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Spallation Neutron Source

A Summary of the

Breakout Session on Basic Mechanical Properties

held as part of the JINS Workshop on Application of Neutron Scattering to Materials Science and Engineering Oct. 1-3, 2001, Oak Ridge, Tennessee

T. M. Holden and C. R. Hubbard
Discussion Leaders

Documented by

Xun-Li Wang
Experimental Facilities Division
701 Scarboro Road
Oak Ridge, TN 37830

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The breakout sessions were held as part of the JINS workshop on Application of Neutron Scattering to Materials Science and Engineering, Oct. 1-3, 2001 in Oak Ridge, Tennessee. The session on Basic Mechanical Properties was led by C. R. Hubbard and T. M. Holden. A list of session participants is given in the Appendix.

Since the 1980's, neutron diffraction has been used to study stress and texture. Much of the research in the early days was strongly oriented towards mechanical engineering, involving the determination of residual stress distribution in components and the use of residual stress data in the design and life predictions. Opportunities for fundamental research began to emerge when it became evident that some of the experimental data could not be understood within the framework of continuum theory and simple thermal-mechanical simulations.

In a broad sense, basic research in mechanical properties can be defined as finding the correlation between the structure and macroscopic mechanical properties. The influence of crystal structure, or phase transformation, is the subject of another breakout session and has been summarized separately. In the absence of a phase transformation, the mechanical properties are closely related to the microstructure. The role of neutron diffraction is then to characterize the relevant features of the microstructure that influence the mechanical properties of the material. The list of these features includes, but not limited to,

(1) Strain/stress, a measure of the dimensional distortion;
(2) Texture, a measure of the change in grain orientation
(3) Dislocation density, a measure of the substructure
(4) Grain morphology

In general, the strain, stress, dislocation density, and grain morphology are also grain-orientation dependent, i.e., textural quantities. The grain-orientation dependent stress is also known as the intergranular stress. Neutron diffraction is well suited for the determination of strain, stress, and texture. Dislocation density can be determined by analyzing the detailed peak shape profile of broadened diffraction peaks. Limited information on grain size and shape may also be obtained from the peak shape analysis. When the grains of interest are of nanometer size, small angle neutron scattering will become a useful tool. Simultaneous diffraction and small angle scattering measurements, as would be realized with the VULCAN diffractometer at the SNS, will be an added advantage for characterizing the microstructure of deformed grains.

It should be noted that neutron diffraction as a tool for characterizing the microstructure is complementary to electron microscopy. For fine-grained polycrystalline materials, neutron diffraction is a bulk probe. The measurement volume is usually on the order of ~mm^3 and the highest spatial resolution will be limited to ~0.1 mm even with the SNS VULCAN diffractometer. Thus, a neutron diffraction measurement typically samples over thousands of grains, providing a statistical representation of the measured quantity. For materials consisting of large grains, neutron diffraction provides an opportunity to study the behavior of individual grains. The ability to measure multiple reflections allows the investigation of the evolution of texture and intergranular stress, simultaneously. Together, these two quantities give insight on how grains of different orientations deform at different stages of deformation. Finally, with neutron diffraction, in-situ experiments can be readily realized. In fact, load frame and furnaces are becoming part of standard suite of sample environment at many neutron scattering facilities. The lately commissioned SMARTS diffractometer at LANSCE will allow, for the first time, in-situ study of uni-axial deformation at elevated temperatures.

In this session, a number of issues were discussed in the use of neutrons for basic research of mechanical properties. The text below is intended to capture key issues or aspects that have not been documented previously [1].

Deformation of polycrystalline materials

Over the last decade, intergranular stress, along with texture, has been used as fingerprints to understand the deformation in polycrystalline materials. Theoretical studies were carried out to understand the intergranular stress data obtained with neutron diffraction. Most of the theoretical works were based on the numerical methods, in which the stress states of individual grains are modeled by considering the physical mechanisms that produce the residual stress during plastic and/or elastic deformation. These numerical methods include Taylor model [2], self-consistent models [3-5], and the finite element method [6]. However, while these models are able to produce a qualitative description of the stress states in individual grains with different orientations, the calculated strains sometimes differ from the experimental values by more than 30% [5], especially for samples subjected to a large degree of plastic deformation. New models therefore must be developed in order to quantitatively understand the intergranular stress and hence the deformation behavior in polycrystalline materials.

On the experiment side, most experiments were performed for simple metals under uni-axial loading. Few studies have been carried out for materials under large, multi-axial deformations, including rolling, forging, equal-channel angular pressing, swaging, and extrusion, which all have technological importance. For some alloys, phase transformation occurs during deformation and its effect on the intergranular stress, especially the orientation relationship between the different phases, is largely unexplored. Ultimately, the experimentally determined intergranular stress will establish the guidelines for the development of theoretical models for quantitative simulation of the deformation behaviors at large deformations.

Complex materials represent another major research opportunity in intergranular stress. Composite materials is one example. By understanding how the applied load is partitioned between the matrix and reinforcement, a primary advantage of composites, new insights into cyclic fatigue will be obtained. Other interesting material systems include intermetallics, bulk amorphous materials, nanocrystalline materials, and carbon nanotubes. There is already evidence that different deformation mechanisms operate when the grain size is reduced to nanometers range.

In addition to the texture and intergranular texture, the dislocation density is also an important measure of the deformed state of the grains. The orientation anisotropy of the dislocation density, or stored energy, provides information on the grain substructure in the deformed state. Additionally, it is also a driving force for recrystallization texture which will be discussed below.

High temperature behaviors

Materials are often processed at high temperatures and their high temperature behaviors have a pronounced influence on their mechanical properties at application temperatures. For this reason, materials behaviors at elevated temperature must be well characterized and in-situ, time-resolved experiments are the key.

Recrystallization is a phenomenon exhibiting many unanswered questions and it plays a significant role in controlling the properties of commercial alloys. Recrystallization proceeds by nucleation and growth processes. If any of these processes is selective with regard to crystallographic orientation, a crystallographic texture develops. In-situ measurements of texture evolution will provide detailed information regarding grain growth and movement during recrystallization. By correlating the change in intergranular stress and stored energy, it will be possible to reveal the roles of intergranular stress and stored energy on the recrystallization process. These measurements will lead the way for the development of models for reliable prediction of recrystallization texture. In the same manner, in-situ experiments will help to understand the transformation texture due to diffusion-controlled phase transformation. The ultimate goal of recrystallization studies in practical applications is to find ways to modify and control the textures in industrial productions.

Creep occurs at sufficiently high temperatures, which may alter the mechanical properties of the materials at application temperatures. With equipment that allows in-situ loading at elevated temperatures, such as the one available on SMARTS at LANSCE, the effects of creep may be addressed. Moreover, the process of cavitation may be characterized with small angle neutron scattering, at least in the initial stage of cavity growth. Often, phase transformation occurs during heat-treatment. Even if a phase transformation does not occur, subtle changes in the crystal structure may take place during the heat-treatment. For example, in Mo-Mo_3Si composites, composition analysis reveals that there is a transfer of Si from the Si-enriched Mo phase to Mo_3Si during the heat-treatment at 1600°C, which may be partially responsible for the cracking.

The study of high temperature behaviors is critical for understanding the solidification process. Typically, during solidification, the temperature field within the specimen is non-uniform. The coupled interaction between the temperature, time, atmosphere, stress, and phase stability leads to a gradient in chemical composition, microstructure, and stress field. The challenge has been to describe these interactions comprehensively. Thus for experimental studies of the solidification process, the experiment will have to be done in-situ, fast, and perhaps spatially resolved as well. With the unprecedented intensity at the VULCAN diffractometer, carefully designed experiments may be carried out to investigate the solidification process in, for example, the welding and heat-affected zones of a weld. Despite years of progress towards understanding the rich variety of metallurgical phenomena happening in the welds, some of the basic issues in welding research have not been addressed, including the effect of phase transformation, influence of precipitation, effect of recrystallization and recovery, development of microvoids and cavities, and the correlation between the microstructure and residual stress.

Environmental effects

Here the term environmental effect is referred to anything but high temperature, which has been discussed earlier. Radiation changes the mechanical properties through the modification of the microstructure. For example, helium bubbles are generated in irradiated components. Because helium is essentially insoluble in metals, it is thermodynamically favorable for the entrapped helium to precipitate as bubbles/microvoids/cavities along grain boundaries. Helium bubbles nucleate, grow and coalesce rapidly at grain boundaries under combined actions of high temperature and tensile stresses. A practical implication of this effect is that during repair welding of irradiated components, brittle rupture occurs along grain boundaries when the cohesive strength of the grain boundary (weakened by growing helium bubbles) can no longer bear the shrinkage-induced internal tensile stress during cooling of the weld. In order to account for the effect of helium bubbles in computer models for simulating repair welding, it is essential the process the nucleation and growth of the helium bubbles are characterized and understood.

Hydrogen embrittlement is another longstanding issue. Because the amount of hydrogen in the trapped region is small (~ ppm), the detection of it has been quite difficult. Lately, some progress has made using the incoherent elastic scattering from hydrogen, but quantifying the amount of hydrogen at a given location remains a challenge.

Often materials also experience thermal-chemical treatment either during fabrication or use. For example, carburization and nitriding have long been used to improve the fatigue properties of steel components by introducing compressive stresses in the surface layer. With appropriate sample environments, the entire thermal-chemical process can be studied in-situ with neutron scattering. In the case of nitrided steel, it has been found that at long nitriding times, the surface stress turns into tensile. Computer simulation suggests that this was due to the dissolution of carbides in the nitrided layer. To verify this prediction, simultaneous diffraction and small angle neutron scattering measurements will be desirable.

Relation of stress to failure

Previous studies of failure were mostly based on the continuum theory, where the stress field is continuous and varies over a macroscopic scale. The effect of intergranular stress has been considered but mainly as an aide for the interpretation of the experimental stress data determined with x-ray or neutron diffraction. It is unclear how the intergranular stress is directly related to failure. Basic research involving in-situ tensile and fatigue tests will be needed to address the effect of the intergranular stress on tensile fracture and fatigue life. Since the presence of tensile stress is necessary for stress-corrosion cracking to occur, it is conceivable that the intergranular stress influences the cracking of materials subjected to a corrosive environment. Indirectly, the intergranular stress affects the materials performance in several ways. In alloys where the phase transformation is selective with respect to the crystallographic axes, for example, the intergranular stress may radically alter the properties of a material via the influence of its transformation behaviors. Also, it has been suggested that the intergranular stress plays an important role in the recrystallization texture.

Characterization of materials of large grains

Ultimately, the local interactions between and within individual grains determine the mechanical property of the polycrystalline materials. In this regard, materials made of large grains (grain size ~ 0.1 mm or greater) offer a great opportunity to probe basic interactions in mechanical properties at the level of individual grains with neutron diffraction. Particular issues include the orientation of grains, the inter- and intra-granular stresses, features across the interface and transition zones, and chemistry and microstructure changes during crystal growth. Experimental techniques are presently being developed to enable complete characterization of individual grains in the three-dimensional microstructure. Example materials system includes the technologically important nickel-based superalloys used in gas turbine engines.

Other Issues

While the technique for measurements of single grains are being developed, a code of standard practice have been drafted under the auspices of VAMAS for measurements of residual stress in fine-grained samples. Results from round-robin measurements with four sets of carefully selected specimens (a shrink fitted ring and plug, a ceramic composite, a multi-pass weld, and a shot-peened plate) were used to produce the draft standard. Once the standard is issued, it may be used as guidelines for novice users in the future.

Development work is needed for methods leading to unambiguous determination of the macro stress when significant intergranular stress is present. When a large number of reflections is measured, as is the case with pulsed neutron sources, the macro stress is an 'average' of all measured reflections which could be handled in the Rietveld analysis software if a proper model for the 'averaging' is developed. Lately, a new analysis method, the Spherical Harmonic Approach, has been proposed which greatly facilitated the separation of the macro and intergranular stresses via the construction of the stress orientation distribution function.

User-friendly data analysis software is a key for quick turn-around of a user project. Efforts should be given to the development of software that will instantaneously reduce the data to a format suitable for further analysis with conventional software.

References

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APPENDIX

List of Session Participants

Bourke, Mark
Los Alamos National Laboratory
MS H805
Los Alamos, NM 87545
USA
Tel. 505-665-1386
Fax 505-665-2676
bourke@lanl.gov

Brown, Don
Los Alamos National Laboratory
MST-8, MS-H805
Los Alamos, NM 87545
USA
Tel. (505) 667-7904
Fax (505) 665-2676
dbrown@lanl.gov

Gallego, Nidia C.
Oak Ridge National Laboratory
Metals and Ceramics
Bethel Valley Road
Oak Ridge, TN 37830
USA
Tel. (865) 241-9459
Fax (865) 576-8424
gallegonc@ornl.gov

Gnaupel-Herold, Thomas
NIST/University of Maryland
Center for Neutron Research
100 Bureau Dr., Stop 8562
Gaithersburg, MD 20899-8562
USA
Tel. (301) 975-5380
Fax (301) 921-9847
thomas.gnaeupel-herold@nist.gov

Holden, Tom M.
Los Alamos National Laboratory
MS H805
Los Alamos, NM 87545
USA
Tel. (613) 584-4373
holdent@magma.ca

Horton, Joe
Oak Ridge National Laboratory
Metals and Ceramics
P.O. Box 2008
Bldg 4500S MS 6133
Oak Ridge, TN 37831-6133
USA
Tel. (865) 574-5081
Fax (865) 574-4066
hortonja@ornl.gov

Hubbard, Camden
Oak Ridge National Laboratory
High Temperature Materials Laboratory
Bldg. 4515, MS 6064
Oak Ridge, TN 37831
USA
Tel. (865) 574-4472
Fax (865) 574-3940
hubbardcr@ornl.gov

Larson, Bennett C.
Oak Ridge National Laboatory
Solid State Division
Bldg 3025
Oak Ridge, TN 37830
USA
Tel. (865) 574-5506
Fax (865) 574-4143
bcl@ornl.gov

Marschman, Steven C.
Pacific Northwest National Laboratory
Radiochemical Science and Engineering
P.O. Box 999, Msin P7-27
Richland, WA 99352
USA
Tel. (509) 376-3569
Fax (509) 376-9781
steve.marschman@pnl.gov

Stoica, Alexandru D.
Oak Ridge National Laboratory
Spallation Neutron Source Project
701 Scarboro, Room 240
Oak Ridge, TN 37831
USA
Tel. (865) 574-0350
Fax (865) 241-5177
stoicaad@ornl.gov

Wang, Xun-Li
Oak Ridge National Laboratory
Spallation Neutron Source Project
701 Scarboro Road
Oak Ridge, TN 37830
USA
Tel. (865) 574-9164
Fax (865) 241-5177
wangxl@ornl.gov

Wang, Yandong
Oak Ridge National Laboratory
Spallation Neutron Source Project
701 Scarboro, Room 240
Oak Ridge, TN 37831
USA
Tel. (865) 574-0337
Fax (865) 241-5177
wangy@ornl.gov

Wang, Jy-An
Oak Ridge National Laboratory
P.O. Box 2008
MS6370
Oak Ridge, TN 37830
USA
Tel. (865) 574-2274
Fax (865) 574-4018
wangja@ornl.gov

Yang, Wenge
Oak Ridge National Laboratory
Solid State Division
P.O. Box 2008, MS 6030
Oak Ridge, TN 37830
USA
Tel. (865) 574-6505

Zhang, Ying
Oak Ridge National Laboratory
Metals and Ceramics
P.O. Box 2008
Oak Ridge, TN 37830
USA
(865) 574-4452
(865) 241-0215
zhangy@ornl.gov