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Investigation on temporal evolution of the grain refinement in copper under high strain rate loading via *in-situ* synchrotron measurement and predictive modeling

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ABSTRACT

Synchrotron X-rays are integrated with a modified Kolsky tension bar to conduct *in situ* characterization of the grain refinement mechanism operating during the dynamic deformation of metals. Copper with an initial average grain size of 36 μm is refined to 6.3 μm when loaded at a constant high strain rate of 1200 s^{-1}. Synchrotron measurements revealed the temporal evolution of the grain refinement mechanism in terms of the initiation and rate of refinement throughout the loading test. A multiscale finite element based recrystallization model has been developed to predict the grain size evolution occurring during the dynamic deformation process. The model accurately predicts the initiation and temporal evolution of the refinement phenomenon with a predicted final average grain size of 2.4 μm.

1. Introduction

Ultra fine grained (UFG) metallic materials have received much attention in recent years due to their enhanced strength and hardness properties [1]. Severe plastic deformation (SPD) processing techniques have been reported to successfully fabricate these nanocrystalline structures by the grain refinement phenomenon [2,3]. The mechanism of grain refinement involves disintegration of grains brought about by the annihilation and arrangement of dislocations through cross-slip and climb. The dislocations arrange to form a dislocation cell structure within the initial grains. The evolution of these dislocation cells during deformation results in a misorientation build-up across the cell walls. Furthermore, once the misorientation value exceeds 10–15°, which is the accepted value for a high-angle boundary, the cell is replaced by a recrystallized grain or a fine grain resulting in grain refinement [4].

Estrin et al. [5] presented a model that predicted the strain hardening behavior of crystalline materials at large strains. The model simulated the dislocation substructures as composite structures made up of dislocation walls and interiors with corresponding evolution laws for the dislocation densities. It was a unified two-dimensional formulation based on the physical mechanisms operating at the dislocation scale. Tóth et al. [6] extended it to a general three-dimensional formulation to predict the strain hardening behavior and dislocation density evolution in copper for various loading conditions.

Equal channel angular pressing (ECAP) is a popular metal processing method to produce ultrafine grained materials with enhanced properties [7]. Baik et al. [8,9] later adopted the dislocation density-based material model to predict the grain refinement phenomenon in equal channel angular processing (ECAP) of IF steel and aluminum respectively. Lemiale et al. [10] showed the model's compatibility under high strain rate impact conditions. The above-mentioned models predicted the evolution of the dislocation mechanisms but did not simulate the microstructural evolution in terms of the grain disintegration by the misorientation accumulation process.

One of the early studies by Derby [11] showed a relation between the steady state recrystallized grain size and the corresponding deformation stress based on an exhaustive survey of experimental studies. The relation is based on measured grain sizes after deformation and it is found that the trends for most metals fall within a narrow range allowing for the development of an empirical relation. However, one of the major challenges faced during the

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study of high strain rate severe plastic deformation processes such as equal channel angular pressing (ECAP) [7], high pressure torsion (HPT) [12], and accumulative roll bonding (ARB) [13] is that though experimental data regarding the microstructural changes indicate grain refinement, very limited data is available with respect to the flow stress behavior of the materials undergoing these processes. Consequently, the mechanism of grain refinement with respect to the initiation and rate of evolution is not known for these high rate deformation processes.

The Kolsky bar or Split Hopkinson bar is a common tool used to characterize the dynamic flow stress behavior of materials [14]. Recently, the Kolsky bar was integrated with the high-speed X-ray imaging capabilities of Synchrotron radiation to track the damage initiation and evolution of a dynamically deforming specimen. This novel data acquisition technique allows for *in situ* monitoring of the microstructure evolution occurring during dynamic loading experiments via ultrafast X-ray diffraction [15,16]. In this study, the experimental approach is developed to track the initiation and temporal evolution of the grain refinement mechanism developed in pure copper when it is deformed at a constant high strain rate. The experiments designed integrate ultra-fast Synchrotron X-rays with a Kolksy bar set-up allowing for *in situ* correlation between the flow stress behavior and the corresponding dynamic grain refinement mechanism developed in the material simultaneously.

The continuous X-rays generated by synchrotron radiation at the Advanced Photon Source (APS) at Argonne National Laboratory have been utilized to track and analyze the *in situ* damage and grain size evolution during the high strain rate loading of copper samples. Furthermore, a probabilistic dislocation density based grain size evolution model has been developed and compared with the experimental measurements.

The first section of the paper details the experimental setup, results from the Kolsky bar and collected images and diffraction patterns. The second section of the paper details the model development and predicted results compared against the collected experimental data.

2. Experimental procedure and results

2.1. Experimental setup

A Kolsky tension bar is utilized to characterize the dynamic response of copper loaded at a constant high strain rate of 1200 s^{-1} which occurs in the realm of 200–300 μs. A modified Kolsky bar has been integrated with the high-energy X-rays at the 32-ID-B beamline of the APS at Argonne National Laboratory to conduct microstructure evolution monitoring via simultaneous high-speed phase contrast imaging (PCI) and X-ray diffraction. The details of the experimental setup in terms of the design of the modified Kolsky bar with the integration of the synchrotron X-rays are reported in Refs. [15,16].

A schematic of the setup is shown in Fig. 1. The setup allows for in-line phase contrast imaging and *in situ* diffraction detection simultaneously. The phase-contrast images are collected using a scintillator-coupled high-speed camera (FastCam SA-Z, Photron USA Inc). The diffraction detection system consists of a scintillator, an image intensifier (Quantum Leap, Stanford Computer Optics Inc), and a high-speed camera (FastCam SA-Z, Photron USA Inc).

The X-ray beamline utilized is equipped with an undulator of period 1.8 cm and length 2.4 m [16]. The undulator gap determines the flux and energy spectrum of the incident X-ray photons. In this study, an undulator gap of 11 mm is chosen for which the first harmonic energy is 23.66 (keV). The calculated raw energy spectrum and the spectrum with absorption effects are shown in Fig. 2.

The collected X-ray diffraction patterns were analyzed using the in-house program *HiSPoD* [17]. The program simulates and analyzes diffraction patterns obtained from polycrystalline samples. For conducting simultaneous imaging and diffraction, the diffraction detector has to be positioned at some offset angle from the incident direction without blocking the transmitted beam. In this experiment, the area detector is placed at an offset angle of 15.4° from the incident beam direction to obtain the strongest diffraction peaks from the sample. A schematic of the diffraction geometry pertaining to the experimental setup is shown in Fig. 3, where O is the point where the X-ray beam and sample interacts; O' is the transmitted beam position on the detector plane; A is the scattered beam position on the detector; q is the scattering wavevector and 2θ is the diffraction angle [17]. For the given detector location and X-ray energy (1st harmonic, 23.66 keV), the scattering vector q and azimuthal angle φ at each pixel position on the detector is calculated using the software *HiSPoD* and shown in Fig. 3.

2.2. Experimental results

The Kolsky bar is utilized to perform high strain rate tensile tests on copper samples with length of 10 mm, width of 1 mm and thickness of 0.07 mm at a constant strain rate of 1200 s^{-1} repeated ten times. The details of the bar setup and properties are reported in Ref. [16]. The incurred strain rate and strain from the sample have been calculated based on the Kolsky bar relations [14] as follows:

$$\dot{\varepsilon} = \frac{c_b}{l_s}(\varepsilon_I - \varepsilon_R) \tag{1}$$

$$\varepsilon = \int_0^t \dot{\varepsilon} d\tau = \int_0^t \frac{c_b}{l_s}(\varepsilon_I - \varepsilon_R) d\tau \tag{2}$$

where c_b is the incident bar acoustic speed which is 5051.86 m/s for the current set up, l_s is the sample gauge length which is 1 mm for the conducted experiments, ε_I is the recorded incident bar strain and ε_R is the recorded reflected bar strain. Due to size restrictions, the transmission bar is replaced by a load cell which allows for the direct detection of the sample force signal [16]. The axial force (F) is detected directly and the corresponding engineering stress is given by $\sigma = \frac{F}{A_0}$, where A_0 is the initial cross-sectional area of the sample.

Fig. 4(a) shows the recorded set of incident and reflected wave forms from the strain gauge during a single loading experiment. Fig. 4(b) shows the calculated (Equation (1)) strain rate history which is a constant 1200 s^{-1}. The accuracy of the recorded stress-strain response is validated by obtaining a constant strain rate signal. Furthermore, the average true stress-true strain response is shown in Fig. 5 with demarcated points at which the diffraction patterns and images were recorded. Error bars marked on the curve show that the observed uncertainty in measurements is negligible. Five phase-contrast images and diffraction patterns are captured during the loading test at a frequency of 20 kHz with exposure times of 250 ns and 20 μs respectively. Concurrently, for the approximate 200 μs loading regime, images and diffraction patterns were captured every 50 μs. The dynamic range of the imaging and diffraction detectors is 10-bit. The field of view includes a pixel resolution of 2 μm and the volume being probed is 0.00672 mm^3. The sequence of phase-contrast images and the corresponding diffraction patterns are shown in Fig. 6. The images indicate the fracture point of the sample during the dynamic loading experiment and the diffraction patterns are analyzed to study the *in situ* temporal grain size evolution in the samples.