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Center #: 10/24-6-R8194-0A0
Contract#: N00014-94-1-0726
Prime #: 
Subprojects #: N
Main project #: 

Project unit: MSE
Project director(s): STOCK S R

Sponsor/division names: NAVY
Sponsor/division codes: 103

Award period: 940701 to 970630 (performance) 970630 (reports)

Sponsor amount
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Title: NONDESTRUCTIVE 3-DIMENSIONAL X-RAY DIFFRACTION TOMOGRAPHIC MICROSCOPY OF...

PROJECT ADMINISTRATION DATA

OCA contact: E. Faith Gleason 894-4820
Sponsor technical contact GEORGE R. YODER/CODE 331 (703)696-4401
ONR BALLSTON TOWER ONE 800 NORTH QUINCY STREET ARLINGTON, VA 22217-5660

Security class (U,C,S,TS): U
Defense priority rating : NA
Equipment title vests with: Sponsor GIT X

Administrative comments - INITIATION OF ONR PROJECT NO. E-18-X02
Closeout Notice Date: 27-OCT-1997

Project Number: E-18-X02

Doch Id: 34224

Center Number: 10/24-6-R8194-0A0

Project Director: STOCK, STUART

Project Unit: MSE

Sponsor: NAVY/OFC OF NAVAL RESEARCH

Division Id: 3314

Contract Number: N00014-94-1-0726

Contract Entity: GTRC

Prime Contract Number:

Title: NONDESTRUCTIVE 3-DIMENSIONAL X-RAY DIFFRACTION TOMOGRAPHIC MICROSCOPY OF...

Effective Completion Date: 30-SEP-1997 (Performance) 30-SEP-1997 (Reports)

Closeout Action:

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NOTE: Final Patent Questionnaire sent to PDPI
During the first year of the program, an African-American graduate student (Mr. C.R. Patterson II) began his MS research under this program in Fall 1994. A second US student (Mr. J.D. Haase), who has worked under this program as an undergraduate, will begin his studies (aimed at a PhD) in Fall 1995. It is expected that Mr. Haase will graduate in the top one-quarter of his class at Georgia Tech. Research experience for a second undergraduate (Ms. V. Snyder) has been funded through this AASERT program. While remaining near the very top of her class academically and participating in this research, Ms. Snyder has found time to be the Editor-in-Chief of Blueprint, the award-winning Georgia Tech yearbook, and to play rugby with world-class teammates. The goal of attracting high quality students to research in areas of interest to DoD has been quite successfully realized.

Research during the first year of this program has focused on using synchrotron polychromatic microbeams to map microtexture in three-dimensions in Al-Li 2090. Development of the novel techniques required for three-dimensional mapping and assessment of the precision of these techniques has been the major activity. The attached reprint [1] illustrates the approach and, in order to avoid repetition, these details are not described here. Dr. Stock’s group made three data collections trips to the Stanford Synchrotron Radiation Laboratory (SSRL) during the year (in 12/94, 2/95 and 6/95); the trip lengths were between seven and ten days. Mr. Patterson participated in two trips and Mr. Haase in one trip.

The experiments which were part of this effort were concentrated on microbeam mapping on a number of compact tension samples. Beam diameters of 1 mm, 100 µm and 30 µm were used to examine side-grooved samples in which fatigue cracks had already been propagated. Several samples without side-grooves were also characterized before crack propagation. Most of microbeam mapping was done with the beam incident normal to the samples’ faces, but some work was done at a series of inclinations up to 60° from the normal to the surface. The angular distribution of diffracted intensity was recorded initially on film, but, during the last two trips, reusable image storage plates were used as the two-dimensional detectors. It is desirable to use image storage plates because the data is recorded directly in digital form suitable for numerical analysis, because the mapping is much more rapid than with film due to the storage plates’ far greater sensitivity and
the shorter time required to process each diffraction pattern and because storage plates have several decades greater linear range than film.

It is very important to establish the precision limits of a new technique such as polychromatic x-ray diffraction tomography. To this end, a model experiment was developed as part of Mr. Patterson's MS thesis. The goal of the experiment was to determine how well one can separate the contributions from two volume elements (in Al-Li 2090) in a thicker sample using the pattern of diffracted intensity recorded on a two-dimensional detector and its changes with increasing sample-detector separation.

Two 1 mm thick pieces were sectioned parallel to the surface of a plate of Al-Li 2090. Plate one was from the surface and plate five was from the center of the plate. As has been discussed by NRL researchers, there are two very different textures at these depths [2]. In the first part of the experiment, plates one and five were mounted on a holder so that the polychromatic x-ray microbeam would pass first through plate one and then through plate five. The plates were separated by 7 mm and the beam was normal to the plates. The angular distribution of intensity diffracted from the two plates was recorded with the image storage plate at numerous separations from the pair of plates. In principal, this information should be sufficient to allow one to determine which parts of the pattern were from plate one and which were from plate five. As at least forty grains were contributing to the image and there was considerable asterism, the situation is somewhat complicated.

As a check to the precision possible, plate five was rotated out of the beam and the pattern of diffracted intensity was recorded from plate one at the same detector positions as were used with the pair of plates. The third part of the experiment involved removing plate one and rotating plate five back into the beam in precisely the same position as it was in the first part of the experiment. Then the pattern of diffracted intensity was recorded at the same positions as were used during the first two parts of the experiment. Analysis of the results is currently underway.

References


HIGH RESOLUTION SYNCHROTRON X-RAY DIFFRACTION TOMOGRAPHY OF POLYCRYSTALLINE SAMPLES

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²Stanford Synchrotron Radiation Laboratory, SLAC, Stanford Univ., Stanford, CA

ABSTRACT

The macroscopic response of polycrystalline materials to loading depends on both the spatial distribution of strain and the variation of microtexture on the scale of 100 μm. Nondestructive measurements are needed if the three-dimensional evolution of strain is to be studied. This paper describes approaches for high resolution synchrotron polychromatic x-ray diffraction tomography of polycrystalline materials. Preliminary experiments are reported on partially cracked compact tension samples of Al-Li 2090 and on model samples of randomly-packed, millimeter-sized pieces of Si wafers. Polychromatic beams collimated to 100 μm diameter have been used, and the distribution of diffracted intensity has been collected on high resolution x-ray film as well as on image storage plates. The depths of diffracting volume elements are determined from the changes in the spatial distribution of diffracted intensity with varying sample to detector separation.

INTRODUCTION

There are many materials problems in which knowledge of the three-dimensional distribution of strain or of microtexture is important for understanding the mechanisms underlying the materials' macroscopic response to conditions such as fatigue encountered during service. In particular, to follow evolving phenomena, it is necessary to interrogate the interior of the sample nondestructively. One example, illustrated below, is the fatigue behavior of Al-Li 2090. In this alloy, fatigue crack growth rates are unusually low compared to other Al alloys. This is related to a characteristic macrotexture in the center of plates which produces a rough, asperity dominated crack face and significant crack deflection [1]. A result of the roughness is a phenomenon known as crack closure. i.e., premature contact of the crack faces during the unloading portion of the fatigue cycle which leads to a reduced driving "force" for crack propagation and lower crack propagation rates [2]. The prominent macrotexture in Al-Li 2090 has been correlated with the geometry of asperities on fatigue crack faces [1], but the role of microtexture on the path of fatigue cracks through the solid has not been investigated.

Methods of mapping microtexture or strain at sample surfaces include electron beam techniques utilizing Kikuchi patterns [3] and x-ray microbeam diffraction [4]. Due to the penetrating power of x-radiation, x-ray microbeams may also be used to map microtexture in transmission; this allows the sample's entire volume to be mapped nondestructively, an important advantage if the role of evolving strain accumulation ahead of the crack is also of interest. X-ray microbeam mapping of the three-dimensional variation of strain in polycrystals and of microtexture, i.e., x-ray diffraction tomography remains to be developed. This paper presents principles of diffraction tomography with synchrotron polychromatic x-radiation, and preliminary results are reported on the two extremes of polycrystalline samples which would be encountered, large-grained samples in which the diffraction images of individual grains can be resolved and
small-grained samples in which the grains' images form an essentially continuous, although nonuniform, distribution of diffracted intensity.

EXPERIMENTAL METHODS

X-ray polychromatic diffraction tomography can be performed in transmission using microbeams and involves the following. For ease of discussion of the technique, it will be assumed that the sample is in the form of a relatively thin plane, and the x-ray microbeam is incident normal to the plate and to the two-dimensional detector.

- Mapping in the plane of the sample involves an x-y translation of the beam. All grains within the column (x,y) illuminated by the beam will diffract different photon energies in different directions, and a two-dimensional detector measures the angular distribution of diffracted intensity \( I_0(r_0, \phi_0) \), where \( r_0 \) and \( \phi_0 \) are polar coordinates of the detector pixels and \( r_0 \) is measured from the incident beam (see Fig. 1 and 2).

- For position \((x,y)\) the depth \( z \) from which volume element \( dV \) diffracts is determined by ray tracing: varying the sample-detector distance changes \( I_0(x_0,y_0) \) as shown in Fig. 1.

- The pattern of diffracted intensity is recorded with and without a filter possessing an absorption edge in the wavelength range of interest for the sample being studied: the conflicting requirements of high transmissivity for rapid data collection and rapidly decreasing x-ray flux for energies much beyond the critical energy (4.7 keV for bending magnets at SSRL [5]) must be carefully considered for each combination of material and sample thickness. With a filter in the beam, a large change in contrast appears between images of grains diffracting x-rays with wavelengths on either side of the absorption edge [6]. This allows one to differentiate, for example, between diffraction from \{111\} by 1.00 Å x-rays and \{200\} by 0.87 Å x-rays. If an energy sensitive, high spatial resolution, two-dimensional detector were available, a filter would be unnecessary.

- Strain within a grain producing a particular diffraction spot or within the grains producing a particular portion of the diffraction pattern is determined using a post-sample analyzer crystal (see Fig. 6). Normally strain is measured with a monochromatic beam, and

![Figure 1. Depth of dV from diffraction of polychromatic x-rays.](image-url)

Figure 1. Depth of dV from diffraction of polychromatic x-rays. Different detector positions (labeled 1-3) allow diffraction of x-rays with energy \( E_i \) from near-surface dV to be separated from diffraction of energies \( E_i < E_i \) or \( E_i > E_i \) by dV deeper within the sample. Depending on detector position, diffracted intensity from interior dV can appear closer or farther from the incident beam than that of entrance surface dV despite diffracting photons with lower energy than the entrance surface dV.
The two examples described below represent the extremes which might be encountered in polycrystal samples: large-grained samples and small-grained, highly anisotropic specimens. Examination of the former allows ray tracing techniques to be refined while the latter allows techniques to be developed for mapping the spatial variation of microtexture in highly textured samples. The large-grained sample was fabricated by cleaving approximately 1 mm x 1 mm pieces of a Si wafer, by randomly orienting the pieces of Si in a 5 mm diameter capillary tube (a standard thin-walled, low absorption glass tube for powder diffraction) and by fixing the pieces in place with lacquer. The small-grained sample was a compact tension sample machined from the center 2 mm of a 12.5 mm thick plate of Al-Li 2090 T8E41 [7]. The diffraction experiments were performed with bending magnet radiation on Beam Line 2-2 of SSRL. The storage ring energy was 3.0 GeV, and data was collected at beam currents between 20 and 100 mA. The primary two-dimensional detector for diffraction tomography were Fuji HR-IIIN Imaging Plates (20 cm x 25 cm dimensions), although Kodak SR-5 film (13 cm x 18 cm dimensions) was used to record the data shown below for the polycrystalline Si sample and Polaroid P57 film was used during setup. Typical exposures for diffraction from 2 mm thick Al-Li 2090 were about 7 x 10^2 mA·sec for Type 57 film, 1 x 10^3 mA·sec for SR-5 film and 2 x 10^4 mA·sec for the image plate. Subsequent to exposure the films were developed and the image plates were read with 100 µm spatial resolution and 1024 levels of contrast in a Fuji BAS-2000 Imaging Plate Scanner. Detailed descriptions of imaging plate characteristics and scanner capabilities appear elsewhere (8,9). The SR-5 film was digitized using a Molecular Dynamics microdensitometer, and the pixel size and contrast levels were 100 µm and 1024 respectively, for the resulting digital image.

Large areas were scanned with a 1.0 mm diameter collimator while a 0.1 mm diameter collimator was used for small fields of view. The Si polycrystal data discussed below was taken with the 1 mm diameter collimator while both collimators were used with the Al-Li 2090 sample. In some exposures of the Al-Li sample a 75 µm thick Mo filter was placed between the storage ring and the collimator in order to identify where on the diffraction pattern grains were diffracting radiaun at the Mo K-edge (λ = 0.620 Å). In all cases, the imaging plate or film was centered on the transmitted beam.

RESULTS AND DISCUSSION

Microtexture within Al-Li 2090 varies significantly from position to position as the imaging plate data in Fig. 2 shows. The lighter pixels in Fig. 2 represent higher diffracted intensity, the patterns were collected with a 1 mm collimator from positions 2 mm apart, and "A" and "B" indicate the changing microtexture revealed with the beam normal to the sample surface.

Results of ray tracing for three "grains" of the polycrystalline sample are shown in Fig.
3. The data was recorded on SR-5 film with a 1 mm collimator and subsequently digitized. Each diffraction spot is an x-ray diffraction topograph of a piece of Si and maps the spatial distribution of the piece's diffracting power. A particular feature in each grain's images was used to extrapolate to the origin of the diffracted beam within the sample. As it is difficult to measure sample-detector separation directly with precision better than about 2 mm, a detector reference position is defined and the detector is positioned by hand on an optical rail relative to the reference with precision of about 0.25 mm. Least squares fitting of \( r_0 \) as a function of detector position is used to determine where within the sample the diffracted beam originated. Seven or more detector positions are used, and the standard deviation of the grain positions within the sample (i.e., the horizontal axis in Fig. 3) are 0.2 to 0.3 mm for this data.

The imaging plate data in Fig. 4 shows the difference in the distribution of diffracted intensity with and without the Mo filter (the left and right images, respectively). The positions diffracting the wavelength of the Mo K-edge show the expected change in intensity, and the numerals 1-4b label the edge positions for five different hkl. The calculated values of the diffraction angle are compared to those expected for the Al reflections (Fig. 5), and the agreement is excellent.

![Figure 2. Microtexture from positions 2 mm apart (1 mm collimator, imaging plate).](image)

![Figure 3. Separation \( r_0 \) between diffraction spots and incident beam vs. detector position.](image)
There is a small systematic difference between the two sets of angles, but this is due to a slight inaccuracy in the estimate of the detector-sample separation which was physically measured and not determined by regression as were the positions of the Si grains 2, 5 and 8 (Fig. 3).

The diffraction profiles of several grains within the large-grained Si sample and from an Al single crystal have been measured in the transmission geometry using an analyzer after the sample (Fig. 6). The Si 111 reflection from a TaSi/Si eutectic composite crystal was used as the analyzer for the 200 Al reflection shown in Fig. 6 for intensity diffracted from the analyzer crystal as a function of rotation of the analyzer crystal. The TaSi/Si crystal provides a higher bandpass and higher integrated reflectivity [10] than a Si crystal and is, therefore, preferred over Si when sample peak widths are much greater than the Darwin curve width. A 1 mm diameter collimator was used and the counting time was 1 sec per data point. Background is very high in the example shown in Fig. 6, but this can be improved by more carefully shielding the detector and analyzer crystal from the ambient radiation in the hutch. High quality diffraction peak profiles in the transmission geometry, are possible with collimator diameters of 100 μm, and data collection with smaller collimators should be possible with more complete shielding.

Figure 4. Diffracted intensity with and without Mo filter (left and right images, respectively).

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<tr>
<td>4a</td>
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<tr>
<td>4b</td>
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<tr>
<td>5</td>
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<td>59.7</td>
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<tr>
<td>or 420</td>
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Figure 5. Expected and calculated diffraction angles of six hkl for the wavelength of the Mo K-edge for the images shown in Fig. 4.

CONCLUSIONS

The development of x-ray diffraction tomography for three-dimensional microtexture and strain mapping is described. The data illustrates the several steps in diffraction tomography.
ACKNOWLEDGMENTS

This research was supported by the Office of Naval Research (Grants N00014-89-J-1708 and N00014-94-1-0306), and the experiments were performed at SSRL which is operated by the Department of Energy, Office of Basic Energy Sciences. We thank Dr. E. Morgan of the Department of Pharmacology, Emory University for use of the microdensitometer.

REFERENCES

June 28, 1996

Dr. George R. Yoder
ONR-Code 331
ONR
Ballston Tower One
800 North Quincy Street
Arlington, VA 22217-5660

Dear Dr. Yoder:

Enclosed are annual reports for AASERT Program N00014-94-1-0726, "Nondestructive 3-Dimensional X-ray Diffraction Tomography of Stress/Strain Distribution around Fatigue Cracks in Al-Li 2090."

Sincerely yours,

Stuart R. Stock
Associate Professor
GRANT NUMBER: N00014-94-1-0726

FORM A2-2

FELICITATION AWARDS FOR SCIENCE & ENGINEERING RESEARCH TRAINING (AASERT) REPORTING FORM

Department of Defense (DOD) requires certain information to evaluate the effectiveness of the AASERT program. By accepting this Grant Modification, the Grantee agrees to provide the information requested below to the Government's technical point of contact by each annual anniversary of the AASERT award date.

Grantee identification data: (R & T and Grant numbers found on Page 1 of Grant)

a. Georgia Institute of Technology
   University Name

b. N00014-94-1-0726
   Grant Number

c. met-00a1---01
   R & T Number

d. Stuart R. Stock
   P.I. Name

e. From: 7/2/95 To: 6/30/96
   AASERT Reporting Period

E: Grant to which AASERT award is attached is referred to hereafter as "Parent Agreement."

Total funding of the Parent Agreement and the number of full-time equivalent graduate students (FTEGS) supported by the Parent Agreement during the 12-month period prior to the AASERT award date.

a. Funding: $ 80,440
b. Number FTEGS: 1.5

Total funding of the Parent Agreement and the number of FTEGS supported by the Parent Agreement during the current 12-month reporting period.

a. Funding: $ 83,277
b. Number FTEGS: 2.0

Total AASERT funding and the number of FTEGS and undergraduate students (UGS) supported by AASERT funds during the current 12-month reporting period.

a. Funding: $ 33,000
b. Number FTEGS: 1.0
c. Number UGS: 0

VERIFICATION STATEMENT: I hereby verify that all students supported by the AASERT Award are U.S. citizens.

Principal Investigator

June 28, 1996

Date
Mr. J.D. Haase began his studies in Fall 1995. At the end of Spring 1996, he had completed 23 of the 30 hrs of course required for his MS degree with an overall grade point average of 3.6/4.0, despite having a very heavy research load.

Research during the second year of this program continued using synchrotron polychromatic microbeams to map microtexture in three-dimensions in Al-Li 2090. Development of the novel techniques required for three-dimensional mapping and assessment of the precision of these techniques has been the major activity. The attached preprint illustrates the progress made in the first one-half year, and, in order to avoid repetition, these details are not described here. Dr. Stock’s group made four data collection trips to the Stanford Synchrotron Radiation Laboratory (SSRL) during the year (in 2/96, twice in 5/96 and 6/95); the trip lengths were between five and seven days.

The experiments which were part of this effort were concentrated on microbeam mapping on a number of compact tension samples. Beam diameters of 1 mm, 100 µm and 30 µm were used to examine samples in which fatigue cracks had already been propagated. Several samples without side-grooves were characterized before and after crack propagation. Most of microbeam mapping was done with the beam incident normal to the samples’ faces, but some work was done at a series of inclinations up to 60° from the normal to the surface. The angular distribution of diffracted intensity was recorded on reusable image storage plates. It is desirable to use image storage plates because the data is recorded directly in digital form suitable for numerical analysis, because the mapping is much more rapid than with film due to the storage plates’ far greater sensitivity and the shorter time required to process each diffraction pattern and because storage plates have several decades greater linear range than film. Access to the image plate reader has been a major problem during the last six months: on two of the runs reader failure or access problems limited data collection to only 20% of what would be normally collected. The laboratory (SSRL) has moved rapidly to address these problems and we are confident that matters will improve.
It is very important to establish the precision limits of a new technique such as polychromatic x-ray diffraction tomography. To this end, a model experiment was developed as part of Mr. Haase's MS thesis. The goal of the experiment was to determine how well one can separate the contributions from adjacent volume elements (i.e., 40 µm thick grains in Al-Li 2090) in a thicker sample using the pattern of diffracted intensity recorded on a two-dimensional detector and its changes with increasing sample-detector separation. A wedge-shaped piece was sectioned parallel to the surface of and from the center of a plate of Al-Li 2090. Analysis of this data is presently underway, but the preliminary results are very encouraging. A new image plate reader should be coming on line with roughly twice the spatial resolution of the present system, and this should further improve depth resolution.
HIGH RESOLUTION SYNCHROTRON X-RAY DIFFRACTION TOMOGRAPHY
OF LARGE-GRAINED SAMPLES

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¹School of Materials Sci. and Eng., Georgia Inst. of Technology, Atlanta, GA
²Stanford Synchrotron Radiation Laboratory, SLAC, Stanford Univ., Stanford, CA

ABSTRACT

In order to understand the macroscopic response of polycrystalline structural materials to loading, it is frequently essential to know the spatial distribution of strain as well as the variation of micro-texture on the scale of 100 µm. The methods must be nondestructive, however, if the three-dimensional evolution of strain is to be studied. This paper describes an approach to high resolution synchrotron x-ray diffraction tomography of polycrystalline materials. Results from model samples of randomly-packed, millimeter-sized pieces of Si wafers and of similarly sized single-crystal Al blocks have been obtained which indicate that polychromatic beams collimated to 30 µm diameter can be used to determine the depth of diffracting volume elements within ±70 µm. The variation in the two-dimensional distribution of diffracted intensity with changing sample to detector separation is recorded on image storage plates and used to infer the depth of diffracting volume elements.

INTRODUCTION

The techniques for nondestructively determining the three-dimensional distribution of microtexture have been outlined previously [1]. In this approach, the distribution of diffracted intensity on a two-dimensional detector is recorded for a number of well-defined sample-to-detector separations. By measuring the separation \( r_i \) between a given diffraction spot in the transmission Laue pattern and the position where the incident polychromatic beam is incident on the detector for each sample-to-detector separation \( d_i \) and using least-squares fitting of \( r_i \) vs. \( d_i \), each diffracted beam is traced back to its origin within the sample. In other words, for a given \( x,y \) position on the sample, the depth \( z \) of the several grains intercepted by the incident beam are determined by ray-tracing. This is a tomographic method in the sense that multiple projections of diffracted intensity recorded by the detector are used to reconstruct the three-dimensional distribution of grain orientations within the sample.

Image plates, used in this work, are an ideal detector for this application, offering a wide field of view, much higher sensitivity than film and a dynamic range far wider than that of film [3,4]. The two model samples examined represent extremes which might be encountered: samples containing little strain but producing patterns which are rich with diffraction spots (Si single-crystal cubes) and samples containing heavily strained grains and producing streaks in the diffraction patterns (Al single-crystal cubes). The precision in determining depths of diffracting grains were investigated for the Si and Al samples using 100 and 30 µm diameter collimators. Typical data from the Si sample is used to illustrate the procedures and the precision which can be obtained for large-grained samples using image plates.
Two samples were examined in this study. The first consisted of ~1 mm pieces of a Si wafer dropped into a 5 mm diameter thin-walled glass capillary tube. The second consisted of similarly sized pieces of an Al crystal. Lacquer was used to fix the pieces in place.

The diffraction experiments were performed with bending magnet radiation on Beam Line 2-2 of SSRL. The storage ring energy was 3.0 GeV, and data was collected at beam currents between 20 and 100 mA. The two-dimensional detectors for diffraction tomography were Fuji HR-IIIN Imaging Plates (20 cm x 25 cm dimensions), and exposures were on the order of 2 x 10^3 and 1 x 10^4 mA·sec for the 100 and 30 μm collimators, respectively. In all cases, the imaging plate was centered on the transmitted beam. Subsequent to exposure the image plates were read with 100 μm spatial resolution and 1024 levels of contrast in a Fuji BAS-2000 Imaging Plate Scanner. Detailed descriptions of imaging plate and scanner capabilities appear elsewhere [3,4].

Transmission Laue patterns were recorded with a 100 μm diameter or a 30 μm collimator at a number of different positions x,y on each sample (Fig. 1). Sample translation was controlled with precision better than 10 μm using two sample translators. The image plates were positioned at 6-8 different sample-to-detector separations d with a third linear translation table. One should note it is relatively difficult to measure d directly; instead the separation between successive exposures was set with a precision of about 10 μm, and d was determined by ray-tracing. As a result, the horizontal axes of r vs. d plots (e.g., Fig. 4) are given relative to the translator zero position; depths within the sample are found by comparing the different positions. As relatively few diffraction spots were observed in the Laue patterns of the Al sample, r was measured between the position or positions within each spot having maximum intensity and the position (on the image plate) of the transmitted beam for each sample-to-detector separation. Least-squares fitting was used to fix the depth from which the diffracted beam originated. In the case of the Si sample, the large number of spots dictated that it was more efficient to fit the spots of a zone with an ellipse and that the extrapolation be performed using each ellipse’s foci. A 75 μm thick Mo filter was placed between the storage ring and the collimator in order to identify where on the diffraction pattern grains were diffracting radiation at the Mo K-edge (λ = 0.620 Å); this was used primarily with the Al sample.

Fig. 1. Schematic of the ray-tracing method.
RESULTS AND DISCUSSION

Figure 2 shows a typical diffraction pattern recorded with the 30 \( \mu \text{m} \) diameter collimator from sample containing the Al cubes. The large amount of strain in each grain produces diffraction spots which are, in fact, streaks. The number beside each streak identifies it for ray-tracing and for correlation with other streaks from the same grain. Figure 3 shows a typical pattern from the Si sample recorded with a 100 \( \mu \text{m} \) diameter beam; the ellipses are superimposed to indicate the zones used in ray-tracing. Figure 4 shows an example of \( r_i \) as a function of \( d_i \) for the Si sample. Eight positions were used (left hand plot), and the results of the extrapolation to \( r_i=0 \) indicated that six grains (right hand plot) were responsible for producing the zones identified in Fig. 3. The uncertainty in each grain depth averaged about \( \pm 70 \mu\text{m} \) (one standard deviation) for both the Al and Si samples; and, surprisingly, there was no significant difference for the two collimator sizes. A detector with finer pixel size may be required to reveal differences between the two collimator sizes. The spread in positions identified for streaks or zones from the same grain was found to be no greater than \( \pm 60 \mu\text{m} \), and the range averaged about \( \pm 30 \mu\text{m} \) for all the grains investigated in the two samples. At first glance it may seem unexpected that the origin of streaks could be found with the same precision as sharply-defined spots; using the pixels of each streak with the maximum intensity, for example, for the extrapolation is the key to obtaining greatest precision. These pixels were readily found in exposures at different \( d_i \), and the procedure appears to be very robust. Equivalent precision could be obtained using the position within each streak diffracting the wavelength of the absorption edge, but, as not all streaks diffract a range of wavelengths spanning the absorption edge, this would limit the number of streaks which could be used and would increase the required exposure times by a factor of three. Microtomography of the two samples revealed that cubes were present at the positions found to originate the streaks and zones (e.g., Fig. 4), and no additional material was found to be present in the incident beam paths investigated.

![Fig. 2 (left) Diffraction pattern comprised of streaks from the sample containing Al cubes and Fig. 3 (right) Diffraction pattern of the Si cubes sample with zones indicated by ellipses.](image-url)
CONCLUSIONS

The investigation of two model samples comprised of single-crystal cubes in a thin-walled container revealed that polychromatic synchrotron x-ray microbeams can be used to determine the depths originating diffracted intensity within uncertainties of ± 60 µm in depth, at least for large-grained samples. Large-grained samples are easier to analyze than small-grained samples because there are relatively few, higher intensity diffraction spots; whether the long beam paths through large grains contribute significantly to the uncertainty in depth is being investigated.

ACKNOWLEDGMENTS

This research was supported by the Office of Naval Research (Grants N00014-94-1-0306 and N00014-94-1-0726), and the experiments were performed at SSRL which is operated by the Department of Energy, Office of Basic Energy Sciences.

REFERENCES


Fig. 4. Plot of $r_1$, separation between ellipse foci and transmitted beam position, as a function of $d_1$, distance between sample and film, (left); and origins of diffracted beams revealed by ray-tracing (right). The positions on the horizontal axis are those on the translator, and the 5 mm diameter sample lies between 606.5 and 611.5 mm positions.
October 17, 1997

Dr. George R. Yoder  
ONR-Code 331  
ONR  
Ballston Tower One  
800 North Quincy Street  
Arlington, VA 22217-5660

Dear Dr. Yoder:

Enclosed are annual reports for AASERT Program N00014-94-1-0726, "Nondestructive 3-Dimensional X-ray Diffraction Tomography of Stress/Strain Distribution around Fatigue Cracks in Al-Li 2090."

Sincerely yours,

Stuart R. Stock  
Associate Professor
Stuart R. Stock
Associate Professor
School of Materials Science and Engineering
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Three US citizens' graduate work was supported during the past year: Mr. J.D. Haase, Mr. J.R. Witt (2 quarters) and Mr. George Butler. The first two students concentrated on microtexture measurements in Al-Li 2090 T8E41 while the third followed a tangential line investigation on microtexture development within grains in copper samples after complex loading histories. Mr. Witt took a job with Boeing in June 1997, Mr. Butler will finish his PhD in March, 1998 and Mr. Haase will leave in December 1997 to found a CNC machining company. Three US citizens have been employed as undergraduate research assistants: Mr. Justin Clark, Ms. Stephanie Sprague and Mr. Tony Watt. All three are currently enrolled in the School, although Justin has begun MS work.

Research during the third of this program continued using synchrotron polychromatic microbeams to map microtexture in three-dimensions in Al-Li 2090. There were two very important advances made during the past year and both have been communicated to the community in a recent national meeting presentation and in two manuscripts submitted for peer review. First, through x-ray microbeam diffraction mapping, the center portions of plates of Al-Li 2090 T8E41 were found to consist, to a significant degree, of regions where the grains were so highly oriented that they might be accurately termed near-single-crystalline. The centers of these plates are well known to have a very sharp, well developed average texture or macrotexture, but the extent to which these grains are clustered together and the extent to which the material consists of these quite large, discrete regions does not appear to have noted previously. Identification of this type of "mesotexture" appears to an important clue in efforts to improve fatigue prediction methodologies for a wide range of alloy systems.

The second main advance is that transitions in microtexture were correlated with asperity formation in samples fatigued under $R=0.1$ ($\sigma_{\text{min}}/\sigma_{\text{max}}$). Preliminary analysis of x-ray microbeam data collected during the past year for several cracked samples may provide guidance to improving the isotropy of mechanical properties of Al-Li 2090 T8E41. A preprint of a paper under peer review for Advances in X-ray Analysis is attached. A second, more detailed paper will be submitted to Acta Mat within about one week.

In research somewhat tangential to the main effort under this program, the microbeam techniques were found to provide a rapid method of quantifying the amount of microtexture on the subgrain
level from copper samples which underwent complex loading histories. Quantifying this subgrain microtexture is important to understanding why the various numerical models of texture evolution predict much sharper textures than are observed experimentally. This very exciting work indicates that the x-ray microbeam diffraction mapping of texture can be done at a synchrotron radiation source. We have demonstrated that data for the range of orientations within ten or more grains can be acquired within a couple of hours in a form which allows direct interpretation. A minimum of sample preparation is required, opening the possibility of observing the same collection of grains throughout their evolution. The information provided is roughly equivalent to that obtained through time-consuming transmission electron microscopy or through orientation imaging microscopy with a scanning electron microscope.
Three dimensional Microbeam Diffraction Tomography

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ABSTRACT
The macroscopic response of polycrystalline materials to loading depends on both the spatial distribution of strain and the variation of microtexture on the scale of 10 - 100 µm. Nondestructive measurements are needed if the three-dimensional evolution of strain is to be studied. This paper describes the approach developed for high resolution synchrotron polychromatic x-ray diffraction tomography of polycrystalline materials. Pinhole collimators have been used to produce microbeam diameters of 100, 30 or 10 µm. The angular distribution of diffracted intensity is collected on image storage plates, and changes in the spatial distribution of diffracted intensity with varying sample to detector separation are used to determine the depth of the diffracting volume element which produced a particular portion of the pattern. Results for several types of samples are summarized here, including: a five millimeter diameter capillary tube filled with randomly-packed, millimeter-sized pieces of an Si wafer, a similar capillary tube filled with millimeter sized pieces of Al crystals, and plates and wedges of Al-Li 2090 with different orientations and thicknesses.

INTRODUCTION
There are many materials problems in which knowledge of the three-dimensional distribution of strain or of microtexture is important for understanding the mechanisms underlying the materials' macroscopic response to conditions such as fatigue cycling. In particular, to follow evolving phenomenon, it is necessary to interrogate the interior of the sample nondestructively. Such non-invasive probing of samples' interiors is necessary, for example, in the study of fatigue crack paths and propagation rates in Al-Li 2090. In this alloy, fatigue crack growth rates along certain plate orientations are unusually low compared to those of other Al alloys [1,2]. This is related to a characteristic macrotexture in the center of plates which produces a rough, asperity dominated crack face and significant crack deflection [3]. A result of the roughness is a phenomenon known as crack closure, i.e., premature contact of the crack faces during the unloading portion of the fatigue cycle which leads to a reduced driving "force" for crack propagation and lower crack propagation rates [4]. The prominent macrotexture in Al-Li 2090 has been correlated with the geometry of asperities on fatigue crack faces [3], but the role of microtexture on the path of fatigue cracks through the solid has not been investigated.

Methods of mapping microtexture or strain at sample surfaces include electron beam techniques utilizing Kikuchi patterns [5] and x-ray microbeam diffraction [6]. Due to the penetrating power of x-radiation, x-ray microbeams may also be used to map
microtexture in transmission, this allows the sample's entire volume to be mapped nondestructively, an important advantage if the evolving strain accumulation ahead of the crack is also of interest. X-ray microbeam mapping of the three-dimensional variation of microtexture is the focus in this report, and the principles of diffraction tomography with synchrotron polychromatic x-radiation are reported below along with a brief summary of the results to date [7-10].

One particular goal of this work is determining whether ray tracing and current image plate technology allow location of adjacent grains within the interior of samples of engineering materials. If the adjacent grains cannot be located with sufficient certainty, then location of diffracting volume elements within two or three grain diameters may be sufficient for understanding why specific fatigue crack paths occur in complex materials like Al-Li 2090.

APPROACH
Mapping in the plane of the sample is quite straight-forward: a simple x-y translation of the beam across the sample is used. All grains within the column (x,y) illuminated by the beam will diffract different photon energies in different directions, and the angular distribution of diffracted intensity $I_d(r_d, \phi_d)$ is recorded on a two-dimensional detector, where $r_d$ and $\phi_d$ are polar coordinates defining the position of each pixel on the detector where intensity $I_d$ is measured. Note that $r_d$ is defined relative to the position of the incident beam and that the incident beam is normal to the detector plane.

For any position (x,y) the depth z from which volume element dV diffracts is determined by ray tracing: varying the sample-detector distance produces changes in $I_d(x_d, y_d)$ if the dV within the irradiated column of material are not identical. This is a tomographic method in the sense that the three-dimensional distribution of grain orientations in the sample is reconstructed from multiple projections of diffracted intensity recorded by the detector.

The pattern of diffracted intensity is recorded with and without a filter possessing an absorption edge in the wavelength range of interest for the sample being studied: the conflicting requirements of low absorption for rapid data collection and rapidly decreasing x-ray flux for energies much beyond the critical energy (4.7 keV for bending magnets at SSRL [11]) must be carefully considered for each combination of material and sample thickness. With a filter in the beam, a large change in contrast appears between images of grains diffracting x-rays with wavelengths on either side of the absorption edge [12]. This allows one to differentiate, for example, between diffraction from {111} by 1.00 Å x-rays and {200} by 0.87 Å x-rays. If an energy sensitive, high spatial resolution, two-dimensional detector were available, a filter would be unnecessary.

SAMPLES AND EXPERIMENTS
Figure 1 shows a transmission Laue pattern obtained from a 2 mm thick sample cut from the center of a plate of Al-Li 2090. A 100 µm pinhole collimator was used, and about forty grains, through the thickness of the sample, are traversed by the beam. Well-defined texture is present, but individual Laue spots (or streaks, given that the sample was rolled) cannot be resolved and this type of sample is not the place to start developing such ray tracing techniques.

In the first experiments to investigate how
well depths could be determined with the ray tracing method, two model large-grained samples were investigated. One model sample was a five millimeter diameter capillary tube filled with randomly-packed, millimeter-sized pieces of a Si wafer, and the second was a similar capillary tube filled with millimeter sized pieces of Al crystals (Fig. 2). Individual Laue spots were resolved, and the deformed cubes in the Al sample and the essentially undeformed cubes in the Si sample represented the two extremes of deformation which might be encountered in polycrystalline samples.

The thinner portion of a wedge-shaped sample of recrystallized Al-5% Cu was studied after experience was gained on the capillary samples. The diameter of the equi-axed grains averaged about 250 µm, and the spots were relatively sharp, indicating little strain was present. Note that examining wedge-shaped samples allows one to easily vary the number of grains and, hence, the range of thicknesses, contributing to a given set of images.

Wedge-shaped samples from the central 2 mm of Al-Li 2090 plates are currently being examined. Two geometries are of interest (Fig. 3): the first with the grains in the same orientation relative to the incident beam as in compact tension samples (i.e., as in Fig. 1) and the second with the beam parallel to the plane of the grains. The latter geometry greatly decreases the number of grains traversed by the beam; this simplifies the ray tracing analysis and also helps with identifying the extent of grains during mapping along the directions perpendicular to the incident beam.

Data were collected with polychromatic bending magnet radiation at SSRL BL 2-2 (3.0 GeV, currents between 20 and 100
mA). Pinhole collimators with 100, 30 or 10 µm diameters have been used. Fuji HR-IIIN Imaging Plates (20 cm x 25 cm dimensions) were used until the 96-97 run; exposures were on the order of $2 \times 10^3$ and $1 \times 10^4$ mA·sec for the 100 and 30 µm collimators, respectively. Subsequent to exposure the image plates were read with a pixel size and contrast in a Fuji BAS-2000 Imaging Plate Scanner. During the 96-97 run, Fuji BAS-2500 SR Imaging Plates (20 cm x 40 cm) were used and the plates were read with 50 µm pixel size and 256 levels of contrast in a Fuji BAS-2500 system. In all cases, the imaging plate was centered on the transmitted beam.

Between five and eight different sample-to-detector separations $d_i$ were used in the ray tracing. The axis of the linear translation table used to control $d_i$ was parallel to the incident beam to better than $1^\circ$. It is quite difficult to measure $d_i$ with the required precision directly; instead the separation between successive exposures was set with a precision of about 10 µm, and $d_i$ was determined by ray-tracing. As a result, the horizontal axes of $r_i$ vs. $d_i$ plots (e.g., Fig. 7) are given relative to the translator zero position; depths within the sample are found by comparing the different positions using least-squares fitting.

Relatively few diffraction spots were observed in the Laue patterns of the capillary tube containing Al cubes, the recrystallized Al plate and of the wedges of Al-Li 2090; therefore, $r_i$ was measured between the position or average of positions within each spot with maximum intensity and the position (on the image plate) of the transmitted beam for each sample-to-detector separation. Many more spots were seen from the tube containing the Si cubes, and the ellipses formed by different zones from different crystals were quite prominent. In this case, the foci of each ellipse were identified and used in the ray tracing process. Initial experiments [7-9] used a beam stop to keep the incident beam from exposing the image plate. The position of the incident beam was fixed by aligning the shadow of the beam stop in each exposure. This procedure sufficed for the capillary tube samples but was inadequate for determining the depths of different $dV$ in either the recrystallized Al sample or the Al-Li 2090 samples. For these samples the thickness of the beam stop was substantially reduced so that a weak image of the incident beam was recorded on the image plate.

The largest uncertainty in the ray tracing process may be from the positioning of the image plate in the film holder. While the image plate is clamped firmly between two aluminum plates, it may still be warped, bent or tilted. Another important requirement in recording transmission Laue patterns using synchrotron radiation is use of a neutral filter (1-3 mm of Al) before and after the image plate; without this shielding scattered radiation overwhelms the diffraction pattern.

RESULTS AND DISCUSSION

Figure 1 shows a Laue pattern of a 2 mm thick section of a compact tension sample of Al-Li 2090. Approximately 40 grains were intercepted by the beam. The streaks from individual grains are resolved in Fig. 4 which is from the wedge-shaped sample of Al-Li 2090 with the same orientation of grains as the compact tension sample (see top diagram of Fig. 3). In contrast, Fig. 5 shows a Laue pattern of a thin section of the recrystallized Al sample which was recorded with the 10 µm collimator. Note the prominent zones and relatively sharp spots in Fig. 5 compared to the elongated streaks in Fig. 4 or 1.
Results on the model sample of Al cubes and the sample of Si pieces indicate that all of the diffracted beam extrapolate back to positions within blocks of material (revealed by x-ray computed tomography [13]) [8]. The uncertainty for the origin of any given diffraction spot or ellipse was ± 70 µm. Further, the range of origins of streaks or ellipses from one grain was ± 60 µm. [8]

The situation is a bit more complicated for the recrystallized Al sample. Figure 6 shows the separation between several diffraction spots for this sample as a function of detector position. Most of the spots cluster closely together, but two of the spots are unrealistically far from the others, in fact outside the sample. Simulations have shown that this effect can be from the presence of subgrains within a single grain or of two or three spatially-separated grains with nearly identical orientations. The cumulative diffraction peak profile can be altered from one detector position to another so that, if one assumes a single origin for the diffracted intensity, the resulting origin will be displaced a significant depth from the actual; origin(s). An example of such a cluster of diffraction spots from Al-Li 2090 is shown in Fig. 7 for five detector-sample distances.

Fig. 4. Transmission Laue pattern from a thin portion of a wedge-shaped Al-Li 2090 sample, in the orientation pictured at the top of Fig. 3.

Fig. 5. Transmission Laue pattern from a thin section of a wedge-shaped sample of recrystallized Al recorded with a 10 µm diameter collimator.

Fig. 6. Origins (upper plot) determined by ray tracing for data from the sample shown in Fig. 5 (lower plot).

Fig. 7. Clustered diffraction spots from Al-Li 2090 grains with nearly identical orientations (top, Fig. 3).
Observing grains along the rolling direction in the wedge-shaped sample (bottom schematic of Fig. 3) allows the orientation of adjacent grains to be determined unambiguously. In Fig. 8, diffraction patterns from adjacent grains are shown, and from this data the elements of the rotation matrix relating the two grains can be determined. This work is just beginning, but there seems to be no reason not to be optimistic that the relationship between orientations of many adjacent grains, i.e., microtexture, will be soon available. Answering the question of whether fatigue crack path in Al-Li 2090 depends on the specific microtexture encountered by the crack or on stochastic wandering of the crack must await analysis of this data.

**SUMMARY**

Methods for three dimensional microtexture mapping were outlined, and the results to date suggest that the depths of diffracting volumes may be located to better than 100 µm, even in samples as complex as Al-Li 2090. While the ultimate depth resolution in the ray tracing procedure may prove to be insufficient to locate adjacent grains, it appears that it will be possible to locate diffracting volumes to within two or three grain diameters.

**ACKNOWLEDGMENTS**

We gratefully acknowledge the support of the Office of Naval Research (Grants N00014-94-1-0726 and -0306), and the experiments were performed at SSRL which is operated by the Department of Energy, Office of Basic Energy Sciences. We thank Dr. Zofia Rek of SSRL for her assistance.

**REFERENCES**

13. Courtesy of J. C. Elliott, P. Anderson and G. R. Davis, Queen Mary and Westfield College, University of London.
Fig. 8. Transmission Laue patterns recorded from positions 20 µm apart in a thin wedge of Al-Li 2090 in the orientation shown in the bottom of Fig. 3 (i.e., coplanar with the rolling direction.)
Final Report for AASERT Program N00014-94-1-0726, "Nondestructive 3-Dimensional X-ray Diffraction Tomography of Stress/Strain Distribution around Fatigue Cracks in Al-Li 2090."

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A number of US citizens were supported during the three years of this program. In the first year, Mr. Curtis Patterson II was working on his MS, and Mr. Jake Haase and Ms. Victoria Snyder were employed as undergraduate assistants. In year two, Mr. Patterson left Tech to take a job with Pratt and Whitney (before finishing his thesis, he may defend it in March 1998) and Mr. Haase became a graduate student in this program. Three US citizens’ graduate work was supported in the third year: Mr. J.D. Haase, Mr. J.R. Witt (2 quarters) and Mr. George Butler. The first two students concentrated on microtexture measurements in Al-Li 2090 T8E41 while the third followed a tangential line investigation on microtexture development within grains in copper samples after complex loading histories. Mr. Witt took a job with Boeing in June 1997, Mr. Butler will finish his PhD in March, 1998 and Mr. Haase will leave in December 1997 to found a CNC machining company. Three US citizens have been employed as undergraduate research assistants: Mr. Justin Clark, Ms. Stephanie Sprague and Mr. Tony Watt. All three are currently enrolled in the School, although Justin has begun MS work.

Research during the first year focused on making x-ray diffraction microbeam measurements routine. The principle focus was on how well microtexture from the outer and center parts of intact, full-sized plates of Al-Li 2090 could be separated by recording the pattern of diffracted intensity recorded on a two-dimensional detector as a function of increasing sample-detector separation. The model sample consisted of two 1 mm thick pieces sectioned parallel to the surface of a plate of Al-Li 2090. Plate one was from the surface and plate five was from the center of the plate, the plates spaced 3 mm apart and the x-ray beam was at normal incidence. As has been discussed by NRL researchers, there are two very different textures at these depths. The ray tracing approach allows one to determine within which of the plates diffracted streaks originated.

Research during the second year of this program continued using synchrotron polychromatic microbeams to map microtexture in three-dimensions in Al-Li 2090. Development of the novel techniques required for three-dimensional mapping and assessment of the precision of these techniques has been the major activity. The experiments which were part of this effort were concentrated on microbeam mapping on a number of compact tension samples. Beam diameters of 1 mm, 100 µm and 30 µm were used to examine samples in which fatigue cracks had already been propagated. Several samples without side-grooves were characterized before and after crack propagation. Most of microbeam mapping was done with the beam incident normal to the samples' faces, but some work was done at a series of inclinations up to 60° from the normal to
the surface. The angular distribution of diffracted intensity was recorded on reusable image storage plates because the mapping is much more rapid than with film due to the storage plates’ far greater sensitivity and the shorter time required to process each diffraction pattern and because storage plates have several decades greater linear range than film. The model experiment developed to explore how well one can separate the contributions from adjacent volume elements (i.e., 40 \( \mu \)m thick grains in Al-Li 2090) showed considerable problems remained with the approach; these experiments continued into the third year. A wedge-shaped piece was sectioned parallel to the surface of and from the center of a plate of Al-Li 2090. Data consisted of the pattern of diffracted intensity recorded on a two-dimensional detector and its changes with increasing sample-detector separation. Analysis of this data is presently underway, and image readout with 0.05 mm pixels improved the precision with which the measurements could be made.

Research during the third of this program continued using synchrotron polychromatic microbeams to map microtexture in three-dimensions in Al-Li 2090. Mapping with a 0.01 mm diameter collimator became rapid and routine. There were two very important advances made during the past year and both have been communicated to the community in a recent national meeting presentation and in two manuscripts submitted for peer review. First, through x-ray microbeam diffraction mapping, the center portions of plates of Al-Li 2090 T8E41 were found to consist, to a significant degree, of regions where the grains were so highly oriented that they might be accurately termed near-single-crystalline. The centers of these plates are well known to have a very sharp, well developed average texture or macrotexture, but the extent to which these grains are clustered together and the extent to which the material consists of these quite large, discrete regions does not appear to have noted previously. Identification of this type of “mesotexture” appears to an important clue in efforts to improve fatigue prediction methodologies for a wide range of alloy systems.

The second main advance in the third year is that transitions in microtexture were correlated with asperity formation in samples fatigued under \( R = 0.1 \) (\( \sigma_{\text{min}} / \sigma_{\text{max}} \)). Preliminary analysis of x-ray microbeam data collected during the past year for several cracked samples may provide guidance to improving the isotropy of mechanical properties of Al-Li 2090 T8E41. A near final draft is attached to summarize our current understanding, and it be submitted to Acta Mat within about one week.

In research somewhat tangential to the main effort under this program, the microbeam techniques were found to provide a rapid method of quantifying the amount of microtexture on the subgrain level from copper samples which underwent complex loading histories. Quantifying this subgrain microtexture is important to understanding why the various numerical models of texture evolution predict much sharper textures than are observed experimentally. This very exciting work indicates that the x-ray microbeam diffraction mapping of texture can be done at a synchrotron radiation source. We have demonstrated that data for the range of orientations within ten or more grains can be acquired within a couple of hours in a form which allows direct interpretation. A minimum of sample preparation is required, opening the possibility of observing the same collection of grains throughout their evolution. The information provided is roughly equivalent to that obtained through time-consuming transmission electron microscopy or through orientation imaging microscopy with a scanning electron microscope.
GRANT NUMBER: N00014-94-1-0726

FORM A2-2
ENTATION AWARDS FOR SCIENCE & ENGINEERING RESEARCH TRAINING (AASERT)
REPORTING FORM

Department of Defense (DOD) requires certain information to evaluate the
tiveness of the AASERT program. By accepting this Grant Modification,
bestows the AASERT funds, the Grantee agrees to provide the information
stated below to the Government's technical point of contact by each annual
versary of the AASERT award date.

Grantee identification data: (R & T and Grant numbers found on Page 1 of Grant)
a. Georgia Institute of Technology
   University Name
b. N00014-94-1-0726
   Grant Number
c. R & T Number
   Grant Number

d. _________
   P.I. Name
e. From: 7/1/96 To: 9/30/97
   AASERT Reporting Period

Next to which AASERT award is attached is referred to hereafter as "Parent Agreement."

Total funding of the Parent Agreement and the number of full-time
ment graduate students (FTEGS) supported by the Parent Agreement during
2-month period prior to the AASERT award date.
a. Funding: $80,440
b. Number FTEGS: 15

Total funding of the Parent Agreement and the number of FTEGS supported
Parent Agreement during the current 12-month reporting period.
a. Funding: $83,277
b. Number FTEGS: 10

Total AASERT funding and the number of FTEGS and undergraduate students
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a. Funding: $102,741.50
b. Number FTEGS: 10
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ATION STATEMENT: I hereby verify that all students supported by the
AASERT award are U.S. Citizens.

Principal Investigator

Date
X-ray Microbeam Mapping of Microtexture Related to Fatigue

Crack Asperities in Al-Li 2090

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ABSTRACT:

In certain orientations, Al-Li 2090 T8E41 cracks more slowly in fatigue than other aluminum alloys due to roughness-induced closure linked to the average texture or macrotexture. For this application, three-dimensional, non-destructive measurement of microtexture and strain evolution within samples was developed using synchrotron polychromatic x-ray microbeam diffraction [1-4]; its use in mapping the microtexture of fatigue crack asperities in Al-Li 2090 is the subject of this report. Groups of adjacent grains with nearly identical orientations are found at numerous locations in the plate centers, and this type of "mesotexture" appears closely tied to asperity formation. Changing arrangements of 111 diffraction spots relative to the samples' rolling and crack propagation directions are found to correspond to the transitions between a relatively planar section of the crack and an adjacent asperity.

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INTRODUCTION:

Knowledge of the three-dimensional distribution of stress, strain, microtexture, etc. is sometimes necessary in order to understand the complex macroscopic behavior of today's engineering materials. More specifically, understanding the role of microtexture and the evolution of strain in response to monotonic and cyclic loading would lead to improved models for predicting a material's macroscopic behavior. One such method under development, three-dimensional microbeam x-ray diffraction tomography, is being used to investigate Al-Li 2090, an engineering alloy with interesting properties. In this alloy, fatigue crack growth rates along certain plate orientations are unusually low compared to those of other Al alloys [5,6]. This is related to a characteristic macrotexture (i.e., the texture averaged over a large number of grains) in the center of the plates which produces a rough, asperity dominated crack face and significant crack deflection [7]. The prominent macrotexture of the center of plates of this Al alloy has been correlated with the geometry of asperities on fatigue crack faces [7], but the role of microtexture on the path of fatigue cracks through the solid appears to have received little attention.

Orientation Imaging Microscopy (OIM) is an alternative to the microtexture mapping method used in this work, and it determines crystal orientation from the distribution of backscattered electrons in a scanning electron microscope or SEM [8,9]. The relatively shallow interaction volume of electrons prevents this method from examining the interior of bulk samples without destructive sample preparation. Another drawback of the OIM method is that it requires special sample preparation; for example, polishing damage must be removed. In contrast, the x-ray microbeams described below allow the sample's entire volume to be mapped nondestructively, an important advantage if the evolving strain accumulation ahead of the crack is also of interest.

The goal of the work reported here is to understand how local changes in the microtexture lead to the low fatigue crack growth rate in Al-Li 2090. To this end, transmission Laue patterns using synchrotron x-ray microbeams were recorded at numerous positions on a compact tension sample. The commissioning of the new high brightness sources ESRF (European Synchrotron Radiation Facility) and
APS (Advanced Photon Source) have stimulated others to employ similar approaches to mapping microtexture [e.g., 10,11]. Emphasis in this paper is on identifying what features or changes in features within the diffraction patterns correlate asperity formation. In other words, the approach is to determine the location and orientation of individual grains or groups of similarly aligned grains related to asperity formation in Al-Li 2090. Comparison of the observed microtexture associated with a particular large asperity and the macrotexture of Al-Li 2090 demonstrates the present results are consistent with earlier reports.

BACKGROUND:

One result of the crack face roughness in Al-Li 2090 is crack closure, the phenomenon where the crack faces come into contact prematurely during unloading of the sample (i.e., before the minimum stress of a fatigue cycle is reached) or where the crack faces remain in contact much longer than expected during loading [12]. Crack closure, in combination with significant crack deflection which increases the total crack length, leads to a reduced driving "force" for crack propagation and lower fatigue crack growth rates [13].

In 12.7 mm thick plates of Al-Li 2090 T8E41, the macrotexture of the center of the plate is very different from that in the outer sections [7], and the surfaces of fatigue cracks are much rougher, with larger, steeper asperities in the center than in the outside of the plate. The center region texture is characterized by two strong preferred orientations {123}<634> and {110}<112> and one weak preferred orientation {112}<111>; and Yoder et al [7] have shown that the orientations of the faces of the asperities are related to this macrotexture. Using the macrotextural information contained in pole figures, it was determined that the angle between the two faces of large asperities corresponds to that between high density {111} orientations in the pole figure. Thus, the shape of asperities is a direct consequence of the macrotexture.
Given that the morphology of the crack surface relates to the macrotexture present, the question remains: whether asperities in the center of plates of Al-Li 2090 form at random within the volume of material through which the crack is constrained to grow (by the notch) or whether variation in microtexture within this volume dictates where large and small asperities develop. In other words, does the crack choose its path to avoid grains or groups of grains with certain orientations or to grow through individual grains, groups of grains or specific orientations of grain boundaries. The resulting large crack deflections and accompanying fatigue crack closure, which has been measured macroscopically [5,6] and with high resolution x-ray computed tomography [14-16], are responsible for the very low fatigue crack growth rates in the L-T orientation. Side-grooves can minimize crack deflection (Fig.1a), but very large deflections are the rule otherwise (Fig. 1b).

EXPERIMENTS:

Compact tension samples were examined in this study of microtexture: identification of the microtexture producing crack deflection and asperity formation in Al-Li 2090 requires examination of samples with geometries that can be compared with those of other investigators. The compact tension samples had a thickness of 2.7 mm and were machined from the center of plates of Al-Li 2090 T8E41; the specific dimensions are discussed elsewhere [16,17], but the scaling was in accordance with ASTM E-399-83 [18]. Fatigue cracks were grown in L-T oriented compact tension samples (i.e., loading along the L direction and crack propagation along the T direction) with $R = 0.1$ (i.e., $\sigma_{\text{min}}/\sigma_{\text{max}}$), 5 Hz frequency and haversine wave form. Most of the samples tested to date had side grooves which minimized crack deflection and allowed valid comparisons to be made with other’s observations of stress intensity range, etc. A number of samples were examined before crack propagation, several were examined after fatigue cracks had extended 6-7 mm and a few specimens were studied after fracture. This report focuses on the samples which were fractured.
In the fractured samples, the volume of material adjacent to the crack was cut from the sample so that the specimen could be examined in the transmission parallel to the rolling direction (L) (Fig. 2). With sample thicknesses (along L) greater than 3mm, the number of overlapping diffraction streaks made analysis difficult and required impractically long exposure times.

The microtexture was mapped in the various compact tension specimens by translating the sample along the two orthogonal axes perpendicular to the beam by fixed increments and recording the resulting transmission Laue pattern. Prior to microbeam data collection, the surfaces of the fractured compact tension samples were viewed optically to locate asperities so that microtexture data collection could concentrate on areas of interest. For the fractured compact tension specimens, most of the Laue patterns were recorded with the incident beam parallel to the rolling direction (L). In this "parallel" geometry (Fig. 2), the beam was translated along the short transverse (S) direction for various positions along the transverse (T) axis. The fractured compact tension specimens were also examined with the incident beam parallel to the short direction (i.e., perpendicular to the face of the plate and in the "perpendicular" geometry) in order to investigate whether asperities could be located in unfractured, unsectioned compact tension specimens. In samples where both fracture surfaces are available, matching locations on each face were studied with the x-ray microbeams.

All diffraction data were collected with polychromatic bending magnet radiation at Stanford Synchrotron Radiation Laboratory (SSRL) beamline 2-2 (3.0 GeV, beam currents between 20 and 100 mA). Pinhole collimators with 100, 30 or 10 µm diameters have been used. Exposure levels were on the order of $2 \times 10^3$ and $1 \times 10^4$ mA·sec for the 100 and 30 mm collimators, respectively. The collimator was placed 55 cm from the sample in order to minimize the effect of scatter from the edges of the pinhole. The ~20 arcseconds of vertical divergence of the beam was enough to broaden the beam from the 10 µm diameter collimator to 80 µm vertically at the sample position. Image storage plates [19,20] were used to record the polycrystalline Laue patterns. Initially, 20 x 25 cm plates were read with 100 µm pixel size
and 1024 levels of contrast in a Fuji BAS-2000 Imaging Plate Scanner. Once a Fuji BAS-2500 Imaging Plate Scanner became available, all data was collected on this system on plates with an area of 20 x 40 cm and read with 50 µm pixel resolution and 256 levels of contrast. Additional levels of contrast could be obtained in the BAS-2500 system at the cost of much larger data file sizes, but 256 levels of contrast provided adequate range for these experiments. In some of the patterns, a filter with an absorption edge in the wavelength range of interest was placed before the collimator in the beam in order to introduce a sharp change in contrast in the polycrystalline diffraction pattern [21]. The use of a filter allows one to index the pattern (Fig. 3).

RESULTS AND DISCUSSION:

In the fractured compact tension specimens, the thickness was chosen so that the number of grains in the beam path along the rolling direction (i.e., in the parallel diffraction geometry) would be minimized. The appearance of complex streaks is evident in microtexture mapping in the parallel geometry and greatly complicated the process of determining the orientation of specific diffracting regions. Since the grain size in Al-Li 2090 along the L direction is on the order of 1 mm and along the S direction is on the order of 50 µm, diffraction patterns from the samples would be expected to consist of streaks from several grains, but not several tens of grains. The separation between sampling positions was chosen so that streaks from each grain would be present in more than one diffraction pattern (i.e., to reveal gradual changes in microtexture) and ranged from 20 to 100 µm. Grouping of 111 streaks is observed in both the parallel and perpendicular geometries, and this suggests that results obtained in the two experimental geometries can be correlated. This is important since the parallel geometry allows straight-forward diffraction pattern interpretation and beam position correlation with asperity locations determined by scanning electron or optical microscopy.

Figure 4 juxtaposes a map of microbeam positions and an SEM micrograph of the fracture surface viewed along the loading axis (i.e., normal to the nominal fracture plane). One side groove is
visible at the top of the micrograph, and the crack propagated from left to right. The very large asperity described below lies adjacent to the side groove and extends almost the entire length of the micrograph. Identifying specific streaks or clusters of streaks associated with asperities in the perpendicular geometry is much more difficult without the guidance of the results of mapping in the parallel geometry.

Figure 5 shows the same area of the sample as Fig. 4, but the sample is viewed at a large angle of tilt. The white dashed line indicates the position of a scan of the beam along the sample's S direction. Note the large asperity at the top of the SEM micrograph. The center portions of two diffraction patterns are shown to the right of the fractograph, and the arrows indicate the position where each was recorded. In Fig. 3 and Figs. 5 - 9, the diamond at the center of each Laue pattern is a lead beam stop whose thickness was chosen to attenuate most (but not all) of the incident beam; and increasing diffracted intensities are indicated by the darkening of the pattern. The abrupt change of texture at the edge of the asperity is seen by the quite pronounced change in 111 streak position to the right of the beam stop. Within the volume of asperity, the orientation of the 111 streaks varied little from the upper pattern of Fig. 5. Outside the asperities, the 111 streaks either had different orientations or were not present.

A series of diffraction patterns in the parallel geometry and spanning the asperity along the S direction of the sample is shown in Fig. 6. Only the central portion of the patterns are shown, and the difference in diffraction patterns taken from the asperity volume are easily seen compared to those of nearby planar regions of the crack face. This type of mapping allows one to map out the entire asperity along both the S and T direction. Figure 7 shows a series of diffraction patterns taken in the same geometry but with the translation between exposures being along the sample’s T direction (i.e., along the length of the asperity). The microtexture revealed by the 111 streaks change little over the 250 μm of translation.

Figure 8 shows a series of diffraction patterns of the same asperity shown in Fig. 7 but recorded in the perpendicular geometry (i.e., with the incident x-ray beam parallel to the S direction and normal to
the face of the plate). The positions of diffraction patterns in Fig. 8 are separated by 20 µm translations along the sample's T direction. This demonstrates that the microtexture producing the 111 streaks parallel to or nearly parallel to the plate's T direction in the parallel diffraction orientation also can be unambiguously seen in the perpendicular geometry as horizontal 111 streaks. It is likely, therefore, that volumes of material with the proper microtexture to form asperities can be located nondestructively in samples prior to crack growth. Whether or not such volumes of material actually form asperities or significant crack deflection, however, depends on many other factors, including whether the advancing crack will intersect the volume.

It is also important to ascertain whether the microtexture inside and outside of the asperity described above is consistent with the expected average texture. Figure 9 shows an experimental 111 pole figure (after [7]) from the central portion of plates of Al-Li 2090 T8E41. The section of the diffraction patterns just inside and just outside the asperity (Fig. 5) are reproduced, and the arrows link the diffraction patterns with the orientation in the pole figure producing the characteristic streaks. Seeing that these orientations produce exit beams in the proper directions and with the proper diffraction angles requires locating the entrance and diffracted beams (S₀ and S₁₁₁, respectively) coplanar with and at an angle \( \pi/2 - \theta_0 \) from the plane normal. Consider the solid rectangle in the right portion of the pole figure as an idealized representation of orientations comprising this portion of the macrotexture, and note that the top and bottom of this region represent those orientations which will produce diffraction streaks at the greatest angle with respect to the transverse direction. Since \( S₀ \) is along \( L \) for the diffraction patterns shown to the right of the pole figure, constructing a stereographic projection with the reference direction along \( L \) (shown below the pole figure) allows straightforward comparison. The rectangle in the pole figure rotates to the position shown and exit beams \( S₁₁₁ \) corresponding to the inner-most corners of the solid rectangles are indicated by the dashed lines leading to the outermost corners of the open triangles (on the back of the projection). From this representation of the texture, one expects 111 streaks to be at an angle no greater than ±25° from \( T \); experimentally the angles are ~±20° in the upper pattern and ~
25° in the lower pattern. Note that there will be a spread of Bragg angles observed, and 90° - θ_b will be 80° for the innermost corner of the rectangle in the stereographic projection. This Bragg angle (10°) corresponds to diffraction of a wavelength of 0.812 Å, and the rest of the grains with 111 orientations represented by the rectangle are expected to diffract at shorter wavelengths. When the molybdenum filter is inserted in to the beam, the position of its K-edge (0.62 Å) intersects the 111 streaks near the transverse direction in some of the diffraction patterns, and this confirms the identification of the portion of the macrotexture related to the microtexture inside and outside of the asperity.

A consistent picture emerges of the relationship between microtexttrue, macrotexture, asperity formation and "choice" of crack path of the central portion of plates of Al-Li 2090 T8E41. Groups of adjacent, highly-oriented, plate-like grains form near-single-crystal regions within the plate, and this spatial distribution of microtexture is a specific type of mesotexture which defines favored crack paths. In other words, macrotexture describes large-scale average texture (e.g., an average over the sample), microtexture is used to describe the orientations present at the subgrain scale or in a grain-by-grain average and mesotexture is used to the average preferred orientation over an assembly of adjacent grains. The present observations are consistent with the distribution of misorientation angles measured across grain boundaries for this material [22]. The data presented above suggest that fatigue cracks tend to propagate crystallographically within or adjacent to these highly-oriented volumes and that crack deflection is likely at the boundary of such a region. The relatively high probability that adjacent pancake-shaped grains are nearly aligned leads one to expect fracture features with aspect ratios and orientations similar to that of the individual grains. The correlation between macrotexture and the {111} faces of asperities is therefore not surprising. Near-single-crystal regions consisting of multiple grains will tend to act like single crystals, slip across grain boundaries within this region will relatively easy and the resulting fracture surface will be crystallographic with {111} faces. Thus, the spatial distribution of
these highly-oriented regions appears to govern whether or not asperities are present on fatigue crack surfaces in this alloy / heat treatment and where on the surfaces these asperities form.

The observation of mesotexture is consistent with the very low fatigue crack growth rates seen in Al-Li 2090 samples tested in the L-T orientation. Crystallographic crack propagation tends to be considerably slower than transgranular cracking [23]. Further, forcing the fatigue crack out of the nominal plane of the notch would also be expected to slow propagation rates since the area of the fatigue crack would be greatly increased, while the effective crack length would be unchanged. These factors, when coupled with decreased driving "force" from roughness induced crack closure, explain the unusually low fatigue crack growth rates in Al-Li 2090.

CONCLUSIONS:

The characteristic microtexture of Al-Li 2090 T8E41 that leads to the formation of asperities and crack closure in fatigue crack growth appears to be a specific type of mesotexture present in this material: sets of adjacent grains are so highly aligned that they may be regarded as nearly single crystalline volumes. A relationship between macrotexture and a specific asperity on the fatigue crack surface was identified. Thus, the size, shape and location of these asperities was shown to depend on microtexture, mesotexture, and macrotexture. When coupled with high resolution x-ray computed tomography observations of the crack face contact as a function of crack face geometry and stress intensity (in the interior of the samples) [14-17], the present observations complete the link between the orientation of individual grains / groups of adjacent grains, macrotexture, the formation of asperities, the positions where crack faces contact as a function of applied stress and macroscopic manifestations of the crack closure process in Al-Li 2090 T8E41. Further elaboration of details such as amount and spatial distribution of near-single-crystalline regions is needed before numerically realistic models may be assayed. Fortunately gathering such information is quite straight-forward with the x-ray microbeam techniques used in this work. The development of these x-ray microtexture mapping techniques is so
encouraging that it is not imprudent to suggest that it should be possible to non-destructively explore the three-dimensional microtexture within intact macroscopic samples of complex materials such as Al-Li 2090.

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18. ASTM Standard E-399-83.
Figure 1. Crack profiles on the faces of Al-Li 2090 compact tension samples: that on one face is shown in black and on the other in gray. The crack profile at both faces of the grooved sample (CT-2) is shown in (a) while (b) shows the crack path on the front and back surface a sample without side grooves (CT 31). The notch tip is at the left.

Figure 2. Experimental geometry with incident x-ray beam parallel to the rolling direction (L). The transverse plate direction T is horizontal. The sample is a section from a fractured compact tension of Al-Li 2090. Incident and exit beams are shown, and the diffracted beams exit the fracture surface.

Figure 3. Two diffraction patterns at the same location on compact tension sample CT21. These patterns were recorded in the perpendicular geometry, (i.e., along the short plate direction), the transverse plate direction T is horizontal. A 10 µm diameter collimator was used to form the microbeam, and the pattern on the right was made by placing a 75 µm thick molybdenum filter in the incident beam path. The presence of this filter causes a sharp change in contrast for diffraction of wavelengths above and below 0.62 Å, i.e., at the wavelength of the Mo K-edge. The darker the pixel value in this figure, the greater the diffracted intensity. The white diamond in the center of the pattern is the beamstop and, by design, the incident beam penetrates it. The separation between the exit surface of the sample and the detector was approximately 245 mm.

Figure 4. Map of microbeam position (top) at the same scale as the SEM fractograph (bottom) of compact tension sample CT21 containing the large asperity described in the text and in Fig. 5-8. The viewing perspective is normal to the surface, the crack propagated from left to right and one of the side grooves appears at the top of the micrograph.

Figure 5. SEM fractograph of the region of the compact tension sample shown in Fig. 4 but at a high angle of tilt. The large asperity appears at the top, and the dashed line marks the line along which the microbeam was scanned. The central portion of diffraction patterns just within (top right) and just outside (bottom right) the asperity are shown to the right of the fractograph; the two positions are identified by the arrows. The darker the pixel value in these diffraction patterns, the greater the diffracted intensity. The white diamond in the center of the pattern is the beamstop. These diffraction patterns were recorded with a separation of about 245 mm between the exit surface and the detector.
Figure 6. A series of five diffraction patterns showing the characteristic change in microtexture in and around an asperity in CT21. The images were taken in 50 µm translation steps with a 10 µm diameter collimator beam and translation along the short rolling direction (S) across the asperity. The sample was oriented so that the incident x-ray beam is parallel to the rolling direction. Pattern b through d are from the asperity region, and the gray scale and sample-detector separation are the same as in Fig. 5.

Figure 7. A series of five diffraction patterns recorded with the 10 µm diameter collimator and showing the <111> streaks comprising the characteristic change in microtexture from an asperity region. The images are from positions within the asperity separated 50 µm apart along the transverse rolling direction (T), and the gray scale and sample-detector separation are the same as in Fig. 5.

Figure 8. Portions of perpendicular geometry diffraction patterns recorded at five different positions along the length of an asperity. This asperity was also shown in Fig. 6 and 7. Translation was in 20 µm steps along the plate’s T direction and the 111 streaks lie along this direction. A 10 µm diameter collimator was used to form the microbeam.

Figure 9. Orientation of 111 diffraction streaks related to the macrotexture of the center of plates of Al-Li 2090 T8E41. The 111 pole figure (after [7]) is shown in the upper left, and the portion of the macrotexture producing the 111 streaks associated with the asperity is represented schematically by the solid rectangle in the right side of the pole figure. The centers of two diffraction patterns show the 111 streaks just outside (top right) and inside the asperity (lower right). The L-oriented stereographic projection (lower left) is used to relate the experimentally observed 111 streaks to the macrotexture: the locus of expected diffracted beam directions (i.e., the streaks) from the orientations within the solid rectangle are indicated by the elongated triangles nearly parallel with the T axis.
Nondestructive 3-Dimensional

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Unlimited

Syndochron X-ray microbeams were used to characterize the three-dimensional distribution of microtexture associated with asperity formation during fatigue crack propagation in Al-Li 2090 T8E41. Nondestructive techniques were developed, and beams formed by a 10 µm diameter collimator were applied. Focus was on the center portion of 12.5mm thick plates where macrotexture correlates with asperity geometry. A very distinct type of mesotexture was found: multiple adjacent grains have nearly identical orientations and form substantial volumes of near-single-crystal (NSC) material. Transitions between differently oriented NSC volumes or an NSC region and more randomly oriented grains seem to bound asperities.