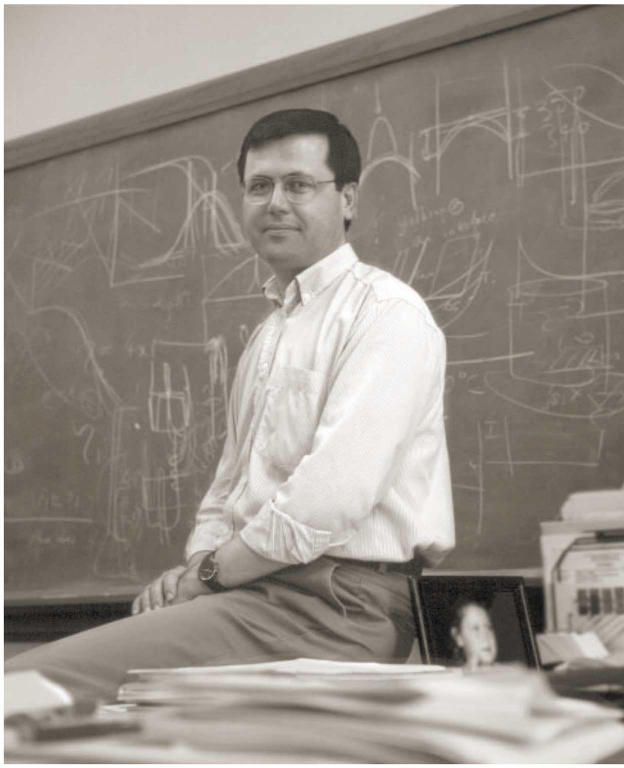
Understanding Material Deformation: Insights into the Inner Workings of Complex Materials

by Ersan Üstündag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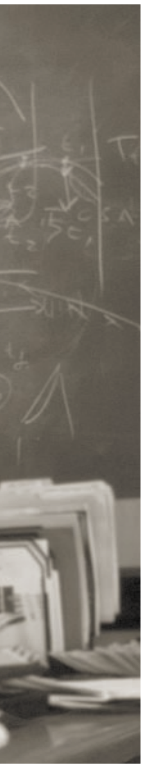
In most engineering calculations, the mechanical performance of structures or components is estimated under the assumption that the material is homogeneous or can be represented by a continuum. Although this assumption is often sufficient, it prevents a true understanding of deformation mechanisms, as most structural materials are actually composites (comprised of multiple phases) and/or polycrystals (composed of many grains). It turns out that the interactions between phases and grains largely determine the overall behavior of the material. These interactions occur over multiple length scales, from nanometers to centimeters. Any experimental technique that intends to fully characterize material deformation must be sensitive to such a scale range. The technique must also be non-intrusive, as it should not cause damage while interrogating the material. Another important requirement is that the technique should allow *in-situ* studies, that is, monitoring of material deformation under a variety of conditions, such as applied load, temperature, or atmosphere.

Diffraction is a powerful technique for material characterization, and easily satisfies these requirements. Especially attractive methods are x-ray and neutron diffraction, as they provide *in-situ* information about internal strains (and indirectly, stresses), crystallography (to help identify different phases), and texture (or preferred grain orientation). Diffraction techniques use a material's crystalline lattice as an "internal gauge," and are therefore sensitive to changes occurring on the atomic scale. In addition, when a large sampling volume is chosen, contributions from many regions are included in the overall "signature" of the material, leading to an effective averaging or bulk characterization. X-ray and neutron diffraction can be used independently or in a complementary manner, as the former can probe sub-micrometer regions while the latter is more suitable for *in-situ* bulk studies on the scale of millimeters to centimeters.

In our research, we employ both x-ray and neutron diffraction for a complete, multiscale characterization of material deformation. Our aim is to develop accurate constitutive laws describing the behavior of a composite or a polycrystal. Accurate description of constitutive behavior is crucial for successful modeling of material behavior, including prediction of expected lifetime. We anticipate that our models will be valuable to engineers designing and constructing complicated structures or devices as varied as jet turbine engines, cars, buildings, satellites, and electronic chips.

This report details one important aspect of our research, namely the use of neutron diffraction in deformation studies. It also describes our recent efforts to design and construct a dedicated engineering neutron spectrometer called SMARTS. Much more than a catchy acronym (standing for Spectrometer for MAterials



Research at Temperature and Stress), SMARTS is currently unique in the world. It is the first instrument specifically designed for engineering stress/strain studies at a spallation neutron source. Located at the Los Alamos Neutron Science Center (LANSCE) in New Mexico, it was commissioned in 2001. It is funded by the Department of Energy (Office of Basic Energy Sciences), and was built by a team led by the author.

SMARTS is expanding the use of neutron diffraction to a wider range of engineering problems than was previously possible. With its extensive array of *in-situ* capabilities for sample environments, it enables measurements on small (1 mm^3) or large (1 m^3) samples. Ease of access to the sample bay is one significant new feature. Components with dimensions up to 1 meter and mass up to 1,500 kilograms can be positioned precisely in the path of the neutron beam. Permanently mounted alignment theodolites provide a simple and efficient way to position samples or equipment to within 0.01 mm. Achieving this level of precision is critical for stress-strain measurements; misalignments of more than 0.1 mm can result in significant pseudo-strain artifacts.

A furnace and load-frame suite allows research on materials under extreme loads (60,000 pounds or 250 kN) and at extreme temperatures (1,500°C or 2,700°F). *In-situ* uniaxial loading on samples 1 cm in diameter at stresses over 3 GPa under vacuum or in a controlled atmosphere is now routine. This represents a significant increase over previous standards. Some of the exciting capabilities provided by SMARTS include measurements of spatially resolved strain fields; phase deformation, and load transfer in composites; the evolution of stress during high-temperature fabrication; and the development of strain during reactions or phase transformations.

The layout of SMARTS is shown in Figure 1. At LANSCE, neutrons are produced by spallation, which involves accelerating protons to very high energies toward a tungsten target, then collecting the polychromatic neutrons that form. These neutrons pass through a water moderator, which reduces their energies to a range suitable for diffraction. After passing through the T_0 chopper, a device which further removes fast neutrons and the gamma flash (to minimize background), the thermal neutrons reach the guide. The guide is coated with ^{58}Ni, and, via the process of near-total reflection, keeps most of the neutrons in the beam path. The guide terminates at the inner surface of the cave wall. Two aperture sets (located between the exit of the guide and the sample) permit the beam cross-section to be defined continuously in shape and area between 1 and 100 mm^2.

When the neutron beam penetrates a sample, some of the neutrons interact with atoms in the material and scatter in all directions. Some of these reach one of the two detector banks centered on the horizontal plane at 90° to the incident beam. Each detector consists of three panels with a total of 192 ^{3}He gas-filled aluminum tubes. Interactions between the neutrons and ^{3}He in the detector tubes produce ^{4}He plus gamma radiation and ionize the gas, creating a cascade of electrons with associated charges. These charges are digitized and converted electronically to patterns of intensity versus scattering angle. Data from the tubes are combined to provide time-of-flight neutron-diffraction patterns. Analysis of the diffraction patterns is carried out with a least-squares fitting routine called the Rietveld method. Data acquisition is based on virtual memory extension technology and uses web-based visualization and control software.

Experiments can be controlled remotely from the user's laboratory (anywhere in the world), and real-time data analysis can be accomplished with a unique software package called *Expert System*. This software represents a radical new approach to experiment planning and execution in the neutron-diffraction field. For the first time, the experimenter has a chance to optimize an experiment according to his/her needs and predict results even before starting. Moreover, during the experiment, data are

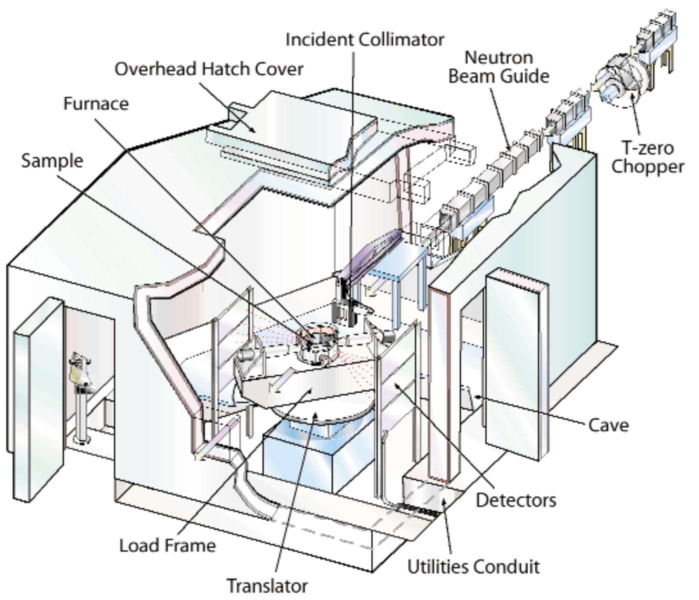


Figure 1. Neutrons from the moderator pass through a series of collimating apertures before entering the neutron guide. A T_0 chopper removes fast neutrons and gamma flash that would otherwise contribute unwanted background. Slow thermal neutrons continue down the guide to the entrance of the SMARTS cave (about 5 x 6 m in size). On exiting the guide, neutrons pass to the center of the cave where some are scattered by the sample to the detectors. Samples or ancillary systems are placed directly on the translator, which can accommodate up to 1,500 kg, move in three orthogonal directions, and rotate about a vertical axis. Theodolites provide precise optical triangulation and alignment capability for equipment or samples. Here, the load-frame-furnace suite is shown on top of the translator. In some experiments where a three-dimensional sampling volume is desired, radial collimators are inserted between the detectors and the sample. When used with the incident collimation, selection of an appropriate radial collimator defines a sampling volume for spatially resolved measurements.

analyzed in real time, allowing a quick assessment of the results. Figure 2 describes the interactions between the user and the various components of Expert System.

First, the user is asked to input detailed material data and the strain error desired. The software then simulates the expected diffraction pattern. This calculation incorporates realistic models of the instrument optics so that the simulated pattern is truly representative of the sample. In addition, based on the experimental parameters (e.g., tension vs. compression) and specimen characteristics (e.g., monolith vs. composite), Expert System will soon be able to perform several mechanics calculations that will simulate the stress-strain behavior of the material. This is necessary to determine the optimum data-collection points so that all critical events during a material's deformation (for instance, its yield point) can be captured. Another planned upgrade involves optimization of experimental conditions using inverse problem analysis. This will yield the mathematically most optimum set of data points required to obtain a desired outcome in the shortest possible time. The latter issue is important since beam time is very expensive. For this reason, the current version of Expert System includes real-time data analysis to determine the exact time when enough data have been collected to satisfy the user's specified initial error value.

Expert System was mostly programmed by a group of undergraduate students led by Richard Karnesky (BS '02, past president of Ricketts House) who is now pursuing a PhD degree at Northwestern University. Other contributors include Justin Fox (currently a senior in E&AS) and Dr. Bjorn Clausen (of Los Alamos). The software was written in Java, so it can be used on different computing platforms and run over the Internet. We expect it to be adopted by various national facilities, both for neutron and x-ray diffraction. When this occurs, robust data comparison between these facilities will be achieved for the first time. This is also expected to lead to standardization of engineering stress/strain measurements using diffraction. The outcome will likely be a rapid growth of the field and its application to a multitude of materials science and engineering

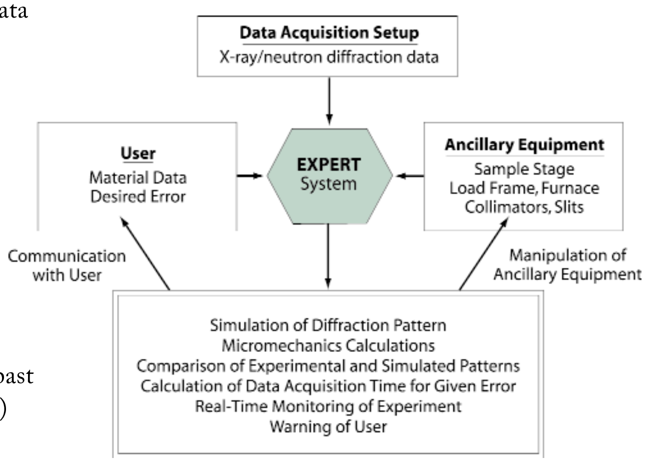


Figure 2. Schematic illustration of the working principle of the SMARTS Expert System software.

problems in both academe and industry.

During the commissioning phase, we used SMARTS for a variety of projects. In a study funded by NASA, we investigated high-temperature deformation mechanisms in structural ceramics and ceramic-matrix composites. Some of these materials are already in use in new jet turbine engines, but before they can be employed further, it is necessary to understand their "creep" behavior. Creep refers to permanent (i.e., inelastic) deformation at high temperatures. This understanding will allow us to construct advanced models that predict the lifetime of these materials under demanding conditions (temperatures above 1,200°C, highly corrosive atmospheres, and so on). Since SMARTS is able to provide temperatures similar to those found in a jet turbine, we collected *in-situ* crystallographic data for the first time for one of the most important structural ceramics, Si_3N_4. The diffraction data (including lattice plane specific strains) were used in a self-consistent model to calculate the elastic stiffness tensor of this material at this temperature—a calculation previously unattainable. In late 2002, additional Si_3N_4 tests were conducted in the creep regime. The results suggest that grain rotation and boundary sliding are active creep mechanisms. This is the first time that they have been observed *in situ.* The data are now being used to develop a new mechanics model.

We have also used SMARTS to study bulk metallic glass (BMG) matrix composites developed at Caltech by Professor Bill Johnson's group. These composites retain the high strength of BMG but improve it further by providing ductility and damage tolerance. Our aim was to understand deformation mechanisms in these composites and to identify the best reinforcement material and its morphology. Some BMG matrix composites require applied stresses over 2 GPa to fully observe their deformation. However, since they include heavy elements (such as zirconium and tungsten) that absorb x-rays, neutron diffraction (and SMARTS specifically) is the only technique available to study *in-situ* deformation of the reinforcements under high applied stress.

Due to its amorphous nature, the BMG matrix cannot be interrogated directly with diffraction to obtain lattice-strain data. However, we were able to use diffraction data to develop new mechanics models (finite-element or self-consistent) that allowed deduction of the behavior of the BMG matrix. We showed that in all composites, the metallic reinforcements yield first and then start transferring load to the BMG matrix. The matrix later deforms by initiating multiple shear bands that make it "plastic," enhancing the overall ductility of the composite. The full micromechanical details of these events are still not fully understood however. To achieve greater understanding, we have started working on model specimens suitable for high-energy x-ray diffraction studies. By combining the neutron-diffraction data we have obtained so far with the spatially resolved x-ray diffraction data, we intend to elucidate the complete, multiscale deformation mechanisms in BMG matrix composites.

In short, the SMARTS system we have built together with the Expert System software allow unprecedented experimental capabilities that are revolutionizing our ability to characterize materials *in situ* under a variety of environmental conditions close to what materials will actually encounter. This is expected to lead to a better understanding of how various materials fail, and how we can improve the design of practical systems, such as aircraft, cars, engines, buildings, and even microdevices, to avoid such failure.

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There is more on Professor Üstündag at
http://www.matsci.caltech.edu/people/faculty/ustundag_e.html
and more about his project at
http://smarts.caltech.edu