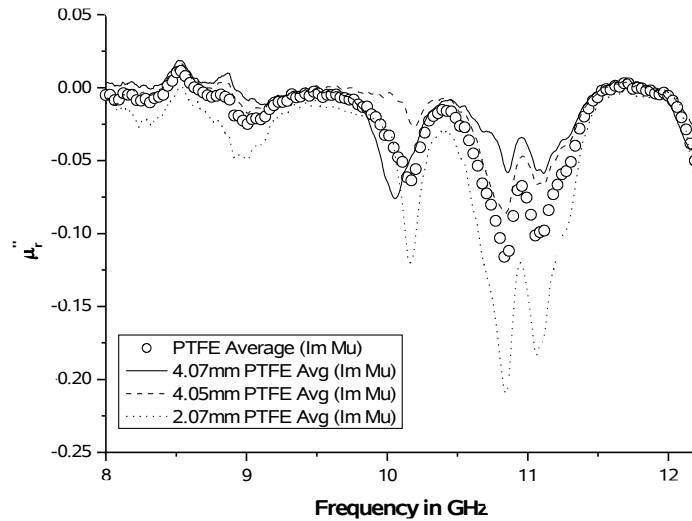


Figure 15:- WR90 Measured PTFE Imaginary Component of Permeability

For the real components sample to sample variation was low with fluctuations in measured data a result of higher order waveguide modes being supported. This was due to unevenness in thickness across the widths of the samples and the tendency of the 2.05mm thick sample to flex and be slightly misaligned when inserted into the test-cell. As expected the real component of permeability was nominally unity while the real permittivity averages just in excess of 2.0 with a minimum of 1.91543 and a maximum of 2.11801. This compares favourable with DuPont's 1MHz value of 2.1

and the value of 2.08 measured by von Hippel (1961) at 10GHz. Qualitative confirmation of the low degree of uncertainty in the NRW obtained real component of permittivity was provided by measuring PTFE sample cut from the same stock using Kobayashi and Katoh's (1985) rod-resonator technique. Right circular cylindrical samples of diameter 23.0mm and lengths 11.5, 23.0 and 46mm (all dimensions $\pm$ 0.05mm) were machined to give TE_{011} , TE_{012} and TE_{014} modes calculated to resonate at a nominal 12GHz using Kobayashi and Tanaka's (1980) mode charts.

Rod-resonator measurements of the nonmagnetic samples used the experimental arrangement shown in figure 16. Two 75mm diameter Copper discs with finely machined and then polished finishes were used to short-circuit each TE_{01n} resonator in turn. Power was coupled to and from the right circular cylindrical samples using loops formed from the centre conductors of short lengths of semi-rigid coaxial cable – see figure 16. The degree of coupling to a sample was gradually reduced by increasing the probe-resonator distance until the Q of the TE_{01n} mode stopped increasing. Once this point was reached the probes were held in place with 'grub' screws drilled into the probe mounting blocks. The resulting values of centre frequency and Q for a pair of TE_{011} and TE_{01n} samples, 'n' being an integer greater than or equal to two, were recorded. These values were used along with the sample dimensions and endplate diameters as inputs to a C program. This used the Newton-Raphson method to solve iteratively the eigenvalue equations of the TE_{011} and TE_{01n} modes used and in so doing determined the two samples' real components of permittivity.

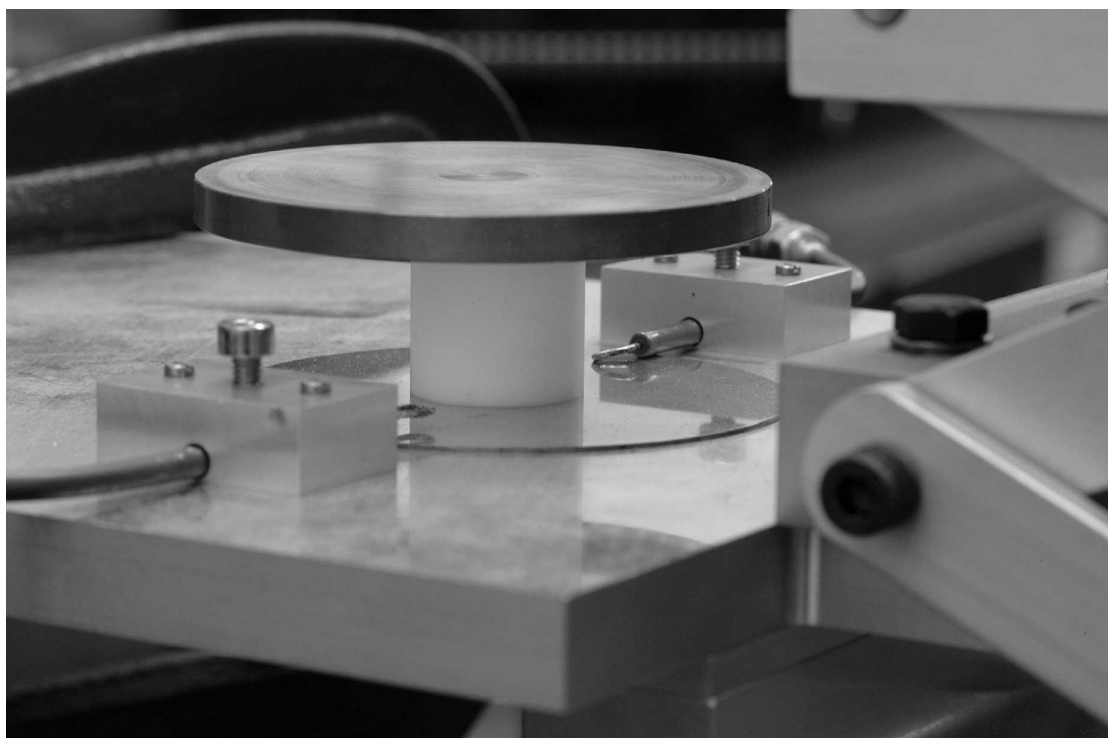


Figure 16:- Nonmagnetic Rod-Resonator Measurement Arrangement

Also solved by the software were the simultaneous equations for dielectric loss tangent derived by Kobayashi and Katoh (1985) for the case of endplates of unknown conductivity. Using the uncertainties in measured centre frequency, Q and sample dimensions along with the calculated errors caused by the use of finite diameter endplates overall uncertainty bounds for the samples' complex permittivity were calculated by the software.

As shown by figures 17, 18 and 19 the actual measured modes all lay within 38MHz of each other. Calculated values of real permittivity at 12GHz were 2.0452 ± 0.0128 , 2.0378 ± 0.00352 and 2.0360 ± 0.0022 for the TE_{011} , TE_{012} and TE_{014} modes respectively.

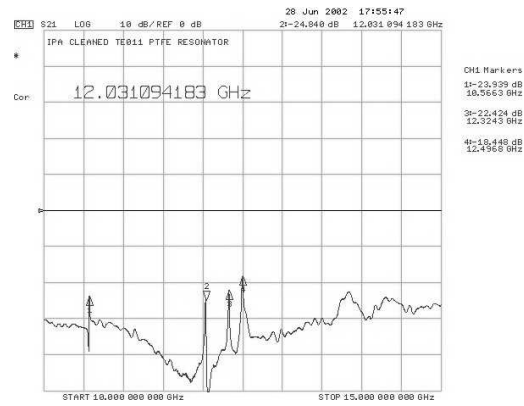


Figure 17:- TE₀₁₁ PTFE Resonator Response



Figure 18:- TE₀₁₂ PTFE Resonator Response

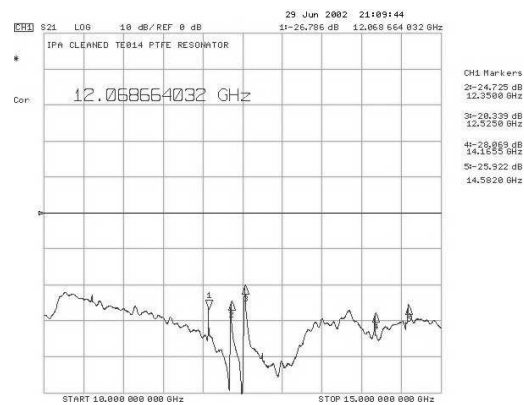


Figure 19:- TE₀₁₄ PTFE Resonator Response

These values compare very favourably with the WR90 data. Further confidence in the accuracy of the T / R technique was provided by the very good agreement between the measurements of the air and PMMA samples with values published by von Hippel (1961) and Riddle et al (2003).

The shortcomings of the T / R technique, poor sensitivity to losses, is shown by figures 13 and 15. DuPont stated that the imaginary component of permittivity for PTFE at 1MHz was 2.1×10^{-4} or smaller ($\tan \delta_e < 1 \times 10^{-4}$) while the 12GHz TE₀₁₄ resonator measured value was 5.8×10^{-4} ($\tan \delta_e = 2.849 \times 10^{-4} \pm 2.922 \times 10^{-4}$). The data presented in figure 13 shows values up to two orders of magnitude larger than this as well as nonphysical positive values. These two features were a combination of waveguide conductor losses dominating those of the material and truncation and/or rounding errors in the NRW software implementation. Figure 15 is similarly in error as the nonmagnetic PTFE should have a zero imaginary component of permeability across the measurement band. Similar responses were produced when the air and PMMA samples were measured so subsequent NRW T / R results show only the real components of permittivity and permeability.

4.2 Properties of Dielectric Host and Garnet Loading Materials

Figures 20 and 21 show the T / R measured permittivity and permeability for Crystalbond™ 509, the host dielectric used in all of the composites. At the lower end of the WR90 band the permittivity is fairly constant at around 3.125. This was very similar to the results from the SU-8 photoresist initially investigated and seemingly a fairly typical value in general for epoxies and many polymers – see von Hippel (1961).

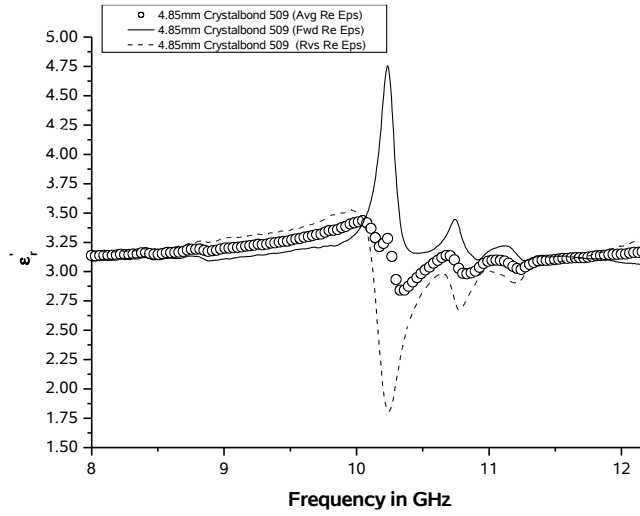


Figure 20:- WR90 Measured Permittivity of Crystalbond™ 509

As would be expected for a nonmagnetic material the permeability of figure 21 is unity across the measurement band. The rise in both permittivity and permeability with frequency with large resonances in both forward (S_{11} , S_{21}) and reverse (S_{22} , S_{12}) measured data resulted from the presence of a meniscus at the guide walls helping to support higher order modes. Predictions of composite material properties, as explained later, used the measured responses shown here and consequently show

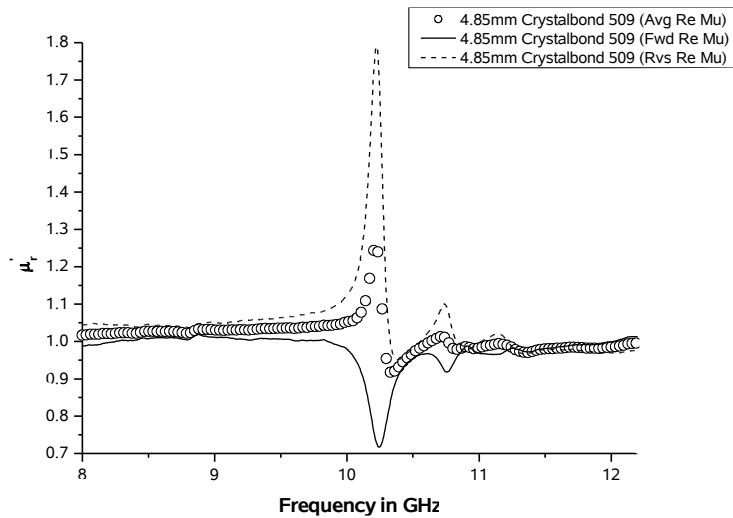


Figure 21:- WR90 Measured Permeability of Crystalbond™ 509

similar, albeit smaller, resonances at the same frequency.

Figures 22 and 23 show the measured permittivity and permeability for an 2.02mm thick unmagnetised sample of 1200 Gauss (95.52 kAm⁻¹) saturation magnetisation Indium-substituted garnet C4A. For comparison with the WR90 permittivity is the manufacturer's 9.6GHz cavity perturbation measured real permittivity which was assumed to be independent of frequency. Airgaps between the T / R measured sample and the larger, transverse, dimension of the waveguide proved impossible to fill with Silver loaded grease and were not sufficiently uniform for mathematical compensation using the equations given by Baker-Jarvis et al (1993). These E-plane gaps impact most significantly upon the permittivity of samples measured in rectangular waveguide and certainly contributed to the discrepancy, around 12.5% at the low end of the WR90 band, between the T / R data and the manufacturer's stated permittivity. The measured values of C4A permeability are presented alongside values calculated using Schloemann's (1970) equation.

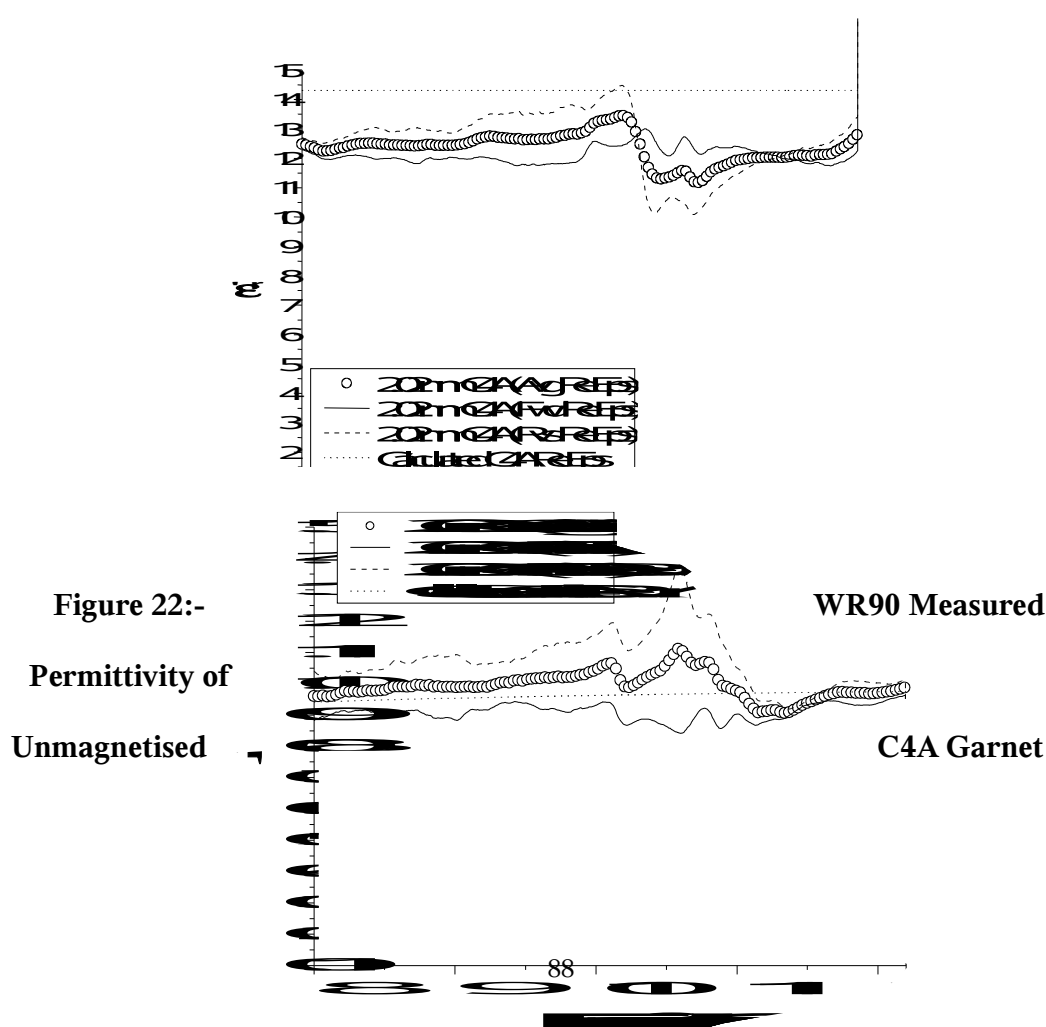


Figure 23:- WR90 Measured Permeability of Unmagnetised C4A Garnet

The averaged measured permeability compares reasonably well with the theoretical prediction allowing for the presence of H-plane airgaps which would cause an increase in measured permeability.

T / R measured permittivity and permeability of an unmagnetised 2.01mm thick sample of 1600 Gauss (127.36 kAm^{-1}) saturation magnetisation substituted garnet C6A are shown in figures 24 and 25. This sample had fewer and smaller airgaps when inserted into a WR90 test-cell but had suffered some surface flaking to the front face when being ground down to size. This is thought to be the cause of the more unstable and resonant behaviour of the forward measured properties. As with the C4A sample the measured permittivity is around 10 – 15% lower than the manufacturer's value while the measured C6A permeability is a better match to the theoretical values.

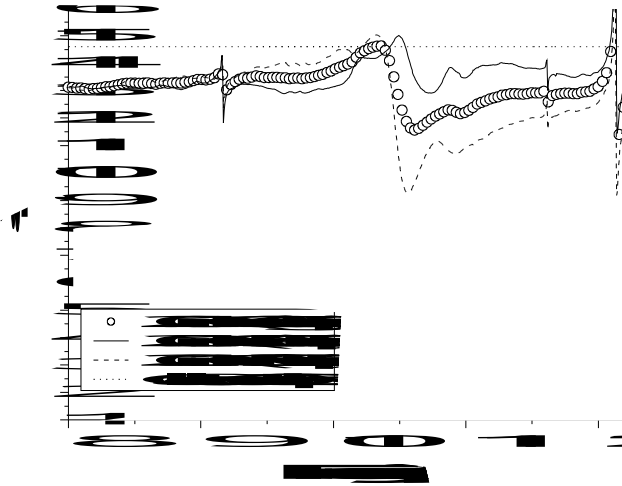


Figure 24:- WR90 Measured Permittivity of Unmagnetised C6A Garnet

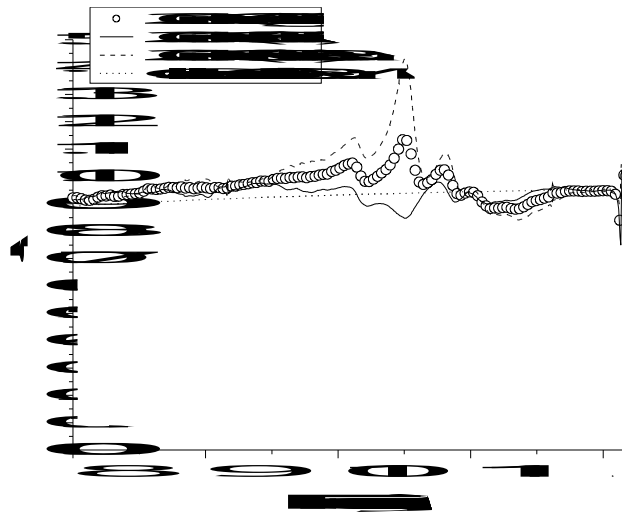


Figure 25:- WR90 Measured Permeability of Unmagnetised C6A Garnet

Figures 26, 27 and 28 show the measured permittivity and permeability of the 2.02mm thick C4A sample when subject to DC magnetic bias fields. These were applied transverse to both the direction of wave propagation in the test-cell and the fundamental rectangular waveguide TE_{10} mode's H-field component. Measurements were made with the sample biased by first one and then three permanent magnets. As a result of the transverse magnetisation the measured garnet permeability was both

scalar and reciprocal and could be expressed in terms of the tensor permeability components κ and μ as shown in equation 14 below – see also Lax and Button (1962).

$$\mu_{effective}^* = \frac{(\mu^*)^2 - \kappa^2}{\mu^*} \quad (14)$$

Aside from a small, said to account for no more than 5% change, frequency perturbational term equation 14 above is identical to the expression produced by curve-fitting finite-difference calculated inductances for microstrip lines on gyromagnetic substrates – see Massé and Pucel (1972). Plotted alongside the measured properties are the manufacturer's measured permittivity as well as permeabilities calculated using the above equation. Values of κ and μ^* were calculated using Rado's (1953) and Green and Sandy's (1974)^{1,2} equations respectively. Discussions with the manufacturer of C4A and C6A revealed that the nearest equivalent material to C4A was the Aluminium-substituted YIG G-1210 produced by Trans-Tech. Green and Sandy's (1974)^{1,2} empirically derived power law for this material was therefore used to calculate the μ'' terms in equation 14. No nearest equivalent combination of gyromagnetic resonance linewidth and saturation magnetisation to C6A could be found in Green and Sandy's self-styled catalogue of loss parameters so the μ'' terms were calculated from the power law for Trans-Tech's G-113 pure YIG but with the saturation magnetisation reduced from 1780 to 1600 Gauss (127.36 kAm⁻¹). Values of effective scalar permeability were calculated for the two garnet samples at four difference DC magnetic bias fields. These are grouped in pairs in the following figures as the Hall probe measured field strengths at the bottom, closest to the magnet(s), and top of the test-cells varied from 1024.3

Gauss (81.5 kAm⁻¹) to 739 Gauss (58.8 kAm⁻¹) for one magnet and from 1150 Gauss (91.5 kAm⁻¹) to 838.3 Gauss (66.7kAm⁻¹) for three magnets.

Airgaps between the C4A sample and the larger transverse dimension of the waveguide were filled by extending the guidewalls inwards to meet the sample edges using 101.8µm (4 mil) thick Copper foil. The presence of the Copper shim led to a small discontinuity in the waveguide dimensions in the front face of the test-cell hence S_{11} and the forward measured properties showed larger and higher Q resonances which carry through into the averaged measurements. The measured permittivity in figure 26 is essentially identical, as expected, for the two different bias conditions. The measured permeability in figures 27 and 28 does not match the predicted values for either bias condition but as will be seen later this sizeable discrepancy does not degrade the accuracy of the effective medium theory predictions to nearly the same extent.

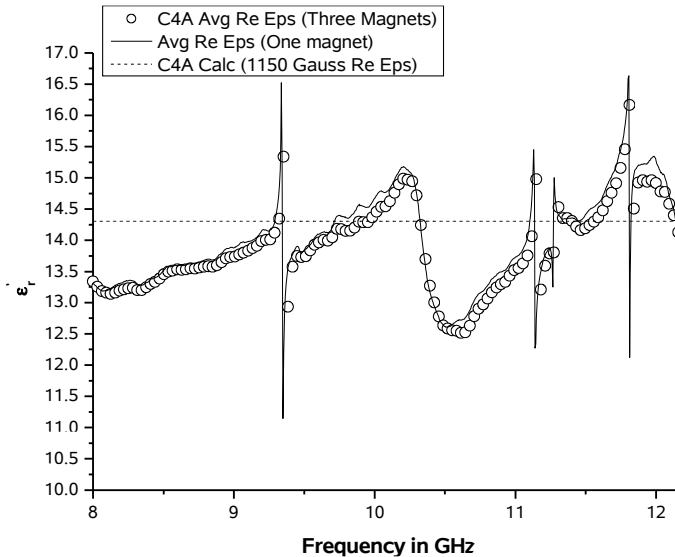


Figure 26:- WR90 Measured Permittivity of Transverse Magnetised C4A Garnet

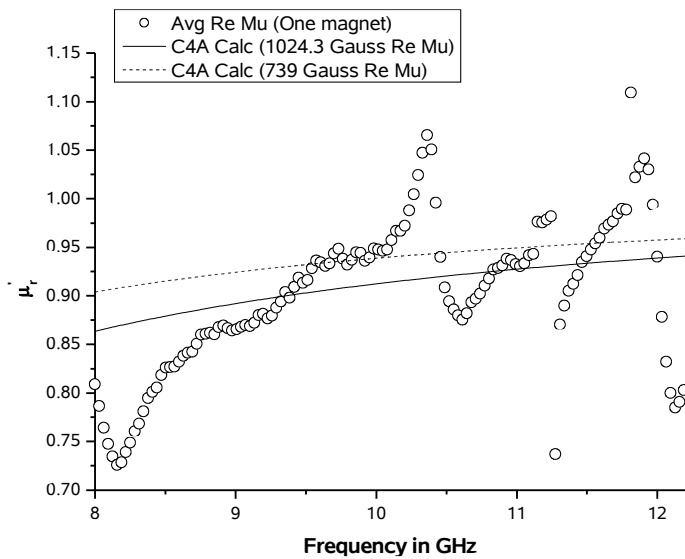


**Figure 27:- WR90 Measured Permeability of Transverse Magnetised C4A
Garnet**

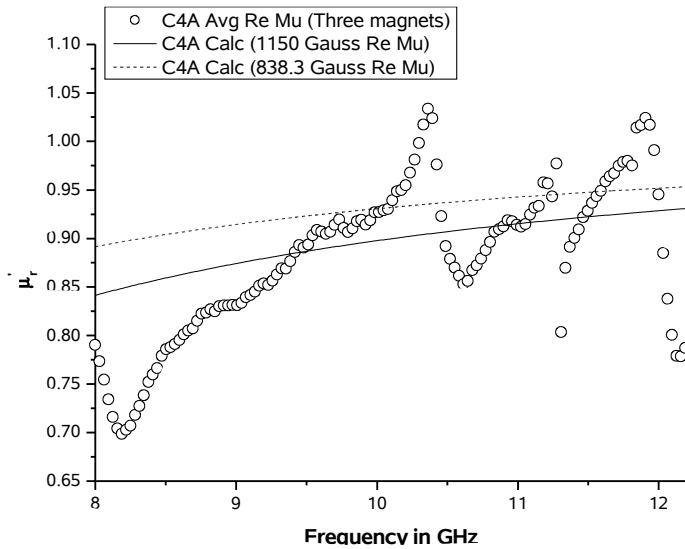


**Figure 28:- WR90 Measured Permeability of Transverse Magnetised C4A
Garnet**

Figures 29, 30 and 31 show the T / R measured permittivity and permeability of a 2.01mm thick C6A sample subject to the same transverse DC magnetic bias fields as the C4A sample described above. The C6A sample fitted more closely into its WR90 test-cell than the C4A specimen consequently the measured data displays considerably fewer resonances and, at least for the permittivity, a much better fit with the manufacturer supplied value. Once again the measured permeability of graphs 30 and 31 compares extremely poorly with the values calculated using Green and Sandy's (1974)¹ formulae. Assuming a magnetically saturated sample and using Polder and Wills' (1949) tensor permeability expressions in conjunction with equation 14 lead to calculated effective permeabilities, not shown, equally in error albeit as under- rather than overestimates.

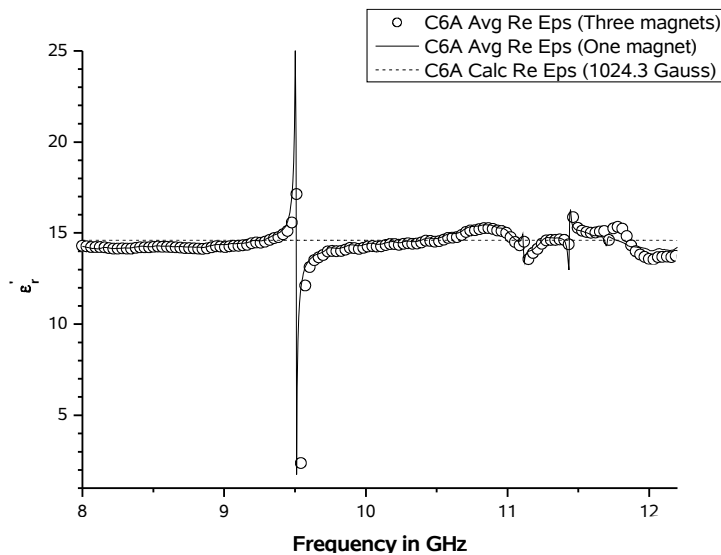


Figure 29:- WR90 Measured Permittivity of Transverse Magnetised C6A Garnet

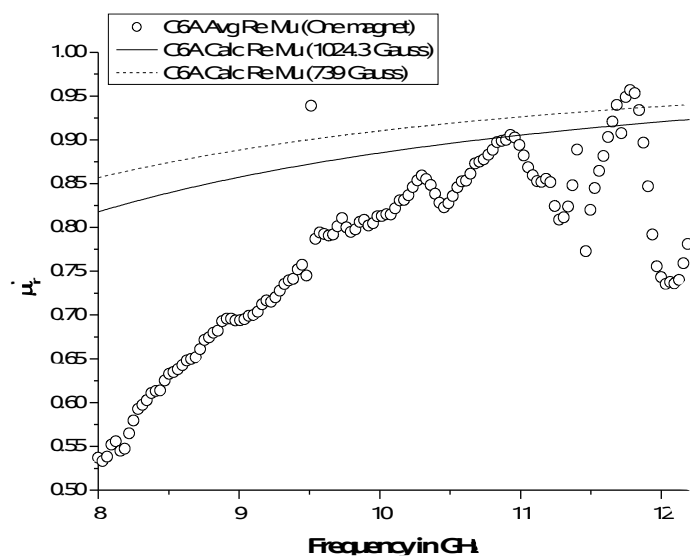


Figure 30:- WR90 Measured Permeability of Transverse Magnetised C6A

Garnet

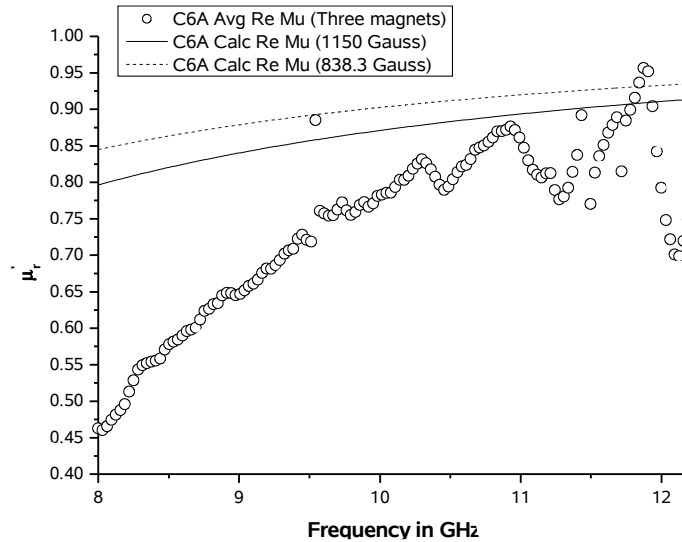


Figure 31:- WR90 Measured Permeability of Transverse Magnetised C6A

Garnet

4.3 Properties of Unmagnetised Composite Materials

Figures 32 through 111 inclusive present the T / R measured permittivity and permeability of unmagnetised binary composites formed from Crystalbond™ 509 and either C4A or C6A powder. Software was written to combine complex permittivity and permeability data for the designated host and inclusion materials to make predictions as to the resulting electromagnetic properties of the composite. For a given inclusion volume fraction calculations were made using both the Bruggeman and NIST-modified Maxwell-Garnett effective medium theories. In the case of the NIST model calculations were performed twice with the rôles of host and inclusion exchanged in order to give upper and lower bounds for the predicted composite properties – see Geyer et al (1994). Based upon the measured and manufacturer

supplied permittivity data and a stated maximum garnet particle size of $5\mu\text{m}$ it was assumed that the garnet particles were completely penetrable to incident electromagnetic radiation over the WR90 band i.e. $G(k_p a)$ in equations 5 and 6 was unity. All of the model predictions labelled 'Calc' used a combination of the measured Crystalbond™ 509 properties with the garnet manufacturer's complex permittivity data and complex permeabilities calculated using Schloemann (1970) and Green and Sandy's (1974)^{1,2} equations as described earlier. Following effective medium theory predictions of composite properties for 0.1 to 0.9 (inclusive) garnet volume fractions it was decided that only the 0.3 and 0.4 volume fractions would be experimentally investigated. These were chosen based upon easy to fabricate microstrip line dimensions calculated using Pucel and Massé's (1972) formulae assuming 10.00075GHz operating frequency, Z_0 of 50Ω , $1\mu\text{m}$ thick metallisation and a 1mm thick substrate. The ultimate intention being to fabricate and test the tunability of half-wavelength gap-coupled two-port linear microstrip resonators.

Figure 32 compares the measured permittivity for a 0.3 volume fraction C4A composite with predictions made using the measured properties of the individual components.

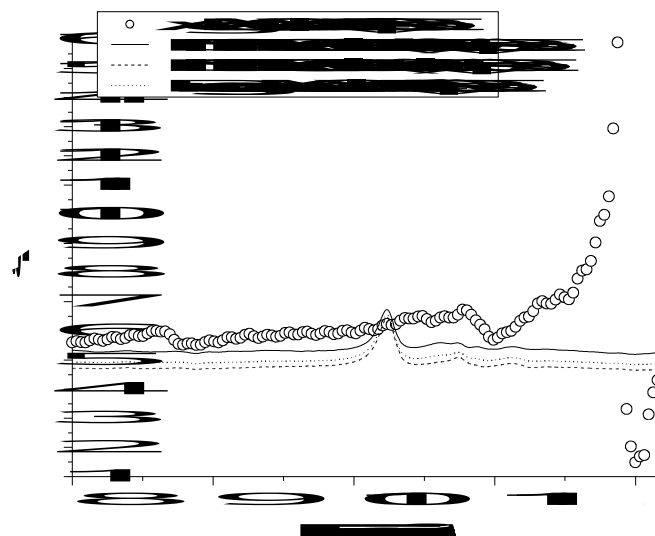


Figure 32:- Comparison of Measured Permittivity of Unmagnetised 0.3 Volume Fraction C4A Composite with Effective Medium Theory Predictions

Of the predictions the modified Maxwell-Garnett model with the C4A designated as host gives the best match of the three in predictions to the measured data in figure 32 above. The model underpredicts the measured permittivity which is perhaps unsurprising given the discrepancy between the manufacturer supplied real permittivity and the WR90 measured value already shown in figure 22. The difference between predicted and measured properties stays below 12.5% from 8.2 to 10.45GHz as shown in figure 33 which is limited to 12GHz due to the onset of higher order waveguide modes leading to nonsensical NRW calculated properties.

Referring to figure 34 use of the same effective medium theories but supplied with the manufacturer permittivity and calculated permeabilities gives a small improvement in

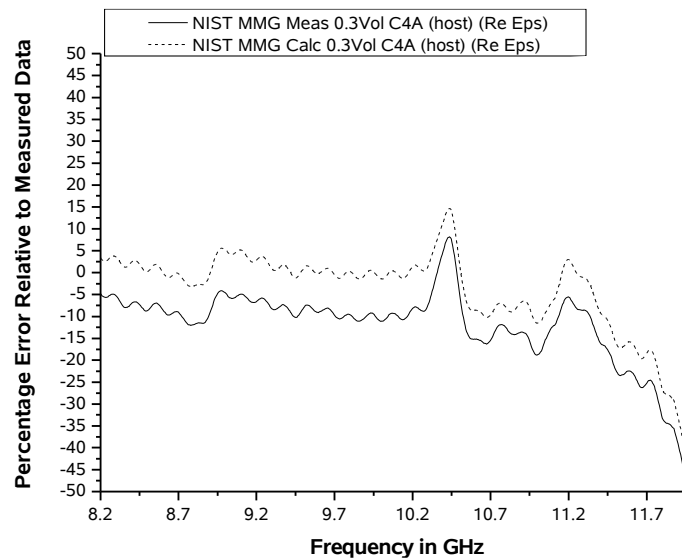


Figure 33:- Percentage Errors in Predicted Permittivity of 0.3 Volume Fraction C4A Composite

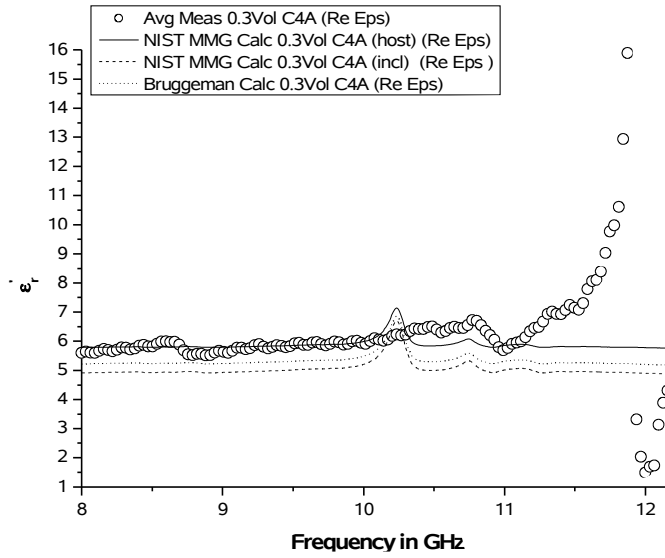


Figure 34:- Comparison of Measured Permittivity of Unmagnetised 0.3 Volume Fraction C4A Composite with Effective Medium Theory Predictions

the match with the measured permittivity as shown by the percentage error in figure 33 above. As with Geyer et al's (1994) work the Bruggeman predictions, using both measured and manufacturer provided permittivities, lay about midway between the upper and lower limits established by the modified Maxwell-Garnett predictions.

Figures 35 and 36 show the measured composite permeability against the predictions using both measured and calculated garnet properties. In both cases the differences in values for the two Maxwell-Garnett calculations and the Bruggeman model so small as to not be visible when plotted.

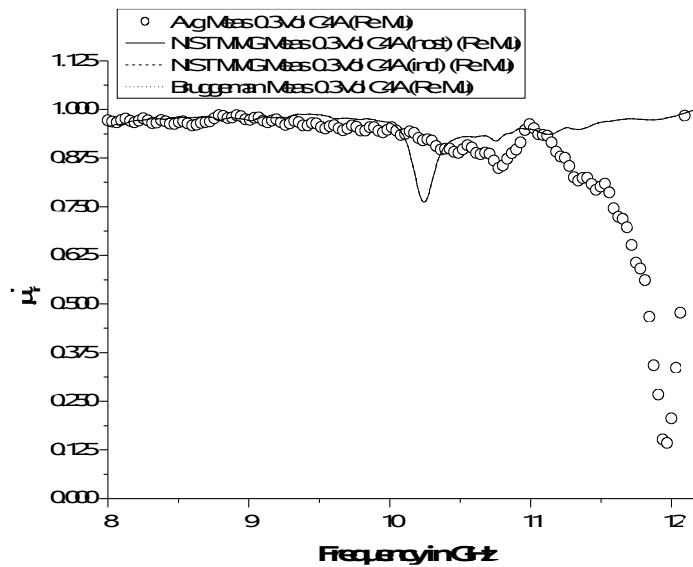


Figure 35:- Comparison of Measured Permeability for Unmagnetised 0.3 Volume Fraction C4A Composite with Effective Medium Theory Predictions

In terms of the match between the measured and predicted properties the difference is less than 5% from 8.2 to 10.45GHz for both the measurement and calculation based garnet data – see figure 37. The sudden drop in accuracy from 10.2 to 10.45GHz in figure 37 is an artifact from the measured Crystalbond™ properties being carried through the effective medium theory calculations. The level of agreement between the effective medium theories and composite properties degrades rapidly above 11.2GHz due to overmoding of the filled waveguide test-cell.

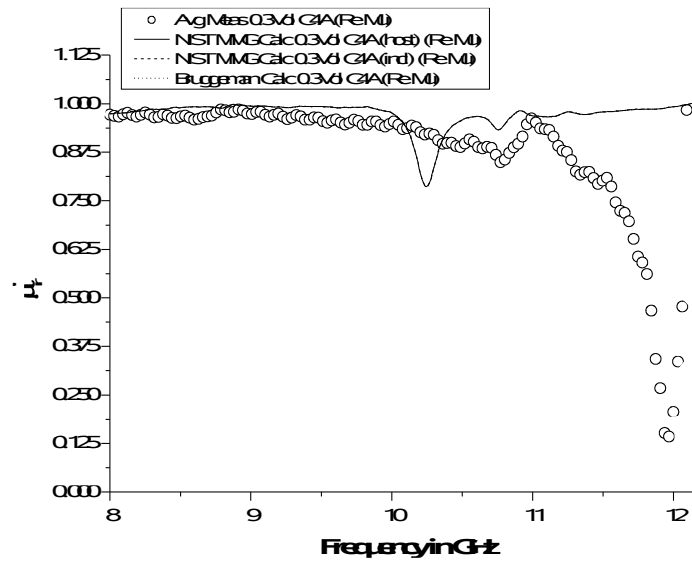


Figure 36:- Comparison of Measured Permeability for Unmagnetised 0.3 Volume Fraction C4A Composite with Effective Medium Theory Predictions

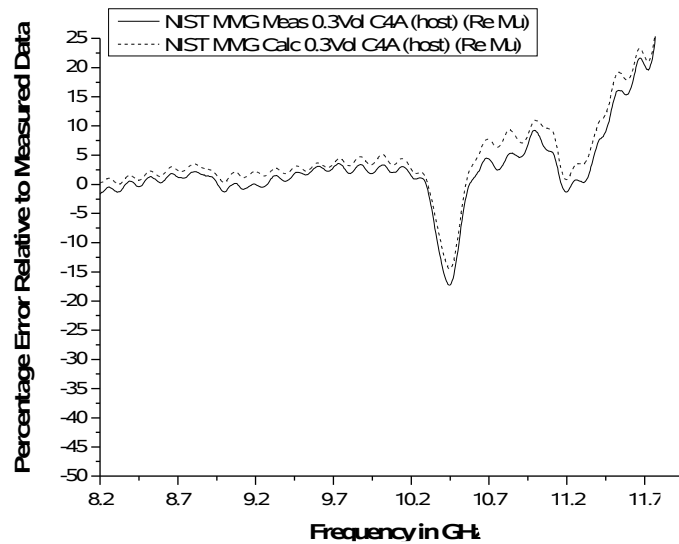


Figure 37:- Percentage Errors in Predicted Permeability of 0.3 Volume Fraction C4A Composite

Figures 38 and 39 compare the measured and predicted permittivity for an unmagnetised 0.4 volume fraction C4A composite. As with the 0.3 volume fraction material the modified Maxwell-Garnett model with the C4A as host gave the best fit when using T / R measured C4A permittivity – see figure 40.

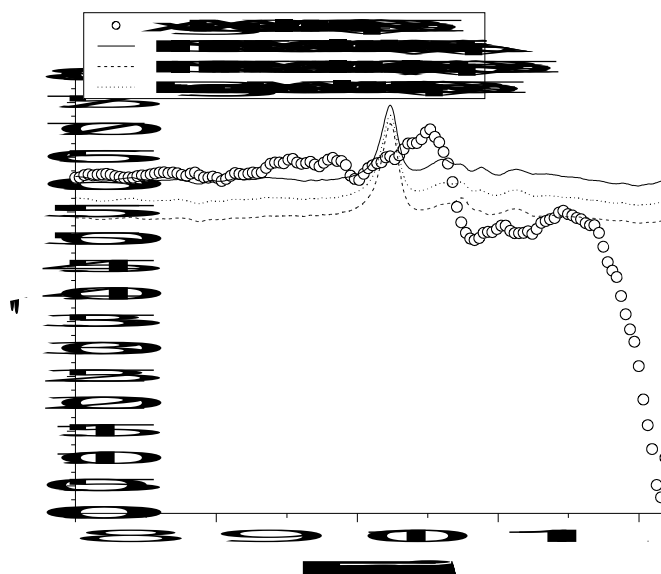


Figure 38:- Comparison of Measured Permittivity for Unmagnetised 0.4 Volume Fraction C4A Composite with Effective Medium Theory Predictions

Referring to figure 39 which used the manufacturer's permittivity data an almost equally good match is obtained using the Bruggeman model. This would seem to be in agreement with Geyer et al's (1994) findings that for approximately equal volume fractions of ferrite loaded dielectrics the Bruggeman model had the best predictive ability.

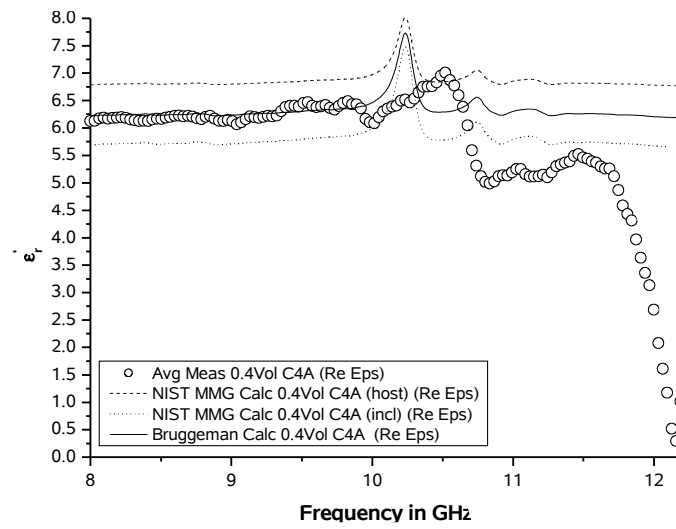


Figure 39:- Comparison of Measured Permittivity for Unmagnetised 0.4 Volume Fraction C4A Composite with Effective Medium Theory Predictions

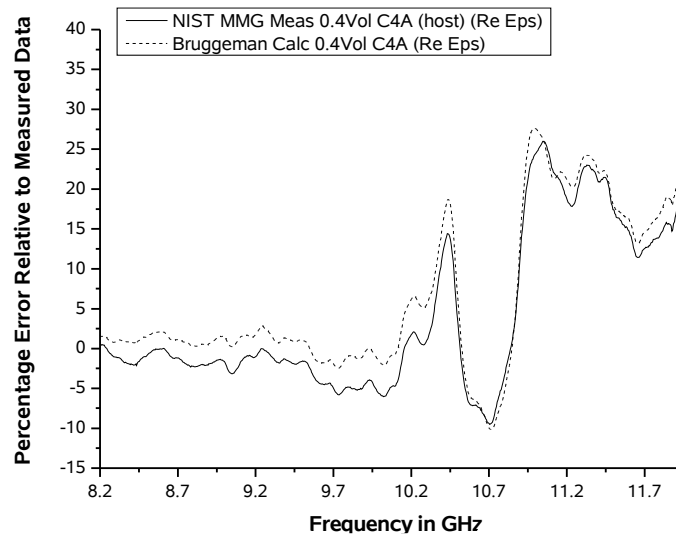


Figure 40:- Percentage Errors in Predicted Permittivity of 0.4 Volume Fraction C4A Composite

This change from that encountered with the 0.3 volume fraction composite is possibly due to the garnet inclusions no longer meeting the assumptions of the Maxwell-Garnett model namely a regular lattice-like distribution of equally sized particles throughout the dielectric host material. Perhaps somewhat surprisingly both of the models just discussed as best fitting the measured composite properties give almost the same percentage errors relative to the T / R measured composite permittivity as shown by figure 40. The difference being less than 10% from 8.2GHz to just above 10.2GHz where the aforementioned Crystalbond™ resonance occurs.

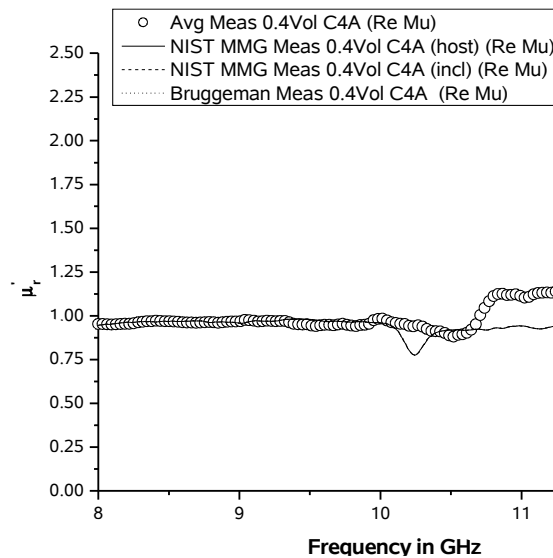


Figure 41:- Comparison of Measured Permeability for Unmagnetised 0.4 Volume Fraction C4A Composite with Effective Medium Theory Predictions

Looking at figures 41 and 42 of the 0.4 volume fraction permeability there is only a marginal difference again between the measured data and the two groupings of predictions. The error in the effective medium theory predictions remaining below 5% from 8.2GHz to the Crystalbond™ induced resonance around 10.45GHz. Above this point comparisons cannot be made as the actual composite no longer supported just the TE₁₀ fundamental waveguide mode.

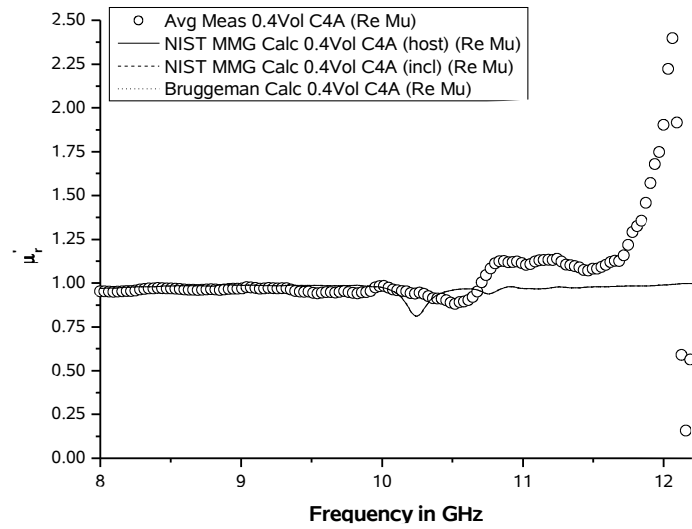


Figure 42:- Comparison of Measured Permeability for Unmagnetised 0.4 Volume Fraction C4A Composite with Effective Medium Theory Predictions

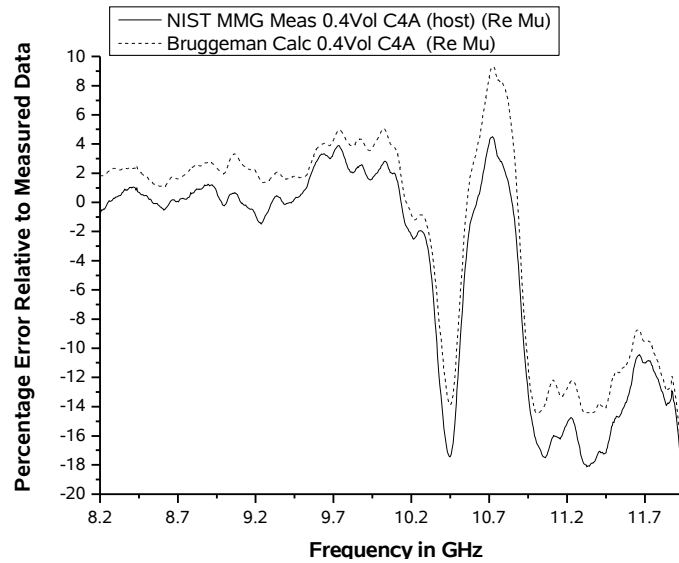


Figure 43:- Percentage Errors in Predicted Permeability of 0.4 Volume Fraction C4A Composite

Looking at the unmagnetised C6A composites a similar although not identical set of results were produced. Figures 44 and 45 show the measured permittivity of a 0.3 volume fraction C6A composite alongside the effective medium theory predictions.

As with the C4A composites the best fit between the measured composite permittivity and predictions made using T / R measured C6A permittivity is given by the modified Maxwell-Garnett model with C6A designated as host – see figure 46.

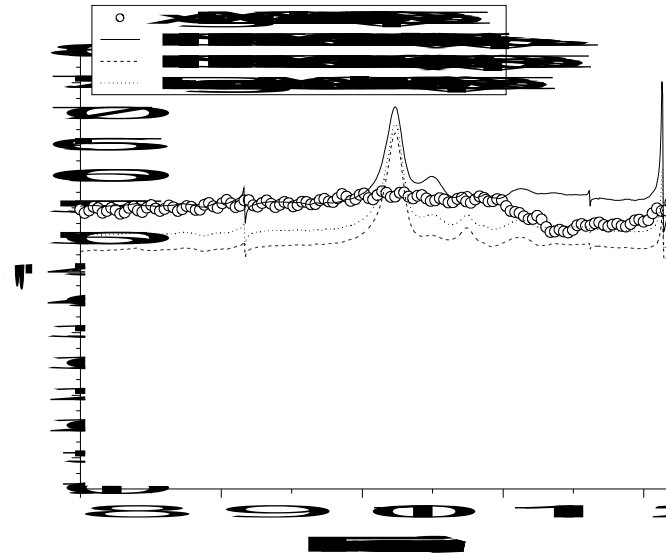


Figure 44:- Comparison of Measured Permittivity for Unmagnetised 0.3 Volume Fraction C6A Composite with Effective Medium Theory Predictions

Leaving aside the Crystalbond™ induced resonance the difference between the predicted and measured composite permittivity is less than 5% from 8.2 to 10.7GHz and within 15% out to 12GHz.

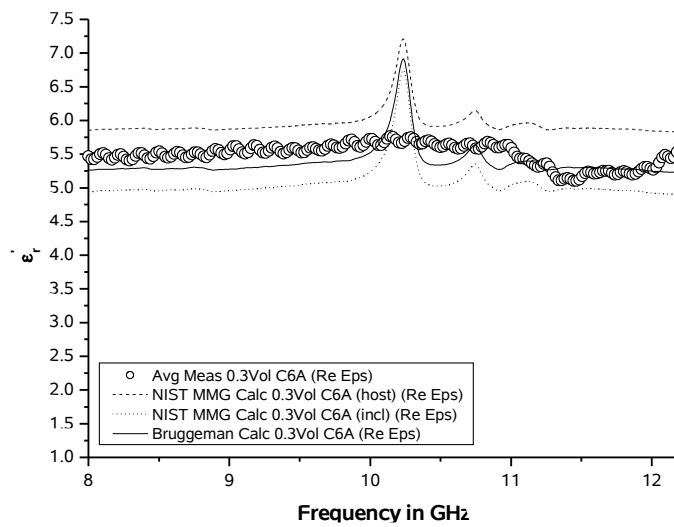


Figure 45:- Comparison of Measured Permittivity of Unmagnetised 0.3 Volume Fraction C6A Composite with Effective Medium Theory Predictions

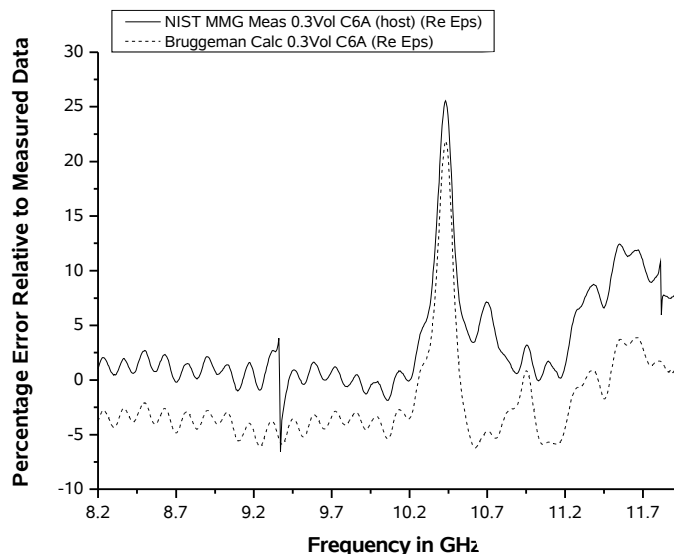


Figure 46:- Percentage Errors in Predicted Permittivity of 0.3 Volume Fraction C6A Composite

Referring to graph 45 comparing measured composite permittivity with predictions made using the manufacturer's stated C6A permittivity the best match is provided by the Bruggeman model. As shown in figure 46 the percentage difference between the

Bruggeman prediction and the measured permeability is also within 5% from 8.2GHz up to the resonance centred around 10.45GHz. Following this the model continues to stay well within 7.5% of the actual T / R measured permeability around half the error in the modified Maxwell-Garnet model prediction using WR90 measured C6A properties. Both graphs 47 and 48 comparing measured composite permeability with the effective medium theory predictions show very similar responses with a very good match to the T / R data. Ignoring the resonance the percentage error for the modified Maxwell-Garnet model, measured C6A permeability and the garnet as host, and the Bruggeman model, calculated permeability, stays well within 5% from 8.2GHz out to 12GHz – see figure 49.

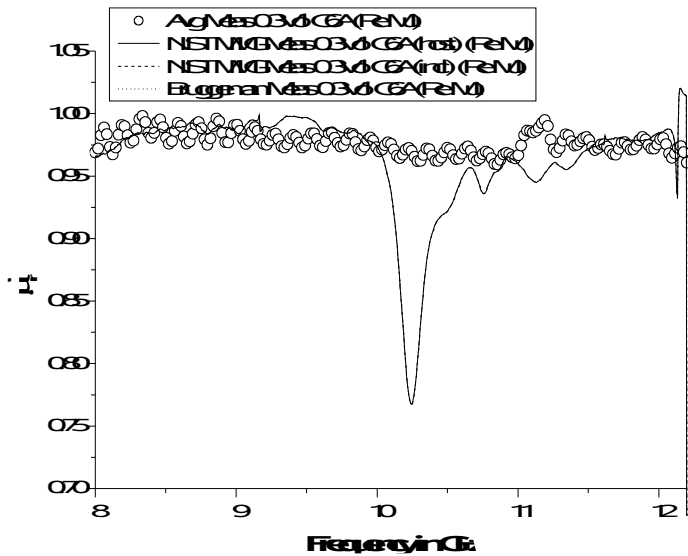


Figure 47:- Comparison of Measured Permeability of Unmagnetised 0.3 Volume

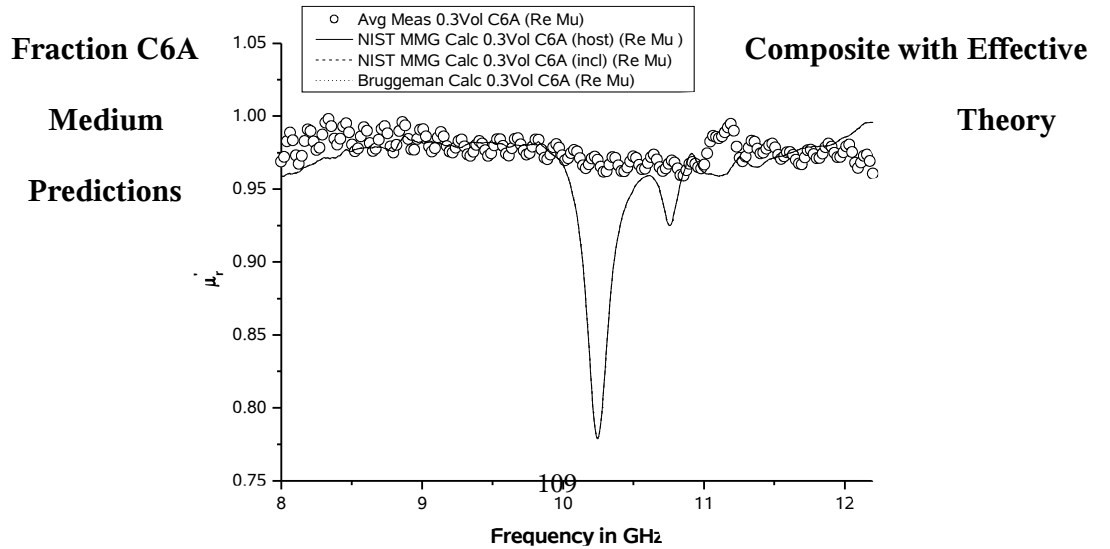


Figure 48:- Comparison of Measured Permeability of Unmagnetised 0.3 Volume Fraction C6A Composite with Effective Medium Theory Predictions

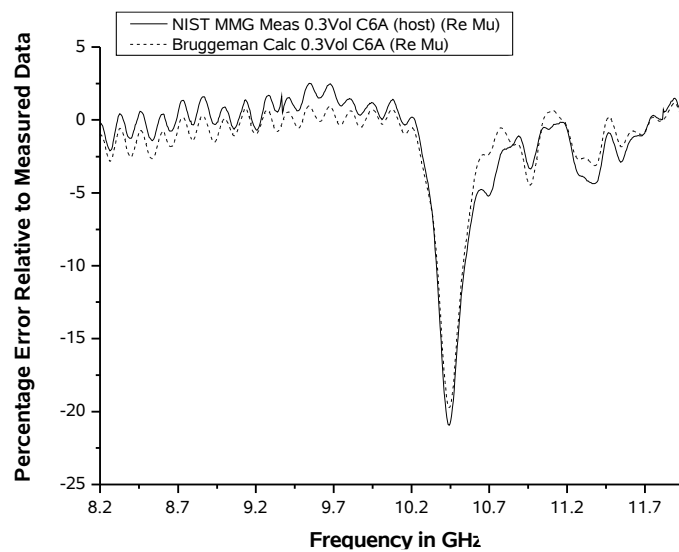


Figure 49:- Percentage Errors in Predicted Permeability of 0.3 Volume Fraction C6A Composite

Measured and predicted properties of an unmagnetised 0.4 volume fraction C6A composite are shown in figures 50 to 55 inclusive. An identical set of comparisons

between measured and predicted properties to those obtained with the same volume fraction C4A composites is demonstrated. The modified Maxwell-Garnett model using T / R measured permittivity with the garnet as host and the Bruggeman model using the manufacturer’s permittivity data giving the best fit to the measured composite permittivity – see figures 50 and 51.

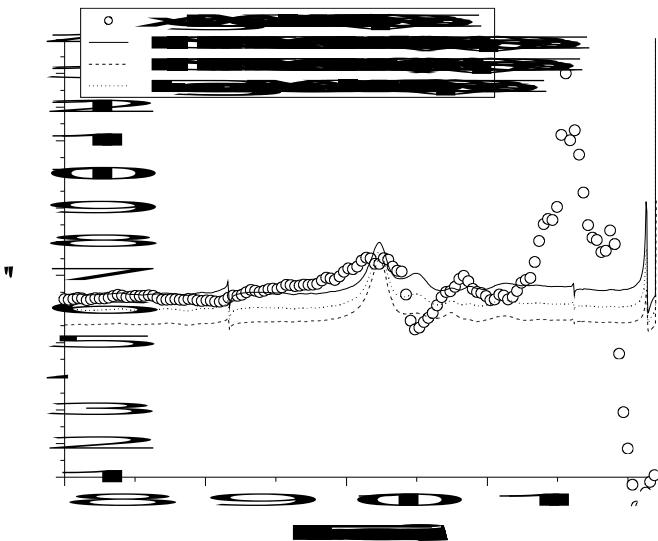


Figure 50:- Comparison of Measured Permittivity of Unmagnetised 0.4 Volume Fraction C6A Composite with Effective Medium Theory Predictions

In terms of percentage errors relative to the measured composite permittivity the two aforementioned predictions give nearly identical performance as shown in figure 52. They are slightly more accurate than for the 0.4 volume fraction C4A composite with the errors remaining less than 10% from 8.2GHz to around 11.45GHz.

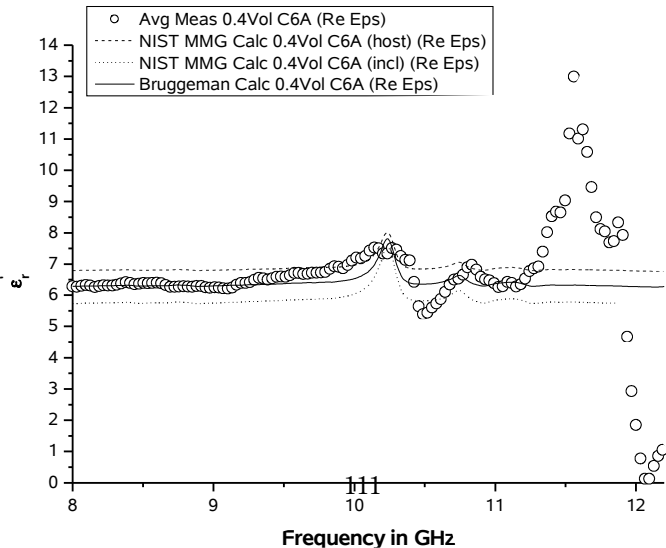


Figure 51:- Comparison of Measured Permittivity of Unmagnetised 0.4 Volume Fraction C6A Composite with Effective Medium Theory Predictions

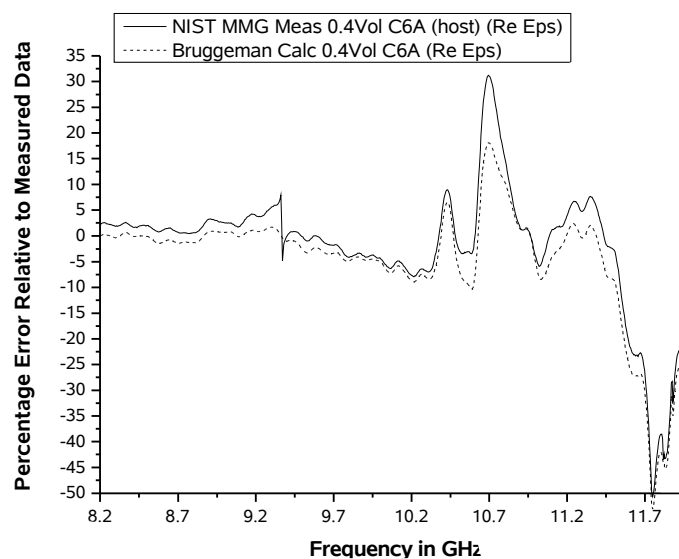


Figure 52:- Percentage Errors in Predicted Permittivity of 0.4 Volume Fraction C6A Composite

Figures 53 and 54 compare the measured and predicted permeabilities for the composite. Both the predictions using measured and calculated C6A permeabilities give extremely similar responses the Bruggeman model being slightly superior in both cases. All of the effective medium theory predictions overestimate the

composite’s permeability somewhat. As with the 0.4 volume fraction C4A composite the error relative to the T / R data is less than 5% from 8.2GHz to around 10.45GHz and then remains within 15% to 11.45GHz and the propagation of higher order waveguide modes – see figure 55.

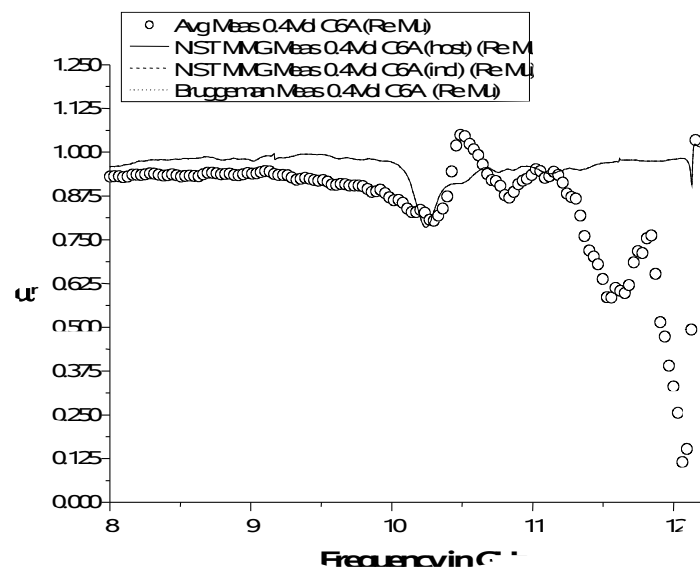


Figure 53:- Comparison of Measured Permeability of Unmagnetised 0.4 Volume Fraction C6A Composite with Effective Medium Theory Predictions

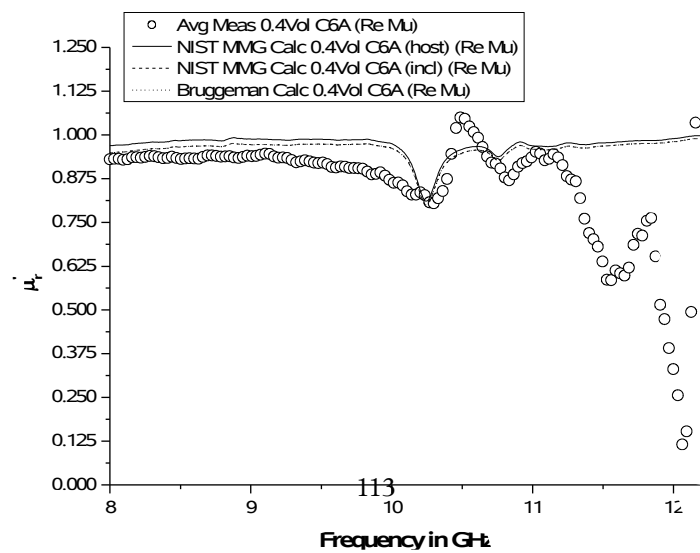


Figure 54:- Comparison of Measured Permeability of Unmagnetised 0.4 Volume Fraction C6A Composite with Effective Medium Theory Predictions

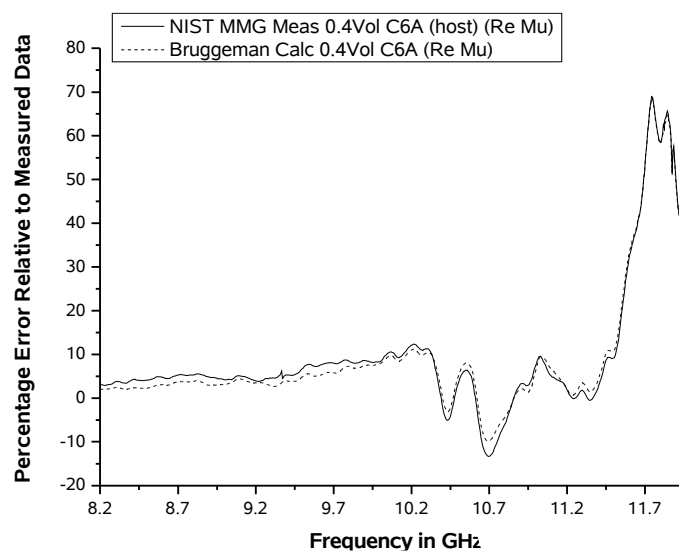


Figure 55:- Percentage Errors in Predicted Permeability of 0.4 Volume Fraction C6A Composite

4.4 Properties of Transversely Magnetised Composite Materials

Figures 56 and 57 compare the measured permittivity of a 0.3 volume fraction C4A composite transversely magnetised by a single permanent magnet with effective medium theory predictions. As with the unmagnetised sample the best match with the T / R data using both the WR90 measured C4A permittivity and the

manufacturer's value is provided by the modified Maxwell-Garnett model with the C4A designated as the composite's host material.

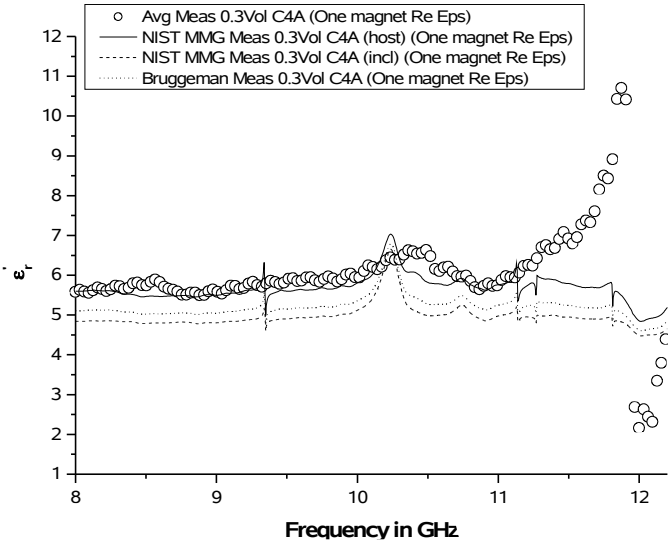


Figure 56:- Comparison of Measured Permittivity of Magnetised 0.3 Volume Fraction C4A Composite with Effective Medium Theory Predictions

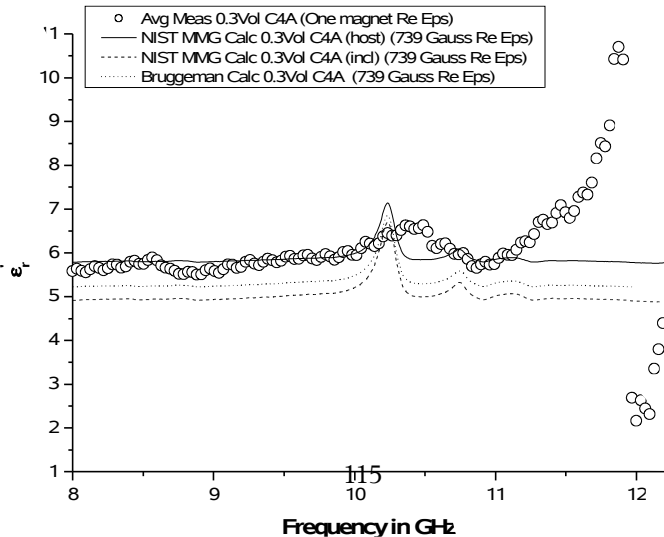


Figure 57:- Comparison of Measured Permittivity of Magnetised 0.3 Volume Fraction C4A Composite with Effective Medium Theory Predictions

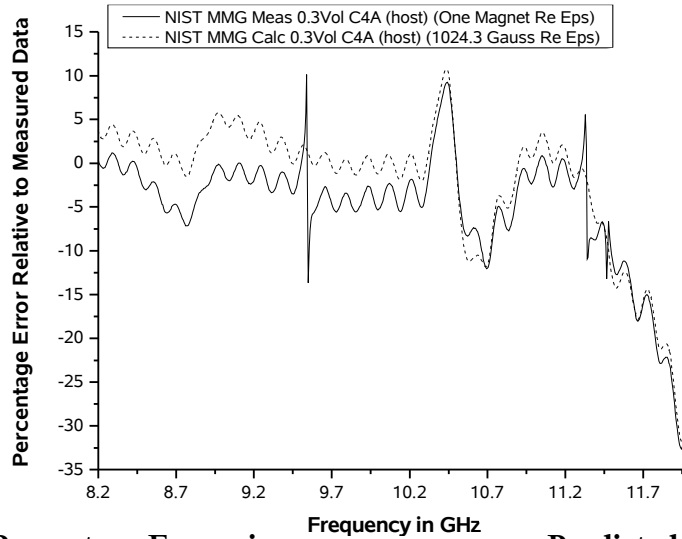


Figure 58:- Percentage Errors in Predicted Permittivity of Magnetised 0.3 Volume Fraction C4A Composite

Reassuringly, T / R measured composite permittivity is, as expected, all but identical to that measured with the unmagnetised sample shown previously in figure 32. As shown by figure 58 the percentage error in the Maxwell-Garnett model predictions relative to the measured composite permittivity remains less than 15% from 8.2GHz, ignoring the Crystalbond™ resonance, to around 11.7GHz and the onset of higher order mode induced NRW equation irregularities. This is a similar level of agreement to that obtained for the unmagnetised data on this composite – see figure 33.

Figures 59, 60 and 61 compare the measured permeability of the sample with the effective medium theory predictions. As with the measurements on pure garnet samples predictions made using calculated effective scalar permeabilities for the garnet component were calculated using both the maximum and minimum measured DC bias flux densities of 1024.3 Gauss (81.53 kAm⁻¹) and 739 Gauss (58.82 kAm⁻¹) respectively. In graphs 59, 60 and 61 the effective medium theory predictions using the Bruggeman and modified Maxwell-Garnett models are so close as to be indistinguishable.

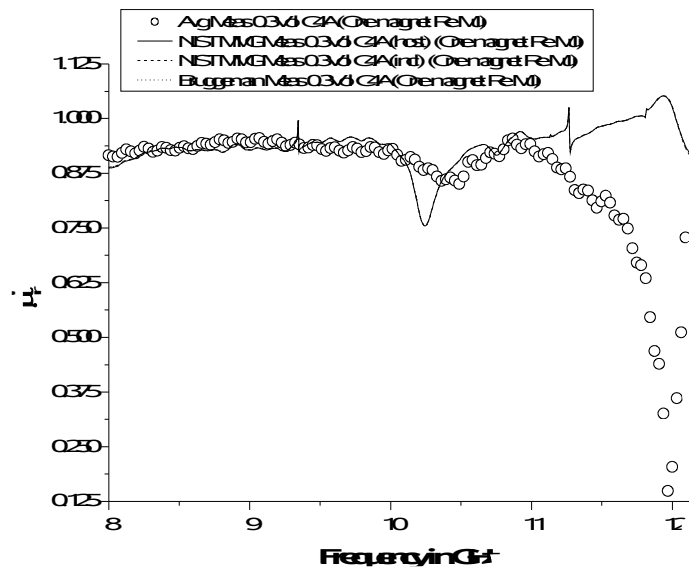


Figure 59:- Comparison of Measured Permeability of Magnetised 0.3 Volume Fraction C4A Composite with Effective Medium Theory Predictions

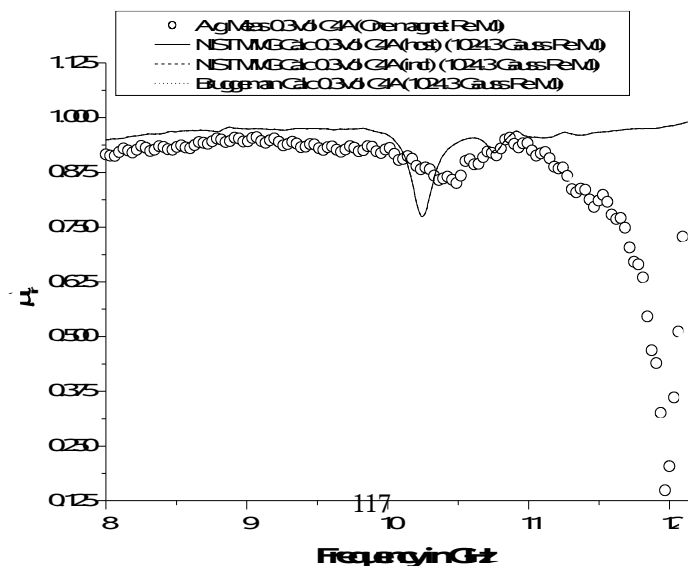


Figure 60:- Comparison of Measured Permeability of Magnetised 0.3 Volume Fraction C4A Composite with Effective Medium Theory Predictions

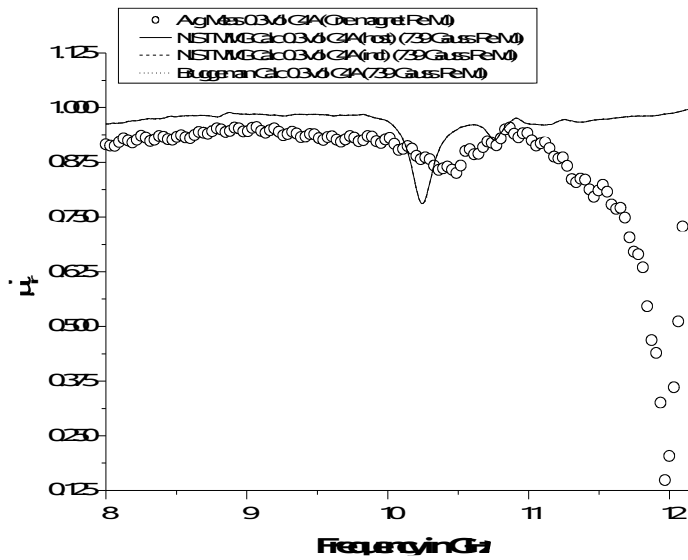


Figure 61:- Comparison of Measured Permeability of Magnetised 0.3 Volume Fraction C4A Composite with Effective Medium Theory Predictions

Using the T / R measured permeability of a transversely magnetised C4A sample in the modified Maxwell-Garnett model with C4A designated as host gives a very good match with the measured composite permeability. The difference between the two, ignoring the resonant peak, remaining less than 5% from 8.2GHz to around 11.2GHz as show in figure 62. This is in spite of the large discrepancy observed earlier in figure 27 when comparing the measured permeability of a transversely magnetised C4A sample with the calculated effective scalar permeability values. Using

calculated values of magnetised garnet effective scalar permeability in the effective medium theory equations the best fit to the measured composite permeability is given assuming a uniform DC magnetic flux density of 1024.3 Gauss (81.53 kAm⁻¹) – see figures 60 and 61. Using this data in the modified Maxwell-Garnett model with the C4A designated as host gives an identical level of accuracy in prediction to that observed using the T / R measured garnet permeability. The error again remaining less than 5% from 8.2 to around 11.2GHz – see figure 62.

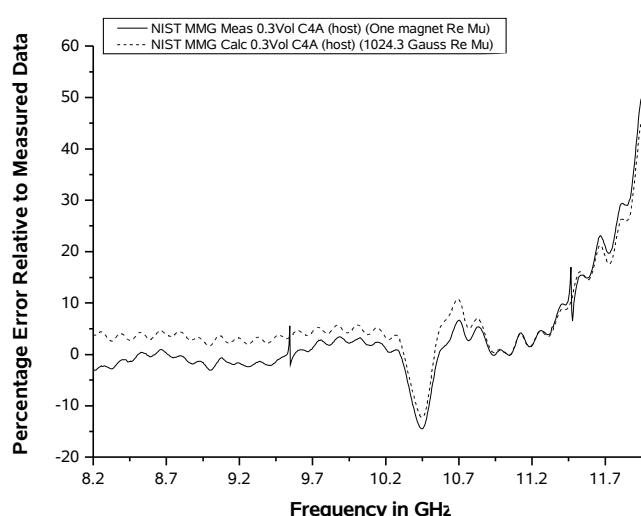


Figure 62:- Percentage Errors in Predicted Permeability of Magnetised 0.3 Volume Fraction C4A Composite

The measured permittivity of a 0.4 volume fraction C4A composite transversely magnetised by a single permanent magnet shows the same trends demonstrated by the unmagnetised material. The modified Maxwell-Garnett model using T / R measured C4A permittivity with the garnet as host and the Bruggeman model using the manufacturer's permittivity data once again gives the best match with the measured composite permittivity – see figures 63 and 64.

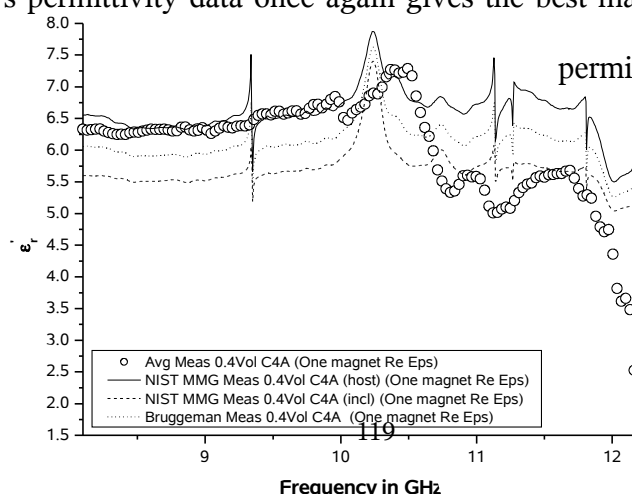


Figure 63:- Comparison of Measured Permittivity of Magnetised 0.4 Volume Fraction C4A Composite with Effective Medium Theory Predictions

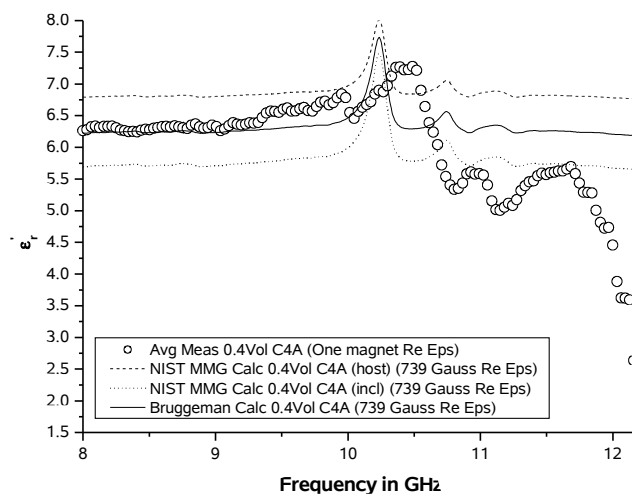


Figure 64:- Comparison of Measured Permittivity of Magnetised 0.4 Volume Fraction C4A Composite with Effective Medium Theory Predictions

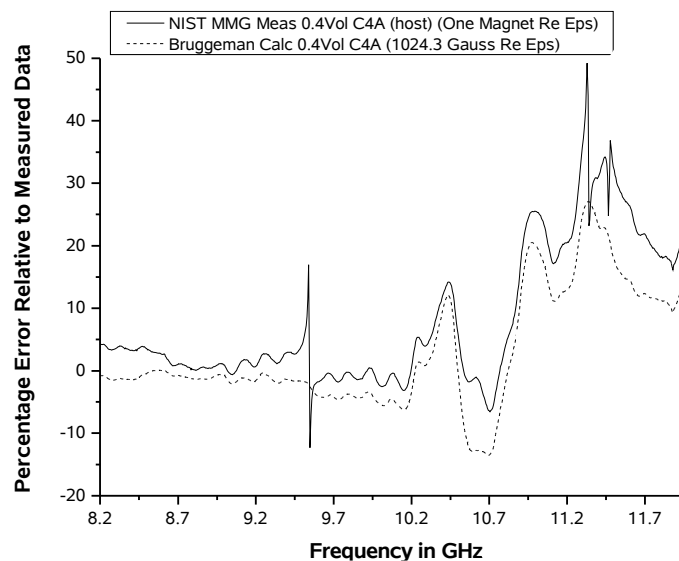


**Figure 65:- Percentage Errors in Predicted Permittivity of Magnetised 0.4
Volume Fraction C4A Composite**

As with the magnetised 0.3 volume fraction C4A composite the error in the predicted permittivity is less than 5% from 8.2GHz to around 10.2GHz with the Bruggeman prediction performing particularly well – see figure 65. Looking at figure 66 showing the composite’s permeability the predictions using the T / R measured permeability of the magnetised C4A, regardless of the effective medium theory used, give a good match at the low end of the WR90 band. The percentage error in the predictions remaining less than 5% from 8.2GHz to the first resonance at around 10.45GHz – see figure 69. The effective medium theory predictions using calculated C4A permeabilities in figures 67 and 68 once again overestimating the actual measured composite values. Once again the higher bias flux density calculated data gives the best fit with the error relative to the T / R measured composite permeability staying around 5% from 8.2GHz to around 10.45GHz – see figure 69.

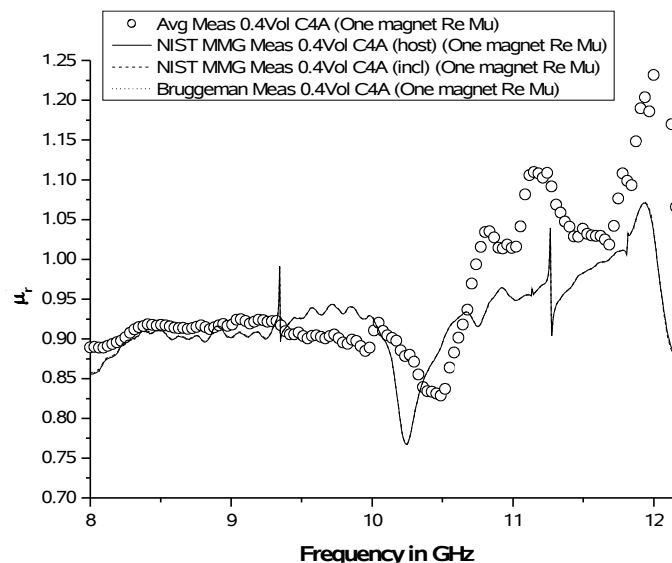


Figure 66:- Comparison of Measured Permeability of Magnetised 0.4 Volume Fraction C4A Composite with Effective Medium Theory Predictions

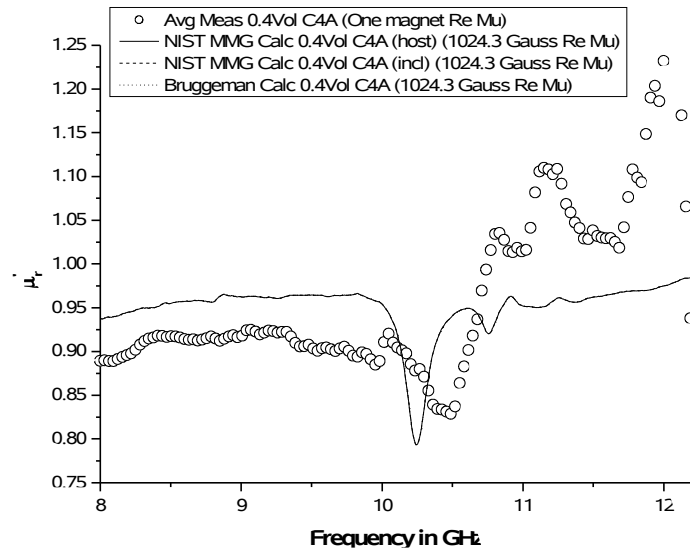


Figure 67:- Comparison of Measured Permeability of Magnetised 0.4 Volume Fraction C4A Composite with Effective Medium Theory Predictions

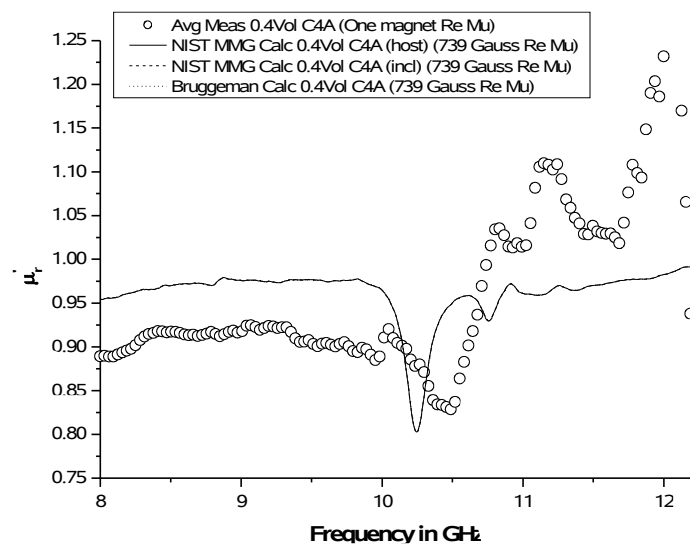


Figure 68:- Comparison of Measured Permittivity of Magnetised 0.4 Volume Fraction C4A Composite with Effective Medium Theory Predictions

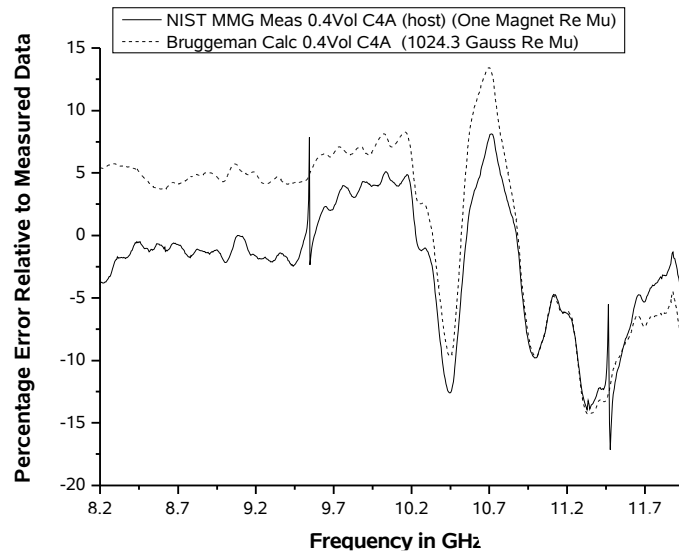


Figure 69:- Percentage Errors in Predicted Permeability of Magnetised 0.4 Volume Fraction C4A Composite

Using three magnets to increase the transverse DC magnetic flux density brought about no change in the observations regarding the predictive abilities of the Bruggeman and modified Maxwell-Garnet effective medium theories. For the 0.3 volume fraction C4A composite the best matches to the measured permittivity were again produced by the modified Maxwell-Garnett model with the garnet as host when using both the T / R measured C4A permittivity and the manufacturer's permittivity data – see figures 70 and 71. The change in accuracy of the two predictions relative to the T / R measured composite permittivity is negligible in comparison with that produced with a single DC permanent magnet as shown in figure 72. This is to be expected given that the permittivity should be and is unaffected by the change in the transverse bias field.

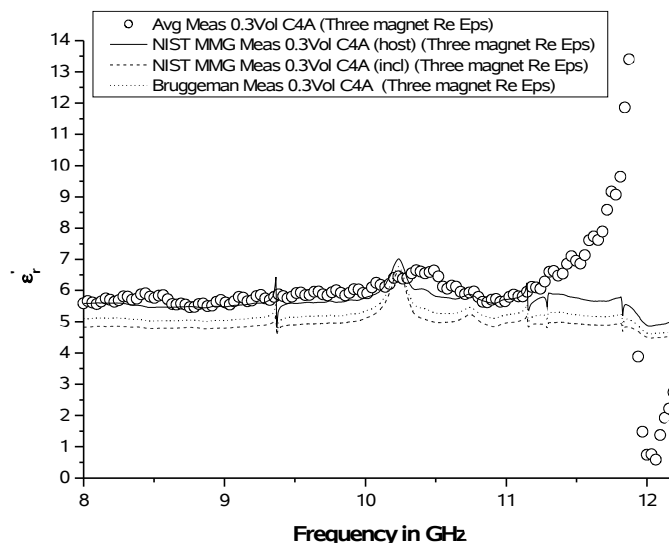


Figure 70:- Comparison of Measured Permittivity of Magnetised 0.3 Volume Fraction C4A Composite with Effective Medium Theory Predictions

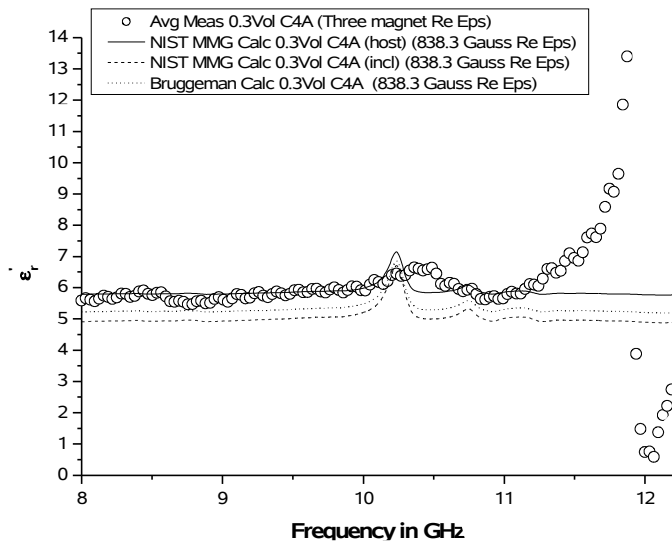


Figure 71:- Comparison of Measured Permittivity of Magnetised 0.3 Volume Fraction C4A Composite with Effective Medium Theory Predictions

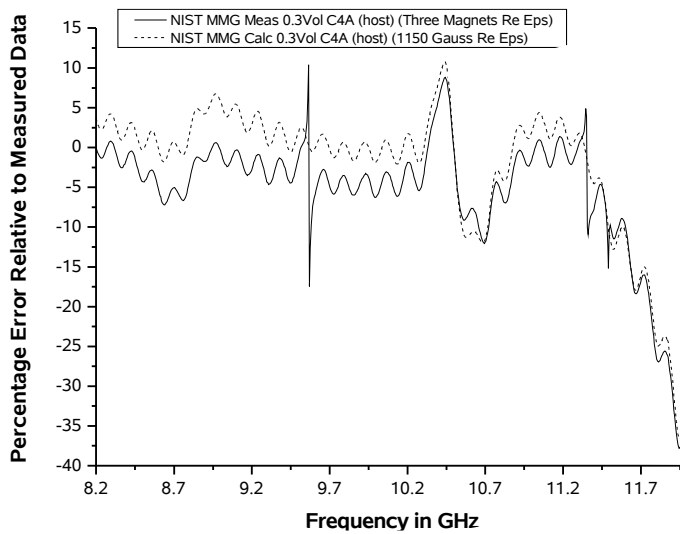


Figure 72:- Percentage Errors in Predicted Permittivity of Magnetised 0.3 Volume Fraction C4A Composite

Comparing the measured permeability data with the effective medium theory predictions in figures 73, 74 and 75 the same pattern encountered with the lower DC bias field is shown. Predicted composite permeabilities using T / R measured magnetised C4A permeabilities once again slightly underestimate the actual composite's permeability while those predictions made using theoretical values of effective C4A scalar permeability overestimate, the higher flux density data providing the closer match – see figures 74 and 75. In terms of percentage errors relative to the measured composite permeability there are very minor changes with the increased bias field. The modified Maxwell-Garnet model using measured garnet permeability displays a minor, 1 – 2%, degradation in accuracy while the Bruggeman model using calculated C4A permeabilities remains essentially unchanged – see figure 76.

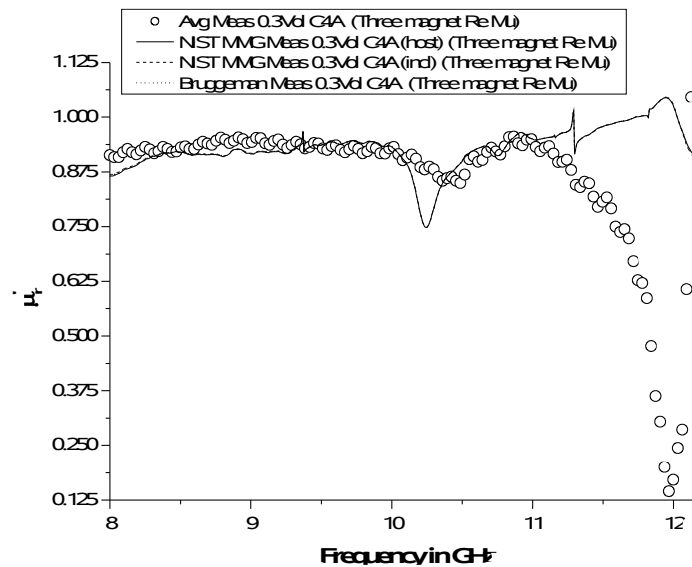


Figure 73:- Comparison of Measured Permeability of Magnetised 0.3 Volume Fraction C4A Composite with Effective Medium Theory Predictions

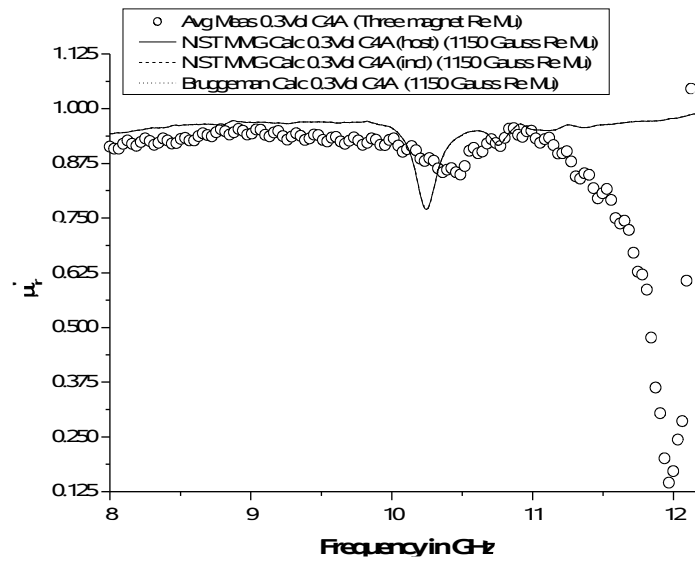


Figure 74:- Comparison of Measured Permeability of Magnetised 0.3 Volume Fraction C4A Composite with Effective Medium Theory Predictions

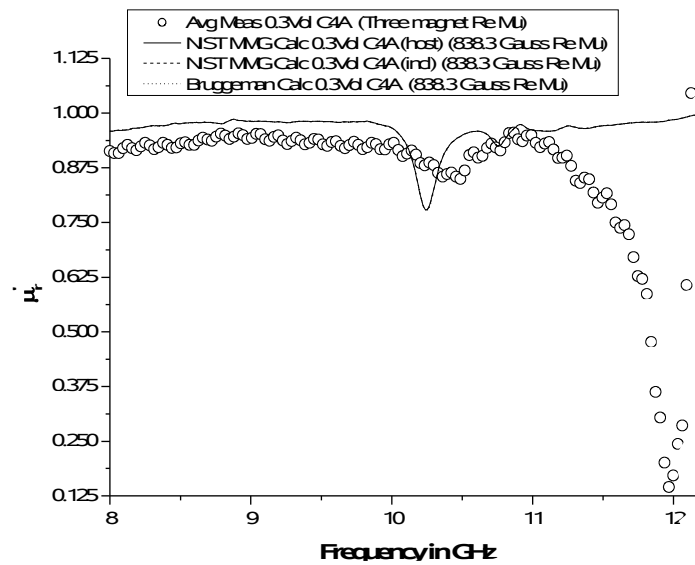


Figure 75:- Comparison of Measured Permeability of Magnetised 0.3 Volume Fraction C4A Composite with Effective Medium Theory Predictions

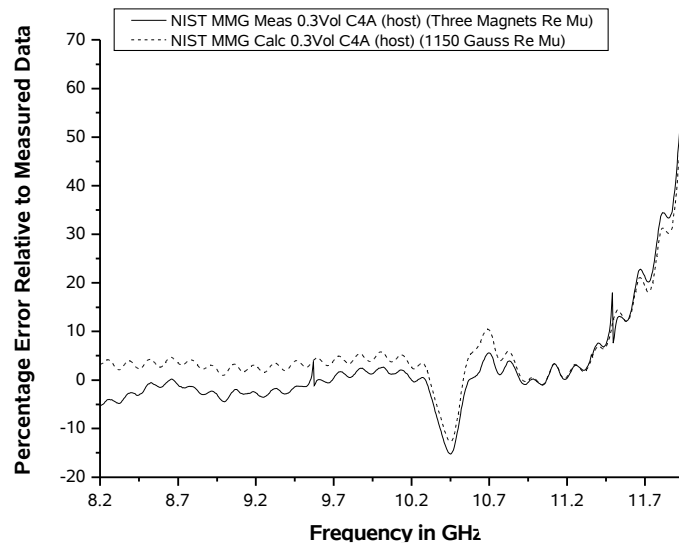
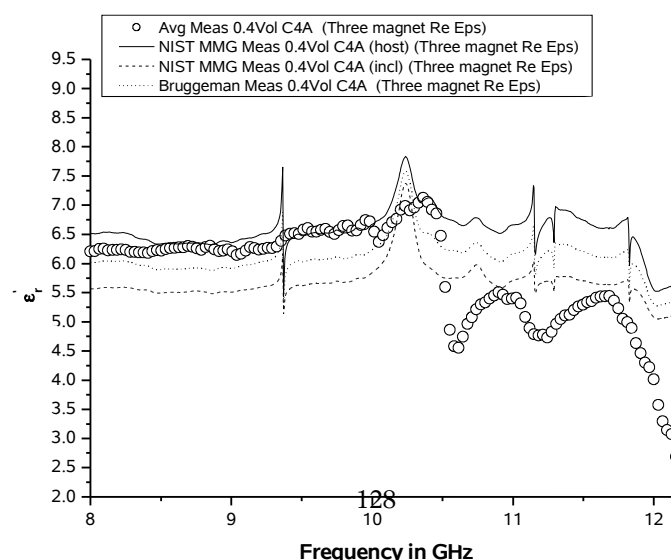


**Figure 76:- Percentage Errors in Predicted Permeability of Magnetised 0.3
Volume Fraction C4A Composite**

The results for the 0.4 volume fraction C4A composite were similarly unchanged by the increase in the bias field. Once again the best match to the measured composite permittivity being provided by the modified Maxwell-Garnet model using T / R measured C4A permittivity with the garnet designated as host and the Bruggeman model using the manufacturer's permittivity data – see figures 77 and 78. There is a small improvement in the match between the Bruggeman prediction and the measured composite permittivity from 8.2GHz to 10.7GHz and a correspondingly small, 1 – 2%, degradation in the match with the modified Maxwell-Garnett model – see figure 79.



Figured 77:- Comparison of Measured Permittivity of Magnetised 0.4 Volume Fraction C4A Composite with Effective Medium Theory Predictions

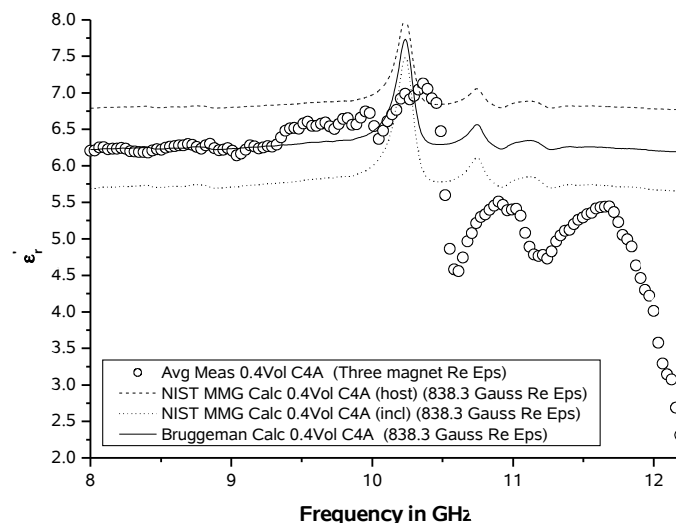


Figure 78:- Comparison of Measured Permittivity of Magnetised 0.4 Volume Fraction C4A Composite with Effective Medium Theory Predictions

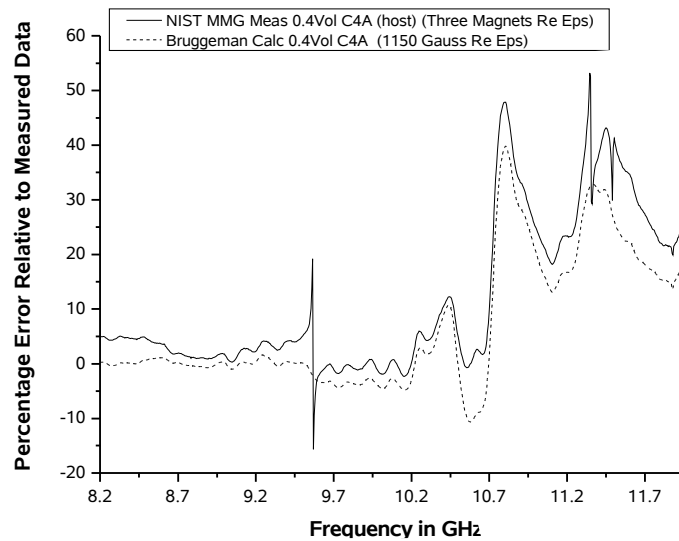


Figure 79:- Percentage Errors in Predicted Permittivity of Magnetised 0.4 Volume Fraction C4A Composite

Permeability data for the 0.4 volume fraction C4A composite is shown in figures 80, 81 and 82. As with the higher bias field 0.3 volume fraction data the changes are fairly minor. The modified Maxwell-Garnett prediction using T / R measured magnetised C4A permeabilities performs more poorly in relation to the measured composite permittivity with the higher bias field. The percentage error in the predicted values having increased by up to 5% over the 8.2GHz to 10.2GHz region – see figure 83. The Bruggeman model using theoretical values of C4A permeability seems to give exactly the same error when using three rather than one biasing DC magnet.

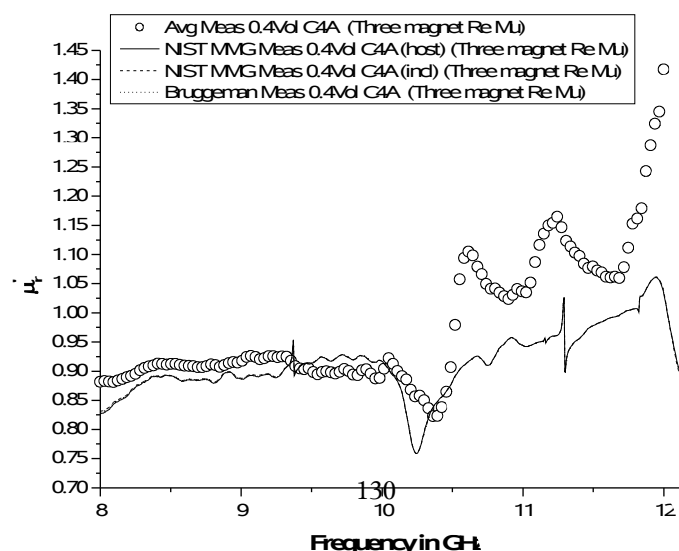


Figure 80:- Comparison of Measured Permeability of Magnetised 0.4 Volume Fraction C4A Composite with Effective Medium Theory Predictions

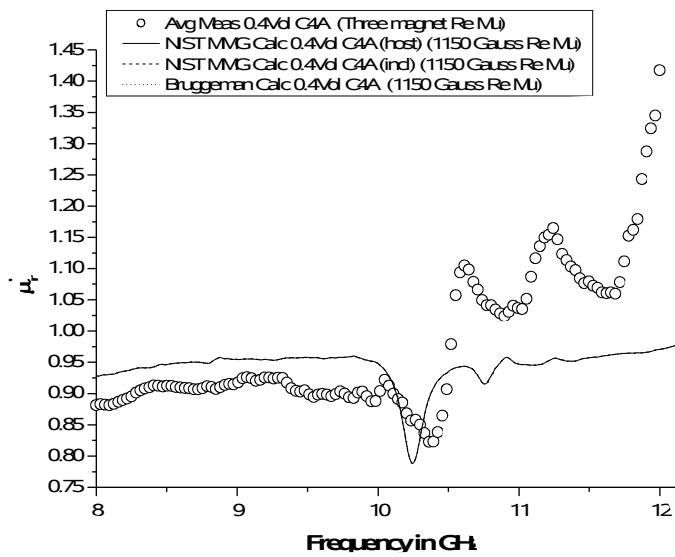


Figure 81:- Comparison of Measured Permeability of Magnetised 0.4 Volume Fraction C4A Composite with Effective Medium Theory Predictions

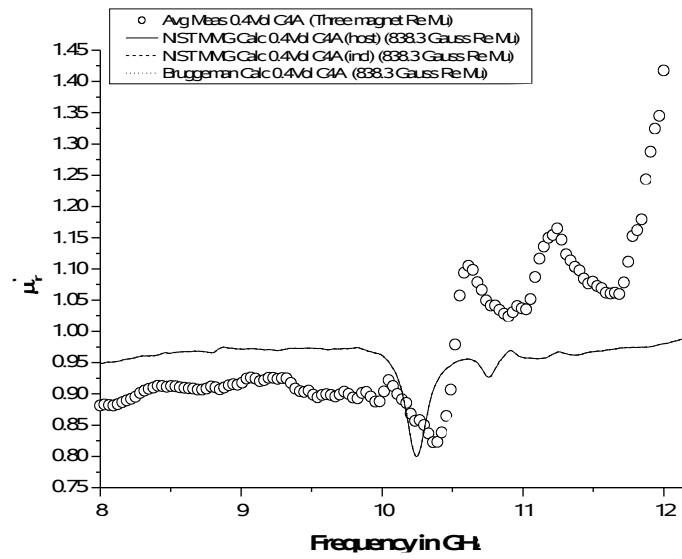


Figure 82:- Comparison of Measured Permeability of Magnetised 0.4 Volume Fraction C4A Composite with Effective Medium Theory Predictions

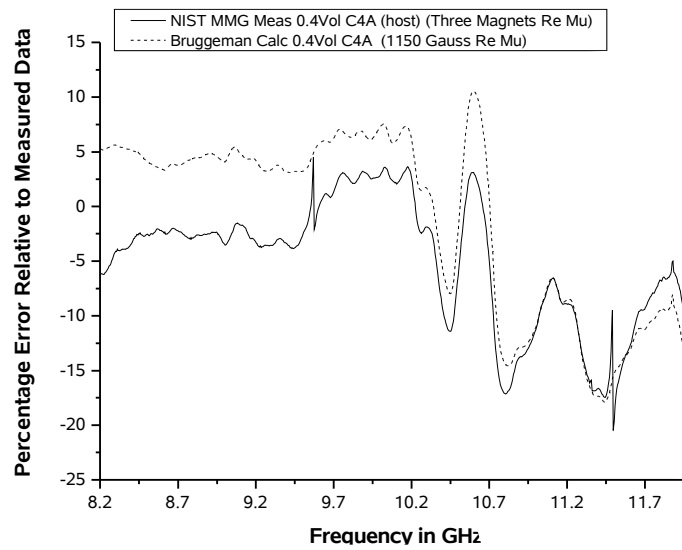


Figure 83:- Percentage Errors in Predicted Permeability of Magnetised 0.4 Volume Fraction C4A Composite

Figures 84 and 85 compare the T / R measured permittivity of a 0.3 volume fraction C6A composite transversely magnetised by a single permanent magnet with effective medium theory predictions. Continuing to use the modified Maxwell-Garnett model with the C6A as host using T / R measured permittivity and the Bruggeman model supplied with the manufacturer's permittivity gives a slight decrease in predictive accuracy. As shown in figure 86 the error in both predictions relative to the measured composite permittivity, unchanged from the unmagnetised sample, is around 5% from 8.2GHz to the sharp resonance at 9.7GHz. This is around 2.5% less accurate than was the case for the unmagnetised sample with a similar decrease in accuracy as the measurement frequency rises – see figures 46 and 86.

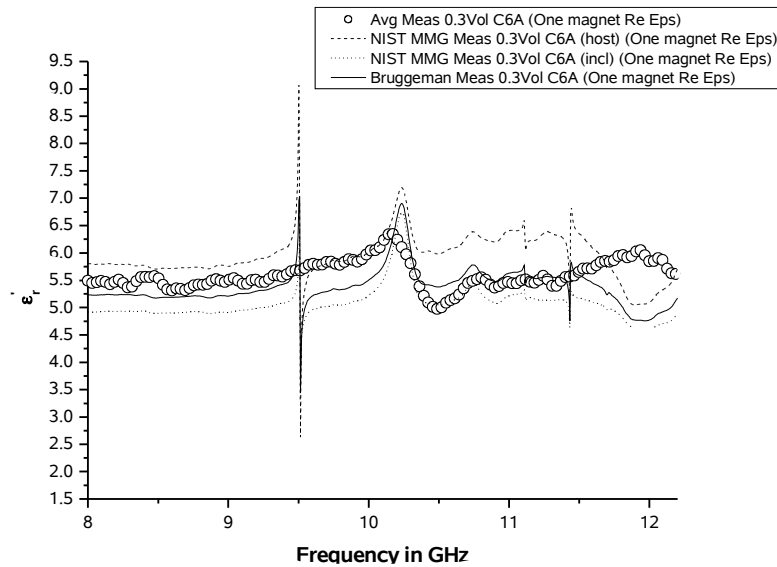


Figure 84:- Comparison of Measured Permittivity of Magnetised 0.3 Volume Fraction C6A Composite with Effective Medium Theory Predictions

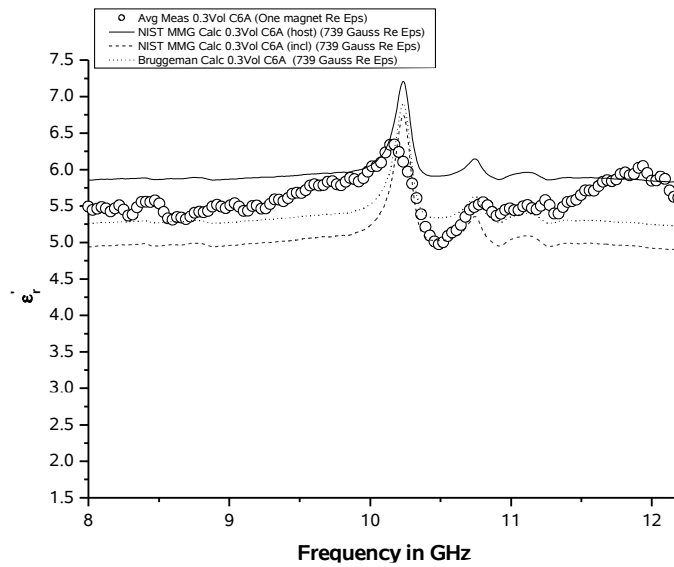


Figure 85:- Comparison of Measured Permittivity of Magnetised 0.3 Volume Fraction C6A Composite with Effective Medium Theory Predictions

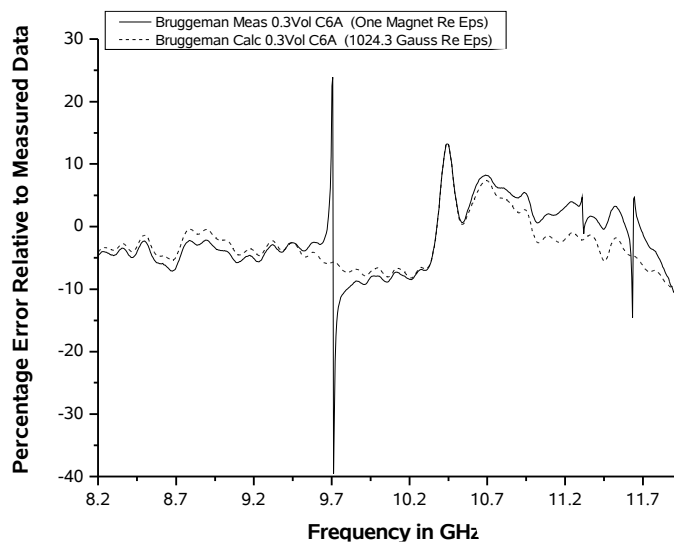


Figure 86:- Percentage Errors in Predicted Permittivity of Magnetised 0.3 Volume Fraction C6A Composite

The permeability comparisons of figures 87, 88 and 89 do not show the same level of match as was observed for the unmagnetised composite. As with all previous permeability comparisons there is negligible difference in the values predicted by the Bruggeman and modified Maxwell-Garnett models. Also repeated is the underestimation of the composite permeability when using T / R measured C6A properties in the effective medium theory calculations, overestimation when using theoretically calculated garnet properties with the closer match produced when using garnet permeabilities calculated at the highest bias field. In terms of the accuracy of the predictions relative to the measured composite there is around a 2.5% degradation in accuracy in the predictions using calculated garnet properties from 8.2GHz to 11.7GHz while the measurements based prediction suffers an initial 12.5% increase in error from 8.2 to 8.45GHz before following the pattern just described – see figure 90.

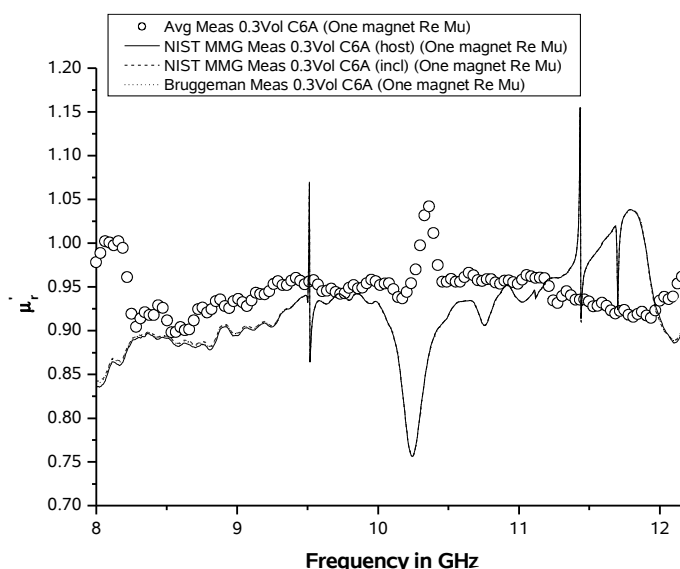


Figure 87:- Comparison of Measured Permeability of Magnetised 0.3 Volume Fraction C6A Composite with Effective Medium Theory Predictions

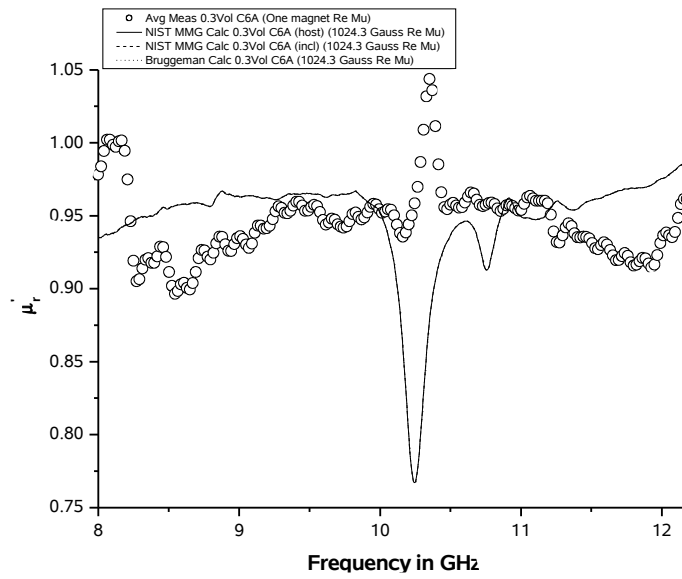


Figure 88:- Comparison of Measured Permeability of Magnetised 0.3 Volume Fraction C6A Composite with Effective Medium Theory Predictions

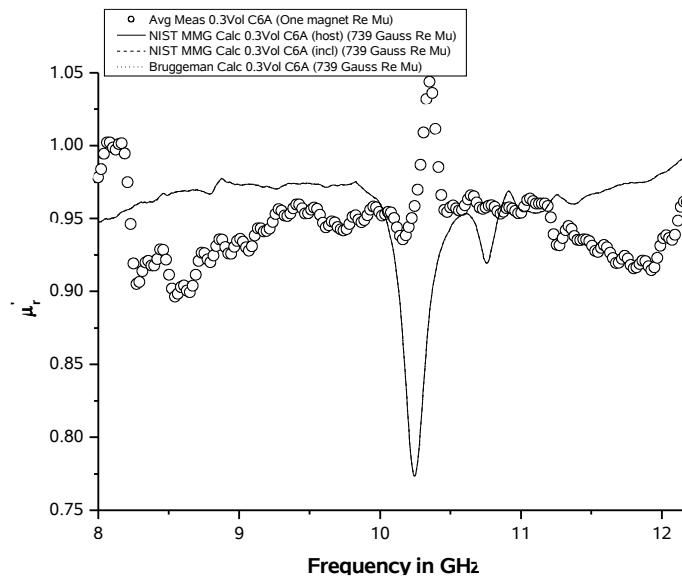


Figure 89:- Comparison of Measured Permeability of Magnetised 0.3 Volume Fraction C6A Composite with Effective Medium Theory Predictions

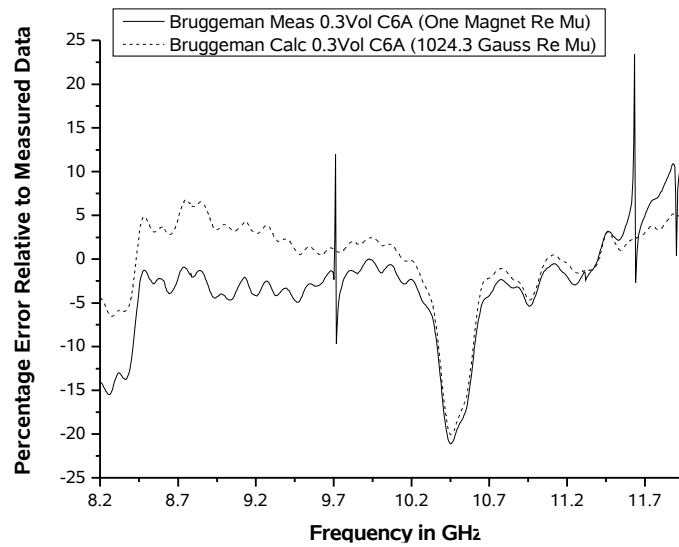


**Figure 90:- Percentage Errors in Predicted Permeability of Magnetised 0.3
Volume Fraction C6A Composite**

The measured permittivity of a transversely magnetised 0.4 volume fraction C6A composite in figures 91 and 92 is essentially identical to that of the unmagnetised composite – see figure 50. What has changed is which of the effective medium theory predictions gives the best match to the measured data. For the magnetised composite this is now the Bruggeman model regardless of the source of the C6A input permittivity data. With the unmagnetised sample this was only true when using the manufacturer’s permittivity value not the T / R measured permittivity which worked best with the modified Maxwell-Garnett model – see figure 50. The most likely cause of this change is the much closer match observed and discussed earlier between the manufacturer’s permittivity and the value measured with a transversely magnetised C6A sample. In terms of the error in the Bruggeman predictions of the magnetised composite permittivity, leaving aside the high Q resonance at 9.7GHz, there is no real change relative to the unmagnetised case. As shown in figure 93 the

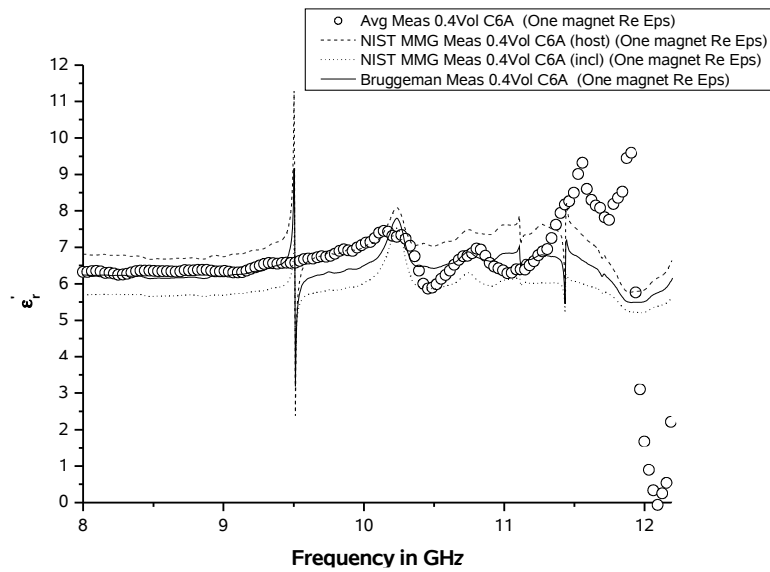


Figure 91:- Comparison of Measured Permittivity of Magnetised 0.4 Volume Fraction C6A Composite with Effective Medium Theory Predictions

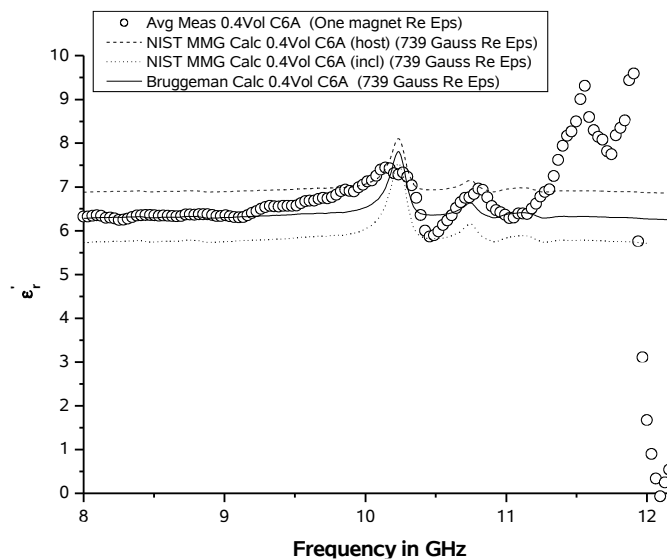


Figure 92:- Comparison of Measured Permittivity of Magnetised 0.4 Volume Fraction C6A Composite with Effective Medium Theory Predictions

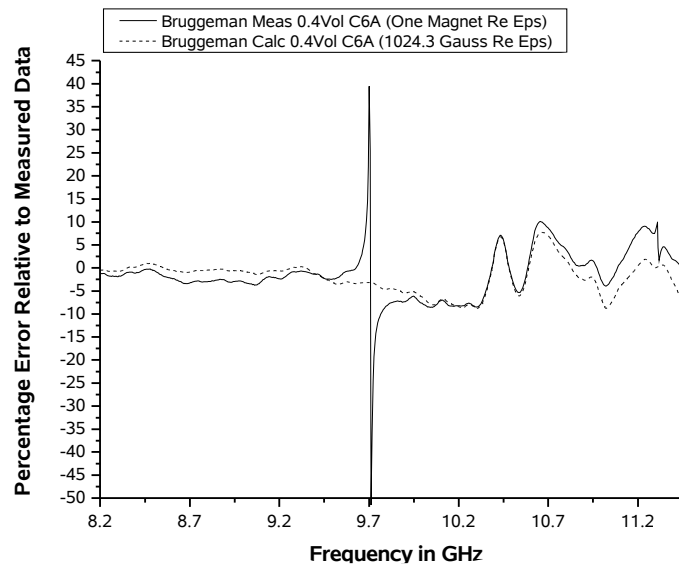


Figure 93:- Percentage Errors in Predicted Permittivity of Magnetised 0.4 Volume Fraction C6A Composite

predicted permittivities remain within 10% of the measured value from 8.2GHz to 11.2GHz.

T / R measured composite permeability is shown alongside the effective medium theory predictions in figures 94, 95 and 96. Continuing to use the Bruggeman model as having the best predictive ability for the transversely magnetised composite there is some noticeable degradation in the accuracy of the predictions compared to what was achieved for an unmagnetised 0.4 volume fraction sample. When using the T / R measured permeability of a transversely magnetised C6A sample in the Bruggeman model there is an increase in the error of the model's prediction by around 2.5% from 8.2GHz to around 9GHz – see figure 97. Thereafter though the error relative to the measured permeability remains within 7.5% out to 11.2GHz which is an improvement over that observed with the unmagnetised composite – see figure 55.

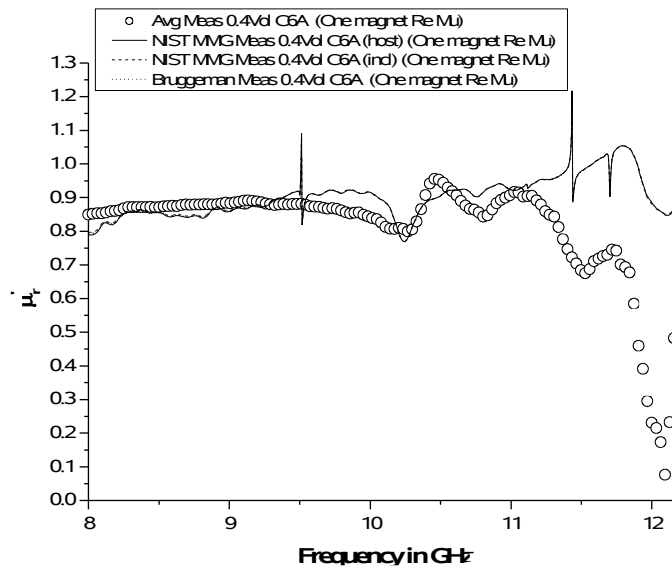


Figure 94:- Comparison of Measured Permeability of Magnetised 0.4 Volume Fraction C6A Composite with Effective Medium Theory Predictions

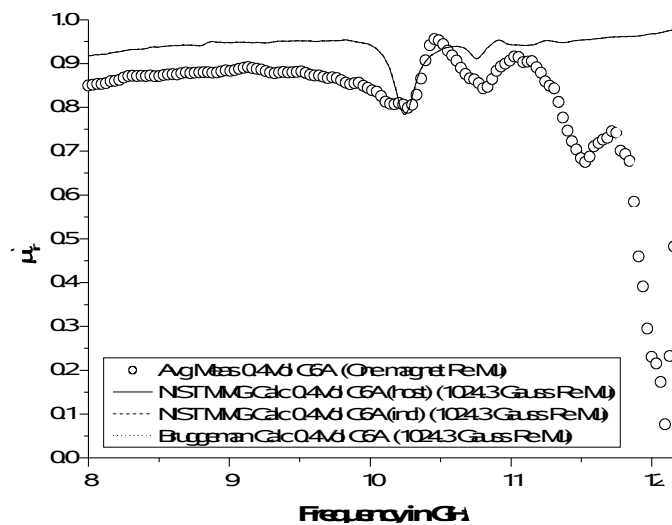


Figure 95:- Comparison of Measured Permeability of Magnetised 0.4 Volume Fraction C6A Composite with Effective Medium Theory Predictions

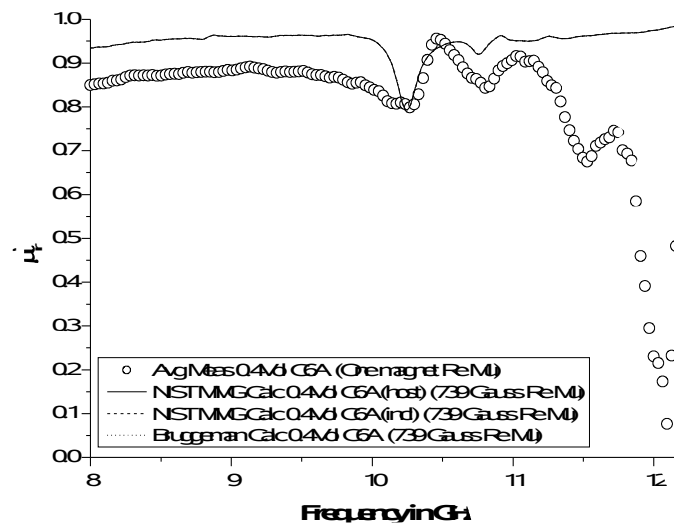


Figure 96:- Comparison of Measured Permittivity of Magnetised 0.4 Volume Fraction C6A Composite with Effective Medium Theory Predictions

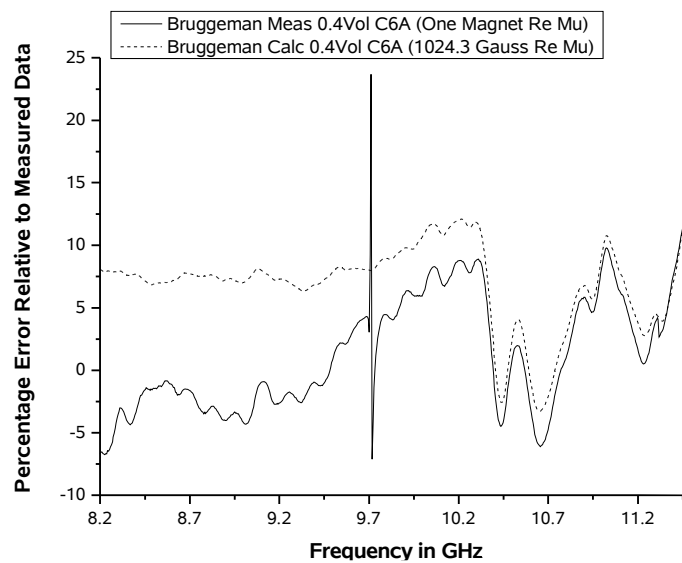


Figure 97:- Percentage Errors in Predicted Permeability of Magnetised 0.4 Volume Fraction C6A Composite

Using theoretical values of C6A permeability in the Bruggeman model there is also a decrease in the accuracy of predicted properties. In comparison with the unmagnetised sample the Bruggeman predictions for the magnetised sample are around 5% less accurate, 7.5% in error relative to the T / R measured composite permeability, from 8.2GHz to 9.7GHz but from this frequency onwards the accuracy of prediction is the same as for the unmagnetised case.

As with the C4A composites described earlier increasing the transverse magnetic bias field had no real impact upon the effective medium theory predictions. Figures 98 and 99 compare the T / R measured permittivity of a 0.3 volume fraction C6A composite transversely magnetised by three permanent magnets.

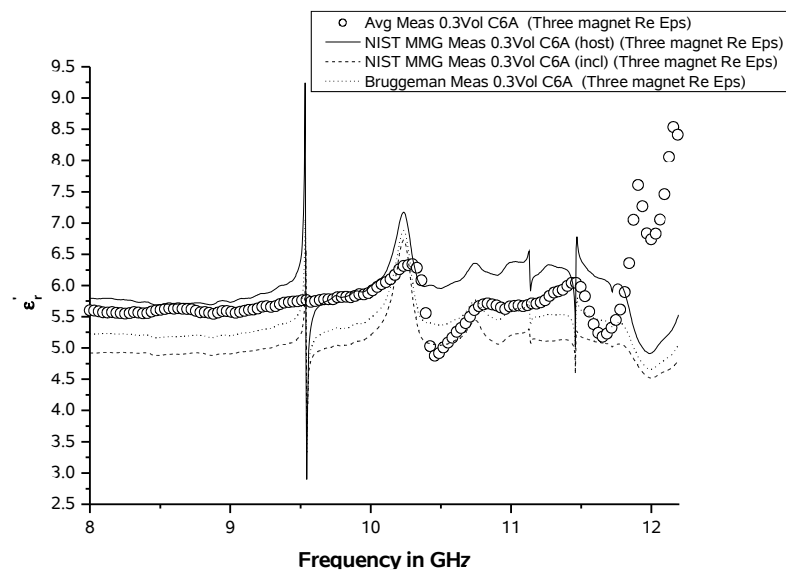


Figure 98:- Comparison of Measured Permittivity of Magnetised 0.3 Volume Fraction C6A Composite with Effective Medium Theory Predictions

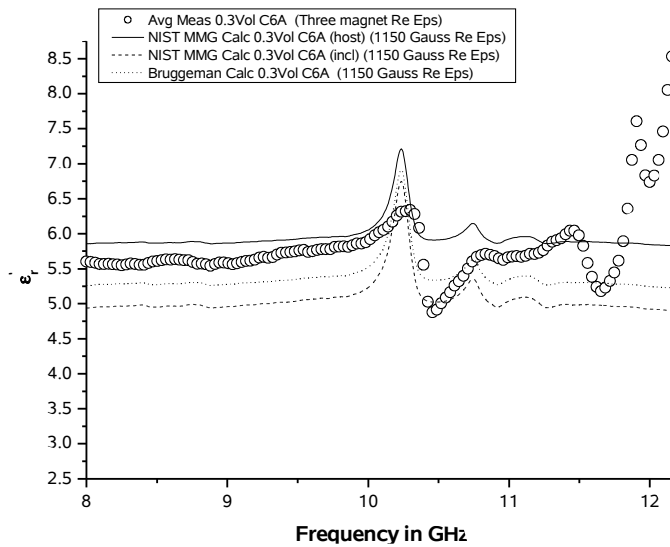


Figure 99:- Comparison of Measured Permittivity of Magnetised 0.3 Volume Fraction Composite with Effective Medium Theory Predictions

As expected the measured permittivity is the same as for a single biasing magnet however looking at figure 99, predictions made using the manufacturer's permittivity data, it is no longer clear cut which effective medium theory best predicts the composite's permittivity. Evaluating the error in both the Bruggeman and modified Maxwell-Garnett models (C6A as host), the latter using both T / R measured and manufacturer supplied permittivities, reveals equal levels of inaccuracy in the two models' predictions – see figure 100. The predicted permittivities are within 5% of the measured values in all three cases, albeit with occasional deviations of greater than 10%, from 8.2GHz to 11.7GHz. Referring back to figure 86, effective medium theory errors at the lower bias field, shows an equivalent level of performance.

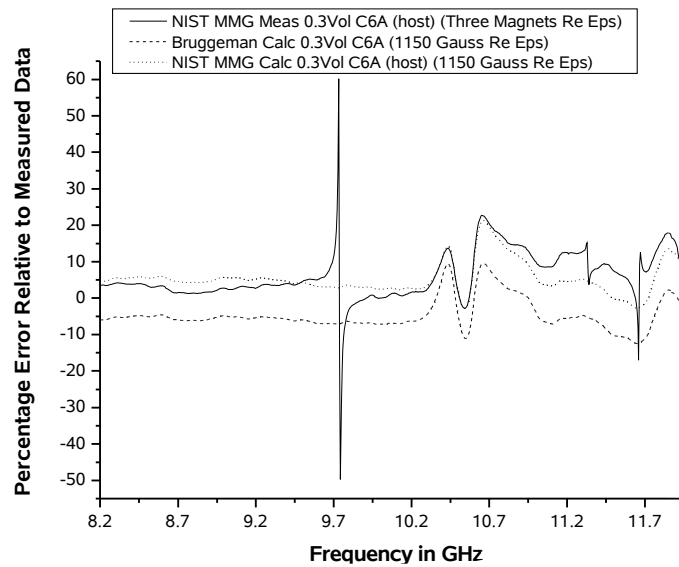


**Figure 100:- Percentage Errors in Predicted Permittivity of Magnetised 0.3
Volume Fraction C6A Composite**

Figures 101, 102 and 103 compare the composite's measured permeability with effective medium theory predictions based upon both T / R measured and theoretical values of magnetised C6A permeability. As with all of the other magnetised composite samples the best match to the measured permeability using theoretical garnet properties is given when using the maximum measured transverse magnetic flux density which for three magnets was 1150 Gauss (91.54 kAm^{-1}) – see figure 102. Also repeated from both the magnetised and unmagnetised samples is the fact that only one of the three theoretical garnet permeability predictions made using the Bruggeman and modified Maxwell-Garnett models is actually visible in figures 102 and 103.

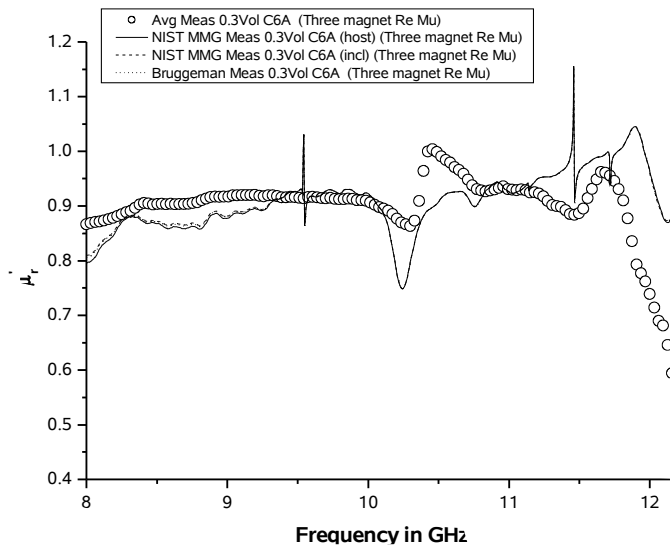


Figure 101:- Comparison of Measured Permeability of Magnetised 0.3 Volume Fraction C6A Composite with Effective Medium Theory Predictions

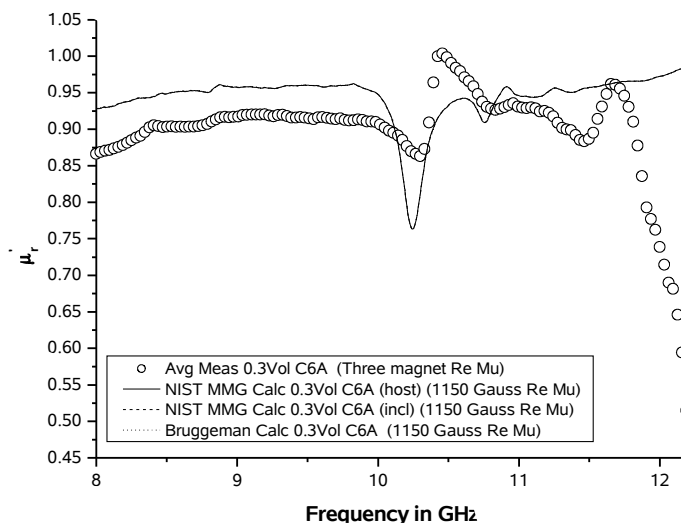


Figure 102:- Comparison of Measured Permeability of Magnetised 0.3 Volume Fraction C6A Composite with Effective Medium Theory Predictions

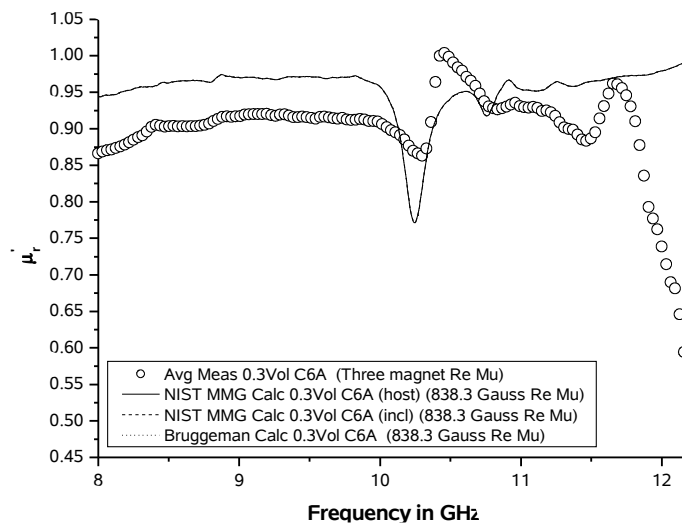


Figure 103:- Comparison of Measured Permeability of Magnetised 0.3 Volume Fraction C6A Composite with Effective Medium Theory Predictions

In terms of the percentage errors in the effective medium theory predictions relative to the T / R measured permeability of the composite there is very little difference in the result with three magnets compared to that achieved with just one – see figures xx and 104. Between 8.2 and 8.45GHz the three magnet predictions using the modified Maxwell-Garnett model with measured C6A permeabilities (garnet as host) is twice as accurate as the Bruggeman model was with measured C6A permeabilities at the lower bias field. From 8.45GHz upwards there is no real difference in accuracy between the three magnet predictions and the one magnet data presented previously. Increasing the bias field seems to have absolutely no effect upon the accuracy of the effective medium theory predictions when using theoretical garnet permeabilities. Errors in predictions essentially remaining within 10%, ignoring the Crystalbond™ resonance at 10.2GHz, from 8.2GHz to around 11.5GHz – see figure 104.

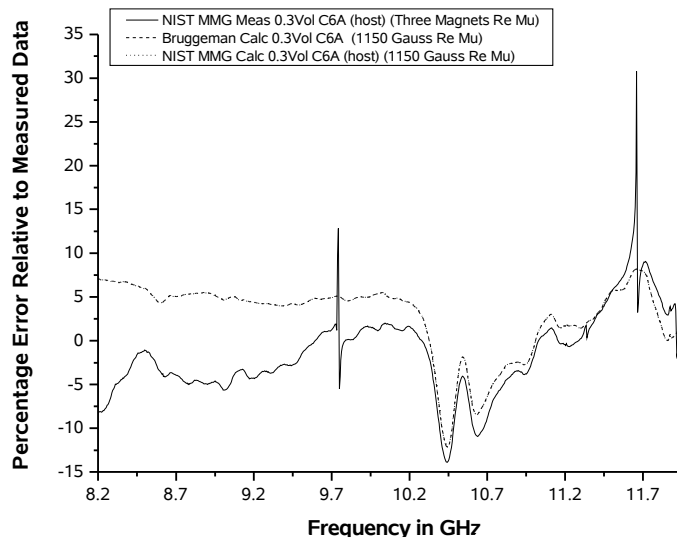


Figure 104:- Percentage Errors in Predicted Permeability of Magnetised 0.3 Volume Fraction C6A Composite

Figures 105 and 106 show the T / R measured permittivity of a 0.4 volume fraction C6A composite transversely magnetised by three permanent magnets. The data is essentially identical to that produced at the lower bias field – see figures 91 and 92. With the higher bias field the Bruggeman effective medium theory, regardless of the source of the permittivity data, gives the best match to the measured composite permittivity. The percentage errors in the two sets of Bruggeman predictions in figure 107 are also identical to those produced at the lower bias field i.e. error on the order of 1% from 8.2GHz to 9.7GHz and then within 10% from 9.7GHz to the onset of higher order mode resonances at 11.45GHz.

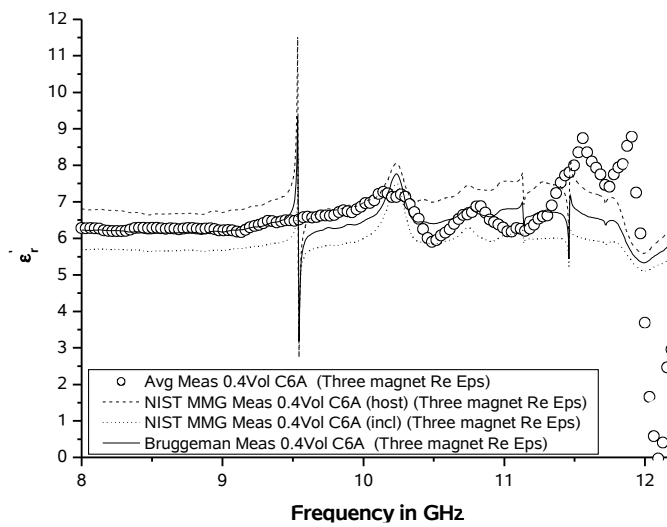


Figure 105:- Comparison of Measured Permittivity of Magnetised 0.4 Volume Fraction C6A Composite with Effective Medium Theory Predictions

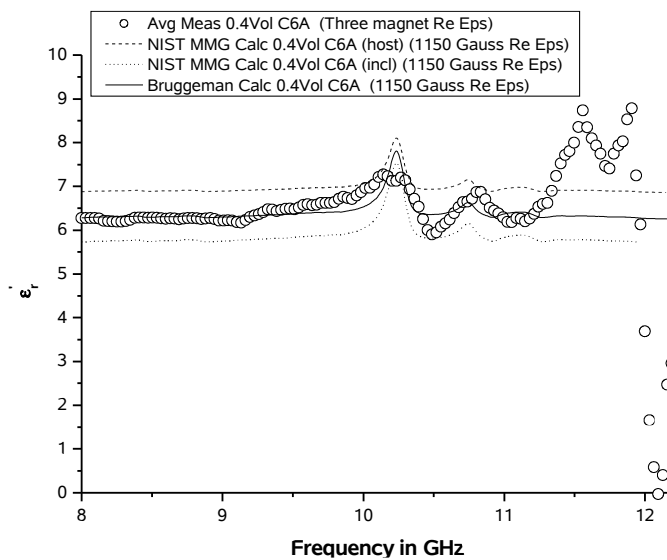


Figure 106:- Comparison of Measured Permittivity of Magnetised 0.4 Volume Fraction C6A Composite with Effective Medium Theory Predictions

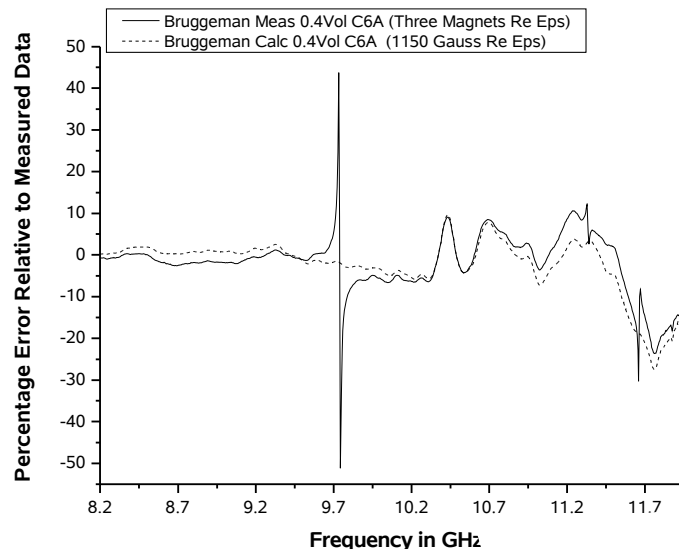


Figure 107:- Percentage Errors in Predicted Permittivity of Magnetised 0.4 Volume Fraction C6A Composite

The permeability data for the 0.4 volume fraction composite in figures 108, 109 and 110 also shows very little change with increased transverse magnetisation. The Bruggeman model using T / R measured permeability of a transversely magnetised sample again underestimating the actual composite properties while effective medium theory predictions using theoretical values of garnet permeability once again are overestimates. The percentage error in the measurement and theoretical garnet property based predictions are also essentially unchanged from the lower bias field condition. There is a 1 – 2% increase in the error of both Bruggeman predictions at 8.2GHz, to 10% and 7.5% respectively, after which the accuracy is identical to that of the lower magnetisation sample i.e. predictions within 10% of the T / R measured composite permeability from 8.2GHz to 11.2GHz – see figures 97 and 111.

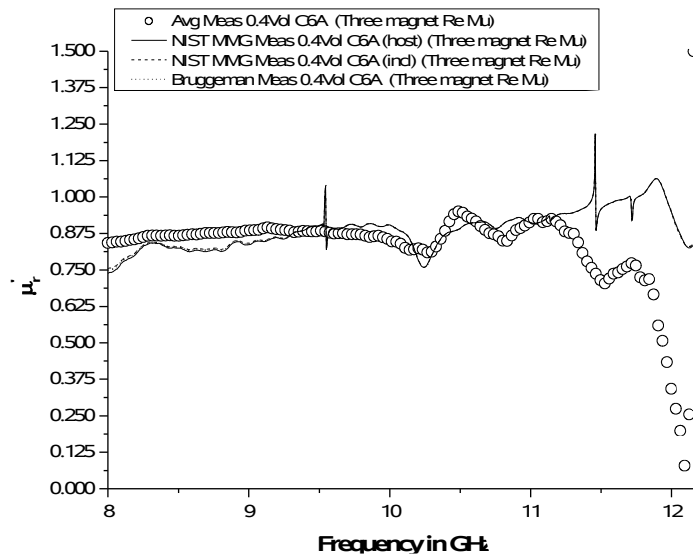


Figure 108:- Comparison of Measured Permeability of Magnetised 0.4 Volume Fraction C6A Composite with Effective Medium Theory Predictions

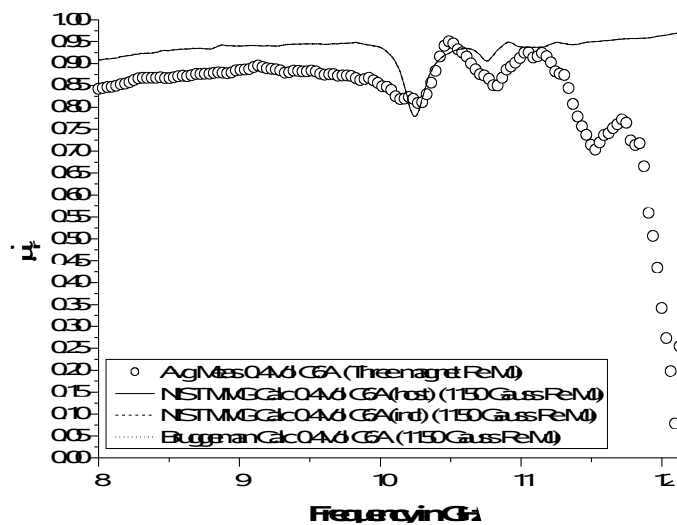
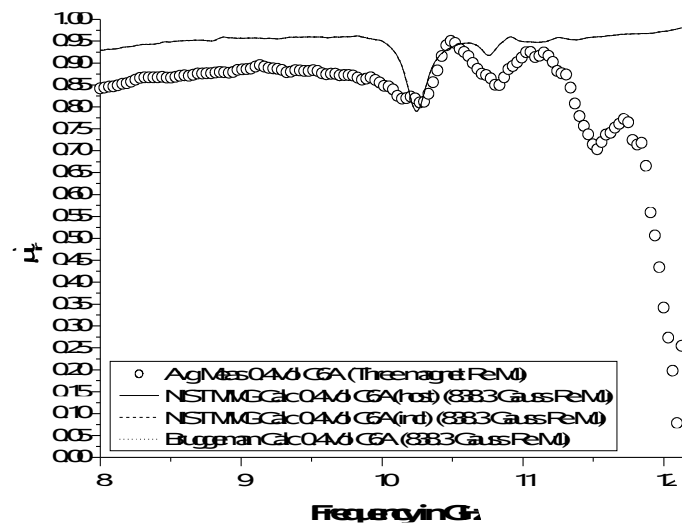


Figure 109:- Comparison of Measured Permeability of Magnetised 0.4 Volume Fraction C6A Composite with Effective Medium Theory Predictions



Figured 110:- Comparison of Measured Permeability of Magnetised 0.4 Volume Fraction C6A Composite with Effective Medium Theory Predictions

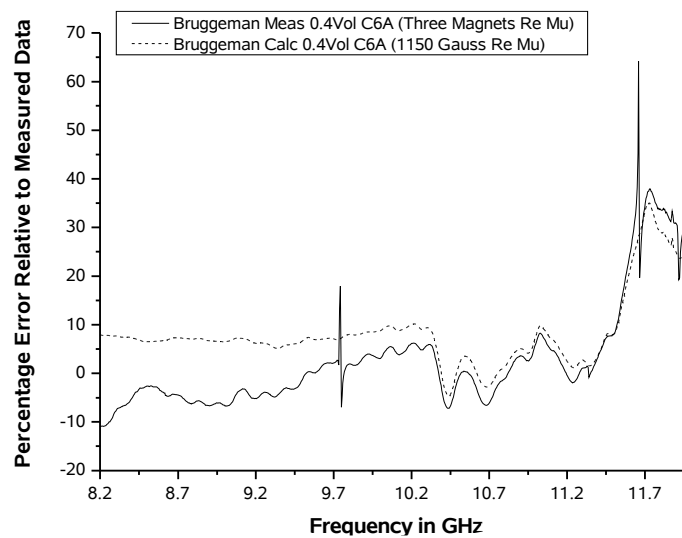


Figure 111:- Percentage Errors in Predicted Permeability of Magnetised 0.4 Volume Fraction C6A Composite

The preceding pages have demonstrated the successful design and implementation of a two-port WR90 rectangular waveguide system for the characterisation of solid dielectric, garnet and garnet-loaded composite samples. The system was used to confirm the suitability and accuracy of the Bruggeman and NIST-modified Maxwell-Garnett effective medium theories in predicting the permittivities and permeabilities of unmagnetised and transversely magnetised garnet-loaded composites. These comparisons demonstrated that in general the modified Maxwell-Garnett provided the best match to observed composite properties when supplied with T / R measured garnet properties and with the garnet specified as host material. Predictions made using the Bruggeman effective medium theory supplied with the manufacturer's permittivity data and theoretical garnet permeabilities were found to be as accurate and occasionally more accurate than those of the modified Maxwell-Garnett model using actual T / R measured garnet properties. In both cases predicted permittivities and permeabilities were within 5% of measured values from 8.2GHz to between 10.2 and 10.95GHz.

For the two combinations of volume fraction and Indium-substituted microwave garnets considered it seems to be possible to make reasonably accurate, within 15% worst-case error across the whole WR90 band, predictions of the properties of both magnetised and transversely magnetised composites using just the Bruggeman effective medium theory and information normally supplied by a manufacturer's datasheet i.e. a single frequency value of permittivity and the materials' saturation magnetisations. The highly accurate nature of the predictions possible from 8.2GHz to between 10.2 and 10.45GHz is put into perspective by the effect of the $\pm 5\%$ uncertainty in garnet permittivities and saturation magnetisations specified by the manufacturer. Propagating these values through both the Bruggeman and modified

Maxwell-Garnett (Ca-V garnet as host) effective medium theories gives worst case uncertainties in predicted permittivities of $\pm 0.749\%$ and $\pm 1.129\%$ respectively for 0.3 volume fraction composites. Worst case uncertainties in predicted 0.4 volume fraction composites permittivities are slightly higher at $\pm 1.026\%$ and $\pm 1.1327\%$ for the Bruggeman and Maxwell-Garnett models respectively.

The impact of uncertainty in the substituted garnets' saturation magnetisations upon predicted permeabilities for magnetised composites is most significant at the beginning of the WR90 band closest to the materials' gyromagnetic resonances. This is in part because Rado's (1953) expression for the permeability tensor components κ , which is only relevant to the calculation of effective scalar permeability for magnetised garnets, was found by Green and Sandy (1974)¹ to be up to 10% in error for some materials. Uncertainty in predicted values of magnetised permeability resulting from variability in saturation magnetisations and errors in Rado's model have worst case values of $\pm 1.086\%$ for 0.3 volume fraction C4A composites and $\pm 1.452\%$ for 0.4 volume fractions. Uncertainties are slightly higher for the C6A composites due to the garnet's higher gyromagnetic resonance frequency. Worst case uncertainties in predicted permeability are therefore $\pm 1.411\%$ for 0.3 volume fractions and $\pm 1.883\%$ for 0.4 volume fraction composites.

The potential uncertainties in predicted permittivities and permeabilities presented while numerically small are actually very significant in the context of the normally less than 5% difference between actual T / R measured composite properties and 'datasheet' predictions made using the Bruggeman effective medium theory. This finding demonstrated the validity of applying the Bruggeman and modified Maxwell-

Garnett effective medium theories to predicting properties of magnetised and unmagnetised garnet loaded composites and gave confidence in the accuracy of the T / R measured properties and their suitability for use in the design of tuneable passive planar devices.

Chapter 5: Microstrip Resonator Measurements

This chapter describes the design and implementation of a measurement system to characterise linear microstrip resonators subject to DC magnetic fields applied parallel to the direction of wave propagation. The approach to the design of linear, gap-coupled microstrip resonators on microwave garnet loaded composite substrates is described. Results for unmagnetised and axially magnetised 0.3 and 0.4 volume fraction C4A and C6A garnet based composite substrates is presented. Details of substrate fabrication and microstrip line deposition are given in Appendix II.

5.1 Experimental Setup

Complex permittivity and permeability data of unmagnetised 0.3 and 0.4 volume fraction composites measured at 10.00075GHz in WR90 waveguide was used to design half-wavelength linear microstrip resonators. The assumed duality relationship between magnetic and dielectric microstrip properties of Pucel and Massé (1972) was applied to the design of the microstrip resonators. Hammerstad's (1975) equations were employed instead of Wheeler's (1965) conformal mapping derived expressions for the characteristic impedance and dielectric filling factors of microstrip lines which were used by Pucel and Massé. According to Collin (1992) Hammerstad's improved accuracy versions of Schneider's (1969) quasi-TEM analysis give calculated effective dielectric constants within 1% of an analytical solution of the integral equation analysis of microstrip. A software implementation of the Hammerstad equations, suitably modified to use the magnetic duality relationship, was used to calculate the guided wavelengths and characteristic impedances of microstrip resonators assuming 1 μ m thick metallisation and a 1mm substrate thickness. This was done for both forward (S_{11} , S_{21}) and reverse (S_{22} , S_{12}) measured material properties for microstrip linewidths of 1.3, 1.5, 1.55 and 1.6mm. Linewidths for the two datasets giving characteristic impedances closest to 50 Ω were selected for fabrication – see Appendix II for details. Resonator lengths were shortened from the initial exact calculated half- wavelength values to take account of the additional capacitance introduced by the input and output coupling gaps. These were chosen to try and ensure only light resonator loading, intended insertion losses of 15 to 20dB, to minimise Q degradation and frequency shifting. The capacitance compensating length change to the actual resonators was calculated using the closed form expressions given by Gupta et al (1996). The dimensions of the linear

microstrip resonators fabricated are summarised in table 1. As described in Appendix II all eight resonators used 320 μ m wide coupling gaps.

Garnet Material and Volume Fraction	Resonator Linewidth in Millimetres	Resonator Length in Millimetres
C4A 0.3	1.5	7.3046
C4A 0.3	1.5	7.2574
C4A 0.4	1.3	7.0738
C4A 0.4	1.3	7.0738
C6A 0.3	1.6	7.54718
C6A 0.3	1.6	7.24618
C6A 0.4	1.5	7.13845
C6A 0.4	1.3	7.0391

Table 1: Dimensions of Fabricated Composite Microstrip Resonators

Composite microstrip resonator testing used 3.5mm coaxial-microstrip transitions mounted on nanometre resolution optical fibre x-y-z alignment stages – see figure 112. The transitions and substrates were bolted to a section of optical breadboard so that probe positioning and substrate magnetisation were highly repeatable.

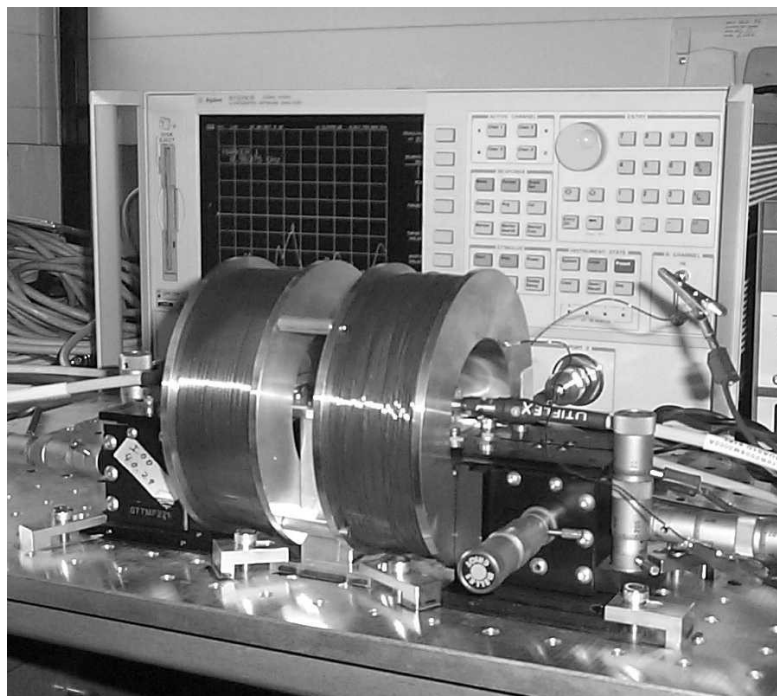


Figure 112:- Magnetised Microstrip Resonator Test Setup

As shown in figure 112 magnetic tunability of the resonators was examined using a Helmholtz coil arrangement of two air-cored solenoids to generate DC magnetic bias fields. The composite substrate under test was bolted to a grounded Aluminium support pillar located in the centre of the bore of the two solenoids. This ensured that the DC magnetic field generated by the Helmholtz coil was aligned axially with the microstrip coupling lines and resonator and parallel to the direction of wave propagation i.e. perpendicular to the microwave H-field component. The individual 1265 turn solenoids were driven by the bridge rectified outputs of two step-down transformer driven variacs capable of providing up to 74V DC at 7A. Using a transverse Hall probe measurements at the centre of the microstrip resonators showed this to correspond to a maximum axial DC magnetic bias of 1059 Oerstads (81.351 kAm^{-1}) which is almost identical to the maximum field generated by a single permanent magnet in the T / R waveguide measurements. Due to the unregulated nature of the coil power supply maximum achievable bias fields varied slightly due to heating induced changes in the coils' resistance.

Before carefully lowering the coaxial-microstrip transition probes into contact with the coupling lines a full two-port OSLT coaxial calibration of the VNA was made at the test cable connectors to implement a full twelve term error correction – see Marks (1997). 1601 equally spaced datapoints from 4 – 20GHz were used for both calibration and measurements with tunability of the design resonance when subjected to axial DC magnetic bias fields examined over a narrower recalibrated measurement bandwidth. Both S_{21} and S_{12} of the resonators was observed confirming the reciprocal nature of the devices' response. As with the WR90 T / R measurements

the VNA was configured to phase-lock with its internal reference oscillator at each measurement point to ensure consistency between composite samples.

To confirm that any observed tuning of resonator centre frequency was solely due to changes in substrate permeability and not a result of movement of the x-y-z stages a 1.5mm wide, 7.3046mm long linear microstrip resonator was fabricated on a 1.27mm thick Rogers RT / Duroid 6006 microwave dielectric substrate with 17.5µm Copper cladding using standard photolithographic techniques. This nonmagnetic ceramic-loaded PTFE composite substrate was selected as its real component of permittivity of 6.15 ± 0.15 was approximately midway between that of the 0.3 and 0.4 volume fraction garnet composites under investigation. As shown in the broadband, 4 to 20GHz, plot of S_{21} the resonance of interest was located just below 9GHz – see figure 113.

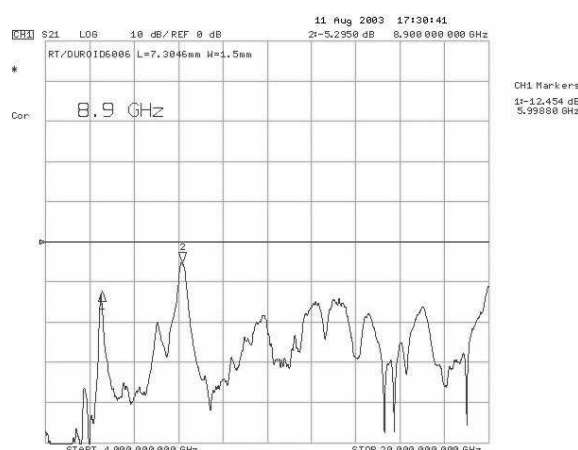


Figure 113:- Broadband Response of 7.3046mm Long RT / Duroid 6006 Linear Microstrip Resonator

Narrowing the VNA measurement bandwidth enabled the resonance centre frequency to be precisely located and stored in the VNA memory. Application of the maximum

possible axial DC magnetic bias field to the device gave no appreciable shift in centre frequency as shown in figure 114 where the magnetised and unmagnetised resonances are indistinguishable.

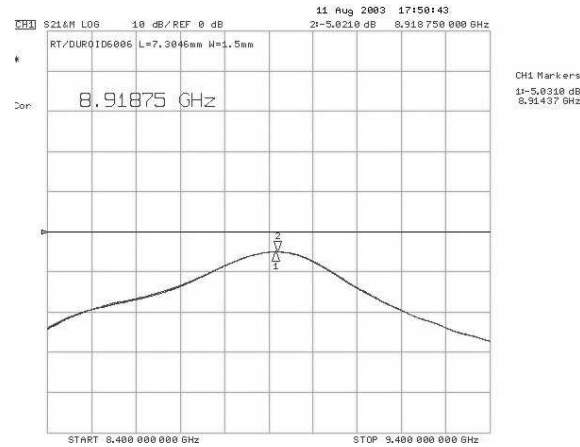


Figure 114:- Magnetised and Unmagnetised Response of 7.3046mm Long RT / Duroid 6006 Linear Microstrip Resonator

This confirmed the mechanical advantage of the micrometer driven x-y-z stages and ruled out movement of the coaxial-microstrip probes as the cause of any subsequently observed tuning of composite resonator centre frequencies.

5.2 Microstrip Resonators on 0.3 Volume Fraction C4A Composite Substrates

Figure 115 shows the unmagnetised broadband transmission response of the a 7.3046mm long microstrip resonator deposited on a 0.3 volume fraction C4A composite substrate. Reducing the measurement bandwidth considerably showed the resonance centre frequency to be 9.99128125GHz which is just 0.095% lower than the design value of 10.00075GHz – see figure 116. Q_o of the unmagnetised resonance was calculated to be 58.79.

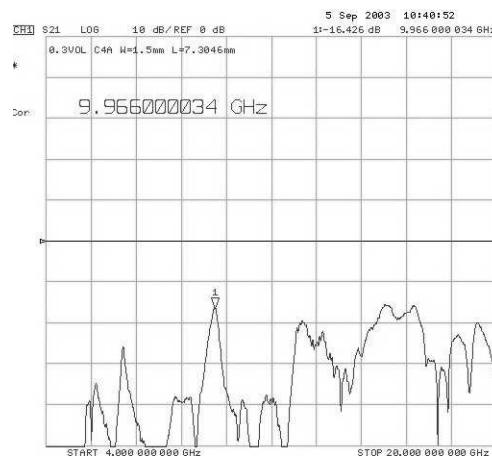


**Figure 115:- Unmagnetised Response of 7.3046mm Long Microstrip Resonator
on a 0.3 Volume Fraction C4A Composite Substrate**

As expected application of axial DC magnetic bias fields using the Helmholtz coil pair caused the measured resonant frequency to increase due to the induced decrease in the garnet fraction's permeability – see figure 117. The centre frequencies,

insertion losses and values of Q_0 for the four magnetisation conditions examined are summarised in table 2.

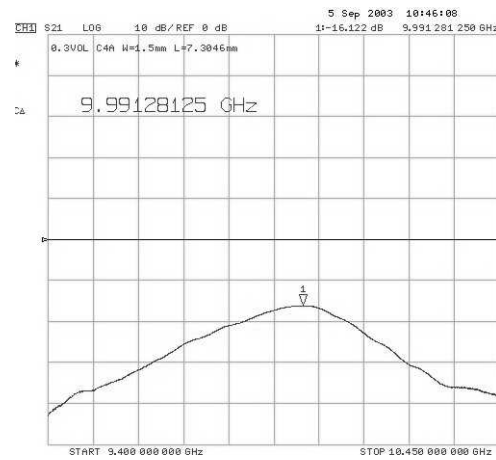


**Figure 116:- Unmagnetised Response of 7.3046mm Long Microstrip Resonator
on a 0.3 Volume Fraction C4A Composite Substrate**

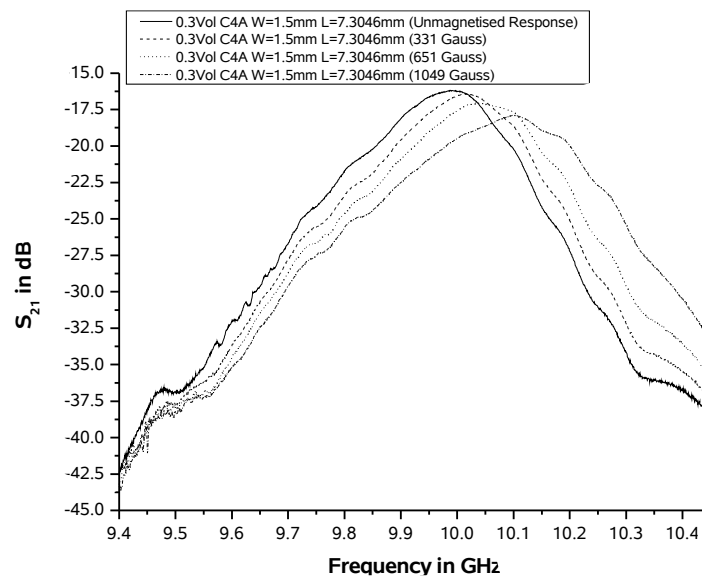


**Figure 117:- Response of 7.3046mm Long Microstrip Resonator on 0.3 Volume
Fraction C4A Composite Substrate to Axial DC Magnetic Bias**

Axial DC Bias in Gauss (kAm^{-1})	Centre Frequency in GHz	Insertion Loss in dB	Q_o
0	9.99128125	16.177	58.79
331 (26.348)	10.01753125	16.402	56.57
651 (51.820)	10.0365625	17.046	52.95
1049 (83.500)	10.10153125	17.889	43.68

**Table 2:- Changes in Observed Resonance due to Axial Magnetic Bias of
7.3046mm Long Microstrip Resonator on 0.3 Volume Fraction C4A Composite
Substrate**

The centre frequency of the resonance can be seen to increase by 0.263%, 0.453% and 1.103% relative to the unmagnetised case for the 331 G (26.348 kAm^{-1}), 651 G (51.820 kAm^{-1}) and 1049 G (83.5 kAm^{-1}) bias conditions respectively.

The second resonator deposited on a 0.3 volume fraction C4A substrate was designed using the reverse (S_{22} , S_{12}) T / R measured properties of the unmagnetised composite and was slightly shorter in length at 7.2574mm but with the same 1.5mm wide lines. The broadband response of the device though was very similar to the previous sample – see figure 118. Somewhat counter intuitively the unmagnetised resonant frequency is lower with the shorter resonator although at 9.8904125GHz it was still only 1.103% lower than the design frequency – see figure 119. In conjunction with the data from the previous resonator this suggests that the T / R measured permittivity and permeability of the unmagnetised 0.3 volume fraction C4A composites corresponds very well with those of the microstrip substrates.



**Figure 118:- Unmagnetised Response of 7.2574mm Long Microstrip Resonator
on a 0.3 Volume Fraction C4A Composite Substrate**

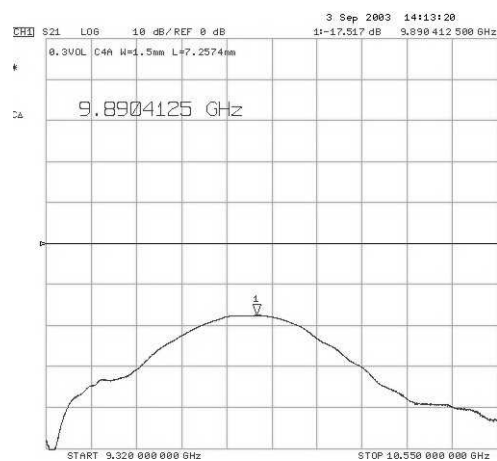


**Figure 119:- Unmagnetised Response of 7.2574mm Long Microstrip Resonator
on a 0.3 Volume Fraction C4A Composite Substrate**

The response of the second, 7.2574mm long, resonator to axial DC magnetic bias fields is also very similar to that of the first sample – see figure 120. The insertion losses though are 1.35dB higher for the unmagnetised condition and remain all but constant as does Q_0 with increasing levels of magnetic bias – see table 3. The lower Q_0 values are most likely due to higher conductor losses, substrate properties having been visibly demonstrated as nearly identical, from insufficient Copper film thickness. As described in Appendix II evaporated Copper conductor thicknesses were found to vary between 0.716 μ m to 0.56 μ m this latter value being less than the skindepth at the unmagnetised resonance frequency. The tunability of the resonance

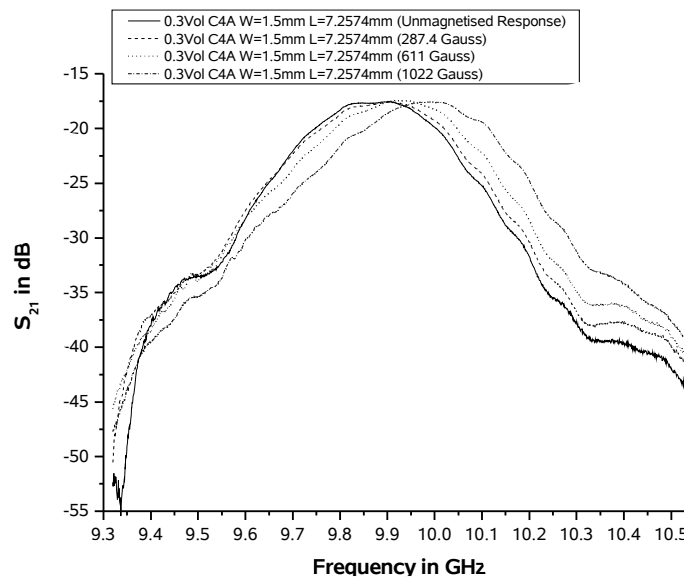


Figure 120:- Response of 7.2574mm Long Microstrip Resonator on 0.3 Volume Fraction C4A Composite Substrate to Axial DC Magnetic Bias

Axial DC Bias in Gauss (kAm^{-1})	Centre Frequency in GHz	Insertion Loss in dB	Q_o
0	9.8904125	17.520	40.38
287.4 (22.877)	9.901175	17.570	39.82
611 (48.636)	9.9180875	17.440	41.49
1022 (81.351)	10.011875	17.570	39.53

**Table 3:- Changes in Observed Resonance due to Axial Magnetic Bias of
7.2574mm Long Microstrip Resonator on 0.3 Volume Fraction C4A Composite
Substrate**

resonance is also very similar to that of the first 0.3 volume fraction C4A sample with increases of 0.109%, 0.280% and 1.228% for the 287.4 G (22.877 kAm^{-1}), 611 G (48.636 kAm^{-1}) and 1022 G (81.351 kAm^{-1}) bias conditions respectively. For both lengths of resonator it should clearly be possible using the test setup used to tune the resonance to match the design value exactly.

5.3 Microstrip Resonators on 0.4 Volume Fraction C4A Composite Substrates

Both of the resonators fabricated on 0.4 volume fraction C4A composite substrates had identical dimensions with 1.3mm wide lines and a 7.0738mm long resonator. The broadband measured responses of the two samples have principal resonances lower than the design value of 10.00075GHz possibly suggesting a discrepancy between the WR90 T / R measured permittivity and permeability and the actual substrate properties – see figures 121 and 122. Referring back to figures 51 and 53 in chapter 4 reveals a small spurious resonance induced decrease in T / R measured average permittivity and an associated increase in the real component of permeability around 10GHz which could be responsible.

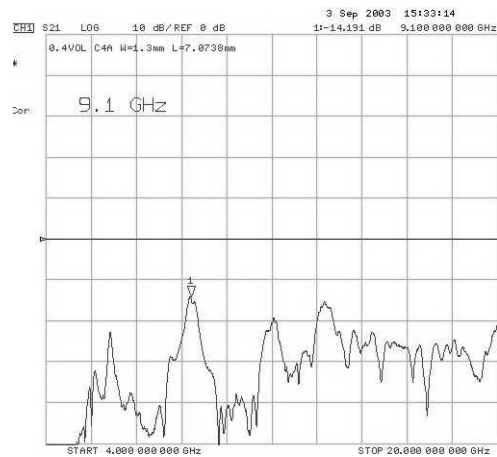


Figure 121:- Response of Unmagnetised 7.0738mm Long Microstrip Resonator on 0.4 Volume Fraction C4A Composite Substrate Designed Using Forward Measured T / R Properties

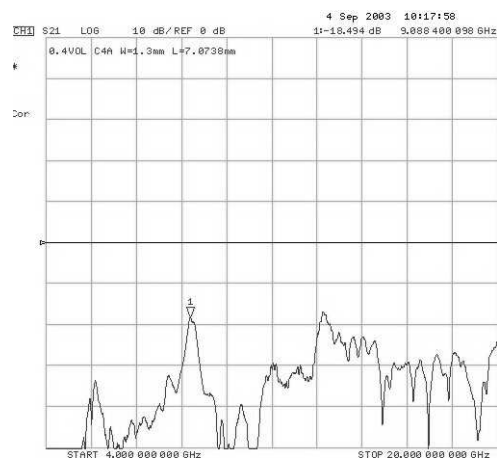


Figure 122:- Response of Unmagnetised 7.0738mm Long Microstrip Resonator on 0.4 Volume Fraction C4A Composite Substrate Designed Using Reverse Measured Properties T / R

Narrowing the VNA measurement bandwidth showed the unmagnetised centre frequencies of the two resonators to be 9.262883125GHz and 9.1024625GHz – see figures 123 and 124. These are 7.378% and 8.982% lower respectively than the design frequency.



Figure 123:- Response of Unmagnetised 7.0738mm Long Microstrip Resonator on 0.4 Volume Fraction C4A Composite Substrate Designed Using Forward Measured T / R Properties

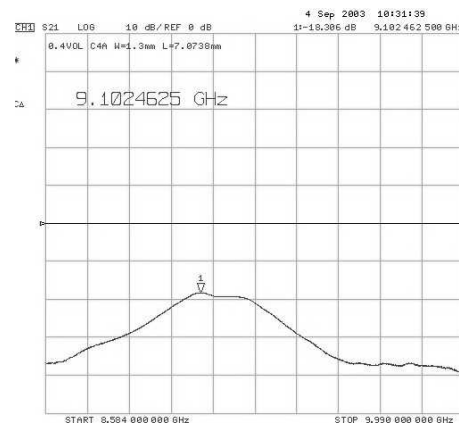


Figure 124:- Response of Unmagnetised 7.0738mm Long Microstrip Resonator on 0.4 Volume Fraction C4A Composite Substrate Designed Using Reverse Measured T / R Properties

The impact of axial magnetic bias fields upon the centre frequency of the first resonator sample is shown in figure 125 and table 4.

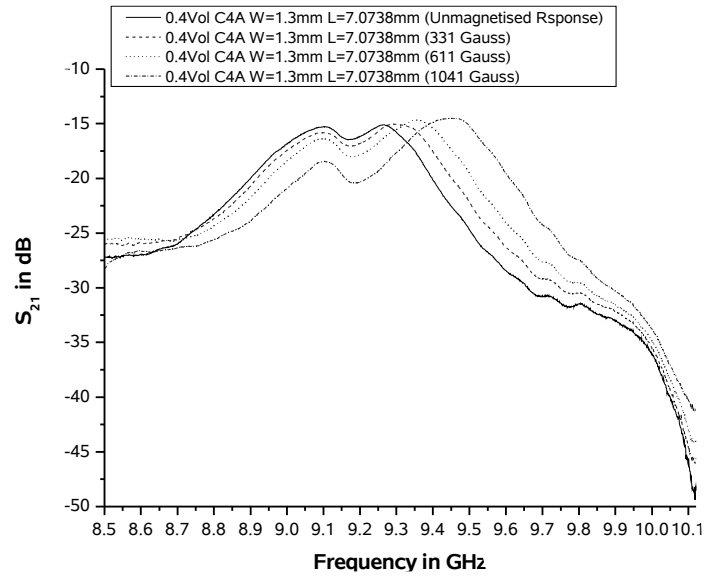


Figure 125:- Response of 7.0738mm Long Microstrip Resonator on 0.4 Volume Fraction C4A Composite Substrate to Axial DC Magnetic Bias

Axial DC Bias in Gauss (kAm^{-1})	Peak Frequency in GHz	Insertion Loss in dB	Q_o
0	9.262883125	15.121	56.57
331 (26.348)	9.29429	15.038	48.46
611 (48.636)	9.352038125	14.623	47.22
1041 (83.500)	9.4685475	14.505	46.32

Table 4:- Changes in Observed Resonance due to Axial Magnetic Bias of 7.0738mm Long Microstrip Resonator on 0.4 Volume Fraction C4A Composite Substrate to Axial DC

For the unmagnetised and 331 G (26.348 kAm⁻¹) bias conditions values of Q_0 were estimates only due to the splitting of the resonance which made determination of –3dB points difficult. As with the 0.3 volume fraction C4A resonators Q_0 values decreased with increasing bias field however the increasing axial magnetisation did also serve to isolate a single resonant peak. The smaller resonance located at approximately 9.1GHz remaining independent of the DC bias field. In relation to the peak frequency of the unmagnetised resonator tunabilities of 0.339%, 0.962% and 2.22% were achieved with 331 G (26.348 kAm⁻¹), 611 G (48.636 kAm⁻¹) and 1041 G (83.500 kAm⁻¹) bias conditions respectively.

Similar double-peaked asymmetrical resonances were also produced by the second 0.4 volume fraction C4A composite sample. Its response to axial DC magnetic bias fields is shown in figure 126 and summarised in table 5. Insertion losses were 3 to 4dB higher than for the previous sample which when combined with the asymmetric response resulted in lower values of Q_0 . The only exception to this was the response when the substrate was subject to a 1005 G (79.998 kAm⁻¹) bias. Tunability for this second 0.4 volume fraction substrate was considerably higher than for the first sample. Increases in peak resonant frequency of 1.834%, 2.520% and 3.360% were achieved using axial biases of 325 G (25.870 kAm⁻¹), 641 G (51.023 kAm⁻¹) and 1005 G (79.998 kAm⁻¹) respectively. The increased tuning range available is possibly a result of differences in the homogeneity of the two 0.4 volume fraction composite substrates with potentially a greater concentration of the garnet fraction in the second sample being located underneath the microstrip lines. This would give a locally increased real component of permittivity, with an associated small decrease in permeability, that would both decrease the unmagnetised resonant frequency and

show a greater interaction between the microwave signal's H-field and the axial DC bias.

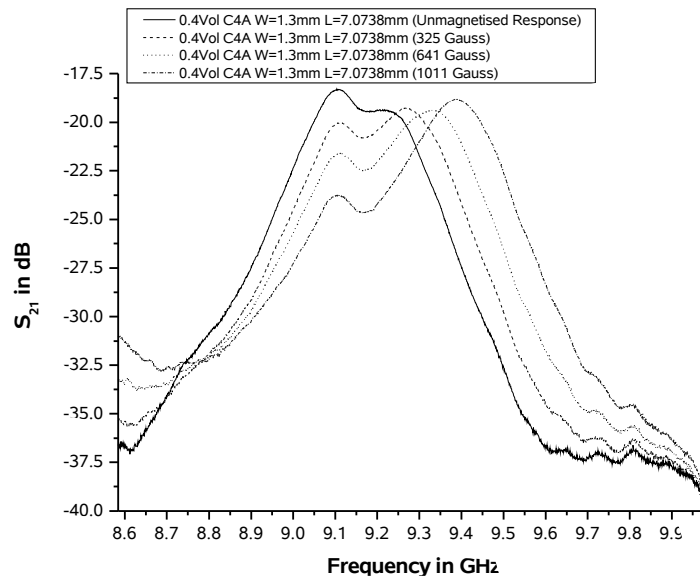


Figure 126:- Response of 7.0738mm Long Microstrip Resonator on 0.4 Volume Fraction C4A Composite Substrate to Axial DC Magnetic Bias

Axial DC Bias in Gauss (kAm^{-1})	Peak Frequency in GHz	Insertion Loss in dB	Q_o
0	9.1024625	18.309	41.32
325 (25.870)	9.269425	19.259	32.32
641 (51.023)	9.33181625	19.376	43.65
1005 (79.998)	9.4082675	18.818	50.27

Table 5:- Changes in Observed Resonance due to Axial Magnetic Bias of 7.0738mm Long Microstrip Resonator on 0.4 Volume Fraction C4A Composite Substrate

5.4 Microstrip Resonators on 0.3 Volume Fraction C6A Composite Substrates

Changing to C6A garnet powder as the loading material higher tuning factors were expected of both the 0.3 and 0.4 volume fraction composite resonators. This is due to the material's higher saturation magnetisation and consequent higher gyromagnetic

resonance frequency. The material is therefore operated in a region where its effective scalar permeability changes more rapidly in response to the axial magnetic bias field – see Lax and Button (1962). Figure 127 shows the broadband measured response of the 7.54718mm long resonator deposited on a 0.3 volume fraction C6A composite substrate. The error in the forward (S_{11} , S_{21}) T / R measured properties again seemingly very small given that the unmagnetised resonator centre frequency is only 0.331% lower than intended at 9.96767375 – see figure 128. The response of the application of axial DC magnetic bias fields is shown in figure 129 with the actual centre frequencies, insertion losses and Q_0 values listed in table 6.

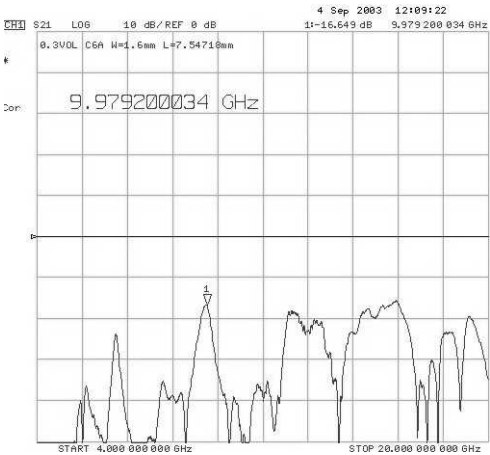


**Figure 127:- Response of Unmagnetised 7.54718mm Long Microstrip Resonator
on a 0.3 Volume Fraction C6A Composite Substrate**

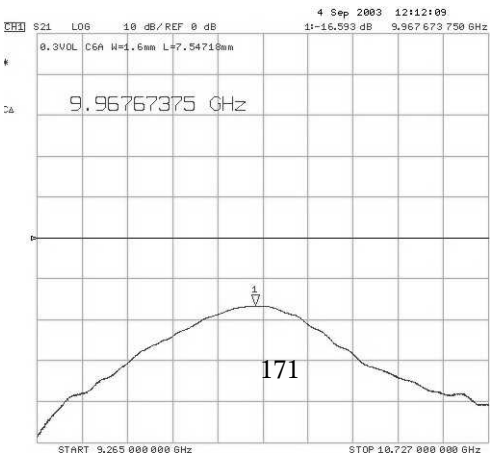


**Figure 128:- Response of Unmagnetised 7.54718mm Long Microstrip Resonator
on a 0.3 Volume Fraction C6A Composite Substrate**

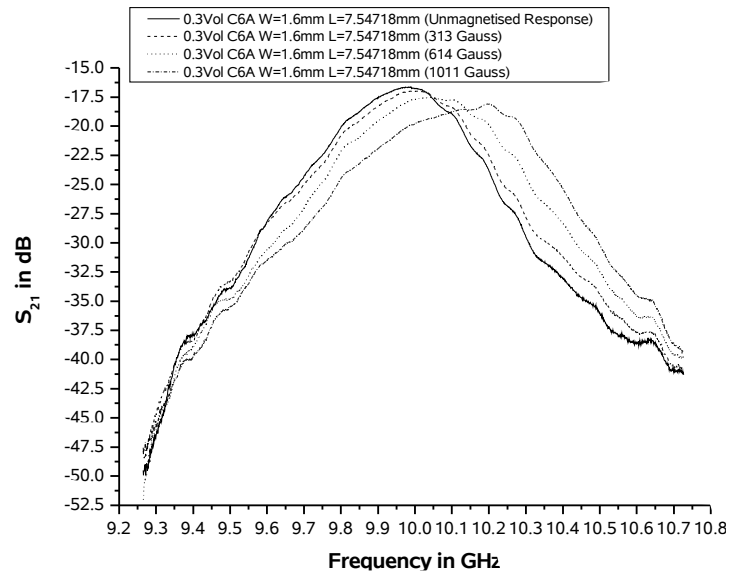


**Figure 129:- Response of 7.54718mm Long Microstrip Resonator on 0.3 Volume
Fraction C6A Composite Substrate to Axial DC Magnetic Bias**

Axial DC Bias in Gauss (kAm^{-1})	Centre Frequency in GHz	Insertion Loss in dB	Q_o
0	9.96767375	16.678	37.93
313 (24.915)	9.98412125	17.022	37.19
614 (48.874)	10.03986	17.519	31.49
1005 (79.998)	10.20158375	18.067	31.54

**Table 6:- Changes in Observed Resonance due to Axial Magnetic Bias of
7.54718mm Long Microstrip Resonator on 0.3 Volume Fraction C6A Composite
Substrate**

Tunability of the device at 313 G (24.915 kAm^{-1}) is very similar to that obtained with the 0.3 volume fraction C4A substrate with a 0.165% increase in the measured centre frequency relative to the unmagnetised case. At the next two bias levels the degree of tuning exceeds that of the equivalent C4A composites with 0.724% and 2.347% increases in centre frequency at 614 G (48.874 kAm^{-1}) and 1005 G (79.998 kAm^{-1}) respectively. Insertion losses of the resonator were not greatly different from those of the C4A composite resonators yet values of Q_o were up to 25% lower.

This higher level of loss was also present in the second and slightly shorter resonator, 7.24618mm long, deposited on a 0.3 volume fraction C6A composite substrate. The broadband response of the resonator is show in figure 130. As with the C4A design that used reverse (S_{22}, S_{12}) T / R measured composite permittivity and permeability values to determine line widths and lengths the unmagnetised resonant frequency was more significantly in error being 2.779% lower than the design value – see figure 131.



**Figure 130:- Response of Unmagnetised 7.24618mm Long Microstrip Resonator
on a 0.3 Volume Fraction C6A Composite Substrate**

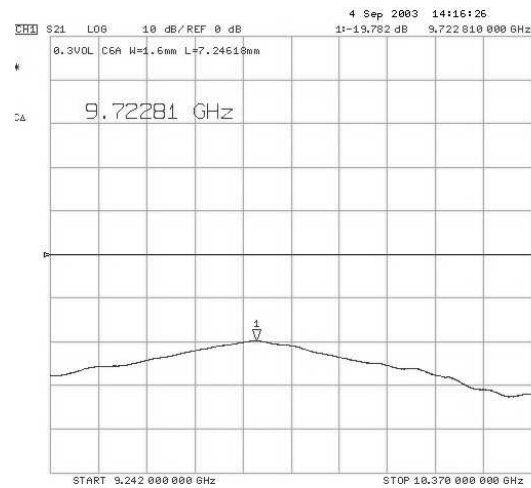


**Figure 131:- Response of Unmagnetised 7.24618mm Long Microstrip Resonator
on a 0.3 Volume Fraction C6A Composite Substrate**

Figure 132 shows the increase in resonant frequency and changing resonance shape as the axial DC bias field was increased. The actual changes in centre frequency, insertion loss and Q_0 are detailed in table 7.

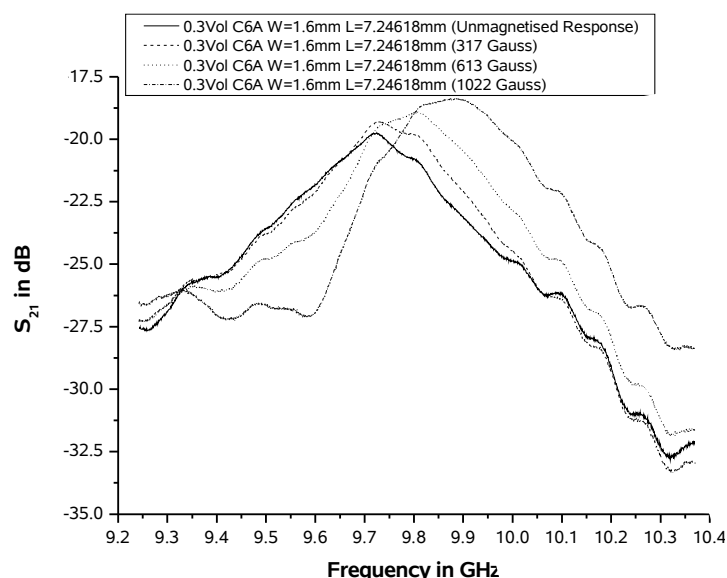


Figure 132:- Response of 7.24618mm Long Microstrip Resonator on 0.3 Volume Fraction C6A Composite Substrate to Axial DC Magnetic Bias

Axial DC Bias in Gauss (kAm^{-1})	Centre Frequency in GHz	Insertion Loss in dB	Q_0
0	9.72281	19.765	32.12
317 (25.233)	9.726335	19.302	34.05
613 (48.795)	9.806705	18.914	32.21
1022 (81.351)	9.888485	18.412	33.65

Table 7:- Changes in Observed Resonance due to Axial Magnetic Bias of 7.24618mm Long Microstrip Resonator on 0.3 Volume Fraction C6A Composite Substrate

As can be seen in figure 132 tunability at 317 G (25.233 kAm⁻¹) was all but negligible with a 0.036% increase in centre frequency observed. At 613 G (48.795 kAm⁻¹) the centre frequency is 0.863% higher relative to the unmagnetised case which is a greater frequency shift than was achieved with the first 0.3 volume fraction C6A resonator. At 1022 G (81.351 kAm⁻¹) the centre frequency is 1.704% higher while the actual resonance itself has changed shape quite significantly. Insertion losses dropped by 1.353dB from the unmagnetised response to that produced at maximum DC bias however Q_o remained all but constant at the lowest levels to be measured for any of the eight resonators tested. The higher C6A saturation magnetisation would give larger values of magnetic loss tangent than for the C4A material however the difference so far above the gyromagnetic resonance frequency would give minimal difference between the two substituted garnets' performance. This is confirmed by the performance of the two 0.4 volume fraction substrates which both gave consistently high values of Q_o as will be seen.

5.5 Microstrip Resonators on 0.4 Volume Fraction C6A Composite Substrates

The broadband response of the first, longer and wider, of the two microstrip resonators deposited on a 0.4 volume fraction C6A composite substrate is shown in figure 133. The centre frequency of the resonance at 9.26216GHz is almost exactly the same as was produced by the equivalent C4A resonator designed using forward T / R measured composite properties. The C4A sample resonated 7.378% lower than the design frequency while the C6A composite shown here operated 7.385% lower than intended – see figure 134. The measured values of S₂₁ for the resonator when subject to axial magnetic bias fields are shown in figure 135. The resonance retains its symmetry with increasing DC magnetic fields and shows the highest consistent values of Q_o – see table 8.

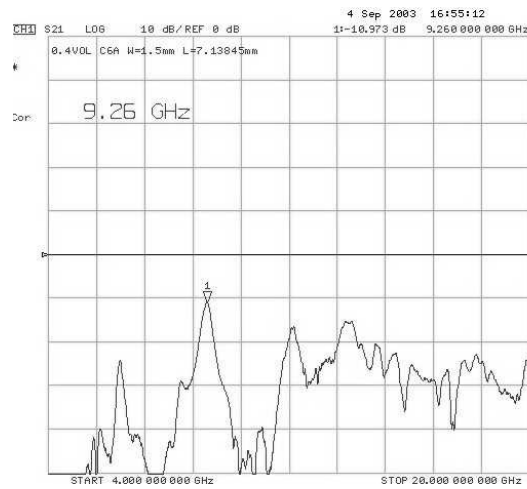


**Figure 133:- Response of Unmagnetised 7.13845mm Long Microstrip Resonator
on a 0.4 Volume Fraction C6A Composite Substrate**

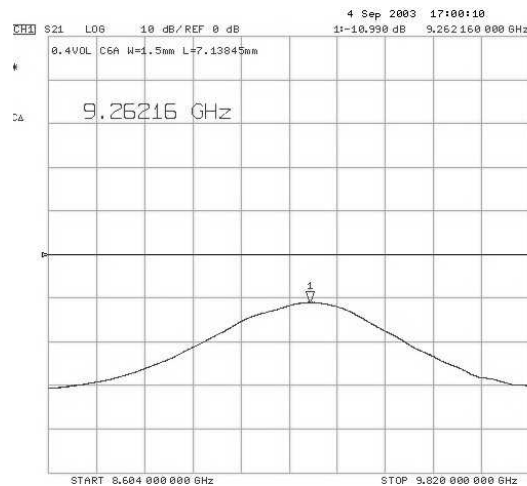


**Figure 134:- Response of Unmagnetised 7.13845mm Long Microstrip Resonator
on a 0.4 Volume Fraction C6A Composite Substrate**

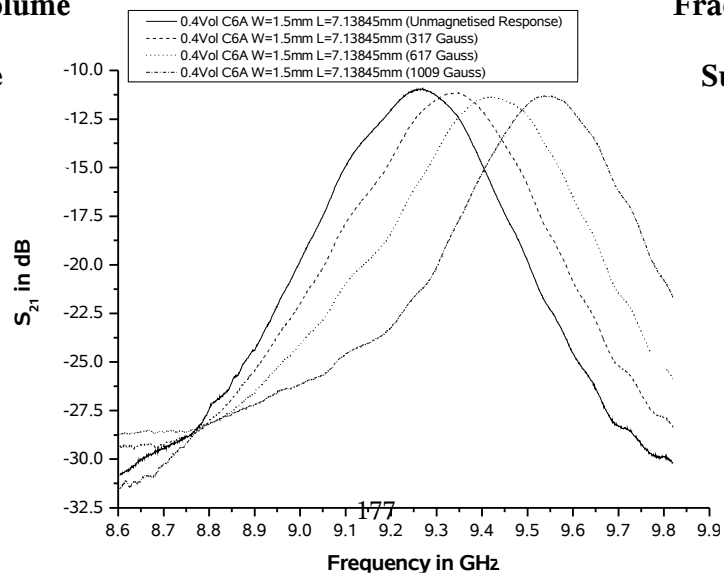


Figure 135:- Response of 7.13845mm Long Microstrip Resonator on 0.4 Volume Fraction C6A Composite Substrate to Axial DC Magnetic Bias

Axial DC Bias in Gauss (kAm^{-1})	Centre Frequency in GHz	Insertion Loss in dB	Q_o
0	9.26216	10.965	50.43
317 (25.233)	9.3488	11.150	52.27
617 (49.113)	9.41948	11.373	50.15
1009 (80.316)	9.5578	11.367	53.93

Table 8:- Changes in Observed Resonance due to Axial Magnetic Bias of 7.13845mm Long Microstrip Resonator on 0.4 Volume Fraction C6A Composite Substrate

The centre frequency of the resonance can be seen to increase by 0.935%, 1.699% and 3.192% relative to the unmagnetised response for bias levels of 317 G (25.233 kAm⁻¹), 617 G (49.113 kAm⁻¹) and 1009 G (80.316 kAm⁻¹) respectively. As would be hoped for this is a greater degree of tunability than was achieved from the equivalent C4A resonator – see figure 125.

The second and shorter resonator fabricated on a 0.4 volume fraction C6A composite substrate also demonstrated remarkable similarities with its C4A equivalent. The broadband response of the 1.3mm wide and 7.0391mm long resonator on the C6A composite substrate is shown in figure 136. Referring to the narrowband measurement data in figure 137 the actual centre frequency located at 9.19326625GHz is 8.074% lower than the design value. The 0.4 volume fraction C4A resonator similarly designed using reverse (S_{22} , S_{12}) T / R measured values of composite permittivity and permeability was in error by a similar amount – see figure 138.

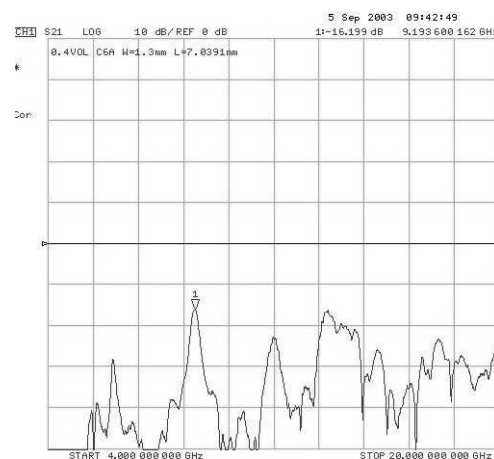


**Figure 136:- Response of Unmagnetised 7.0391mm Long Microstrip Resonator
on a 0.4 Volume Fraction C6A Composite Substrate**

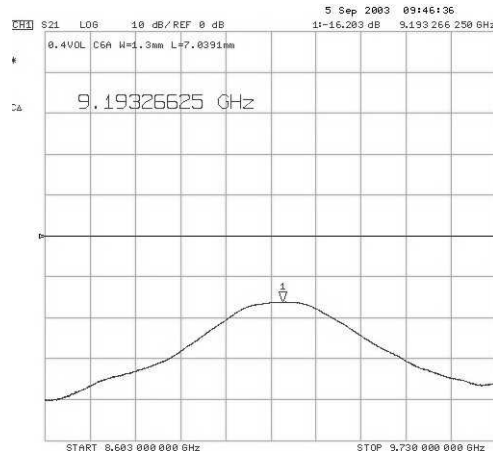


**Figure 137:- Response of Unmagnetised 7.0391mm Long Microstrip Resonator
on a 0.4 Volume Fraction C6A Composite Substrate**

Application of axial DC magnetic bias fields gives a similar increase in measured resonant frequency to that observed previously with the first 0.4 volume fraction C6A composite substrate – see figures 135 and 138. Increases in resonant frequency of 0.736%, 1.877% and 3.900% are achieved using biases of 317 G (24.994 kAm⁻¹), 631 G (50.228 kAm⁻¹) and 1059 G (84.296 kAm⁻¹) respectively. Q_0 is again high and relatively independent of frequency with the insertion losses decreasing gradually with bias field strength – see table 9. Some change in the resonance shape is apparent with increasing bias levels with a smaller extraneous resonance becoming apparent around 9.1GHz as was the case with the resonators fabricated on 0.4 volume fraction C4A composite substrates – see figures 125 and 126.

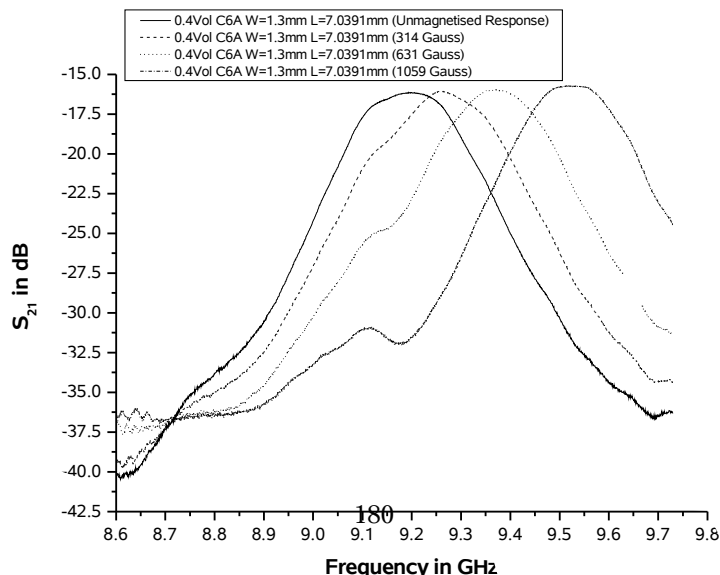


Figure 138:- Response of 7.0391mm Long Microstrip Resonator on 0.4 Volume Fraction C6A Composite Substrate to Axial DC Magnetic Bias

Axial DC Bias in Gauss (kAm^{-1})	Centre Frequency in GHz	Insertion Loss in dB	Q_o
0	9.19326625	16.178	47.40
314 (24.994)	9.26088625	16.069	49.29
631 (50.228)	9.365838125	15.988	51.73
1059 (84.296)	9.551793125	15.814	52.37

Table 9:- Changes in Observed Resonance due to Axial Magnetic Bias of 7.0391mm Long Microstrip Resonator on 0.4 Volume Fraction C6A Composite Substrate

This chapter has successfully demonstrated the design, fabrication and testing of frequency agile linear microstrip resonators on composite substrates. In so doing it has widened the scope of Pucel and Massé's (1972) work on microstrip lines with bulk gyromagnetic substrates to include microwave garnet loaded composites. Applying their duality assumption to Hammerstad's (1975) microstrip design formulae has enabled the very accurate choice of line widths and lengths to achieve a given resonant frequency. Using values of permittivity and permeability from unmagnetised composites measured using the T / R technique in WR90 waveguide it has proved possible to produce 0.3 volume fraction C4A resonators within 0.11% of their intended resonant frequency. Resonators fabricated on 0.3 volume fraction C6A composite substrates resonated within a worst-case error of 2.779% relative to the design value. Designs for resonators on 0.4 volume fraction composite substrates using forward measured T / R material properties proved less accurate than the 0.3 volume fraction resonators. All but identical errors in resonant frequency of 7.378% and 7.385% were measured with the 0.4 volume fraction C4A and C6A designs respectively. Performance was similar for resonators designed using reverse T / R measured composite properties with unmagnetised resonant frequencies 8.982% and 8.074% lower than intended for C4A and C6A loaded substrates respectively. The extremely consistent magnitude of the errors, regardless of garnet inclusion material, and the highly accurate performance of the 0.3 volume fraction designs raises the possibility of the presence of some systematic error(s) in the T / R data for 0.4 volume fraction composites.

Q_0 values of the fabricated resonators varies between a minimum of 32.12 and a maximum of 58.79. This is lower than had been hoped but in the context of Copper conductors of a single skindepth or less in thickness and a dielectric host not normally

employed for microwave devices the results are encouraging. Looking at Gopinath's (1981) calculations of the maximum achievable Q_0 for linear half-wavelength microstrip resonators on purely dielectric substrates a composite material optimised for minimum host dielectric loss, minimum radiation loss and using thick metallisation should be capable of a maximum Q_0 of between 150 and 200 at 10GHz. This estimation seems plausible in light of the Q_0 of 166 measured by Tsutsumi and Fukusako (1997) for a Copper resonator deposited on a monocrystalline YIG film grown on a Gadolinium Gallium Garnet substrate.

Relating the observed levels of resonator tunability to the changes in WR90 T / R measured permeability samples is not trivial. Due to the unregulated nature of the DC power supply for the Helmholtz coil pair heating of the coil windings caused fluctuations in current drawn. Consequently, the maximum DC magnetic bias levels were inconsistent from sample to sample varying from a minimum of 1005 G (79.998 kAm⁻¹) to a maximum of 1059 G (84.296 kAm⁻¹) these two being respectively lower and higher than the transverse magnetisation available from the single magnet WR90 T / R measurement setup. Using Pucel and Massé's (1972) approach in conjunction with Hammerstad's (1975) microstrip equations gives calculated magnetic filling factors of 65.61%, 66.65% and 67.13% for linewidths of 1.3, 1.5 and 1.6mm respectively. These values might reasonably be expected to set the upper limits on the proportion of the WR90 observed tunability achievable with the microstrip devices. Both of the 1.3mm wide resonators on 0.4 volume fraction C4A substrates demonstrated 56.97% and 53.2% of the WR90 tunability out of a maximum postulated 65.61%. The 1.6mm wide resonators on 0.3 volume fraction C6A substrates demonstrated 59.31% and 60.99% of the WR90 samples' tuning with a potential maximum of 67.13%. However, evidence contrary to the predicted filling

factor limit on tunability comes from both of the 0.3 volume fraction C4A and 0.4 volume fraction C6A designs. All four samples demonstrating proportions of the WR90 tunability exceeding the microstrip filling factors. With the aforementioned bias problems and the small number of samples investigated a definitive conclusion as to the relationship between the tunability of composite samples measured in WR90 waveguide and used as microstrip substrates cannot be reached. A new multi-tier development of an existing planar materials measurement technique will be described in the following chapter as a means to investigate the tunability link as well as to help optimise it and resonator Q_o as functions of substrate thickness, linewidth and composite formulation.

Chapter 6: Conclusions and Further Work

6.1 Conclusions

The aim of this thesis has been to investigate the potential use of binary composite materials for the production of planar frequency-agile microwave devices. Work has concentrated on the use of Calcium Vanadium Iron Garnet loaded thermosetting resin. From a review of the available literature the Bruggeman and NIST-modified Maxwell-Garnet effective medium theories were selected for investigations into their ability to predict the permittivity and permeability of unmagnetised and transversely magnetised garnet loaded composites. The effective medium theories were supplied with permittivity and permeability data measured in WR90 waveguide for the composites' constituents using the T / R technique and NRW equations as well as manufacturer supplied garnet permittivity and simple empirical model calculated garnet permeabilities. Measurements on 0.3 and 0.4 volume fraction composites demonstrated that predicted composite properties using a combination of the Bruggeman model with the manufacturer's permittivity and calculated permeability data was sufficient to give highly accurate predictions. Discrepancies with the composites' measured permittivity and permeability remained less than 5% over around half of the 4.2GHz WR90 bandwidth. Over the whole of the waveguide band errors in predicted material properties remained within 15%. This implied that for the cases considered accurate predictions of composite permittivity and permeability were possible without recourse to any form of microwave materials measurement. Analysis of the impact of uncertainty in manufacturer supplied permittivity saturation magnetisation revealed that it could account for up to up to 20% of the observed differences between measured properties and those predicted using the Bruggeman effective medium theory.

Suitability of the composite materials for tuneable device use was investigated by using them as substrates for linear gap-coupled microstrip resonators. Choice of resonator lengths and linewidths to give an unmagnetised resonance at 10.00075GHz applied Pucel and Massé's (1972) work on microstrip lines with bulk ferrite and garnet substrates to Hammerstad's (1975) microstrip design formulae. Results for resonators with 0.3 volume fraction garnet composite substrates confirmed the accuracy of the T / R measured composite properties used in the design process with worst case errors in unmagnetised resonant frequency of -0.095% and 1.103% for C4A composites and -0.331% and -2.779% for C6A composites. The magnitude of the errors for 0.4 volume fraction composites was higher but remarkably consistent between the two materials with lower and upper error limits of -7.378% and -8.982% respectively. This consistency suggesting the presence of a systematic error in the waveguide measured properties of the 0.4 volume fraction composites. Measured unmagnetised resonator Q_0 levels varied from a minimum of 32.12 to a maximum of 58.79 with an average of 45.62. Values were lower than hoped for due in part to the thin Copper conductors used. Examination of Gopinath's (1981) work on half-wavelength microstrip resonators suggested that an optimised design could achieve a maximum Q_0 of between 150 and 200.

Application of DC magnetic fields parallel to the direction of wave propagation i.e. axial to the microstrip lines caused increases in measured resonant frequency. At maximum applied bias levels the devices showed maximum increases of 1.228% in resonant frequency for 0.3 volume fraction C4A materials; 3.36% for 0.4 volume fraction C4A composites; 2.347% for 0.3 volume fraction C6A composites and 3.9% for 0.4 volume fraction C6A composites. It was speculated that the magnetic filling

factors of the microstrip lines would set the upper limit on the proportion of T / R measured tunability of composite permeability that the microstrip resonators could achieve. Limited number of samples and inconsistent bias field strengths prevented a link from being established conclusively either way.

The work described has demonstrated that garnet loaded composites are a viable route to the production of frequency agile planar microwave devices. Accurate predictions of composite permittivity and permeability without recourse to measurement proved possible using existing effective medium theories using what amounted to the information provided by a manufacturer on a datasheet. This approach provides the microwave engineer with flexibility in that the permittivity and tunability can be tailored to the application, no complicated or expensive ceramic production techniques are required and there is the possibility of producing novel three dimensional structures. Location of the microwave garnet component within the host dielectric offers the potential for production of a high temperature superconductor compatible composite with user determined properties.

6.2 Further Work

As a result of the work carried out there are a number of areas warranting further investigation to try and clarify uncertainties and to develop the composite idea further. The first area for attention is the waveguide material measurement system. In order to truly establish the accuracy of the Bruggeman model's predicted permittivities and permeabilities when using 'datasheet' information the source of uncertainty in the measurement system need reducing and quantifying. One of the principle means of accomplishing this is to overcome the problems that result of the support of higher order modes in the waveguide test-cells. This can be achieved through use of Baker-Jarvis et al's (1993) multiple point orthogonal distance regression method to calculate sample permittivity and permeability from measured S-Parameters. This approach places a higher weighting factor on the fundamental mode data in its optimisation routine leading to greatly reduced sensitivity to higher order modes and the spurious calculated material properties that they can result in. The approach also determines the uncertainties in calculated permittivity and permeability due to systematic uncertainties in sample length, test-cell length and the location of the sample within a test-cell. Errors in measured S-Parameters can themselves be reduced relative to their present values through use of a multiple line standard TRL calibration. This should make use of the new optimal solution calibration algorithm developed by Williams et al (2003)^{1,2} which outperforms the current most accurate VNA calibration procedure in existence developed by Marks (1991). Williams et al's (2003)^{1,2} use of orthogonal distance regression routines determines the uncertainty limits of the resulting calibration coefficient and vector error corrected measurement data. This should reveal the exact levels of accuracy available from the effective medium theory models as well as helping to determine if the errors in the unmagnetised 0.4 volume

fraction microstrip resonators' resonant frequencies was due to inaccuracies in the NRW calculated composite properties.

Optimising microstrip linewidths to maximise transversely biased tunability and determine any link between microstrip filling factors and the proportion of WR90 measured permeability tuning available from a microstrip device requires a dual approach. Firstly, a fully regulated high current power supply is required to drive the existing Helmholtz coil pair used to provide DC magnetic bias to the microstrip devices. This would prevent thermal induced changes in coil resistance from giving rise to changes in applied bias field. Secondly, use should be made of the following hitherto unpublished measurement technique for determining the effective permittivity and permeability of microstrip lines on bulk or composite garnet / ferrite substrates subject to DC magnetic bias fields applied transverse to the microwave H-field. This method, confirmed as valid by Janezic (2003) of the US National Institute of Standards and Technology, is a multi-tier development of the microstrip method for determining the permittivity of thin-films developed by Janezic et al(2003). The new approach proceeds as follows:

1. Perform a multiple line TRL calibration using Williams et al's (2003)^{1,2} new optimised algorithm with lines fabricated on a low-loss substrate with a low dispersion permittivity such as Fused Quartz or Sapphire.
2. Find the frequency independent line capacitance from measurement of the line standards' DC resistance using the method of Williams and Marks (1991). Use the calculated capacitance along with the propagation constant determined as a

by-product of the TRL calibration to determine the characteristic impedance of the line standards – see Marks and Williams (1991).

3. Fabricate multiple microstrip line standards of the width of interest on the garnet loaded composite substrate under test.
4. Use a Helmholtz coil or permanent magnet(s) to apply a DC magnetic bias field of sufficient magnitude parallel to the microwave H-field component on the microstrip lines to cause substrate saturation. This will result in unity permeability and zero magnetic losses – see Green et al (1968). The substrate effectively functions now as a purely dielectric material enabling both its effective and actual permittivity to be calculated.
5. Perform a multiple line TRL calibration using the magnetically saturated lines of step 4. Determine the lines' actual characteristic impedance ($Z_{o \text{ saturated}}$) for this condition using the reference calibration and characteristic impedance of steps 1 and 2 using the calibration comparison method of Williams et al (1991).
6. From a knowledge of the substrate thickness and microstrip linewidths used in step 4 use either a set of conformal mapping derived closed-form expressions or a finite series analytical approximation to the integral equation to calculate the capacitance (C_{air}) of the lines assuming an air substrate – see Collin (1992)
7. Use the values of $Z_{o \text{ saturated}}$ and C_{air} to calculate the effective substrate permittivity (ϵ_{eff}) using equation 15. At this point the method of Janezic et al (2003) can be used to calculate the actual permittivity of the composite substrate.

$$Z_{o \text{ saturated}} = \sqrt{\frac{\mu_o \epsilon_o}{\epsilon_{eff}}} \frac{1}{C_r} \quad (15)$$

8. Having stored the calculated values of $Z_{o \text{ saturated}}$ and ϵ_{eff} the bias field is removed and the sample allowed to demagnetise.
9. Repeat the multiple line TRL calibration using the composite substrate lines for conditions of DC magnetic bias applied transverse to the microwave H-field i.e. parallel to the direction of wave propagation. Calculate the characteristic impedance of the lines ($Z_{o \text{ transverse}}$) using the calibration comparison method but with the data from step 5 and $Z_{o \text{ saturated}}$ as the new reference calibration.
10. Calculate the effective permeability (μ_{eff}) of the transversely magnetised microstrip lines in step 9 using equations 16 and 17. Potentially at this point it may be possible to calculate the effective WR90 permeability of the substrate using a finite difference analysis of microstrip lines on gyromagnetic substrates - see Pucel and Massé (1972)

$$\begin{aligned} \frac{Z_{o \text{ transverse}}}{Z_{o \text{ saturated}}} &= \sqrt{\frac{\mu_{eff} \epsilon_o}{\epsilon_{eff}}} \frac{1}{C_r} \\ \therefore \mu_{eff} &= \frac{Z_{o \text{ transverse}}^2}{Z_{o \text{ saturated}}^2} \quad (17) \end{aligned}$$

The preceding novel method offers several advantages over microstrip linear half-wavelength or ring resonator techniques. The first benefit is the extremely broad

measurement bandwidth available, on the order of tens of GHz due to the use of the straight microstrip lines and the multiple line TRL calibration. Additional benefits of the use of straight microstrip lines are greatly simplified fabrication, absence of fringing fields or the need to take account of changing gap-coupling factors. Selection of the new optimised multiple line TRL calibration algorithm of Williams et al (2003)^{1,2} to carry out this procedure as stated earlier automatically gives uncertainty limits on the calibration and measurement data which can be linked with uncertainties in line dimensions to calculate the resulting uncertainties in effective substrate properties.

The new measurement technique also enable contributing loss factors of the microstrip lines to be determined from their lumped element equivalent circuit terms L, C, R and G. These terms can be calculated from the lines' propagation constant and characteristic impedance – see Marks and Williams (1991). For the saturated sample case the conductor losses (dimensionless quantity) are given by equation 18 while the contribution due to the dielectric loss tangent of the substrate is given by equation 19 – see Janezic et al (2003).

$$\text{conductor losses} = \frac{R}{\omega L}$$

$$\text{dielectric losses} = \frac{G}{\omega C} \quad (19)$$

For an unmagnetised substrate or when subject to transverse DC magnetic bias the dielectric loss term should remain unchanged while any incremental increase in the lumped element R and L terms and associated loss factor in equation 18 will be due to the contribution from the magnetic loss tangent of the garnet / ferrite inclusions in the composite substrate. The actual utility of this information may however be limited when dealing with thin-film substrates due to metallisation losses

greatly overshadowing those due to the materials' loss tangents – see Janezic et al (2003).

Changes in the host dielectric used for composite fabrication for one with a very low real component of permittivity and loss tangent would provide greatly improved device performance. It would enable the investigation of a much wider range of garnet / ferrite filling factors while still achieving a low overall composite permittivity. Two candidate materials for use as the host material are the non crystalline PTFE-like amorphous fluoropolymers Teflon-AF™ from DuPont and Cytop™ from Asahi Glass Company. These have low real components of permittivity of 1.89 – 1.93 at 10GHz and 2.04 – 2.05 from 1 to 25GHz respectively. Their dielectric loss tangents are also low with DuPont (1997) reporting values less than or equal to 3×10^{-4} for Teflon-AF™ while the loss tangent for Cytop™ quoted by Asahi (2002) as being less than 8×10^{-4} . The amorphous fluoropolymers have the ability to be deposited as thin or thick-films by a multitude of techniques including spin-coating, thermal evaporation, pulsed laser ablation and chemical vapour deposition – see Asahi (2002), Kiesbye et al (1998), Sharangpani et al (1997)^{1,2} and Cho et al (1994, 1995). Bulk samples can be formed by dip casting or thermal extrusion – see Asahi (2002) and DuPont (1997). Combined with the aforementioned electrical properties, the same chemical resistance as conventional PTFE and low (less than 200°C) processing temperatures garnet / ferrite loaded amorphous fluoropolymers offer the potential to overcome the problems of integrating epitaxial ferrite and garnet thin-films compound semiconductor microwave integrated circuits described by Glass (1988). Maximum tuneable device Q_0 could be achieved by replacing the garnet powder inclusions with nanometre scale single crystals of microwave garnet for minimum magnetic loss tangents. This is already possible for pure YIG using the

sol-gel method developed by Vaquero and Lopez-Quintela (1997) which may be extendable to include the substituted garnets as well. Using the approach and materials outlined above the design and production of low-loss and magnetically tuned frequency agile microwave ‘systems-on-a-chip’ using monolithic integrated microwave circuits (MMICs) would become a reality.

Appendix I :- Nicolson-Ross-Weir Derivation

The Nicolson-Ross-Weir (NRW) equations enable the calculation of the complex permittivity and permeability of an unknown material sample entirely filling the cross-section of a reflectionless airline from the measured S-Parameters. The link between measured S-Parameters and material properties is derived by considering the multiple reflections of a unit amplitude wave incident upon the air-sample interfaces within the waveguide – see figure A1.

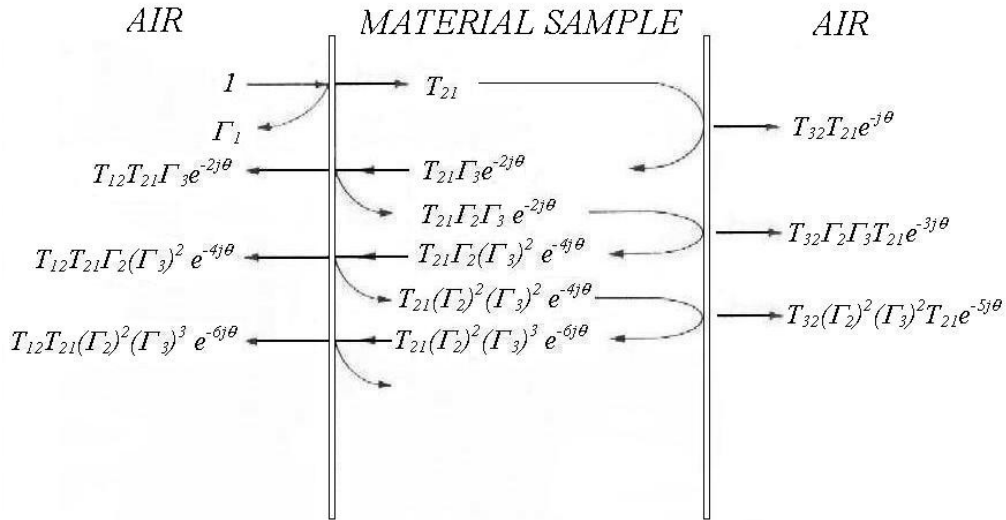


Figure A1:- Multiple reflections at the air-sample interfaces within a filled waveguide

As described by Collin (1992) when the wave is incident upon the first, left most, air-sample interface there is a partial reflection of amplitude Γ_1 with the remaining portion T_{21} travelling through the sample to strike the second interface. Once again there is a partial reflection of amplitude $\Gamma_1 T_{21} e^{-2j\theta}$ with the remainder of amplitude $T_{32} T_{21} e^{-j\theta}$ being transmitted. The sum of the reflections occurring at the first air-sample interface is equal to the input reflection coefficient Γ_{IN} of the filled section of

waveguide. The first three terms of the infinite number comprising Γ_{IN} are shown in equation A1:

$$\Gamma_{IN} = \Gamma_1 + T_{12}T_{21}\Gamma_3 e^{-j2\gamma d} + T_{12}T_{21}\Gamma_3^2\Gamma_2 e^{-j4\gamma d} + \dots \quad (A1)$$

Equation A1 can be further simplified as follows:

$$\Gamma_{IN} = \Gamma_1 + T_{12}T_{21}\Gamma_3 e^{-2j\theta} \sum_{n=0}^{\infty} \Gamma_2^n \Gamma_3^n e^{-2jn\theta} \quad (A3)$$

$$\sum_{n=0}^{\infty} r^n = \frac{1}{1-r}$$

$$\Gamma_{IN} = \Gamma_1 + \frac{T_{12}T_{21}\Gamma_3 e^{-2j\theta}}{1 - \Gamma_2\Gamma_3 e^{-2j\theta}} \quad (A5)$$

Substitutions can be made for T_{12} , T_{21} , Γ_2 , Γ_3 and $e^{-j2\theta}$ in order to express S_{11} , which is the equivalent to Γ_{IN} , in terms of the normalised complex reflection coefficient for a wave incident upon the air-sample interface with an infinitely thick material sample Γ_1 and the a complex exponential containing the material's propagation constant γ - see equations A6 to A9.

$$\Gamma_2 = \Gamma_3 = -\Gamma_1 \quad \text{where} \quad \Gamma_1 = \frac{Z-1}{Z+1} \quad (A6)$$

$$z^2 = e^{-2j\theta} \quad \text{where} \quad \theta = \gamma d \quad (A7)$$

$$T_{12} = 1 + \Gamma_2 \quad (A8)$$

$$T_{21} = 1 + \Gamma_1 \quad (A8)$$

$$S_{11} \equiv \Gamma_{IN} = \frac{\Gamma_1(1 - z^2)}{1 - \Gamma_1^2 z^2} \quad (A9)$$

An identical procedure is used to calculate the sum of all of the wave components transmitted completely through the material sample. This summation is equal to the measured value of S_{21} for the filled section of waveguide – see A10 through A12.

$$Total \ Transmitted = \frac{T_{32}T_{21}e^{-j\theta}}{1 - \Gamma_2\Gamma_3e^{-2j\theta}} \quad (A10)$$

$$where \ T_{32} = 1 + \Gamma_2 \quad (A11)$$

$$S_{21} \equiv Total \ Transmitted = \frac{z(1 - \Gamma_1^2)}{1 - \Gamma_1^2 z^2} \quad (A12)$$

The measured values of S_{11} and S_{21} are now expressed in terms of two unknowns Γ_1 and z whose values are determined by the complex permittivity and permeability of the material sample under test. Calculating the values of these two unknowns and hence the material properties involves the use of the sum and difference of the S-Parameters as follows.

$$V_1 = S_{21} + S_{11} \quad (A13)$$

$$V_2 = S_{21} - S_{11} \quad (A14)$$

$$V_1 V_2 = S_{21}^2 - S_{11}^2 = \frac{z^2 - \Gamma_1^2}{1 - \Gamma_1^2 z^2} \quad (A15)$$

$$V_1 - V_2 = 2S_{11} = \frac{2\Gamma_1(1 - z^2)}{1 - \Gamma_1^2 z^2} \quad (A16)$$

$$X = \frac{1 - V_1 V_2}{V_1 - V_2} = \frac{1 + \Gamma_1^2}{2\Gamma_1} \quad (A17)$$

$$\sqrt{X^2 - 1} = \frac{\Gamma_1^2 - 1}{2\Gamma_1} \quad (A18)$$

$$\Gamma_1 = X \pm \sqrt{X^2 - 1} \quad (A19)$$

The choice of sign in A19 is therefore made so as to give a physically plausible solution i.e. $|\Gamma_1| \leq 1$. Having done this the characteristic impedance of the filled section of line can be calculate by rearranging equation A6 to make Z the subject.

With Γ_1 determined it can be used in conjunction with S_{11} and S_{21} to determine the value of the complex exponential z.

$$V_1 - \Gamma_1 = \frac{(1 - \Gamma_1^2)(z - z^2 \Gamma_1^2)}{1 - \Gamma_1^2 z^2} \quad (A20)$$

$$1 - V_1 \Gamma_1 = \frac{1 - z\Gamma_1 + z\Gamma_1^3 - \Gamma_1^2}{1 - \Gamma_1^2 z^2} \quad (A21)$$

$$\frac{V_1 - \Gamma_1}{1 - V_1 \Gamma_1} = \frac{(1 - \Gamma_1^2)(z - z^2 \Gamma_1^2)}{1 - \Gamma_1^2 z^2} \times \frac{1 - \Gamma_1^2 z^2}{(1 - \Gamma_1^2) - z\Gamma_1(1 - \Gamma_1^2)}$$

$$\frac{V_1 - \Gamma_1}{1 - V_1 \Gamma_1} = \frac{z(1 - z\Gamma_1)}{1 - z\Gamma_1} = z \quad (A22)$$

As shown previously in A7 the complex exponential term z is related to the complex propagation constant of the material under test which can now be explicitly calculated by making use of de Moivre's theorem – see Stephenson (1990).

$$z^n = (\cos \theta + j \sin \theta)^n$$

$$\frac{1}{z^n} = (\cos \theta + j \sin \theta)^{-n}$$

$$\frac{1}{z} = e^{\gamma d} \quad (A23)$$

$$\ln \frac{1}{z} = \gamma d \quad \text{where } d \text{ is the sample thickness}$$

$$\frac{1}{d} \ln \frac{1}{z} = \gamma \quad (A24)$$

As described by Lederer (1990) knowledge of the sample length, propagation constant and the normalised characteristic impedance of the filled section of waveguide provides all of the information required to determine the material's complex permittivity and permeability. The exact expressions used to give the material properties depend upon the relationship between the normalised characteristic impedance of the filled line when supporting the waveguide's fundamental mode of propagation and the normalised intrinsic impedance of the material sample. The two most commonly used waveguides for microwave T / R characterisation of materials namely coaxial line and rectangular waveguide are discussed here.

The fundamental mode of propagation in a coaxial waveguide is the TEM mode and as such the normalised impedance of the filled line section is the same as that of an infinitely long material sample in freespace – see equation A25.

$$Z \equiv Z_{\text{infinite material}} = \sqrt{\frac{\mu_r^i}{\epsilon_r^i}} \quad (\text{A } 25)$$

The propagation constants are also identical so use can therefore be made of A24 using the approach used by Lederer (1990).

$$\gamma = j \frac{2\pi}{\lambda_o} \sqrt{\mu_r^i \epsilon_r^i} \quad (\text{A } 26)$$

$$N_m = \text{material refractive index} = \sqrt{\mu_r^i \epsilon_r^i} \quad (\text{A } 27)$$

$$N_M = -j \frac{\lambda_o}{2\pi} \gamma \quad (\text{A } 28)$$

The material's complex permittivity and permeability are calculated by combining A25 and A27 as follows:

$$\mu_r^i N_m Z_m \quad (\text{A } 29)$$

$$\epsilon_r^i = \frac{N_m}{Z_m} \quad (\text{A } 30)$$

In a rectangular waveguide the guided wavelength λ_g differs from the freespace value λ_o according to A31. λ_c is the low-frequency cutoff wavelength of the mode in

question. When using the NRW equations this is the TE₁₀ mode which has a value of λ_c equal to twice the longest transverse dimension of the waveguide.

$$\lambda_g = \frac{\lambda_o}{\sqrt{1 - \left(\frac{\lambda_o}{\lambda_c}\right)^2}} \quad (A\ 31)$$

The characteristic impedance of an air-filled section of rectangular waveguide is also different to the freespace value according to A32 while the characteristic impedance of the sample filled section is given by A33.

$$Z_{AIR} = \sqrt{\frac{\mu_o}{\epsilon_o}} \frac{\lambda_g}{\lambda_o} \quad (A\ 32)$$

$$Z_{SAMPLE} = j \frac{2\pi f \mu_r \mu_o}{\gamma_{guide}} \quad (A\ 33)$$

$$where \gamma_{guide}^2 = \gamma^2 + \left(\frac{2\pi}{\lambda_c}\right)^2 \quad (A\ 34)$$

The normalised characteristic impedance Z in A6 therefore relates to the dielectric and magnetic properties of the sample filling the waveguide as follows:

$$Z = \frac{Z_{SAMPLE}}{Z_{AIR}} = j \frac{2\pi f \mu_r \mu_o}{\gamma_{guide}} \times \frac{\lambda_o}{\lambda_g} \sqrt{\frac{\epsilon_o}{\mu_o}} \quad (A\ 35)$$

$$\mu_r^{\dot{i}} = \frac{\gamma_{guide} Z \frac{\lambda_g}{\lambda_o}}{j \frac{2\pi}{\lambda_o}} \quad (A\,36)$$

$$N_m = \frac{1}{j \frac{2\pi}{\lambda_o}} \sqrt{\gamma_{guide}^2 - \left(\frac{2\pi}{\lambda_c}\right)^2} \quad (A\,37)$$

$$\epsilon_r^{\dot{i}} = \frac{N_m^2}{\mu_r^{\dot{i}}} \quad (A\,38)$$

The numerical shortcoming of the NRW equations comes in A24 where the natural logarithm of a complex number has to be calculated. Choice of the correct root is essential to correctly calculating the complex permittivity and permeability of the sample under test.

Appendix II: Sample Preparation

To try and fabricate low-loss magnetically tuneable binary composites both the dielectric host and garnet loss tangents had to be minimised. For combined lowest magnetic and dielectric losses Indium substituted Ca-V garnet powders were used as loading materials. The two saturation magnetisation formulations chosen were predetermined by the decision to use two-port rectangular waveguide T / R measurements as the principle means of material characterisation. WR90 waveguide was available and could be readily machined to form a TRL calibration kit thereby fixing the material characterisation and microstrip device design bandwidths to be 8.2 – 12.4GHz. Combining this with a desire to minimise losses and make use of Pucel and Massé's (1972) magnetic microstrip substrate analysis required the ratio of garnet gyromagnetic resonance frequency to operating frequency to be less than or equal to 0.75. The Indium substituted CA-V powders selected therefore had saturation magnetisations of 1200 Gauss (95.52kAm⁻¹) and 1600 Gauss (127.36kAm⁻¹) for ratios at 10GHz of 0.336 and 0.448 respectively. The characteristics sought after in the host dielectric were a low, less than five, real component of permittivity to give filling factor flexibility without recourse to extremely narrow microstrip lines when trying to obtain Z₀ of 50Ω; unreactivity with the garnet powders; the ability to be easily shaped into three-dimensional forms such as rectangular blocks for waveguide testing and microstrip substrates. Ideally, the host dielectric was to have a loss tangent on the order of 10⁻³ or smaller.

The first material considered was Cotronics Corporation's RTC60, a slip-castable 96% Alumina powder. Unlike conventional ceramics this required no high temperature sintering instead a two hour bake at 110°C to remove any water

remaining after twenty-four hours setting at room temperature. Predominantly composed of Alumina it was thought that the RTC60 would be a low-loss host. Attempts to quantify this and determine its real component of permittivity using both two-port WR90 T / R and rod resonator techniques proved otherwise. Losses were very high and the real component of permittivity was found to vary quite considerably. This was attributed to a combination of inconsistent samples density, air pocket removal proved difficult, and the fact that the material was originally intended for refractory applications such as furnace tray fabrication. In order to have a sufficient degree of thermal shock resistance and good continuous high temperature durability the RTC60 formulation most likely contained a mixture of conductive, hence lossy, clays and glassy phases. Difficulties encountered in trying to make WR90 waveguide samples were estimating the degree of post-cure shrinkage and the very soft, easily damaged, nature of the material. Airgaps between the sample and waveguide walls were therefore unavoidable enabling high order evanescent modes to be supported causing nonsensical permittivity and permeability values to be calculated by the NRW equations. Further investigation of the material was not pursued.

The second host dielectric considered was Microchem SU8-500 negative photoresist. Originally developed by IBM for the manufacture of advanced CMOS devices the resist is based upon Shell Chemicals' EPON SU8 epoxy resin dissolved in an organic solvent with an ultra-violet (UV) photoinitiator – see Shaw et al (1997). The resist has a very low optical absorption in the near-UV part of the spectrum giving it a high degree of optical sensitivity. This has brought widespread use in the micromachining community where complicated three-dimensional structures with near perfect $\sim 89^\circ$ wall profiles and aspect ratios as high as 14:1 have been demonstrated – see Acosta et

al (1996). Little characterisation of SU8's dielectric loss seems to have been done at microwave frequencies although time –domain spectroscopy data from Arscott et al (1999) claimed a highly promising loss tangent of 6.3×10^{-6} at 1THz. A review of the experimental data by Lucyszyn (2001) disputed this value instead calculating dielectric loss tangents of 0.14 at 1THz and 0.08 at 100GHz. This latter value compared quite favourably with the loss tangent of 0.02 ± 0.001 reported by Collins et al (1998) from measurements in 75 – 110GHz rectangular waveguide. Despite this value being two orders of magnitude higher than the maximum manufacturer reported dielectric loss tangent of the garnet powders it was decided to try and fabricate garnet loaded SU8 composites.

Due to the aforementioned optical sensitivity and high aspect ratio performance of SU8-500 fabrication of pure resist samples that accurately filled the internal cross-section of rectangular WR90 waveguide was not problematic. Difficulties were experienced in producing samples thicker than the $\sim 500\mu\text{m}$ achieved in a single resist processing cycle. This alone took seven hours of cleanroom time and with no simple linear scaling of processing time with sample thickness a multiple layer approach to developing samples several millimetres in thickness was not really feasible. Fabrication of solid cylinders of pure SU8 resist tens of millimetres in diameter and length for rod-resonator measurements had to be abandoned as an unrealistic proposition.

Attempts to fabricate garnet loaded resist samples also met with difficulties. The WR90 specimens proved to be firmly bonded to the glass discs on which they had been spin coated. This was felt to be a result of incomplete crosslinking of the SU8 epoxy due to scattering and / or absorption of the UV light by the garnet powders.

Increasing exposure times as well as exposing through the glass substrate to illuminate the sample bases proved ineffective in overcoming the problem. A variety of 'lift-off' coatings on top of the glass substrates were subsequently tried including DC magnetron sputtered films of Gold and Barium-Calcium-Copper-Oxide (BaCaCuO) superconductor precursor. The Gold films were totally ineffective, no lift-off occurring, while the BaCaCuO precursor etched easily away in 63% Nitric acid enabling the samples to be removed from the substrate. Due to the low degree of UV initiated crosslinking the post-developer rinsed samples were extremely thin, curling up at the edges and cracking when free of the substrate – see figure A2. To try to avoid this further attempts were made with samples coated in pink dental wax to provide mechanical support prior to removal of the precursor layer. The samples could then be handled with tweezers and the substrate side UC exposed. The samples again fractured which was thought to be due to differential contraction when crosslinking. Aluminium foil wrapped glass discs were also tried as substrates for spin coating. Following resist processing the samples were wax coated and the Aluminium etched away in a lightly heated 7.5M Potassium Hydroxide (KOH) solution.



Figure A2:- Incompletely Crosslinked Garnet Loaded SU-8 Composites

When removed from the Aluminium the backs of the samples were found to be uneven and full of voids. The same problem occurred when using Silicon wafers as a sacrificial substrate indicating poor alkali tolerance as the cause of the problem rather than lack of substrate flatness.

The final dielectric investigated, and subsequently used, was Aremco Products' Crystalbond™ 509. This thermosetting adhesive was originally intended for mounting hard, brittle materials such as ferrites and other ceramics for diamond machining. Crystalbond™ 509 is solid and machinable with conventional steel tooling at room temperature, dissolves in Acetone and can be molded when heated above 121°C. Fabrication of composites using the solid adhesive was very straightforward requiring it to be crushed into a fine powder with a mortar and pestle. A WR90 test-cell resting on an Aluminium foil covered hotplate was packed with the powdered dielectric. Raising the temperature of the hotplate to around 150°C melted the Crystalbond™ which was gently stirred to remove air bubbles. An identical technique was used to produce the composites with the garnet and dielectric powders mixed in the desired volumetric ratios before being packed into the WR90 test-cell. After cooling, the Aluminium foil was peeled from the back of the test-cells which were clamped in a lathe and excess material, the test-cells having been deliberately overfilled, machined away to leave the sample faces flush with the waveguide flanges. This helped to reduce uncertainties in sample length as well as reducing any

surface unevenness that could have helped support high order evanescent waveguide modes.

WR90 waveguide samples of Indium substituted Ca-V garnets C4A and C6A were diamond cut from 50mm diameter round circulator substrates rejected by the manufacturer due to edge chipping and the presence of pinholes. The resulting rectangular samples' edges and corners were hand ground using a series of increasingly fine slurries of Silicon Carbide. Final finishing to obtain a close fit inside the waveguide with minimal airgaps used fine synthetic Diamond loaded paste.

Attempts were made to produce solid garnet cylinders for rod-resonator measurements of complex permittivity and scalar permeability using pre-sintered powders from the manufacturer. These were carefully mixed with Polyvinyl Alcohol (PVA) and Polyethylene Glycol (PEG) dissolved in warm water, dried and crushed to a fine powder with a mortar and pestle. The PVA was used as a binding agent to help the garnet granules pack closely together and adhere to one another when diepressed to help increase the density of and mechanical strength of greenbody samples. As described by Ring (1996) the PEG was used as a plasticiser to decrease the glass transition temperature (T_g) of the PVA. Above T_g the PVA would flow easily under pressure so decreasing the actual pressure needed to diepress the garnet powder into a green body sample.

The PVA-PEG coated garnet powder was poured into a nonmagnetic Phosphor Bronze uniaxial press and compressed at 100MPa for ten minutes before release – see figure A3. Phosphor Bronze was used for the press body to overcome problems of green body fracture experienced when samples were removed from an all-steel press.



Figure A3:- Diepress for Uniaxial pressing of TE_{01n} Garnet Resonators

This was attributed to increased adhesion of the powder to the press walls which had been magnetised by the machining process and friction between the press walls and punch. The internal press diameter was carefully bored oversize to try and exactly compensate for the sample shrinkage occurring during sintering. The aim was to remove the need for diamond grinding of the sintered samples to obtain the 12mm outer diameter calculated from the manufacturer supplied material properties and Kobayashi and Tanakas's (1980) mode charts to give a nominal 10GHz TE_{01n} resonance well separated from the surrounding modes. An initial estimate of 20% was made for the sintering induced shrinkage and an appropriately sized press produced. Trial sintering of both powders with the design revealed the actual shrinkage to be approximately 14.3%. All subsequent samples were therefore made using a 14mm internal diameter press.

Green body samples were subjected to a three stage heat treatment in air to both dry them and burn-out the organic binder and plasticiser. Early attempts at fabrication had omitted the high temperature stage in the binder burnoff leading to incomplete

removal of the organic chemicals and the presence of large voids in the final sintered material. Sample sintering was at first carried out in a vacuum furnace with a 100% Oxygen atmosphere for the last 100°C of the heating ramp and the first hour of the six hour sinter. Attempts at rod-resonator measurements of the resulting samples showed none of the expected mode structure due to extremely high losses totally different from those reported by the powder manufacturer. Scanning electron microscope (SEM) images of the both the exterior and, after grinding, interior of the samples showed obvious surface melting and cracking and the presence of large voids within the material – see figures A4 and A5. In comparison SEM images of one of the rejected C6A circulator substrates provided by the manufacturer show no porosity or melting with a very even grain structure – see figure A6.

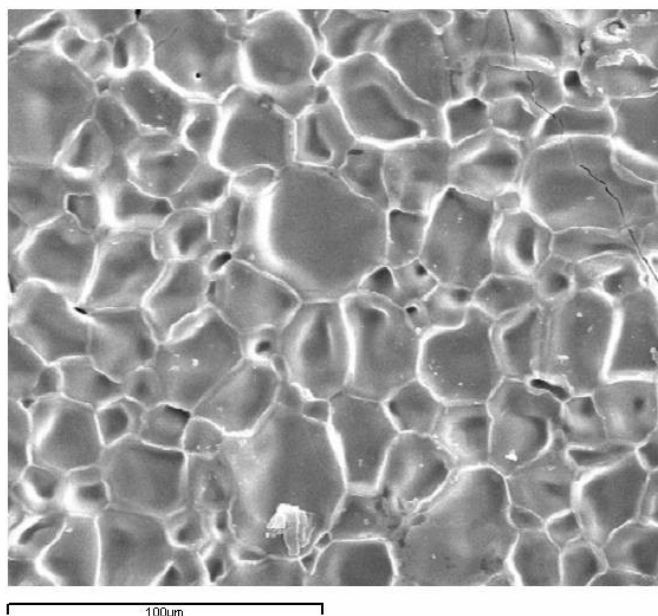


Figure A4:- SEM Image of Exterior of Vacuum Furnace Sintered C6A

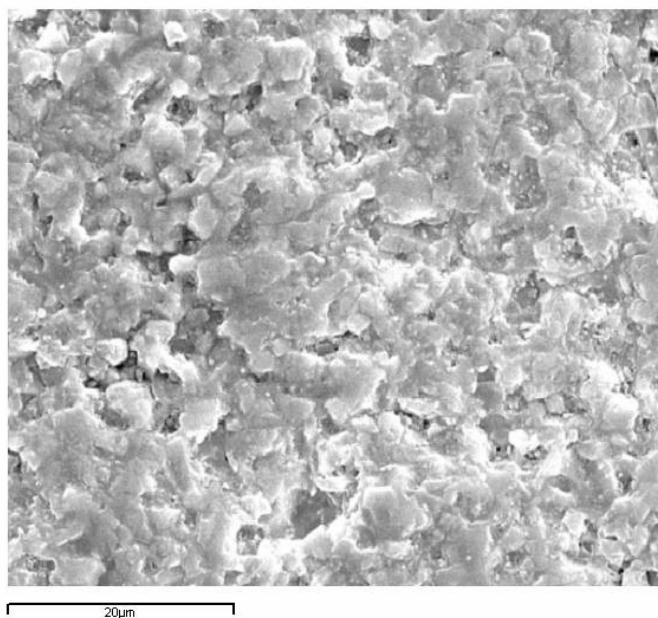


Figure A5:- SEM Image of Interior of Vacuum Furnace Sintered C6A

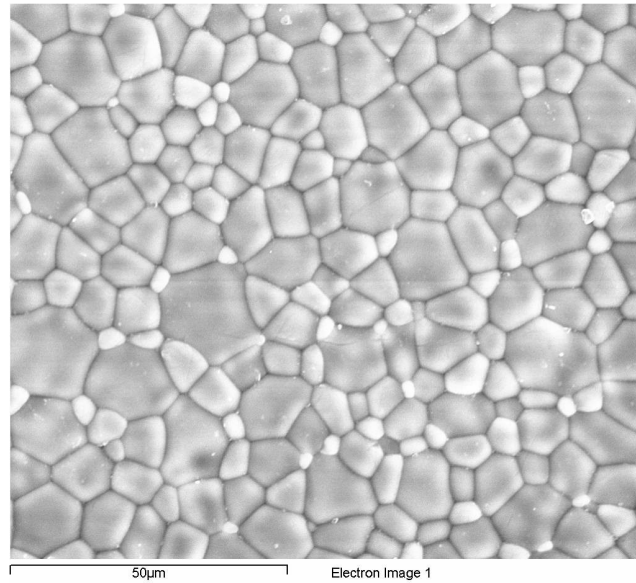


Figure A6:- SEM Image of Manufacturer Supplied C6A

X-ray diffraction analysis of the vacuum furnace sintered resonator samples in comparison with the manufacturer supplied circulator substrates revealed totally different stoicheiometries – see figure A7.

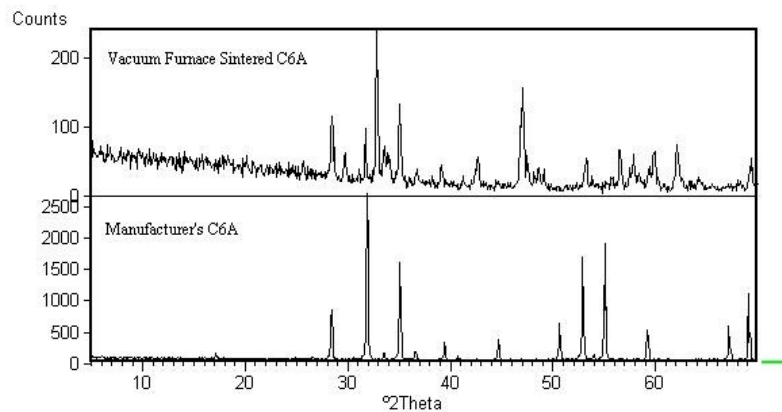


**Figure A7:- Comparison of XRD Responses of Vacuum Furnace Sintered C6A
and Manufacturer's C6A**

Further XRD analysis indicated that the high levels of loss were not only due to the absence of the correct substituted YIG material and the presence of voids and cracks but that the vacuum furnace sintering was caused the samples to contain a large proportion of electrically conductive Haematite (Fe_2O_3) and Calcium Oxides – see figure A8.

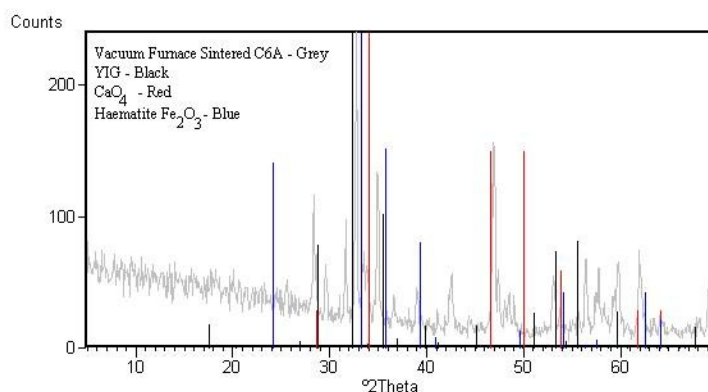


Figure A8:- XRD of Vacuum Sintered C6A Showing Presence of Haematite and Calcium Oxides

Careful searches of the available substituted Ca-V garnet literature and subsequent informed questioning of the garnet powder manufacturer revealed that sintering needed to be carried out in an Oxygen enriched air atmosphere. Difficulties in achieving the required sintering conditions and ensuring optimum green body sample density lead to the abandonment of attempts to produce bulk sintered TE_{01n} resonators.

To demonstrate the suitability of the garnet loaded composites for tuneable planar device fabrication straight microstrip resonators were fabricated on 0.3 and 0.4 volume fraction C4A and C6A loaded substrates. These were produced in much the same way as the pure Crystalbond™ WR90 samples described previously. Crystalbond™ and garnet powders were mixed in the desired volumetric ratios and

then packed into Aluminium mold / substrate holders to form 50mm x 50mm x 1mm substrates – see figure A9. These were placed onto a hotplate with removable endplates screwed into the mold / substrate holders. With the temperature raised to around 150°C the Crystalbond™ component melted and, as with the WR90 specimens, gentle stirring used to give a uniform garnet distribution.

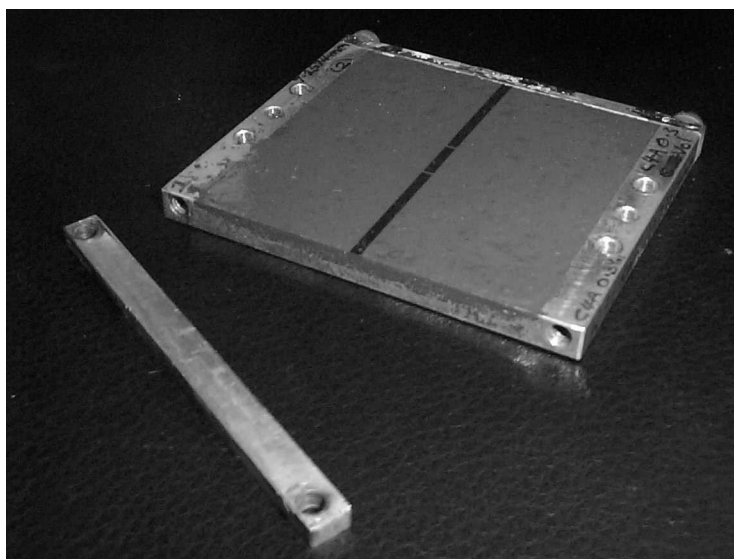


Figure A9:- Mold / Sample Holder for Composite Substrate Fabrication and Testing

The mold / substrate holders were all deliberately overfilled with composite so that after cooling the excess could be machined away to give a flat substrate of uniform 1mm thickness. Following machining the samples were returned briefly to the hotplate to reflow the composite so as to remove any small voids and give a smooth surface.

Due to the solubility of Crystalbond™ in Acetone, and by implication in most organic solvents, the microstrip lines were defined using physical masks rather than by photolithography. Initial attempts at mask fabrication using 80μm thick Beryllium

Copper (Be-Cu) sheet with coupling gaps one tenth of the linewidths, minimum gap size $130\mu\text{m}$, proved unsuccessful due to a lack of mechanical strength in the etched Be-Cu sheet. Use of $178.15\mu\text{m}$ thick Be-Cu with $320\mu\text{m}$ wide gaps for all designs overcame the robustness problem. To avoid overetching of the Be-Cu and to preserve the intended device dimensions the resonator designs were photolithographically defined on both sides of the Be-Cu allowing double sided etching. The resulting coupling lines, coupling gaps and resonators were found to be within $20\mu\text{m}$ of the design dimensions. The Copper microstrip lines were deposited onto the substrates by thermal evaporation through the 'windows' etched into the Be-Cu sheets. To minimise linewidth increases the masks were tightly screwed into their respective mold / substrate holders to give as flush a fit as possible. The evaporated metallisation thicknesses were found to vary from $0.56\mu\text{m}$ to $0.716\mu\text{m}$ which is on the order of one skindepth ($0.661\mu\text{m}$) at 10.00075GHz .

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