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Characterization of beryllium deformation using in-situ x-ray diffraction

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Abstract
Beryllium's unique mechanical properties are extremely important in a number of high performance applications. Consequently, accurate models for the mechanical behavior of beryllium are required. However, current models are not sufficiently microstructure aware to accurately predict the performance of beryllium under a range of processing and loading conditions. Previous experiments conducted using the SMARTS and HIPPO instruments at the Lujan Center(LANL), have studied the relationship between strain rate and texture development, but due to the limitations of neutron diffraction studies, it was not possible to measure the response of the material in real-time. In-situ diffraction experiments conducted at the Advanced Photon Source have allowed the real time measurement of the mechanical response of compressed beryllium. Samples of pre-strained beryllium were reloaded orthogonal to their original load path to show the reorientation of already twinned grains. Additionally, the in-situ experiments allowed the real time tracking of twin evolution in beryllium strained at high rates. The data gathered during these experiments will be used in the development and validation of a new, microstructure aware model of the constitutive behavior of beryllium.

I. INTRODUCTION

Beryllium has unique mechanical properties that make it valuable in many high performance applications. However, the mechanical behavior of beryllium is sensitive to both microstructure and mechanical loading conditions. Current models for the mechanical behavior of materials like beryllium are not microstructurally aware and development of next generation microstructurally aware models requires detailed data on the mechanical response of beryllium.

Previous studies of the beryllium using ex-situ neutron diffraction have provided insight into the basic behavior of beryllium at stress, for example texture development as a function of strain rate, but due to the long exposure times required for neutron diffraction experiments, real time analysis of deformation at higher strain rates has not been possible.[1]

High x-ray fluxes available at third generation synchrotron sources like the Advanced Photon Source have enabled a new generation of in-situ diffraction experiments. In-situ techniques allow the real time measurement of the mechanical response of materials and thus provide the experiment data necessary to develop and validate new, microstructure aware models.[2]

II. EXPERIMENTAL MATERIALS AND METHODS

A. Sample Preparation

Experiments were conducted on samples of hot rolled beryllium. The beryllium material was prepared using a proprietary process with the following general procedure. Beryllium powder is first hot-pressed into a large billet. Thin slabs are then cut from the powder-formed billet and then cross rolled at elevated temperature until they reach a final thickness of ~5mm. **[1]**

Compression samples were then machined from the rolled plate using electric discharge machining (EDM) for use during compression experiments. The samples were then deformed to prepare them for further analysis using in-situ diffraction. Several samples were pre-strained at Los Alamos National Laboratory to approximately 10, 15, and 20% compressive strain at a rate of 2000/s. These samples, as well as virgin, unstrained samples, were then deformed in-situ at the Advanced Photon Source.

B. In-situ diffraction procedure

Samples underwent in-situ diffraction experiments at the 1ID beamline of the Advanced Photon Source.

During the first series of diffraction experiments, samples of as-rolled beryllium were deformed at varying strain rates ranging from 10^{-3}/s to 1/s. Powder diffraction measurements were recorded in-situ for the duration of the loading event to characterize the microstructural response of the material. Additionally, crystal texture data was gathered post-deformation to more accurately characterize changes in the microstructure of the material.

During the second series of diffraction experiments, samples of pre-strained beryllium were reloaded at a quasi-static strain rate of $2*10^{-3}$/s. The re-loading was performed orthogonal to the original strain path to characterize the effect of strain path changes on the material. Additional powder diffraction measurements were taken of the deformation of as-rolled samples of beryllium to allow comparison with the pre-strained material.

III. DATA ANALYSIS AND RESULTS

A. Analysis of Synchrotron Diffraction Data

The analysis of synchrotron diffraction data requires several distinct steps to convert raw diffraction data into usable information.

The detector used in these experiments generates a two dimensional image of the diffraction pattern. When a polycrystalline material is placed into a monochromatic x-ray beam, the x-rays diffract through this sample. The diffracted x-rays form a series of concentric rings

radiating outward in a Debye Scherrer cone. When the Debye-Scherrer cone intersects the flat detector a diffraction pattern similar to that shown below in Figure 1 results. [3]

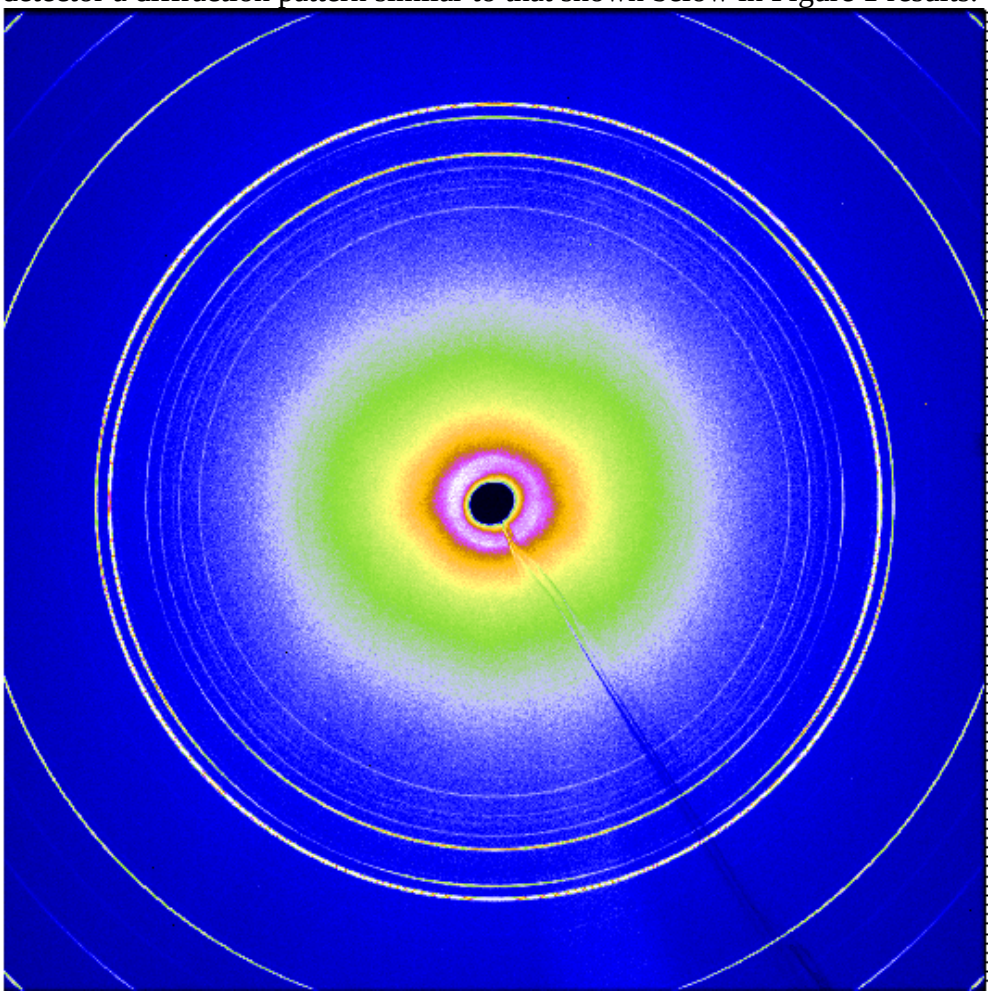

Figure 1: 2D diffraction pattern of beryllium

The exact configuration of the detector must be calibrated before the rest of the diffraction data may be analyzed. The approximate position and configuration of the detector are known, but several parameters, including the sample to detector distance, the XY center of the beam on the detector, and two directions of detector tilt must be determined before analysis can be proceed. Ceria (CeO_2) is used as a standard because its diffraction pattern and crystal structure are well understood. After fitting the detector parameters to the ceria diffraction pattern, a test integration, or caking, was performed using the Fit2D analysis software.[4, 5] This process converts the two dimensional diffraction pattern into a series of one dimensional spectra, as shown on the following page in Figure 2, by integrating the full diffraction pattern into a series of 24 fifteen degrees slices. The integrated diffraction pattern is then broken into twenty four individual spectra, as shown in Figure 3. The spectra were then analyzed using the General Structure Analysis System developed

at Los Alamos National Laboratory.[7] The quality of the calibration is then determined by plotting the lattice spacing of the ceria as a function of angle.

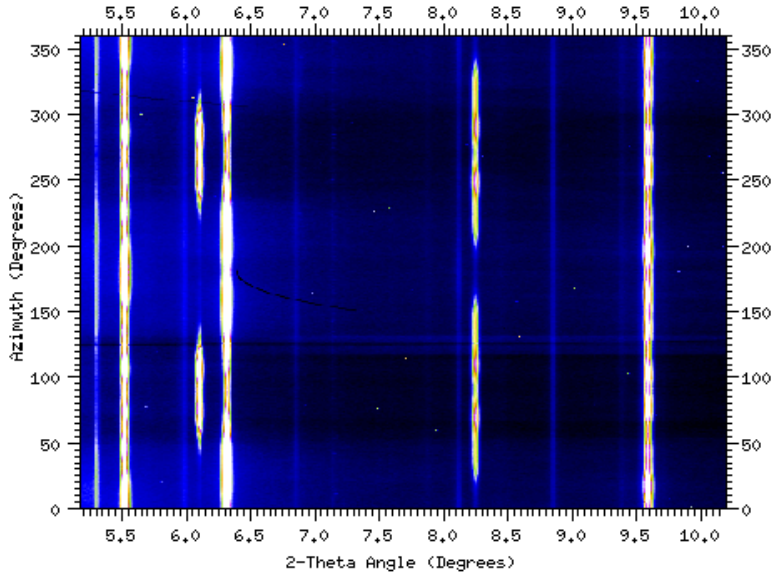

Figure 2: Fully integrated diffraction pattern of Be

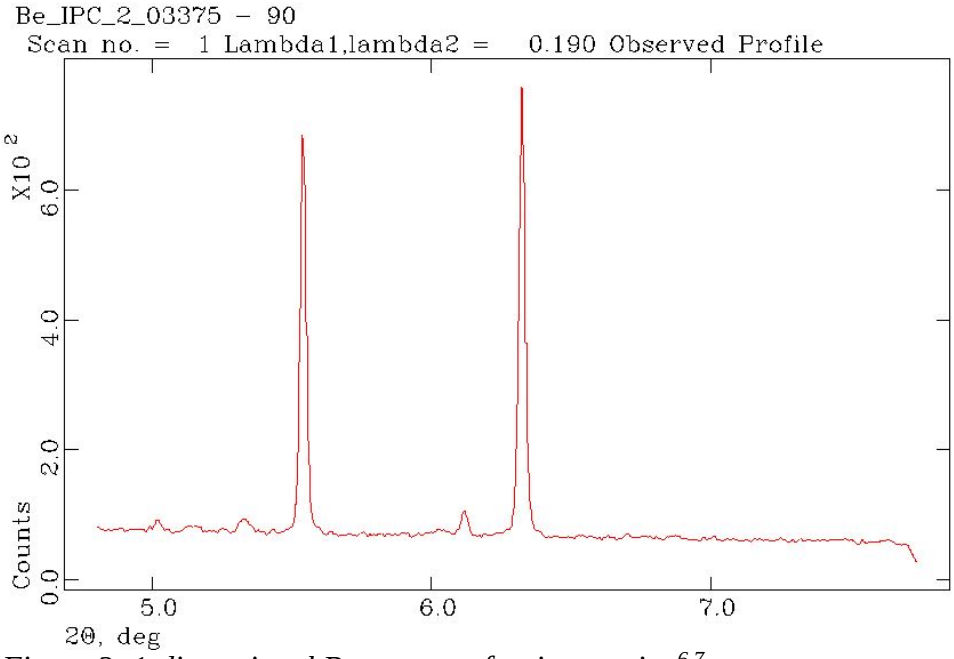

Figure 3: 1-dimensional Be spectra after integration[6,7]

After calibration using the ceria standard, the beryllium diffraction data was then analyzed using the same process. A series of batch processing utilities has been developed to assist the data reduction process.[8] First, APSbin was used to integrate the diffraction patterns and prepare them for further processing. Second, another batch processing utility, APSspf, was used to perform single peak fits on the 1 dimensional spectra. This process fits several parameters to individual peaks in the 1-d spectra including the peak position, peak intensity, and peak width.

The results from the peak fitting are then used to calculate lattice strains using Equation 1. The calculated lattice strains for each angle are then average with the lattice strains of the angle

180° around the Debye-Scherrer cone. This helps to minimize the effect of systematic errors, including small errors in the initial calibration.

$$\epsilon = \frac{d - d_0}{d_0} \quad (1)$$

These represent elastic strains in the material resulting from distortion of the crystal lattice. It is difficult to accurately determine the equilibrium lattice parameters, d_0, of this material, so for the purpose of this analysis, the reference state of the material was assumed to be at the beginning of the in-situ loading event. This assumption neglects the presence of residual lattice strains in the material, both from the initial hot-rolling of the material and the subsequent pre-deformation of the samples.

Additionally, macroscopic force and displacement data were used to calculate macroscopic stress and strain curves for each sample. The macroscopic engineering stress was calculated using Equation 2, using the data from the test frame and the initial sample dimensions. The crosshead displacement of the load frame and the compliance of the loading apparatus and sample were used in Equation 3 to calculate the macroscopic plastic strain.

$$\sigma^{eng} = \frac{F_{load\ cell}}{A_0} \quad (2)$$

$$\epsilon^{eng}_{plastic} = \frac{\Delta l}{l_0} - Compliance_{load\ frame+\epsilon_{elastic}} \quad (3)$$

B. Results

1. Macroscopic Mechanical Response

The macroscopic mechanical response of beryllium can provide insight into the active deformation processes. For example, the macroscopic stress strain curve shown below in Figure 4, shows the macro response of as-rolled beryllium. This response differs from that of the pre-strained beryllium shown in Figure 5.

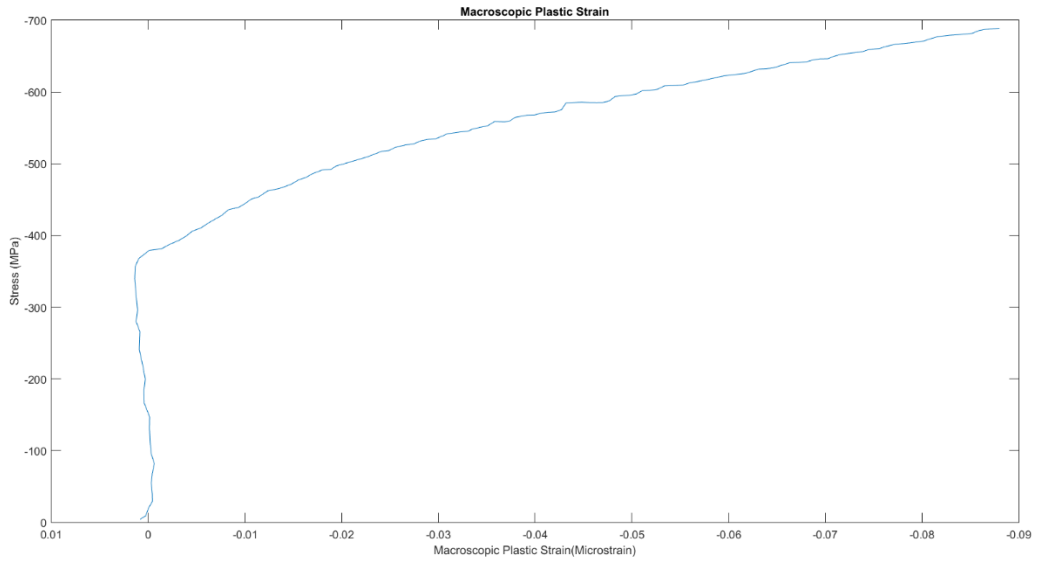

Figure 4: Macroscopic Stress-Plastic Strain Curve for As-Rolled Beryllium

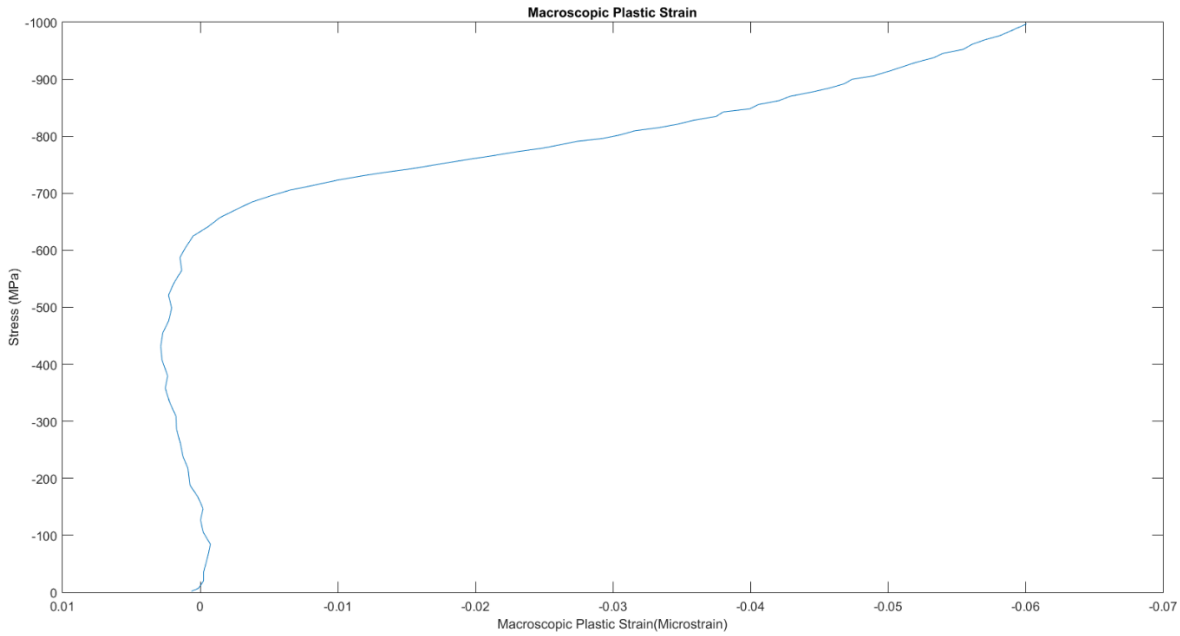

Figure 5: Macroscopic Stress-Plastic Strain Curve for Pre-Strained Beryllium

Both the virgin and pre-strained samples show elastic regions and pronounced yield points, but their post yield behavior differs. The pre-strained sample has a sigmoidal stress strain curve, which is indicative of twin nucleation or re-orientation.[9] The different response of the two materials signifies the importance of processing history to the mechanical behavior of poly-crystalline beryllium.

2. Poisson's Effect

One of the unique properties of beryllium is its extremely small Poisson's ratio of 0.032. Poisson's ratio relates the axial strain imposed on a material to the induced transverse strain from that compressive or tensile strain. Materials with a large, positive Poisson's ratio will experience comparatively larger transverse strains than a low Poisson's ratio material under equivalent axial strains. The Poisson's ratio of beryllium is an order of magnitude lower than most typical metals, and this is reflected in the lattice strains shown on the following page in Figure 6.

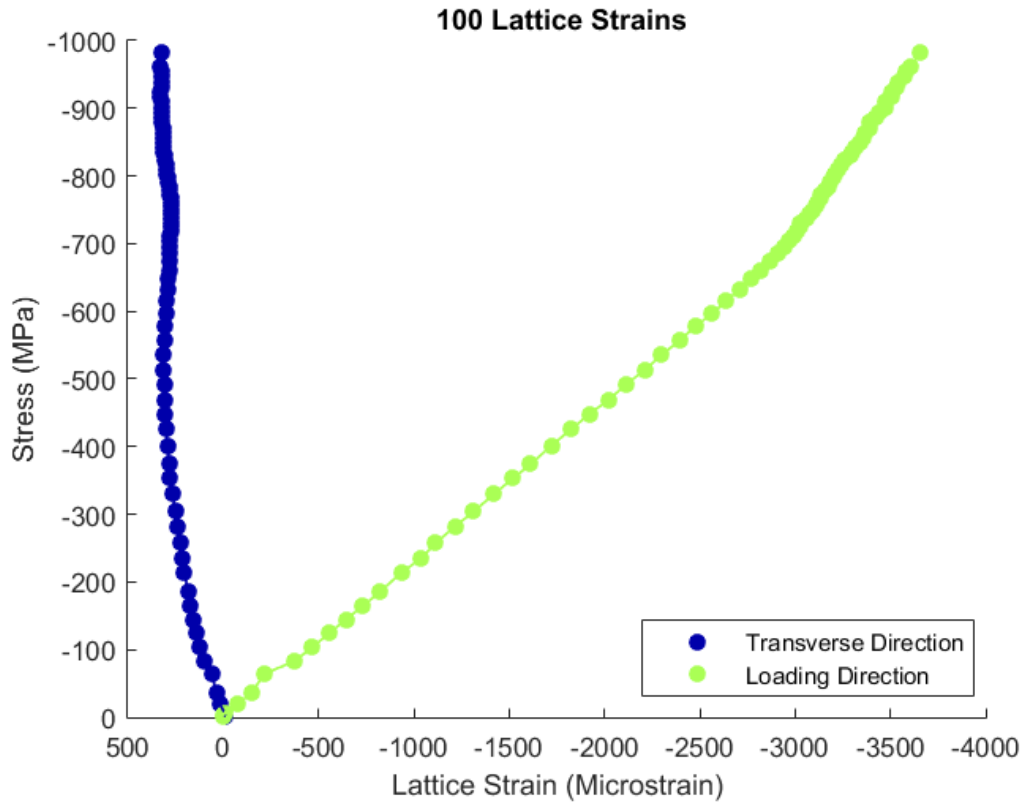

Figure 6: Lattice strains showing Poisson effect in beryllium

Compression loaded samples experienced large compressive strains along the loading direction with comparatively smaller strains along the direction transverse to the strain. The visible Poisson's effect in the diffraction data mirrors the same Poisson's effect for the macroscopic material.

3. Detection of twinning at high strain rates

The role of deformation twinning during the deformation of beryllium at high strain rates is a known phenomenon, previously characterized using the SMARTS instrument at the Lujan Center. During the first series of experiments at the APS, samples of as-rolled beryllium plate were compressed using a large range of strain rates. Samples compressed at high strain rates underwent twinning during the deformation. This is shown in Figure 7, where new 002 peaks appear parallel to the loading direction at high stresses. This is consistent with twinning of the material as the strong initial texture of the material does not allow diffraction of those peaks prior to twinning of the material. In Figure 7, the loading direction is represented by the 90° and surrounding peaks. The direction transverse to the loading direction is represented by the 0° and surrounding peaks. As the peaks in the loading direction do not appear until partway through the loading event, their reference state was determined using a sinusoidal fit of reference lattice spacing as a function of angle.

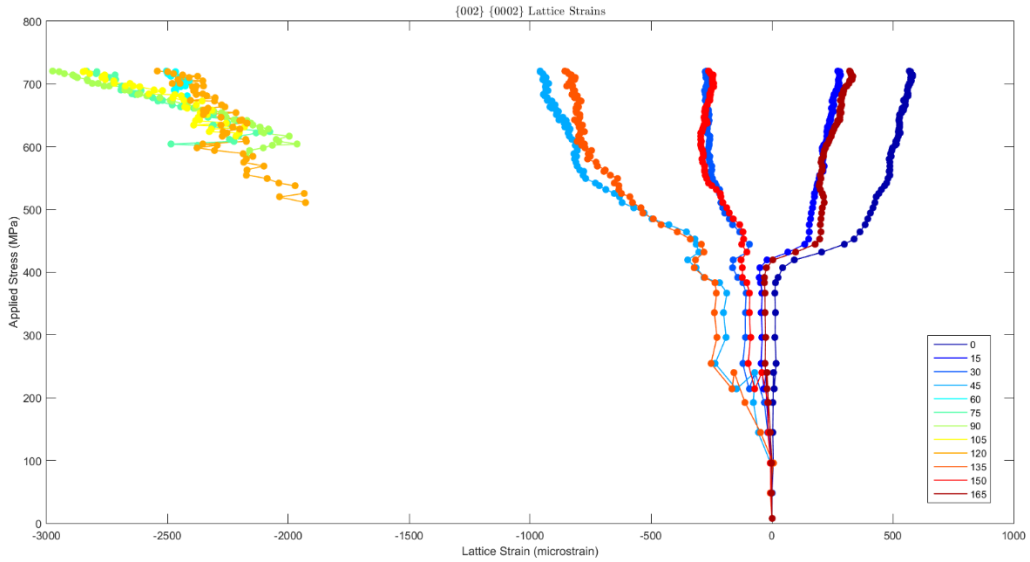

Figure 7: Lattice Strains Showing Twinning

As the material is deformed at high strains, the dominant deformation mechanism changes from dislocation based slip to deformation twinning. This process creates new 002 poles that are oriented parallel to the loading direction. The as rolled material does not have 002 poles parallel to the loading direction due to the strong initial texture, but as the material begins to twin, new peaks appear in the diffraction pattern. The use of in-situ diffraction techniques allows the real-time tracking of microstructural evolution during the deformation event.

4. Comparison of virgin and pre-strained samples at quasi-static strain rates

The second run of experiments allowed the comparison of the dominant deformation mode of virgin, i.e. as-rolled and pre-strained beryllium samples. These samples were loaded at quasi-static strain rates of $2*10^{-3}$/s. The as-rolled and pre-strained samples exhibited different deformation modes during the loading event.

The pre-strained samples were placed into the load frame with the IP direction parallel to the x-ray beam. Consequently, the twinning induced 002 poles were parallel to the beam and did not contribute to the diffraction pattern. As the material was loaded, the intensity of the 002 peak parallel to the loading direction increased significantly, as shown below in Figure 8.

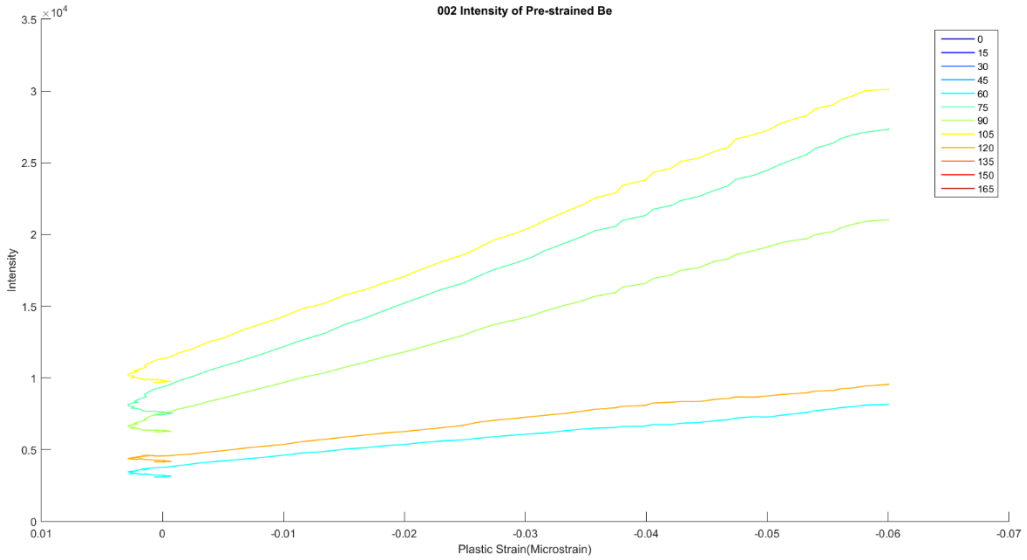

Figure 8: Pre-strained Intensity Plot

This is indicative of twin reorientation of grain initially with the basal pole parallel to the beam to an orientation where the basal pole is aligned with the loading direction. In pre-twinned beryllium, the material will reorient existing twins to accommodate plastic deformation rather than through slip.

By contrast, experiments performed on as-rolled beryllium at similar strain rates do not result in an equivalent increase in 002 pole intensity parallel to the loading direction as shown in Figure 9. Due to the strong initial texture of the as-rolled beryllium, the 002 peak is not present along the loading direction, which is consistent with rolled material. Additionally, the increase in 002 pole at 30 and 150 degrees is indicative of pyramidal slip, which is one of the possible slip modes of poly-crystalline beryllium.

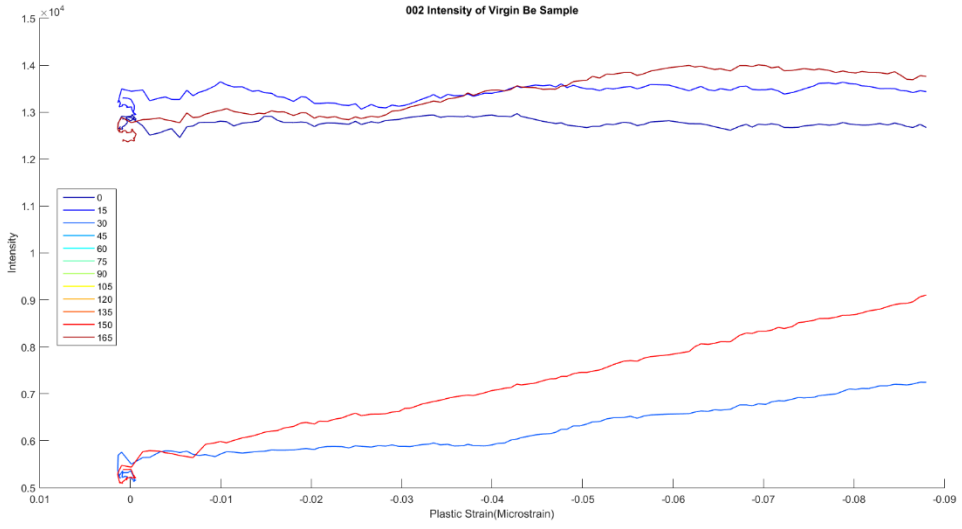

Figure 9: As-rolled Intensity Plot

IV. CONCLUSIONS

Beryllium has many applications in high performance aerospace and mechanical applications. Consequently, models that can accurately predict the mechanical behavior of beryllium are extremely useful. However, current models are not microstructure or processing aware, and require further development to incorporate these factors.

In-situ studies of beryllium deformation conducted at the Advanced Photon Source in 2010 and 2014 were used to analyze the behavior of a sample of rolled plate beryllium strained under a variety of conditions with different processing histories. The original in-situ studies were conducted to characterize the relationship between dominant deformation mechanism and the strain rate. At slow strain rates, the as-rolled beryllium will deform via crystallographic slip while at higher strain rates, the material will accommodate strain through deformation twinning. These results were consistent with earlier experiments conducted using neutron diffraction at the Lujan Center, but these experiments were the first real time characterization of twinning processes in beryllium.

Additional experiments were then conducted in 2014 at the Advanced Photon Source comparing the behavior of pre-strained and as-rolled beryllium at quasi-static strain rates. The pre-loaded samples were strained at rates high enough to ensure a large twin fraction. During secondary loading, the already twinned material deformed primarily through the reorientation of already twinned grains rather than through slip based processes. The as-rolled samples did not display evidence of twinning and small changes in the diffraction pattern were indicative of deformation through pyramidal slip. These results show the importance of material history in predicting the mechanical behavior of beryllium during deformation.

The results of these experiments will be used in the development of a new EPVSC model at the University of New Hampshire. This model is more microstructurally aware than the previous generation of models, incorporating experimental data from both in-situ experiments conducted at the Advanced Photon Source and ex-situ experiments conducted at the Lujan Center. However, the experimental data set is limited to a fairly narrow set of processing and deformation conditions. For example, the current set of experimental conditions is comprised of experiments conducted under simple uniaxial compression. Additional experiments under less arbitrary stress state constraints will improve the resulting model.

Additionally, capturing full diffraction patterns at high strain rates is difficult due to the detector read-out time. However, region of interest scans, where only a portion of the diffraction pattern is scanned, are a promising method of analyzing deformation at high strain rates. A limited number of region of interest scans were taken during the experiments in 2010, but there have been problems correlating the observed diffraction data to the behavior of the material. Improvements in detector read out time will allow measurement of constitutive response at higher strain rates, but this requires an entirely new detector from the existing system in use at 1ID, and it is unknown when these improvements will occur.

V. ACKNOWLEDGEMENTS

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