

MATERIALS CHARACTERIZATION BY MICROFOCUSSED HIGH ENERGY X-RAYS

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Abstract

Micro-focussed high energy synchrotron radiation has been employed for the structural characterization within polycrystalline bulk materials. Essential features of the developed techniques are: (i) the observation of true bulk properties as opposed to surface effects such as strain relaxation or abnormal grain growth (ii) the definition of 3 dimensional gauge volumes down to micrometer length scale and (iii) fast data acquisition enabling *in-situ* experiments during thermo-mechanical processing. X-ray energies from 50 to 90 keV have been employed enabling bulk investigations even in materials of medium and high atomic number.

The main instrumental components are broad band micro-focussing optics, a sample stage with a high concentricity rotation axis, sample environment (deformation devices and furnaces), 2 dimensional detectors for parallel data acquisition, and conical slit cells which are placed behind the sample. Techniques have been developed to define a gauge length parallel to the incident beam within the sample. Software has been developed aiming at on line data reduction.

Engineering strain/stress and texture mappings were performed and in this case statistical assemblies of grains or domains are observed. Narrow gauge dimensions are of interest to follow strain/stress and texture gradients as they typically exist in *e.g.* coatings or at buried interfaces. Examples include strain gradients across buried interfaces in CuNi multilayers, the strain evolution during the selective surface aging of a β -titanium alloy, and measurements of the triaxial strain tensor across torsion samples. The grain averaging is improved by sample oscillation perpendicular to the strain gradient.

The behavior of polycrystalline materials under thermo-mechanical processing is often described by phenomenological laws. The observation of individual grains or domains should provide decisive tests of the underlying physical principles. Examples of such observations will be presented.

The kinetics of individual nuclei during recrystallization of an Al alloy have been observed. It is found that the nucleation time and the growth rate differ substantially from nucleus to nucleus which is not explained by current models.

The orientation of the grains and the shape of their boundaries have been mapped within bulk samples. The nondestructive nature of the technique therefore provides the initial sample configuration prior to processing.

Plastic deformation has been studied by observation of the rotation pathway of individual grains during uniaxial tension. The experimental grain rotations are inconsistent with classical Taylor and Sachs models.