ULTRASONIC EXAMINATION OF TI-6-4 AND NITRIDED TI-6-4 MATERIALS

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INTRODUCTION

The work reported herein is a continuation of studies into the detection and characterization of grain boundaries which have become enriched by the accumulation of undesirable elements or second-phase precipitates. These conditions have been associated with embrittlement in several alloys, and may radically alter engineering properties of the bulk material. Grain boundaries can become enriched, or decorated, during heat treatments, including welding, associated with fabrication or in-service repair. Additionally, embrittlement can occur during the service life of components exposed to intense radiation.

Material samples showing structure similar to the grain boundary enrichment being studied were recently produced by compacting Ti-6-4 microspheres with a hot isostatic press (HIP) process [1]. Microspheres, free of surface contamination, were compacted into samples showing ordinary Ti-6-4 microstructure, which involves somewhat randomly oriented alpha phase crystals, along with small amounts of the beta and acicular alpha phases. We treated scattering from this material with a theory developed for scattering in multiphase polycrystalline materials; scattering is produced by both the inherent anisotropy of single crystals of each species, and by the difference between each species and the macroscopic average properties of the material [2,3]. Other microspheres were exposed to nitrogen at elevated temperatures, and developed an outer layer of titanium nitride, which is denser, and has higher wave speeds than the core of Ti-6-4 [4]. When these microspheres were compacted into solid specimens, the nitrided material remained in place, forming regions analogous to a decorated grain boundary. We treated the scattering problem for nitrided microspheres as if each were a single isotropic grain with a shell of second-phase material [5].

Theoretical and experimental work considered isolated microspheres of both kinds, and consolidated materials formed from microspheres of both kinds. The two analyses are tied together by the Independent Scatterer Model [6], which predicts rms grain noise by summing the energy scattered by each scatterer in an aggregate, rather than forming the coherent sum of scattered displacement fields.

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THEORY OF SCATTERING FROM ISOLATED SPHERICAL SCATTERERS

The classical solution for scattering of a plane elastic wave from an isotropic spherical scatterer embedded in an isotropic host was given by Ying and Truell [7]. For incident longitudinal waves, the problem is symmetric about the direction of propagation of the incident wave (hence no dependence on the azimuthal angle), and separates in the remaining two variables (radius and polar angle). The equation in the polar angle turns out to be independent of material properties, and because of this, the process of matching stresses and displacements at spherical boundaries involves only the equation in radial position. This property of the Ying and Truell solution has been exploited to solve for shelled spherical scatterers, and has been extended to the case of spherically orthotropic shell materials [5]. In the present case, we have modelled isolated Ti-6-4 microspheres embedded in metallographic mounting plastic as isotropic spherical scatterers in an isotropic host, and nitrided Ti-6-4 microspheres in the same plastic as isotropic spheres with an isotropic shell.

The Ying and Truell solution (extended to the case of shelled spherical scatterers) was used to predict the scattering amplitude, $A(\omega)$, for scatterers having dimensions and physical properties similar to the experimental set of microspheres. For representative cases, a time domain response was predicted by taking the inverse Fourier transform of scattering amplitude (which gives an impulse response for the scatterer), and convolving this with a reference interface echo [8]. In these simulations, the reference echo was captured with a 2 nanosecond sampling interval; to reconstruct the time domain impulse response with the same sampling interval it was necessary to calculate scattering amplitude out to $(ka)_{max} = (\pi a) / (\tau C_L)$, where a is the scatterer's outer radius (mm), τ is the sampling interval (microsec), and C_L is the longitudinal wave speed in the host (mm/microsec).

Numeric difficulties associated with the separation of variables solution [9] were handled by slightly perturbing material parameters at points where the original parameters gave rise to numeric instability. This problem, which is more evident with thin-shelled scatterers than with solid scatterers, has not yet been resolved in a rigorous fashion. It occurred at isolated values of ka above about 30, when the condition number associated with matrix operations required by the Ying and Truell method of solution indicated a loss of all available precision. To put this problem in context, wavenumbers calculated at the transducer's nominal center frequency were 37 for the average Ti-6-4 microsphere, and 29 for the average nitrided microsphere.

Time domain reconstructions show a direct reflection and a second peak which may be related to propagation of a surface-type wave. It will later be shown that excellent agreement between theory and experiment exists for the time delay between these two waveform features. The amplitude of the second peak, however, is weaker in practice than would be predicted by the theory used here; possible explanations for this discrepancy include the use of a focused transducer (theoretical model presumes incident plane longitudinal waves), and an incomplete model for the plastic-to-metal interfacial bond (the theoretical solution assumes perfect bonding of perfectly spherical surfaces).

MEASUREMENTS OF SCATTERING FROM ISOLATED SCATTERERS

Microspheres of Ti-6-4 and nitrided Ti-6-4 were cast in Buehler Transoptic plastic, a transparent material commonly used for mounting metallurgical samples. The microspheres, whose diameters ranged from 0.165 mm to 0.523 mm, were arranged on a square grid with 0.1" (2.5 mm) spacing between rows and columns. The plane of this grid lay 0.1" (2.5 mm) below the plastic's surface. Each plastic sample prepared contained 117 microspheres, although optical inspection of the sites revealed that some contained contamination, and some particles were noticeably prolate. The majority of sites, however, contained one very spherical particle. The diameters of these particles was measured optically, using a Mitutoyo Toolmaker's Microscope with accuracy estimated at ±0.003 mm.

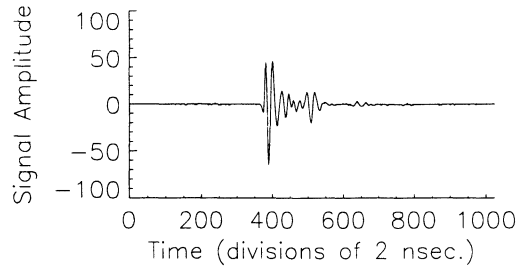

Figure 1. Experimental echo from a 0.292 mm nitrided microsphere.

Ultrasonic inspection of the isolated microspheres was performed with several focused transducers, the most satisfactory of which was a Panametrics V3384 50 MHz transducer with a 0.5" (12.7 mm) focal length in water. At each inspection site, the transducer was carefully centered over the microsphere by peaking up the echo, with the height of the transducer adjusted to ensure the particle was in the focal zone of the transducer. The resulting echo was then averaged 16 times by the digitizing oscilloscope (Tektronix 7603 scope with 7D20 Digitizer), and captured as a 1024 point waveform with a 2 nanosecond sampling interval. A representative experimental waveform is shown in Figure 1.

For each of the microspheres, the time delay between the first and second major negative peaks of the experimentally obtained signal was measured, and correlated with the microsphere's diameter, measured optically. Figure 2 shows this data for the two sets of microspheres, and emphasizes the very small difference in properties, when set in a host that is quite different from the microspheres. Nonetheless, the average ratio of diameter to time delay was slightly different for the two types of particles, and this difference was also predicted from time domain reconstructions.

Table I gives material properties used in calculating the theoretical responses for Ti-6-4 and nitrided Ti-6-4 microspheres, and Table II presents theoretical and experimental results for 0.292 mm diameter microspheres embedded in Perspex. Calculations for the nitrided microsphere assumed a 0.013 mm thick titanium nitride shell, which is consistent with measurements made on SEM images of consolidated powder metallurgy samples.

The generally good agreement between theory and experiment suggests that the models of Ti-6-4 and nitrided Ti-6-4 microspheres as isotropic and shelled isotropic scatterers, respectively, are reasonable, and that the material properties we used are representative. The importance of these conclusions is that the independent scatterer model requires some knowledge of scattering amplitude, which in the case of the nitrided microspheres, is derived from the extended Ying and Truell solution for layered spherical scatterers.

Table I. Material Properties Used for Time Domain Reconstructions.

Material	Density (gm/cm^3)	C_L (mm/microsec)	C_T (mm/microsec)
Perspex	1.18	2.73	1.43
Ti-6-4	4.49	6.191	3.186
Nitrided Ti-6-4	4.621	7.902	4.350

Table II. Comparison of values for the time delay between waveform peaks.

Microsphere Material	Theoretical Diameter/Delay (mm/microsec)	Experimental Diameter/Delay (mm/microsec) (Average±Std. Deviation)
Ti-6-4	1.207	1.217±.041
Nitrided Ti-6-4	1.264	1.256±.086

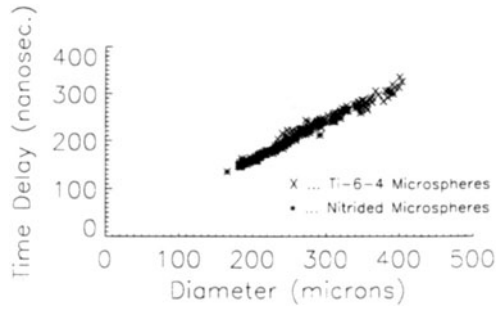

Figure 2. Time delay between waveform peaks versus microsphere diameter.

INDEPENDENT SCATTERER MODEL PREDICTIONS FOR CONSOLIDATED SAMPLES

The Independent Scatterer Model of Margetan et al [6] has been widely used to predict absolute grain noise in polycrystalline materials. In it's original form, a material dependent parameter called the "Figure of Merit" (FOM) was defined in such a way that it was independent of the measurement system, and a practical procedure for calculating the FOM was presented. This procedure, which involves the use of an easily obtained reference measurement, was later used as the basis for a predictive model [10] which, using the measured reference signal and calculated values for FOM as inputs, gives predictions for the rms grain noise voltage as a function of time. The general form for the FOM, in the case of discrete scatterers is:

$$FOM = \sqrt{n} \cdot |A(\omega)|$$

where n is the number of scatterers per unit volume. In the case of the nitrided Ti-6-4 microspheres, the scattering amplitude (in the backscattered direction) was directly available as a function of frequency from the analytical solution. It is important to note that scattered power (proportional to the square of rms voltage) adds for each scatterer, according to the Independent Scatterer Model; in the case of a distribution of scatterer sizes or compositions, we may think of a Figure of Merit for each scatterer, and add the squares of their FOM's. Using Margetan's model, a prediction for grain noise was made using the average size of a nitrided Ti-6-4 microsphere to calculate both the backscattered scattering amplitude and the number of scatterers per unit volume (i.e. the reciprocal of the average scatterer's volume).

For grain noise due to the random orientation of anisotropic crystals, Rose [2] calculated backscattered power in the Born approximation for weak scatterers and early time (before the incident field is significantly attenuated), using a probability distribution on grain size to characterize the point-to-point correlation of elastic constants. The resulting expression for power implied independent scatterers and gave an explicit expression for the Figure of Merit. Rose later extended this analysis to multiphase materials [3], again invoking assumptions appropriate to independent scatterers, and assuming that the crystallites of each phase had independent, random orientations. This latter analysis yielded the expression shown below:

$$FOM^2 = 8\pi \sum_i f_i (R_i + Q_i) \frac{k^4 a_i^3}{(1 + (2ka_i)^2)^2}$$

where the summation is over phases in the material, f_i is the volume fraction of the i-th phase, R_i accounts for phase contrast (density and wave speed differences between each phase and the macroscopic average material), Q_i accounts for the anisotropy of single crystals of the i-th phase, and a_i is a characteristic crystallite dimension for the i-th phase.

This expression was used to calculate the FOM for Ti-6-4, which has alpha, beta, and acicular alpha phases. Initial calculations used a characteristic dimension appropriate to the unconsolidated Ti-6-4 microspheres; this dimension, however, is not representative of the structures within each microsphere. Grain noise predictions under these conditions were smaller than observed. Later calculations explored the effect of using a smaller characteristic dimension, and found that grain noise increases with decreasing scatterer size, as is seen from the high frequency limit of Rose's expression.

MEASUREMENTS OF GRAIN NOISE FOR CONSOLIDATED SAMPLES

Ultrasonic scattering from consolidated samples was measured using focused, 50 MHz ultrasonic transducers (Panametrics models V390 and V3384), a wideband pulser/receiver (Panametrics UA5600), and a digitizing oscilloscope (Tektronix 7603 scope with 7D20 digitizer). The three samples that were used were consolidated from a) all Ti-6-4 microspheres, b) all nitrided Ti-6-4 microspheres, and c) a 50/50 mix of Ti-6-4 and nitrided Ti-6-4 microspheres. The samples were prepared by polishing one face to provide a smooth entry surface. Each data set consisted of 256 waveforms, each of which represents the average of 16 individual echoes (to reduce electrical noise). The transducer moved approximately 20 microns laterally between waveforms. Individual waveforms in each ensemble were aligned at the trailing edge of the interface echo to avoid the variance that arises from trigger jitter and similar temporal shifts [11].

At each time index, the average and variance of the signal were calculated. The average should contain only stationary contributions associated with the transducer (e.g. buffer rod reverberation); some transducers are better than others. The V3384 was chosen for the grain noise measurements reported below because its average signal was nearly devoid of stationary artifacts. The variance signal results from position dependent details of the microstructure, and its square root is termed "grain noise."

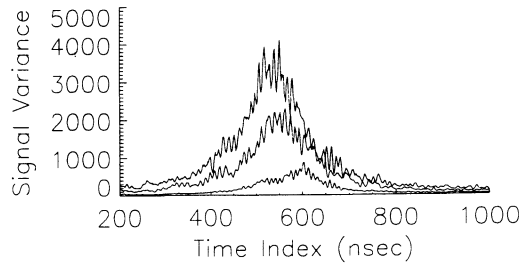

Figure 3. Variance of waveform ensembles for three compacted specimens: nitrided Ti-6-4 particles (highest), 50/50 mix of particles (middle), and plain Ti-6-4 particles (lowest).

Experimental results for the three samples described above are shown in Figure 3. The most prominent feature of the variance curves shown is that the peak increases with increased fraction of nitrided microspheres. With only three samples it is difficult to assert that the peaks vary linearly with composition, as would be predicted from the independent scatterer model, but the peak variance for the sample consolidated from a 50/50 mixture of Ti-6-4 and nitrided Ti-6-4 microspheres is just about the average of the other two values. Another feature of the variance curves is that the peak occurs earlier in time for samples with more nitrided particles. According to Margetan's model, the grain noise signal should peak for sound scattered from the transducer's focal zone. The observed shift in peak position is consistent, therefore, with a higher effective velocity in the nitrided material, and with higher attenuation (which would favor the return of scattering from nearer scatterers).

Numerical predictions of the Measurement Model, when run with material parameters similar to those encountered experimentally are encouraging. For samples composed of all nitrided microspheres, the measured peak grain noise was 56 millivolts while the prediction based on equation (2) and the results of theoretical scattering amplitude calculations was 51.8 millivolts. For samples compacted from plain Ti-6-4 microspheres, the measured peak grain noise was 26 millivolts, while the prediction based on equation (3) with a characteristic dimension appropriate to the average microsphere size was 8.6 millivolts. As pointed out earlier, the proper characteristic dimension is probably smaller than the one used for the present calculations. Moreover, it is not clear that the assumption of randomly and independently oriented crystallites of the various phases is appropriate to microstructures developed during the rapid solidification of particles.

CONCLUSIONS

Ultrasonic scattering from Ti-6-4 and nitrided Ti-6-4 microspheres was studied experimentally and theoretically. The microspheres were considered first in isolation (mounted in metallurgical mounting plastic) and then in specimens compacted by a hot isostatic pressing procedure. For both the plain and nitrided Ti-6-4 microspheres, experimentally obtained waveforms were in generally good agreement with theoretically calculated time-domain reconstructions based on Ying and Truell's canonical solution. Predictions of grain noise for the compacted specimens were made, using theoretically calculated scattering amplitudes to calculate a Figure of Merit in the case of the nitrided microspheres, and using Rose's expression for FOM in the case of the plain Ti-6-4 microspheres. Again, quantitative agreement between theory and experiment was good. The ability to discriminate between samples compacted from Ti-6-4 microspheres, nitrided Ti-6-4 microspheres, and a mixture of both has been demonstrated. The primary difference between these samples was the composition of the grain boundaries; bulk densities and wavespeeds were little affected, but grain noise rose considerably with grain boundary "contamination." Quantitative details of the grain noise measurements agree well with theoretical predictions, suggesting that assumptions of the Independent Scatterer Model are reasonable for these kinds of grain noise studies.

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