Spatially Resolved MicroDiffraction Analysis of the Plastic Deformation in the Shock Recovered Al Single Crystal

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Abstract
A spatially resolved diffraction method with a sub micrometer-diameter beam and 3D differential aperture technique together with MD simulations, SEM and OIM analysis are applied to understand the arrangements of voids, geometrically necessary dislocations and strain gradient distribution in samples of Al (123) single crystal shocked to incipient spallation fracture. We describe how geometrically necessary dislocations and effective strain gradient alter white beam Laue patterns of the shocked materials. We show how to quantitatively determine the orientation and density of geometrically necessary dislocations in the shock recovered Al samples being initially oriented for single slip.

Introduction
Strong shock waves result in the transition from elastic to plastic compression. As a result of dislocation motion and the strong interaction between dislocations and elastic waves, an initially random dislocation distribution becomes unstable and forms a correlated dislocation arrangement into dislocation walls. Some fraction of the dislocations may remain randomly distributed, and the rest form various correlated groupings and more organised disclination arrangements. Regions with geometrically necessary dislocations may form causing local lattice curvature.

Aluminum has been the object of numerous shock experiments. Reshock and release of shock - compressed aluminum was studied previously by Lipkin and Asay[1]. Recently developed in-situ time resolved x-ray diffraction relates in-situ pseudo Kossel line broadening with dislocation density during the shock experiments[2 - 4]. Effect of stress triaxiality on void growth in dynamic fracture of metals was studied with molecular dynamic simulations[5]. We complement these results with white (polychromatic) X-ray microbeam study of the meso-scale geometrically necessary dislocation arrangement in the (123) Al single crystal. The single crystal Al samples were shocked to incipient spallation fracture on the LLNL light gas gun as was done by Stevens *et.al.*[16]

Experimental
The x-ray synchrotron measurements were made with a 3D X-ray crystal microscope[6] based on polychromatic (white) beam with the energy range 6 - 25 eV. This method allows for true 3D mapping of the crystalline phase, orientation and plastic deformation with unprecedented spatial resolution. A region of interest is identified using an optical microscope and the polychromatic x-ray beam is focused onto the sample. The orientation at each position of the crystal is precisely determined by an automated indexing program. Because polychromatic microdiffraction produces Laue images from

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each deformation cell with slightly different orientation intercepted by the x-ray beam, the typical problem is an overlap of many Laue patterns that must be disentangled. The process of disentangling can be carried out either by a direct experimental technique called differential aperture microscopy[6] or by pattern matching/ absorption sensitive methods. Once the Laue pattern from a subgrain volume has been obtained and the indices of each reflection determined, powerful single-crystal methods can be employed to determine the unit cell parameters. Although differential aperture microscopy (DAXM) can often resolve Laue image into single crystal-like patterns, when the density of unpaired or "geometrically necessary" dislocations (GNDs) is high, streaking is observed even for microsize regions in DAXM resolved patterns[7 - 9]. Typically streaking in Laue patterns indicates the presence of lattice rotations within the probed region as a result of the GNDs formation. Data collection has been carried out using microbeam Laue diffraction on beamline 34ID at the Advanced Photon Source. Dimensions of the beam were 0.5 by 0.5 microns with a penetration depth of ~ 500 micron. Details on the experimental setting and data collection can be found elsewhere[6 - 9].

The sketch of the shocked Al single crystal and the geometry of initial and diffracted white beam radiation and the microbeam geometry are shown in the Fig. 1. Oriented for single (123) Al single crystal with initial dimensions: diameter 5 mm and thickness 5 mm (123) Al single crystal was cut along the shock direction and analyzed at different locations of the sample cross section (Fig 1b).

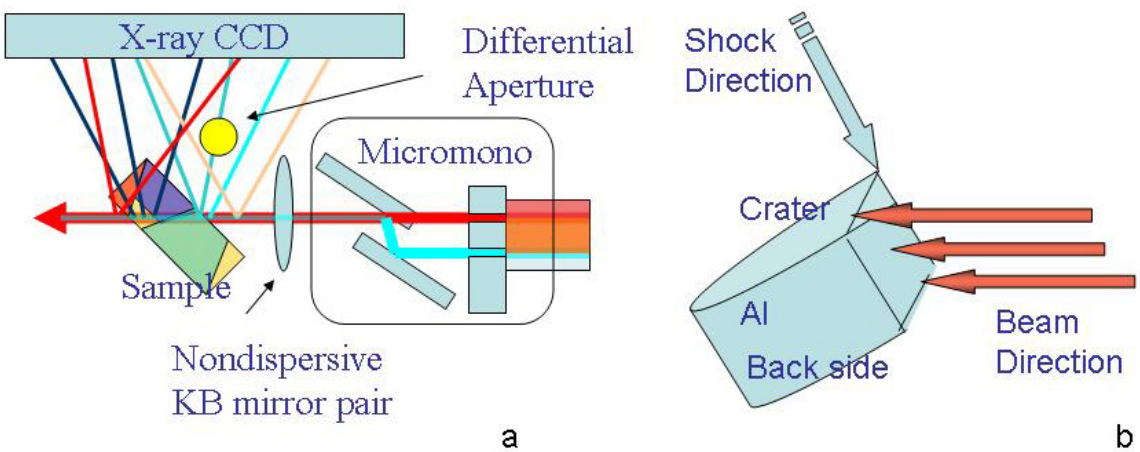

Figure 1. 3D polychromatic microbeam geometry: wire (yellow circle) is shadowing different portions of the diffracted intensity on a CCD (a); sketch of the Al single crystals orientation during shock experiment and orientation relative to microbeam (b).

Results and Discussion

A number of experimental studies of plastic deformation under different loading conditions show that typically the cell-wall structure with alternating regions of high and low dislocations density is formed[8 - 13]. The dislocation walls contain high density of dislocations, and cell interiors have very low dislocation population. Shock often causes formation of high density of voids in materials[16].

An approach to characterize the dislocation structure and determine the activated slip systems by analyzing the streaked Laue pattern using a multiscale hierarchical framework is described in detail in the Refs. [7 - 9]. In the white beam diffraction the full width at half maximum of the streak (ξ direction) $FWHM_{\xi} \equiv \delta m_{\xi}$ is a function of the

misorientation within the probed region caused by geometrically necessary dislocations. It depends on the orientation and the number of unpaired or geometrically necessary dislocations (GNDs) in the probed volume. The orientation of the GNDs influences the character of Laue spot. In the transverse direction ν, the $FWHM_\nu \equiv \delta m_\nu$ depends on the total number of all dislocations (GNDs and statistically stored) per unit length and usually $\delta m_\xi >> \delta m_\nu$.

Under multiple slip the Laue intensity distribution depends on the dislocation density tensor ρ_{ij}. The element of dislocation density tensor can be written as:

$$\rho_{ik} = \tau_i b_k \delta(r). \tag{1}$$

The magnitude of the vector, $\boldsymbol{\tau}$, is the net number of dislocations having Burgers vector, $\boldsymbol{b}$, crossing unit area normal to $\boldsymbol{\tau}$ [14]. Following the approach[15] in the framework of strain gradient plasticity, the total GNDs and GNBs density tensor ρ_{ij} relates to the strain gradient tensor η_{lmk};

$$\varepsilon_{ilm}\eta_{lmk} = -\rho_{ik}; \qquad \eta_{lmk} = \partial^2 u_k \big/ \partial x_l \partial x_m \tag{2}$$

Here ε_{ilm} is the anti-symmetric Levi-Civita tensor. Under multiple slip, intensity distribution around each reciprocal lattice point and the streak direction ξ of the Laue spot depends on the aforementioned dislocation density tensor. Here u_k, the k-th component of the displacement field $\boldsymbol{u}$ for any unit cell, is due to all dislocations in the crystal.

We calculate the total displacement of the i-th cell $\boldsymbol{u}_i$ from the equilibrium positions $\mathbf{R}_i^0$ corresponding to the undeformed crystal using continuum elastic theory. This displacement is due to all dislocations and is defined using random numbers c_t by the equation[6 - 8]:

$$\mathbf{u}_i = \sum_t c_t \mathbf{u}_{it}; \qquad c_t = \begin{cases} 1, (+) \\ 0, (-) \end{cases} \tag{3}$$

If there is a dislocation at position $t, c_t = 1$, and for positions without dislocations $c_t = 0$. Dislocation density tensor ρ_{ik} gives the sum of the Burgers vectors of the dislocations whose Burgers vector and line direction are directed parallel to x_k and x_i axes (k, i =1, 2, 3), respectively. These are set as an initial input parameter for simulation of the Laue pattern. By simulating Laue images with different GND slip systems, we can determine the set of slip systems that are best fit to the local lattice curvature and experimentally determined Laue image. The analysis of the dislocation substructure for multiple slip deformation includes the following steps:

- Determination of the local lattice curvature (misorientation axes and angle) from the experimental Laue pattern at each probed location.
- Simulation of Laue pattern using these parameters to check its identity with the original experimental image.
- Performing least square fit of the streak profile find the components of the dislocation density tensor.

The polychromatic x-ray microdiffraction (PXM) technique is applied to a complicated dislocation structure arising from the shock induced plastic/elastic deformation of the Al (123) single crystal. The microbeam-Laue diffraction reveals several distinct zones located at different depth under the shock front (indicated by several red arrows in Fig.1 b). Pronounced streaking of Laue images are observed in the zones 1 and 6 close to the front and back surface of the after shock (123) Al single crystal. (Fig. 2).

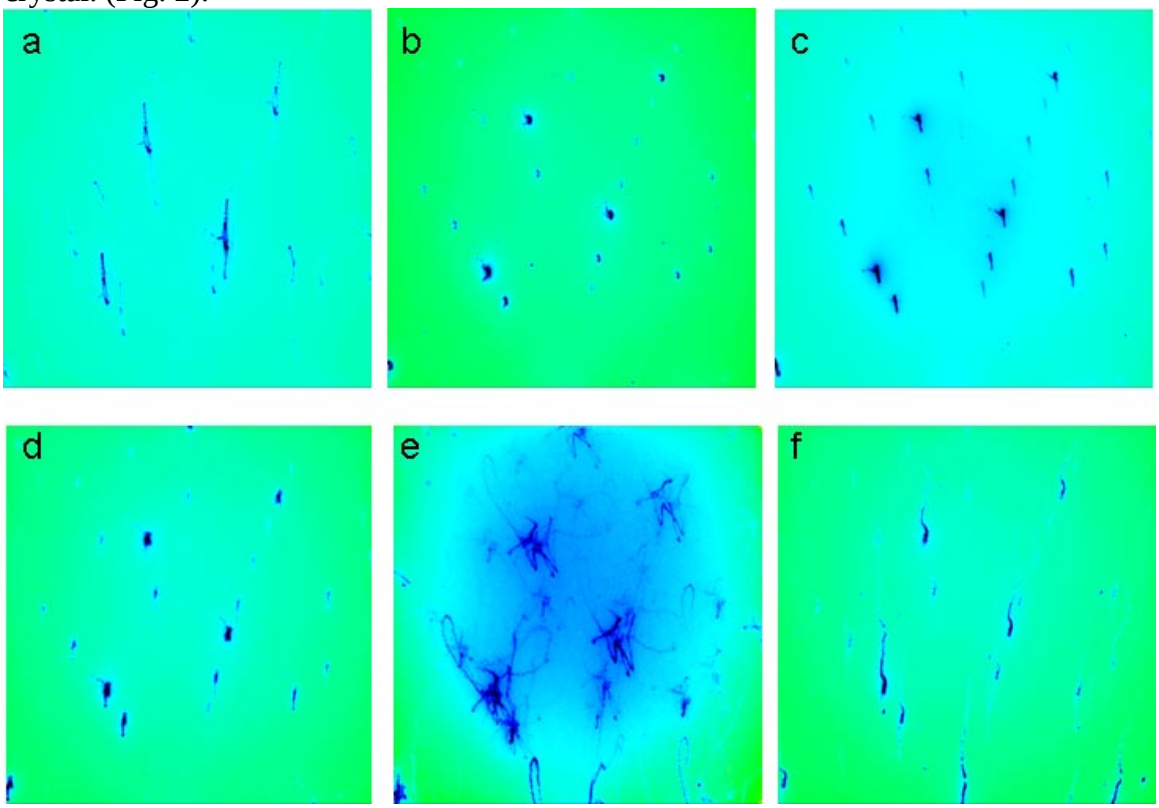

Figure 2. Laue images obtained at different depth under the shock front of (123) Al single crystal: a) zone 1 near the shock front; b).zone 2;c) zone 3; d)zone 4; e)zone 5 with corresponding to the region of intensive void formation; f) zone 6 near the back surface of the shocked single crystal.

Much less streaking is observed in the zone 2, 3, 4 located approximately in the upper half of the sample. The most interesting images correspond the zone 5 (Fig. 2 e). This zone corresponds to the region in which X-ray tomography, SEM and OIM indicated intensive void formation (Fig.3).

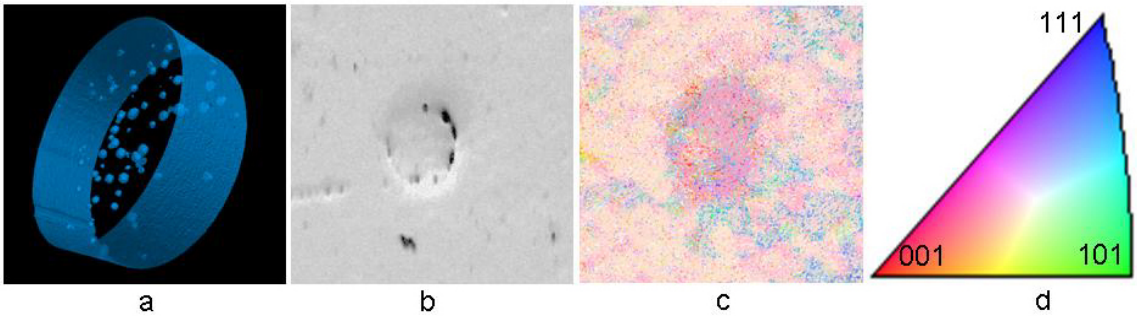

Figure 3. X-ray tomography shows region with high concentration of mesoscale size voids in the spall region (a), SEM image of the void at the surface of the cross section of the after shock Al single crystal chosen for PXM analysis, OIM image of the orientation change around the same void (c) and color scale of orientation distributions (d).

To get a better understanding of the reasons for such a peculiar shape of the Laue image the 3D depth resolved measurements were performed. These differential aperture measurements allow tracking the change in the intensity at different depth along the beam. They showed that in this region alternating local lattice rotations take place (Fig.4). We argue that such alternating local rotations are caused by the presence of voids creating regions of plastic deformation in the vicinity of the void. This result agrees well with SEM and OIM observations.

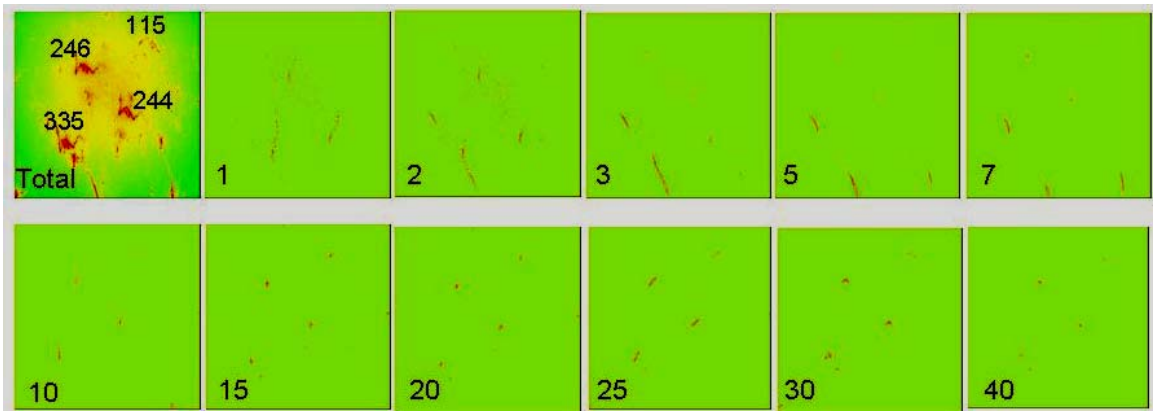

Figure 5. Total (left top corner) and depth resolved Laue patterns from the spall region with voids. Depth is shown in microns at each depth resolved images. Total Laue pattern is indexed for convenience.

Conclusions

The microbeam-Laue diffraction reveals several distinct zones located at different depths under the shock front. Pronounced streaking of Laue images are observed in the zones close to the front and back surface of the after shock (123) Al single crystal. They indicate the formation of geometrically necessary dislocations being consistent with a single slip mode in the near surface regions. In the depth of the Al single crystal the portion of geometrically necessary dislocations in the dislocation ensemble reduces, and in the same time the portion of statistically stored dislocations increases. An intensive mesoscale void formation is observed in the region close to the center of the samples, which is accompanied by a peculiar complicated shape of the Laue diffraction images.

The density and organization of dislocations is presented as a function of depth under the shock front as well as comparison to SEM on the same samples.

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