P.184 Effect of ultrasound on the structure of amorphous zirconia gels

L Almasy¹, N N Gubanova², A Ye Baranchikov³, G P Kopitsa², B Angelov⁴, A D Yaprinytsev⁵, L Rosta¹ and V K Ivanov⁵

¹Wigner RCP, Hungary, ²Petersburg Nuclear Physics Institute, Russia, ³Kurnakov Institute of General and Inorganic Chemistry, Russia, ⁴Institute of Macromolecular Chemistry, Czech Republic, ⁵Moscow State University, Russia

In the present work, we have studied for the first time the combined effect of both sonication and precipitation pH on the structure of amorphous zirconia gels precipitated from zirconium(IV) propoxide. The techniques of small-angle neutron and X-ray scattering (SANS and SAXS) and low temperature nitrogen adsorption provided the integral data on the changes in the microstructure and mesostructure of these materials caused by ultrasonic treatment. ZrO₂·xH₂O synthesized under ultrasonic treatment was found to possess very structured surface characterized by the surface fractal dimension 2.9 – 3.0 compared to 2.3 – 2.5 for the non US-assisted synthesis, and possess higher specific surface area, while the sizes of the primary particles remain unchanged.

P.185 High fidelity physics based constitutive model for metals and alloys at high temperature and pressure

D Bansal, A Aref and G Dargush

SUNY at Buffalo, USA

Equations of state (EoS) have been re-derived to integrate the contribution of each phonon mode with temperature and pressure (T&P) as opposed to average contribution from the entire reciprocal space, in calculating the constitutive model properties of metals and alloys at high T&P. The derivation relies on basic principles of thermodynamics with insights from quantum mechanics to characterize the motion of the atoms inside the lattice. Further, relationship between EoS and inelastic neutron scattering data has been established such that constitutive properties at moderate to high T&P can be directly calculated from either phonon density of states (DoS) or phonon dispersion relation.

Early research has shown that change in Phonon DoS with T&P has different degree of dependence on longitudinal and two transverse modes, and by averaging the normal modes, we lose the valuable information of constitutive properties along different directions, which restricts us to precisely model the behavior of materials where anisotropy is high. In this work, we have verified the constitutive model at high T&P for Al, Ni and Fe by using experimental inelastic neutron scattering data and derivation is promising. Derivation is free of any empirical parameters and solely has physics principles.

Existing constitutive material models at high T&P are largely empirical and little is known about accuracy and robustness beyond their calibrated range. In many critical applications (i.e. nuclear vessels, submarines) the need for having reliable and accurate constitutive model that represent extreme load conditions of high T&P is essential and of great national interest, and the limited predictive capabilities of existing models pose a high risk.
P.186 Neutron diffraction in a validation of in-situ vibratory stress relief applied to welded steel plates

V Davydov¹, A Wahlen², A Matt² and H Van Svygenhoven¹

¹Paul Scherrer Institute, Switzerland, ²University of Applied Sciences Northwestern, Switzerland

Residual stresses in metal work pieces induced either by heat treatment, machining or welding are often a cause of a premature failure of engineering components and thus have to be kept under control. There are several methods currently used to reduce the development of uncontrolled residual stresses. Typically, heat treatments at higher temperatures allow plastic or visco-plastic flow of elastic residual stresses, which reduce the mean residual stress level but result in distortions of welded or machined components. To decrease or to remove residual stress without causing a distortion of an engineering component, a vibratory stress relief (VSR) technique is applied during or after welding. To validate experimentally the relaxation of residual stress, four welded samples of austenitic steel with dissimilar VSR treatment were examined by neutron diffraction at POLDI materials science time-of-flight diffractometer, at SINQ neutron spallation source at PSI. The obtained results exhibited no influence of VSR treatment on the reduction of residual stress in the examined specimens.

P.187 Anisotropic properties of the newly developed Mg-RE alloys by neutron diffraction

W Gan and Y Huang

Helmholtz-Zentrum Geesthacht, Germany

Tensile and compression deformation were first performed on the newly developed high Mg-Re alloys, both of which demonstrated high ductility. Bulk texture evolution of the deformed alloys were then investigated at STRESS-SPEC with thermal neutron diffraction. Results showed that existed a basal plane type preferred orientation in the tensile and compression deformed specimens. However, the texture intensity was lower as that of the commercial deformed Mg alloys. Maximum intensity was gradually increased with the tensile and compression strains; and much stronger with the compressed strains.

Weak texture intensity showed that the new Mg-RE alloys demonstrated relatively low anisotropic property, which means a high potential application. Detailed discussions will be given in current contribution.

P.188 Atomic structure and elastic properties of amorphous steels: Neutrons + quantum chemistry

V Kazimirov

Joint Institute for Nuclear Research, Russia

The local atomic structure and elastic properties of multicomponent Fe-based metallic glasses have been investigated by neutron diffraction and ab initio molecular dynamics. A comparison of neutron radial distribution functions allowed the difference in local atomic organization for chemically different glasses to be explained in terms of the atomic size effect. The conclusions drawn on the basis of “empirical” consideration were confirmed by the results of first-principles simulations. The structural models obtained in the course of ab initio molecular-dynamics simulations were used for the modeling the elastic properties. The results of the simulations revealed a close agreement with the experiment. This allowed us to clarify a role of particular chemical elements in improving the ductility of particular compositions.
P.189 Investigating the sintering stress of heavy Element materials by means of SANS

J Lechelle, M-H Mathon², G Cecilia³, L Aurore³ and E Belval-Haltier²

¹CEA/DEN/Cad/DEC/SPUA/LMPC, France, ²CEA/DSM/IRAMIS/LLB, France, ³CEA/DEN/DEC/SPUA, France

Nuclear oxide fuel is fabricated by means of a powder metallurgy route with a sintering stage during which porosity is almost eliminated whereas the material reaches its target density. A final density of ca. 95% of the theoretical density aims to keep free volume to trap gaseous fission products avoiding their localisation between fuel pellets and the cladding where they could act as a thermal barrier. This porosity is also that beyond which a further evolution could occur in reactor (porosity evolution). Sinterability of fuel pellets is of major importance both for fuel fabrication and for prediction of fuel further potential shrinkage during the beginning of its life in reactor. It can be quantified by means of the sintering stress that would have to be applied on a pellet to prevent it from shrinking. This quantity can be expressed on a theoretical basis as a function of porosity mean curvature and porosity total surface per volume unit of material. In appropriate conditions, the nanoporosity characterization can be obtained in the case of heavy elements, impenetrable to X-rays, by means of small angle neutron scattering. Independently of pore size the total solid/gas interface can in any case be measured by SANS. Bigger pores, on a micrometer scale, can be obtained by SEM, and isolated bigger ones by optical microscopy. This approach has been used in order to compare samples of fuels with and without Cr₂O₃ as a doping agent, in the frame of GEN3 fuel studies. With Cr₂O₃ addition since 1550°C the surface area of the solid/gas interface per volume unit was that obtained without doping agent at 1750°C and half of that at 1550°C without doping agent.

P.190 Quantitative analysis of martensite phase in stainless steel by using neutron diffraction

A Maneewong¹, B S Seong², E Shin³, Y S Han², W C Woo² and K H Lee²

¹KAERI/UST, Korea, ²KAERI, Korea

Neutron diffraction technique is very powerful tool in understanding the information of multi-phases in higher advanced steels used for industrial applications. Neutrons make it possible to measure non-destructively in bulk sample with various sample environments such as furnace, loading devices, pressure cells and magnetic fields. In some cases, X-ray, TEM and APT are complementarily used to get the local information in the sample. In this presentation, the quantitative analysis of and the partitioning of the martensite phase induced by thermal and mechanical process by neutron scattering techniques performed at HANARO will be introduced.

P.191 Structural analysis of steel rust using compact small-angle neutron scattering instrument

Y Oba¹, T Wakabayashi², T Nakayama³ and M Sugiyama¹

¹Kyoto University Research Reactor Institute, Japan, ²Kobelco Research Institute, Japan, ³Kobe Steel, USA

Nanostructure of steel rust, which is composed of iron oxyhydroxide and oxide particles, is important because it is strongly related with the corrosion resistance in weathering steels. To develop high performance steels, the detail analysis of the nanostructure in the steel rust is required. Small-angle neutron scattering (SANS) is one of the best techniques to characterize those nanostructures. However, poor machine time prevents the widespread use of the SANS. To overcome this problem, effective use of compact neutron source is needed. The SANS instrument KUR-SANS installed at Kyoto University Research Reactor (thermal power: 1-5 MW) is a typical compact SANS instrument. Though the neutron flux is lower than that in general neutron facilities, longer measurement time provides enough data to analyze the nanostructures. In this study, the nanostructure of steel rust was investigated by SANS.
As a model for the real steel rust on the weathering steels, poorly crystallized Fe-Ti mixed oxide particles synthesized by hydrolysis and freezing method was measured.[1] The SANS measurements were performed using KUR-SANS.

In the SANS profiles, the scatterings of individual Fe-Ti mixed oxide particles and agglomerates of the particles are observed. With increasing the content of Ti, the size of the particles decreases. We also performed the SANS measurements in wet conditions because water adsorbed on the steel rust plays a key role in the corrosion resistance. As a result, we clearly demonstrated the importance of the compact neutron source.


P.192 Evolution of the arrested austenite in metamagnetic shape memory alloys by powder neutron diffraction

J I Pérez-Landaínó, V Recarte, V Sánchez-Alarcos, C Gómez-Polo, E Cesiari and J A Rodríguez-Velamazán

1Universidad Pública de Navarra, Spain, 2Universitat de les Illes Balears, Spain, 3Instituto de Ciencia de Materiales de Aragón, Spain / Institut Laue-Langevin, France

Metamagnetic Shape Memory Alloys transform from a ferromagnetic austenite into a weak magnetic martensitic phase on cooling below a characteristic transformation temperature ($T_m$). The transformation temperature decreases with increasing magnetic field and the direct Martensitic Transformation can be inhibited (arrested) by the application of the field. In this work, the evolution during heating of the arrested austenite in Ni-Mn-In-Co metamagnetic shape memory alloy will be analyzed by in situ neutron diffraction. The retained austenite at low temperatures under a magnetic field evolves to martensite as soon as the field is removed or reduced. The reduction in the applied field, apart from an instantaneous phase change, leads to a time dependent evolution of the transformed fraction.

P.193 Residual stresses in continuously cast steel slabs

M Schöbel and G Requena

1TU München, Germany, 2TU Wien, Austria

Steel slabs produced by continuous casting allow fast processing. The molten metal is poured into a channel through a nozzle and bent by rolling while solidification takes place. The partly solidified slab suffers a combination of internal stresses by thermal gradients, phase transformation and externally applied deformation. A complex superimposed stress situation is responsible for crack formation during cooling and further manufacturing. The current simulation models take into account thermal stress calculations, which deliver the starting stress condition to predict crack formation and failure.

Non-destructive neutron diffraction was applied in-situ on a large continuously cast steel slab, with an initial thermal gradient to simulate solid state cooling. Neutrons were chosen as probe particles due to their high penetration depth in metals to enable diffraction on coarse grained cast structures. In-situ strain scanning was performed to determine the temperature dependent strains as a function of depth in the slab during cooling. Additional stress mapping at room temperature revealed the 3D macro stress state after casting.

The stresses originating from the austenite (f.c.c.) to ferrite (b.c.c.) phase transformation are dominant, during and after cooling. The volume increase of ~1% by phase transformation produces high macro stress gradients which superimpose to the thermal stresses. The phase sensitive strain analysis of the in-situ experiment during cooling, was correlated to the final 3D stress state, measured in the as cast condition. Compared to the thermal model, an overall inverse stress situation was observed, as a consequence of transformation gradients during production.
P.194 Misfit in Inconel type superalloy

P. Strunz, M Petrenec, V Davydov, P Beran and J Polák

1Nuclear Physics Institute, Czech Republic 2Institute of Physics of Materials ASCR Brno, Czech Republic, 3Materials Science and Simulation, ASQ/NUM, PSI, Switzerland, 4Nuclear Physics Institute ASCR Řež, Czech Republic

Nickel based superalloy Inconel is a natural composite consisting of $\gamma'$ precipitates (L12) with an ordered structure coherently embedded in a $\gamma$ solid solution (fcc). An important parameter for the characterization of microstructural changes is misfit - the relative difference between lattice parameters of $\gamma$ matrix and $\gamma'$ precipitates. For Inconel alloys, the misfit is generally less than 0.5%.

Neutron diffraction offers a unique tool for investigation of misfit in the bulk of superalloys containing large grains (Inconel: up to several millimeters). X-ray diffraction can be hardly used due to insufficient number of grains in the gauge volume.

Misfit in IN738LC alloy was examined in POLDI TOF diffractometer both ex-situ and in-situ at elevated temperatures using ILL-type furnace. A careful out-of-furnace measurement yielded the lattice parameters of both $\gamma$ and $\gamma'$ phases at room temperature ($a_\gamma = 3.58611(10)$ Å and $a_{\gamma'} = 3.58857(17)$ Å, misfit $6.9(6) \times 10^{-4}$).

The in-situ measurement at elevated temperatures allowed to assess the evolution of lattice parameters in $\gamma$ and $\gamma'$ up to 1000°C. Using these data, the temperature dependence of the misfit was calculated. The misfit parameter decreases with increasing temperature until it reaches zero value at temperature around 800°C. Above 800°C, it becomes negative.

P.195 Investigation of residual stress in straightened steel pipes and plates

K Theis-Bröhl, S Raatz, P Staron, M Hofmann, M Kaack, C Genzel and K Theis-Bröhl

1University of Applied Sciences Bremerhaven, Germany, 2Helmholtz-Zentrum Geesthacht, Germany, 3Forschung-Neutronenquelle Heinz Maier-Leibnitz, Germany, 4Salzgitter Mannesmann Forschung GmbH, Germany, 5Helmholtz-Zentrum Berlin, Germany

In our study we compare several non-destructive evaluation methods to determine the residual stress in steel components due to a demand from the industry for reliable on-site testing. The chosen samples are low alloy steel pipes and plates. The pipes contain residual stress due to different levels of a straightening process which leaves a visible helix on the surface. The plates were rolled and heat treaderd. We used neutron diffraction at STRESS-SPEC at the FRM II (Munich) for measuring absolute values of residual stress in the bulk of our samples.

The pipe samples have a wall thickness of 4 mm. The measurements show the highest residual stresses approximately 1 mm under the surface. The residual stresses correlate with the levels of straightening and change their values up to 400 MPa through the wall thickness. We compared the results with several non-destructive evaluation methods like synchrotron diffraction, magnetic Barkhausen noise, harmonic analysis and ultrasonic testing. We could show that each of these indirect measuring methods has limitations (for example different penetration depths), but can be used to estimate the residual stress in an industrial environment with on-site testing after a calibration with an absolute method.

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