Dielectric measurements from 10^{-5} to 10^{11} Hz

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Abstract

This paper describes the use of two dielectric measurement systems to provide complex dielectric constant data over the frequency range 10^{-5} to 10^{11} Hz for a wide range of materials. A Hewlett Packard 8510C Vector Network Analyser system is used for frequencies above 0.5 GHz. This system uses the Nicholson-Ross-Weir technique to extract both complex dielectric constant and magnetic permeability from samples loaded into coaxial and/or waveguide transmission lines. A Novocontrol BDS Broadband Dielectric Spectrometer is used for frequencies up to 1.8 GHz. The Novocontrol system also enables temperature dependent measurements over the range -160 to 400 °C and the measurement of magnetic permeability over the frequency range 1 MHz to 1.8 GHz.

Measurements are presented on insulating, conducting and magnetic materials. Both polymeric and ceramic materials have been characterised. Lower frequency/temperature dependent data is used to study both conduction and polarisation processes at a fundamental level, where a wide range of theoretical models can be used to fit the measurement results. Higher frequency data is more often used in the electromagnetic design of materials such as those used in electromagnetic shielding applications. The implications of sample preparation are also discussed in terms of their impact on measurement uncertainty.

1 Introduction

The low observable materials group at DERA Farnborough has been involved in the microwave characterisation of materials for over ten years following a requirement to predict the microwave reflectivity of composite multi-layer structures.(1) To achieve this a database of complex permittivity and permeability measurements on a wide range of dielectric and magnetic materials has been established. The majority of this data has been obtained using a Hewlett Packard 8510C Vector Network Analyser system covering the frequency range 0.5 to 110 GHz. The group have recently acquired a Novocontrol BDS Broadband Dielectric Spectrometer to extend its measurement range down to 10^{-5} Hz. The techniques are subject to the requirements of BS EN ISO 9001(2) and re-accreditation to NAMAS standard(3) of the Hewlett Packard system over the frequency range 0.5 to 18 GHz is currently being pursued following a major laboratory relocation programme. This paper aims to provide an overview of the measurement techniques and the diversity of materials that can be characterised.

2 The dielectric/magnetic test systems

2.1 Frequency range 0.5 to 110 GHz

The measurements are fully automated and are undertaken using a Hewlett-Packard (HP) 8510C Vector Network Analyser. A HP8515A S-Parameter Test Set is used in conjunction with 7 mm coaxial and WG14, WG16, WG18 and WG20 waveguide transmission line sections. For higher frequency measurements using WG22, WG24, WG25 and WG27 waveguide transmission lines, S-parameter test sets have been constructed from the appropriate waveguide components. Figure 1 presents a schematic view of the measurement system. The measured S-parameters are converted into complex relative permittivity (ε_r^*) and permeability (μ_r^*) using a transmission line method.(4,5) The specific technique currently used by the laboratory is that of Nicolson-Ross-Weir (NRW)(6,7) as given by equations (1) and (2). Previous papers(8,9) enlarge on the traceability and uncertainty aspects of this system relating to the ISO9001 and NAMAS quality standards. Samples for use with coaxial transmission lines are required to have a toroidal geometry, where as those for use with waveguide are rectangular in cross-section.

$$\varepsilon_r^* = \frac{\left(\frac{1}{\Lambda^2} + \frac{1}{\lambda_c^2}\right)\lambda_0^2}{\mu_r^*} \tag{1}$$

$$\mu_r^* = \frac{1+\Gamma}{\Lambda(1-\Gamma)\sqrt{\frac{1}{\lambda_0^2} - \frac{1}{\lambda_c^2}}} \tag{2}$$

with

$$\Lambda = \frac{j}{\left(\frac{-1}{2\pi d}\right)\ln(T)}$$

$$T = \frac{S_{11} + S_{21} - \Gamma}{1 - (S_{11} + S_{21})\Gamma}$$

$$\Gamma = \kappa \pm \sqrt{\kappa^2 - 1}$$

$$\kappa = \frac{S_{11}^2 - S_{21}^2 + 1}{2S_{11}}$$

where S_{mn} are the measured scattering parameters, d is the sample thickness, λ_0 is the free-space wavelength and λ_c is the cut-off wavelength of the transmission line.

2.2 Frequency range 10^{-5} Hz to 1.8 GHz

The Novocontrol BDS is based around two impedance analysers which enable dielectric measurements over 15 orders of magnitude in frequency. A Hewlett Packard 4291 is used for frequencies above 1 MHz and a Solartron 1260 is used for frequencies below 10 MHz. The combination of these instruments has the obvious advantage of offering an order of magnitude overlap in frequency. The dynamic range of the latter is further enhanced using the Novocontrol 'Broadband Dielectric Converter', extending the upper impedance limit from 100 MΩ to 200 TΩ and increasing the resolution in loss tangent from <4 mrad to <0.1 mrad. A temperature control system is also included which enables measurements over the temperature range -160 to 400 °C. Finally, measurements of magnetic permeability are afforded above 1 MHz through the use of the Hewlett Packard 16454A test fixture. Figure 2 presents a schematic view of the measurement system. The complex relative dielectric constant (ε_r^*) is derived, assuming an ideal parallel plate capacitor (although corrections for fringing fields can be enabled), as the ratio of the capacitance with the sample between the plates (C_P^*) to the capacitance of the empty cell (C_0) (as given by equation (3)). The complex relative permeability (μ_r^*) is similarly derived as a ratio of the sample inductance (L_S^*) to the inductance of the empty cell (L_0), as given by equation (4). Samples for dielectric measurements with this system are required to have disk geometries, those for magnetic measurements have toroidal geometries.

$$\varepsilon_r^* = \frac{C_P^*}{C_0} \tag{3}$$

$$\mu_r^* = \frac{L_S^*}{L_0} \tag{4}$$

3 Considerations for sample preparation

Sample preparation is an essential element for all dielectric measurements. In particular, for the measurement techniques reported here sample thickness is used in the derivation of the dielectric/magnetic properties from the measured data (either complex impedance or scattering parameters). In this case, the uncertainty in the measurement of this dimension is directly transferred to the result. For transmission line measurements over the range 0.5 to 18 GHz it has been shown(9) that measurement uncertainty is dominated by the contribution from the uncertainties of sample thickness and of positioning the sample at the calibration plane when the combination of these uncertainties is of the order of 50 μm. This limit will become even more stringent at higher frequencies since the dimensional uncertainty relates to the phase uncertainty which, for a fixed dimensional uncertainty will scale with increasing frequency. Air gaps between the sample sides and the walls of the transmission line can also have a significant affect on the measured results. Again, previous work has shown that 10 μm air gaps can be tolerated for measurements over the frequency range 0.5 to 18 GHz. Following these observations the laboratory routinely imposes dimensional tolerances of ±5 μm on its sample preparation.

In the case of lower frequency dielectric measurements, the flatness of the sample is critical since electrical contact is made to the sample faces as part of the measurement process. Therefore, it is recommended(10) that the surfaces of the sample are metallised, preferably by evaporation with gold. This is particularly important for more highly insulating materials and materials with large dielectric constants. Corrections may also be applied to account for the non-ideal nature of the sample capacitor (that is the existence of fringing fields) when suitable control is placed on the capacitor geometry. This approach is favoured over the use of guard electrodes when the sample capacitor geometry can be controlled and when measurements are required over such wide frequency ranges. The magnitude of the contribution to the measured capacitance due to the fringing fields (and hence the magnitude of the correction) can be minimised by optimising the electrode diameter, electrode thickness and sample thickness.(10)

In addition, for both measurement systems the sample thickness needs to be selected to minimise measurement uncertainty since there exist optimum ranges over which the respective equipments can make measurements of the scattering parameters or impedance. For transmission line measurement optimum sample thicknesses occur at odd integer multiples of the wavelength in the material, λ_g, as given by equation (1).

$$\lambda_g = \mathrm{Re}\left(\frac{1}{\sqrt{\frac{\varepsilon_r \mu_r}{\lambda_0^2} - \frac{1}{\lambda_c^2}}}\right) \qquad (5)$$

For dielectric measurements with the Novocontrol system, the optimum sample thickness is different for the two measurement ranges and is quantified by ideal capacitances which can be used to calculate appropriate sample thicknesses (d), again using an estimate of the sample's dielectric constant using equation (6). The ideal capacitance range for frequencies below 10 MHz is 50-200 pF, for the higher frequency range the capacitance should ideally lie between 2 and 10 pF.

$$d = \varepsilon'_r \varepsilon_0 \frac{\pi D^2}{4C} \qquad (6)$$

where C is the sample capacitance, ε'_r is the real component of the samples relative permittivity, ε_0 is the permittivity of free space and D is the sample diameter.

4 Measurement results and discussion

4.1 Conducting polymers

Conducting polymers offer potential for electromagnetic screening and antistatic applications.(11) Measurement of their frequency and temperature dependent conductivity and dielectric constant enables an understanding of the conduction and polarisation mechanisms(12-14) leading to optimisation of their synthetic design, higher frequency data is required to quantify and design their screening effectiveness.(12,15)

Figure 3 presents a comparison of the dielectric spectra for doped and undoped polyaniline with that of polytetrafluoroethylene (PTFE). The latter is included for comparison and as a check of system performance. Two types of PTFE sample was used. The sample used for measurements above 1 MHz was die-pressed from powder, the sample used for lower frequency measurements was taken from a PTFE sheet. Measurements of PTFE in the range 0.5 to 18 GHz show the limitations of the transmission line technique in measuring materials with loss factors (ε") less than 0.01. Nevertheless, the measured values are in good agreement across the band and with values recorded in the literature(16).

Figures 4 shows, in more detail, the frequency and temperature dependence of the dielectric properties and conductivity for the doped polyaniline material. These measured results were analysed in an earlier paper(17) to determine which conductivity model provided the best fit. The first observation is that the dispersion in the complex permittivity as a function of frequency is indicative of a material incorporating hopping charges.(18) The exponent of the frequency dependence for the imaginary component of the complex permittivity at 303 K is also close to -1 which is consistent with a dc conductivity dominated loss mechanism. However, the strong low frequency dispersion in the real permittivity is due to a separate dielectric polarisation process. This is currently unidentified but could be due to ionic diffusion or interfacial effects. Finally, analysis of the temperature dependent conductivity suggests a good fit to both Austin-Mott variable range hopping or Sheng's thermal fluctuation induced tunneling models for temperatures below 223 K. Further experimentation is required to confirm the applicability of either model.

4.2 Other materials

To demonstrate the application of the measurement techniques to a wider range of materials: Figures 5 and 6 present complex permittivity data for a fibre reinforced plastic material, a Barium 'M' Hexaferrite and Macor ceramic; Figure 7 presents the magnetic permeability of another Barium 'M' Hexaferrite composition. The measurements of the ceramic samples (shown in Figures 6 and 7) were used in a measurement intercomparison with NPL.(9)

Figure 5 demonstrates the evolution of conductivity within the material as the temperature is increased. This conductivity is likely to be due to absorbed impurities which may include water. The figure also shows an evolving dielectric relaxation, the frequency of which is increasing with temperature. This is assigned to motion of the polymer chain, the mobility of which is increasing with temperature resulting in the increasing relaxation frequency.

The permittivity data shown in Figure 6 for the ferrite and Macor ceramics demonstrates the differences in the dielectric spectra for materials with (the ferrite) and without (Macor) a strong conduction mechanism. The nature of the conduction mechanism in the ferrite could be further probed using temperature dependent measurements as shown above for polyaniline.

Finally, the magnetic permeability spectra given in Figure 7 demonstrates a completely different process, namely the ferromagnetic resonance phenomenon which arises from the coupling of energy to the precessional motion of the magnetic moment of the ferrite crystal lattice.

5 Conclusions

This paper demonstrates the power of ac dielectric and magnetic measurements in terms of the wide range of materials that need to be characterised for a similarly diverse range of applications. The paper also highlights the care that needs to be exerted in the area of sample preparation to establish confidence in the measurement results.

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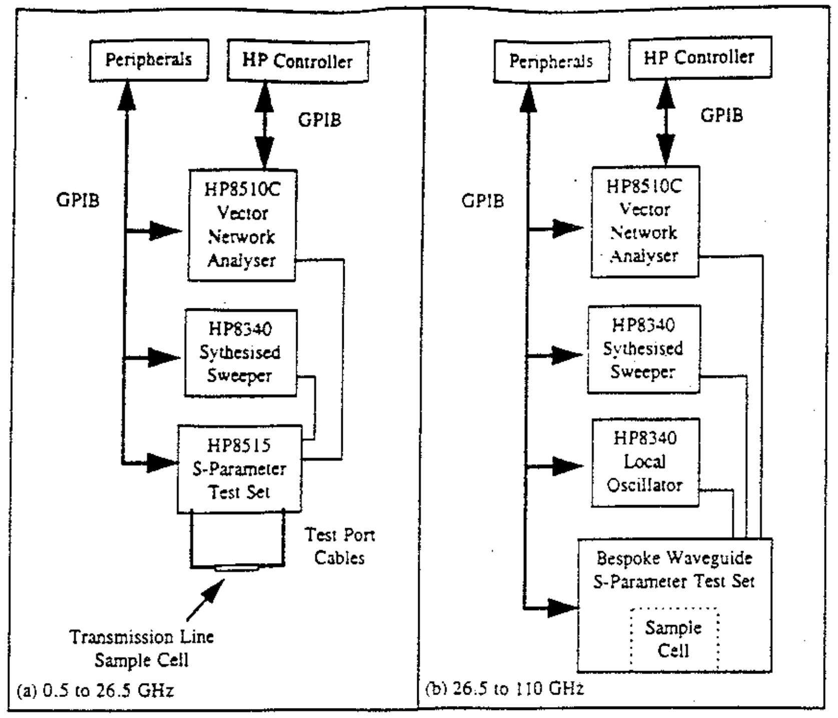

Figure 1: Schematic view of dielectric/magnetic test system for frequencies 0.5 to 110 GHz

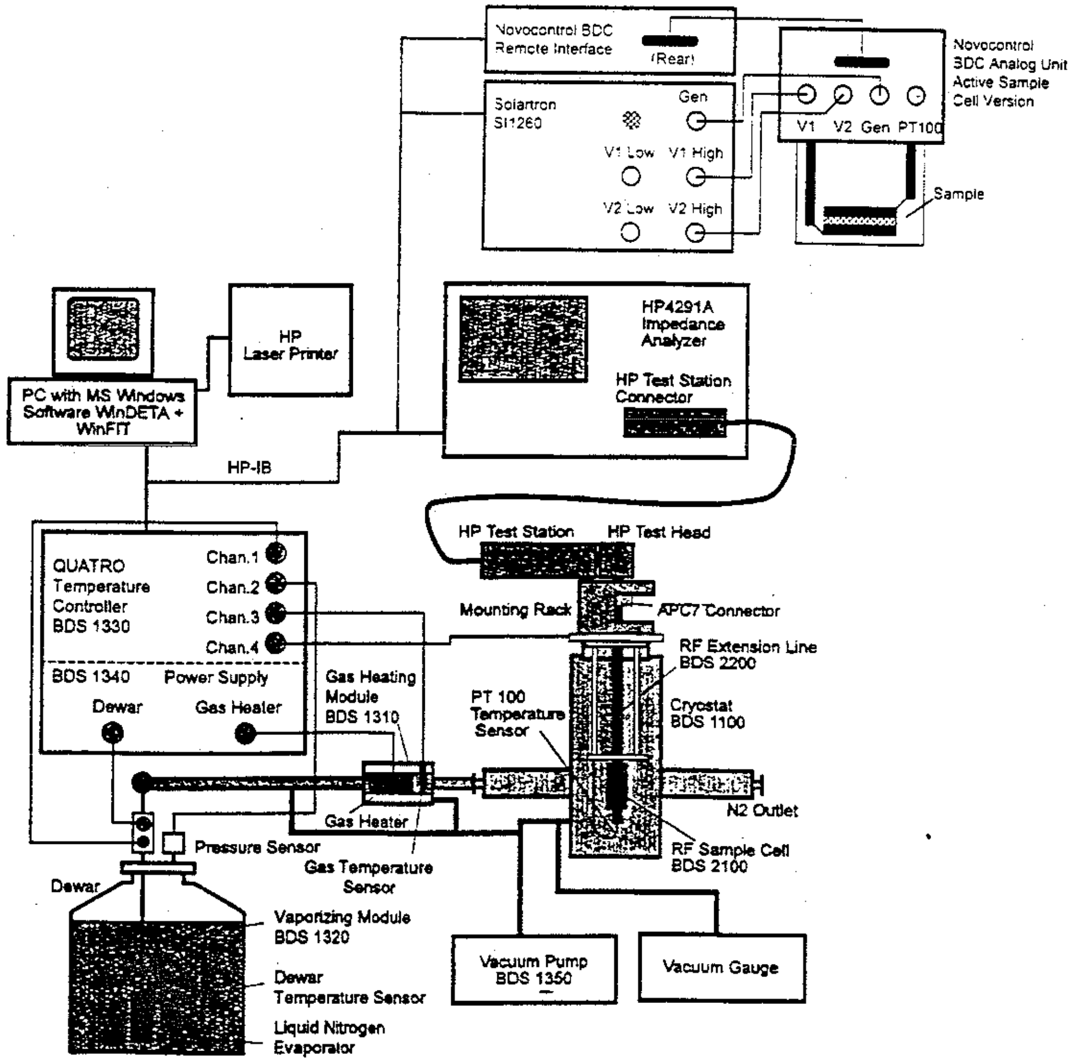

Figure 2: Schematic view of Novocontrol Concept 15 Broadband Dielectric Spectrometer[10]

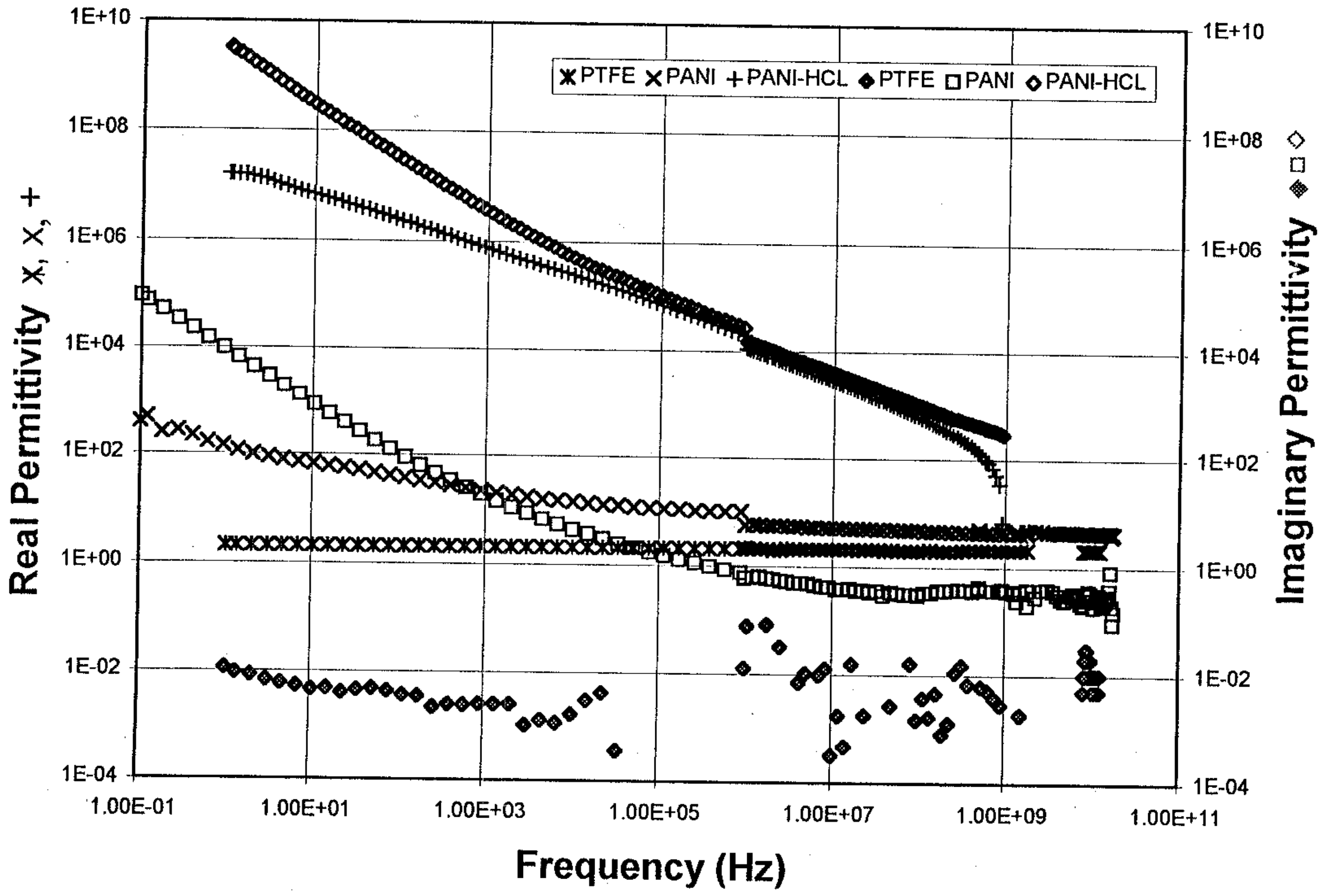

Figure 3: Dielectric properties of doped and undoped polyaniline, and polytetrafluoroethylene

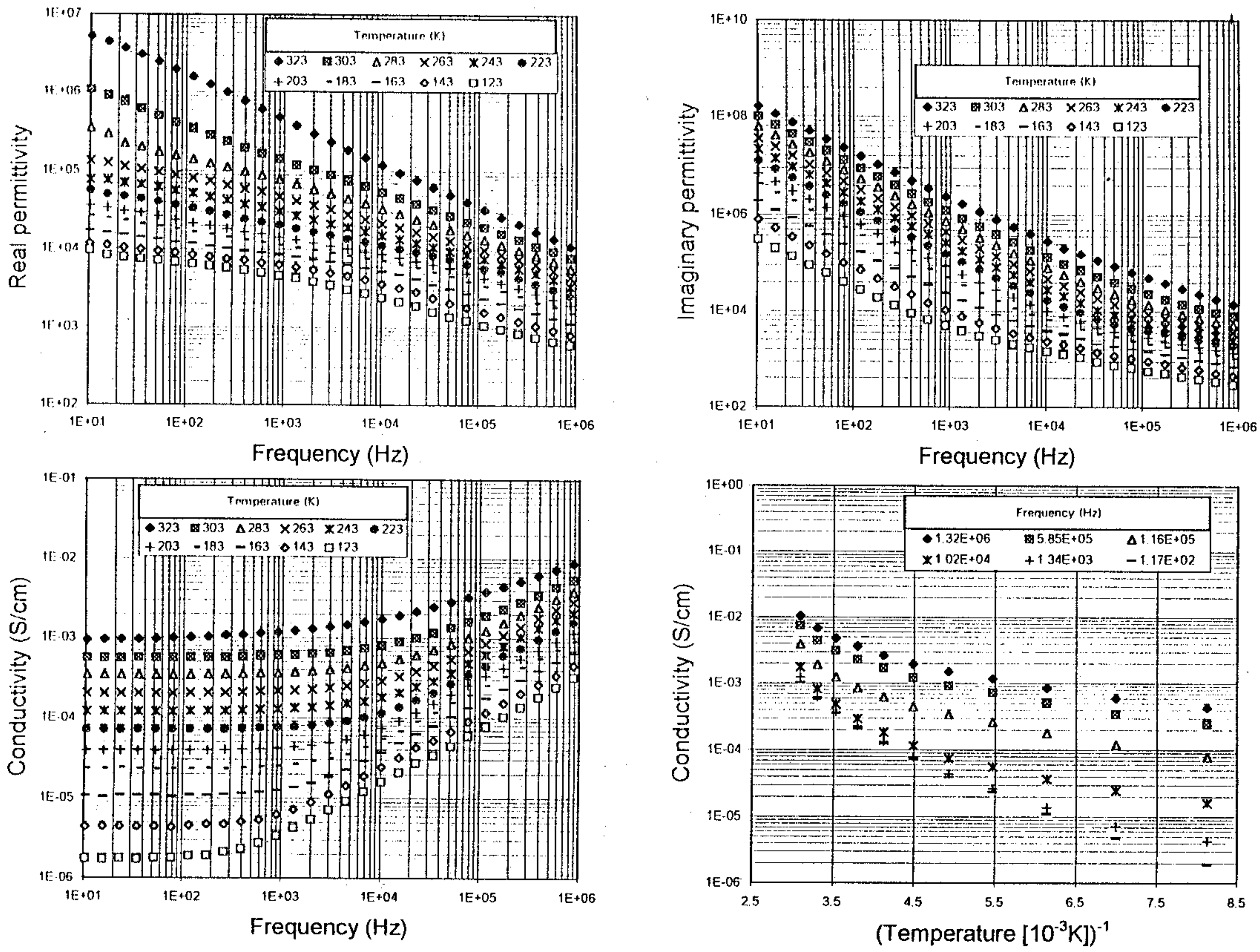

Figure 4: Frequency and temperature dependence of real permittivity and conductivity for doped polyaniline

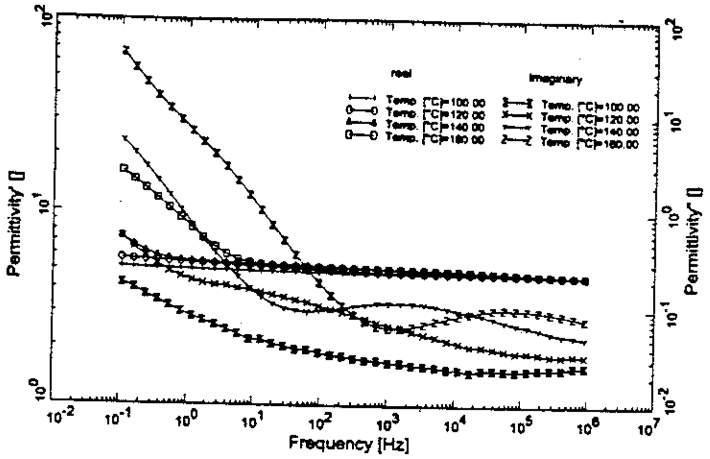

Figure 5: Dielectric properties of a fibre reinforced plastic

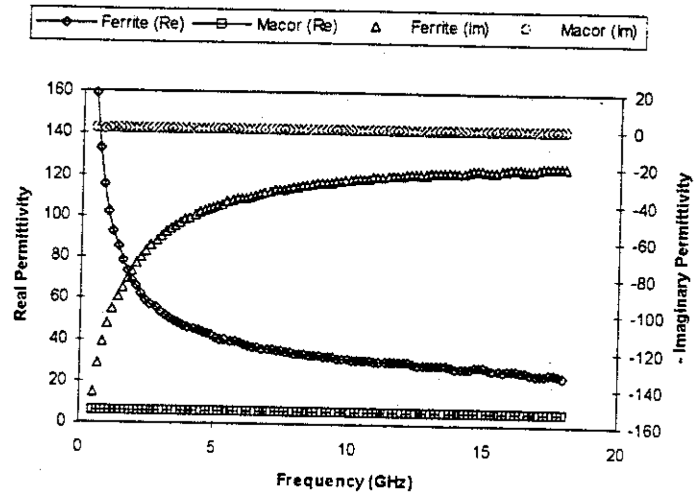

Figure 6: Dielectric properties of ferrite and macor ceramics

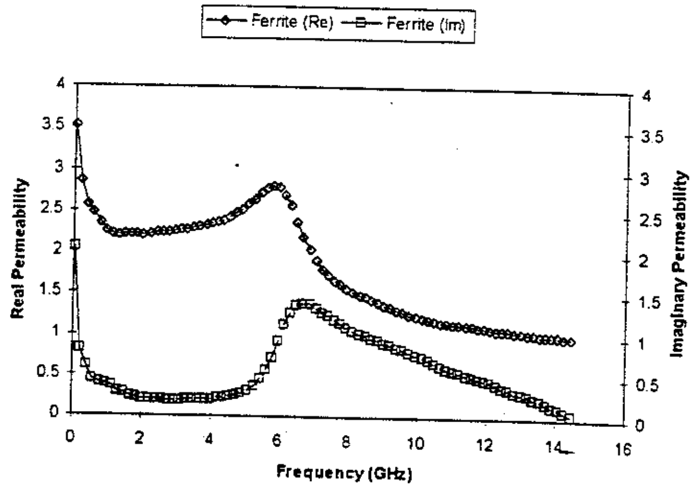

Figure 7: Magnetic properties of a barium M hexaferrite