Tutorial on Engineering Applications of Neutron Diffraction

Lecture 1: Introduction to Neutron Diffraction

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Neutron diffraction has long been recognized as a valuable technique for analytical interpretation of crystalline and amorphous structures. As will be demonstrated in subsequent presentations in this workshop, the applications are far-reaching, and in fact, continually expanding. New instruments are emerging at existing facilities, and soon at the Spallation Neutron Source (SNS) at Oak Ridge National Laboratory, that should dramatically expand the importance of neutron diffraction to applied science and engineering research. This introductory presentation will provide an underpinning for other presentations, by reviewing the mathematical and physical basis for neutron diffraction, distinctions between x-ray and neutron scattering, and the features and uses of Reactor- and Spallation-based neutron sources. This will include descriptions of single crystal and polycrystalline diffraction, along with small angle and disordered materials diffraction. A brief review of the components of powder diffractometers will introduce the audience to the important issue of optimizing instrument designs to maximize performance and best serve the scientific needs of the neutron scattering community. We will review the many unique properties of neutron scattering that complement x-ray scattering. Among these are the high penetrating power of neutrons (up to 15 cm into samples), enhanced scattering contrast between neighboring elements (e.g., Mn vs. Fe, H vs. D) and unusually strong scattering from light elements (H, D, Li, O). The high penetration power of neutrons, for instance, is critical for studies of sub-surface strain gradients, chemical reactions or phase transformations in materials under industrially-relevant conditions, and bulk microstructure (e.g., preferred orientation, strain) measurements in macroscopic materials. Some examples will be described to illustrate these features.

Lecture 2: Introduction to Neutron Stress Measurements in Engineering Components

Aaron Krawitz
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University of Missouri, Columbia, Missouri USA

The scattering of neutron beams by engineering materials offers a means of measuring stress in actual engineering components. The method is nondestructive and enables determination of complete stress tensors to depths of several millimeters below the surface, in gage volumes down to about one cubic millimeter. Furthermore, stress or strain may be measured in specific phases, and in desired crystallographic and physical directions, of engineering components. Since the initial use of neutrons to measure stress 20 years ago, the method has developed steadily in the US and Europe, and, more recently, in Asia. A number of engineering instruments are now in operation at reactor and pulsed source, with VULCAN - here at the Spallation Neutron Source - promising to be the world leader. The basic physical ideas enabling neutrons to be used to determine residual (and applied) stresses in engineering components are presented. A small set of concepts about diffraction, stress and strain, neutrons, and engineering materials allows the capabilities and potential of neutron stress (and texture) measurement to be appreciated. These concepts will be used to introduce the notion of diffraction to measure stress, and the use of neutrons in particular. A number of experimental aspects will be addressed, followed by some examples.

Lecture 3: Quantitative Texture Analysis by Neutron Diffraction

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Real materials, as found in industrial components, are rarely single crystals, nor powders. They are typically polycrystalline aggregates, with preferred orientations of crystallites as a result of anisotropy in materials processing routes. As single crystal properties are often direction-dependent, the preferred orientations of crystallites can therefore lead to anisotropy in the behaviour of bulk materials, with regard to elastic properties, thermal expansion, yield behaviour, creep and so on. Diffraction is a powerful method for the quantitative analysis of the preferred orientations of crystallites in polycrystalline materials - i.e. of crystallographic texture. Neutron diffraction is particularly well suited for analysis of texture because neutrons probe the interior of bulk material and are often able to provide meaningful scattered-intensity information at every orientation of a specimen. This introductory presentation will outline the concepts of relative orientation of crystal-fixed and specimen-fixed coordinate systems, orientational probability distributions, the series-expansion method for representing an orientation distribution function, techniques for measuring texture by diffraction, presentation of data, and subsequent analysis, along with a few illustrative examples.

Lecture 4: Probing the Microstructure of Industrial Components

Stephen Spooner
Oak Ridge National Laboratory

Residual strain scanning is the pre-eminent application of neutron scattering to industrial components. The influence of grain size and texture on the feasibility and design of neutron scattering experiments is illustrated in cases of industrial strain scanning. The presentation is expanded to a more general discussion of advantageous neutron scattering properties that permit the consideration of additional applications of neutron scattering. The use of small angle scattering for the characterization of a variety of metallurgical structures is featured.

Dinner Speaker: Social Implications of Stress Analysis

I. C. Noyan
IBM Research Division, T. J. Watson Laboratory, Yorktown Heights, NY

Stress analysis with diffraction techniques can be used on structures with very different sizes. Such analysis is critical to efficient design, and efficient design may influence human life on the global scale. In this talk, we will discuss stress effects in microchips and aircraft structures.

Lecture 5: Development of an International Standard for the Measurement of Residual Stress by Neutron Diffraction

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Residual stresses can have an important influence on the performance of engineering components. In order to quantify their effect it is necessary to know their magnitude and distribution. Neutron diffraction is a relatively new method for determining residual (and applied) stresses in crystalline materials. It is similar to the X-ray technique for surface determinations. However because neutrons are not charged, neutron diffraction can be used to obtain residual stresses non-destructively to a depth of several centimetres in most materials of practical interest. No standard is currently available for making these measurements. An international project, under the auspices of VAMAS (Versailles Agreement on Advanced Materials and Standards), Technical Working Area 20 (TWA 20) was initiated in January 1996 to carry out the under-pinning research necessary to develop a standard. The investigation involved most of the neutron sources worldwide, which are capable of making the measurements. A series of 'round-robin' specimens including a shrink fit aluminium alloy ring and plug assembly, a ceramic matrix composite, a nickel alloy shot-peened plate and a ferritic steel weldment were examined. These examples were chosen to establish the range of application of the technique. In each case a protocol was specified which each participating group was required to follow. Measurements were made at each neutron source independently. The results were then collected together and statistical analyses carried out to determine the reliability of the results. The study was supported by a European (EU) project RESTAND (Residual Stress Standard using Neutron Diffraction) which was started in December 1997 to demonstrate the usefulness of the technique to a range of practical applications and to develop confidence in the method for industry. This presentation gives the background to the VAMAS TWA 20 and RESTAND projects. It outlines the main findings and indicates the precautions that are required to achieve accurate positioning and alignment of specimens (and components) in the neutron beam and the analysis required to obtain reliable results. It will be shown that special attention is needed in dealing with near-surface measurements because of surface aberration effects. It is demonstrated that, provided the recommended procedures are followed, a positional tolerance of ± 0.1 mm can be achieved with an accuracy in strain of $\pm 10^{-4}$ to give a resolution in residual stress of ± 7 to 20 MPa in most materials of practical interest. Some examples of the residual stress distributions that have been measured in a variety of components are included to illustrate how they can be used in engineering design and for making component lifetime predictions.

Lecture 6: The Challenges of Stress Measurement by Neutron Diffraction

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In principle, the fact that neutrons penetrate easily into most engineering materials is the key that opens the doorway for straightforward, reliable, "fool-proof" scanning of residual stresses in intact engineering components. However, one must be aware that elastic constants and intergranular (type II) stresses can vary significantly with (hkl); thus, the choice of diffraction peak can have a crucial effect on the assessment of macroscopic residual stress distributions. Also, strain is often evaluated by comparing crystal lattice spacings of the object in question with that of a "stress-free" reference specimen. One must be aware of the issues that might undermine the reliability of stress-free reference values. Crystallographic texture (preferred orientations of crystallites in polycrystalline materials) can be an advantage, a nuisance or a potential killer for reliable stress measurements. Specimens with a coarse microstructure (by which we mean that there is a very sparse population of grain orientations within the sampling volume) demand techniques that no longer assume the measurement of "powder peaks". Analysis of steep, sub-surface stress gradients, in the depth range of 0.1 to 1.0 mm continue to stretch the capabilities of neutron stress scanners. Evidently, the unwary practitioner of neutron stress scanning might stumble into a number of traps. However, the challenges that arise at the frontiers of "routine residual analysis" provide the stimulus for some of the cutting-edge developments that are happening today.

Symposium on Fundamental Studies Of Materials Phenomena Using Neutrons

Neutron Residual Stress Mapping Facility

Cam Hubbard
Oak Ridge National Laboratory

SMARTS and HIPPO Capabilities

Mark Bourke
Los Alamos National Laboratory

Materials Science and Engineering Facilities at Chalk River For Academic and Industrial Researchers

John Root
National Research Council of Canada
Chalk River Laboratories
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Neutron Scattering at Intense Pulsed Neutron Source

Jim Richardson
Argonne National Laboratory

The Engineering Program at the NIST Reactor

Thomas Gnäupel-Herold and Henry J. Prask
National Institute of Standards and Technology

VULCAN at the SNS

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The VULCAN diffractometer at the SNS is designed to tackle a broad range of problems in materials science and engineering. The primary use of this instrument is for stress-related studies, including mapping of the residual stress distribution in components and the determination of deformation behaviors under applied load. Other uses include in-situ measurements of materials kinetics in chemistry, stress, texture, and microstructure. For mapping experiments, the measurement time with a sampling volume of 1 mm^3 will be minutes. This will make possible parametric studies of components subjected to different fabrication or operating conditions. Load-frame and furnaces will be an integrated part of the instrument. With its high flux and a large detector array, VULCAN is also ideally suited for the study of texture and transformation kinetics. Equipped with a 2D area detector, which would allow simultaneous measurements of small angle neutron scattering data, VULCAN will become a great tool for understanding the intricate interplay between stress, texture, and microstructure in materials.

Studies of Plastic Deformation and Recrystallization in Metals By Neutron Diffraction and Other Methods

Dorte Juul Jensen
Center for Fundamental Research: Metal Structures in Four Dimensions
Materials Research Department, Riso National Laboratory
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Neutron instruments developed and built for studies of thermomechanical processes in materials are described. This includes a texture instrument for fast in-situ measurements, and an instrument for measurements of internal stresses. Both instruments make use of a position sensitive detector. Various examples of applications illustrating the potentials of the instruments are reviewed, and the necessity for neutron diffraction in these investigations is discussed. The examples are mainly limited to plastic deformation and recrystallization, but also examples of more direct practical applicability are included. In the last part of the talk, the potentials of other methods based on electron microscopy and synchrotron x-ray diffraction for material science investigations are shortly discussed, and a suggestion for a future next generation neutron instrument is finally given.

Small-Angle Neutron Scattering Studies of Porosity in Ceramics

Andrew J. Allen
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Single and multiple small-angle neutron scattering (SANS), when used together with electron microscopy, precision density measurement and other techniques, are particularly powerful in addressing microstructure characterization issues in porous ceramic systems. For example, direct, statistically-significant descriptions of the pore microstructure of a ceramic as a function of sintering can be obtained over an extended structural scale range from one nanometer up to one micrometer. Anisotropic extensions of these methods enable microstructure issues to be addressed in preferentially-aligned ceramic morphologies such as occur in coatings. The advent of near-surface SANS studies even allows microstructures to be characterized in sub-millimeter thick thermal barrier coatings *in situ* on the substrate. These neutron scattering applications will be discussed in relation to building an understanding of the significant properties of various ceramic systems.

A Neutron Diffraction and Self-Consistent Modeling Study Of the Deformation of Beryllium.

Don Brown
Los Alamos National Laboratory

We have completed an *in-situ* neutron diffraction loading study of the deformation of beryllium in uniaxial tension and compression. We have observed an asymmetry in the deformation of beryllium between tension and compression and have tried to relate this to thermal residual stresses developed on cooling due to the anisotropic coefficient of thermal expansion of beryllium. The results of the neutron diffraction study are compared to self-consistent model predictions for the purpose of validating the model. Finally, the understanding gained by studying the deformation in uniaxial stress fields will be applied to a more complex part in a three-dimensional stress field, that is, a set of girth welded beryllium rings.

Dinner Speaker: Coherent and Incoherent Viewpoints of Scattering from Materials

Brent Fultz
California Institute of Technology

Scattering can be elastic or inelastic, and coherent or incoherent. We can form four combinations of these word pairs (elastic, coherent), (elastic, incoherent), (inelastic, coherent), (inelastic, incoherent), and the neutron experimenter can make use of all of them. This complexity is not typical of most work with x-ray or electron scattering, which tend to use only the pairs (elastic, coherent) and (inelastic, incoherent). Diffraction experiments (elastic, coherent) are an expedient channel for neutron scatterers to make contact with most of the community of materials science and engineering, but the large expansion of neutron capabilities in the U.S. promises entirely new directions for materials research. It is fun to speculate on where this could go. As a field of study, materials science and engineering is itself not entirely coherent. The evolution of internal stress during a phase transformation would fall comfortably under the umbrella of materials science, but what about studies to find energy gaps in dispersive magnetic excitations? I will present handy, although opinionated, rules-of-thumb for separating real materials scientists from the imposters.

Fundamental Interactions in Martensitic Transformations Revealed By Inelastic Neutron Scattering

S. M. Shapiro
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Inelastic neutron scattering is a unique tool for revealing the strength and the origin of interatomic interactions. This has been demonstrated for over 40 years in the study of dramatic temperature dependent phonon behavior occurring in structural phase transitions. Materials that exhibit shape memory effects have very many useful applications and undergo a type of transition known as "Martensitic Transformation". This will be demonstrated in a simple thermodynamic experiment. Details of neutron experiments on some simple systems will be presented. The results, combined with theoretical calculations, will show the origin of the transformation is the strong electron-phonon coupling occurring in these metallic alloys.

Local Disorder and Neutron Scattering: from Catalyst To High-Temperature Superconductors

Takeshi Egami
University of Pennsylvania

The purpose of structural studies in materials science is not the structure determination *per se*, but understanding the structural underpinning of the properties so that we can improve them. For this reason, determination of the crystal structure often is not sufficient, since many properties are controlled by more subtle features of the structure, such as defects and other deviations from perfect periodicity. In fact many of the materials of current technological interest are complex crystals with internal disorder, and controlling the nano-scale local structure is the key to optimizing the properties. Conventional crystallographic methods that rely upon the analysis of Bragg peaks are not ideal in dealing with such issues. Instead we have been using the method of atomic pair-density function (PDF) analysis, which has been widely used in the study of liquids and glasses. We discuss how such an approach made an impact to the study of real materials, ranging from ceria/zirconia catalyst support in automotive exhaust control, to colossal magnetoresistive oxides and high-temperature superconductors.

Dynamics of Disordered Materials*

David L. Price
Argonne National Laboratory, Argonne, IL 60439 and CNRS-CRMHT, Orleans 45071, France

This talk will review the insight into the dynamics of disordered crystalline materials, glasses and liquids provided by inelastic and quasielastic neutron scattering. It will begin with a review of the various techniques and the time and energy scales that they probe. This will be followed by some illustrative examples of recent work at Argonne and elsewhere on vibrations and relaxation processes in disordered crystalline phases such as plastic crystals and fast-ion conductors; phonons and low-frequency excitations in glasses; and translational and orientational relaxation in molecular liquids and polymer melts. *Work supported by the Office of Science, US Department of Energy, under Contract W31-109-ENG-38.

Applications of Neutron Diffraction to Residual Strain Related Problems

Judy Pang
Metals & Ceramics Division, Oak Ridge National Laboratory

Neutron diffraction has been used for residual strain measurements for more than a decade. The ability of neutrons to penetrate deeply through a wide-range of materials makes neutron diffraction an ideal tool to measure residual strain development in components in bulk non-destructively. The underlying principle of diffraction also means that neutron diffraction is an atomic strain gauge; hence the residual strains of different phases and grains of different orientations can be identified separately. Applications of neutron diffraction in the study of the generation of residual strains during inertia welding of Ni superalloys and the development of interphase strains in multiphase materials will be described.