Analysis of the Stress-Free Interplanar Spacing during Solution Heat Treatment of 319 Al Alloy Engine Blocks using In-Situ Neutron Diffraction
ID: 70676
Type: Student
Speaker: Anthony Lombardi, Ryerson University, Canada
Author(s): A. Lombardi, D. Sediako, C. Ravindran, R. MacKay
Aluminum alloy engine blocks have successfully replaced ferrous materials in order to maximize weight savings and improve vehicle fuel efficiency. However, the development of an optimal heat treatment process is required to improve engine block casting integrity and prevent potential problems such as in-service cylinder distortion. Optimization of heat treatment parameters requires an in-depth study to determine how residual stresses are relieved with time during solution heat treatment. In order to perform this analysis, however, in-situ neutron diffraction must first be carried out on stress-free samples of the same composition and processing history as the engine blocks to account for factors such as thermal expansion and changes in lattice parameter due to dissolution of secondary phases. The results from this study suggest that thermal expansion caused the largest change in d0 spacing, while prolonged exposure to solution temperatures of 470 and 500 °C did not cause significant changes in d0.

Effect of constraint on room temperature deformation of Zircaloy-2
ID: 73934
Type: Student
Speaker: Dale Campbell, Queensu University, Canada
Author(s): D. Campbell
In-situ neutron diffraction experiments under biaxial compression were conducted on a warm rolled Zircaloy-2 plate at room temperature. A biaxial stress state was achieved through the loading of one principal direction while a second principal direction was constrained. All six possible combinations of loaded and constrained principal directions, relative to the parent plate texture, were investigated with lattice strains obtained. Experimental results were compared with an elastic-plastic self consistent (EPSC) model to further interpret deformation mechanisms and texture evolution under the constrained conditions. Through the use of a genetic algorithm the Voce hardening parameters associated with the EPSC, which incorporated self and latent hardening, are optimized. Results are compared with parameters obtained from unconstrained uniaxial tests.

Effect of heat treatment on the residual strain levels in MIG welded AR200 steel joints
ID: 70761
Type: Contributed
Speaker: Lukas Bichler, University of British Columbia, Canada
Author(s): V. Neykov, D. Sediako, L. Bichler
AR200 steel is frequently used to fabricate complex structural components (e.g., for fabrication of custom-built auto bodies) due to its economical attractiveness. However, from the fabrication point of view, the AR200 steel contains elevated amounts of Manganese (Mn), Silicon (Si) and Carbon (C), leading to Carbon equivalent (Ce) of 0.42wt%, which represents a challenge specifically for the weldability of the AR200 steel. In this research, AR200 C-sections were welded using MIG technique and heat treated at 400, 500 and 600 °C. The influence of the heat treatment on the AR200’s microstructure was studied using quantitative image analysis and SEM-XEDS techniques. Further, residual strain mapping was carried out using neutron diffraction, and the influence of heat treatment on the levels of residual strain and the extent of heat affected zone was quantified.
Experimental cell for neutron reflection on Lithium Manganese Oxide to study the electrode/electrolyte interface.
ID: 72461
Type: StudentPoster
  Speaker: Brian Kitchen, University of Michigan, USA
  Author(s): B. Kitchen
Lithium-ion batteries are plagued by capacity losses as well as aging during the lifetime of the battery. A significant hurdle to advancing lithium-ion batteries lies with uncertainty in the creation, evolution, and composition of interfacial structures. The uncertainty is a result of sensitivity to destructive testing as well as layer thickness on the order of several to tens of nanometers. This work is intended to develop a wet sample cell for neutron reflection studies of the interface, along with operating as an electrochemical half cell. Neutron reflection is ideal to study the interface between cathode electrode materials and the electrolyte as neutron reflection is capable of sub-angstrom resolution in the normal direction which is far superior to any other density depth probe.

In-situ chemistry mapping of hydrogen storage materials by neutron diffraction
ID: 72481
Type: Invited
  Speaker: Edward Payzant, ORNL, USA
  Author(s): E. Payzant, R. Bowman, T. Johnson, S. Jorgensen
Neutron diffraction was used to nondestructively study the microstructures for two hydrogen storage media systems. In the first case, sodium alanate based hydrogen storage is a vehicle-scale candidate system developed by Sandia/GM. Neutron scattering was used to determine the distribution of phases in the storage media at different hydrogen loading levels, to help understand the absorption/desorption of hydrogen in large-scale systems. This study also included a 3D neutron tomographic study of the microstructure. In the second case, tin-doped lanthanum nickel alloys have been studied at JPL for space-based applications, for which the gradual degradation of the material due to segregation and disproportionation of phases is a known problem. A regenerative process developed to restore the storage properties of these alloys was studied, using in-situ neutron diffraction to relate the microstructure to the thermodynamic simulations.

In-situ Neutron Powder Diffraction on TiF3- catalyzed Magnesium for Hydrogen Storage Applications
ID: 70881
Type: Contributed
  Speaker: Roxana Flacau, Canadian Neutron Beam Centre, NRC Canada, Canada
  Author(s): R. Flacau, X. Tan, M. Danaie, H. Fritzksche, D. Mitlin
In-situ powder neutron diffraction was used to study the hydrogen/deuterium sorption behavior and microstructure of Mg powders catalyzed by 15 wt.% TiF3. Using our in-house designed and built in-situ top-loading furnace system we can simultaneously perform pressure-composition-temperature (PcT) measurements and neutron powder diffraction measurements. Diffraction patterns were collected during various stages of Mg to MgD2 phase transformation and vice versa. The presence of Ti fluoride catalyst appears to modify the thermodynamics of Mg-D in a way that the uptake of deuterium in α-Mg has become energetically more favorable and the β-MgD2 has become energetically less favorable. This modification in the system's thermodynamics may result in the observed extended deuterium solubility limit for TiF3-catalyzed Mg.
In-situ neutron reflectometry studies on Mg-based hydrogen-storage materials
ID: 70985
Type: Contributed
Speaker: Helmut Fritzsche, NRC, Canada
Author(s): H. Fritzsche, W. Kalisvaart, D. Mitlin

Our neutron reflectometry studies on Mg, Mg-Al, Mg-V-Cr, and Mg-Fe-Cr films with various catalyst layers nicely show how the unique capabilities of neutron reflectometry help to gain deeper insight into the hydrogen absorption and desorption processes. Using thin films as model systems makes it possible to discriminate between the catalytic surface effect of a cap layer on top of a Mg film and the catalytic bulk effect of alloying Mg with other elements. The scattering length density profile of the thin film system as determined from fitting of the reflectivity curves allows to reveal hydrogen/deuterium gradients within the thin films and to monitor structural changes of the film as e.g. expansion, surface/interface roughness, and alloying of the layers. Neutron reflectometry can be even used to study the reaction kinetics of deuterium absorption because the resulting increase of the critical scattering vector can be measured very fast.

Metallurgical studies with the HIPPO beam-line at LANSCE
ID: 71226
Type: Keynote
Speaker: Sven Vogel, Los Alamos National Laboratory, USA
Author(s): S. Vogel, T. Tomida, D. Brown, M. Okuniewski, M. Reiche

We present highlights of metallurgy-related research on the HIPPO beam-line, a general purpose neutron diffractometer. The TTT-diagram of 947; uranium-10wt.% molybdenum was re-assessed, following the concurrent decomposition to a-U and metastable intermetallic U-Mo phases. As a second example, we discuss the phenomenon that the transformation texture after a phase transformation cycle, such as ferrite/austenite/ferrite, reproduces nearly the initial texture (texture memory). We studied the texture in a steel sheet during the transformation cycle via HIPPO’s unique capability to measure the bulk texture in situ at elevated temperatures and could validate a model predicting the texture changes. Finally, we will present our new high temperature deformation stage. A Zr-2.5Nb sample was heated from the initial 945;\946; composition into the 946; field, then compressed by 20%, and cooled back into the room temperature composition. Our new device allows deconvolution of heating, phase transformations, deformation, and cooling effects on the texture.

Micromechanics of Elasto-Plastic Deformation of Duplex Stainless Steel Under Biaxial Loading
ID: 73795
Type: Student
Speaker: Andrew Poshadel, Cornell University, USA
Author(s): A. Poshadel, M. Gharghouri, P. Dawson

In situ neutron diffraction experiments coupled with polycrystalline finite element simulations have been used to understand the micromechanical response of duplex stainless steel (LDX-2101) under biaxial loading applied to hollow tubular specimens using a combination of internal pressurization and axial loading. Lattice strain data were acquired for five biaxial conditions ranging from uniaxial tension to balanced biaxial loading. Inflections in the lattice strain plots provide important information pertaining to the relative order in which crystals yield and relative crystal hardening rates. For the finite element simulations, traction boundary conditions corresponding to the experimental load history were specified on the surfaces of a virtual polycrystal. The model incorporates equations for crystallographic slip, elastic anisotropy, and the evolution of material state. The simulated fiber-averaged lattice strains are compared to experimental lattice strains to validate the model. The model also yields stress, plastic deformation rate, and slip system hardness at the grain level.
Neutron Diffraction in the Toolbox for Materials Science and Technology
ID: 70568
Type: Keynote
Speaker: John Root, National Research Council of Canada, Canada
Author(s): J. Root

Through microscopy, X-rays and other optical technologies, materials researchers can acquire an impressive amount of information at the micro- and nanometre scales. Neutron diffraction is not suitable for imaging small details. However, it is relatively straightforward with neutron diffraction to determine truly representative averages of the bulk material, such as the volume fractions of phases, preferred orientations of crystallites, and spatial distributions of internal stresses. When researchers need complex environments to place specimens in conditions of relevance, for example combining deformation and temperature, neutron diffraction may be the most powerful tool to obtain insights about the microstructural response of materials to those conditions, as they are applied. This presentation will illustrate how neutron diffraction complements the job that can be done with surface-imaging methods, and why neutron diffraction is such an effective addition to the toolbox for materials science and technology.

Neutron diffraction investigation of phase compositions in as-received and modified Zr-2.5Nb pressure tube materials
ID: 70762
Type: Contributed
Speaker: Amy I Fluke, Atomic Energy of Canada Limited, Canada
Author(s): R. Fong, A. Fluke, R. Flacau

Zr-2.5Nb alloy is used for the pressure tubes in CANDU reactor fuel channels. Three phases (α, β, and γ) of zirconium are known to exist in Zr-2.5Nb alloy pressure tube materials. An as received pressure tube is composed mainly of α-phase and approximately 8% β-phase material. In the last step of production, the pressure tube is given a steam-autoclave treatment (i.e., thermal ageing at 400°C for 24 h). This treatment results in decomposing the β-phase into an Nb-enriched β-phase and a metastable γ-phase material. In this present experiment, as received pressure tube materials were modified by high-temperature annealing and thermal ageing treatments to enhance the performance of the material (e.g., fracture toughness, creep, etc.). This paper presents a neutron diffraction investigation into the phase compositions in the modified materials at room temperature using the C-2 high-resolution neutron powder diffractometer at the Canadian Neutron Beam Centre.

Neutron diffraction studies of residual stresses around gouges and gouged dents in pipelines
ID: 74082
Type: Contributed
Speaker: Lynann Clapham, Queen’s University, Canada
Author(s): L. Clapham, T. Gnaeupel-Herold, M. Zarea

Neutron diffraction measurements were conducted at the NIST reactor on three gouged dents in X52 pipeline sections. Gouges contained in pipeline sections were termed BEA161 (primarily a gouge with little denting), and BEA178 (mild gouging, very large dent). Measurements were also conducted on a coupon sample – P22. For moderate gouges (BEA161, P22) the residual stress field was highly localized around the immediate gouge vicinity (except where there was some denting present). The through wall stresses were neutral or moderate hoop and axial stresses (50-100MPa) at the outer wall (i.e. at the gouge itself) gradually becoming highly compressive (up to -600MPa) at the inner wall surface. The other sample (BEA178) exhibited a very mild gouge with significant denting. In these gouges the residual stress distribution was very complex and was not localized to the gouge vicinity. Further studies are needed to quantify the behavior in these large, complex damage situations.
Neutron Diffraction Study and EVPSC modeling of the Deformation Behavior of a Solid-Solution-Strengthened Mg-Al Binary Alloy Subjected to Uniaxial Loading  
ID: 73775  
Type: Contributed  
Speaker: Michael Gharghouri, National Research Council, Canada  
Author(s): S. Lee, M. Gharghouri, J. Luo, H. Wang, P. Wu, W. Poole, W. Wu, K. An  
In-situ neutron diffraction has been used to measure lattice strains for different grain orientations as a function of applied load for a solid-solution-strengthened Mg-9wt.%Al binary alloy subjected to uniaxial tension. The neutron diffraction data have been used to optimize the adjustable slip and twinning system parameters in an elastic-viscoplastic self-consistent model of polycrystal plasticity. The simulations reproduce the trends in the neutron diffraction lattice strain data and the macroscopic stress-strain curve very well, showing how the activities of the various slip and twinning mechanisms vary during the course of the test. A clear distinction is made between hard grain orientations, in which the slip and twinning mechanisms are difficult to activate, and soft grain orientations, which are favorably oriented for slip and twinning. The hysteresis loops observed during unloading and reloading are shown to be due mainly to basal slip in favorably oriented grains.

Neutron Imaging of Hydrogen in Steels  
ID: 73150  
Type: Contributed  
Speaker: Axel Griesche, Federal Institute for Materials Research and Testing (BAM), Germany  
Author(s): A. Griesche, E. Dabah, I. Manke, N. Kardjilov, T. Kannengiesser  
Transmission images with neutrons show a high contrast between hydrogen and iron when using state-of-the-art scintillator materials and digital cameras. Monitoring the 2D hydrogen distribution evolution with good temporal resolution is possible if a high flux of cold neutrons is provided. The mass transport of hydrogen can be quantified by converting the intensity distribution of the images in a hydrogen concentration distribution by using standards with known composition and similar thickness. Then neutron radiography can be used quantitatively as a non-destructive measurement technique to determine diffusion coefficients, e.g. for hydrogen in steel. We report about measurements at the Research Reactor BER II in Berlin. The capabilities and limitations, as well as perspectives of this method will be discussed and illustrated with selected examples.

Neutron Powder Diffraction of the Mg6Pd Alloy for Hydrogen Storage  
ID: 71445  
Type: Student  
Speaker: Julien Lang, Hydrogen Research Institute, Canada  
Author(s): J. Lang, J. Huot  
Magnesium and magnesium-based alloys are of high interest in hydrogen storage research as they have a high hydrogen absorption capacity and are abundant. Their reaction kinetics, however, are slow, requiring high operating pressures and temperatures. To improve magnesium’s hydrogen absorption properties, palladium was ball milled with magnesium to create the Mg6Pd alloy. Known to reversibly absorb hydrogen in three disproportional reactions;Mg6Pd + 2.35H2 + energy <=> Mg3.65Pd + 2.35MgH2             Mg3.65Pd + 2.35MgH2 + 1.15H2 + energy <=> 0.5Mg5Pd2 + 3.5MgH2 0.5Mg5Pd2 + 3.5MgH2 + 1.5H2 + energy <=> MgPd + 5MgH2, we hydrided the sample at the end of each reaction. Neutron powder diffraction was made on these partially hydrided samples to study the evolution of the microstructure with hydrogenation. The effect of iron as a catalyst for Mg6Pd’s activation will also be presented, including a neutron powder diffraction study of the sample.
Quantitative analysis of elastic and plastic response of advanced Mg alloys during creep at 170 ºC using neutron diffraction techniques  
ID: 70778  
Type: Contributed  
Speaker: Lukas Bichler, University of British Columbia, Canada  
Author(s): L. Bichler, D. Sediako  
In this research, the creep resistance of wrought magnesium alloys (AE42, AJ32, EZ33 and ZE10) developed for elevated temperature automotive applications was investigated. Neutron diffraction was used to measure the creep strain (elastic) along with an extensometer measurement of the total (plastic) deformation in the axial and transverse directions at 170°C. Optical and SEM-XEDS analysis was subsequently used to analyze microstructural constituents of each alloy. The alloy’s creep resistance was mainly related to grain boundary sliding at elevated temperatures.

Residual Stress Tensor in a Compact Tension Weld Specimen  
ID: 73789  
Type: Contributed  
Speaker: Michael Gharghouri, National Research Council, Canada  
Author(s): Y. Traore, S. Paddea, M. Gharghouri, P. Bouchard  
Neutron diffraction has been used to determine the full residual stress tensor along the expected crack path in a creep resistant austenitic stainless steel (Esshete 1250) compact tension weld specimen. A destructive slitting method was used on the same specimen to determine the stress intensity factor profile associated with the residual stress field as a function of crack length. Deformations of the cut surfaces were also measured to generate a contour map of the residual stresses in the intact specimen. The distributions of transverse residual stress measured by the three techniques are in close agreement. The neutron diffraction measurements show that exceptionally high residual stress triaxiality is present at crack depths likely to be used for creep crack growth testing, and confirm that lattice strain measurements in just the three specimen orthogonal directions would have been sufficient to provide a reasonably accurate characterization of the stress state in the specimen.

Residual Stresses in Semi-Permanent Mold Engine Head Castings – A Neutron Diffraction Study  
ID: 70750  
Type: Contributed  
Speaker: Dimitry Sediako, National Research Council Canada, Canada  
Author(s): M. Walker, D. Hess, D. Sediako  
Cylinder head castings for internal combustion engines require high tensile and fatigue strength to be able to handle the loads imposed on them during the engine combustion cycle. The demands for lighter, more efficient, vehicles will only increase the material demands in these castings. In the 300 series heat treatable cast aluminum alloys, precipitation hardening is accomplished by first solutionizing the casting at ~500oC, followed by rapid cooling (quenching). The quenching process introduces significant residual stresses in the component, therefore reducing the service load that the material can sustain. A neutron diffraction analysis was performed on two semi-permanent mould castings of a cylinder head. One casting was quenched using water and the other was air quenched. The neutron diffraction experiment compared the strains/stress profiles along the web area between the intake and exhaust valve ports indicating the main differences in strain/stress distribution for the two alternative quenching practices.
Solidification Analysis of an Al-5wt% Cu Alloy Using In-Situ Neutron Diffraction
ID: 70936
Type: Contributed
Speaker: Francesco D’Elia, Ryerson University, Canada
Author(s): F. D’Elia, C. Ravindran, D. Sediako, R. Donaberger
An understanding of the solidification mechanism of Al-Cu alloys is vital to enhancing the use of these alloys in industry. In this study, in-situ neutron diffraction was used to characterize the solidification of an Al-5wt%Cu alloy. Neutron diffraction patterns were collected in a stepwise mode during solidification between 660 °C and 450 °C. The nucleation of the Al phase and Al2Cu phase was successfully detected. The solid and liquid volume fractions of these phases were determined from the change of intensity of neutron diffraction peaks over the solidification interval. Further, the results of neutron diffraction also showed good agreement with calculations obtained from FactSage software. This study aims to better understand the solidification mechanism of Al-Cu alloys, with a view to eliminating the formation of defects during solidification and thereby, enhancing the use of Al-Cu alloys in industrial applications.

Solidification of Mg-9Al alloy using in-situ neutron diffraction
ID: 70766
Type: Student
Speaker: Abdallah Elsayed, Ryerson University, Canada
Author(s): A. Elsayed, D. Sediako, C. Ravindran
In-situ neutron diffraction has been used to examine the solidification behavior of an Mg-9Al alloy. A sample of Mg-9Al was heated to 620 °C and slowly cooled to 300 °C in a stepwise fashion while simultaneously collecting thermal and neutron scattering intensities. Optical microscopy of the solidified samples revealed the presence of primary Mg and eutectic Mg17Al12 phases both of which were detected by a variation in neutron scattering intensities. The neutron diffraction data accurately described the fraction solid growth of crystallographic planes over the entire solidification regime. The research demonstrates possibilities in using neutron diffraction to examine the solidification behavior of Mg alloys for further understanding of nucleation, eutectic formation and solid phase evolution.

The CO2 adsorption dynamics in flexible zeolitic imidazolate framework 7
ID: 71144
Type: Student
Speaker: Pu Zhao, University of Cambridge, United Kingdom
Author(s): P. Zhao, S. Redfern, G. Lampronti, G. Lloyd, E. Suard
ZIF-7 has recently been shown to undergo unspecified structural phase transitions upon loading and unloading of CO2 guest molecules into the porous framework. These transitions assumed to be due to a “breathing effect” of the benzimidazolate framework, and refer to the process as “guest-induced gate-opening”. Previous results demonstrate the structural transformation of ZIF-7 on CO2 adsorption and desorption, however, neither determination was provided of the structure in the host material nor detail was given on the relationship between CO2 guest molecules and the crystalline host structure. In our recent work, with neutron scattering technique, structure variations in deuterated ZIF-7 upon CO2 loading and the binding relation between CO2 guest and ZIF-7 host were investigated under ambient external environment. Combined with computational modeling, the dynamic carbon dioxide adsorption process of the potential rationalised ZIF-7 framework was established for further study of ZIFs’ efficient CO2 capture, storage and subsequent release.
Use of neutron diffraction for the development of BCC alloys for hydrogen storage
ID: 72239
Type: Contributed
Speaker: Jacques Huot, UQTR, Canada
Author(s): J. Huot, P. Jain, M. Tousignant

Ti-based BCC solid solutions are promising material for hydrogen storage applications which need high volumetric capacity and room temperature operation. Up to now the main systems considered have been Ti-V-Mn and Ti-V-Cr. By atomic substitution the working temperature and hydrogen capacities could be tuned up to a certain level. Using only X-ray diffraction for structural identification does not provide information about hydrogen localization. Therefore, neutron diffraction is essential for complete determination of this class of hydrides. In this communication we will discuss the use of neutron diffraction for the development and understanding of hydrogenation mechanism in BCC alloys. After a brief review of the work performed on Ti-V-Cr and Ti-V-Mn alloys we will show our recent results on a new class of alloys Ti-Nb-Cr and Ti-Nb-Mn.

Using Neutrons to Study Radioactive Materials
ID: 73262
Type: Invited
Speaker: Ron Rogge, National Research Council, Canada
Author(s): R. Rogge, M. Gharghouri, I. Swainson, J. Root, D. Sears

Located at the Chalk River Laboratories of AECL, the Canadian Neutron Beam Centre (CNBC) has access to the unique infrastructure and specialized staff of the Nuclear Laboratory. Shielded cells enable neutron diffraction studies on highly radioactive samples. Two examples will be presented. Reactor Fuel: Al-U<sub>3</sub>Si dispersion fuel has been used for almost two decades, with stable behaviour and no defects. However, little is known about the phase composition and crystal structure of Al-U<sub>3</sub>Si fuel after irradiation to high burnup. Diffraction measurements reveal that only the Al crystalline phase is evident, indicating that the fuel particles and reaction products have become amorphous. Stainless Welds: Weld bead-on-plate specimens made of 304 and 316L stainless steel were neutron-irradiated to high fluence with activity of order 10<sup>12</sup> Bq. A series of samples were examined by neutron diffraction. The values of residual stress transverse and longitudinal to the weld bead decreased with increasing neutron dose.