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LONG WAVELENGTH ULTRASONIC CHARACTERIZATION OF INCLUSIONS IN SILICON NITRIDE

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ABSTRACT

The size and material content of Fe and Si inclusions in Si₃N₄ have been measured by means of pulse-echo scattering of elastic waves in the frequency range 5 to 100 MHz. The inclusions were of 100 µm and 400 µm nominal diameters and were located 3 mm deep in the Si₃N₄. The electronic noise was reduced by signal averaging and, in some cases, the noise due to grain scattering was reduced by averaging over transducer position. The scattering amplitude A(ω) and the impulse function R(t) were obtained by a desensitized deconvolution of the reference waveform obtained by reflecting the transmitted pulse from the back surface of the sample in a defect-free region. Comparison of theory and experiment are given for A(ω) and estimates of flaw size and material content are presented.

INTRODUCTION

Silicon nitride and other ceramics have a greater fracture strength at high temperatures than do metals. They are, therefore, candidates for use as turbine blades in new high temperature turbofan engines. The fracture strength of silicon nitride in known to depend not only on the size, shape and orientation of inclusions it contains, but also on the material an inclusion is made of. For example, the critical flaw size for an Si inclusion in hot pressed Si₃N₄ is 40 µm whereas for an Fe inclusion it is 150 µm and a WC inclusion reduces the fracture strength of the bulk Si₃N₄ hardly at all.

The most promising method for detecting and characterizing these defects is ultrasonics. There are two experimental ultrasonics tasks being performed in parallel under this program. These tasks are roughly divided into the two frequency regimes where the ultrasonic wavelength is small compared to the defect size (Stanford, described elsewhere in the proceedings) and comparable to or large compared to the defect size (Science Center, reported here). This is natural division both in terms of the experimental techniques required and the wave scattering theories used to interpret the ultrasonic waveforms. The same set of silicon nitride specimens containing defects of various known sizes and types are being studied in each task as well as by acoustic microscopy (Sonosan, also described in this proceedings). It may turn out that elements of all of these measurements are required to characterize the defects.

A third task (also described elsewhere in this proceedings) considers the statistical aspects of the ultrasonic flaw characterization, flaw distribution, component stress history and the consequences of falsely accepting or rejecting a component in formulating a probabilistic accept/reject criterion.

EXPERIMENTAL PROCEDURE

The overall experimental approach, described in Fig. 1, required obtaining and characterizing seeded specimens, developing measurement capabilities, analyzing the experimental data to extract features which are a consequence of the characteristics of the defect and then using these features in an inversion formalism to obtain a probabilistic defect description.

Specimen Characterization. Hot pressed Si₃N₄ specimens containing specified inclusions were ordered from Norton Co. for use in developing the ultrasonic defect characterization techniques. Ten each of 1 in. diam. x 1/4 in. thick specimens containing Si, Fe and C inclusions 100 µm and 400 µm in diameter were ordered but 16 of each type were delivered. Only a few specimens of each type met the specified density of > 3.20 gm/cm³ as seen in Fig. 2. Each of the 96 specimens was therefore packaged and labeled to retain its individual identity. Master lists were maintained showing the correspondence between specimen numbers and the specimen density, thickness, wave velocities, and defect locations as this information became available.

Instrumentation. Some instrumentation development was required to extend present ultrasonic capabilities to higher frequency and greater sensitivity. First, a scanning liquid bath system was designed and constructed to accommodate two transducers, both of which are able to be positioned at an angle with respect to the specimen surface. Also, the specimen is mounted in a goniometer so all sides of the defect may be observed.

Secondly, new techniques have been devised util-
Fig. 2 Distribution of densities among the hot pressed $\text{Si}_3\text{N}_4$ test specimens.

using unfocussed broadband pulser-transducer-buffer combinations using lithium niobate transducers and fused silica or 5061-T6 Al (aluminum) buffer rods, and pulsers using transistors operating in the avalanche mode to deliver high voltage delta function pulses to the transducers.

Scanning Bath System. The $x$, $y$ scanning system is shown schematically in Fig. 3. The specimen is placed in the immersion tank (A) which is mounted to a goniometer (B). Transducer holders (D, E) are to be positioned at an angle with respect to the specimen. These angles are measured with the transducer goniometers (G). Transducer holder E can also be moved away from holder "D" by its own separate holder, goniometer device (F). Knob "H" is a positioning knob to insure that the transducers are normal to the specimen in that plane. "I" is the arm which is mounted on the $x$, $y$ scanning device and is able to move in the "z" direction. Computer controlled scanning in raster steps as small as 25 $\mu$m can be accomplished using focused or unfocussed transducers in either the pulse-echo or pitch-catch modes. This system was prepared for use in the 100% volume inspection of the critical regions of $\text{Si}_3\text{N}_4$ turbine blades. The present measurements on the seeded specimens were made, however, using only contact transducers in pulse-echo at 0° and 15° incidence angles.

Broadband Transducer System. In the past, a modified Panametrics pulser-receiver was used for the detection of the defects. This system, however, has characteristics which can be greatly improved upon. First, the receiver amplifier has an upper cutoff frequency of 35 MHz which does not allow the characterization of flaws (275 microns; secondly, the transmitter pulser has restrictions on its rise time; and third, the use of long, unmatched (up to 4 ft.) coaxial cables can result in unwanted reverberations in the transducer signal at higher frequencies which can give erroneous results in the characterization of the defects.

To solve these problems, pulser-transducer-buffer systems were constructed that were mounted in a manner to minimize circuit inductance and designed to match 50 ohm coaxial cable with 50 ohm attenuators and broadband amplifiers. The pulser is a transistor operating in the avalanche mode whose circuit layout construction uses high frequency strip line techniques (i.e., all leads are very close to ground planes and have parallel plate construction). The lead to the transducer is kept as short as possible and is the major source of system inductance. However, the resonant circuit consisting of the transducer-system capacitance and inductance is heavily damped and does not seem to cause any problems (Fig. 4).

Fig. 4 Pulser-TR switch schematic.

The pulser can be tuned to the transducer by changing the discharge capacitor ($C_1$) and the resistors $R_1$, $R_2$, $R_3$ are altered to give a 50 ohm output impedance. The attenuators and amplifiers are wide band (attenuators have 0-1 GHz bandwidth and the amplifiers are 5 MHz to 1 GHz with ~30 dB gain) with low noise figures (4.5 dB).
Because of the limited bandwidth of any single transducer, several had to be constructed to cover the required frequency range. The reason for this can be seen in Table I where the range of frequencies corresponding to $0.2 < k_0 a < 3$ are shown for various defect sizes within the range of interest. Because $k_0$, rather than frequency determines the nature of scattering from a defect, transducers suitable for a 400 µm diameter defect are unsuitable for characterizing a 40 µm diameter defect.

Table I
Frequency (MHz) - $k_0$ Correspondence
For Typical Defect Diameters

<table>
<thead>
<tr>
<th>d (µm)</th>
<th>VALUES OF $k_0$</th>
</tr>
</thead>
<tbody>
<tr>
<td>400</td>
<td>1.6 8 24</td>
</tr>
<tr>
<td>200</td>
<td>3.2 16 48</td>
</tr>
<tr>
<td>100</td>
<td>6.4 32 96</td>
</tr>
<tr>
<td>40</td>
<td>15.5 78 235</td>
</tr>
</tbody>
</table>

Table II lists the transducer-buffer combinations which were constructed and used in making the measurements. In most cases they were made by bonding a longitudinal lithium niobate crystal onto the end of the buffer rod, lapping the crystal to the desired thickness (resonance frequency), then providing an electrode and backing material for critical damping to increase its bandwidth. The buffer length and electrode diameter were chosen so that the defects in the seeded specimens would be in the far-field radiation pattern of the transducer over its frequency bandwidth. Figure 5 shows the characteristics of two of these transducers, obtained by a pulse echo measurement of a plane reflector. A matching 15° aluminum wedge was bonded to the face of the 40 MHz LiNbO$_2$-buffer combination to get the plane surface reflection shown in Fig. 5.

Table II
Transducers and Buffers Which Have Been Constructed

<table>
<thead>
<tr>
<th>TRANSDUCER</th>
<th>BUFFER</th>
<th>TYPE</th>
<th>FREQUENCY (MHz)</th>
<th>DIAM. (µm)</th>
<th>MATERIAL</th>
<th>LENGTH (cm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PANAMETRICS</td>
<td>0.04</td>
<td>0.32</td>
<td>15</td>
<td>1.27 Al</td>
<td>2.74</td>
<td></td>
</tr>
<tr>
<td>PZT</td>
<td>0.17</td>
<td>0.32</td>
<td>25</td>
<td>0.95 F.S</td>
<td>1.27</td>
<td></td>
</tr>
<tr>
<td>LiNbO</td>
<td>0.17</td>
<td>0.32</td>
<td>40</td>
<td>0.95 Al</td>
<td>1.27</td>
<td></td>
</tr>
<tr>
<td>LiNbO</td>
<td>0.17</td>
<td>0.32</td>
<td>50</td>
<td>0.95 Al</td>
<td>1.27</td>
<td></td>
</tr>
<tr>
<td>LiNbO</td>
<td>0.17</td>
<td>0.32</td>
<td>60</td>
<td>0.95 F.S</td>
<td>1.27</td>
<td></td>
</tr>
<tr>
<td>LiNbO</td>
<td>0.17</td>
<td>0.32</td>
<td>70</td>
<td>0.95 F.S</td>
<td>1.27</td>
<td></td>
</tr>
</tbody>
</table>

Fig. 5 Examples of Transducer Characteristics.

Measurements. Several specimens of each type were scanned and the coordinates of any ultrasonic indications above the noise level were compiled. The number of indications range from 0 - 14 per specimen. No indications were observed in the samples containing carbon inclusions. This could be due to conversion (during the hot pressing) of the carbon seed to silicon carbide which has a similar acoustic impedance to the host material.

For the strongest indications, a variety of measurements were conducted. The measurements include normal incidence pulse-echo scans across the defect at close-spaced intervals (< 0.1 mm), and measurements at 30° incidence with the sample being rotated to 0, 90, 180, and 270 degree azimuthal angles using the Al wedge transducer-buffer system. Experiments were conducted on both sides of the sample. Figure 6 shows the raw waveforms for a 100 µm Si inclusion and a 400 µm Fe inclusion, respectively. Note that, as expected, the echo for the larger flaw has a longer time duration.

Data Analysis. Two features were extracted from each waveform: $A_0$ and $d$. They are derived from the low and intermediate frequency portion of the scattering spectrum. The reason for this choice is that for flaws which are as
irregular and spatially and chemically inhomogeneous as inclusions in Si$_3$N$_4$ are known to be; the high frequency portion of the spectrum will be dominated by fine structural details of the flaw whereas the overall size and material properties of the flaw will be easily accessible at lower frequencies.

$A_2$ is the coefficient of frequency squared in the long wavelength ($ka \ll 1$) regime. In this regime, $A_2$ completely defines the scattering of ultrasound from the flaw in a given direction. Therefore, by combining the values of $A_2$ for a variety of directions, it has been demonstrated that it is sometimes possible to obtain estimates of the size, shape and orientation of the defect. In addition, the sign of $A_2$ indicates whether the acoustic impedance of the flaw is greater or less than that of the host material.

The second feature which is extracted from the data is "d" which is an estimate of the defect radius in a given direction obtained using the one dimensional Born inversion algorithm. The Born inversion uses the intermediate frequency ($ka$-1) part of the data as well as the low frequency phase data.

The extraction of $A_2$ and d was performed in the frequency domain, although equivalent time domain algorithms can be written and may prove more efficient computationally. The raw waveforms have had a reference waveform from a flaw-free region of the sample subtracted from them, and have then been Fourier transformed. The spectrum of the incident pulse was then divided out of the measured spectrum, yielding the normalized spectrum. $A_2$ was extracted by plotting the spectrum in a log-log manner and finding the intercept of the slope-of-2 region. d was extracted by time centering the spectrum using the low frequency flaw center, applying the Born inversion, and estimating radius as the area of the characteristic function divided by its peak.

Inversion. The inverse problem of finding the flaw properties given the measured features is handled by a statistical inversion algorithm. The input data consists of one or more pairs of $A_2$ and d. By making use of the ability to calculate $A_2$ for any spheroidal geometry, the algorithm iteratively arrives at best estimates of the scatterer geometry and type given the input data. Appropriate confidence measures are also included in the output.

The inversion algorithm can be used in either of 2 different methods. In the first, only one normal incidence pulse echo measurement of the flaw is performed. This is the data that would be available if a part were being rapidly scanned. In the second method of using the algorithm, a set of pulse echo measurements from various angles are made, as would be done in a detailed inspection of a suspected flaw. The resulting set of $A_2$ and d pairs is input to the inversion algorithm and estimates are made of the size, shape, orientation and material content of the spheroid which best fits the flaw. Generalization of the algorithm from spheroidal to ellipsoidal geometry would be straightforward.

RESULTS

Comparison of Measurements with Theory. Figure 7 shows theoretically calculated frequency ($ka$) spectra and impulse response functions for spherical inclusions of Si and Fe in Si$_3$N$_4$, respectively. $A_2$ describes the parabolic region of the frequency curves below $ka = 0.5$. In each case, the impulse response function shows a front surface echo as well as miscellaneous later echoes. In the case of Fe, the front surface echo is small and the late arriving echoes extend well beyond the right edge of the figure.

The first notable observation about the data is that for 2 different 400 $\mu$m Fe inclusions, there is no detectable front surface echo. Although the expected front surface echo is small for an Fe inclusion, it should still have been detectable. A likely explanation of this is that the very irregular front surface of the flaw will not specularly reflect the high frequency.
components of which the front surface echo is largely composed. This should not appreciably affect the low frequencies for which the wavelength is larger than the scale of the irregularities. The complete absence of a front surface echo suggests that some other as yet not understood mechanism may also be present. For the 100 μm Si inclusion studied, the incident sound pulse is not quite short enough to allow us to determine if the front surface echo is missing. However, if it were missing, this would have larger effect than in the case of Fe.

In order to explore the implications of a missing front surface echo, we have calculated the frequency spectra expected for spherical inclusions if the front surface echo is windowed out of the impulse response of the flaw. Figure 8 is a composite presentation of these comparisons. It shows, for 100 μm Si and 400 μm Fe inclusions, various frequency spectra and, below them, the deconvolved impulse response of the measured data. The solid curve in the frequency spectra is the normalized spectrum of the measured waveform. The dashed curve in each case is the calculated spectrum for a spherical inclusion (including front surface echo). The dotted curve is the calculated spectrum with the front surface echo windowed out.

For the case of Fe, the small size of the front surface echo means there is little difference between the two theory curves. The measured spectrum agrees reasonably with the theory. In the case of Si, the spectrum is greatly affected since about half of the impulse response has been windowed out. In particular, the windowed spectrum looks like the unwindowed spectrum of a smaller flaw. The measured flaw spectrum seems to be more like the windowed (front surface echo missing) spectrum. However, remember that the theory curves are based on the nominal size of the flaw. The photomicrograph of this flaw after sectioning (see Fig. 11 and accompanying discussion) leaves open the question of whether there is a systematic lack of front surface echoes in the low frequency regime. In the absence of more definitive evidence, we have done the flaw characterizations assuming the presence of the front surface echo.

Determination of Flaw Size and Material. The inversion algorithm requires as inputs the set or sets of A<sub>2</sub> and d, a priori error estimates for them and the list of candidate inclusion materials to be considered. By observing the sign of A<sub>2</sub>, the algorithm can immediately limit its consideration to those candidate materials whose acoustic impedance is on the same side of the host material's acoustic impedance as that of the flaw being measured.

The output of the inversion algorithm is presented graphically in Fig. 9 and 10. These figures show the joint probability of flaw radius and flaw material plotted vs radius. Therefore, each curve is the probability of the flaw having a given radius assuming that the flaw is made of the given material. The area under each curve is therefore the probability of the flaw being made of the corresponding material. These probabilities are listed for each curve and are the basis for deciding which material the flaw is made of. The results obtained from these plots are summarized in Table III.

<table>
<thead>
<tr>
<th>NOMINAL MATERIAL</th>
</tr>
</thead>
<tbody>
<tr>
<td>APPROACH FLAW RADIUS (μm) MATERIAL PROBABILITY FLAW RADIUS (μm)</td>
</tr>
<tr>
<td>I Fe 300 Fe 0.71 109</td>
</tr>
<tr>
<td>I Si 50 Si 0.66 20</td>
</tr>
<tr>
<td>II Si 50 Si 0.44 20.95 4</td>
</tr>
</tbody>
</table>

Table III
Results

For the case of Fe, the small size of the front surface echo means there is little difference between the two theory curves. The measured spectrum agrees reasonably with the theory. In the case of Si, the spectrum is greatly affected since about half of the impulse response has been windowed out. In particular, the windowed spectrum looks like the unwindowed spectrum of a
Figure 9 contains the results for a nominally 400 \( \mu m \) diameter Fe inclusion. Method 1 (one pulse echo waveform, assumed sphericity) was used. The probability of the flaw being Fe is 71\% and the most likely diameter is 410 \( \mu m \). This estimate is within 3\% of size determined by destructive examination.

**Figure 9 Joint probability of flaw type and radius for nominal 200 \( \mu m \) radius Fe inclusion.**

Figure 10 shows the probability curve for a nominal 100 \( \mu m \) diameter Si inclusion treated by method 1. The probability of it being Si is 60\% and the most likely diameter is 52 \( \mu m \).

**Figure 10 Joint probability of flaw type and radius for nominal 50 \( \mu m \) Si inclusion.**

Another set of results for the same Si flaw are given in the table, this time using method 2 (several measured waveforms). The flaw is again called Si and it is estimated to be slightly oblate (26 \( \mu m \times 33 \mu m \)) and slightly tilted (4\°) from the surface normal.

**Figure 11 Photomicrograph of this Si flaw after sectioning. It is revealed to be 2 very inhomogeneous regions close to one another. The observed radii are seen to be less than the nominal 50 \( \mu m \) radius of the Si seed, due no doubt to the dispersing of the original spherical seed which occurred during hot pressing. Both methods of analysis estimate a 26 \( \mu m \) radius in the direction perpendicular to the plane of the photograph, where an exact measurement is not available. However, judging by the lateral radii visible in the photograph, these estimates, together with the 33 \( \mu m \) lateral radius estimate, are somewhat low, but are probably within 40\% of the correct values. This result is gratifyingly good, considering how much the actual flaw differs from the simple model flaws upon which the analysis is based.**

Two comments are in order here. First, for a pair of flaws interrogated together, a radius estimate based on \( A_2 \) alone should increase only by the sixth root of 2. Also, the radius estimate from the Born inversion for normal incidence should be little bigger than for each of individual regions alone because of their positioning next to one another. Secondly, recall the lack of measurable front surface echo mentioned above and the large effect it had on the frequency spectrum of Si inclusions. Because the spectral features are offset to higher frequencies when there is no front surface echo, this would lead to underestimation of \( A_2 \) and \( d \) and therefore to underestimation of flaw size. It is possible that this is occurring here.

**CONCLUSIONS**

It has been shown that we can determine the size and material content of inclusions in silicon nitride with reasonable accuracy by using as few as one low frequency pulse echo ultrasonic waveform. The material of which the inclusion is made is identified correctly from among a small set of likely candidates. The flaw size was accurately estimated for a large (400 \( \mu m \)) Fe inclusion and somewhat underestimated for a small (100 \( \mu m \)) Si inclusion. The causes of this underestimation are under investigation.
These results provide a significant advancement in the state-of-the-art of characterizing flaws in ceramics, especially in determining the material content of the flaw.

ACKNOWLEDGMENT

This research was sponsored by the Center for Advanced NDE operated by the Rockwell International Science Center for the Advanced Research Projects Agency and by the Air Force Materials Laboratory under Contract No. F33615-76-C-5180 and by the Rockwell International Independent Research and Development Program.

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