HIGH-FREQUENCY BULK WAVE MEASUREMENTS OF STRUCTURAL CERAMICS

Stanford University
Stanford, CA 94305

ABSTRACT

A 50 MHz C-scan imaging system is under construction for fast defect detection. A high-frequency (150-450 MHz) A-scan system is used for host material evaluation, and defect characterization. Several signal processing schemes such as time and space averaging, Wiener filtering, diffraction and propagation loss corrections are used in the process of defect characterization. Further modifications of the exact theory of scattering from spherical inclusions are made to ease the process of defect identification.

C-SCAN SYSTEM

A C-scan imaging system is being built to scan ceramics for interior defects. Initially, the device will operate at 50 MHz with a partially focused transducer to produce a beam approximately 1 mm in diameter. The device will be capable of scanning in both the x and y directions along the surface of a ceramic. By operating in a reflection mode, it should be possible to determine both the depth and the transverse position of a flaw. Eventually, it is intended to operate at frequencies as high as 400 MHz and to use the same system to evaluate the size and nature of the flaw. By using a moveable point source as a synthetic aperture system, i.e., a focused beam focused on the surface of the ceramic, we expect eventually to be able to reconstruct an image of the flaw on the computer.

The mechanically-scanned system being designed for this purpose has to satisfy the following requirements:

1. the system should be computer compatible, and it should be possible to control the whole system operation from a computer terminal; and

2. the mechanical scans should be accurate to about 70 \( \mu \)m or less (\( \pm 1 \% \) at 100 MHz).

Thus, defect location and evaluation could be eventually carried out on one system. We believe that such a system will combine the advantages of A-scan with those of the acoustic microscope, but be able to examine internal as well as surface defects. For this application, steps of the order of one or two wavelengths in the X and Y directions are usually required.

Go-To Mode - In this mode, the transducer can be moved to any location of interest in its field. The position is dialed on thumb-wheel switches and is found with an accuracy of \( \pm 8 \mu \text{m} \). The scan can be continued from this location in any of the modes previously described.

Flag-Stop Mode - In this mode, the scan stops when a defect is located. A discriminator set by thumb-wheel switches is used to decide at what level a signal is considered to be due to a defect. The discriminator also gives out a signal to drive the Z-axis (pen up, pen down) of an X-Y recorder.

A combination of these modes of operation may be the best way to use the system. For instance, a slow scan at a moderate speed can be used to determine the rough location of the defect. A go-to command followed by a fine-step scan is used to determine the correct location of the defect. A flag-stop can be used to stop the scan over the defect location (when the signal is maximum). The X-Y location of the defect is displayed on digital displays in the main panel and/or the plot of an X-Y recorder. The A-scan display is continuously monitored on a cathode ray oscilloscope which can be used to determine the location of the defect in the depth of the sample.

HIGH-FREQUENCY A-SCAN SYSTEM

A schematic diagram of the A-scan system used in our study is shown in Fig. 1. The bottom end of the buffer rod is polished with a radius of curvature of 20 cm. Contact to the ceramic under study is made by pushing the curved end of the buffer rod against the ceramic without using a contacting layer such as gold. The transducer is excited with a 30 V, 2 nsec electric pulse in order to obtain broad bandwidth operation (150-450 MHz).

The received signal at the transducer is passed into a sampling oscilloscope which yields an output that is a slowed down version of the pulse. A Biomation analog-to-digital converter digitizes the slowed down pulse for insertion into a PDP-11/34 minicomputer. The computer is used to take fast Fourier transforms and correct the received signal.

Signal Processing Methods - The signal processing schemes we use to correct and improve the signals from the defects are:

Increment Mode - Scanning is carried out by stepping from one point to the next. The step size can be varied from 50 \( \mu \)m to 1 cm with an accuracy of \( \pm 1 \% \)um. The mechanical settling time per step is 3 msec. In this mode, the number of rf samples taken per step is settable from 0-999. The speed of the scan is controlled by the operator.
Loss of power due to diffraction between two identical piston transducers of radius $a$ as a function of their distance apart in units of $a^2/\lambda$. 

1. Time domain averaging.
2. Space domain averaging.
3. Wiener filtering.
4. Propagation loss correction.
5. Diffraction loss correction.

Time domain averaging consists of reading the signal $N$ times at a certain location, then taking the average of the sum of the $N$ signals. The amplitude of the signal from a defect relative to random noise is improved by $\sqrt{N}$. Space domain averaging consists of reading the signal at $N$ different locations on the sample, then taking the average of the $N$ signals. The criteria for the maximum step over the sample is to keep the signal from a defect almost constant, i.e., the defect is kept within the radius of the beam. This type of averaging improves the signal-to-grain scattering noise level. Wiener filtering is a deconvolution scheme used to remove the effect of the transducer response. This technique, which has been described in the previous report, consists mainly of dividing the received signal by the impulse response of the transducer while taking account of the presence of noise in the system. Propagation and diffraction loss corrections take care of acoustic beam spreading and propagation loss through the host material itself. By using the techniques described, we have been able to improve the signal-to-noise ratio in the system, and look at the impulse response of defects separated from the effect of the transducer and the propagation media.

Propagation Loss Measurements - One important step in the NDE of ceramics is to evaluate the quality of the host material itself. If the host material is porous or has large grains and does not meet fracture strength requirements, there is no point in looking for isolated defects and attempting to characterize them. For this test, we carry out a propagation loss measurement vs. frequency, as described in an earlier publication.

The most important development in the last year has been to calculate the exact diffraction loss for all ranges of normalized distances from the transducer. This loss can be written as

$$
\langle p \rangle = 2\pi \int_0^\infty \frac{J_1^2(y)}{y} e^{-\gamma y} \sin\theta dy
$$

where $\langle p \rangle$, $\langle p_o \rangle$ are the average values of the pressure over the area of the output transducer at a distance $z$, and at the input transducer, respectively. $S = z/a^2$ where $a$ is the transducer radius, $k$ is the wave number, and $J_1(y)$ is a Bessel function of the first kind and first order.

The diffraction loss $L$ is therefore

$$
L = 20 \log \frac{\langle p \rangle}{\langle p_o \rangle}
$$

Fig. 1 shows a plot of the diffraction loss versus $S$, for $S$ varying from 1 to 100. When the medium is anisotropic, the loss can be found simply by changing $S$ to $(1-2b)S$ where $b$ is an anisotropy coefficient defined as

$$
b = \frac{(C_{33}-C_{13}-2C_{44})(C_{33}+C_{13})}{2C_{33}(C_{33}-C_{44})}
$$

For a material with hexagonal symmetry, such as sapphire (the buffer rod), $b = .16$. For isotropic materials, these formulae are reliable for $S << 1$, provided $ka >> 10$. For anisotropic materials, however, the accuracy of the formula is better for $S > 1$.

We repeated our measurements of propagation loss in hot pressed NC 132 silicon carbide and at different locations in the same sample. Fig. 2 shows the results of some of these measurements. The loss is about 3 dB/cm at 300 MHz and follows an $f^4$ dependence, as would be expected from Rayleigh scattering theory. The value of the propagation loss at 300 MHz and the $f^4$ dependence were consistent for good hot-pressed silicon nitride samples. For inferior samples, we will discuss our results on some "bad" or lossy samples. It will be noted that, especially in the lower frequency range, it is vital to correct for diffraction.

Scattering Theory - The type and size of defects is determined by comparing their back-scattered power spectra to theory. The theoretical calculations for various types of spherical inclusions in a silicon nitride host matrix were calculated using the theory developed by Ying and Truell, and Johnson and Truell, using techniques similar to those of Richardson and Cohen. A PDP 11-34 minicomputer was used to carry out the calculations.
We found that the time taken to calculate the back-echo period for a scattering structure is much simpler for a vacancy than for an inclusion. The reason is that the inclusion has several types of resonances, whereas a vacancy has only one. The vacancy gives rise to a back-scattered echo from its front surface, and thereby gives rise to the quasi-periodic back-scattering structure. On the other hand, a wave can propagate through the middle of the inclusion and be scattered from its back surface as either a shear or a longitudinal wave. The return echoes interfere with each other, thus giving rise to a far more complicated form for the scattering cross-section as a function of frequency.
impulse responses of the defects. The various waves are shown schematically in Fig. 4. It is obvious from Figs. 3 and 4 that different inclusions give rise to different return echo responses in both the frequency and time domains, thus making it possible to differentiate between different types of defects. It is also obvious that a void can be directly differentiated from inclusions because its spectrum is very flat with frequency, i.e., there is only a strong return echo from its front surface. Thus, it is simple to classify defects as to whether they are voids or inclusions.

If a defect is a void, it is typically not possible to detect the second echo due to the leaky wave propagating around it. This we believe is due to the fact that the inside surface of the void is rough and scatters the circumferential wave before it reaches the transducer. The size of a void is then determined from a measurement of the echo amplitude. Another distinction to be made is whether a void is a "volumetric" void or a flat crack. This is typically done by measuring the thickness of the void either by measuring its location from the two sides of the sample, or by measuring the distance from the back wall to the mirror image of the defect with respect to the back wall.

If the defect is an inclusion, we may use the signal processing schemes described above to compare the back-scattered spectrum of the flaw to theory and decide on its type and size. In our efforts to confirm the theory with samples having seeded defects, a new problem arose: the seeded samples are usually porous resulting in a large amount of back-scattering from grain boundaries and, of course, a large amount of propagation loss in certain cases. The effect of this grain scattering is that the resultant noisy signal tends to obscure the front face echo, which is relatively weak compared to the back face echo. In some cases space averaging enabled us to reduce the effect of grain scattering and isolate the front face echo. In many other cases, this was not possible, and we had to decide on the type and size of inclusions using the defect back-face echoes only.

We simulate this case in the theory by gating out the specular reflection from the front face in
the time domain, then Fourier transforming the remainder of the signal into the frequency domain. The results of this calculation are shown as dashed lines in Figs. 3a, b and c. Notice that as there is no longer interference between the back-face and front-face echoes, the high-frequency ripples in the time domain, then Fourier transforming the remainder of the signal into the frequency domain. is no longer interference between the back-face lines in Figs. 3a, b and c.

Results - We used a number of seeded NC 132 silicon nitride disks to confirm our theory on detection and characterization of defects. The samples were seeded with iron (Fe), silicon (Si) and carbon (C) inclusions. The sizes of the inclusions were either 100 µm or 400 µm in diameter. We tested seven of these samples and the results are shown in Table I.

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Type</th>
<th>Size of Seeded Incl.</th>
<th>a (a/sec)</th>
<th>Vp (km/sec)</th>
<th>Vs (km/sec)</th>
<th>No. of Defects</th>
<th>No. of Voids</th>
<th>No. of Cracks</th>
<th>Size of Inclusions (µm)</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>67</td>
<td>Fe-100</td>
<td>1.3</td>
<td></td>
<td>3.13</td>
<td>300</td>
<td>5</td>
<td>3</td>
<td>2</td>
<td>30 µm (average)</td>
<td>High density.</td>
</tr>
<tr>
<td>66</td>
<td>Si-100</td>
<td>2.5</td>
<td></td>
<td>2.5</td>
<td>300</td>
<td>5</td>
<td>3</td>
<td>30 µm (average)</td>
<td>High density.</td>
<td></td>
</tr>
<tr>
<td>93</td>
<td>Si-100</td>
<td>1.2</td>
<td></td>
<td>2.5</td>
<td>300</td>
<td>4</td>
<td>3</td>
<td>0.1 µm (average)</td>
<td>Low density.</td>
<td></td>
</tr>
<tr>
<td>32</td>
<td>Fe-100</td>
<td>1.2</td>
<td>1.6</td>
<td>3.2</td>
<td>300</td>
<td>4</td>
<td>3</td>
<td>0.3 µm (average)</td>
<td>Low density.</td>
<td></td>
</tr>
</tbody>
</table>

Judging by the number of defects found in the seeded samples, we observe that over 90% of all defects found were either voids or appeared to be cracks, which in fact were later found to be BN discs. Many voids may not occur in unseeded samples and certainly do not occur in high quality NC 132 material.

We concentrated our detailed quantitative interpretation on the measurements of sample #1, the sample with isolated inclusions. After processing all the signals from the defects, as described, we found that three of the defects in the sample were Si inclusions 100 µm in diameter. This result is in agreement with the type and size of inclusions with which the sample was said to be seeded.

At the time of writing, the sample had been partially polished to the locations of some of the defects we found. The results indicate that we were able to detect the defects present in the portion of the sample examined. Moreover, all the cracks we detected were found at the prescribed locations. They were in reality thin disks of BN, a material used in the pressing operation, which is relatively harmless as far as its effect on fracture is concerned. Flat empty cracks do not occur in hot pressed Si₃N₄, and hence when "cracks" are detected by our acoustic method, they must be expected to be thin disks filled with a low acoustic impedance material such as BN. Hence, for hot pressed Si₃N₄, all the detected cracks are thin disks of BN.

In another example, we decided that one of the voids we detected was actually two voids separated by 50 µm in depth. This decision was made because two echoes of equal amplitude were detected with ~10 nsec separation in time. The actual defect is shown in Fig. 3b. The defects consist of two voids, as predicted, the two voids being connected by a porous region.
Some of the defects detected were of a new and unexpected type. They corresponded to a series of echoes with a short time separation between them (10-15 nsec), as would be expected from granular scattering. One such defect is shown in Fig. 5b. This defect turned out to consist of regions of concentrated MgO inclusions, a material used in the hot pressing operation to increase the density of the ceramic. It was thought that this material would disperse and not give rise to any problems; however, it obviously does not always do so. This unexpected defect, which was first detected clearly by acoustic techniques, is likely to have an important effect on the fracture characteristics of these ceramics.

There are a few more defects present in the samples, namely Si. We are presently awaiting the results of the polishing to compare with our acoustic measurements. At the present time, the polishing process has reached only one defect. The results obtained on this one defect are not encouraging, because the Si seed turned out to be porous, and of a larger size than had been predicted by our theory. This would be expected because the theory was based on the supposition that the defect was a sphere of Si. Here it was not spherical, nor was it pure Si. It remains to be seen whether the other Si seeds have changed in the same way.

**CONCLUSIONS**

We have developed two systems to locate and characterize defects in ceramics. We have used a set of signal processing schemes to improve the signal-to-noise (electronic noise and grain-scattering noise) ratio and to correct the signals received from defects. We believe that our theoretical and experimental techniques have not been fully tested yet, because the seeded samples we had were inferior to hot pressed, unseeded silicon nitride. However, we feel confident that our technique will work well on high quality samples of the type that should be used in real turbines. One difficulty is that artificially seeded materials always appear to be far worse in quality than the natural unseeded samples with naturally occurring defects present.

It appears from our initial results that the seeded defects which occur are not always spherical or simple in nature. This presumably will be the case with naturally occurring defects. Thus, although there are acoustic signatures associated with the different types of defects that are present, it will be necessary to develop further acoustic methods to provide a complete unequivocal quantitative identification of the size and type of all defects present. Naturally occurring inclusions of biphasic materials of awkward shape are the most difficult to identify. For a complete analysis, it will probably be necessary to resort to the imaging techniques which we are presently developing.
REFERENCES


8. J. M. Richardson, D. Cohen, Rockwell Science Center (Private Communication).


ACKNOWLEDGMENT

This work was supported by the Advanced Research Projects Agency under Rockwell International subcontract RI74-20773.
SUMMARY DISCUSSION
(P. Khuri-Yakub)

Wolfgang Sachse (Cornell University): One comment and a question. It's nice to see that someone else has found Weibull useful for classifying certain defects, but my question is: you said you made the diffraction correction. You mean to say that you found what is the diffraction of a sound wave as it leaves the buffer rod into the sample?

P. Khuri-Yakub: No. When you want to make a correction loss, if you're at high TS, and if you look at all the published data, you find it goes up to four or five.

Wolfgang Sachse: That's for a piston radiator?

P. Khuri-Yakub: That is a piston transducer, too.

Wolfgang Sachse: I'm not sure, but anyhow --

P. Khuri-Yakub: We just took it farther because we needed to get that right correction there.

Jim Rose (Univ. of Michigan): You cut off your theory of KA of 1. Does that imply in your data analysis you don't use the low-frequency information?

P. Khuri-Yakub: It depends on the size of the transducer (inaudible), the only reason why we did that was because we plotted on a log-log scale (inaudible).

# #