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Georgia Tech's new Manufacturing Research Center and the Electronic Industries Association (EIA) are co-sponsoring a seminar on what may be the most important problem facing electronics manufacturers today: component solderability.

The seminar, to be held October 5 and 6 in Atlanta, is intended for two groups: engineers and managers responsible for assembly of electronic components and researchers in materials science and chemistry who can make contributions toward resolving the problems.

Attendees will hear presentations from two key industry authorities: Mike Wolverton, a materials specialist at Texas Instruments Defense Systems Electronics Group in Dallas; and Dr. Gordon Davy, a soldering specialist at Westinghouse Defense Center in Baltimore.

The seminar begins at noon on Thursday, October 5, 1989, and continues all day Friday at the Lanier Plaza Hotel and Conference Center in midtown Atlanta. Attendees will learn about Georgia Tech's research agenda in manufacturing technology, and have an opportunity to tour the Tech campus.

For more information and registration, contact Joe T. Franklin, Jr., Electronic Industries Association, 1722 Eye Street, N.W., Suite 300, Washington, D. C. 20006 Telephone: (202) 457-4930.
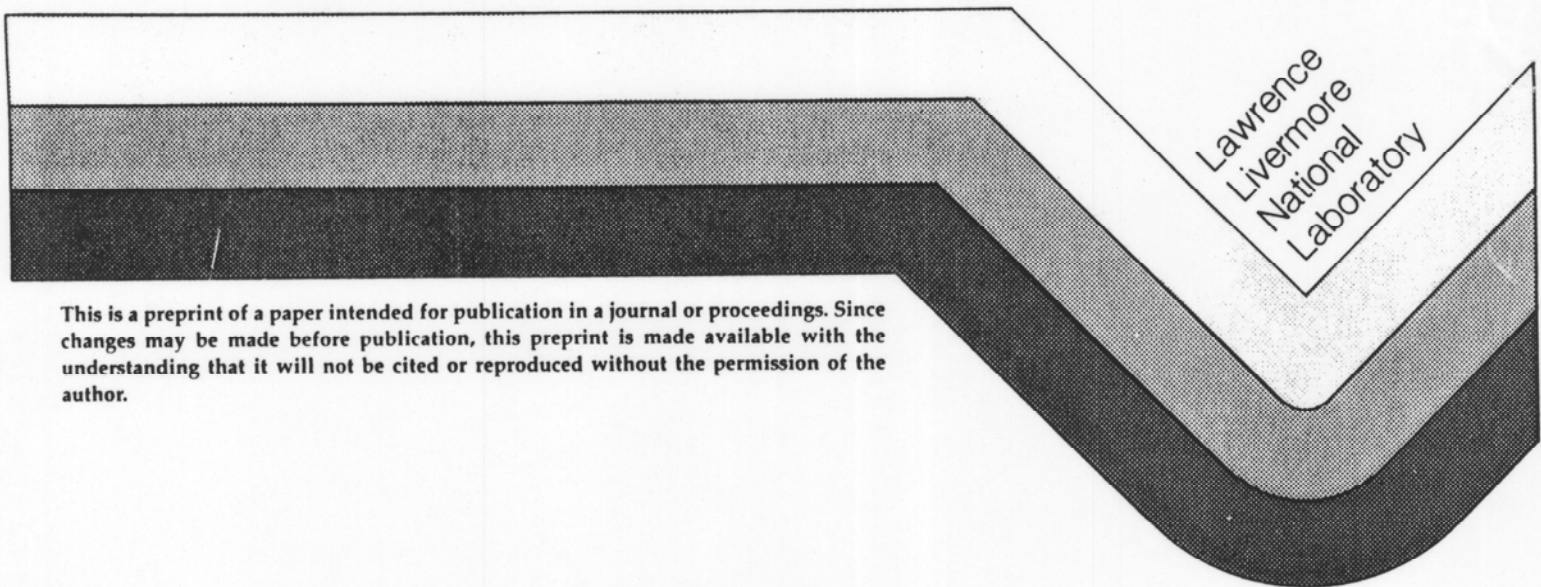
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August 25, 1989

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ABSTRACT

X-ray tomographic microscopy (XTM) utilizes intense, highly collimated synchrotron radiation to nondestructively image materials structures in three dimensions. The spatial resolution of the technique approaches that of conventional optical microscopy, but XTM does not require polished surfaces or serial sections. We present the results of an XTM investigation of a composite material composed of silicon-carbide fibers in an aluminum matrix. The results reveal the aluminum/silicon-carbide interfaces and show microcracks running along many of the interfaces as well as in the matrix. Excellent contrast is observed between the silicon-carbide sheath of the fiber surrounding the graphite core and the coating at the fiber-matrix interface. The sensitivity to small changes in the linear absorption coefficient allows nondestructive imaging of variations in chemistry between graphite and silicon carbide and between silicon carbide and aluminum. The experimentally determined values of the absorption coefficients of these phases are identical to values published in the literature. For the first time, XTM will allow observation of damage accumulation and crack growth *during* deformation testing. The availability of such data will greatly improve our understanding of failure in advanced materials.

I. INTRODUCTION

Advanced composites are beginning to fill important niches as structural materials. Continuous-fiber, metal-matrix composites, for example, provide an unprecedented combination of high specific stiffness and strength, high thermal and electrical conductivity, and high dimensional stability over wide temperature ranges.[1] The utilization of composite materials for primary structural applications requires adequate design margins and lifetimes under all anticipated loading conditions and environments. This goal demands sound understanding of the possible failure mechanisms, an understanding that necessitates a broad experimental data base. The prohibitive expense required to produce such a data base frequently prevents evaluation of new composite systems; indeed, using present methods, it is impractical to gain confidence in the fracture and fatigue properties even of familiar composites.

Isolating the fundamental failure mechanisms of composites from the confusion of information created by incidental specimen-to-specimen variations is extremely difficult. Present methods used for determining the spatial variation in density and for characterizing damage in composites with micrometer resolution rely on destructive analysis, usually performed *post mortem*, and cannot readily ascertain the order in which the observed features developed. Serial sectioning, for example, is destructive, extremely time consuming and inefficient. Furthermore, in the case of metal matrix composites, surface relief during polishing tends to obscure interfacial cracks that might be present in the sample and may introduce additional cracks.

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Clearly, a technique for nondestructively imaging microstructures of materials *in situ*, especially a technique capable of delineating the time evolution of chemical changes or damage, would greatly benefit studies of materials processing and failure. In this paper, we describe a recently developed technique for nondestructively characterizing microstructures in three dimensions. This technique, x-ray tomographic microscopy (XTM), has been applied to metal matrix composites and has imaged microcracks and the interface between the ceramic fiber and the metal matrix. This paper discusses these results.

II. BACKGROUND

XTM differs from conventional optical and electron-beam microscopy in that the sample need not be harmed prior to characterization of its internal microstructure. There is no requirement for flat optical surfaces or thin sections, and it is thus possible to examine materials in their unaltered state. XTM is based upon the same principle as is used in medical computed tomography (CT), in which the x-ray attenuation coefficient, μ, at a point $r_{x,y,z}$ in a material can be determined from a finite set of x-ray attenuation measurements (projection data) taken at different angles. The projection data is the transmitted x-ray intensity reaching a position-sensitive detector after passing through the sample. This data, which is directly related to the materials microstructure, is given by

$$ I = \int S(E) \left\{ \exp\left[-\int \mu(x,y,z,E) dl \right] \right\} dE \quad , \tag{1} $$

where $S(E)$ is the energy spectrum of the x-ray source, and $\mu(x,y,z,E)$ is the energy-dependent attenuation coefficient at a single point on the projection. The integral is taken along a straight path dl through the sample. Because a synchrotron beam can be made nearly monochromatic with photon energy E_0, the energy spectrum can be approximated by a delta function, and Eq. (1) reduces to the familiar form of the Radon transform,[2]

$$ \ln\left(\frac{I_0}{I}\right) = \int \mu(x,y,z,E_0) dl \quad . \tag{2} $$

Measurements of the attenuation through the sample as a function of angle are used to numerically invert Eq. (2) to solve for $\mu(x,y,z,E_0)$.[3] The number of angular views considered sufficient for this inversion (reconstruction) is approximated, using simple geometric arguments, by

$$ R \, \Delta\Theta = W \quad , \tag{3} $$

where R is the maximum outward extent of the sample from the center of rotation, $\Delta\Theta$ is the suggested angular increment, and W is the projection width. A typical value for R with the present XTM is 2 mm, and W is 5 μm. The angular increment sufficient for the reconstruction, using these dimensions, is approximately 0.2°. In practice, however, an increment of 0.5 to 1.0° is usually used because of limited beam time.

Conventional CT measurements involve collecting absorption information for a single cross-sectional slice through a material. Spatial resolution is achieved either by collimating the incident beam (using a pinhole) and then rastering the beam across the sample,[4,5] or by using a position-sensitive, linear, photodiode array to measure all of the projection data for a single angular

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view in parallel.[6] Aside from its relative simplicity, the advantage of the pinhole technique is that the resolution, to first order, is determined only by the size of the collimator. The primary disadvantage of using the pinhole is that most of the incident radiation is thrown away. The rastering technique, therefore, is extremely time consuming.[7] Acquiring the data for the reconstruction of a single cross section of a sample takes upwards of 12 h. This limits the utilization of the pinhole approach for three-dimensional analysis and precludes real-time studies.

The linear photodiode array recently has been used in a number of CT devices designed to operate on conventional x-ray sources.[8] The widespread application of linear photodiode arrays results from both their ease of use and their ability to acquire more than a thousand projected rays simultaneously. The parallel acquisition of data improves the speed of the measurements nearly a thousandfold, and the accumulation of enough data to reconstruct a single slice becomes measured in minutes rather than days. In spite of these advantages, however, there are drawbacks to the use of the photodiode array for ultrahigh-resolution characterization.[9] The first of these drawbacks is that the photodiode array is noisy and is subject to nonlinearities. The noise limits the dynamic range and, therefore, the maximum contrast that can be studied in a sample. The nonlinearities introduce ring-like artifacts in the reconstructions, which can further reduce the usefulness of the information obtained. Finally, even with reducing the data-acquisition times for a single slice from days to minutes, it still requires days to obtain enough information for three-dimensional sample visualization.

The XTM instrument we have developed, described in detail elsewhere,[10,11] overcomes most of the disadvantages encountered using conventional CT methods. The XTM requires a source of parallel x rays (synchrotron radiation) and consists of a sample stage, a detector system, and an analyzing computer. In operation, a double-crystal monochromator selects a narrow energy range of x rays from the primary beam [$(\Delta E/E) < 10^{-3}$]. The x rays pass through a sample positioned on a rotation stage, then pass through a second, magnifying x-ray optic[12] (if further magnification and filtering are desired), and are converted to visible light by means of a single crystal scintillator. The visible-light image is magnified and recorded on a low-noise, two-dimensional, charge-coupled-device (CCD) detector. The sample is rotated by a small angle, and another two-dimensional absorption image is obtained. This process continues until 180° of sample rotation has been recorded and stored in a computer. Tomographic software converts x-ray absorption profile data into two-dimensional reconstructions of the linear attenuation coefficients in the sample interior. These values are rendered into a three-dimensional view by means of high-speed, digital, image-processing computers.

A major advantage of the XTM over the pinhole or photodiode techniques is that all of the data for three-dimensional imaging can be acquired in parallel. Data-acquisition times have been reduced to a few hours, and improvements in mechanical and electrical hardware indicate that measurement times less than 1 h will soon be realized. Also, the cooled CCD has a tremendous dynamic range ($>10^3$) and is not as subject to nonlinearities as photodiode arrays are. The present XTM has a three-dimensional resolution of better than 10 μm, and the next generation of x-ray optics and synchrotron storage rings is expected to allow micrometer and submicrometer resolution.[13] In addition, when elemental[14] or phase-mapping[15] details are desired, excellent chemical contrast can be obtained by recording data at two x-ray wavelengths (usually above and below a characteristic absorption edge) and performing image subtraction to enhance chemical or phase-specific information. Conventional CT technology typically uses a polychromatic beam from conventional tube sources, which effectively precludes quantitative chemical analysis.

Since its development, XTM has been used to investigate coatings in supported catalysts[16] and porosity in oil shale and coal.[17] Only with the recent advances in XTM technology, however, has it become possible to image complex composite materials successfully.

III. EXPERIMENT AND DISCUSSION

In the present study, a $1.5 \times 1.5 \times 10$-mm specimen of an Al/SiC composite was examined by means of XTM on the 31-pole wiggler beamline at the Stanford Synchrotron Radiation Laboratory (SSRL).[18] An x-ray energy of 21 keV was selected for good sample transparency and x-ray contrast between SiC and Al. A set of 97 contiguous cross sections of the composite was examined with the XTM. Figure 1(a) shows a single slice with a projection width and slice thickness of 5.6 μm; the spatial resolution is much better than 10 μm. Data was collected with 5-s exposure times at 1° intervals, a much smaller interval than that suggested by the use of Eq. (3). This undersampling leads to some blurring of the image; nevertheless, the 32-μm-diameter graphite cores and surrounding 140-μm-diameter SiC sheaths are clearly visible. The slight mottling in Fig. 1(a) is caused by either the statistical noise in the image (~2%) or actual variations in the materials composition.

Cracks running longitudinally in the plane of the fiber plies are evident in Fig. 1(a). These cracks, which do not penetrate the fibers, are similar to those observed in polished sections of the composite [Fig. 1(b)] and may, therefore, be a result of processing. This material is fabricated by means of a plasma-spray technique and is consolidated at high temperatures. It is possible that mismatches in thermal-expansion coefficients may create residual stresses that open these cracks during cooling, or that the material is not being completely consolidated. It is interesting to note, in Fig. 1(b), that surface relief and plastic flow of the matrix during polishing have apparently obscured the interfacial cracks that are observed in Fig. 1(a). This result highlights one advantage of XTM over conventional metallography.

Figure 2(a) is a planar cut through the same sample as in Fig. 1, but parallel to the fibers. In this ply, the fibers are regularly arranged, and few cracks are present. Figure 2(b) is a similar plane taken through a ply that is heavily populated with cracks. Figure 2 demonstrates that the cracks tend to run along the fiber-matrix interface and between nearest-neighbor fibers rather than across the plies of the composite.

Values of μ calculated and obtained by using XTM are compared in Table I for the various components of the composite. No corrections or normalizations have been made to the measured values of μ. All constituents are assumed to be fully dense in the calculations, and the SiC is presumed to be stoichiometric. The agreement between the XTM data and the calculated values is excellent, and the small differences between calculation and experiment are probably the result of slight departures from the nominal compositions of each phase, as well as inaccuracies in the tabulated cross sections.

The largest difference between observed and calculated values of μ is for SiC. As we discuss later, our results show that the fiber sheaths are probably not stoichiometric. If, for the sake of argument, one assumes that the structure and chemical composition of the sheaths are the same as reported elsewhere[22] (carbon core surrounded by 40 μm of 94 wt. % SiC and 6 wt. % C, which in turn is surrounded by 40 μm of stoichiometric SiC), a somewhat lower average μ would result. Weighting the contributions of the different compositions by the volume fraction of each, one obtains $\mu = 8.75 \text{cm}^{-1}$, which is identical to the observed value.

The exterior Al that surrounds the composite is 99.9% pure. The interior, on the other hand, is comprised of foils and plasma spray of 6061 aluminum. The calculated value of μ in Table I for 6061 aluminum is based on the nominal composition for 6061 alloy, but considerable chemical variation is allowed in both commercially pure and 6061 aluminum. Also, at the temperatures and times under which this composite was consolidated, significant diffusion of minor elemental constituents is anticipated[23] and would minimize any differences in absorption coefficients between the two alloys of aluminum. Nevertheless, the measured μ values show a

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scaling similar to that calculated. This gives us confidence that XTM can be made very sensitive to subtle changes in alloy composition. Therefore, it should be possible to use XTM to study, at least in coarse detail, interfacial chemistry *in situ*.

Figure 3 is a view of a single fiber that shows the graphite core and the SiC sheath surrounding it. The change in columnar SiC subgrain size produced during the growth of the sheath is evident, as is the SCS-8 coating in the final few micrometers near the Al interface. The variation in μ across the fiber is extracted from the noise in the silicon carbide data (Table I) by averaging over many volume elements. The radially averaged line trace of the XTM data in Fig. 4 graphically shows the change in carbon concentration across the fiber and is consistent with reported variations of stoichiometry.[24,25]

IV. FUTURE DIRECTIONS

Efforts are now being focused on improving the resolution and sensitivity of XTM. Achieving a spatial resolution of 1 μm and better will require improved sample-positioning capabilities and either larger CCD formats or smaller samples. This latter point is demonstrated with the data in Table II, where maximum sample sizes are given for 1-μm resolution as a function of the commercially available CCDs.

Table II indicates that only the largest-format CCD arrays are useful for XTM imaging of composites. In this study, we used the Kodak 1320 × 1035 CCD with a magnification of 2.4×. This allowed imaging of a 1.5 × 1.5-mm section of composite. Samples smaller than this are of much less practical value, and it would be extremely important to be able to image 1-cm-diameter samples with a resolution of 1 μm. An ideal XTM would not be constrained to keeping the entire sample within the field of view, as is done now; rather, it should be possible to vary the magnification so that only the region of interest is displayed with high resolution. This, however, will require more research into reconstruction algorithms.

Another avenue for future work is the design of *in situ* tensile stages and environmental chambers for imaging samples under load or high temperature. Presently, all measurements are made at ambient room conditions. If a sample is stressed or a crack is introduced, it must first be removed from the XTM stage for stressing and then replaced in the XTM for characterization. Because cracks may close in the absence of an applied load, direct visualization of damage prior to failure may be difficult unless *in situ* load cells are used. The development of these stages and chambers is under way.

V. CONCLUSIONS

This application of XTM is the first time a nondestructive technique has had sufficient spatial resolution and sensitivity to image the microcracking and interfacial structure of a metal-matrix/ceramic-fiber composite in three dimensions. Because monochromatic synchrotron radiation was used, it is possible to unambiguously measure the absolute values of the x-ray attenuation coefficient for the different structures in the composite. The excellent agreement between the measured and calculated attenuation coefficients indicates that the XTM technique can be used not only to image high-contrast features such as microcracks, but also the time evolution of chemical changes within a material.

We must emphasize that these XTM images have been taken of *unaltered* interior surfaces. XTM is nondestructive, and no surface preparation is involved in the characterization. Because of this, direct observation of crack nucleation and growth is possible throughout *bulk* specimens *during* deformation testing. The availability of such data should have a profound effect on the modeling of damage accumulation in metal-matrix composites and other advanced materials.

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VI. ACKNOWLEDGMENTS

TABLE I. Calculated and observed values of the linear absorption coefficient (μ), in cm^{-1}.

Materials	μ_{calc}[19,20]	μ_{obs}	$\dfrac{\mu_{calc}}{\mu_{obs}}$
Crack	0	0.07 ± 0.045	— *(0.88 ± 0.44)*
Graphite	0.85 (0.86)	0.85 (0.86)	0.97 (0.98)
Aluminum (pure)	7.90 (7.95)	8.09 ± 0.21	0.98 (0.98)
Aluminum (6061)[21]	7.96 (8.26)	8.15 ± 0.18	0.98 (1.01)
Silicon carbide	9.21 (8.85)	8.70 ± 0.73	1.06 (1.02)

TABLE II. Largest samples that can be imaged with XTM at resolution of 1 μm.

CCD	Format[a]	Pixel size (μm)	f_n (lp/mm)[b]	Magnification[c]	Largest sample (mm)
Kodak	1320×1035	6.8	74	7×	1.3
Kodak	2K × 2K	9.0	56	9×	2.0
TI4849	390 × 584	22.0	23	22×	0.6
Thompson	384 × 576	23.0	22	23×	0.5

[a] Number of pixels per row and column in the CCD
[b] f_n is the Nyquist frequency.
[c] Minimum necessary to achieve 1-μm spatial resolution, assuming the discreet sampling of the CCD is the only limit.

REFERENCES

1. M. F. Amateau, J. Composite Mat. **10**, 279 (1976).
2. G. T. Herman, *Image Reconstruction from Projections: The Fundamentals of Computerized Tomography* (Academic Press, New York, 1980).
3. Q. C. Johnson, J. H. Kinney, U. Bonse, and M. C. Nichols, Mat. Res. Soc. Symp. Proc. **69**, 203 (1986).
4. J. C. Elliott and S. D. Dover, J. Microscopy **126**, 211 (1982).
5. J. C. Elliott and S. D. Dover, J. Microscopy **138**, 329 (1985).
6. F. H. Seguin, P. Burstein, P.J. Bjorkholm, F. Homburger, and R. A. Adams, Appl. Opt. **24**, 4117 (1985).
7. S. R. Stock, A. Guvenilir, J. C. Elliott, P. Anderson, S. D. Dover, and D. K. Bowen, in *Advanced Characterization Techniques for Ceramics* (American Ceramic Society, Westerville, Ohio, in press).
8. M. K. Cueman, L. J. Thomas, C. Trzaskos, and C. Greskovich, in *Review of Progress in Quantitative Nondestructive Evaluation*, D. O. Thompson and Dale E. Chimenti, eds. (Plenum Press, New York, 1989), Vol. 8A, p. 431.
9. J. H. Kinney, Q. C. Johnson, U. Bonse, R. Nusshardt, and M.C. Nichols, SPIE **691**, 43 (1986).
10. U. Bonse, Q. Johnson, M. Nichols, R. Nusshardt, S. Krasnicki, and J. Kinney, Nucl. Instrum. Methods **A246**, 644 (1986).
11. J. H. Kinney, Q. C. Johnson, R. A. Saroyan, M. C. Nichols, U. Bonse, R. Nusshardt, and R. Pahl, Rev. Sci. Instrum. **59**, 196 (1988).
12. U. Bonse, R. Nusshardt, F. Busch, Q. C. Johnson, J. H. Kinney, R. A. Saroyan, and M. C. Nichols, Rev. Sci. Instrum. (in press).
13. T. Hirano, K. Usami, and K. Sakamoto, Rev. Sci. Instrum. (in press).
14. L. Grodzins, Nucl. Instrum. Methods **206**, 541(1983).
15. J. H. Kinney et al., Appl. Opt. **25**, 4583 (1986).
16. M. C. Nichols, J. H. Kinney, U. Bonse, Q. C. Johnson, R. A. Saroyan, R. Nusshardt, and F. Busch, Rev. Sci. Instrum. (in press).
17. B. P. Flannery, H. Deckman, W. Roberge, K. D'Amico, Science **237**,1439 (1987).
18. The storage ring SPEAR was running at 3.3 GeV and nominally 30 mA. The wiggler field was 1.0 T, only 60% of its rated field strength.
19. E. F. Plechaty, in "Tables and Graphs of Photon-Interaction Cross Sections," Lawrence Livermore National Laboratory, Livermore, Calif., UCRL-50400, Vol. 6, Rev. 3 (1981).
20. *International Tables for X-Ray Crystallography* (Kynoch Press, Birmingham, England,1972), Vol. 4, pp. 61–66.
21. The nominal composition of this alloy is from *Metals Handbook Desk Reference* (American Society for Metals, Metals Park, Ohio, 1961), 8th ed., Vol. 1, p. 945.
22. P. Martineau, M. Lahaye, R. Pailler, R. Naslain, M. Couzi, and F. Creuge, J. Mat. Sci. **19**, 2731 (1984).
23. W. G. Friche, Jr., Scripta Metall. **6**, 189 (1972).
24. S. R. Nutt and F. E. Wawner, J. Mat Sci. **20**, 1953 (1985).
25. B. A. Lerch, D. R. Hull, T. A. Leonhardt, *As-Received Microstructures of a SiC/Ti-15-3 Composite*, National Aeronautics and Space Administration, Washington, D.C., NASA TM-100938 (1988).