MICROMECHANISMS OF DAMAGE AND FAILURE IN POLYCRYSTALLINE MATERIALS FROM X-RAY AND NEUTRON DIFFRACTION

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ABSTRACT: The room-temperature plastic behavior of several FCC alloys was examined with in-situ neutron-diffraction and with polychromatic X-ray microbeam diffraction (PXM). The measurements characterize the local dislocation density distribution as a function of loading and combined with modeling, provide insights into damage and failure in polycrystalline materials. Both, monotonic-tension and low-cycle-fatigue experiments were conducted as a function of stress. The plastic behavior during deformation is discussed in light of the relationship between the stress and dislocation-density evolution. The observed dislocation density evolution finds that the monotonic tensile and low-cycle-fatigue samples have similar dislocation densities at small strain, but that latter have much lower dislocation densities than the former at high strain.

INTRODUCTION: Plastic deformation in polycrystalline solids is the focus of intense research due to an increasing demand for high-performance structural materials. Of central importance is the need to understand the mechanisms that control damage and which ultimately lead to failure. The recent development of powerful X-ray and neutron sources allows nondestructive materials characterization at the length scales needed to advance our understanding of damage nucleation and evolution. For example, nondestructive three-dimensional (3D) measurements of submicron strain and unpaired dislocation-density distributions are now possible due to an emerging class of instrumentation: the 3D x-ray crystal microscopes. These instruments use ultra-intense synchrotron x-ray sources and advanced x-ray optics to probe polycrystalline materials with submicron x-ray beams. By employing polychromatic x-ray microbeams (PXM) and a virtual pinhole camera method, called differential aperture microscopy, 3D distributions of the local crystalline phase, orientation (texture) and elastic and plastic strain tensor distributions can be measured with submicron resolution in all directions [Larson *et al.* 2002; Ice and Barabash, 2007].

Neutrons also provide valuable information about damage processes and can be used to study in-situ bulk deformation within gauge volumes that are sufficiently large to provide good statistical information, but are sufficiently small to avoid surface or geometrical complications. Because neutrons are nondestructive, measurements can be made in-situ on a single sample, which avoids the difficulties of ex-situ or multiple specimen

experiments [Huang *et al*., 2007]. Here we report on x-ray microdiffraction measurements made on world's first 3D X-ray Crystal Microscope on station 34-ID-E at the Advanced Photon Source (APS). We also report on related measurements made on the Spectrometer for MAterials Research at Temperature and Stress (SMARTS) at the Los Alamos Neutron Science Center (LANSCE). The combination of these two diffraction methods provides insights into the plastic behavior of our samples and their defect evolution with load history.

PROCEDURES, RESULTS, AND DISCUSSIONS: The HASTELLOY® C-22HS™ nickel-based superalloy samples (Ni-21Cr-17Mo in weight percentage) were hot-rolled and mill annealed at 1,080°C. TEM studies find that this processing dissolves most alloying elements and precipitates and results in a faced-center cubic (FCC) solid-solution alloy. Strong texture in the alloy was not observed. The average grain-size of this alloy is about 90 μm.

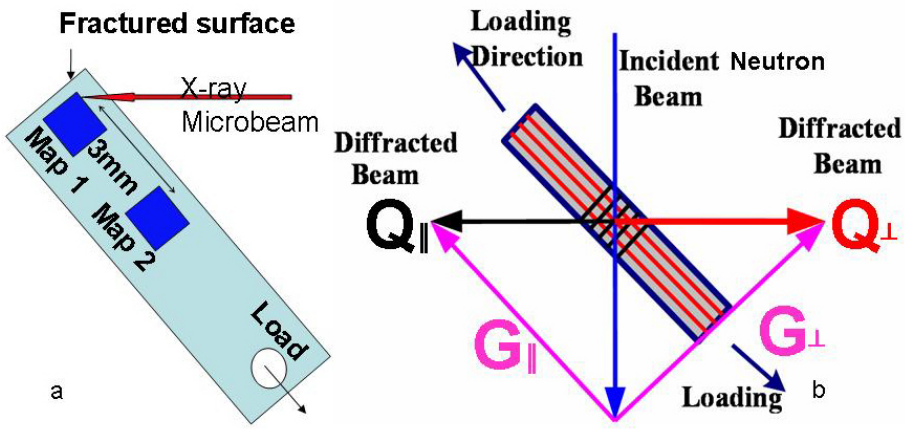

Figure 1. Scheme of the two mapped areas for PXM analysis (a) and two different probing directions in neutron diffraction parallel and perpendicular to loading direction (b).

In-situ neutron diffraction measurements were made on the samples during loading to follow the dislocation density evolution with load history. The low-cycle-fatigue experiments were conducted with a minimum strain and maximum strain of -1% and +1%, respectively. The frequency of the loading cycle was 0.5 Hz. The neutron-diffraction data were collected during the fatigue experiments at: 1st, 4th, 8th, 12th, 30th, 50th, 100th, 250th, 500th, 1,000th, 1,250th, 1,500th cycles and failure. For these studies, the specimens were aligned at 45° to the incident neutron beam with two detectors fixed at $2\theta = \pm 90^{o}$ (Fig.1 b). The gauge volume of the in-situ neutron measurements is 120 mm^3. The total dislocation density was estimated from broadening of the peak widths. Neutron diffraction data show that in the same (not very large) true stress region (400~600 MPa), the hardening of the monotonic tension and cyclic loading produce similar dislocation densities ($10^8 \sim 10^9$ cm^{-2}). However, at larger stresses, greater geometrically necessary dislocations density (GNDS) is found with monotonic tensile deformation. To further understand the origin of these differences, the samples were studied ex-situ with high-resolution polychromatic x-ray microdiffraction (PXM).

The geometry for the polychromatic X-ray microbeam (PXM) measurements is shown in Figs.1a. The gauge volume in PXM measurements was $0.5 \times 0.5 \times 50\mu m^3$. PXM measurements measure the local distribution of GNDs and lattice rotations. Measurements of the samples after failure probed two different areas: one area was located near the fractured surface (map 1 in Fig.1a) and one area was centered 3mm from the first area (Fig.1a). The two areas were mapped with 2D and 3D scans at 1 μm pitch. Both the GND density and deviatoric strain tensor distributions were obtained in the mapped regions. After 15% monotonic tensile straining, the GNDs density is so high that Bragg spots are smeared almost beyond detection. With low-cyclic-fatigue (LCF) loading, the diffracted Laue radiation could be measured up to the failure. The most interesting results were observed in the boundary region between grains (Fig. 2). In the boundary region all Laue spots are streaked (elongated) (Fig. 2c) indicating the presence of strong strain gradients in the immediate vicinity of the boundary. This buildup of defects near grain boundaries has important implications for eventual failure and can be compared to emerging theoretical models.

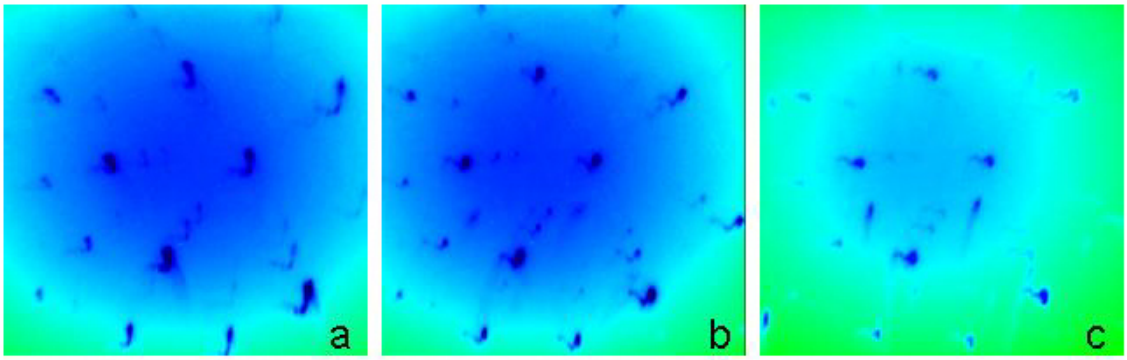

Fig. 2. Laue patterns in the boundary region between two grains within the Map 1 in the region of the sample near the fractured surface after cyclic failure: (a) 10 μm from the boundary; (b) partially covering the boundary; (c) in the middle of the boundary region.

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