Ultrasonic Flaw Detection in Ceramics

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ULTRASONIC FLAW DETECTION IN CERAMICS

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The work I shall be describing this afternoon is a recently initiated joint study between Stanford University and the Science Center to determine the viability of ultrasonics as a failure prediction technique for structural ceramic materials.

To put this work in perspective, I thought it would be worthwhile spending a few moments at the start of the talk describing failure prediction techniques in ceramics, in general. In many ways they are similar to those employed for metals, but there are three or four key differences, and I think these should be emphasized.

The first point to realize is that the critical flaws are typically rather small. The reason they are small is that ceramics have a much lower toughness than comparable metallic systems. Specifically, fracture mechanics data for structural ceramic materials can be used to compute the size of the initial flaw that would lead to a failure (as a function of the stress level) assuming that 10,000 hours is the requisite component lifetime (Fig. 1). If the maximum tensile stress is assumed to be 300 MPa, then the critical flaws at room temperature range from 80 microns to about 30 microns. At higher temperatures, the critical flaws are even smaller, e.g., 10 microns for the best commercially available silica nitride at 1400°C.

The high temperature curve is dotted in the high stress regime because it has been calculated on the premise that the propagation of a pre-existing defect is analogous to that of the large crack used to obtain fracture mechanics data. However, when the flaws are on the same scale as the grains, microstructurally related slow crack can occur. In the extreme case, if the flaw is smaller than the grain size, the requisite toughness is the single crystal toughness; whereas, when the flaw is large, the toughness is given by the polycrystalline value.

The next problem arises because the slow crack growth exponents in ceramics are fairly large, which means that 90 percent of the time to failure is spent while the flaw is growing from its initial size to a size approximately 10 percent larger. There, it is indeed required to detect flaws essentially as small as indicated on Fig. 1.

The third feature is illustrated by Fig. 2; not all flaws are equally deleterious. For example, an inclusion with a lower elastic modulus and a lower thermal expansion coefficient than the matrix can be extremely deleterious; whereas an inclusion with a similar modulus and expansion coefficient to the matrix would be quite innocuous. Hence, it is not sufficient to determine the size of the defect; it is also required to determine something about its character. Otherwise, satisfactory components would be eliminated.

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These problems have, in the past, limited the application of ultrasonics to failure prediction, and alternate failure prediction methods have been tried first. The first technique is a statistical technique, which attempts to characterize the fracture statistics; recently a first principles theory has been developed for this purpose. The problem with fracture statistics for failure prediction derives from the large number of samples that have to be used to achieve a high confidence and a low failure probability. Also, the flaw population must be invariant from batch to batch; this is often an invalid assumption. The statistical method thus has major shortcomings.

Another approach which has been tried with much greater success is that of overload proof testing. With this approach, the component is subjected to a stress state that simulates the inservice stress state, but at a larger level than that to be experienced in service. Then, provided that it is unloaded under conditions where no additional crack growth can occur, there is an unequivocal assurance that the component will last for a specified time during service. But proof testing is limited to fairly simple geometries, and the approach cannot easily be applied to many real components. So, there is still a requirement to develop a direct flaw detection technique that is capable of detecting flaws in the pertinent size range. Of all the techniques that are available, we believe that ultrasonics has the greatest promise.

Backscattering calculations from spherical cavities in silicon nitride (Fig. 3) indicate that useful information about the size of the defect is obtained in the range $k \approx 1$, suggesting that frequencies in the range of 100 to 200 MHz are required. Having established that high frequencies are important, it is firstly required that transducers be available for this frequency range. A pertinent transducer configuration consists of zinc oxide chemically vapor deposited onto a sapphire buffer, (Fig. 4). These transducers have center frequencies of the order of 250 MHz and a bandwidth of 150 MHz. A short pulse is also required and the pulsing and the protection circuitry for this purpose have been developed.

The next question is whether these materials are too attenuating at these frequencies. A sampling of some data (Fig. 5) indicates that in the fine grain nearly fully dense structural ceramics, the attenuation seems to be fairly reasonable up to at least 200 MHz and probably up to even 400 MHz.

Figure 3. The backscatter cross section for spherical cavities in silicon nitride.

Figure 4. A schematic of the high frequency transducer system.

Figure 5. Attenuation data obtained for three silicon nitrides.
Having established that attenuation is reasonable, some calculations have been performed of the relative amplitude received at the transducer, as a function of frequency for typical defects in structural ceramics. From these it appears that, with the particular transducer developed in this study, it should be able to detect defects at least as small as 50 microns at frequencies at least as large as 100 MHz. These are approaching the important range.

These are just initial calculations, and experiments are needed to confirm these expectations. Initially, the depth resolution at high frequencies has been demonstrated for a 135 micron thick glass slide (Fig. 6). This indicates that resolutions at least as small as 30 microns could be possible with this technique. Next, defect studies have been performed on optically transparent magnesium oxide and on a silicon nitride block. Figure 7 shows a typical example of the reflection from small defects in silicon nitride. Note that they are well above the background. Hence, at this stage, the method looks promising, and we might indeed be able to use ultrasonics as a failure prediction technique in fine grained, fully dense ceramics; but, of course, there are two or three key steps to be explored before we can determine the range of applicability of this method.

Figure 6. A demonstration of the depth reduction at 200 MHz obtained on a glass slide.

Figure 7. The scattering obtained from several defect types in silicon nitride.
DISCUSSION

PROF. JOHN TIEN (Columbia University): One of the problems with structural ceramics is that they are not pure materials; additives must be used to enhance sintering ability and, unfortunately, these become segregated at grain boundaries and the thickness of these materials may control the creep properties or strength at high temperatures. Is there any hope for ultrasound to interrogate grain boundary films?

DR. EVANS: I do not believe that existing theory tells us anything about this interaction of ultrasound with grain boundary films. However, we are preparing a series of model systems based on PZT to look at attenuation effects.

DR. PAUL FLYNN (General Dynamics): Do you anticipate any problem in detecting flaw sizes of the size you are looking at here near or on the surfaces?

DR. EVANS: Gordon Kino and Lazlo Adler have some ideas on the use of surface waves to detect surface or near surface defects, and we will incorporate these in our program.

DR. OTTO BUCK (Rockwell Science Center): Do you have a homogeneous distribution of defects within your materials?

DR. EVANS: It depends very much on the materials. For the materials of greatest interest, the defects are large inclusions and large pores, and they are randomly distributed throughout the material. But their separation is many orders of magnitude larger than their diameter. So they should be identified on an individual basis.

MR. ROY SHARPE (Harwell Labs): It may be a bit heretical in an acoustic meeting like this, but isn't it possible to detect these flaws by something like radiography? It's possible to get resolutions of that order right now, quite easily.

DR. EVANS: Radiography was one of the first techniques tried for these materials because it was a well established technique that was capable, certainly, of detecting fairly dense inclusions (such as tungsten carbide) in the 100 micron range; but it was quite incapable of detecting many other inclusions (such as silicon and silicon carbide) smaller than a few millimeters. With microfocus x-rays, the resolution is superior and defects as small as 25 microns can be resolved, but again, many of the important inclusions do not produce a detectable contrast in the appropriate (10-100 micron) size range.

PROF. GORDON KINO (Stanford University): I think the other problem with radiography is that when you get down to the root of the turbine blade, access is difficult, and I think the use of surface waves might be much easier.

PROF. TIEN: Thank you, Tony.