New Mechanical Testing Methods for Structural Materials at Small Size Scales

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Abstract

This paper describes new experiments designed to characterize the micromechanical response of structural materials. As a way to calibrate microscale constitutive models and to understand grain scale elastic-plastic deformation, our group worked closely with CHESS personnel to develop a high energy x-ray method for measuring lattice Strain Pole Figures (SPFs) *in situ* in the A2 experimental station at CHESS. Results from experiments and crystal-based finite element simulations on copper are presented in the paper. The distribution of stresses predicted by the model closely compares to the experimental result. Motivated by our success at CHESS, we have begun conducting experiments at the Advanced Photon Source (APS) to characterize individual grains within a deforming polycrystal. We show results for crystals in two processed states of a titanium alloy,Ti-7Al. The stress states change with deformation-likely due to translation of the stress state along the single crystal yield surface. A complicated residual stress state was present in each grain upon unloading. By highly resolving individual diffraction spots within each crystal, we were able to understand the character of dislocation structure formation in each alloy state. These experiments show huge potential as a means of characterizing engineering materials in general. We also believe the experiments – coupled with a crystal-based stress state representation methodology – bring a new measurement capability to the important problem of residual stress.

Introduction

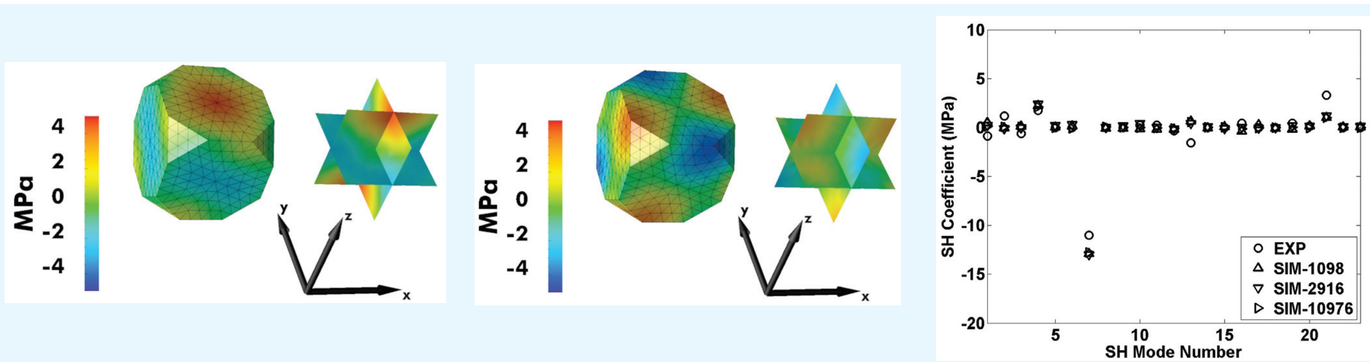
Structural materials play a key role in the products we use every day. From the titanium of a bicycle frame to the nickel-based super alloy that makes up a jet engine component, structural materials are selected for their strength, stiffness, density, corrosion resistance and a myriad of other properties. Mechanical properties are always related to some idealization of material behavior referred to as a constitutive model. From Young's modulus to yield strength and fracture toughness, mechanical properties arise naturally when the response of a structural material to thermal and mechanical loading is viewed from the perspective of a constitutive model. Mechanical properties are measured by applying thermo-mechanical loads to a test specimen then monitoring its response. By knowing both the stimulus and response functions, the properties can be determined. With the mechanical properties known, these experiments – which are referred to as *mechanical tests* – can also be used to monitor the performance characteristics of a structural material. The fidelity of the constitutive model can be verified by subjecting the material to more complicated loading scenarios. The simplest test is uniaxial monotonic loading – pull or compress the specimen past the yield strength to failure. Multiaxial loading experiments, such as tension-torsion tests, can be used to understand material behavior and the validity of the model under complicated stress states, which more closely mimic service conditions. High temperature, corrosive conditions, cyclic loading and loading rate variation can be employed to create a veritable "gauntlet" of service conditions for the material and the model. Success in correlating experimental behavior builds confidence that a constitutive model can be trusted in the actual conditions an engineering component will be subjected to – conditions that cannot be replicated in the laboratory.

Constitutive model development for structural applications has moved down scale for several reasons. Micro-electromechanical systems, MEMS, are the same size as the microstructure. MEMS design requires an understanding of material response on the scale of the microstructure. More importantly, there is a growing need to model deformation – induced microstructure evolution within large scale components. The hope for these microscale formulations– from models of atomic behavior to dislocation models to crystal plasticity – is that some of the behaviors that are mysterious and practically random on the macroscale will become tractable and predictable when the microstructural response is being modeled. These processes include fatigue microcrack initiation, plasticity, recrystallization and phase transformations. Enormous growth has taken place in the general field of multiscale material modeling over the past 10-15 years. The ever-increasing capacity of computing facilities has encouraged theoreticians to model behaviors on increasingly smaller size scales with the hope of creating truly predictive links between material structure, thermo-mechanical properties and measures of performance. These links will eventually enable designers to base allowable (operating loads and temperatures, for instance) on micromechanical properties and analyses. From an experimental perspective, the question becomes whether or not we can reproduce mechanical tests on the microscale. One approach is to conduct traditional mechanical tests on subsized specimens constructed from the microstructure or from components of a micromechanical machine. Another approach is to employ microscopic mechanical instruments such as nanoindentation devices to probe microscale stress-strain behavior. High energy synchrotron x-ray diffraction methods represent a formidable alternative to subscale experiments. Modern area detectors and impressive depths of penetration have made it possible to probe a metallic crystal that lies far beneath the surface of a test specimen and make diffraction measurements quickly. By conducting carefully designed mechanical tests *in situ* during an x-ray diffraction experiment, the crystal lattice plays the role of the engineering strain gage on the macroscale, with the added bonus that the strains indicated by the lattice are elastic. The challenge is the deconvolution of the diffraction data - pulling it apart in order to understand its origin and significance – then recombining it to draw conclusions regarding the deformation of the polycrystalline aggregate.

Our group at Cornell began using high energy x-rays at the A2 experimental station at CHESS to understand the behavior of polycrystalline structural materials and to validate micromechanical constitutive models. Working closely with beamline personnel, we designed and fabricated our own mechanical loading stage and have had significant success interrogating polycrystalline aggregates for distributions of lattice strain and stress[1,2]. Since that time, we have moved farther downscale to probe individual crystals within an aggregate at the Advanced Photon Source (APS). This paper describes our experiment developed at A2 designed to measure lattice strain distributions within a loaded polycrystalline aggregate. By determining the lattice strain tensor at each orientation, we are able to use Hooke's law to calculate the orientation-averaged stress tensor distribution. Coupled with crystal-based simulations, these data have proven invaluable for improving our understanding of deformation partitioning in polycrystalline alloys. The second type of experiment, in which the deformation of individual crystals within the aggregate is tracked, is part of the High Energy Diffraction Microscopy (HEDM) suite of experiments at beamline 1-IDC at the APS. When full spatial resolution is attained, the HEDM experiment will likely become the "gold standard" for microscale stress measurements.

Stress State Distributions in Loaded Polycrystals

In this "powder" experiment , we interrogate an entire polycrystalline aggregate simultaneously. As with traditional mechanical tests, each **continuum** point within the sample is identical. Each diffraction volume that defines a continuum point must contain a statistically relevant number of grains - usually several thousand. In this way, we are not restricted to tracking a particular set of crystals during the *in situ* deformation. This condition is crucial for this experiment and dictates the grain size / beam size relationship. We acquire bands of diffraction data for each family of lattice planes, *{hkl}*, by rotating the loadframe using its built-in goniometer[3]. At A2 we typically use a beam of 50 KeV x-rays with 0.5mm x 0.5mm slits. A set of lattice Strain Pole Figures (SPFs) are measured at discrete loads; then the lattice strain distribution tensor, $\boldsymbol{\epsilon}(\mathbf{R})$, at each lattice orientation, **R**, is determined[4]. Lattice strains are elastic so the stress distribution is calculated using Hooke's law, $\boldsymbol{\sigma}(\mathbf{R}) = \mathbf{C}(\mathbf{R})\boldsymbol{\epsilon}(\mathbf{R})$, where **C** are the single crystal moduli. We know that orientation as well as crystallographic neighborhood contribute to the mechanical response of an individual crystal within an aggregate. Therefore $\boldsymbol{\sigma}(\mathbf{R})$ has the interpretation of being the most likely state of stress at a particular orientation. The $\boldsymbol{\sigma}(\mathbf{R})$ data are ideal for use with a crystal-based modeling framework, such as an elastic-plastic finite element formulation, for understanding the distribution of deformation within the aggregate. One way to evaluate the distribution of each stress component over orientation space - both measured and computed - is by an expansion of spherical harmonic functions, $\sigma_{ij}(\mathbf{R}) = \sum_{l=0}^{\infty} H_l T_l(\mathbf{R})$. Here the $T_l(\mathbf{R})$ are generalized spherical harmonics functions (modes) that satisfy relevant symmetry conditions and form an orthonormal basis over orientation space. The coefficients, H_l, prescribe the contribution of each mode to the stress. Fig. 1 depicts two spherical harmonic modes plotted over orientation space. Fig. 1 also depicts the spherical harmonics coefficients for the shear stress, σ_{xy}, over orientation space from a polycrystalline copper sample loaded in tension to a macroscopic stress of S_{zz} = 180 MPa. Based upon the coefficients, we see that both the experiment and simulation have "chosen" mode 7 as the dominant mode. Even though this component of stress must necessarily integrate to zero over the cross section to match the boundary conditions of the experiment, shear stress values as large as 75-80 MPa are present. The close comparison between the simulation and experiment creates confidence in both, and builds additional trust in the model as it is applied beyond the reach of the experiments. Similar comparison was made



(a) mode 3 (b) mode7 (c) σ_{xy}

Fig. 1: (Left and Center)Two spherical harmonic modes shown distributed over the cubic fundament region of orientation space parameterized using the Rodrigues representation of orientation. The surface of the region along with orthogonal slices through the origin are depicted. (Right) Spherical Harmonics (SH) coefficients vs. mode number for the shear stress component, σ_{xy} as determined from the experimental data (EXP) and computed from the crystal-based finite element simulations (SIM) at a uniaxial stress of S_{zz} = 180 MPa. Three finite element mesh sizes for the virtual test specimen were employed, 1,098, 2,916 and 10,976 crystals. Each crystal contained 48 finite elements.

for other components of stress[5]. From these experiments and simulations, we learned that the microscale stress state can be very different than the macroscopically applied stress. There are many situations (such as fatigue) when an assumption of uniaxial stress is nonconservative. These data enable us to implement the finite element modeling formulation with greater confidence.

Individual Crystal Experiments
The stress distribution data from the *in situ* SPF experiments described in the previous section provide an orientation dependent, statistically relevant approximation of the micromechanical partitioning of the elastic-plastic deformation over a polycrystalline aggregate. As described above, the stress distribution data are an excellent match for the current generation of crystal based simulation formulations. In reality, the response of each crystal within the aggregate is a function of both its orientation and the surrounding crystals that supply the boundary conditions for its deformation. Measurement of the stress state within individual crystals will enable us to eventually quantify the neighborhood effect. The 3DXRD suite of interrogation experiments were first developed by Risoe researchers at the European Synchrotron Radiation Facility (ESRF)[6]. One of the 3DXRD capabilities made it possible to measure lattice strains within an individual crystal within a loaded aggregate[7]. Our group has collaborated with APS sector 1 personnel to further develop this *in situ* lattice strain measurement capability as a part of the beamline 1-IDC suite of experiments called High Energy Diffraction Microscopy (HEDM). Fig. 2 depicts a configuration for conducting two HEDM experiments: individual crystal lattice strain measurements for understanding crystal stress states and high resolution interrogation of individual reflections within the crystal to understand deformation-induced dislocation substructure formation[8].

Titanium Alloys
With their high strength to density ratio and excellent corrosion resistance and temperature range, titanium alloys have enabled some of the most important aircraft design innovations over the past several decades[9]. Single crystal property anisotropies make processing of titanium alloys difficult and create challenging design conditions, however. Our group has conducted a number of studies on titanium. Fig. 2 also depicts the macroscopic stress strain curves for experiments on an α phase (hcp) Ti-7Al alloy in two states. The slow cooling rate that the AC (Air Cooled) material experiences from α / ß transus temperature produces an ordering of the titanium and aluminum atoms over short length scales. Dislocation glide within this Short Range Order (SRO) material results in planar slip and increased strain hardening and greatly affects the room temperature creep behavior of Ti-7Al[10]. When the Ti-7 was quenched quickly in the IWQ (Ice Water Quenched) state, the SRO does not form and the slip is more isotropic. We conducted HEDM experiments - lattice strain measurements and high resolution reflection decomposition - on samples from both material states. The stress strain curves shown in Fig. 2 also depict the macroscopic stress levels where diffraction experiments were conducted. We tracked the deformation of several grains in each specimen but only collected high resolution data on one each. While identical orientations would have been preferred, we chose the subject grains based mostly on the number of clear reflections we were able to capture. The angles between the c axis and the loading axis in the IWQ and AC grains was 28.7°and 81.0°, respectively. Monochromatic 52 KeV x-rays were employed with a beam size of 300 µm x 150 µm (horizontal x vertical) for detector A and 100 µm x 100 µm for detector B. Additional experimental details can be found in[11].

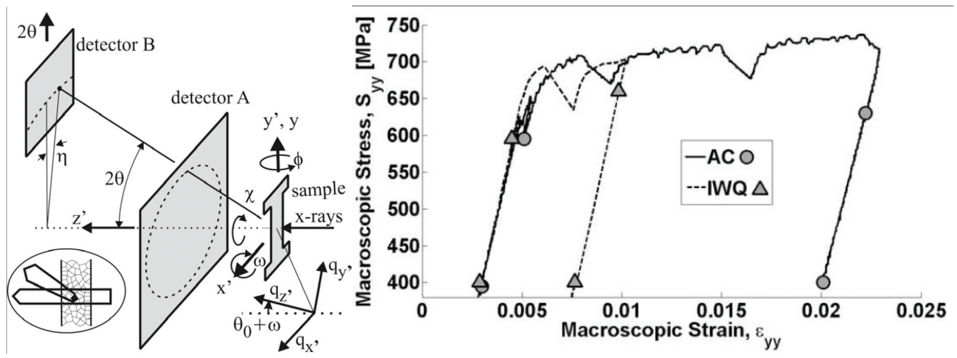


Fig. 2: (Left) HEDM setup at APS beamline 1-IDC. Different detectors were used for the lattice strain measurements and for the high resolution experiments. Each experiment requires a rotation of Φ about the y axis to interrogate relevant scattering vectors. Detector A (approximately 1m from the sample) captures many discrete diffraction spots from multiple *{hkl}*s and the lattice strains are used to determine the full lattice strain tensor for the grain. A single reflection is captured on detector B - very high resolution is possible. The B detector is approximately 8m from the sample. The x', y', z' laboratory and $q_{x'}, q_{y'}, q_{z'}$ reciprocal space coordinate systems are indicated. The y' axis coincides with the sample y direction, the sample x and z directions rotate with Φ. The insert indicates diffraction from a selected bulk grain. (Right) Macroscopic stress-strain response for the AC and IWQ Ti-7Al samples during the *in situ* loading experiment. The symbols indicate stress levels where diffraction experiments were conducted.

Stress State Evolution
Fig. 3 depicts the evolution of the three dimensional stress state within the AC and IWQ grains. Each grain begins with a stress state that very nearly matches the applied uniaxial state. As the load increases and the grains yield, the stress states change. The largest change in each grain's stress state occurs between points 2 and 3, when fully developed plasticity occurs. The stress states become multiaxial - which is very interesting from an elastic-plastic deformation perspective and validates, at least in a qualitative manner, the trends that we observed in the aggregate experiments described above. The direction associated with the largest principal stress is near the macroscopic loading direction, but the other two principal components are not zero, which they would be for a uniaxial stress state. Each specimen was unloaded to 400 MPa, which corresponds to the first stress level. The residual stress state in each grain can be understood by comparing 400el values to 400un values. Even "simple" macroscopic loading conditions like uniaxial tension can produce complex, three-dimensional residual stress states on the crystal scale that **evolve** with straining. Since most residual stress measurement capabilities require an assumption of stress state[12], this indicates a real need for a more sophisticated measurement capability. Processing-induced residual stresses are a huge issue in titanium parts used in aerospace

applications. For instance, it is nearly impossible to predict the fatigue life of a titanium part that contains significant residual stresses.

Resolved Shear Stress

Dislocation glide (yielding on the crystal scale) occurs when the shear stress on one or more of the slip systems (slip plane + slip direction) reaches a critical value. One of the primary hcp slip systems is the basal, $\{0001\}\langle 11\bar{2}0\rangle$. Calculation of the resolved shear stress, τ , involves projecting the stress state onto the slip plane in the slip direction. The typical approach is to assume uniaxial tension as the macroscopic stress state. We have measured the full stress tensor so we do not need to assume a uniaxial stress state. To understand the possible error that might arise with a uniaxial assumption, we calculated τ in the AC grain two ways. First we employ the usual approach: project the macroscopic stress, S_{yy}, onto the slip system. Then we calculate τ using the entire stress tensor. The results are depicted in Fig. 3. The biggest difference is seen between 595 MPa and 630 MPa where fully developed plasticity ensues and the stress state jacks in Fig. 3 are seen to rotate. We actually see a drop in τ between 595 MPa and 630 MPa for two of the three slip systems, even though the macroscopic stress increases by 35 MPa. This seemingly inconsistent result arises due to the topology of the single crystal yield surface. Metallic crystals are restricted to plastic straining only on the prescribed slip systems. To accommodate a general plastic strain rate, therefore, it has been hypothesized that the crystal stress state will actually rotate to activate more slip systems[13,14]. Certainly this evolution can involve a decrease in the value of stress projected onto a slip system. The decrease seen in Fig. 3 is entirely consistent with this rotation of the stress state. To our knowledge, this is the first measurement of the yield surface-induced stress rotation for a crystal embedded within a polycrystalline aggregate. The rotation of the stress state should not be confused with the crystal rotation, which is discussed in the next section.

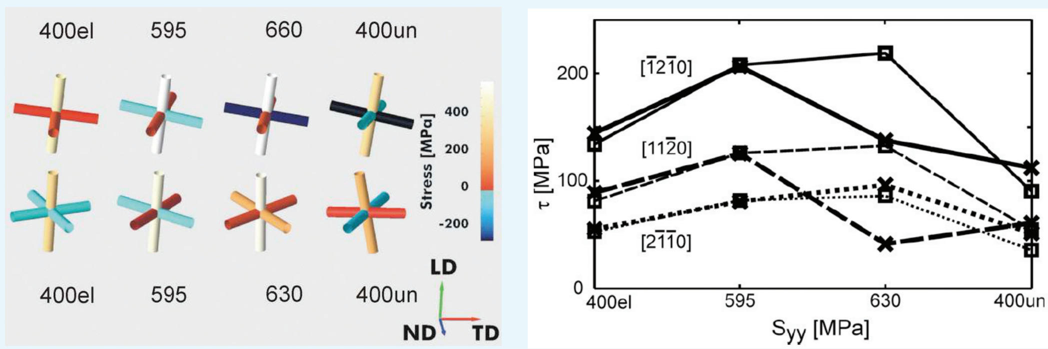
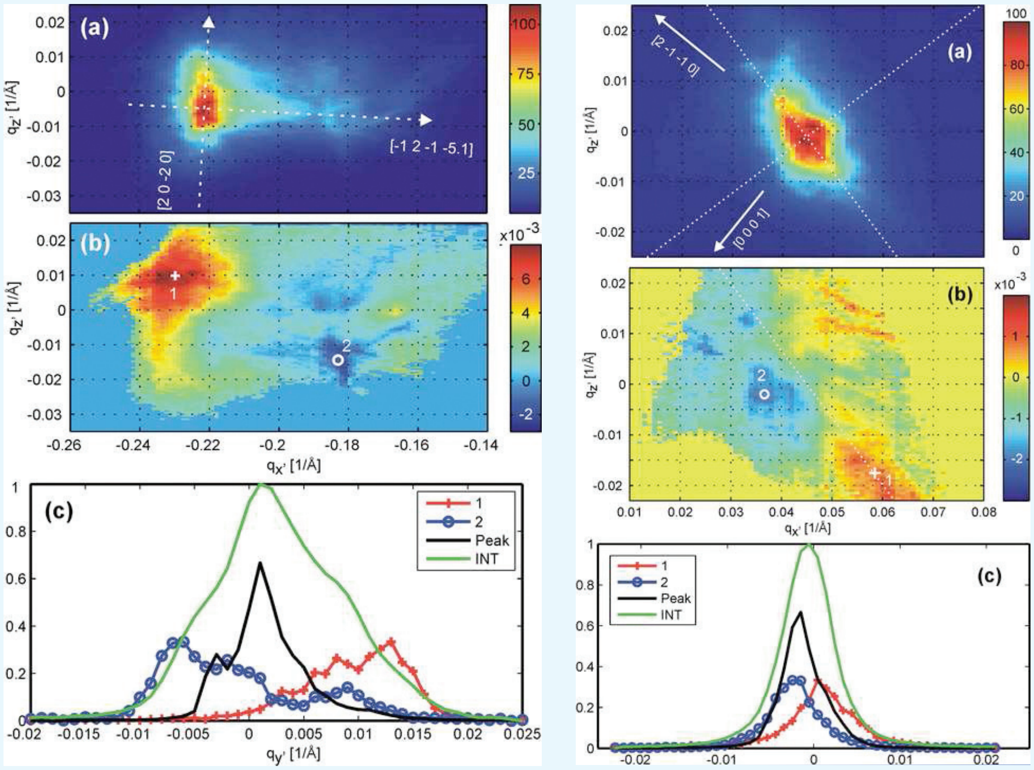


Fig. 3: (Left)Principal axis triads or "jacks" depicting the orientation of the principal stress state at the four indicated macroscopic stress levels (in MPa) for the IWQ (top) and AC (bottom) specimen. The legs of the jacks correspond to the principal directions relative to the specimen Loading Direction (LD), Transverse Direction (TD) and Normal Direction (ND). The colors of each leg and accompanying color scale indicate the principal value. (Right) Resolved shear stress, τ for the 3 basal slip systems of the AC grain as a function of the applied load. The box symbols were calculated assuming a uniaxial stress and the macroscopic stress value. The **X** symbols were calculated using the full stress tensors shown in the figure on the left.

High Resolution Reflection Decomposition

We hypothesized that the SRO-induced differences between AC and IWQ would be visible in the high resolution data. Using detector B, we analyzed individual reflections (spots) associated with the AC and IWQ grains. Basically this study is an extension of traditional peak broadening analysis. The detailed geometric information that can be obtained with high brilliance synchrotron radiation and large sample to detector distances however, enable increased understanding of the deformation-induced dislocation substructure formation that occurs within metallic crystals.

Fig. 4 depicts reciprocal space representations of intensity and lattice strain distribution for individual reflections in the AC and IWQ grains. Fig. 4(a) are intensity maps. An interesting feature of the AC map is the extended "tail" which is consistent with inhomogeneous grain rotation. The intensities integrated over the peak and tail region are roughly the same; hence, the volumes are similar[15]. This would indicate significant dislocation structure formation, which is consistent with TEM micrographs of the material[11]. The intensity distribution for the IWQ reflection is consistent with relatively small grain rotation and homogeneous deformation and distribution of dislocations. Fig. 4(b) depicts the "centroid" of the axial distributions, $q_{y'}$. The centroid values represent the lattice strain in the direction of $q_{y'}$ averaged over the orientation fiber specified by the $q_{x'}$ and $q_{z'}$ coordinates. There is a broader spread in centroid over the AC grain than the IWQ grain. Fig. 4(c) depicts intensity vs. $q_{y'}$ for the maximal values in the centroid maps and the peak intensity point from the azimuthal map. The multiple peaks (black, red and blue) in the AC data are consistent with heterogeneous straining within the grain. From the profiles, the IWQ grain appears to be deforming uniformly. Additional data from multiple diffraction peaks would be necessary to quantify the exact distribution of orientations within the grain - an intragrain Orientation Distribution Function, but the information from the reflections shown here is consistent with the hypothesis regarding short range order and planar slip. From the TEM analysis, we determined that the dislocation density in the AC grain was comparable to that in the IWQ[11]. We used the IWQ integrated profile (green curve) and the restricted random dislocation model developed by Wilkins[16] to confirm the dislocation density. However, an estimate of the AC grain dislocation density from its integrated peak information would grossly overestimate the dislocation density in the AC grain. The broadening in this case comes primarily from the larger structures associated with heterogeneous straining within the grain.



A: AC ($\bar{1}2\bar{1}2$)

B: IWQ ($0\bar{2}20$)

Fig. 4: Reciprocal space maps and intensity profiles for individual reflections from the AC and IWQ crystal. (a) Azimuthal map of diffracted intensity in reciprocal space. The reciprocal space coordinates are depicted in Fig. 2. The dashed lines and Miller indices indicate the directions of the smallest and largest widths at half maximum. (b) Azimuthal map of the centroid of the $q_{y'}$ (d^{-1}) distribution. (c) Normalized intensity vs. $q_{y'}$ for points 1 and 2 in (b) and for the peak intensity value in (a).

Summary and Future Directions

Motivated by traditional mechanical tests, we have conducted experiments that combine mechanical loading with synchrotron x-ray diffraction to understand the micromechanical response of metallic alloys.

• The aggregate-based experiments that we developed at CHESS A2 have become our standard mechanical test. We have characterized copper, titanium, aluminum and nickel alloys. In addition to building a more complete understanding of our error bars and resolution, we are continuing to develop data reduction schemes that have greatly streamlined the SPF experiments and made them more user friendly. In general, any success we have enjoyed with these experiments is due in large part to the environment of encouragement that exists at CHESS and the excellent working relationship we have with CHESS personnel. Our graduation to other techniques and facilities has come about by the confidence we have gained from our CHESS experience.

• In addition to understanding elastic-plastic deformation behavior within a polycrystalline aggregate, we foresee the combined suite of distribution-based SPF diffraction experiments and crystal-based micromechanical representations as a way to understand *residual stresses* in a way that has never been done. Residual stress plays a major role in the processing and performance of wrought metallic alloys. As demonstrated in this paper, the state of stress within an aggregate is a function of macroscopic loading and microscopic mechanical properties. In order to accurately quantify and predict a residual stress state, one must have a way for accounting for both. Our experiments and simulations represent a potential framework capable of such an accounting.

• In addition to monotonic loading, we conduct cyclic experiments - determining SPFs after a certain number of cycles. In collaboration with Peter Revesz at CHESS, we have also developed an experiment for measuring lattice strains in real time using a custom built x-ray shutter system[17].

• The potential of the HEDM experiments is enormous. We are currently working with other 1-IDC users to transform the experimental suite from “heroic efforts”, capable of measuring the response of a handful of grains, to methodologies, capable of characterizing large polycrystalline aggregates of grains. Creating robust data acquisition and reduction software is at the heart of this effort. Plans include combining the lattice strain measurements described above with grain reconstruction experiments[18] to characterize spatially resolved stress fields over a statistically relevant number of grains. Again, the exciting part of this plan is combining such datasets with micromechanical modeling formulations.

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