An Important Step in Microwave Processing of Materials : Permittivity Measurements of Thermoplastic Composites at Elevated Temperatures

H S Ku[+] E Siores, J A R Ball[#] and B Horsfield[#]*

[#] Faculty of Engineering and Surveying, University of Southern Queensland (USQ).
* Professor and Executive Director, Industrial Research Institute Swinburne (IRIS).
+ PhD Candidate, IRIS; Member of Staff, USQ.

ABSTRACT

The dielectric constant, ε′, dielectric loss, ε″, and loss tangent, tan δ, of some commonly used thermoplastics have been measured[1,2,3] at various temperatures and frequencies. They do give some clues to the suitability of microwave processing of certain fibre-reinforced thermoplastic (FRTP) composites but cannot provide definite answers to the problems. Few measurements, if any, of the ε or the tan δ of FRTP composites have been reported in the literature. This paper describes a convenient laboratory based method designed to obtain ε′, ε″ and hence tan δ. The method employs an automatic logic network analyser and is called the waveguide transmission technique or transmission line method and is chosen because it provides the widest possible frequency range with high accuracy and utilised available the hardware and software. The required data were collected at a range of elevated temperatures and over a band of frequencies. The relationship between ε′ and ε″, or the tan δ of the composites and their weldability by microwave energy is also studied and discussed.

INTRODUCTION

The use of high energy rate joining of fibre-reinforced thermoplastic (FRTP) composites using microwaves has yielded promising preliminary results[4,5] and more research is being carried out so that the technology can find its application in manufacturing industries shortly. The material properties of greatest importance in microwave processing of a dielectric are the complex relative permittivity, $\varepsilon = \varepsilon' - j\varepsilon''$, and the loss tangent, $\tan\delta = \varepsilon''/\varepsilon'$. The real part of the permittivity, ε′, sometimes called the dielectric constant, mostly determines how much of the incident energy is reflected at the air-sample interface, and how much enters the sample; but the most important property in microwave processing is the loss tangent, tan δ which predicts the ability of the material to convert the absorbed energy into heat. As these electrical properties vary significantly with temperatures and frequencies, values attained at room temperature may not be appropriately used to predict the microwave-reactiveness of thermoplastic composites at elevated temperatures. This paper describes the measurement of ε and tan δ of three types of random, 33% by weight, glass-fibre or carbon-fibre reinforced thermoplastic composites. The three matrix materials chosen for the study are the commonly used thermoplastics, namely, low density polyethylene (LDPE), polystyrene (PS) and nylon 66. The sensitivity of the logic network analyser to heat resistance prevents measurements of the ε to be taken at a temperature higher than 100°C and the frequencies selected varies from 2.2 GHz to 12.5 GHz, which cover a broadband of the microwave spectrum and its industrial applications.

DIFFERENT MEASUREMENT METHODS FOR ε

Extensive methods[6,7,8] have been reported in the literature for the measurement of ε of materials. A dielectric probe kit is available for the network analyser[9], whereby the probe is placed against the material under test and ε is calculated from the reflection coefficient. Sequeria[10] employs a one-port network analyser reflection measurement on multiple length samples to determine both ε and μ. Pham[11] developed an in-situ monopole antenna probe and a numerical technique which extends the measurement frequency range for ε. Ness[6,12] used two-port transmission measurements made by a semi-automatic network analyser via co-axial line and waveguide and this is the basis for the measurement system used in this paper. A waveguide transmission technique is a convenient laboratory based method which employs a network analyser to obtain ε for a length of sample filled waveguide. ε is calculated off-line from transmission coefficient S_{21} data from the network analyser. A schematic diagram of the measurement set-up and the sample filled test cell is shown in figure 1. From this measurement the sample dielectric constant, dielectric loss and loss tangent were calculated.

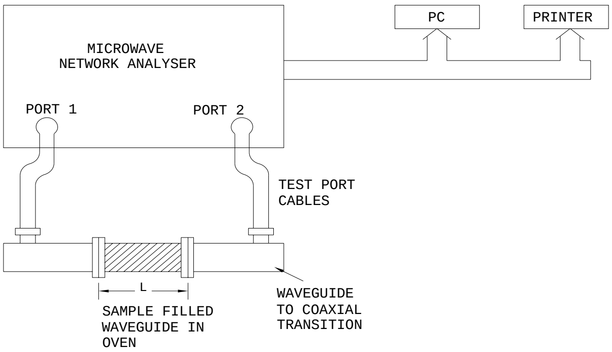

Figure 1. : Equipment Set-up for Waveguide Transmission Technique.

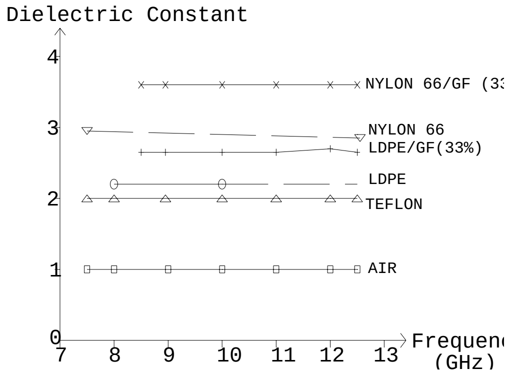

Figure 2. : Dielectric Constants of Air, Teflon, LDPE, LDPE/GF(33%), Nylon 66/GF(33%), Nylon 66 at Room Temperature over Certain Frequencies.

THEORY OF WAVEGUIDE TRANSMISSION TECHNIQUE

The idea of calibration was to get the values of reflected and transmission coefficients with air filled waveguide as standard and then compare the values of those with sample filled ones. During measurements, fixed lengths of waveguide were filled with the FRTP and a range of waveguide sizes was required to cover an almost continuos spectrum of frequencies from 2.2 - 12.5 GHz. The transmission coefficient between the reference planes is[7] :

$$S_{21} = \frac{4\Gamma_1\Gamma_0}{(\Gamma_1+\Gamma_0)^2\exp(\Gamma_1 L) - (\Gamma_1-\Gamma_0)^2\exp(-\Gamma_1 L)} \qquad (1)$$

where $\Gamma_0 = j\beta_0$, the propagation coefficient in air filled waveguide

$\Gamma_1 = \alpha_1 + j\beta_1$, the propagation coefficient in sample filled waveguide

L is the sample length in mm.

The relative permittivity of the FRTP[7] is :

$$\varepsilon = \varepsilon' - j\varepsilon'' = \varepsilon'(1 - j\tan\delta) \qquad (2)$$

For non-magnetic materials the elements of the complex permittivity may be obtained from the propagation coefficient as follows[7] :

$$\varepsilon = [1 + (\beta_1^2 - \alpha_1^2)(a/\pi)^2](c/2af)^2 \quad (3)$$

$$\text{and } \tan\delta = \alpha_1\beta_1 c^2/(2\pi^2 f^2 \varepsilon') \quad (4)$$

where 'a' is the broad dimension of the waveguide and 'c' is the velocity of electromagnetic waves. Since S_{21} is known from measurements, in principle, (1) can be solved for Γ_1, from which the complex permittivity of the sample may be calculated. Ness[6], Sabburg[7] and Ball[13] detailed an iterative technique for getting very accurate estimates of ε'. In this study, however, Newton's Method which is fast-converging and very robust was used. It requires an initial estimate of ε' and ε'' at a particular frequency. Because there are an infinite number of possible solutions, an accurate initial estimate of ε' and ε'' is important, as it enables the iteration process to converge promptly to the required result. Occasionally, the software failed to estimate the initial value of ε' and ε'' and the programme did not converge. In this situation, a manual estimate would be required and the software would then calculate the values of ε' and ε'' at a range of frequencies dedicated to that particular type of waveguide.

WAVEGUIDE SIZES AND SAMPLE LENGTHS

In order to cover the frequency range of 2.2 to 12.5 GHz, four different sizes of waveguides were used, namely : WR90, WR159, WR229 and WR340. Taking waveguide WR340 as an example, the recommended range of frequencies[14] for single (TE_{10}) mode only is from 2.2 to 3.3 GHz. If the loss tangent is not excessive then the wavelength in the sample depends only on the sample relative permittivity or dielectric constant, ε', as[15] :

$$\lambda_g \approx \frac{\lambda_o}{\varepsilon'^{1/2}} \cdot \frac{1}{[1 - (\lambda_o/2a\varepsilon_r^{1/2})^2]^{1/2}} \quad (5)$$

where λ_o is the free space wavelength
and 'a' is the broad side waveguide dimension. Assuming the largest possible frequency, ie frequency = 3.3 GHz, λ_o = 30/3.3 = 9.091 cm and a = 3.4 x 2.54 cm = 8.636 cm. Assuming that the dielectric constant of the sample would lie in the range of 2 to 5 and say, ε' = 2 then (5) yields λ_g = 6.926 cm. For each waveguide size, it was necessary for the samples to be at least three quarters of a guide wavelength long at the highest frequency. This allows the software to obtain sufficient S_{21} phase data to provide an initial estimate of permittivity. The required sample length cannot be determined in advance so it is necessary to make worst case assumptions. The sample length required to determine various dielectric constants are as shown in Table 1.

WR 340		WR 229		WR159		WR 90	
Fre range : 2.2 - 3.3 GHz		3.3 - 4.9 GHz		4.9 - 7.05 GHz		8.2 - 12.4 GHz	
Permittivity	Sample Length	Permittivity	Sample Length	Permittivity	Sample Length	Permittivity	Sample Length
2	52	2	35	2	24.5	2	14
3	41.5	3	28	3	19.5	3	11
4	35.5	4	25	4	17.2	4	9.4
5	31.5	5	22	5	15.1	5	8.5

Table 1. : Waveguide Type, Frequency Range and Sample Length

RESULTS

In order to verify the accuracy of the method, we first measure of materials having known dielectric constants. Air and teflon were chosen for this purpose and their dielectric constants were measured at room temperature. Figure 2 shows that dielectric constants for air and teflon are 1 and 2 respectively at room temperature which coincides very well with data in the literature. This figure also illustrates the dielectric constants of polystyrene (PS) , low density polyethylene (LDPE), glass fibre-reinforced LDPE and carbon fibre-reinforced PS. Figures 3 and 4 show that the higher the temperatures the lower the values for the dielectric constant of LDPE/GF(33%). The dielectric loss of this material is quite low, of the order of 0.02 at 8 Ghz. It has not yet been possible to determine the variation of this over the 8 - 12 GHz band with sufficient accuracy because the same loss is of the order as waveguide wall loss and unpredictable coupling flange losses. However it does appear to increase with frequency and temperature , and is of the order of 0,1 at 12 GHz.

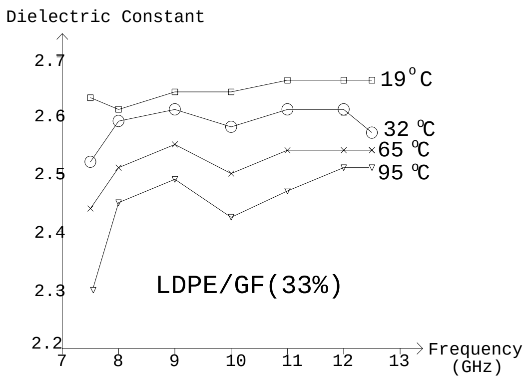

Figure 3. : Dielectric Constants of LDPE/GF(33%) at Elevated Temperatures over Certain Frequencies.

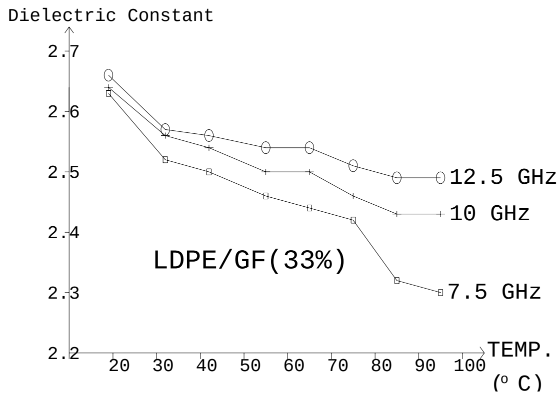

Figure 4. : Dielectric Constants of LDPE/GF(33%) at Different Frequencies over a Range of Temperatures.

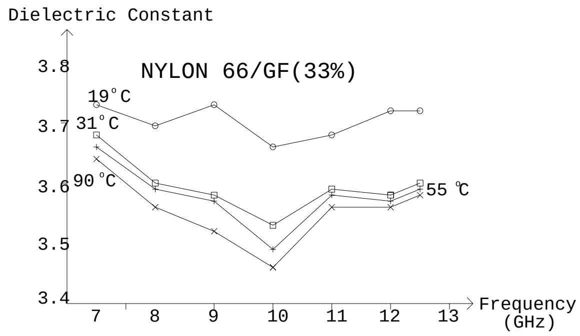

Figure 5. : Dielectric Constants of Nylon 66/GF(33%) at Elevated Temperatures over Certain Frequencies.

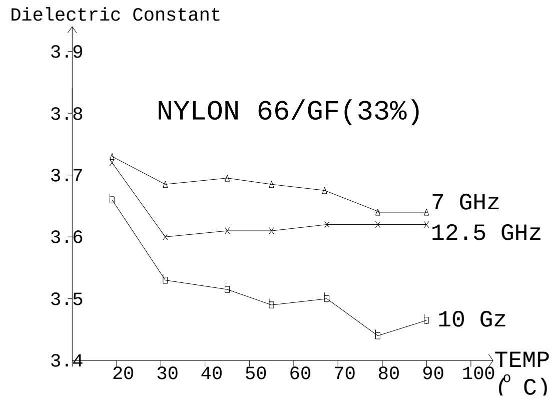

Figure 6. : Dielectric Constants of Nylon 66/ GF(33%) at Different Frequencies over a Range of Temperatures.

With regard to the glass fibre (33%) reinforced nylon 66, the higher the temperature, the lower the values of the dielectric constant as depicted in figures 5 and 6. The trends of the values of the dielectric constants for both materials are the same and they match with the trends for most materials including water[8]. Figures 7 and 8 illustrate the change of loss tangent values of nylon 66/GF(33%) with frequencies and temperatures respectively. The trend shows that the higher the temperatures, the higher the values of the dielectric loss factors but there is no such a trend with frequencies. Referring to figure 8, at a frequency of 7 GHz and a temperature of 90 degrees Celcius, the dielectric loss factor is at a maximum; at this point, a maximum amount of microwave energy which penetrates the sample will be converted to heat to facilitate the joining process but on the other hand, the dielectric constant at this condition is quite high and microwave energy penetration will not be optimum. A compromise for the values of tan δ and ε′ should therefore be chosen to obtain greatest microwave energy penetration and maximum energy conversion to heat. Similar arguments can be made for the LDPE/GF(33%) and welding the material by microwave energy at 12.5 GHz (see figures 3 and 4) and 90 degrees Celsius will not give the optimum result because although tan δ is maximum at this frequency, ε′ is not favourable for the penetration of microwave energy.

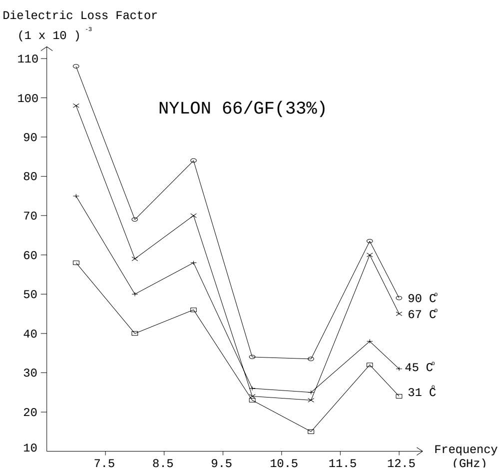

Figure 7. : Dielectric Loss Factors of Nylon 66/ GF(33%) at Elevated Temperatures over Certain Frequencies.

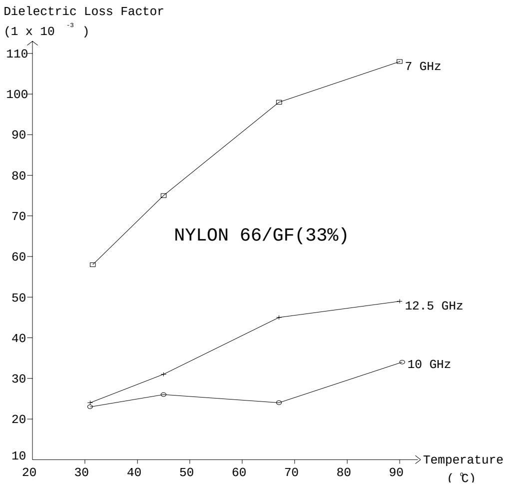

Figure 8. : Dielectric Loss Factors of Nylon 66/ GF(33%) at Different Frequencies over a Range of Temperatures.

CONCLUSION

Information on the microwave dielectric properties of fibre-reinforced thermoplastics is required in order to determine whether they are suitable for microwave processing. Some preliminary data has been presented for 33% glass fibre reinforced LDPE and nylon 66. Those values give a clue on how to choose suitable combinations of parameters for joining glass fibre or carbon fibre thermoplastic materials using microwave energy. The potential benefits of the technology will be to speed up the replacement of thermosetting resins by advanced thermoplastic composites in the structural parts of aeronautical, military and recreational industries.

ACKNOWLEDGEMENT

The authors would like to thank Mr. Chan Ka-hau, Peter for his assistance in collecting some of the data.

REFERENCES

1. Osswald, T.A. and Menges, G, Materials Science of Polymers for Engineers, Hanser/Gardner Publications Inc., 1995, pp. 393-5.

2. Lynch, C.T. (Ed.), CRC Handbook of Materials Science, Volume III, Non-metallic Materials and Applications, CRC Press Inc., 1975, pp.118 - 120.

3. Shackelford, J.F. (Ed.), The CRC Materials Science and Engineering Handbook, CRC Press, 1992, pp. 731 - 9.

4. Ku, S H, Siores E, and Ball J A R (1997) Weldability and Heat Affected Zone (HAZ) Evaluation for High Energy Rate Joining of Thermoplastic Composites Using Microwave, Proceedings of the Eleventh International Conference on Composite Materials, Vol 6, pp 55-64, Gold Coast Australia, 14-18 July.
5. Ku, S H, Siores E, and Ball J A R (1997) Welding of Thermoplastic Composites Using Microwave Energy, Proceedings of CIPR International Symposium, Vol 2, pp 612-9, Hong Kong, 21-22 August.

6. Ness, J., Broadband Permittivity Measurements at Microwave Frequencies, IREE Conf. Dig., (Australia), September 1983, pp. 330 - 2.

7. Sabburg, J, Ball, J.A.R. and J.B. Ness, Broadband Permittivity Measurements of Wet Soils, Proceedings of 1992 Asia-Pacific Microwave Conference, Adelaide, pp.607 - 10.

8. Senko, H., Investigation Into and Control of Microwave Induced Thermal Run-away, PhD Thesis, Deakin University, Australia, Feb 1997.

9. HP85070A Dielectric Probe Kit, 200 MHz to 20 GHz, Hewlett Packard Product 85070A Information, August 1990.

10. Sequeira, H.B., Extracting ε and μ of Materials from Vector Reflection Measurements, Microwave Journal Vol. 34 No. 3, March 1991, pp. 73 - 85.

11. Pham, A.T., In-situ Materials Permittivity Measurement Using a Monopole Antenna, Proc. IREE Conv., (Australia), Sept. 1991, pp. 658 - 61.

12. Ness, J., Broadband Permittivity Measurements Using the Semi-automatic Network Analyser, IEEE Trans. Microwave Theory Tech., Vol. MTT-33, No.11, November 1985, pp.1222 - 6.

13. Ball, JAR, Horsefield, B and et al, Cheese Curd Permittivity and Moisture Measurement Using A 6-Port Reflectometer, Proceedings of 1997 Asia-Pacific Microwave Conference, Dehli, India.

14. HP Coaxial and Waveguide Catalogue and Microwave Measurement Handbook, 1993.

15. Kraus, J.D., Electromagnetics, 4th ed., McGraw-Hill, 1992, pp.655-60.