NATIONAL SCIENCE FOUNDATION
Washington, D.C. 20550

FINAL PROJECT REPORT
NSF FORM 98A

PLEASE READ INSTRUCTIONS ON REVERSE BEFORE COMPLETING

PART I—PROJECT IDENTIFICATION INFORMATION

1. Institution and Address	2. NSF Program	3. NSF Award Number
Georgia Tech Research Corporation Georgia Institute of Technology Atlanta, Georgia 30332-0245	INT	INT-8513629
	4. Award Period From 3/1/86 To 12/31/88	5. Cumulative Award Amount $9,350

6. Project Title
Synchrotron Radiation Studies of the Spatial Distribution of Impurities in Materials

PART II—SUMMARY OF COMPLETED PROJECT *(FOR PUBLIC USE)*

Novel synchrotron radiation techniques were used to study the spatial distribution of impurities in materials. A number of techniques were proposed for investigation, and efforts in the project focused on topographic EXAFS and microtomography. Topographic EXAFS refers to contrast observed on x-ray diffraction images (e.g. topographs) due to the fine structure of the x-ray absorption edge. Microtomography is a very high resolution variant of the familiar medical CAT scanner. Experimental results are reported for crystals of gallium arsenide and hematite and for silicon carbide/silicon nitride and silicon carbide/aluminum composites. The technological and scientific impact of these experiments are also reported.

PART III—TECHNICAL INFORMATION *(FOR PROGRAM MANAGEMENT USES)*

1. ITEM *(Check appropriate blocks)*	NONE	ATTACHED	PREVIOUSLY FURNISHED	TO BE FURNISHED SEPARATELY TO PROGRAM	
				Check (✓)	Approx. Date
a. Abstracts of Theses	X				
b. Publication Citations		X			
c. Data on Scientific Collaborators		X			
d. Information on Inventions	X				
e. Technical Description of Project and Results		X			
f. Other *(specify)* i)presentations at national/ international meetings. ii)proposals resulting from program's accomplishment		X X			

2. Principal Investigator/Project Director Name *(Typed)*	3. Principal Investigator/Project Director Signature	4. Date
Stuart R. Stock		3/12/89

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Form Approved OMB No. 3145-0058

PART IV - SUMMARY DATA ON PROJECT PERSONNEL

NSF Division INT-8513629

The data requested below will be used to develop a statistical profile on the personnel supported through NSF grants. The information on this part is solicited under the authority of the National Science Foundation Act of 1950, as amended. All information provided will be treated as confidential and will be safeguarded in accordance with the provisions of the Privacy Act of 1974. NSF requires that a single copy of this part be submitted with each Final Project Report (NSF Form 98A); however, submission of the requested information is not mandatory and is not a precondition of future awards. If you do not wish to submit this information, please check this box ☐

Please enter the numbers of individuals supported under this NSF grant.
Do not enter information for individuals working less than 40 hours in any calendar year.

*U.S. Citizens/ Permanent Visa	PI's/PD's		Post-doctorals		Graduate Students		Under-graduates		Precollege Teachers		Others	
	Male	Fem.	Male	Fem.	Male	Fem.	Male	Fem.	Male	Fem.	Male	Fem.
American Indian or Alaskan Native	0	0	0	0	0	0	0	0	0	0	0	0
Asian or Pacific Islander	0	0	0	0	0	0	0	0	0	0	0	0
Black, Not of Hispanic Origin	0	0	0	0	0	0	0	0	0	0	0	0
Hispanic	0	0	0	0	0	0	0	0	0	0	0	0
White, Not of Hispanic Origin	0	0	0	0	0	0	0	0	0	0	0	0
Total U.S. Citizens	0	0	0	0	0	0	0	0	0	0	0	0
Non U.S. Citizens	0	0	0	0	0	0	0	0	0	0	0	0
Total U.S. & Non-U.S. ..	0	0	0	0	0	0	0	0	0	0	0	0
Number of individuals who have a handicap that limits a major life activity.	0	0	0	0	0	0	0	0	0	0	0	0

*Use the category that best describes person's ethnic/racial status. (If more than one category applies, use the one category that most closely reflects the person's recognition in the community.)

AMERICAN INDIAN OR ALASKAN NATIVE: A person having origins in any of the original peoples of North America, and who maintains cultural identification through tribal affiliation or community recognition.

ASIAN OR PACIFIC ISLANDER: A person having origins in any of the original peoples of the Far East, Southeast Asia, the Indian subcontinent, or the Pacific Islands. This area includes, for example, China, India, Japan, Korea, the Philippine Islands and Samoa.

BLACK, NOT OF HISPANIC ORIGIN: A person having origins in any of the black racial groups of Africa.

HISPANIC: A person of Mexican, Puerto Rican, Cuban, Central or South American or other Spanish culture or origin, regardless of race.

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THIS PART WILL BE PHYSICALLY SEPARATED FROM THE FINAL PROJECT REPORT AND USED AS A COMPUTER SOURCE DOCUMENT. DO NOT DUPLICATE IT ON THE REVERSE OF ANY OTHER PART OF THE FINAL REPORT.

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b. Publication Citations

1. "X-ray Microtomography with Synchrotron Radiation," D. K. Bowen, J. C. Elliott, S. R. Stock and S. D. Dover, in X-ray Imaging II, SPIE Vol. 691, (1986) 94.

2. "Microtomography of Silicon Nitride/Silicon Carbide Composites," S. R. Stock, A. Guvenilir, T. L. Starr, J. C. Elliott, P. Anderson, S. D. Dover and D. K. Bowen, accepted for Advanced Techniques for Characterization of Ceramics (Am. Cer. Soc./MRS, 1989).

Anticipated Publications, Journal (if known) and Submission Dates

1. "Detectability of Cracks in Continuous-Fiber, Metal Matrix Composites Using Microtomography," T. M. Breunig, S. R. Stock, J. C. Elliott, A. Guvenilir, S. D. Antolovich, P. Anderson, S. D. Dover and D. K. Bowen, est. June 1989.

2. "Processing Induced Porosity and Density Variation in Silicon Carbide/Silicon Nitride Ceramic Matrix Composites," A. Guvenilir, S. R. Stock, J. C. Elliott, T. L. Starr, P. Anderson, S. D. Dover and D. K. Bowen, Phil Mag A, J. Am. Cer. Soc. or J. Mat. Res., est. June 1989.

c. Data on Scientific Collaborators

The various individuals who participated in different parts of the cooperative science project are listed below:

Georgia Institute of Technology

Mr. A. Guvenilir (Materials Engineering)
Mr. T. M. Breunig (Materials Engineering)
Dr. T. L. Starr (GTRI)
Dr. S. D. Antolovich (Materials Engineering)

London Hospital Medical College (LHMC)

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e. Technical Description of Project and Results

1. Introduction

The initial goal of this cooperative science project was to develop and to apply synchrotron x-ray diffraction imaging, microbeam x-ray fluorescence mapping and differential absorption microradiography to the study of the spatial distribution of impurities in materials. The collaborative experiments were to be conducted at Daresbury Laboratory with Dr. D. K. Bowen's group (Department of Engineering, University of Warwick, U.K.). Five types of specimens were identified for study: porous-sintered samples (microradiography), single crystals with composition gradients (diffraction imaging), samples with grain boundary segregation (microbeam x-ray fluorescence) coal (microradiography) and porous materials for medical implants (microradiography).

The unavailability of the Daresbury Synchrotron Radiation Source (SRS) during much of the period in which the experiments were to be conducted.

Installation of the high brightness lattice closed SRS to experimenters from October 1986-June 1987. It has also been exceedingly difficult to obtain time on the topography wiggler line; use of this line is necessary for most of the planned experiments. This has been due, apparently, to a shift in scheduling priorities: beam time has been approved for our experiments but little has been allocated for this class of experiments. Related to the lack of beam time are delays in our collaborator's microbeam fluorescence project; optics development for focusing to 10 μm beam diameter has been protracted.

A second change in the actual research performed was to substitute microtomography for microradiography. Microtomography has developed very rapidly over the last five years, and its advantages over conventional projection radiography are considerable. This, coupled with the fact that our British collaborators are leaders in the field of microtomography, dictated the shift from microradiography to microtomography. Due to the lack of beam time at Daresbury much of the collaborative microtomography has been done using laboratory radiation.

Two of the three techniques have been examined (x-ray absorption and topographic EXAFS methods) and two of the five sample types were studied in detail (porous composite samples and single crystal specimens). Studies of precipitates in coal were abandoned when we learned that other investigators were devoting large efforts [1,2]. The microfluorescence and biomaterial experiments have not yet been pursued (the former, noted above, because of instrumental problems). The following section reviews the techniques, the results of which are summarized and discussed in the third section of the report.

The fourth section describes the technological and scientific impact of the collaborative research.

2. Background

Microtomography

In computed tomography and high resolution variants termed microtomography, the spatial variation of x-ray absorption in a thin slice or cross-section of the sample is recorded for various projection directions or views, the number of which depend on the ultimate resolution desired [1]. The different views are combined via a reconstruction algorithm, and the two-dimensional map of x-ray absorption across the slice is obtained [3,4]. The three-dimensional distribution of absorption in the sample is recovered by stacking successive slices. This approach is much superior to microradiography where features of interest can be obscured if the sample is too thick or if there are too many overlapping features. Cracks and their precursors are much more readily detected with computed tomography because geometric invisibility is no longer possible (geometric invisibility occurs when a crack is viewed from directions other than its plane: virtually no contrast is produced because the path length though the material is unchanged).

Spatial resolution has always been a limitation of tomography apparatus. Medical units have resolution approaching 0.5 mm in most cases, although a specialized industrial unit with 50 μm resolution has been described for samples with dimensions on the order of inches [5,6]. Systems using ribbon-like x-ray beams (from rotating anode sources) and multiple detector arrays are limited to

spatial resolution no better than 25 μm because of the intrinsic dimness of conventional x-ray sources.

Elliott and Dover [7] used the translate-rotate scheme (the specimen is translated across a very narrow diameter beam to obtain each absorption profile, and then the sample is rotated for the next view), a microfocus x-ray generator and a 15 μm diameter collimator to obtain very high resolution microtomographs of human femoral bone. Their apparatus is shown schematically in Fig. 1. Counting time per slice was about 19 h for a 0.8 x 0.8 mm^2 sample (128 points, each counted for 10 s, per projection and 54 projections at 3 1/3° intervals per slice), and undersampling (~130 projections should have been recorded) blurred the image.

Data acquisition rates can be improved with the LHMC scanner if synchrotron radiation is used. Synchrotron radiation is produced when electrons traveling at relativistic velocities are deflected by the bending magnets, wigglers or undulators of a storage ring. The brightness of synchrotron white radiation is at least two orders of magnitude higher than that of characteristic peaks from the most powerful laboratory sources [8]. The broad spectrum of synchrotron radiation allows selection via monochromators of the most appropriate wavelengths for a given specimen; one is not limited to wavelengths of characteristic lines. Spatially-broad and well-collimated beams are natural property of synchrotron radiation and provide a considerable advantage over conventional x-ray sources. Bowen, Elliott, Stock and Dover [9], as part of this cooperative science project, used the LHMC apparatus and synchrotron radiation to study human femur bone and sintered alumina with 4 and 10 μm diameter collimators, respectively. Counting times were 1 s/position (128 positions/profile) for the 2 mm square alumina

specimen, and 64 views of the sample were recorded in about 4 h.

The LLNL and EXXON groups have chosen to use parallel data collection [1,2]. The LLNL group uses a fluorescent screen coupled through a lens to a CCD (charge coupled device) array and have obtained spatial resolution of about 5 μm at the Stanford Synchrotron Radiation Laboratory (SSRL) and at the German Electron Synchrotron Source (DESY) [1]. A schematic of their apparatus is shown in Fig. 2. Many absorption profiles are recorded simultaneously so that the data collection rate is orders of magnitude faster.

Topographic EXAFS

X-ray diffraction topography uses a nearly parallel beam of x-rays to image the diffraction and orientation contrast from crystals or from large grained polycrystalline samples. The contrast mechanisms are similar in most respects to those in TEM. In white beam topography a pattern of Laue spots is formed, each of which is an individual topograph produced by diffraction of a specific wavelength and its harmonics. Dispersion from the finite synchrotron source size or variations in the crystal's orientation (due to bending) leads to a small range of wavelengths being diffraction from different positions of the sample. If the specimen is aligned to diffract a range of wavelength encompassing that of the absorption edge of an element of the sample, large contrast differences can be observed across the specimen [10]. The technique, termed topographic EXAFS, appears to be promising for simultaneous assessment of crystalline and chemical defects in crystals. It may be able to serve as a survey tool to identify specific regions of the crystal for more detailed examination with techniques such as microbeam EXAFS.

3. Description of Samples Examined and Results Obtained

Materials

The materials examined include a commerical alumina refractory, two fiber-matrix composites and two crystals containing composition gradients. The composites studied by microtomography were an aligned-fiber metal matrix composite, SiC/Al, and a chopped-fiber ceramic matrix composite, SiC/Si_3N_4. The crystals examined for topographic EXAFS effects were GaAs, which contained a variation in stoichiometry from seed to tail, and hematite, which is suspected of having a Fe valence change near twin boundaries. Thus, a wide range of sample types were evaluated in this program.

The initial microtomography experiment was on a porous alumina refractory. This sample had pores up to two millimeters in diameter and is typical of commercially available material. The majority of microtomography, however, was on advanced composites where the information obtained would have greater scientific and technological impact.

The addition of SiC or other dispersoids (particles, whiskers or fibers) to silicon nitride matrices offers a potential for considerable improvement of fracture toughness and strength [11]. Apparently, the strong ceramic fibers can prevent catastrophic brittle failure by providing extra energy dissipation during crack advance [12]. The silicon nitride matrix/silicon carbide fiber system has received considerable attention [12-16] particularly since commercial, continuous-polymer derived SiC fibers such as Nicalon provide good chemical compatibility and degradation resistance superior to carbon fibers in high temperature oxidizing environments. Complete densification of sintered or chemical vapor infiltrated (CVI) specimens is frequently impossible, however, due to the formation of stable pores in the interior of grains or to the

enclosure of pores by growth of the matrix on surrounding fibers. The resulting lower density and large number of internal stress concentrators leads to poor mechanical properties. If the porosity cannot be eliminated during processing, most of the anticipated fracture toughness will be lost.

Continuous fiber reinforced SiC/Al MMC are designed for high temperature structural applications in the aerospace industry. Composites similar to these (but as yet undefined) are envisioned for application in the National Aerospace Plane (NASP), the Advanced Tactical Fighter (ATP), manned space stations, etc. A major effect on the mechanical properties of MMC's is the damage induced by thermal cycling. As the structure undergoes a thermal cycle, the coefficient of thermal expansion mismatch between fiber and matrix initiates fatigue damage and debonding at the fiber/matrix interface. The damage is very difficult to detect experimentally because of crack closure when the externally applied loads are removed. The current methods for assessing the damage state are based upon stiffness loss [17-19]. They do not, however, provide an adequate indication of the location and quantity of damage present. Knowledge of the crack initiation and propagation stages, a measure of the total quantity of damage present and a descriptive model for damage evolution are required for prediction of the remaining life of a structure.

The advanced composites which were studied with microtomography were random fiber SiC/Si_3N_4 CMC and continuous fiber SiC/Aℓ MMC. The SiC fibers were 15 and 142 μm in diameter for the CMC and MMC materials, respectively. The CMC was studied to establish sensitivity of microtomography to processing defects, and the MMC was studied to define damage/crack detectability limits for different levels of spatial resolution.

The crystals examined for topographic EXAFS effects were GaAs and Fe_2O_3. Variations in valence of the Fe atoms were expected across the crystal as were variations in stoichiometry in the GaAs crystal. The GaAs crystal was a longitudinal slab from a melt-grown crystal. Variations in stoichiometry produce differences in electrical properties, and it is important to minimize these differences in wafers taken from different portions of a boule. Measuring these chemical differences and comparing them to changes in electrical properties is the first step in evaluating the magnitude of needed improvements in composition control during crystal growth. Anomalous contrast was observed near twin boundaries in topographs of the Fe_2O_3 crystal, and one possible source was localized changes in valence of Fe and a corresponding change in the atomic scattering factor [20]. Our experiments were to test this hypothesis.

Topographic EXAFS

These experiments were performed at the Stanford Synchrotron Radiation Laboratory (SSRL) because of the Daresbury shutdown and because of Dr. Bowen's sabbatical in the U.S. A particular reflection for each sample was chosen to minimize the harmonics contained in the topographic image (e.g. $\underline{h}$=220 for GaAs) and to enhance any topographic EXAFS contrast. The specimen was aligned normal to the beam and then rotated so that the reflection of interest was diffracted in transmission and at a wavelength near that of the absorption edge (Ga for GaAs and Fe for Fe_2O_3). Further alignment was accomplished by recording in situ transmission Laue patterns: with an incident white beam many diffraction spots are formed, each with a different wavelength.

We attempted to monitor the critical reflection using a Brimrose x-ray videocamera system, but the background was too high for the fine structure to

be resolved within the spot. We located the edges of both the GaAs and Fe_2O_3 crystals by exposing a large number of polaroids and observing whether the contrast changed after small rotations. Topographs were recorded at wavelengths on either side of the absorption edges using the minimum rotation steps of the SSRL topography camera. The change in contrast is so gradual that detailed numerical image analysis will be required before this experiment can evaluated. The image analysis will be conducted by the British group using Daresbury Laboratory facilities. The poor performance of the Brimrose videosystem (which, to be fair, may have been malfunctioning) serious hindered the progress of this portion of the program.

Microtomography

The synchrotron of microtomography alumina was conducted at the Daresbury Topographic Wiggler Station under the author's exploratory beam time grant "Synchrotron Computed Tomography Study of the Distribution of Pores in Sintered Alumina." The apparatus used is diagrammed in Fig. 1 and was designed and constructed by Dr. J. C. Elliott, London Hospital Medical College and Dr. D. K. Bowen, University of Warwick. Image reconstruction was done using software of Dr. S. D. Dover, King's College, London.*

The collimator diameter was 10 μm and the translation steps were 15 μm. The counting time was 0.75 seconds per position. Absorption was measured for x-rays with λ=0.37A by means of a post-specimen monochromator. After each profile was obtained, the specimen was rotated by 3.0 degrees, and the process

*For more information, please see the reprints [9] submitted with the annual report.

was repeated until the specimen had been viewed from 180 degrees. In the limited beam time, five slices of the 2x2x10 mm^3 alumina samples were obtained. Four of the five scans were made with a 10 μm diameter collimator and are from adjacent volumes of materials (each slice is separated by 20 μm). The other scan was recorded using a 30 μm diameter collimator. A number of pores and a crack or very large cavity intersecting the surface were seen [9].

Laboratory microfocus radiation was used to study porosity as a function of processing conditions in SiC/Si_3N_4. We recorded a large number of adjacent slices with a 10 μm diameter collimator, and differences in porosity were clearly evident. The sample shown in Fig. 3 and 4 was produced by reaction bonding, was approximately 1 mm^2 in cross-sectional area and contained considerable porosity. Each pixel in the reconstructed images is about 20 and 10 μm on a side in Fig. 3 and 4, respectively. The higher resolution in Fig. 4 is due to the larger number of views recorded for these slices. The lighter areas represent regions of low x-ray absorption, and the slices are reproduced with 256 gray levels. Adjacent slices (numbered) are separated by 20 μm, large trapped pores and highly densified volumes are clearly visible, but individual 15 μm fibers are not resolved in these tomographs. Tomographs were also recorded from a second Si_3N_4/SiC composite which had been processed to eliminate the porosity. As expected, these tomographs revealed little porosity with dimensions greater than 10 μm. A preliminary report on these results should appear shortly [21]; preprints are enclosed with this report.

One key element in the analysis is the resolution of individual fibers whose x-ray absorption is quite similar to the matrix and whose 15 μm diameter is only slightly larger than the ~10 μm pixel size. The random orientation of

the fibers necessitates three-dimensional presentation of the adjacent slices; limited ranges of absorption will be rendered so that the resulting skeletal image will emphasize connected porosity or fibers. The data also allows determination of the average density of the sample; when this part of the analysis is complete, this density can be compared with that determined by macroscopic techniques. The distribution of pore sizes is also currently under investigation. A paper for submission to an archival journal (such as Philosophical Magazine A, Journal of Materials Research or Journal of the American Ceramics Society) will follow the completion of the analysis.

Damage in a continuous fiber SiC/Al MMC has also been studied with laboratory microfocus radiation apparatus; the sample was split parallel to and between the unidirectional fibers by a wedge [22]. The resulting crack stayed between plies of fibers for the most part, although SEM micrographs of the side of the sample revealed some micro-cracking and fiber breakage. The wedge was left embedded in the sample so that a gradient of crack openings and their visibility could be studied with microtomography (Fig. 5). Resolution in the slices was quite poor due to the fact that the wedge required a large sample to collimator separation; the divergence of the beam irradiated a much larger volume than was ideal. The limit of crack detectability cannot, therefore, be determined from this data. We can, however, determine the minimum crack opening displacement which produced significant contrast for this particular pixel size. The volume fraction of crack within the pixel can be estimated from the observed contrast and can be correlated with an extrapolation of crack openings measured at different distances from the wedge. When complete, this work will also be published in the open literature.

4. Scientific and Technological Impact

The power of microtomography for processing defect characterization and for damage determination in MMC and CMC has been demonstrated. This study has shown that microtomography can lead to improved understanding of damage and of processing-related defects in composites. This is a prerequisite for improved life-prediction and process modeling, for wider use of composites and for extensive economic impact of this strategy for obtaining enhanced properties. Better understanding of damage mechanisms and their relationship to processing defects will be the first step to a new generation of high performance composites. The nondestructive nature of tomographic "sectioning" of the sample allows the same specimen to be studied multiple times during processing or deformation tests and allows the clearest identification of the mechanisms controlling macroscopic properties. Sample-to-sample variability, which often plagues composite studies, can now be eliminated.

Higher resolution must be obtained, and better means for presenting the three-dimensional data must be devised. Correlation of tomographic images with well-understood and accepted techniques such as fractography are necessary for the confident interpretation of results. Development of this type of database on relatively complex materials such as composites will lay the groundwork for widespread, routine NDE of composites and of monolithic materials with these techniques. Achievement of 1 μm resolution over millimeter-wide dimensions will fill the gap between electron microscopy and statistical sampling techniques such as small angle scattering on the one hand and macroscopic measuring techniques on the other. The previously inaccessible size range is where microscopic mechanisms link with macroscopic behavior (and associated continuum models), and,

as such, its study is critical for physically based modeling.

Progress in the fundamental understanding of damage accumulation in CMC, PMC and MMC appears very likely if microtomography is developed further. Advances in the technique's sensitivity and spatial resolution must, therefore, be an important goal. Increasing the volume of material which can be studied with microtomography is critical if it is to be used with specimens for which fracture mechanics calculations are valid (e.g. 1 cm^2 cross-sections instead of the 2 mm^2 which are presently feasible). Resolution of 1 μm and contrast better than 4% are goals which can be met in the next few years. The examination of larger diameter samples will also be possible, and it is not too optimistic to envision microtomography studies of samples with 5 mm or greater diameters. Another issue central to NDE of structural composites and to understanding basic failure mechanisms is the role of the loading state in the detectability of cracks. It is essential to know whether complex loading stages will be needed for routine NDE imaging. If damage is hidden by the removal of loads (if cracks or fiber-breaks close when the imposed strain is released), the amount of "invisible" damage or its fractions of the total damage should be established. Finally, the relationship of damage accumulation to processing flaws or to fiber arrangement must be determined.

Effort should concentrate, therefore, on the fundamental aspects of damage initiation and accumulation, on the link between microscopic features and macroscopic mechanical behavior of composites and on microstructure-based modeling of phenomena such as stiffness loss. The understanding developed will be a key to improved composite design and processing, and industrial interactions will be essential in assuring improvements in the next generation of composite

materials. Making use of and refining instrumentation advances made elsewhere, promises exceptional scientific and technological advances for a very modest level of effort. Interpretation of data will be much clearer because the sample samples can be examined many times during the course of the deformation test. This approach of microtomographic damage characterization will, therefore, provide exciting, and perhaps unprecedented, advances in understanding damage initiation and accumulation.

References

1. J. H. Kinney, Q. C. Johnson, U. Bonse, M. D. Nichols, R. A. Saroyan, R. Nusshardt, R. Pahl and J. M. Brase, MRS Bul. **XIII**#1 (1988) 13.

2. B. P. Flannery, H. Deckman, W. Reberge and K. D'Amico, Science **237** (1987) 1439.

3. G. T. Herman, Image Reconstruction from Projections: The Fundamentals of Computerized Tomography, Academic Press (1980).

4. A. C. Kak and M. Slaney, Principles of Computerized Tomographic Imaging, IEEE Press (1988).

5. F. H. Seguin, P. Burstein, P. J. Bjorkholm, F. Homburger and R. A. Adams, Appl. Optics **24** (1985) 4117.

6. R. A. Armistead, Advanced Matls. and Processes/Metals Progress, March 1988, p. 42.

7. J. C. Elliott and S. D. Dover, J. Microscopy **138** (1985) 329.

8. U. Bonse, in: Characterization of Crystal Growth Defects by X-ray Methods (1980) 298.

9. D. K. Bowen, J. C. Elliott, S. R. Stock and S. D. Dover, in: X-ray Imaging II **SPIE 691** (1986) 94.

10. D. K. Bowen, S. R. Stock, S. T. Davies, E. Pantos, Haydn Chen and H. V. Birnbaum, Nature **309** (1984) 336.

11. S. T. Buljan, J. G. Galdoni and M. L. Huckabee, Ceramic Bul. **66** (1987) 347.

12. J. Homeny and W. L. Vaughn, MRS Bul. Oct. 1/Nov. 15 (1987) 66.

13. T. -I. Mah, M. G. Mendiratta, A. P. Katz and K. S. Madiyasni, Ceramic Bul. **66** (1987) 304.

14. R. Lundberg, L. Kahlman, R. Pompe, R. Carlsson and R. Warren, Ceramic Bul. **66** (1987) 304.

15. T. L. Starr, J. N. Harris and D. L. Mohr, in Ceramic Sci Proc. **8** (1987) 985.

16. T. L. Starr, in: Tenth International Conference on Chemical Vapor Deposition (1987) 1147.

17. W. Hwang and K. S. Han, J. Composite Matls. **20** #3, (1986) 125.

18. W. S. Johnson, NASA Tech. Mem 89116, March 1987.

19. J. Aboudi, Composites Science and Technology **28** (1987) 103.

20. D. K. Tanner, private communications (1985).

21. S. R. Stock, A. Guvenilir, T. L. Starr, J. C. Elliott, P. Anderson, S. D. Dover and D. K. Bowen in: Advanced Characterization Techniques for Ceramics (in press).

22. T. M. Breunig, S. R. Stock, A. Guvenilir, J. C. Elliott, P. Anderson, S. D. Dover, D. K. Bowen and S. D. Antolovich, unpublished data.

23. S. R. Stock, J. H. Kinney, T. M. Breunig, U. Bonse, S. D. Antolovich, Q. C. Johnson and M. C. Nichols, in: Synchrotron Radiation in Materials Research (in press).

f. Presentations at National and International Meetings

1. "Microtomography os Silicon Nitride/Silicon Carbide Composites," S. R. Stock, et al., TMS-AIME, 1988 Fall Meeting, Chicago, Illinois, September 29, 1988.

2. "Microtomography of Silicon Nitride/Silicon Carbide Composites," S. R. Stock, et al., Symposium on Advanced Characterization Techniques for Ceramics (Am. Cer. Soc./MRS), San Francisco, California, October 24, 1988.

3. "Application of Microtomography to the Study of Ceramic and Metal Matrix Composites, S. R. Stock, Symposium on Thermal and Mechanical Behavior of Ceramic and Metal Matrix Composites (ASTM), Atlanta, Georgia, November 8, 1988.

4. "Application of Microtomography to Composites," S. R. Stock, et al., Symposium V: Synchrotron Radiation in Materials Research (MRS), Boston, Massachusetts, November 30, 1989.

5. "Microtomography of Damage in SiC/Al Continuous Fiber Composites," T. M. Breunig, S. R. Stock, et al., TMS-AIME, 1989 Annual Meeting, Las Vegas, Nevada, February 28, 1989.

Proposals Resulting from Program Accomplishments

1. "US-UK Cooperative Research - Novel X-ray Methods for Characterization of the Spatial Distribution of Inhomogeneities in Materials," NSF INT-8814774, $15,067, 11/1/88-4/30/92.

2. "US-France Cooperative Research: Characterization of Damage in Composites by Microradiography, Computed Tomography and Microtomography." NSF Proposal INT-8815501, not funded.

3. "Computed Tomography Apparatus for Evaluation of Consolidation during Processing of Monolithic and Composite Ceramics," E. J. Grassmann Trust, $20,000, July 1988.

4. "A Study of the Relationship between Macroscopic Measures and Physical Processes Occurring during Crack Closure," ONR, $303,107, 3/89-2/91.

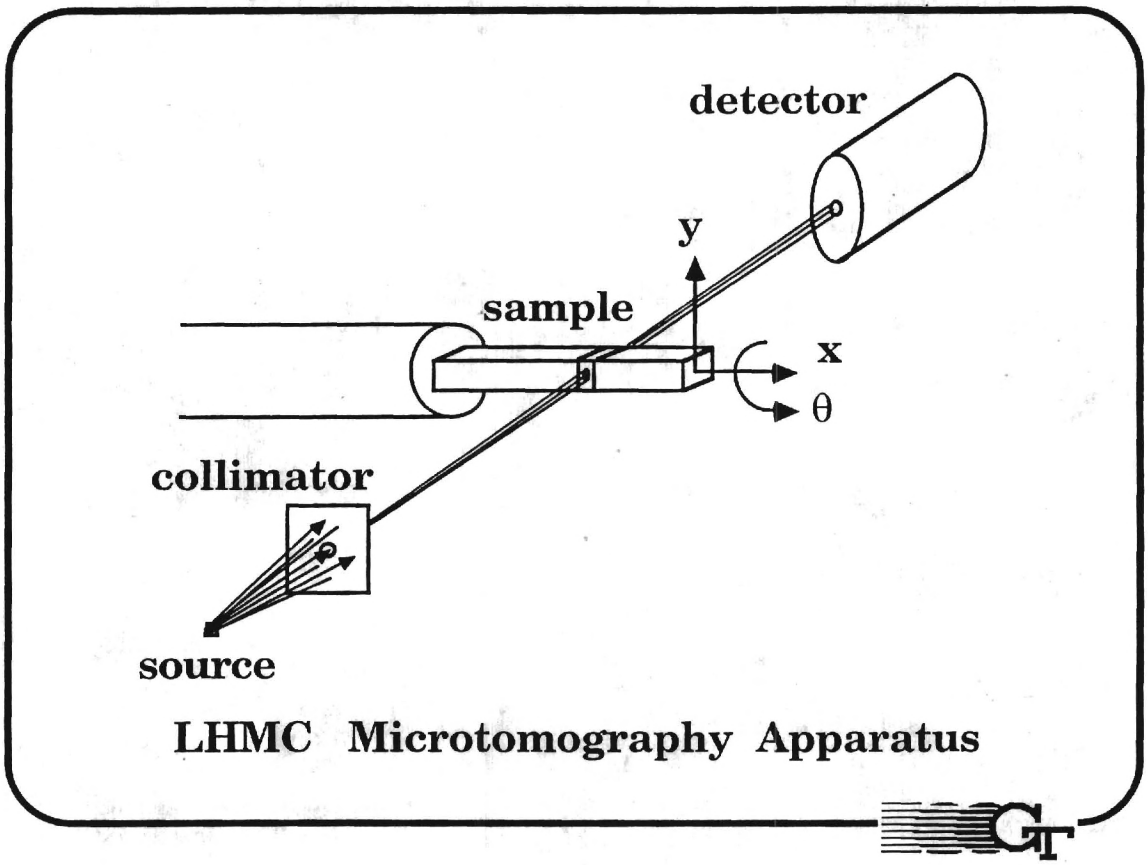

Figure 1. Schematic of the London Hospital Medical College pinhole microtomography apparatus.

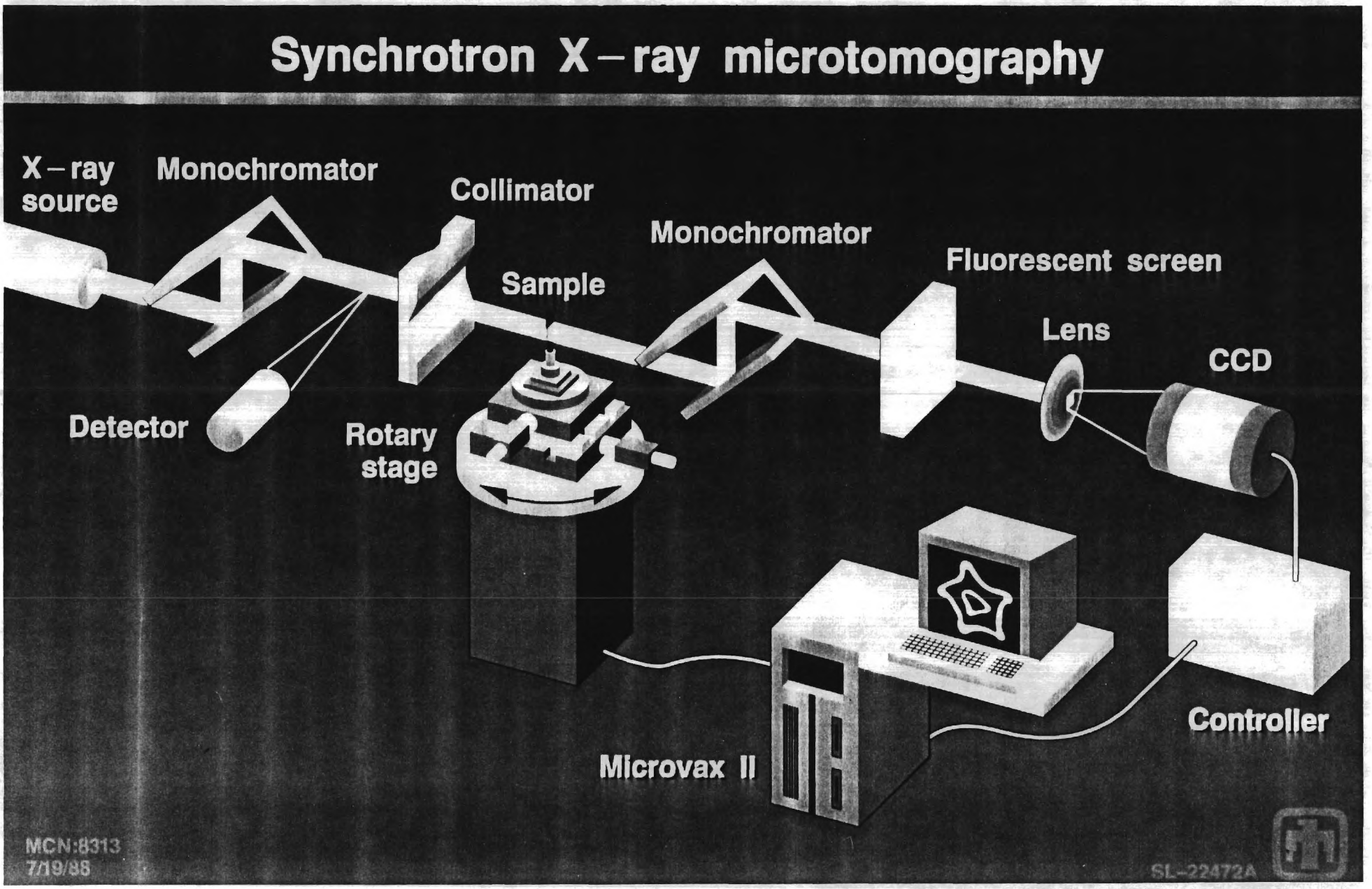

Figure 2. Schematic of the Lawrence Livermore National Laboratory CCD-based microtomography apparatus.

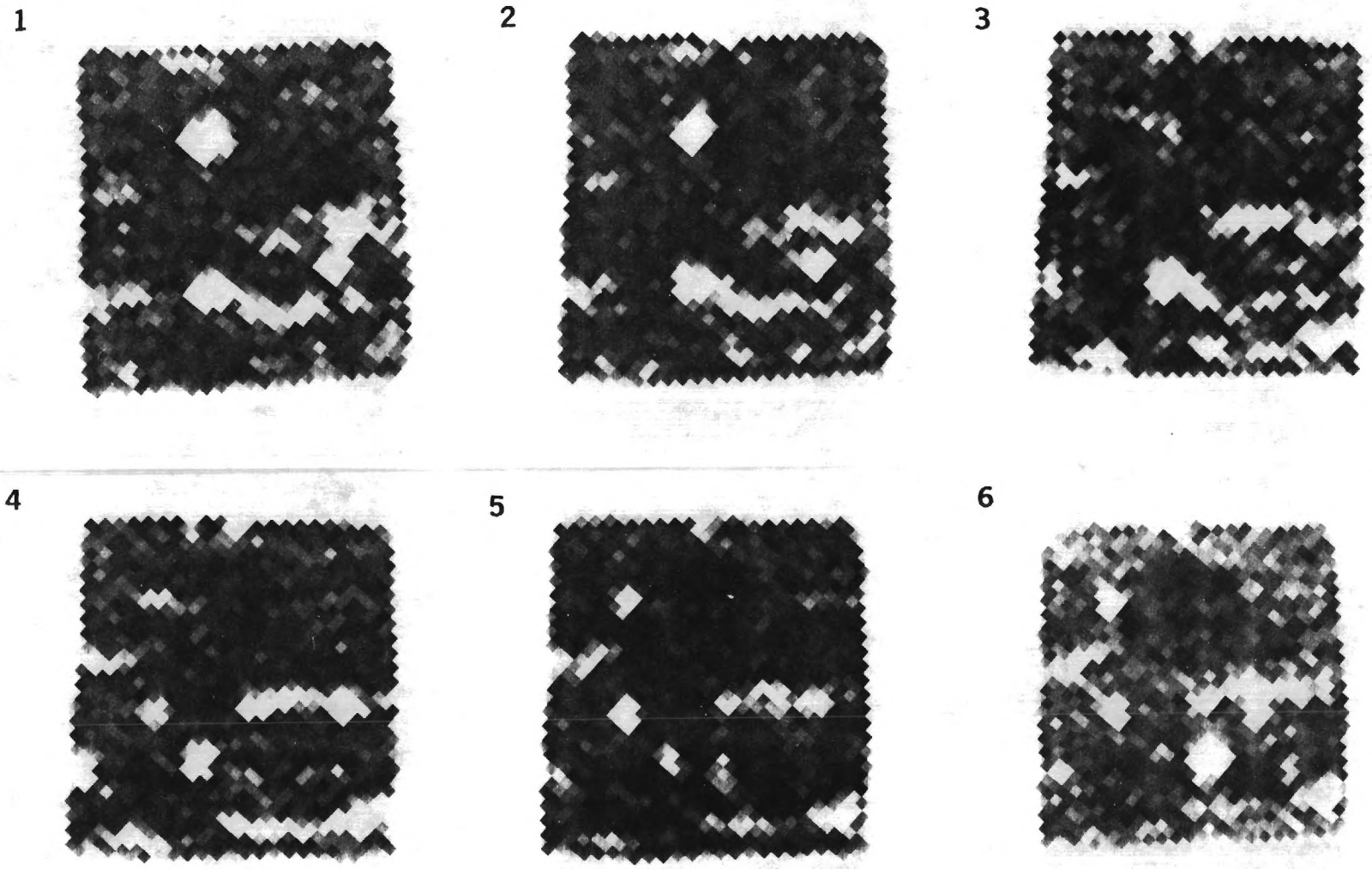

Figure 3. Silicon nitride/silicon carbide composite (1 mm^2 cross-section)
Sample N-1-4 (porous) Reconstructions SIL
10 um dia. collimator, 20 um between slices, 3 deg/ view
Darker pixels have higher x-ray absorption.

Figure 4.

Silicon nitride/silicon carbide composite (1 mm2 cross-section)
Sample N-1-4 (porous) Reconstructions SIL3
10 um dia. collimator, 10 um between slices, 1.5 deg/ view
Darker pixels have higher x-ray absorption.

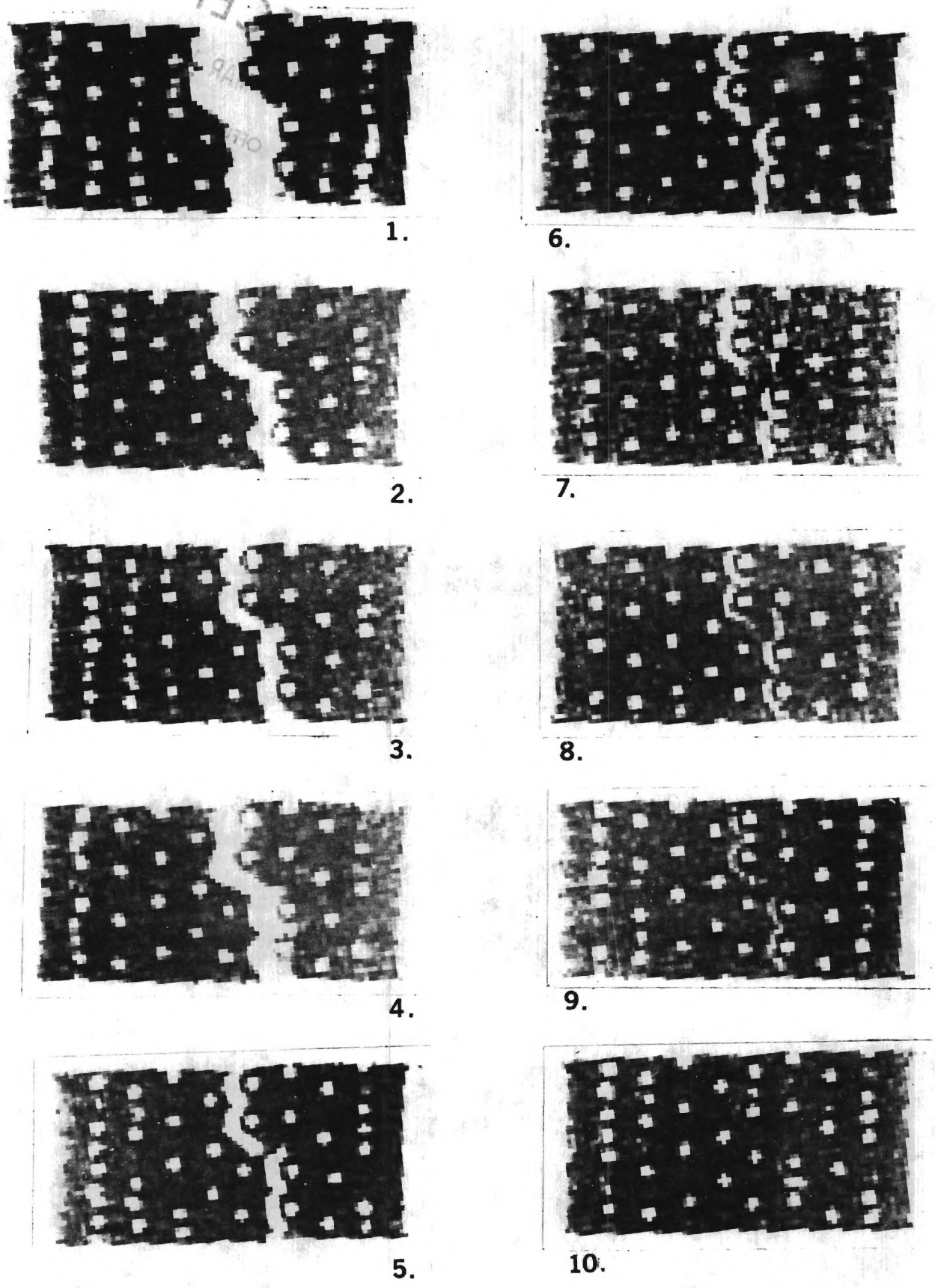

Figure 5. Slices perpendicular to a crack in a SiC/Al MMC, illustrating the variation in crack opening with distance from the wedge. The lower numbers are closer to the wedge.