PHOTOACOUSTIC MICROSCOPY OF CERAMICS USING LASER HETERODYNE DETECTION

Harry I. Ringermacher and Carol A. Kittredge

United Technologies Research Center
East Hartford, Connecticut 06108

INTRODUCTION

In recent years a variety of thermoacoustic techniques have been used to image surface and near surface features in ceramics. These include early gas cell methods [1] as well as the Scanning Electron Acoustic Microscopy (SEAM) technique [2] and more recently the Mirage or Optical Beam Deflection (OBD) methods [3,4]. In the gas cell and Mirage methods, the effect of the outgoing thermal wave on the air boundary at the sample surface is sensed, respectively, by a microphone and by a laser beam. In SEAM the specimen vibration or acoustic wave is sensed directly by a contacting transducer. The gas cell and Mirage methods generate pure thermal images but require long times to generate useful images of areas a few millimeters square. SEAM, on the other hand, is a high signal/noise technique due to the exceptional sensitivity of piezoelectrics and thus requires shorter imaging times. However, SEAM is sensitive to both the thermal wave signal and the local mechanical response of the specimen to the thermal wave and convolutes the two responses. The author has recently demonstrated that the entire image can be dominated by the mechanical response alone [5]. Thus SEAM image interpretation is considerably clearer for such cases. This thermomechanical mechanism is now fairly well understood and its analysis will be presented elsewhere. SEAM is thus an excellent method for imaging ceramics. Using Coordinate Modulation (CM) with SEAM 5mm x 5mm areas of ceramics have been imaged for surface and subsurface defects in 2 minutes. CM requires the electron beam to be dithered a small amount instead of being intensity modulated, the usual approach. This increases image contrast and will be described later.

SEAM has two disadvantages. System preparation in an electron microscope requires an inordinate amount of time and the available specimen space and positioning is limited. Thus it appears desirable to duplicate SEAM using an open laser system while sensing the specimen vibration and/or thermal wave displacement itself to achieve the high signal/noise ratio. Previous work [6] using piezoelectric detection on the specimen has achieved rapid imaging. However, direct contact was required.

In the present work advances in laser interferometry have been applied to noncontact detection of vibration for photoacoustics (PA) thus in principle eliminating the last hurdle towards an ideal flexible imaging system. Many interferometric techniques are available for vibration sensing. References to interferometric techniques abound [7]. All possess, under ideal circumstances, similar sensitivites. Differences arise primarily in the customization of the interferometer for a particular frequency regime. Thus displacement-sensing Michelson interferometry is more suitable for detection of very high frequencies, e.g. ultrasound, since its response is centered about a DC output and is thus very susceptible to low frequency noise. Heterodyne interferometry is more desirable for lower frequency work since an rf carrier is introduced upon which the desired lower frequency signal is impressed thus raising it above the 1/f noise region. Of the various optical heterodyne techniques available, we have chosen Doppler heterodyne interferometry because of its great insensitivity to surface condition and the availability of excellent low noise FM discriminator technology. This technique outputs a signal proportional to the Doppler shift of the return light and hence is a measure of the surface velocity of the specimen. It is therefore very insensitive to the return light intensity requiring only enough to "quiet" the FM discriminator. It has been used with resulting similar signal/noise responses on specular, diffusing and dark surfaces and therefore seems ideal for application to ceramics.

COORDINATE MODULATION

Coordinate modulation has been described in detail previously [5,6] so only a brief summary will suffice. In CM the thermoacoustic source coordinate, rather than its intensity, is modulated. Thus if a laser beam is scanned, for example in the X direction in raster fashion, the beam may be dithered a small amount in the scan direction. The modulation reference signal may be either a square wave or a sine wave, each having specific advantages. The thermoacoustic response to such a modulation source is a null background signal when the source dithers over a locally homogeneous region and a net acoustic output when the source dithers between two local thermally inhomogeneous regions. Since, for typical thermoacoustic frequencies, the acoustic wavelength is much greater than the modulation spacing, the two point acoustic sources cancel in this near field when they both see thermally identical regions. When the two points see thermally differing regions, although the acoustic signals will still be out of phase, they will generate different amplitudes resulting in a net acoustic output. This has been verified to high cancellation levels by the author. This results in exceptional contrast since one is now measuring a small signal against a null background rather than against a constant, large, background with the usual intensity modulation.

A direct 6 dB signal/noise has also been observed by the author when using CM in comparison to AM with all other parameters fixed. This can be simply explained from making full use of the available laser beam rather than losing half its power during chopping and is achieved only when the peak-to-peak modulation amplitude is adjusted to be on the order of the dimension of the response to be modulated (the defect dimension if it is thermally shallow). If modulation is increased much beyond this point, image modulation broadening will occur. Excessive over modulation can actually result in diminished signal/noise. These effects are more fully discussed in [6] with regard to application in thermoacoustics and are in fact well understood and used in other fields such as magnetic resonance for spectral lineshape analysis.

PHOTOACOUSTIC SYSTEM

Figure 1 shows the experimental arrangement of the laser heating source together with the Laser Heterodyne Interferometer (LHI) detector. The Acousto-Optic Deflector is a Matsushita EFL-D250. It is modulated at the desired photoacoustic frequency up to 100 kHz resulting in a dithered beam whose peak-to-peak separation at the target is dependent on the optics between the target and the AOD. The present system allows a modulation separation of up to 0.5 mm while maintaining a 7 μm focal spot. The modulated beam is then raster scanned over the specimen with General Scanning galvanometer mirrors at a rate of around 4mm/sec.

A typical image coverage would be a 4mm x 4mm with 10 μm resolution (400 point scan line). Such an image would take 400 seconds for 400 lines. However when lower image quality was acceptable (200 x 200) or when signal/noise permitted faster scan rates, this time was readily reduced to 120 seconds for modulation frequencies above 300 Hz. The scanning is IEEE 488 controlled from a PDP11/24.

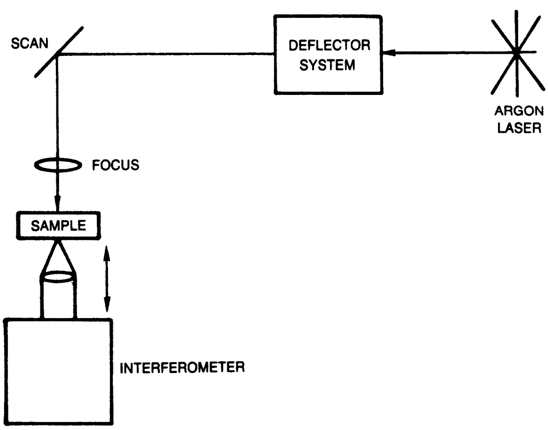

Fig. 1. Photoacoustic system

The specimen is held rigidly in position at its edge. The LHI is focused almost anywhere on the rear of the specimen except near an edge where the vibration must vanish due to the edge mounting boundary conditions. It could also have been focused on the front.

Figure 2 shows the detection electronics. The LHI output is an rf signal at the desired heterodyned carrier frequency. Impressed upon this carrier is the small frequency deviation, varying positive to negative at the modulation rate, arising from the Doppler shift. The carrier plus FM signal is injected into a low-noise, custom designed, FM discriminator where it is converted to an AM signal which is sent to a lock-in amplifier referenced to the modulation frequency.

The interferometer arrangement is shown in Fig. 3. This interferometer is optically heterodyned with both reference and probe arm beams up and down-shifted respectively in frequency by Intra-Action AOMs. The polarizing beam splitter, PBS1, controls the relative intensity between the two arms which must then include additional optics (PBS2 and a quarter-wave plate) for efficiency. The return probe light with the Doppler signal impressed upon it is interfered with the reference beam on a fast pin diode, D. Initial work with this interferometer used 60 MHz and 40 MHz for the reference and probe arms. The carrier at 100 MHz was sent to a commercial FM radio tuner. Although this worked well, the discriminator signal/noise ratio was deemed inadequate and a custom designed limiter-discriminator was used with the 20 MHz carrier created by utilizing the upshifted orders of the AOMS.

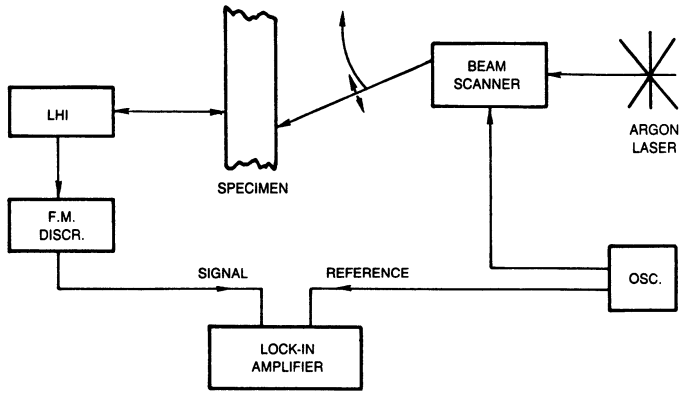

Fig. 2. Detection electronics

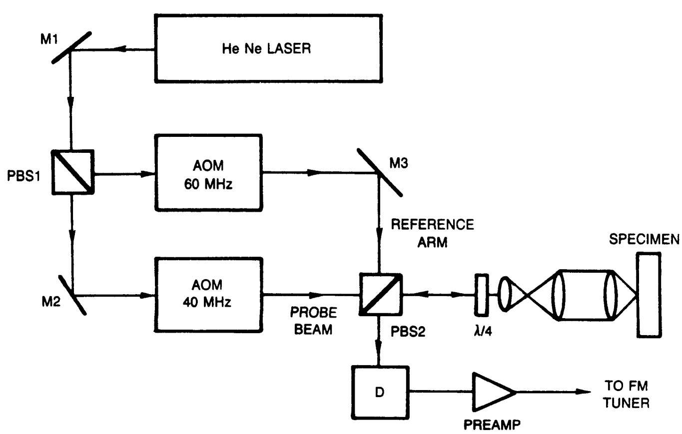

Fig. 3. Laser Doppler heterodyne interferometer

The Doppler frequency deviation is given simply by the Doppler shift formula for light of wavelength λ reflected from a target at velocity V,

$$\Delta f_D = \frac{2V}{\lambda} .$$

The discriminator produces a voltage proportional to the deviation in Hertz. The calibration constant in units of volts/Hz can be determined by injecting into the discriminator a known deviation at the carrier frequency from a frequency synthesizer with FM capabilities. From the above formula and the calibration constant, the actual specimen surface velocity can be determined and hence the displacement for a given PA excitation frequency.

The displacement sensitivity of this interferometer is frequency dependent and has been measured to be a constant 0.0003 Å in a 1 Hz bandwidth for target frequencies above 8 kHz. A proportionately decreasing sensitivity is expected and observed for lower frequencies down to 50 Hz. At 300 Hz, the sensitivity falls to 0.01 Å.

THERMOACOUSTIC IMAGING RESULTS

Figure 4 shows thermoacoustic images for a 500 µm diameter void whose top is located 300 µm below the surface of a SiC ceramic sample. Figure 4a is a standard Scanning Electron Microscope surface image of the defect area showing spider cracks resulting from stresses following the artificial void implantation and specimen densification. Figure 4b is the thermoacoustic image generated by SEAM [5] using a piezoelectric transducer for the thermoacoustic detector. Figure 4c shows the corresponding image produced by the present PA system. The decreased image sharpness is due to the 100 µm laser spot size and 125 µm beam modulation (at 1250 Hz). The thermal diffusion length in SiC at this frequency is approximately 135 µm. The general features present in the SEAM image are visible in the PA image. The cracks are no longer visible because of the above-mentioned sharpness loss. One point of interest is the more quantitative nature of the PA image. A grey-scale calibration in units of Å has been provided since the heterodyne interferometer is self-calibrated. The displacement is indicative of the entire specimen deflection from center to edge in response to the laser "hot spot" thermal expansion. This information could be used together with knowledge of the peak hot spot temperature and modeling to evaluate thermomechanical properties such as the CTE or the modulus.

FUTURE RESEARCH AND CONCLUSIONS

As has been stated, the concept pursued in this program has been to combine the excellent characteristics and control of SEAM imaging with the capability of remote laser detection approaching the sensitivity of piezoelectrics. This goal is close to being achievable with the present system. One particular direction has been the evaluation of ceramic coatings on carbon/carbon (C/C) composites. The coatings can be chemical vapor deposited (CVD) in which case their surfaces are extremely rough, consisting of randomly oriented crystallites. SEAM imaging in this program on such materials has been successful. Figure 5a shows a SEM photo of the crystal-

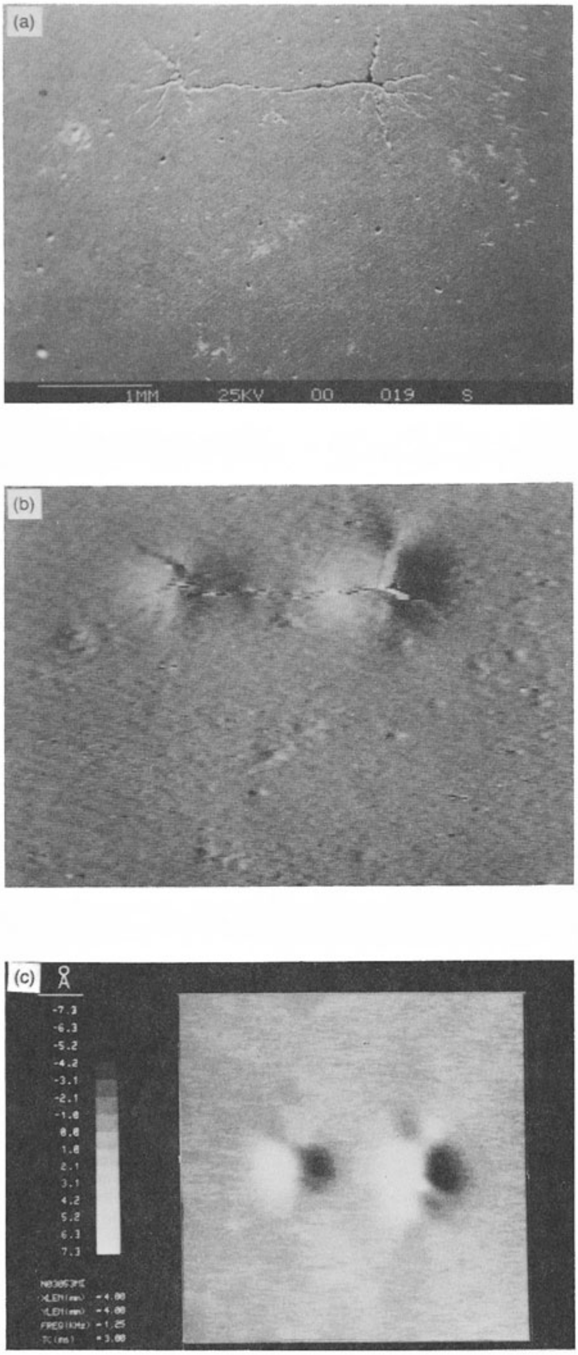

Fig. 4. Thermoacoustic images of 500μm subsurface voids in SiC: (a) SEM photo; (b) SEAM; (c) LLPAM

line surface of a silicon nitride CVD coated C/C composite. Cracking is barely visible within the contrast produced by the crystallites. The SEAM thermoacoustic image of the same area shows the cracking and at the same time has reduced the contrast arising strictly from geometric contour. This was possible using the depth-phasing characteristics of thermoacoustic imaging together with the choice of coordinate modulation characteristics (phase and amplitude). A 600 Hz modulation frequency was used resulting in a thermal diffusion length of 125 μm. The beam modulation width at the specimen surface was 2 μm.

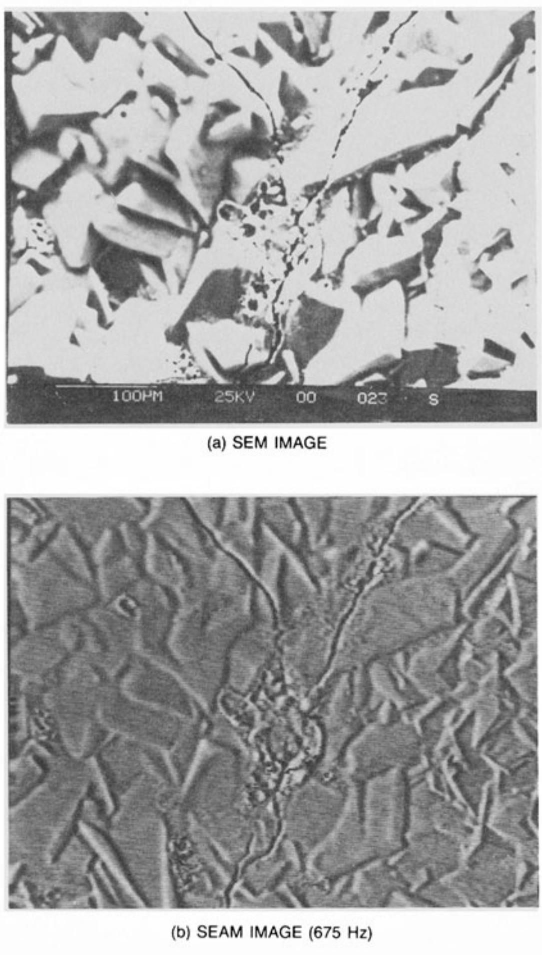

Fig. 5. SEAM thermoacoustic image of CVD silicon nitride on C/C substrate

This type of surface poses problems for most laser thermal imaging sensing methods. It is hoped that use of Doppler interferometry will reduce such surface effects for detection.

In summary, Doppler heterodyne interferometry has been used successfully for subsurface thermoacoustic imaging in ceramic materials. It has proven to be relatively insensitive to surface conditions by permitting a return light intensity variation of as much as 30 dB while retaining a constant image signal/noise ratio. This is due to the method's reliance only on the Doppler frequency shift of the return light. At the same time the technique has demonstrated a sensitivity of up to 0.0003 Å in a 1 Hz bandwidth which is suitable for a rapid scanning capability as demonstrated. This sensitivity can be expected to increase by an order of magnitude as further noise source problems are pursued.

ACKNOWLEDGEMENT

This work was supported in part by DARPA, Contract No. N62269-84-C-0217. The collaboration of Dr.James Strife in providing specimens and specimen preparation in this program is gratefully acknowledged.

REFERENCES

1. Y. H. Wong, R. L. Thomas, and G. F. Hawkins, Appl. Phys. Lett. 32, 538 (1978).

2. E. Brandis and A. Rosencwaig, Appl. Phys. Lett. 37, 98 (1980).

3. A. C. Boccara, D. Fournier and J. Badoz, Appl. Phys. Lett. 36, 130 (1980).

4. J. C. Murphy and L. C. Aamodt, Appl. Phys. Lett. 38, 196, (1981).

5. H. I. Ringermacher and L. Jackman, Review of Progress in Quantitative Nondestructive Evaluation, Vol. 5A, p. 567, 1986.

6. H. I. Ringermacher, IEEE Ultrasonics Symposium, IEEE Cat. #82CH1823-4, Vol. 2, p. 576, 1982.

7. An excellent discussion is presented by: R. M. De La Rue, etal, "Acoustic-Surface-Wave Amplitude and Phase Measurements using Laser Probes," Proc. IEEE, Vol. 119, No. 2, Feb. 1972.

DISCUSSION

From the Floor: Could you give us a little more information on the depth of capability? In the beginning, you sort of said it was limited by the thermal. Do you have any results that show that you could look at things quite deep?

Mr. Harry Ringermacher, United Technologies: If you are talking about the groove, it does appear to be so, but actually, when you get into an analysis of what I was seeing, what the contrast mechanism is on that long groove, it turns out the contrast mechanism is not thermal at all. It's mechanical.

What you are seeing, literally, is an image of the variation of the local stiffness of the plate as the hot spot scans across. So as a result, you are sensitive to quite deep defects if those defects affect the local mechanical properties of the specimen. So in that image, the far end of that groove is 26 mills below the surface, 600 or 700 microns, but the thermal diffusion length is only 25 microns, so there's no way a thermal wave could penetrate.

Typically, I work with frequencies as low as 100 Hertz, and in silicon nitride, that gives you about a couple of hundred micron penetration depth. In silicon carbide, it gives you perhaps a 400 micron penetration depth.

From the Floor: But by imaging, your receptor rays are on the front side. Actually, it doesn't have to go -

Mr. Ringermacher: No, it can be anywhere. You'll see the same signals. It doesn't matter, because the bending is very nearly the same on the front as on the back. As the specimen gets thicker, that bending signal decreases, too. So there's a limitation on when and where you can use that kind of detection. If you go and look at the hot spot itself, you then limit yourself, in thick specimens, to pure thermal imaging.

Mr. John Gospels, Boeing Aerospace Company: As a rule of thumb, what would you say the limits are as far as thickness?

Mr. Ringermacher: As a rule of thumb, the resolution of the object you are trying to image is on the order of the thermal diffusion length, and the depth. They are all about the same number. So if I've got 25-micron thermal diffusion length and I look 25 microns into a piece of some material, you can resolve a 25 micron defect.

Mr. Achenbach, Northwestern: Is some of the energy converted, then, to acoustic energy which interacts with something that is deeper than thermal wave length?

Mr. Ringermacher: Yes. It's actually very simple. Think of a thick specimen with a little hot spot on the surface. You are causing the surface to expand, but the rest of the specimen is cool, so it goes into compression. And if you periodically vary the hot spot, then the whole specimen is vibrating.

Mr. Achenbach: But the acoustic waves play the role of an-

Mr. Ringermacher: You can't even talk about acoustic waves because the acoustic wavelength at 100 Hertz is several feet. So you've got just elastic vibration.

From the Floor: You said your system works by the Doppler shift effect method of displacement and therefore is not sensitive to surface conditions. But if your surface is rough, would you destroy a system by having a little light come back in the problem?

Mr. Ringermacher: By having little?

From the Floor: Yes.

Mr. Ringermcher: You reach a point where insufficient light comes back to achieve quieting in the discriminator.

From the Floor: In a way, you still cannot image rough substances?

Mr. Ringermacher: I've got 40 dB of dynamic range and there's enough light off the rough surfaces. You can defocus the spot a little bit, for example, to average over the roughness and get enough light back to saturate the discriminator. And literally, you just scan the beam over the surface and you can get light intensity changes of a factor of 100 and still saturate the discriminator if you have a good low-noise F.M. detection system.

Mr. Randy Sands: Harry, you mention that you were doing some work with coatings for ceramic. You mentioned briefly that you were looking at coatings for carbon-carbon composites. Can you look at the bottom line between the coating and the composite? Are you able to resolve that, or do you have a difficult problem there, because the composite underneath is not necessarily well-defined?

Mr. Ringermcher: Well, it is a rough problem. That's one of the challenges in the program. The program is fresh off the ground as far as this kind of inspection is concerned. We are looking at 10-mill-thick coatings. That's at the limit of what I can do for frequency. That requires 100 Hertz and lower to penetrate, so there are noise problems with the scanning electron microscope. That's one of the reasons I'm anxious to get up on the laser system, because it doesn't suffer from those problems. There are pumps on the electron microscope that drive you crazy.

But yes, we should be able to see variations at the interface. We are making up standards right now to check that aspect at the boundary.

Oh, I didn't mention this, but one of the important results would be to not only look at thermal defects in the coatings, but to look at coating integrity because this technique is sensitive to mechanical aspects of the coating. If a coating, for example, is weakly coupled to the surface, this will pick it up. So it will pick up both the mechanical aspects of the coating as well as the thermal-related properties.