13th Canadian Neutron Scattering Summer School Experiments

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Overview

The 13th Canadian Neutron Scattering Summer School will be held at Chalk River Laboratories on May 15-19, 2017. The school is organized and hosted by the Canadian Neutron Beam Centre, Canada's neutron beam user facility operated by Canadian Nuclear Laboratories. The school is sponsored by the Canadian Institute for Neutron Scattering (CINS).

The school is aimed at graduate students and post-doctoral researchers with backgrounds in physics, chemistry, or materials science who may have no prior experience in using neutron beams to study materials. The curriculum covers a wide range of scientific areas, such as magnetism, industrial alloys, and thin films.

Students will participate in **two** sessions relevant to their research interests. Each session will include in-depth introductory lectures, hands-on experience on the beamline at the NRU reactor, and detailed data analysis after the experiment. At the end of the summer school, students will be in a position to apply neutron scattering methods to their own research projects.

Important

Students are asked to review the experiment descriptions and select their three most preferred experiments when registering.

Two of the experiments, *Rietveld Analysis* and *Triple-Axis Neutron Scattering Study of the Magnetic Properties of MnF2* include 2 sessions that build upon one another and will take the whole week.

Experiment Schedule

Session 1 (Tuesday-Wednesday)	Session 2 (Thursday – Friday)
• Rietveld Analysis (Part 1) • Triple-Axis Neutron Scattering Study of the Magnetic Properties of MnF_2 (Part 1) • Crystallographic Texture	• Rietveld Analysis (Part 2) • Triple-Axis Neutron Scattering Study of the Magnetic Properties of MnF_2 (Part 2) • In-Situ Deformation of a Zirconium Alloy • Hydrogen and Deuterium Absorption in Thin Ti Films

Hydrogen and Deuterium Absorption in Thin Ti Films (1 session)

Introduction

Neutron Reflectometry (NR) can give the chemical depth profile in thin films with thicknesses ranging from sub-nanometer up to about 200 nm. The high sensitivity of neutrons to hydrogen and deuterium enables NR to detect absolute hydrogen concentrations in the at.% range even in nm-thick layers. Therefore, NR is an ideal tool to study *in-situ* the hydrogen/deuterium absorption and desorption properties of thin films on a nanometer scale, without the need of a calibration sample.

In a NR experiment, the neutron beam hits the surface of a sample at a glancing angle θ and is specularly reflected at the same angle. The intensity of the reflected beam is measured as a function of θ, which usually ranges between 0 and 2°. Based on the index of refraction for neutrons [1,2,3], the reflected intensity is well described by the Fresnel formula for reflected light waves. The optical phenomenon of total reflectivity is observed in NR as well. The critical angle up to which this phenomenon is observed depends on the Scattering Length Density (SLD) of the material, which depends only on the material composition. NR can thus be used to monitor deuterium uptake in a thin film as the SLD increases with increasing deuterium content [4].

Experiment

In this experiment, we will first measure the NR curve of an as-prepared 50 nm thick Ti film deposited on a Si substrate and covered with a 5 nm catalytic Pd layer. We will then expose the Ti film to deuterium at a pressure of 1 bar and re-measure the NR curve to determine quantitatively how much deuterium has been absorbed and how the structure of the film has changed.

Topics to be covered

1) NR technique
2) How to handle hydrogen gas safely
3) Instrument alignment
4) Sample alignment
5) Running a NR experiment
6) Data analysis software (*Parratt32*, *GenX*)
7) How to fit a NR curve

Tools

To get the most out of this experiment, each student should bring a laptop on which *Parratt32* and *GenX* have been installed. These freely available software tools can be downloaded from the internet. Installation instructions will be provided.

References

[1] T. P. Russell, Materials Science Reports **5**, 171-271 (1990).
[2] H. Fritzsche, “Neutron Reflectometry” in “Materials Characterization”, E. N. Kaufmann (Ed), Wiley&Sons (2012), Online ISBN: 9780471266969, DOI: 10.1002/0471266965
[3] H. Fritzsche, “Neutron Reflectometry” in “Neutron Scattering and Other Nuclear Techniques for Hydrogen in Materials”, H. Fritzsche, J. Huot, D. Fruchart (Eds.), Springer (2016), ebook ISBN 978-3-319-22792-3, hardcover ISBN 978-3-540-88587-0
[4] H. Fritzsche, W. P. Kalisvaart, B. Zahiri, R. Flacau, and D. Mitlin, Int. J. Hydrogen Energy **37**, 3540 (2012)

Crystallographic Texture (1 session)

Introduction

Many materials used in every-day applications, from pop cans to I-beams for construction to aircraft fuselages, are manufactured using materials that are themselves made up of many crystals. Each crystal in a *polycrystalline* material is characterized by its *orientation*, which refers to how the atomic planes are oriented with respect to a fixed set of reference axes. The distribution of crystal orientations in a polycrystal – the *crystallographic texture*, or simply *texture* – is of great importance as many crystal properties are *anisotropic* (they depend on the crystal direction), such that the properties of polycrystalline materials can be strongly affected by the texture.
Neutron, X-ray, and electron diffraction techniques are routinely used to measure texture. Since neutrons penetrate deeply into most materials, neutron diffraction is a bulk-averaging technique that easily provides high quality bulk texture measurements with very little sample preparation.

Experiment

In this experiment, pole figures will be acquired for a selection of materials and analyzed to extract orientation distribution functions as well as other texture quantification parameters.

Students will learn

1) How to set up a spectrometer for a diffraction experiment (wavelength and gauge volume selection, spectrometer alignment).
2) How to set up a texture goniometer on the spectrometer.
3) How to acquire pole figures and generate stereographic pole figures.
4) How to generate the orientation distribution function.
5) How to quantify experimental uncertainty

Topics to be covered

1) What is texture and how does it arise during processing of materials?
2) Describing texture
    - The rotation tensor
    - The Rodrigues vector
    - Euler angles and Euler space – sequential rotations about follower axes
    - The stereographic projection
3) Measuring texture
    - Pole figures (intensity maps)
4) The orientation density function
    - What is it?
    - How is it obtained from measured pole figures?
    - What can we do with it?

Tools

To get the most out of this experiment, each student should bring a laptop on which Microsoft Excel (2007-2013) and Python have been installed. A free 3rd-partyPython installer for windows, which includes all of the necessary Python components, will be provided.

In-Situ Deformation of a Zirconium Alloy (1 session)

Introduction

The deformation of metallic materials under the action of applied loads typically is heterogeneous at the crystal scale. The degree of heterogeneity depends directly on the strength of the anisotropy in the mechanical properties of the constituent crystals. Conjugate to the deformation is the stress, which is similarly heterogeneous over an aggregate of grains. Behaviors like yielding and failure are closely associated with stress levels experienced at the crystal level. Measuring the mechanical behavior at the crystal scale is thus critical to developing a quantitative understanding of it. Since neutrons penetrate deeply into most materials, neutron diffraction provides a bulk-average, allowing study of the mechanical behaviours of different crystal orientations in a polycrystalline aggregate.

Experiment

In this experiment, a zirconium alloy will be loaded and unloaded *in-situ* to investigate how different grain orientations deform and contribute to the overall macroscopic deformation of the material. A selection of diffraction peaks will be acquired at a series of programmed loads to study how the plane spacing and diffraction peak intensity change with the applied load.

Students will learn

1) How to set up a spectrometer for a diffraction experiment (wavelength and gauge volume selection, spectrometer alignment).
2) How to set up a load frame on the spectrometer and how the load frame and spectrometer control systems interact to run an *in-situ* experiment.
3) How to program an *in-situ* experiment.
4) How to analyze diffraction data:
    - lattice parameter evolution with applied load, and how the data can be used to determine material parameters such as the critical resolved shear stress for different slip and twinning systems
    - diffraction intensity evolution with applied load, and how to interpret it in terms of twinning activity
    - quantification of experimental uncertainty

Topics to be covered

1) Deformation – what is it?
    - Deformation gradient tensor
    - Graphical introduction to deformation
    - Polar decomposition of the deformation gradient tensor
    - The stretch tensor
    - Mathematical description of strain
    - Elastic vs. plastic strain
2) Stress
    - What is stress?
    - Getting stress from strain
    - Residual vs applied stresses
    - Polycrystal deformation (Voigt, Reuss, Taylor models, self-consistent models)
3) Measuring strain using diffraction-based methods
    - Crystal structure and crystal planes
    - Crystal plane spacing as an internal strain gauge
    - Mapping strain – direction and gauge volume
    - Positioning – how wall scans work
    - Residual and *in-situ* strain measurement
    - Examples of applications of diffraction-based strain measurement

Tools

To get the most out of this experiment, each student should bring a laptop on which Microsoft Excel (2007-2013) has been installed. Custom Visual Basic macros will be provided for data analysis.

Rietveld Analysis (2 sessions)

Introduction

Neutron Powder Diffraction (NPD) is used to determine crystal structures (lattice parameters and distribution of atoms) when large single crystals are unavailable (pretty much always). It can also be used to work out which phases are present in a complex sample, both under static conditions, and during heating and/or cooling under a controlled atmosphere (*in-situ* experiments),for example during welding or chemical reactions. It is also routinely used to study structural and magnetic phase transitions. As a result, the powder diffractometers at every neutron facility are work-horse instruments with the highest user turnover and publication rates.

Data analysis almost invariably involves fitting an entire diffraction pattern, or simultaneously fitting multiple neutron and/or X-ray patterns. The fitting technique, referred to as Rietveld Refinement or Rietveld Analysis (named after the person who pioneered the technique), is the focus of this module.

Experiment

Over the course of the experimental sessions at the NRU reactor, we will gather data on a variety of samples that have been chosen to exploit specific strengths of neutron powder diffraction:

- $Fe_{1+x}Ti_{2-x}O_5$ (pseudobrookite) - Extreme contrast between Fe and Ti allows us to determine how the two elements are distributed among the lattice sites.
- $YBa_2Cu_3O_{7-x}$ – The sensitivity of neutrons to oxygen allows us to determine the vacancies in the famous 1-2-3 High-T_c superconductor
- Fe_2O_3 – The magnetic moment of neutrons allows us to study the magnetic transition in this material (haematite).

X-ray diffraction data will be supplied for comparison and co-refinement.
We will be using the GSAS/EXPGUI package for the analysis as it is thoroughly debugged, freely distributed, and has a mature front end that makes data input relatively painless. In a series of guided sessions we will work through the process of setting up and conducting a Rietveld refinement within GSAS. We will look at the impact of data quality, strategies for optimum refinements, use of constraints, pit falls, and final interpretation of the results.
The primary goal of this module is to ensure that students are sufficiently comfortable analysing data using GSAS that they will be able to use it for their own projects.

Topics to be covered

1) What is a powder diffraction pattern and how is it generated using neutrons or X-rays?
2) Differences and strengths of neutrons and X-rays.
3) Rietveld analysis:
    - History
    - Introduction to GSAS/EXPGUI
    - Calibrating the spectrometer (wavelength, zero, peak profiles)
    - Fitting multicomponent samples
    - Co-refinement of neutron and x-ray diffraction data
    - Co-refinement of multi-wavelength neutron data
    - Fitting using constraints
    - Locating vacancies or atom distributions in disordered compounds
    - A basic introduction to fitting magnetic systems

Tools

Students should install GSAS/EXPGUI on their own laptops. These software packages can be downloaded from the internet (http://www.ccp14.ac.uk/solution/gsas/). Detailed installation instructions will be provided prior to the summer school.

Triple-Axis Neutron Scattering Study of the Magnetic Properties of MnF_2 (2 sessions)

Introduction

In this module, students will learn the experimental and theoretical foundations of neutron scattering and its application in the study of Quantum Materials, through measurements on MnF_2, one of the best-studied antiferromagnets in condensed-matter physics.

Since neutrons with wavelengths similar to interatomic distances are readily available, structural measurements over distances from the shortest hydrogen bonds to macromolecules are possible. Also, since the energies of the neutrons with such wavelengths match the energy scales of many condensed matter systems, it is possible to use them to probe the dynamics of the system. In addition, since the neutron has a magnetic moment, it interacts with unpaired electrons in solids. Thus, the neutron is the probe of choice for investigating magnetic materials, as it often provides crucial information about the magnetic properties of the system that cannot be obtained by other techniques.

Experiment

In this experiment, the C5 Triple Axis Spectrometer will be used to acquire neutron scattering data to characterize the elastic and inelastic magnetic properties of MnF_2. The nuts and bolts of Triple-Axis Spectroscopy (TAS), its many instrumental components, and their functionality will be discussed in detail.

Students will learn

1) How to run a TAS spectrometer:
2) Spectrometer alignment
3) Wavelength selection
4) How to perform data acquisition on a TAS
5) How to analyze and interpret the neutron data to characterize the elastic and inelastic magnetic properties of a MnF2 single crystal

Topics to be covered

1) Introduction to magnetic neutron scattering.
2) Introduction to triple axis spectroscopy (TAS). This introduction will include details of the main components of a triple-axis set-up such as monochromator, analyzer, detector, filters, and collimation.
3) Instruction for setting up and running a triple axis spectrometer for studying static nuclear and magnetic properties of materials in general and in particular single crystals.
4) Performing elastic scattering measurements on MnF_2 single crystal to study static magnetic order (determining the magnetic order transition temperature, moment direction and magnitude).
5) Introduction to inelastic measurements using a triple axis spectrometer for studying dynamic nuclear (phonons) and magnetic (spin-wave excitations) properties of materials in general and in particular single crystals.
6) Performing inelastic measurements on MnF_2 single crystal to study the dynamic nature of its magnetic order (determining spin-wave dispersions, exchange interaction parameters, anisotropy gap and their temperature dependence).

Tools

To get the most out of this experiment, each student should bring a laptop equipped with a graphing/analysis software of their choice (Microsoft Excel, MatLab, Origin…).

Students are strongly encouraged to carefully read the following journal article in preparation for this experiment.

Z. Yamani, Z. Tun, D. Ryan, *Can. J. Phys.*, **88**: 771-797 (2010).