MATERIALS CHARACTERIZATION BY ULTRASONIC ATTENUATION

SPECTRAL ANALYSIS

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INTRODUCTION

The use of ultrasonic techniques for the characterization of microstructural features has attracted much attention in recent years. Although the measurement of ultrasonic parameters has been used for determining material properties for many years, but with the advent of modern signal processing techniques it is possible to extract significantly more information from ultrasonic signals. This has lead to numerous studies being carried out on the effect of microstructures on the propagation of ultrasonic waves and their application to monitoring properties of industrial significance. Examples include the determination of grain size, degree of porosity, amount of second phase particles and measurement of texture and residual stresses. Simultaneously the theoretical models describing the interaction of an ultrasonic wave with these features have been refined.

One of the approaches we have adopted at Harwell is that of studying the broad band ultrasonic attenuation characteristics of materials. This work was originally prompted by the results published by Vary [1] which related an ultrasonic attenuation parameter with the fracture toughness of steels. This would be a significant contribution to the application of fracture mechanics to structural integrity analysis if such a general correlation could be verified. However our work differed in approach in that we attempted to relate the ultrasonic measurements to the materials microstructure and mechanical properties from a fundamental rather than empirical point of view. It should be noted that this approach requires a close collaboration between the ultrasonics specialist, both experimental and theoretical, and the metallurgist or materials scientist. In this paper a review of the experimental and theoretical work carried out at Harwell will be given which and how it relates to the work of others. Some recent results on materials of technological importance will also be presented.

BACKGROUND

The many possible interactions by which an ultrasonic wave loses energy as it propagates through a material have been discussed in detail many times [for example see Refs 2 and 3]. The mechanisms for the loss of energy may be broadly catagorized as those due to absorption processes and those due to scattering processes. Hence the attenuation may be written as

$$\alpha(f) = \alpha_a(f) + \alpha_s(f) \quad (1)$$

where $\alpha_a(f)$ is the absorption contribution and $\alpha_s(f)$ the scattering contribution to the total attenuation, f is the frequency as in general these coefficients are frequency dependent. Examples of absorption processes are dislocation damping, magnetic domain damping and thermoelastic interactions. Scattering can occur from features such as grain boundaries, voids, inclusions, second phase particles and micro and macro cracks. These are considered to be the principle mechanisms operating in the MHz range of frequencies, say 1-100 MHz, where most ultrasonic work is carried out. At very high or low frequencies or very low temperatures other interactions occur, such as phonon-electron scattering, but they may be ignored in most practical applications.

In much of the early work using ultrasonic attenuation measurements for grain size determination the absorption contribution was also ignored and the data interpretted in terms of scattering from the grains only. Whereas the theory for scattering of ultrasonic waves in polycrystals has received a great deal of detailed investigation, the theory for the absorption processes has been somewhat neglected. This is probably due to the lack of experimental data in the MHz frequency range compared to the extensive work carried out at lower frequencies (internal friction measurements). However, if absorption processes make a significant contribution in the frequency range of interest then it becomes vital that the effects of absorption and scattering can be separated if ultrasonic attenuation measurements are to be used for materials characterization.

PROCEDURE AND RESULTS

The measurement procedure adopted was that described by Papadakis [3] in which a broad band buffered ultrasonic transducer is used to generate and receive the ultrasonic pulses. Two backwall echoes and also the buffer-specimen interface echo are collected and analysed. Corrections are made for both diffraction losses and the frequency dependent reflection coefficient of the interface layer. The present analysis system is based on a Panametrics PR5052 pulser-receiver using 20, 50 and 100 MHz transducers and a Tektronix WP1310 signal processing system with a ROM based Fast Fourier Transform. A complete attenuation spectra measurement at a single point on a specimen can be recorded, analysed and displayed in about 2 minutes.

The importance of relating the ultrasonic measurements with the metallurgical state of the sample was noted above and for these reasons much of our early work was carried out on specially prepared samples of Fe-C alloys. The results of this work have been reported previously [4,5,6,7]. The primary metallurgical variables investigated were grain size and carbon content [4,5,6] with later work investigating the morphological states of these alloys [7]. Figure 1 illustrates the effect of varying the amount of carbon in the Fe-C alloy while the grain size remains constant. The results on the grain size experiments could be interpreted in terms of the accepted concepts of Rayleigh, Stochastic and Diffusion scattering provided that the grain size distribution was taken into account [8]. These results may be summarized as follows:

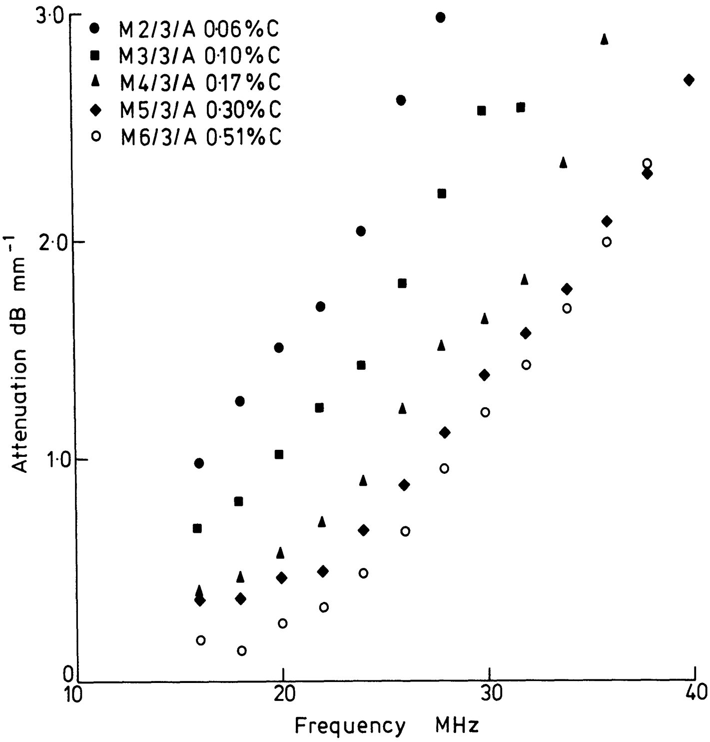

Figure 1 - The Frequency Dependence of the Ultrasonic Attenuation in the Fe-C Alloys

1. Scattering from the grains may be interpreted in terms of the accepted scattering regimes provided the grain size distribution is taken into account. Even if all the grains meet the Rayleigh scattering criteria ($\lambda \gg d$, where λ is the ultrasonic wavelength and d is the (maximum) grain size) it is the largest grains that dominate the attenuation characteristics. The grain size factor in the Rayleigh scattering term is $\langle D^6 \rangle / \langle D^3 \rangle$ where $\langle \rangle$ is the average.

2. Increasing the carbon content of these alloys decreases the ultrasonic attenuation (Figure 1) if the grain size remains constant. It was proposed that this affect was related to the absorption process via the dislocation damping and dislocation pinning.

3. There was no unique relationship between the ultrasonic attenuation measurements and the mechanical properties of the material. In particular an increase in grain size causes an increase in ultrasonic attenuation and makes the material more brittle whereas an increase in carbon content causes a decrease in ultrasonic attenuation but also makes the material more brittle.

From these results it is apparent that even in a relatively simple metallurgical system careful analysis of the ultrasonic measurements is required to relate them to the microstructural feature of interest. More recent work carried out on pure aluminium [9] has demonstrated the role of dislocations in ultrasonic absorption and in this particular material, because of the weak grain scattering, it completely dominates the ultrasonic attenuation characteristics. Figure 2 illustrates the results of a strain-anneal experiment carried out on 5N purity aluminium. The data are presented in the form to be described in the next section but the essential features are obvious.

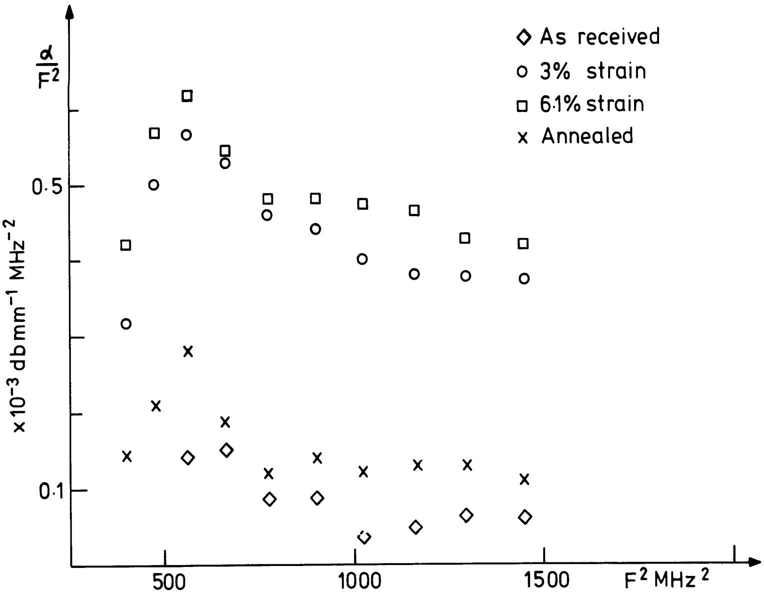

Figure 2 - Attenuation Spectra in Pure Aluminium

During the deformation process the increase in dislocation content causes a significant increase in the level of attenuation (with a related increase in hardness). A subsequent low temperature anneal returns the attenuation to almost its initial value. The exact mechanism for the recovery process is not clear but may be due to the activation of point defects causing pinning of the dislocations. These results may be of relevance to ultrasonic monitoring of early fatigue damage although unpublished work at Harwell has shown the effect to be very small in aluminium alloys. This is consistent with the processes involving the use of alloying elements to strengthen aluminium.

The results described above have concentrated on grain scattering and absorption related to dislocations but other effects were also observed. In particular in the very low carbon Fe-C alloys a significant amount of absorption due to magnetic damping was observed but not quantified. Direct measurements of ultrasonic absorption in steels [10] have shown that both dislocation and magnetic damping can be present. The magnetic contribution may be removed by applying a large magnetic field to the specimen. Similarly results of attenuation measurements on multi-phase materials can be complex to analyse. The following section describes the theoretical models we have developed for the analysis of ultrasonic attenuation data.

THEORETICAL ANALYSIS

There are two approaches to the theoretical analysis of ultrasonic attenuation characteristics. One which enables ultrasonic data to be interpreted in terms of the material variables of interest and the other which allows the ultrasonic characteristics to be predicted if the microstructural variables are known. These may not be mutually exclusive and examples of both approaches are presented here.

Ultrasonic Attenuation Spectral Analysis

Reynolds and Smith [11] have developed a method for analysing attenuation data based on the accepted treatments of grain scattering valid in the Rayleigh, Stochastic and Diffusion regions. The method allows for the effects of grain size distributions as well as a contribution from the absorption processes. The basic principles of this analysis are shown in Figure 3. At low frequencies, the scattering is proportional to the fourth power of the frequency, at higher frequencies it varies as the square of the frequency and at the highest frequencies it becomes independent of frequency. The frequencies at which each type of scattering becomes dominant depends upon the wavelength to grain size ratio but in the general case of a grain size distribution there can be all three types of scattering occurring at an individual frequency. However there will be some frequency at which all the grains will scatter in a Rayleigh fourth power mode. To account for the absorption processes Reynolds and Smith used the basis of the Granato and Luke vibrating string model of dislocation damping [see Ref 12 for a detailed description]. This theory predicts that away from the resonance condition the absorption is preportional to the square of the frequency. The advantages of analysing the data in the form given in Figure 3, viz α/f^2 vs f^2, are therefore apparent. The absorption term becomes a constant term over the whole frequency range and the Rayleigh region a linear portion whose slope is proportional to the $\langle D^6\rangle/\langle D^3\rangle$ moments of the grain size distribution. In the case of a narrow distribution $\langle D^6\rangle/\langle D^3\rangle$ becomes the mean grain volume.

Other attempts have been made to account for absorption losses using a function linear in frequency. In particular in the use of backscattered signals for grain size determination [13] a linear function combined with the Rayleigh function is used. In [14] the concept of an 'effective grain size' is introduced for steels but would also apply to other complex microstructures. This recognizes the fact that the dominant features of the microstructure may not be the same optically as they are ultrasonically. Figure 4 illustrates this for measurements on steels with a martensitic structure. Here it can be seen that the scattering is in the Rayleigh region and that the slope of the A508 type steel is approximately twice that of the A533B steel. This would appear to correlate with the prior austenite grain size of these materials rather than the lathe or lathe packet size. Vary [15] has used a correlation technique to determine the dominant scattering features in a complex titanium alloy. In such complex microstructures it should be possible, if all the elastic constants and orientations are known, to predict the strongest scatters. The extreme example would be voids in a polycrystal where the void would scatter strongly because of the large elastic mismatch but may or may not be the dominant scatterer depending on the volume ratio between them and the grains [16].

Scattering Models

The ability to predict the influence of a particular microstructural feature on the total attenuation due to scattering enables quite complex situations to be analysed. A number of models have been applied and developed at Harwell to aid the understanding of scattering in complex materials. One model [8], mentioned above, uses a simple hard sphere scattering theory to look at the influence of grain size distributions. Most importantly it should be noted that there is no unique solution to the interpretation of an ultrasonic attenuation-frequency curve in terms of grain size. The best that can be achieved is to obtain most probable fits of the data using multi-variable curve fitting techniques. Such techniques have been applied recently to the problem of determining texture pole figures from ultrasonic velocity measurements [17].

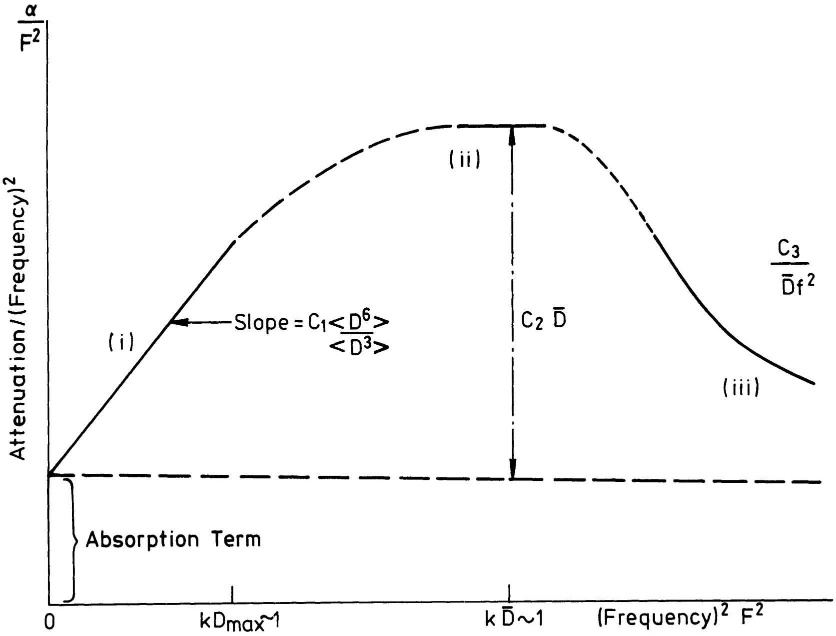

Figure 3 - Ultrasonic Attenution Spectral Analysis

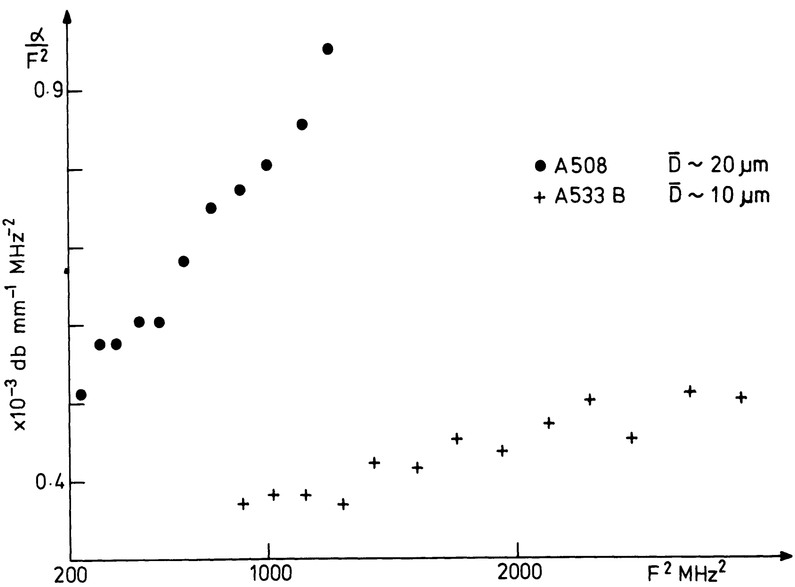

Figure 4 - Attenuation Spectra of A508 and A533B Steels

More complex work has concentrated on a detailed analysis of the scattering occurring in porous, polycrystalline and two phase materials. For discrete scatterers in a homogenous matrix, the theory of Ying and Truell [18] describes the attenuation of longitudinal waves. An examples of the application of this theory is to the experimental data of Kinra et al [19] for a system of lead inclusions in an epoxy matrix. We have applied the theory to grain scattering but it has some restrictions when compared to the simple hard sphered model in dealing with grain size distributions. More detailed models have recently been proposed elsewhere[20,21]. A further problem that has been recently evaluated is that of scattering by a second phase formed by segregation at a grain boundary [22]. This model has been applied to a WC-Co system for which experimental data was also available. Significant discrepancies were found between the theory and experiment which have initially been ascribed to the incomplete knowledge of the single crystal elastic constants of the two phase materials [23].

Applications to Engineering Materials

With the background described above available the application of ultrasonic attenuation spectral analysis to a number of materials of practical interest has been carried out [24]. These include steels, nickel alloys, titanium alloys, cemented carbides and cast irons. In each case significant information can be obtained on some aspects of the structure of the material. For example Figure 5 illustrates measurements made on a sample of Inco 718 Ni-Cr-Fe Alloy forging which can show a phenomena of large grain size variations near the surface. It can be seen that the attenuation spectral analysis clearly indicates a change from Rayleigh (positive slope) to Diffusion (negative slope) scattering over the frequency range of the measurements. Such a change of slope would be a particularly easy parameter to measure on-line.

The technique has also recently been applied to non-metallic systems, plastics and fibre reinforced plastics [25] and it is proposed to use the same technique for the characterization of engineering ceramics.

CONCLUSIONS

The importance of an understanding of the scattering and absorption processes occurring in polycrystalline materials has been discussed when using ultrasonics for materials characterization problems. The work carried out at the National NDT Centre, Harwell to develop this understanding has been described along with the techniques used for data analysis and theoretical modelling. The rapid measurement of ultrasonic attenuation over a wide range of frequencies offers a promising approach

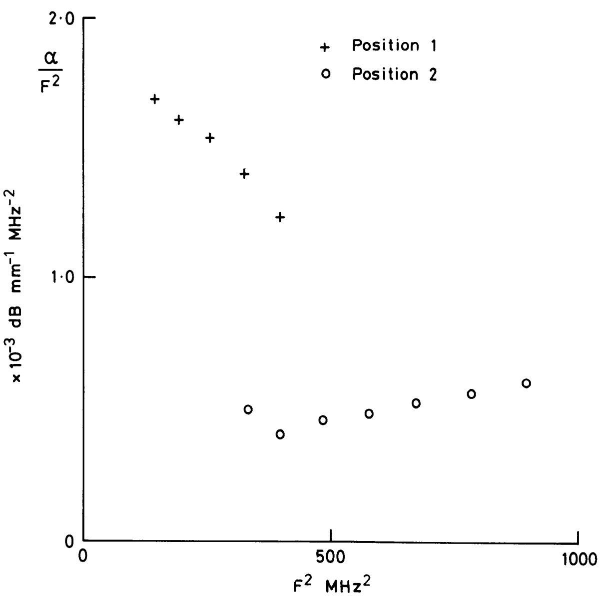

Figure 5 - Attenuation Spectra of a Nickel Alloy Forging

to a number of problems of materials evaluation. The general behaviour of polycrystalline metals follow a clearly indentifiable pattern. It is evident that dislocations play a far larger part in the attenuation of ultrasound in metals than has usually been assumed. However there are significant limitations to the properties that may be determined. Hence it is important when applying such techniques to materials characterization that the problem must be clearly defined in terms of metallurgical microstructure and how it relates to the ultrasonic measurement.

ACKNOWLEDGEMENTS

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REFERENCES

1. A. Vary, Ultrasonic Measurement of Material Properties, in: "Research Techniques in Nondestructive Testing, Vol 4", R.S. Sharpe, ed., Academic Press, New York (1980).
2. R. Truell, C. Elbaum and B.B. Chick "Ultrasonic Methods in Solid State Physics" Academic Press, New York (1969).
3. E.P. Papadakis, Scattering in Polycrystalline Media, in: "Methods of Experimental Physics, Ultrasonics Vol 19" P.D. Edmonds, ed., Academic Press, New York (1981).
4. R.L. Smith, W.N. Reynolds and H.N.G. Wadley, Metal Science 15 554 (1981).
5. R.L. Smith and W.N. Reynolds, J Mat Sci 17 1420 (1982).
6. R.L. Smith, K.L. Rusbridge, W.N. Reynolds and B. Hudson, Mat Eval 41 219 (1983).
7. R.L. Smith and S. Perring, AERE-R10956 (1983).
8. R.L. Smith, Ultrasonics 211 (1982).
9. S. Perring, R.L. Smith, B. Hudson and W.N. Reynolds, Phil Mag A 52 721 (1985).
10. J.P. Monchalin and J.F. Bussiere, Proceedings of Symposium on Non-destructive Methods for Material Property Determination, Plenum Press, New York (1983).
11. W.N. Reynolds and R.L. Smith, J Phys D: Appl Phys 17 109 (1984).
12. A.B. Bhatia, "Ultrasonic Absorption" Oxford (1967).
13. K. Goebbels, Structure Analysis by Scattered Ultrasonic Radiation, in: "Research Techniques in Nondestructive Testing, Vol 4", R.S. Sharpe, ed., Academic Press, New York (1980).
14. H. Takafuji, S. Sekiguchi, T. Iuchi and S. Matsuda, Proceedings of Ultrasonics International 85, 195 Butterworth (1985).
15. A Vary and D R Hall, Mat Eval 41 309 (1982).
16. R.B. Thompson, W.A. Spitzig and T.A. Grey, Review of Progress in Quantitative Nondestructive Evaluation 5, 1643 Plenum Press New York (1985).
17. J. Lewandowski, Ultrasonics 24 73 (1986).
18. C.F. Ying and R. Truell, J Appl Phys 27, 1086 (1956).
19. C.M. Sayers and R.L. Smith, J Phys D: Appl Phys 16 1189 (1983).
20. F.E. Stanke and G.S. Kino, J Acoust Soc Am 75, 665 (1984).
21. S. Hirsekorn, J Acoust Soc Am 79 1269 (1986).
22. C.M. Sayers, Wave Motion 7 95 (1985).
23. C.M. Sayers and R.L. Smith, Proceedings of Ultrasonics International 85, 195, Butterworth 1985.
24. R.L. Smith, AERE-R11885 (1985).
25. W.N. Reynolds, L.P. Schudder and H. Pressman, Polymer Testing 6 325 (1986).

DISCUSSION

Dr. W. Sachse, Cornell: On the attenuation curves that you showed for aluminum as a function of deformation and annealing, how much time was there between the deformation and when you made the measurements? Because the aluminum is known to have terrible time-dependence effects.

Dr. Smith: That one was done as quickly as we could. We deformed it, took it to the measuring system and measured it straightaway.

If I may expand on that a little, there is a potential, I think, for a new measurement system here, because if you want to know the activation energies for the recovery process we can monitor the attenuation as a function of time and temperature.

We have done two experiments, one where we deformed the aluminum and determined the annealing temperature required to return the attenuation to its original level. We also measured the recovery rate at room temperature and by equating those measurements to the time-temperature constant for the process we determined the activation energy. It was about an order of magnitude one but it shows the potential.