APPLICATIONS OF ULTRASONIC MICROSCOPY
TO THE EVALUATION OF CERAMIC MATERIALS

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INTRODUCTION

Modern ceramic materials offer many attractive physical and mechanical properties for a wide and rapidly growing range of industrial applications. But the criticality of many of those applications sets technical challenges to the materials and inspection industries. NDE needs may be classified into three groups: controls on manufacturing processes, characterization of material properties, and detection of defects. Good process control techniques, important to all industries, assume even more importance for many ceramics, especially structural ceramics, because of specific characteristics such as a relatively low fracture-toughness. Apart from detection of discrete defects, techniques will also be needed to monitor uniformity of properties, and to inspect for bond strength, for example. Characterization of physical, chemical, and thermo-mechanical properties will use many of the orthodox techniques of the materials scientist, modified as necessary to accommodate generic differences from other materials. Flaw population studies, and the detection of defects -- cracks, voids, inclusions, delaminations, etc. -- to ensure material and product integrity, will require both unusually high sensitivity and high reliability, due to the unusually small size of defects (of the order of 25 μm diameter) which may be fracture-critical.

SUITABLE INSPECTION TECHNIQUES

Understanding of the capabilities and limitations of NDE techniques allows preliminary selection of promising candidate technologies to meet the challenges posed by ceramics. One such technique is infrared imaging: by monitoring thermal radiation emitted by surfaces of interest, with a scanning infrared-sensitive camera, surface and near-surface anomalies in thermal conductivity are revealed. From such data, variations in thickness and disbonds between layers may be deduced; these techniques are
well-suited to examination of ceramic layers on metallic substrates, such as the thermal-barrier coatings used in the aircraft engine industry.

Conventional radiography or radioscopy, using a standard-size X-ray source (with typical diameter of about 1.5mm), is of only marginal interest, since the best achievable resolution (approximately 2 to 4 line-pairs/mm (lp/mm) is barely adequate to allow detection of the defects of concern. However, using high-resolution digital sensors, even with conventional sources, computed tomography (CT) systems have been shown to have better sensitivity than conventional X-ray systems for detection of some conditions of interest, such as small density variations. They also offer a unique capability for viewing internal structure, measuring wall-thicknesses, etc., otherwise obtainable only with destructive examination methods.

Microfocus X-ray systems, with a source diameter reduced to about 10um, operated with film, fluoroscope or digital sensors, offer resolutions of approximately 20 lp/mm, allowing detection of defects about 10um in diameter (with the actual detectability depending on the contrast in X-ray absorptivities between the defect and the surrounding material, as well as the thickness of the material). This high resolution, coupled with the ready availability of microfocus sources, make this technique of major significance for evaluation of ceramics. Although they are not yet commercially available, the few existing microfocus CT systems offer a prospect of even wider applicability for this technique.

The only other NDE technique which is of major relevance to the inspection of ceramics is ultrasonic (often called acoustic) microscopy: the use of tightly-focused high-frequency sound beams to form images of the point-to-point reaction of material to periodic stress waves. It too can offer a high sensitivity for the detection of small defects, and is often a complementary technique to X-ray.

ULTRASONIC MICROSCOPY

Sokolov [1], of the University of Leningrad, appears to have been the first to recognise, in 1936, that sound waves at frequencies of about 3GHz could produce images with resolution comparable to that of optical microscopes. At the time there was no instrumentation available to permit implementation of his idea, and it was not until 23 years later that the first operating system was demonstrated [2].

In the 1970’s the first practicable high-frequency systems were produced; both were through-transmission systems: Kessler [3] and his co-workers utilized a single stationary transducer generating plane waves, with a scanning laser system to detect minute surface displacements due to the sound beam; Quate [4] and his co-workers developed a system based on a pair of stationary confocal lenses, moving the sample through the sound beam. The former systems are now conveniently referred to [5] as "SLAM’s" (Scanning Laser Acoustic Microscopes), and the latter as "SAM’s" (Scanning Acoustic Microscopes). The frequency of operation of a SLAM is typically 100 to 500 MHz, and of the SAM, 100 to 3000 MHz, although both systems have been operated at frequencies outside these ranges. SAM’s have also been built to operate in a reflection (pulse-echo) mode, using a single transducer; for both transmission and reflection versions, scannable thicknesses are very restricted, due both to the lens design requirements and to the high attenuation of most materials (including the necessary couplants) at these high frequencies; reflection SAM’s are categorized as surface inspection devices [5]. A SLAM is somewhat less restricted, since it does not use the short focal-length and -depth lenses of the SAM, and because it is possible to eliminate all
or most of the couplant, but images of multi-layered objects can be complicated since they represent the entire thickness of the sample.

In 1979, Kessler and Yuhas [6] wrote that "acoustic microscopes use frequencies ranging from 100-3000 MHz". The upper limit has now been passed, using liquid helium as a couplant to reduce attenuation, allowing frequencies up to 8 GHz, and it is now recognized that the lower limit was arbitrary and unrealistic. Gilmore [7,8] appears to have been the first to realize that it was in the frequency region below 100 MHz that most practical industrial applications lay, and that "ultrasonic microscopy" might better be defined as the formation of images using sound beams with widths comparable to or smaller than the features being imaged, independent of frequency. In reality, there is a continuum between lower frequency ultrasonic C-scan images and the ultra-high frequency SAM images.

The work described in this paper has all been performed using a GE NDE Systems & Services's "CAUM 3000" (computer assisted ultrasonic microscope) of the Gilmore type. It uses a single focused transducer, which is scanned relative to a fixed sample. Images are formed from peak signals occurring at specific electronically-gated depths, using an 8-bit (256 level) detector; the transducer is fired on a square grid, with spacing as small as 5μm. Scanned areas can be much larger than for typical SLAM or SAM systems, and the depth of penetration is much less limited. This type of microscope is now often referred to [5] as a C-SAM (C-mode Scanning Acoustic Microscope) - although there is more convenience than justification for distinguishing it from the SAM in this way: both are C-scan systems.

**BEAM PROPERTIES**

The diffraction-limited focal zone dimensions of an ultrasonic beam in water, with wavelength $\lambda_v$, focal length $F_w$, diameter $D$, and can be characterized by the following formulae:

$$\text{focal zone depth} = k_1 \lambda_v (F_w/D)^2,$$

$$\text{focal zone diameter} = k_2 \lambda_v (F_w/D),$$

where the values of $k_1$ and $k_2$ depend on the decrement below peak intensity used to define the zone of interest; if we select -17 dB, $k_1 = 16$ and $k_2 = 2.4$; the focal zone diameter is then equivalent to the full width of the principal lobe of the sound beam. Criteria used in acquiring data for most of the accompanying images were a) to set the pulse index equal to the -1 dB beam width ($k_2 = 0.4$); and b) to gate a layer defined by the -3 dB focal zone depth ($k_1 = 4$).

![Figure 1. GE's "CAUM" Computer Assisted Ultrasonic Microscope](image)
Detectability of defects is determined by their size relative to the beam diameter; by the contrast between their acoustic impedance and that of the surrounding material; and by other factors such as their shape and orientation relative to the sound beam. We may expect that a void considerably smaller than the full sound beam width might be detectable, for example, provided that its echo signal is above noise, whereas an inclusion would be less detectable. Table I summarizes beam properties for several typical CAUM/C-SAM transducers; note that to a first approximation, the beam diameter is the same [8] in water and solids, but the focal depth is shortened in solid in proportion to the ratio of the sound velocities in these two materials.

It may be seen that wide-aperture (small F_/D) 50 MHz transducers have beam diameters offering a good prospect of detecting material features of the order of 20 μm diameter, making them suitable for inspection of structural ceramics. However, it should be noted that many ceramics are highly attenuating at these frequencies, making choice of high-performance low-noise instrumentation essential to the inspection of significant thicknesses of ceramic materials.

RESOLUTION

Kino [9] has offered a modified Rayleigh criterion to be applied to scanned imaging systems: two objects can be resolved if their separation is at least 1.03 (AF_/D). In water, typical values of F_/D are between 0.5 and 15; as shown in Table I, use of a wide-aperture 50 MHz transducer can result in resolutions of the order of 25 μm. In solids, the minimum value of F_/D may be limited by total reflection, at the water/solid interface, of sound coming from the peripheral regions of the transducer. For example, for silicon nitride, F_/D cannot be smaller than 3.4 for compressional waves, or smaller than 1.9 for shear waves. The effects of this limitation are indicated in Table I (for compressional waves; restrictions are less severe for shear waves); although this limits resolution of closely spaced defects, it does not limit their detection.

LIMITATIONS AND REQUIREMENTS

The advantages of ultrasonic microscopy over conventional ultrasonic inspection lie in the much higher lateral and depth-wise resolution, which allow detection of exceptionally small discontinuities; the pinpointing of their location; and the formation of realistically representative images of all but the smallest features, permitting greatly improved interpretation of the data and evaluation of the material properties. These advantages are not obtained without limitations: these are principally concerned with attenuation effects, depth of field, penetration, and the need for large data files. Attenuation in both the couplant and the sample may represent significant limits; indeed, above about 50 MHz, high-frequency attenuation in the couplant reduces the effective frequency of any but very short focal-length transducers to well below their nominal frequency.

Both depth of field and penetration are quite limited by comparison with lower-frequency inspections, an unavoidable consequence of the use of high frequencies and tightly focused sound beams; the ratio F_/D must be kept small to achieve a small beam-width and the best resolution, but transducer diameters are necessarily limited by practical considerations, thus requiring a short focal length. In practice, this means that ultrasonic microscopes of the CAUM/C-SAM type find ideal application to inspection of surfaces (bond-surfaces, for example, or the examination of ceramic surfaces for machining or handling damage). However, volumetric
### TABLE I. EXAMPLES OF BEAM AND RESOLUTION PARAMETERS FOR SCANNING ULTRASONIC MICROSCOPES

<table>
<thead>
<tr>
<th>FREQUENCY (MHz)</th>
<th>5.0</th>
<th>10.0</th>
<th>20.0</th>
<th>50.0</th>
<th>50.0</th>
<th>50.0</th>
<th>50.0</th>
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<tbody>
<tr>
<td>LENS RADIUS [D/2] (mm)</td>
<td>6.35</td>
<td>3.18</td>
<td>3.18</td>
<td>3.18</td>
<td>3.18</td>
<td>3.18</td>
<td>3.18</td>
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<tr>
<td>FOCAL LENGTH [F_w] (mm) Water</td>
<td>50.8</td>
<td>25.4</td>
<td>25.4</td>
<td>25.4</td>
<td>19.1</td>
<td>12.7</td>
<td>5.1</td>
</tr>
<tr>
<td>&quot;f&quot; NUMBER [F_w/D]</td>
<td>4.0</td>
<td>4.0</td>
<td>4.0</td>
<td>4.0</td>
<td>3.0</td>
<td>2.0</td>
<td>0.8</td>
</tr>
<tr>
<td>WAVELENGTH [λ] (μm) Water [a]</td>
<td>296</td>
<td>148</td>
<td>74</td>
<td>29.6</td>
<td>29.6</td>
<td>29.6</td>
<td>29.6</td>
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<tr>
<td></td>
<td>Silicon Dioxide [b]</td>
<td>1192</td>
<td>596</td>
<td>298</td>
<td>119</td>
<td>119</td>
<td>119</td>
</tr>
<tr>
<td></td>
<td>Silicon Nitride [c]</td>
<td>2040</td>
<td>1020</td>
<td>510</td>
<td>204</td>
<td>204</td>
<td>204</td>
</tr>
<tr>
<td></td>
<td>Alumina [d]</td>
<td>2200</td>
<td>1100</td>
<td>550</td>
<td>220</td>
<td>220</td>
<td>220</td>
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<tr>
<td>FOCAL ZONE DIAMETER (μm) -1 dB, water, ceramic</td>
<td>474</td>
<td>237</td>
<td>118</td>
<td>47.4</td>
<td>35.6</td>
<td>23.7</td>
<td>9.5</td>
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<td></td>
<td>-17 dB, water, ceramic</td>
<td>2844</td>
<td>1422</td>
<td>711</td>
<td>284</td>
<td>213</td>
<td>142</td>
</tr>
<tr>
<td>FOCAL ZONE DEPTH (μm) -3 dB, Water</td>
<td>18944</td>
<td>9472</td>
<td>4736</td>
<td>1894</td>
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<td>474</td>
<td>76</td>
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<td>-3 dB, Silicon Dioxide</td>
<td>4704</td>
<td>2352</td>
<td>1176</td>
<td>470</td>
<td>353</td>
<td>237*</td>
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<tr>
<td></td>
<td>-3 dB, Silicon Nitride</td>
<td>2749</td>
<td>1374</td>
<td>687</td>
<td>275</td>
<td>237*</td>
<td>237*</td>
</tr>
<tr>
<td></td>
<td>-3 dB, Alumina</td>
<td>2549</td>
<td>1274</td>
<td>637</td>
<td>255</td>
<td>237*</td>
<td>237*</td>
</tr>
<tr>
<td>RESOLUTION (μm) [compressional wave] Water</td>
<td>1220</td>
<td>610</td>
<td>305</td>
<td>122</td>
<td>92</td>
<td>61</td>
<td>24</td>
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<tr>
<td></td>
<td>Silicon Dioxide</td>
<td>1220</td>
<td>610</td>
<td>305</td>
<td>122</td>
<td>92</td>
<td>61*</td>
</tr>
<tr>
<td></td>
<td>Silicon Nitride</td>
<td>1220</td>
<td>610</td>
<td>305</td>
<td>122</td>
<td>105*</td>
<td>105*</td>
</tr>
<tr>
<td></td>
<td>Alumina</td>
<td>1220</td>
<td>610</td>
<td>305</td>
<td>122</td>
<td>113*</td>
<td>113*</td>
</tr>
</tbody>
</table>

Compressional wave velocity: (a) 1.48; (b) 5.96; (c) 10.2; (d) 11.0 mm/μs
* Aperture limited by total reflection

Inspections are also quite practicable for fairly thin samples, or for restricted areas of thicker samples, such as weld zones. For large volumes of material, high-resolution inspection is still possible, but the inspection time may become large; note, for example, that the volume of material defined by our recommended (-3 dB depth-of-field) gating and (-1 dB beam-width) pulse-spacing is 134 to 248 times smaller for the most tightly focused 50 MHz transducer described by Table I than for the 5 MHz transducer in the same Table, (for the three ceramics listed therein).

If it is necessary to inspect to depths greater than those attainable with the highest near-surface resolution, compromise is possible, sacrificing some resolution for greater penetration. Consideration of the costs of inspection may also suggest relaxing the -3 dB depth-of-field or the -1 dB pulse-spacing criteria, provided that this does not cause unacceptable loss in inspection reliability. The high pulse density needed for high-reliability high-resolution imaging requires a computer with the capability of storing and processing quite large data files: for example, with a 25 μm pulse spacing, each 625 mm scanned requires 1 Mb of storage. Our experience has been that the great majority of practical laboratory applications of the ultrasonic microscope require files of approximately 0.5 to 10 Mb, but this limit can easily be exceeded when examining industrial components.
IMAGING MODES

The CAUM ultrasonic microscope can be operated in a variety of imaging modes, allowing a choice to be made for specific applications. For most applications, a normal-incidence sound beam is used, with the gate set to isolate compressional wave images of front-surface, interior, or backwall detail, or any combination thereof. Non-normal-incidence beams may also be used to give compressional wave images of front surfaces, or either compressional or shear wave images of subsurface features; it has been suggested by Gilmore (8) that the latter give the most reliable information about the integrity of bonded surfaces. For both compressional and shear modes of inspection, the focal point of the transducer can be located at the depth of greatest interest, to enhance sensitivity and resolution at that depth. If desired, the same surface may be scanned several times, with changes in the focus and gate settings.

Finally, if a wide-aperture lens is used, surface wave images may be formed, in effect converting the CAUM from a reflection microscope to a transmission microscope. This requires a transducer large enough that it may be considered to contain annular regions generating rays which will strike the inspection surface at angles equal to the Rayleigh critical angle. A converging cone of rays thus strike the surface, are mode-converted to surface waves, and propagate across the surface, generating leaky waves which are detected by the opposite element of the same annular region which generated them. Not only must the transducer have adequate diameter, but it must be "defocussed" by pushing the focal point sufficiently far subsurface for the compressional wave, directly reflected from the front surface, and the surface wave to be time-resolved, as shown in Figure 2. In this cylindrically convergent surface wave mode, discontinuities of one-third to one full surface wavelength in diameter can cause -3 dB to -20 dB changes in the received signal amplitude; furthermore, surface and near-surface features can be detected with equal probability regardless of their orientation, unlike conventional reflection or transmission surface wave imaging (10).

EXAMPLES OF ULTRASONIC MICROSCOPE IMAGES

Images accompanying this paper illustrate a small sample of the variety of ceramic materials which have been examined, and have been selected to demonstrate the typical imaging quality attainable; the original images are all in colour, and are presented to the operator at a considerably larger magnification than is possible here.
Figure 4. Compressional wave echo from front surface of silicon nitride (magnification: 2.9)

Figure 5. Time-resolved surface wave echo from silicon nitride (magnification: 3.5)

Figure 6. Configuration for test of silicon carbide washers

Figure 7. Backscattered signals from silicon carbide washers (magnification: 3.5)

Figure 8. Compressional wave echoes from interior structure of aluminum oxide (magnification: 3.5)

Figure 9. Compressional wave echoes from far-wall laser damage sites in aluminum oxide (magnification: 3.5)

Figure 4 was obtained by focussing the transducer on the front surface of a small piece of ground and polished silicon nitride. It had been used as a fracture test specimen, and then loaded at one point with a conical indenter. The image of a crack induced by the latter may be seen at top center, and to the right of this is a network of fine cracks associated with the fracture surface, which is to the right. A letter "E" is visible; this was written in thin indelible ink to label the sample. The overall pattern of horizontal lines comes from the grinder. Apart from the label, none of these features was visible to the unaided eye. Figure 5 is of the same specimen, but obtained from the time-
resolved surface wave technique. Visibility of all these surface features is enhanced, but the images are broadened by the entry-circle diameter of the surface waves. Figure 6 indicates the set-up used to test for surface wear on a group of three silicon carbide washers. The transducer was focused at a distance somewhat greater than the point of nearest approach of the washers, and scanned in a plane. Echoes from the unworn washers at the left and center, shown in Figure 7, are obtained only by near-specular reflection. The worn washer at the right back-scatters sound at incident angles up to about 10°; there is also evidence of non-uniform wear in an axial direction.

Figures 8 and 9 result from examination of samples of sintered aluminum oxide bars approximately 2 to 3 mm thick. Figure 8 is an image of internal particles, taken by gating to select only echoes coming from depths between 2.62 and 2.96 mm, and focusing the transducer in the middle of this depth range. (The subsurface focus causes a loss in signal amplitude close to the edges of the bar, which may be seen.) Figure 9 comes from a bar 2.3 mm thick, which had been deliberately damaged on one surface with a laser. The transducer was placed on the opposite side of the bar, and focused on the far wall. The gate was set to just avoid including echoes from the far wall; clear images of each of the shallow damage sites were obtained. This image took full advantage of the low-noise GE instrumentation: the signals were barely detectable; the sample had proved unspectable at this 50 MHz frequency with other instrumentation.

SUMMARY

Worldwide interest in the use of ceramics and ceramic composites as key load-bearing components for gas turbine, aerospace, and other advanced engineering applications, has led to the need for inspection techniques capable of detecting unusually small structural anomalies. One of the few techniques affording such a capability is ultrasonic microscopy, an imaging technique using tightly-focused sound beams at frequencies typically up to 50 MHz.

Capabilities and limitations of all types of ultrasonic microscopes have been outlined to aid in selection of the right type for future applications. We suggest, though, that ultrasonic microscopes of the C-SAM type (such as GE's CAUM) are vital to the examination for structural integrity of a variety of ceramics. They have sufficient sensitivity and resolution to detect and image anomalies in a size range of significance, and can be obtained with manipulation versatility and data handling capabilities compatible with industrial components. At lower frequencies, resolution is inadequate; higher-frequency microscopes of the SAM type generally are too limited in penetration and scanned area to be useful for examining most industrial components. The examples shown are drawn from preliminary feasibility demonstrations on material from a variety of sources, and are intended only to illustrate part of the range of applications. We expect to show more detail in future reports.

REFERENCES

1. S.Sokolov, USSR Patent No. 49 (August 31, 1936)


