ACOUSTIC HARMONIC GENERATION MEASUREMENTS FOR MATERIALS CHARACTERIZATION

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INTRODUCTION

In the first presentation of the Otto Buck memorial session, Dr. Donald Thompson described contributions of Dr. Otto Buck on the theories of finite amplitude waves and elastic constants and his experimental work involving reflections from stress free interfaces and harmonic generation. The speakers following Dr. Thompson described many more of Dr. Buck's contributions in a wide variety of topics. Listening to the sessions speakers talk of their work with Otto made this author realize just how wide Otto's interests and expertise were. Here, we come full circle and discussed the preliminary results of work which relates directly to contributions remembered by Dr. Thompson regarding harmonic generation. This work was initiated by Dr. Buck at the Ames Laboratory and continues there.

The lattice contribution to harmonic generation in single crystals allows the determination of third order elastic constants or certain combinations of constants[1]. Contributions from dislocations and their interactions with microstructural features and interfaces in structural materials demonstrated potential applications as a nondestructive technique for evaluating microstructures. Measurements of acoustic harmonic generation and the nonlinearity parameter, β, have been shown to be sensitive to microstructure variations in materials such as interstitial carbon concentration and martensite tetragonality in steels[2], percent second phase[3] and fatigue induced dislocations[4] in aluminum alloys. From the noted examples, acoustic harmonic generation measurements are seen a cradle-to-grave technique, with applications beginning with materials processing and monitoring the material throughout service. A joint study currently in progress at Ames Laboratory and Oak Ridge National Laboratory (ORNL) is an effort to establish the theoretical and experimental basis for application of harmonic generation measurements in characterizing microstructural evolution and degradation.

CURRENT STUDY - SOLID SOLUTION EFFECTS

The first phase of this collaboration involves the evaluation of the contribution of varying solid solutions to harmonic generation. It is known that solid solution alloying will change the higher order elastic properties of a material, so it is expected that a change in harmonic generation will be seen. The Cu rich side of the Cu-Al alloy system was chosen as a model alloy because of the stability of the α-phase field to approximately 20 at. % Al. To ensure that no second phases contributions would be introduced, only Al concentrations below 15 at. % would be used. Specimens with compositions of Cu and 0, 2, 4, 6, and 8 at. % Al were produced at Ames Laboratory by high pressure gas atomization (HPGA) of the alloy into powder followed by cold isostatic pressing (CIP), hot isostatic pressing (HIP), and a full anneal.

Immersion density measurements were performed to ensure the processed powders had achieved full density. Longitudinal velocity values (required for the calculations of β) and hardness measurements were also made. Density and velocity results are shown in Fig.'s 1and 2, respectively. Metallography of the samples found the powder metal processing techniques produced very consistent grain sizes in the alloyed samples, with grain diameters measured in the 10-40 μm range. The pure Cu sample appeared to have suffered severe grain growth during the high temperature processes. Grains diameters in the pure Cu sample were measured to be in the 150-300 μm range. Immersion ultrasonic attenuation measurements showed attenuation levels to be much higher in the pure Cu sample, a result of the large grain size. The attenuation levels in the pure Cu sample were in fact so high that no harmonic generation measurements were possible. In contrast, attenuation levels were very consistent and relatively low in all alloyed samples. A 0.5 at. % Al sample has been produced in an effort to more closely approximate the pure Cu sample in terms of harmonic generation. Metallography has indicated that the severe grain growth seen in the pure Cu sample has not occurred: data from this sample will be published at a later date. Results from the attenuation measurements are shown in Fig. 4 as plots of the attenuation coefficient versus frequency for the model alloy samples.

ACOUSTIC HARMONIC GENERATION MEASUREMENTS

Harmonic generation measurements are typically performed in a through-transmission type of experiment and involve injecting a monochromatic toneburst into a sample and measuring the absolute amplitudes of the fundamental and second harmonic after the toneburst has propagated the length of the sample. Although other techniques utilizing a capacitance based receiver [5] or a laser interferometer [6] are available for measuring absolute displacements at the output side of the sample, this work makes use of fluid-coupled contact piezoelectric transducers and a reciprocity-based calibration technique. This broadband technique establishes the empirical conversion efficiency for the receiving transducer as a function of frequency and includes the effects of the coupling media, allowing the calculation of absolute displacement amplitudes. Details of the calibration technique have been described elsewhere [7,8]. This contact transducer technique was chosen because sensitivity and signal-to-noise are reasonable and sample preparation is relatively easy, with reasonable results obtained from surfaces prepared by polishing with 600 grit abrasives. For these reasons, it should be easier to implement as a fieldable inspection technique than other methods.

In single crystals, the nonlinearity parameter, β, is obtained from second and third order elastic constants using the relationship shown in equation 1,

$$\beta = -\left(3 + \frac{K3}{K2}\right) \quad (1)$$

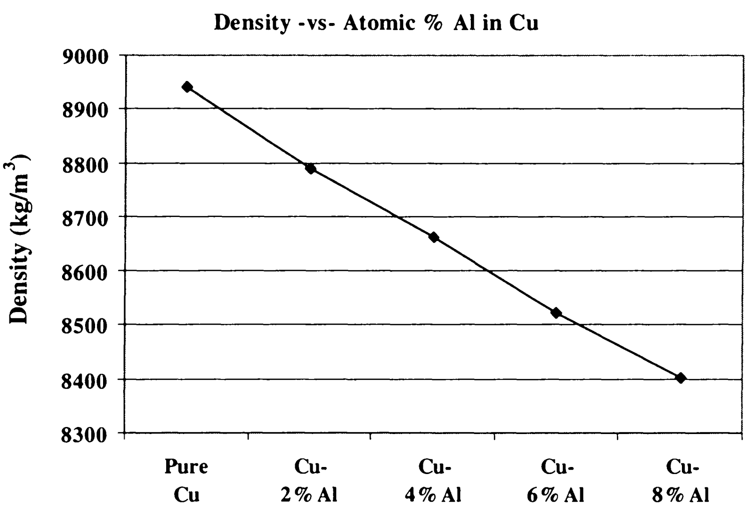

Fig. 1 Density measurement results.

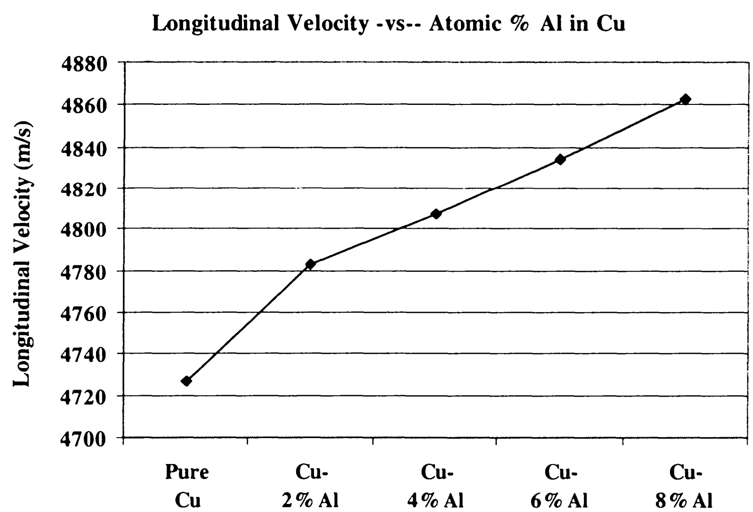

Fig. 2 Longitudinal velocity measurement results.

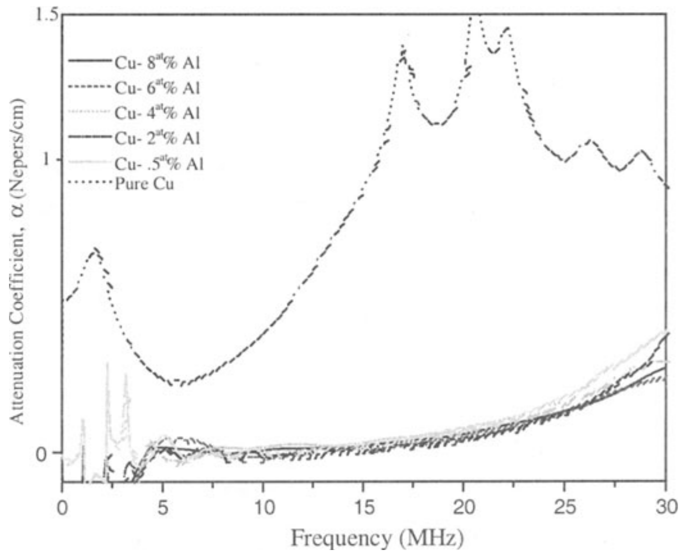

Fig. 3 Attenuation curves for Cu-Al powder metallurgy samples.

with, for example, $K3 = C_{111}, K2 = C_{11}$ for longitudinal waves propagating in the [100] direction. From experimental results, β is calculated using the expression shown in equation 2,

$$\beta_{expt} = \frac{8A_2}{A_1^2 k^2 x}, \tag{2}$$

with the variable x the sample or propagation length and A_1 and A_2 are the fundamental and second harmonic amplitudes, respectively. The symbol k is the wave vector at the fundamental frequency, f_o, where $k=2\pi f_o/c$, and c the longitudinal velocity in the sample. In this work, the fundamental frequency is 10 MHz, with the second harmonic frequency 20 MHz. The term β_{expt} is used because this measured value has experienced attenuation in propagating through the sample. A correction for attenuation[9] is applied to β_{expt} using attenuation coefficients at the fundamental and second harmonic frequencies, α_1 and α_2, respectively, yielding

$$\beta = \beta_{expt} \frac{\delta x}{e^{\delta x} - 1} \tag{3}$$

where $\delta = 2a_1 - a_2$ and x is the sample length.

INITIAL RESULTS

In measurements of the nonlinearity parameter at the Ames Laboratory, varying levels of fundamental amplitude are injected into the sample by the use of a stepped attenuator in-line between the tone burst amplifier and the transmitting crystal. With this data, β is calculated by plotting the second harmonic as a function of the square of the fundamental amplitude and use the slope determined from a least-squares fit of the data in Eq. 2 as the A_2/A_1^2 term. A problem arises when using this technique which results from the use of low fundamental amplitudes (and subsequently, low amplitudes of the second harmonic). Three trials on the Cu - 8at. % Al sample are shown in Fig. 5, where β is plotted as a function of fundamental amplitude. It is common to see β values to increase very quickly below a particular level of the fundamental [8,10], as seen in the curves of Fig 5. This artificial increase in β is known to be the result of the second harmonic amplitude falling below the noise floor, appearing to remain constant for ever decreasing levels of the fundamental amplitude. From Eq. 2, it is clear that if the numerator remains constant while the denominator decreases, the apparent value of β will increase. This effect acts an very nice indicator of the minimum sensitivity of the technique but makes the use of the least-squares fit to the data to calculate β unreliable. In these situations, the asymptotic value of β at the higher fundamental amplitudes is taken as the true value. The average of three trials on each of the model alloy samples is shown in Fig. 6.

One question that arose at this point of the work is what value of β should be expected? For comparison, an "average" β value was calculated from single crystal elastic constants to approximate the value that should be measured in a polycrystalline material. This average was calculated following Chang [11], using Eq. 1 with

$$K3={}^*C_{111}=({}^1/_{35})(15C_{111}+18C_{112}+12C_{144}+72C_{166}+2C_{123}+16C_{456}) \quad (4)$$

and

$$K2={}^*C_{11}=({}^1/_5)(3C_{11}+2C_{12}+4C_{44}). \quad (5)$$

Using single crystal data reported by Cain and Thomas [12], values of 8.82, 8.76 and 8.64 for pure Cu and Cu-Al alloys (0-, 3.1- and 7.4 at. % Al, respectively) were calculated for β. Note that these values are consistent with the high amplitude values (A_1 >10Å) of β from the Ames Laboratory specimens. The β values from the measured (polycrystalline) and calculated (single crystal) results appear to indicate that the contribution to harmonic generation from changes in solid solution is small. The data also shows a need for

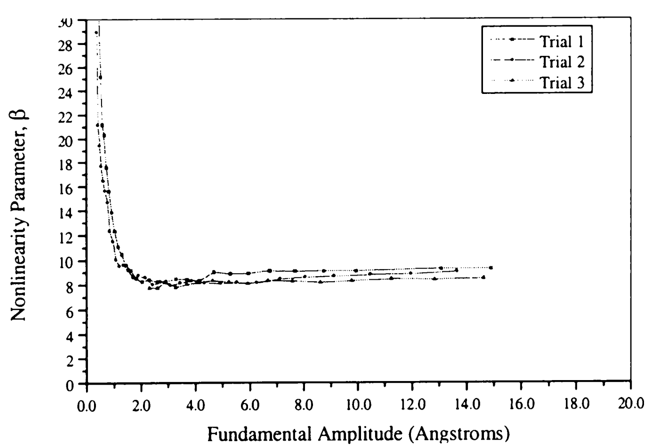

Fig. 4 Results of three trials on Cu-8at% Al powder metallurgy sample

Nonlinearity Parameter -vs- Fundamental Amplitude
Average of three trials per sample

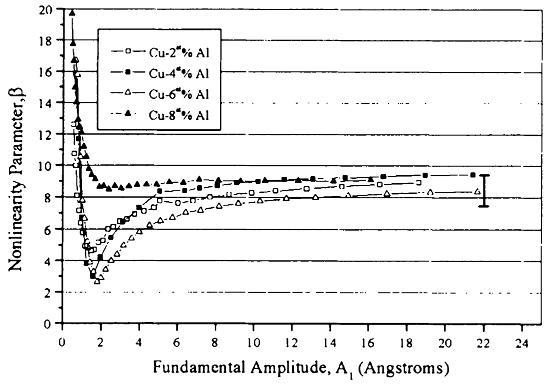

Fig. 5 Average of three trials on Cu-Al powder metallurgy samples

improvements in the measurement technique to minimize error, which is indicated by the bars in Fig. 6.

THE "HOOK"

Upon seeing the data from these initial results on the work, Dr. Buck pointed to the minimum in the curves of the 2-, 4-, and 6 at.% Al samples at low fundamental amplitude levels, with β values around 4, and questioned, "Vats dat hook? (the proper accent is required). Is it real?" A "hook" or minima at low amplitudes had not been observed before and would raise some important fundamental issues.

Recently, a similar minimum has been observed by others in commercial aluminum alloys in harmonic generation measurements using a capacitance microphone as a receiver and in pressure derivative of velocity experiments [13]. With this corroboration the phenomenon is believed genuine, but measurement effects have not been ruled out completely. The mechanism producing this finding is not yet immediately known but these results bring into question previous measurements of third order elastic constants (TOEC's). The β values in the 8-10 range were thought to contain only the lattice contribution to harmonic generation. If the minimum values for b are representative of the nonlinearity of the lattice, previously measured TOEC's may be "contaminated" with some effect. If this is the case, the true solid solution contribution has not been demonstrated. If not, then the mechanism that is suppressing β at low amplitude needs to be identified.

CONCLUSION

It is quite apparent that much work is left to do regarding the development of our understanding of harmonic generation and its applications for characterizing materials, particularly in materials where multiple contributors are active. Future work will involve the study of dislocation effects in the current Cu-Al alloy samples and a subsequent sample group that will be subjected to varying amounts of plastic deformation. Samples will also be produced to study second phase effects, as will specimens that contain combinations of effects. A thorough study of the measurement technique used here is needed so as to rule out measurement effects on results, particularly in the low amplitude areas. An evaluation of the receiver electronics in the measurement circuit will concentrate on ways of increasing signal-to-noise and precision and allow a more complete probe of the low amplitudes.

ACKNOWLEDGEMENTS

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REMEMBERING OTTO

In the introduction, it was noted that the speakers of the session commented on the wide variety of Otto's contributions to science. There was not, however, much variety in the personal messages of the speakers regarding Otto. This would be expected, we all felt the same way about Otto. Otto Buck was a wonderful person to work for and with, and his personal contributions of friendship, wisdom, time, and humor will remain as important to this author as those recorded in journals or proceedings.

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