Workshop Report:

Probing Mechanical Deformation and Failure via Synchrotron X-rays

Workshop at SSRL Users' Meeting, October 8, 2003

A joint SSRL-ALS workshop on "Probing Mechanical Deformation and Failure via Synchrotron X-rays" was held on Oct. 8th, in conjunction with SSRL's Annual Users' Meeting. The workshop began with presentations by the co-organizers, Apurva Mehta and Nobumichi Tamura, on various X-ray techniques utilized for probing materials.

Apurva Mehta indicated that there are two broad types of synchrotron techniques useful for looking at mechanical deformation and failure. The first class of techniques comprises of diffraction probes at various spatial resolution, from sub-micron to mm. This class of techniques gives information about the state of strain, and orientation of grains in the material. Diffraction techniques have been the dominant probes of mechanical deformation so far. Nobumichi Tamura discussed them in depth, paying special attention to the synchrotron-based techniques of diffraction with microprobe. Not only did he show numerous examples to demonstrate the scope of the technique, but he also showed many mechanical deformation applications, which were useful for discussion throughout the workshop. The other class of techniques includes various types of microscopy and 3-D tomography. Special attention was drawn to the advantages of relatively high-energy (15 – 50 keV) phase contrast tomography.

The rest of the workshop was devoted to talks and discussions on how the classical approach to the understanding of mechanical deformation and failure is becoming increasing insufficient if not coupled with information on the effect of deformation at the microstructural level; and how the synchrotron hard x-ray based techniques, probing materials at tens of nanometers to sub-mm scale, are becoming increasingly important in developing a better understanding of materials under stress.

Materials scientists and mechanical engineers, over the last century, have developed a complex framework of theories and models to understand and predict deformation and failure of structural components. Many of these theories are based on the assumption that mechanical materials are homogeneous and continuous. However, a large fraction of structural materials, for instance, most of the metals and all of the ceramics, are polycrystalline, and therefore, inherently discontinuous and highly anisotropic on microns and submicron scales. The effects of the microstructural discontinuity and anisotropy are sometimes visible even in large structures made from these materials. Bill Nix showed how the hardness of a material, he used Cr-Ni-Mo steel as an example, increases with decreasing grain size. Brad Boyce showed examples of how the residual stresses even in a "bulk" materials (of several mm in dimensions) deviate from prediction of the classical models due to incomplete inclusion of hard to model transient and thermal effects. Valentina Imbeni used the example of a NiTi endovascular stent to show how industrial devices with complex geometry invariably experience complex multi-axial loading in vivo, this complex behavior deviates significantly from idealized and computed stress-strain responses. She showed the comparison of the elastic and plastic strain measured at the ALS versus the prediction of a finite–element model used in the biomedical industry for a corner of a sample with a stent-like geometry under load. The comparison showed how the measured stresses deviated from the calculations due to local defects, but more significantly, due to the inability of the models to account for the athermal Austenite to Martensite phase transition. She then showed results of a more fundamental study that indicated that the stress response of NiTi thin tube is dramatically different in tension versus torsion and cannot be explained yet by modeling that does not include microstructural features and the austenite to martensite transformation.

Also, mechanical failure generally occurs via initiation and propagation of crack. Cracks can begin at localized defects or at the point of highest stress concentration. Don Shockey and Jeff Simons showed simulations of crack formation and propagation based on crystal plasticity theory.

Zofia Rek showed images of crack formation, propagation and closure using absorption and phase contrast tomography and discussed advantages and difficulties of phase contrast tomography for visualizing cracks.

An increasingly larger proportion of technological products are becoming miniaturized. The mechanical (and micromechanical) properties of these new devices, for example, thin films or MEMS devices, deviate significantly from the prediction of the classical theory of continuum mechanics. Nobumichi Tamura showed examples of how the stresses in a real MEM device deviated from a classical finite-element calculation. Sometimes these deviations are sufficiently large to cause these devices to malfunction.

A better understanding and ultimately an optimized design of these small scale structures is possible only if we define newer theories, theories that go beyond the assumptions of continuum mechanics and that also include microscopic features such as grain boundaries, slip bands, dislocation walls and directionally anisotropic response of these structures under applied load. Brad Boyce introduced the concept of micro and macrostress to contrast the mechanics occurring at the microstructural level and the measurements of "bulk" mechanical responses in strain gages and force meters.

James Stolken gave a detailed overview of six of the prominent new theories that provide some understanding of the physics behind deviation from continuum mechanics and proposed experiments that could help us discriminate among them.

Monica Barney showed some preliminary results from a set of experiments specifically designed to contrast between two classes of the theories mentioned by Stolken.

Erica Lilleoden and Bill Nix suggested that for a better understanding of nano-scale structures and in general for a better understanding of the deviations from the theory of continuum mechanics, one needs newer techniques to characterize mechanical behavior at the nanoscale level. They proposed fabricating test structures, such as custom engineered thin-films with idealized grain structure, cantilever microbeams, FIB-machined micro-compression structures, and to measure the micro and macrostress response of these structures using micro and nano indentation in-situ in electron microscopes and microprobe x-ray diffractometers. Erica Lilleoden showed many experimental examples, which showed how not only the macrostress response of these structures, but also the microstructural response of the material deviates from the classical theories and current understanding.

The workshop ended with an open discussion session. All the participants agreed that more macrostress studies such the one presented by Brad Boyce on fatigue failure caused by foreign object damage, or the one presented by Valentina Imbeni that showed dramatic deviation of mechanical response of NiTi tubes under multi-axial loading from current mechanics theories must be continued. However, it was felt that there is even a greater need for a fundamental understanding microstructural responses of materials. Two of the greatest challenges in microstress mechanics are the mechanisms of plastic deformation, and crack formation and propagation. The technique of phase contrast tomography combined with micro-probe x-ray diffractometry has a lot of promise for the study of cracks. But a better understanding of plastic deformation, it was felt, is not possible without a better understanding of accumulation and motion of dislocations. Shape and widths of Bragg peaks and dynamical contrast mechanism in electron diffraction are very sensitive to dislocation density, and efforts in relating the measurements of dislocation density to macrostress plastic deformation, the discussion group felt, must be strongly encouraged. It was also pointed out that sensitive probes of dynamical contrast in x-rays, such as x-ray topography, have great advantages over electron microscopy for measurement in a load rig due to the large penetration depth. But these techniques are underdeveloped and infrequently utilized for probing mechanical deformation and more attention should be paid to them in future.