LA-UR- 99-617

Title: GRAIN TRACING AND STRAIN DETERMINATION IN A Be COMPACT TENSION SPECIMEN USING SYNCHROTRON RADIATION

Author(s):
Ravi Varma, MST-8, LANL
Ray Green, LANL
M.D. Garcia, LANL
P.V. Satyam, APS/ANL
W.B. Yun, APS/ANL
J. Maser, ANL
Z. Kai, ANL
B. Lai, ANL
Sunil K. Sinha, APS/ANL

Submitted to:
ICONE-7 CONFERENCE
Tokyo, JAPAN
April 19-23, 1999

Los Alamos
NATIONAL LABORATORY

7th International Conference on Nuclear Engineering
Tokyo, Japan, April 19-23, 1999
ICONE-7350

GRAIN TRACING AND STRAIN DETERMINATION IN A Be COMPACT TENSION SPECIMEN USING SYNCHROTRON RADIATION

Ravi Varma**
Materials Science and Technology (MST-8)

Ray E. L. Green and Michael D. Garcia
Engineering Sciences and Application Division

Los Alamos National Laboratory

and

P. V. Satyam, W. B. Yun*, J. Maser, Z. Kai, B. Lai, and Sunil K. Sinha**
Advance Photon Source

Argonne National Laboratory

1 ABSTRACT

X-ray synchrotron radiation of high (11 KeV) energy and high flux (10^{10} photons per square centimeter per second) has been used to measure strains and polycrystallinity in 6-mm thick polycrystalline beryllium compact tension (CT) specimens at and around the crack tip (for fatigue-precracked sample) or at chevron notch point under load or no-load conditions. We demonstrated the feasibility strain field mapping as well as determining the poly-crystallinity at or near the points of maximum load in beryllium CT specimens. The experimental techniques and results will be discussed.

7th International Conference on Nuclear Engineering
Tokyo, Japan, April 19-23, 1999
ICONE-7000

GRAIN TRACING AND STRAIN DETERMINATION IN A BE COMPACT TENSION SPECIMEN USING SYNCHROTRON RADIATION

Ravi Varma**
Materials Science and Technology (MST-8)

Ray E. L. Green and Michael D. Garcia
Engineering Sciences and Application Division

Los Alamos National Laboratory
PO Box 1663, Mailstop G770
Los Alamos, New Mexico 87545, USA

and

P. V. Satyam, W. B. Yun*, J. Maser, Z. Kai, B. Lai, and Sunil K. Sinha**
Advance Photon Source

Argonne National Laboratory
Argonne, Illinois 60439 USA

*Current affiliation is Lawrence Berkeley Laboratory, Advanced Light Source, Berkeley, California 94720.
**Persons to whom inquiries regarding this paper may be addressed.

We report a method of tracing grains in a polycrystalline beryllium, compact tension specimen by using an 11.0 keV, 250 x 250 μm^2 (spatial resolution) x-ray beam. By determining the angular deviation of the diffraction spots arising from the same grains at different applied stresses, it was possible to map changes in their relative strains. Diffraction measurements from (100) and (101) planes were carried out with a high load of about 2000 N and with a nominal load near 70 N. The ability to map the relative strain and the mosaic spread (with and without load) around the notch point is demonstrated.

1 INTRODUCTION

A truly microscopic understanding of the manner in which stresses are distributed in crystalline materials and lead to fracture can only be achieved by understanding how the magnitude and anisotropy of the stress propagates from grain to grain. For intensity reasons, earlier neutron and x-ray studies of residual stress in materials have generally been confined to fairly large-gauge volumes, encompassing large numbers of grains (Noyan and Cohen 1987). With the emergence of high-brilliance, third-generation synchrotron radiation sources, it is possible by using focusing techniques (e.g., a zone plate) to have a high monochromatic flux with beam sizes down to ~1 μm diameter (Lai et al. 1995, Wang et al. 1995). This makes it possible to study gauge volumes confined to single grains, which could lead to new insights into microstrains in materials. As a first step in this direction, we have carried out microdiffraction measurements on compact tension (CT) 6 mm thick beryllium L-T coupons at and around the notch point, as indicated in Figure 1. Beryllium is a very useful metal for specialized applications in structural and nuclear metallurgy because of its low density, high elastic modulus, elevated melting point, and high heat capacity; it is an integral part of synchrotron beam lines as an optical element (Webster and London 1979, Varma et al. 1997).

In this communication, we present a new approach to investigating residual strains in polycrystalline beryllium by tracing individually identified grains from one applied stress state to another. We use a charge-coupled device (CCD) area detector to detect the diffracted x-ray beam from a gauge volume within the specimen as defined by slits in the incident and scattered beam paths. We obtained the measurement of strain by determining the relative shift of the diffraction peaks. A weak contribution to the diffracted beam from the two edges of the specimen helped us define the sample position with respect to the individual grains being examined.

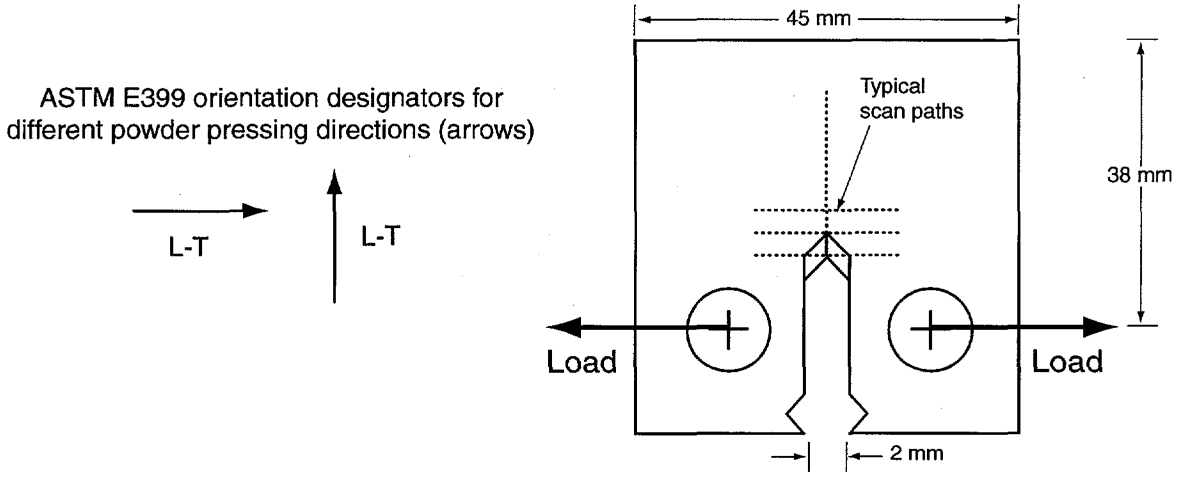

Figure 1. Some details of the compact tension specimen fabricated for stress studies in structural beryllium. Typical paths scanned in the work described here are indicated.

2 EXPERIMENT CONFIGURATION

The experiment was carried out at the 2ID-D undulator beam line of the Advanced Photon Source at Argonne National Laboratory. The schematic experimental setup is shown in Figure 2. We used a slit, S1, to predefine the beam and reduce the background in the experimental hutch, using a double-crystal monochromator (MC) to define the incident energy to be 11.0 keV (λ = 1.127 Å) with an energy resolution of 10^{-3} keV. A slit, S2, located after the MC, defined the incident beam to be 250 x 250 μm^2. We suppressed the higher-order harmonics, which come from the undulator, using a mirror before the MC. The beryllium sample, along with a load cell, was mounted on a set of precision motion stages that allowed translational and rotational manipulation. The load was changed manually. At the notch point, the sample is thinner, and hence the transmitted x-rays had lower attenuation, which made it possible to align the sample accurately. We defined the notch point to have coordinates x = 0 and y = 0, placing the z-axis along the direction of the incident beam. We also used a laser to correct any tilts that resulted from the procedures used to apply stress. A CCD detector (988 × 1024 pixels, 60 μm linear size per pixel) was mounted on the two-theta arm of a two-circle goniometer. The load frame blocked the required lines of site for some of the reflections, but we were able to appropriately orient the sample and demonstrate unfettered access to four reflections, the strongest two of which we used for this study. We placed the detector 587.6 mm from the sample and a slit (S3 in Figure 2) between the CCD active area and the sample 139.6 mm from the sample.

The pixel width in the CCD detector and the characteristics of the incident beam determine the instrumental resolution for measuring the radial and azimuthal angular deviations in the diffraction spots. We used the (100) and (101) reflections in the beryllium material with corresponding resolutions in strain ($\Delta d/d = \Delta\theta \cot\theta$) of 4.0 x 10^{-4} and 3.5 x 10^{-4}. We chose an initial load of 2000 N as adequate to generate plastic response at the notch point but low enough to minimize the possibility of fracturing the specimen. We slowly increased the load manually, monitored the calibrated load cell output until it reached this value, and then later reduced the load to a small nominal value of approximately 70 N. At both loads, the diffraction patterns from (100) and (101) planes were recorded for sampling volumes in and around the notch point by moving the specimen through the beam using the precision translation stages. These data allow the strain changes in the system to be mapped and studied.

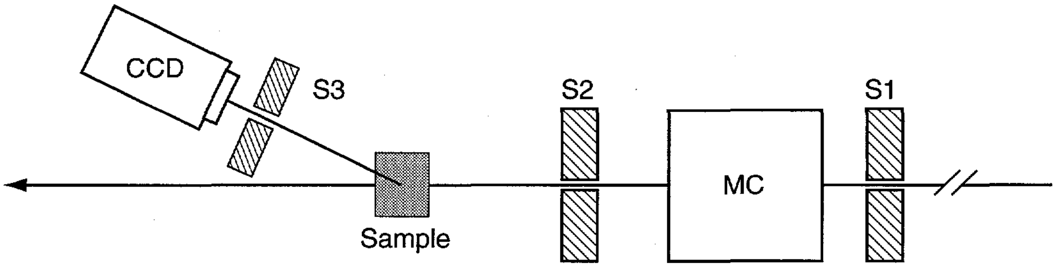

Figure 2. Schematic of the experimental setup used for the measurements.

3 RESULTS

Figures 3a and 3b show sections of representative CCD images in the region where the (100) reflections appear. The diffraction patterns shown in the figures originate from the same nominal sampling volume in the same specimen under two different loads. It is clear from these pictures that the diffraction patterns are similar in the sense that we observe many diffraction spots in nearly the same pattern except for an overall shift in both the radial and azimuthal directions. We saw this behavior in numerous such pairs of images, although isolated spots would occasionally appear in only one image. We interpret this to mean that the sample orientation may have shifted slightly during the change in load and thus shifted the overall image position but that the change in orientation is small enough so that most of the same grains approximate the Bragg condition for both loads. We have thus been able to track individual grains when we change the load on the specimen.

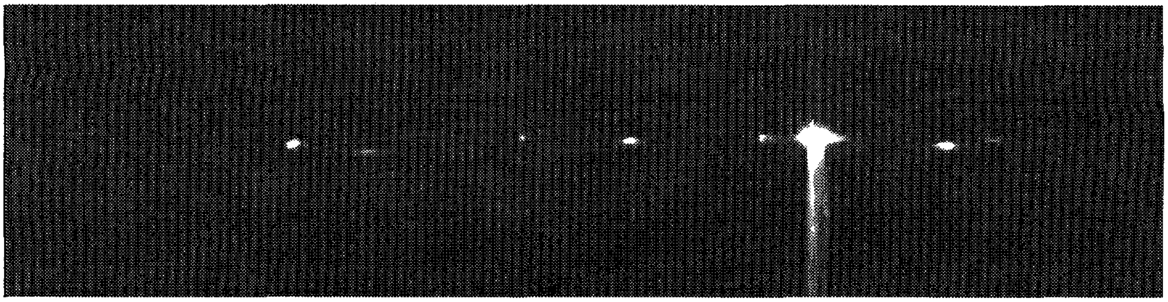

Figure 3a. CCD picture for (100) diffraction at a distance of 1.25 mm away from the notch point for an applied load of 2000 N.

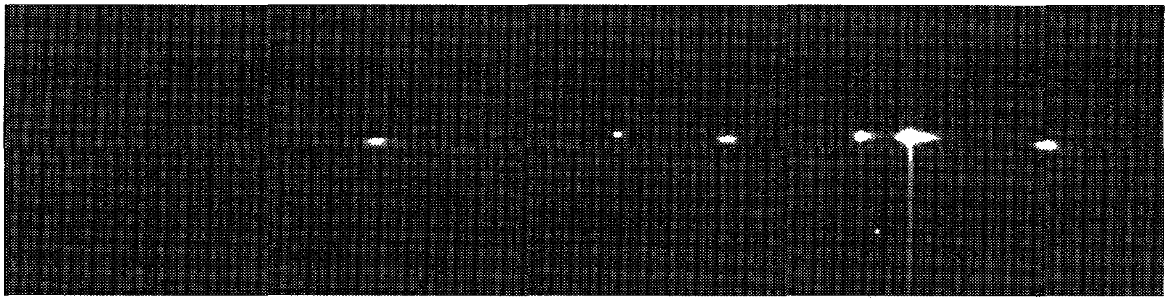

Figure 3b. CCD picture for (100) diffraction at a distance of 1.25 mm away from the notch point for an applied load of 70 N. The pattern is shifted from Figure 3a due to a small change in sample orientation, but is otherwise very similar.

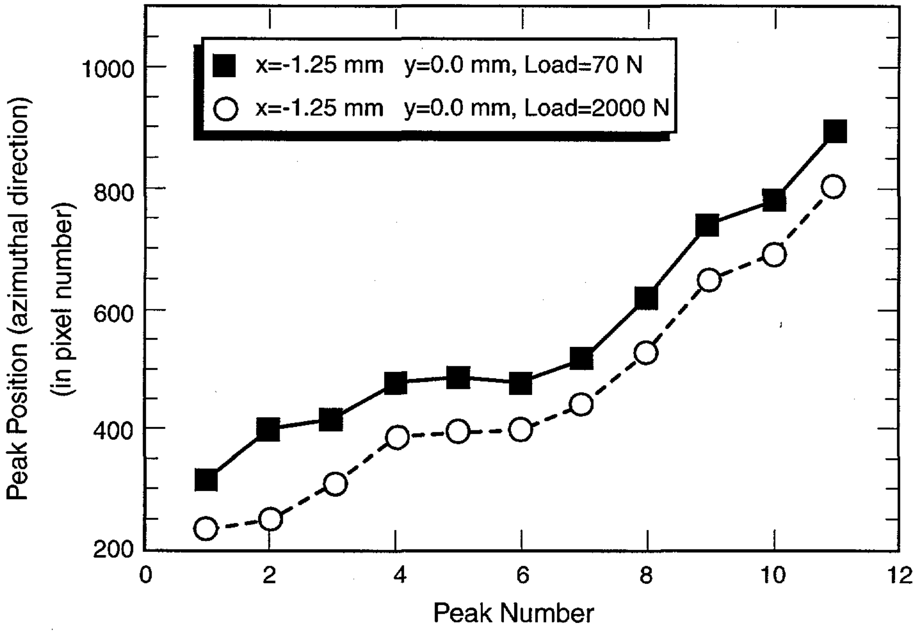

Figure 4. Azimuthal positions of diffraction spots from several of the grains in Figure 3 for two loads on the specimen.

The ability to track individual grains from case to case is exciting if small changes in relative positions can be closely examined. We illustrate this possibility in Figure 4, where we plot the azimuthal positions of diffraction spots from several of the grains in Figure 3 for both of the loads. There is an overall shift related to sample reorientation, but superimposed on the average shift are small differences from grain to grain that contain the information of likely interest, e.g., the spread in microstrain values about an average macrostrain.

Diffuse scattering from the beryllium specimen is apparent above the general background in the CCD data, as can be seen in Figure 5, where we show a line out of the data through one of the intense diffraction spots. The broad plateau region above the background level is interpreted to be the result of diffuse scattering in the target. The edges of this plateau, as projected through S3, can be used to define the surface of the specimen relative to the location of the grain being observed. The full width at half height (FWHM) of this broad peak varied, depending on how near the sampled volume was to the notch point. At the nominal notch point, the observed width of the plateau in the CCD data was only 0.6 mm (10 pixels); the value observed when we sampled in full-thickness regions of the target was approximately 9.0 mm (150 pixels), consistent with the target thickness and geometric factors. Being able to observe this process helps us define the diffracting locations more precisely.

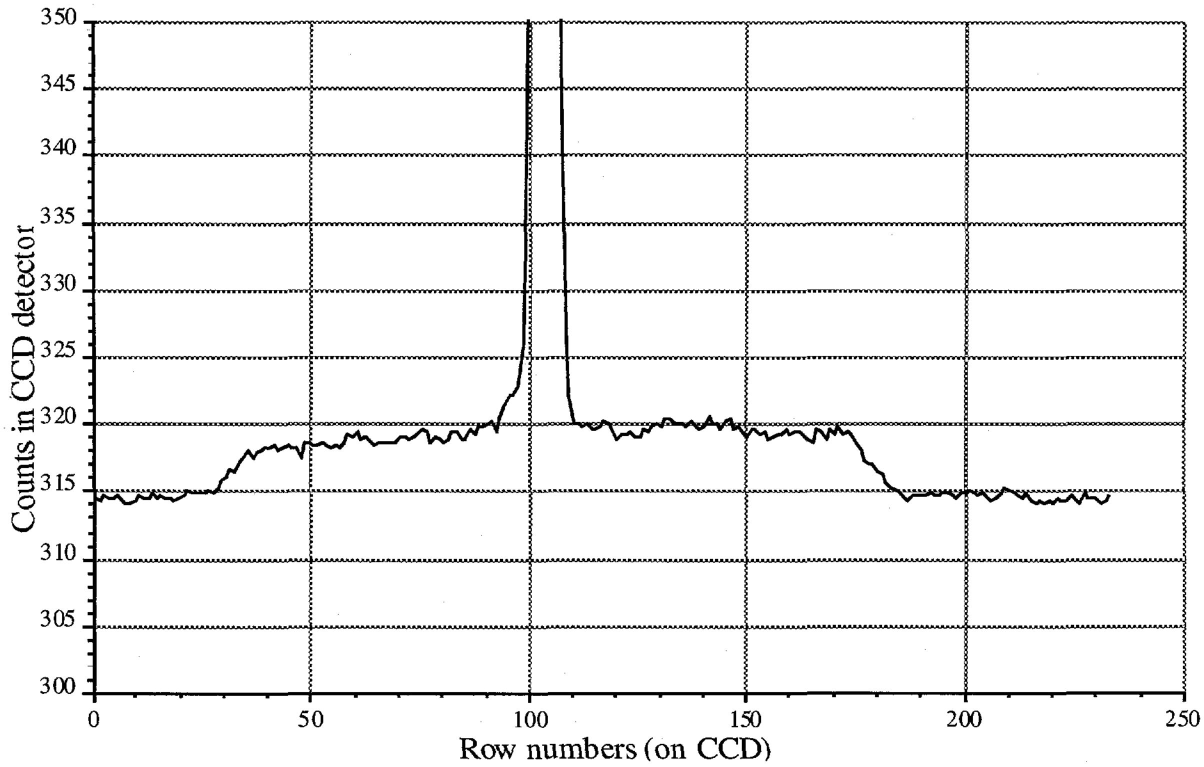

Figure 5. CCD data along one column of the array.

PRELIMINARY STRAIN ANALYSIS AND CONCLUSIONS

For a first-order analysis, we assume that the diffraction peak locations give the microstrain and that the peak shapes give the grain size and grain mosaic spread when scanned along the radial and azimuthal directions, respectively. The microstrain ($\Delta d/d$ in units of 1×10^{-4}) was determined by tracing the shift in peak position (radial direction) of the same diffraction spot with and without load. The limited number of diffracting grains within our sampling volumes and an apparent spread of microstrain values about the macrostrain average expected complicate the analysis and will be one focus of future work. Using simple averages, Figure 6 shows that the microstrain under load increases at the notch point for a scan of the (100) reflection across the notch. In a similar scan across the notch plane but at a depth 1 mm inside the notch point, the observed microstrain values were smaller overall but still indicated a similarly peaked distribution. We also found an increase in the mosaic spread and a reduction in the crystallite size at the notch point. We will report these results in a forthcoming paper.

In conclusion, we have demonstrated tracking of individual grains from one load state to another in beryllium compact tension specimens. We have also observed features in our data that help identify the sampling locations more accurately. Further measurements with smaller beams and improved analysis techniques are planned to map accurately the strains around notch point and around crack tips in precracked specimens.

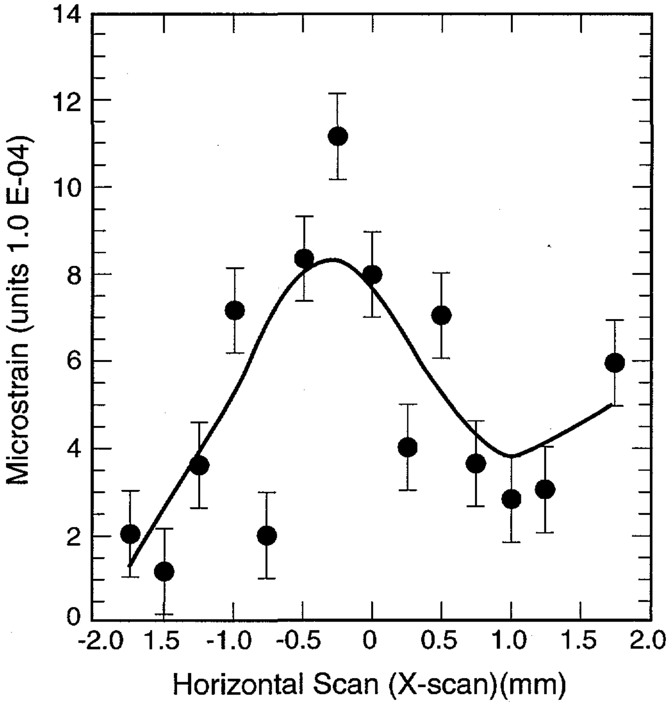

Figure 6. Relative microstrain at 2000 N load as determined in a scan of the (100) *reflection across the notch point.*

ACKNOWLEDGMENTS

This work was supported by the Department of Energy under Contract W-7405-ENG-36 through the Enhanced Surveillance Program Office of Los Alamos National Laboratory. The support from personnel at the Advanced Photon Source of Argonne National Laboratory is gratefully acknowledged.

REFERENCES

Lai, B., et al.: Rev. Sci. Instrum., Vol. 66, 2287 (1995).

Noyan, I. C., and J. B. Cohen: Residual Stress: Measurement by Diffraction and Interpretation, Springer-Verlag, New York (1987).

Varma, R., et al.: *Proceedings of Fifth International Conference on Nuclear Engineering, May 25-29, 1997,* ICONE5-2036, Nice, France (1997).

Wang, J.-D., et al.: Rev. Sci. Instrum., Vol. 66, 1401 (1995).

Webster, D., and G. London: *Beryllium Science and Technology*, Vols. 1 and 2, Plenum Press (1979).