Subsurface defect detection in materials using optical coherence tomography

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Abstract: We have used optical coherence tomography to study the internal structure of a variety of non-biological materials. In particular, we have imaged internal regions from a commercial grade of lead zirconate titanate ceramic material, from a sample of single-crystal silicon carbide, and from a Teflon-coated wire. In each case the spatial positions of internal defects were determined.

OCIS codes: (110.4500) Optical coherence tomography; (120.4290) Nondestructive testing; (290.7050) Turbid media.

References and links

1. R. C. Youngquist, S. Carr, and D. E. N. Davies, "Optical coherence-domain reflectometry: a new optical evaluation technique," Opt. Lett. **12**, 158-160 (1987).
2. K. Takada, I. Yokohama, K. Chida, and J. Noda, "New measurement system for fault location in optical waveguide devices based on an interferometric technique," Appl. Opt. **26**, 1063 (1987).
3. A. F. Fercher, K. Mengedoht, W. Werner, "Eye-length measurement by interferometry with partially coherent light," Opt. Lett. **13**, 186-188 (1988).
4. E. A. Swanson, D. Huang, M. R. Hee, J. G. Fujimoto, C. P. Lin, and C. A. Puliafito, "High-speed optical coherence domain reflectometry," Opt. Lett. **17**, 151-153 (1992).
5. V. M. Gelikonov, G. V. Gelikonov, R. V. Kuranov, K. I. Pravdenko, A. M. Sergeev, F. I. Feldshtein, Ya. I. Khanin, and D. V. Shabanov, "Coherent optical tomography of microscopic inhomogeneities in biological tissues," JETP Lett. **61**, 159 (1995).
6. M. Bashkansky, M. D. Duncan, M. Kahn, D. Lewis, III and J. Reintjes, "Subsurface Defect Detection in Ceramics Using Optical Gated Techniques," Opt. Lett. **22**, 61-63 (1997).
7. J. A. Powell and D. J. Larkin, "Process-induced morphological defects in epitaxial CVD silicon carbide," Phys. Status Solidi B **202**, 529-548 (1997).
8. J. F. de Boer, T. E. Milner, M. J. C. van Gemert, J. S. Nelson, "Two-dimensional birefringence imaging in biological tissue by polarization-sensitive optical coherence tomography", Opt. Lett. **22**, 934-936 (1997).

1. Introduction

Optically-gated reflectometry [1-3] based on low coherence interferometry has found important applications in surface and sub-surface imaging. This is because of the high spatial resolution, the relative simplicity, and the superior signal discrimination of the technique when used in highly scattering material. When this type of optically gated reflectometry is used in a scanning mode it is termed optical coherence tomography (OCT). Until recently, the vast majority of applications for OCT have been in biology [4, 5].

In this work, we use multimedia to demonstrate the application of OCT techniques to the detection of defects and subsurface structures in a variety of non-biological materials. Our OCT apparatus utilizes a design that allows rapid scans in a plane perpendicular (X-Z) or parallel (X-Y) to the sample surface while also maximizing the spatial resolution over the entire scan area. We have used our apparatus to measure sub-surface defects in a number of ceramic materials, in CVD-grown diamond films, and in single-crystal SiC. We have also

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imaged through plastic and composite materials. In most cases, the material under study was highly scattering, which rendered non-gated imaging impossible.

2. Apparatus

The experimental set up for our OCT apparatus has been described elsewhere [6]. In brief, we use 130 μW of 40 nm broad, 1.3 μm light from a light emitting diode (LED). The LED radiation is coupled into a single-mode fiber and split into reference and probe beams in a 50% fiber splitter. The phase of the reference beam is modulated by stretching the fiber a few microns at high speed (~300 kHz) with a piezoelectric transducer. The reference beam is retro-reflected from a mirror back into the fiber and toward the 50 % splitter. The probe beam is focused on the sample with a microscope objective lens. The probe radiation scattered from the sample is collected by the same lens, retraces its path through the fiber, and recombines with the back-propagating reference beam in the 50% splitter. This combined radiation is detected by a fiber-coupled photodiode. The light that has traveled the same distance in both arms of the interferometer is correlated and produces a signal due to the phase modulation.

A galvanometer-controlled mirror is placed before the microscope objective lens and provides rapid scanning in one transverse direction. The sample is scanned in depth or in the other transverse direction with slower, computer-controlled translation stages. Two-dimensional images consisting of 250 x 250 points are captured in less than one second using this scanning technique. All scanning and data acquisition functions are computer controlled, providing near-real-time display of OCT images.

The transverse spatial resolution of our OCT device is approximately 4 μm. This is limited by the quality of the microscope objective that we are using. The depth resolution is given by the correlation length of the radiation from the LED and is approximately 20 μm in air. In a material this resolution is improved by a factor of n, where n is the material index of refraction. In our current apparatus, the size of a two-dimensional scan, in air, can be varied from 500 μm x 500 μm to 4 mm x 4 mm.

3. Experimental results

Our OCT apparatus has been used to image internal defects in a large number of ceramics. One widely-used type of ceramic is piezoelectric lead zirconate titanate material. Defects are a problem in this material both from a structural standpoint and, in the case of acoustic transducers, from an acoustical-energy production standpoint. Figure 1 shows micrographs of a 2 mm thick sample of this type of material. This sample has multi-millimeter-sized discolorations and smaller, barely apparent anomalies at the center of each discoloration. These anomalies are expected to be defects in the ceramic structure, but they cannot be localized or even easily seen using ordinary light microscopy, due to the highly scattering nature of the material. Even destructive testing is not guaranteed to help because of the small defect size and because of the possibility of introducing other defects.

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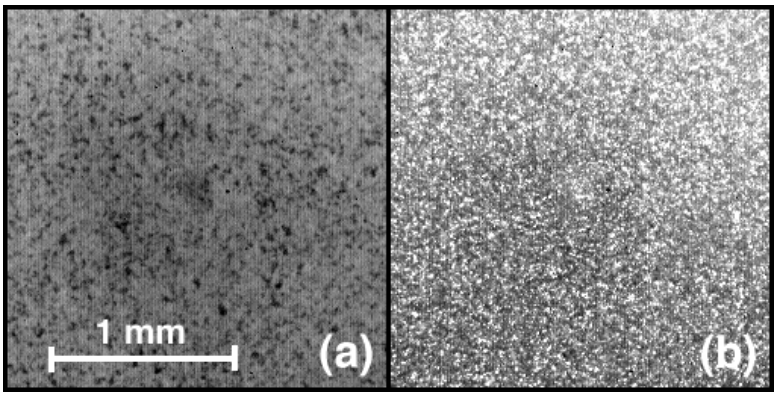

Fig. 1. Reflection (a) and transmission (b) micrographs of a silicon nitride PZT ceramic sample 4 mm thick. There is a defect in the center of the image, and a larger, low contrast discoloration surrounding the defect.

Figure 2 shows a movie made up of a number of OCT scans of the same region of the PZT ceramic. The defect appears slightly to the top and left of center and is seen to arise at a depth of 20 - 30 μm, grow slightly in size, and then begin to disappear at a depth of 140 μm. Each scan was taken in approximately 1 second. The noisy appearance of the scans is due to the intrinsic high scattering from crystal grain boundaries that occur naturally in the ceramic material. The increase in scattering apparent in the defect region could be due to a higher or lower density of material in the defect, to inclusions, or to excess concentrations of one particular component of the ceramic matrix.

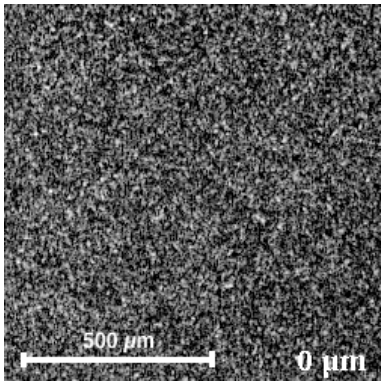

Fig. 2. A movie of 13 separate OCT scans taken at various depths from the same region of the sample shown in Fig. 1. The OCT scans are taken in the X-Y plane (parallel to the surface of the ceramic) at the depths shown in the lower right hand corner of each frame. Zero microns represents the approximate surface of the sample.

We have also studied-single crystal silicon carbide (SiC) samples with our OCT apparatus. This type of SiC is important in high-power and high-temperature electronics but is still prone to various problems during growth and processing. Major problems include pits that form during the growth of epilayers and the formation of 'micropipe' defects, small tubular voids that run through the crystal in a direction normal to the surface [7]. Single-crystal SiC is not highly scattering, but the presence of surface scratches and surface defects, especially in the early stages of inspection and processing, limit the ability to localize, or even see, internal defects. An example of single-crystal SiC material is shown in Fig. 3. This is a transmission micrograph of a region on the sample with numerous surface and sub-

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surface defects. Because of the surface scattering, it is very hard to identify the extent of any but the most superficial defects.

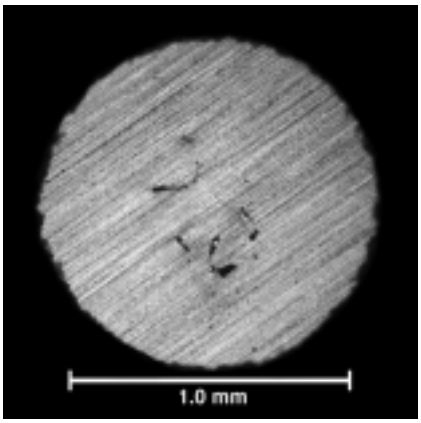

Fig. 3. A transmission micrograph of a single-crystal silicon carbide sample.

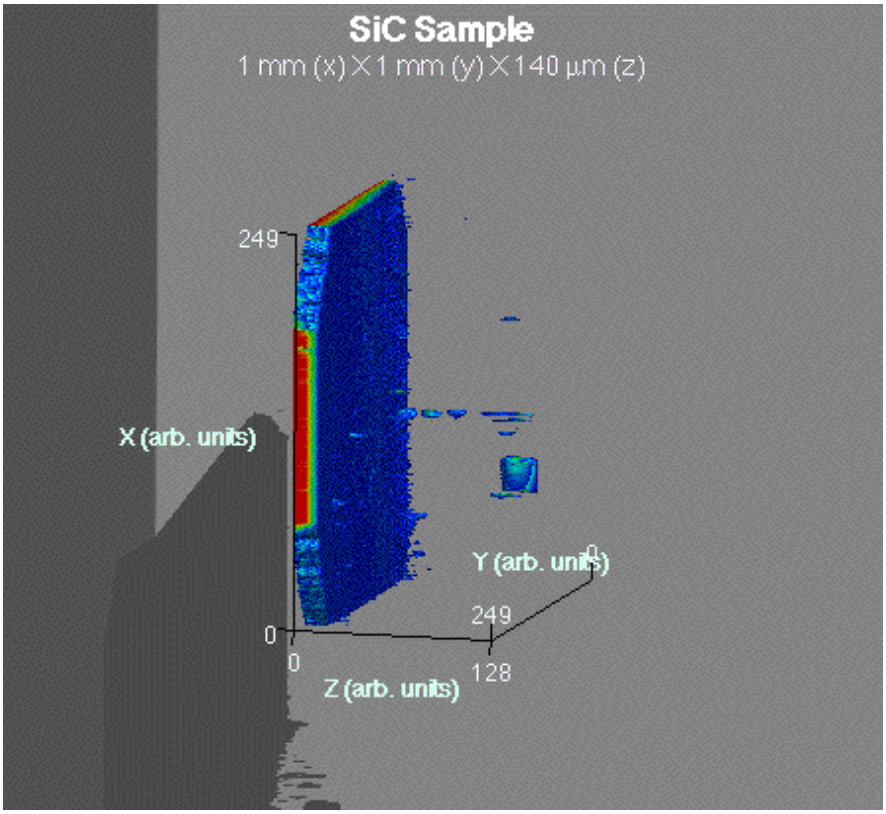

Fig. 4. A movie showing various three-dimensional views of OCT data from 15 different X-Y scans in the silicon carbide sample shown in Fig. 3. The scan range in X and Y is 1 mm . The X-Y scans were taken 10 µm apart in depth. The colored regions represent areas of high scatter.

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We have used our OCT apparatus to take of number of sub-surface scans in the SiC sample shown in Fig. 3. We took 15 separate X-Y scans, at 10 μm depth intervals, from the surface to 140 μm into the sample. They have been combined to form a 3-dimensional representation of the sample, as shown in Fig. 4. In addition, we generated a number of different views of the 3-dimensional data, allowing the data to be shown in a "fly-by" style movie. These different views allow a better understanding of the position and extent of the various structures seen using our technique.

The colors used in Fig. 4 represent the strength of the backscattered signal, with red being the most and blue being the least. The scattering from the surface is very strong, partially due to the roughness and partially due to the index of refraction mismatch with air. Below that, however, there is essentially no scattering except from various internal defects in the bulk SiC crystal. Besides the obvious large defect that is deep in the material, we can see a long, thin defect that is normal to the sample surface. This is a classic micropipe defect. In addition, we can see the exact depth location of other internal defects in the crystal. Such localization of these defects would have been very difficult, if not impossible, using non-gated imaging techniques.

We have also used our OCT apparatus to image through plastic and paint coatings. Plastic coatings on wires are of particular interest since images obtained through the normally opaque plastic layer could be used to detect cracks, corrosion, or complete breaks in the underlying wires. Figure 5 is an example of an OCT scan taken in the X-Z plane through a teflon-coated copper wire. The coating is approximately 400 μm thick and can be seen to consist of a number of layers. Below the bottom layer is a solid strand of copper wire. There is no optical penetration into the wire, of course, but the surface of the wire provides a large return signal when the scanning light is specularly reflected from the metal surface. That is the bright surface seen in the inner region of the structure. In order to create a flaw in this coated wire, we removed the copper wire, cut it completely, and then reinserted the two pieces from opposite ends until they touched. We then took 29 OCT scans of the coated wire and put them into a movie, also shown as Fig. 5. The scans started on one side of the inner wire break, were each separated by 100 μm along the wire, and ended on the other side of the break.

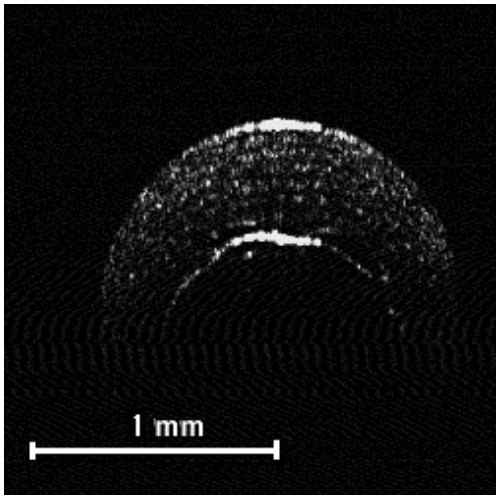

Fig. 5. A movie of 29 separate OCT scans taken at 100 μm intervals along a teflon-coated copper wire. The OCT scans are taken in the X-Z direction (cross-sections of the wire).

In the movie of Fig. 5, the surface of the copper wire is seen to stay at approximately the same radius until frame 14. At that point the surface of the copper wire rapidly drops and is greatly reduced in height by frame 18. From frame 19 until frame 26, a

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new surface appears and grows to fill the inner region of the wire. This sequence of images shows the tapering of one end of the cut copper wire and then the growth of the other, abutted, piece of copper wire.

4. Summary

We have shown how optical coherence tomography can be used as a tool for non-destructive testing and evaluation of ceramic and other materials. This technique is particularly suited to highly scattering material due to the gated nature of OCT. Other applications of OCT in non-biological materials include ball-bearing inspection, diamond film growth monitoring, and thermal-barrier coating analysis. OCT is also intrinsically sensitive to the sample birefringence [8]. Therefore, it should be possible to detect stress-induced birefringence below the surface of various samples. Possible indication of this effect was mentioned previously [6]. We plan to study polarization effects in materials using OCT and apply the results to the nondestructive inspection of ceramics and other materials.

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