T12 - Small-Angle Scattering

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EARLY STAGES OF NUCLEATION AND GROWTH OF GUINIER-PRESTON ZONES IN Al-Zn-Mg ALLOYS

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Al-Zn-Mg alloys are age hardenable at room temperature (RT) due to the formation of Guinier-Preston zones. The temperature of solid solution heat treatment has a strong effect on the kinetics of the early stages of decomposition. The samples of two different alloys have been quenched from 470 or 600 ^{o}C into RT water. The small- and wide angle scattering and diffraction experiments have been started within about 60 to 120 s after quenching. The SAXS patterns are evaluated for the average size and the volume fractions of the Guinier-Preston (GP) zones. From the shifts of the peak positions of the 111 and 200 Bragg reflections the change of the Mg and Zn content of the solid solution during zone formation is evaluated. The offcentre of the diffuse halo appearing around the fundamental 111 and 200 Bragg reflections enables to conclude about the elastic strains produced by the GP zones.

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THE EFFECTIVE WAVELENGTH IN A CONVENTIONAL SANS EXPERIMENT

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In the conventional setup of a small-angle neutron scattering diffractometer the monochromatic beam is produced by a mechanical velocity selector. Usually, the wavelength of the neutron beam is calculated assuming that it is proportional to the reciproc of the selector rotation speed. Due to the finite wavelength spread of the selector transmission function and the non-flat wavelength distribution of the thermalized neutron spectrum, this relation is not precise. In the present work, we carried out calibration measurements using a silver behenate powder sample, at two selector tilt angles. The deviations form the simple reciprocity relation reached few percents, in accordance with the numerical calculations using the measured spectrum and the known selector transmission function. It was shown that the usual way of the wavelength calibration by the selector speed is not sufficienlty accurate; it can affect considerably the precision of the size determination in SANS measurements.

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SANS INVESTIGATION OF PRECIPITATE MICROSTRUCTURE IN NICKEL-BASE SUPERALLOY WASPALOY

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The structure investigation of nickel base superalloys is an important task of materials research. The nickel base superalloys are multicomponent alloy systems, thermodynamically not stable, which are used at severe conditions. The structure stability and creep resistance of superalloys is dependent on the ‘-precipitate morphology and volume fraction of precipitates, which strengthen the -phase matrix. Various techniques (TEM, SEM, X-ray, neutron diffraction) are necessary for a full characterization of superalloy microstructure, especially its ‘ morphology.

The SANS method can contribute substantially to the morphology investigation of ‘ precipitates in nickel base

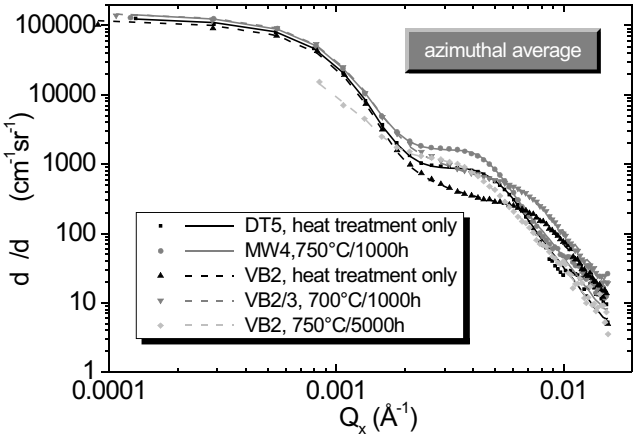

Figure 1: Measured and fitted scattering curves of modified Waspaloy (DT750).

superalloys [1]. The present SANS experiment (SANS-II facility, SINQ, Paul-Scherrer Institut Villigen, Switzerland) was aimed to provided bulk information on the ‘-precipitate characteristics in polycrystalline nickel base superalloy Waspaloy resulting from the modification of the applied heat treatment. This precipitation-strengthened superalloy has standard heat treatment as follows: 1080 °C/4h + 4K/min to 850 °C + cooling to RT + 850 °C/2h + 760 °C/16h. In order to improve its properties as well as to simplify the treatment, a modified procedure (1080 °C/4h + 800 °C/16h, alloy denoted DT750) have been tested recently.

The microstructure resulting from these two (standard and modified) treatments was investigated by SANS. At the same time, the morphological changes (precipitate size and their volume fraction) resulting from the long thermal exposure of the alloys were examined in dependence on the temperature and time of exposure.

The experiment had clearly revealed the secondary precipitation in the standard alloy after the heat treatment. These precipitates, however, are alredy not present (at least not with a significant volume fraction) after the thermal expositions. Fig. 1 displays the measured and evaluated scatteing curves for modified alloy at low *Q*-values. At larger *Q*-values, there was no indication of the secondary precipitation for this modified alloy at all.

The bulk parameters resulting from the evaluation (mean size and volume fraction) are stated in Table 1. It can be noticed that the large difference exists already between the alloys prepared at different places (Saarschmiede, Juelich). Thermal exposition has then an additional effect on the growth of ‘ precipitates.

It has also been found, that the thermal exposure influences the scattering contrast. It could be probably the effect of depleted- zones formation during the precipitate growth and (at longer exposures when the precipitate growth is already not fast) by diffusion in the matrix which promotes its homogenization.

The SANS results clearly showed that there are large differences even between samples of the modified superalloy heat-treated at different facilities (see Table 1). A strong influence of the thermal exposition on the superalloy microstructure was observed which can have an impact on the mechanical properties.

sample name	DT750, heat treatment at	thermal exposition	mean size (Å)	volume fraction
DT5	Saarschmiede	none	1020	0.21
MW4		750°C/1000h	1190	0.27
VB2	Juelich	none	620	0.21
VB2/3		700°C/1000h	750	0.35
VB2		750°C/5000h	1210	0.20

Table 1: Parameters determined by fit to the measured data.

Acknowledgement
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T12 - P214

SANS STUDY OF NANO SIZED PRECIPITATE ON COLD ROLLED STEEL SHEETS

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Ultra-low carbon steel is widely used in the manufacturing of panels and structural parts for automobiles because of good sheet formability. The high formability is achieved by developing the strong {111} fiber texture in the recrystallized steel sheet after cold rolling, which can be more easily obtained in interstitial-free steels. The titanium used for scavenging the carbon and nitrogen atoms in solution also leads the formation of precipitates sush as TiN, $Ti_4C_2S_2$ and TiC. These precipitates, especially nano-sized TiC, also affect the evolution of the texture in the steel sheets. In the previous studies, the size distribution of fine precipitates was evaluated quantitatively by using small angle neutron scattering (SANS) technique for hot rolled and recrytallized steel sheets [1,2]. However the effect of cold rolling on the fine precipitates was nearly studied.

In this study, the Ti-added low carbon steel sheets cold rolled with different reduction rates were investigated by using SANS instrument in HANARO, Korea. Scattering patterns were obtained in the Q-range of 0.008~0.25 by 0.508 nm wavelength neutron. As increasing the reduction rate, the scattering intensity in the Q-range of 0.02~0.06 decreased, while the intensity in the Q-range less than 0.02 increased. The size distribution and volume fraction of fine precipitates were analyzed by using direct model fitting method assuming the precipitates with a log-normal size distribution. As increasing the reduction rate, the volume fraction of 10~20 nm nano-sized inhomogeneities decreased, while that of nano-sized inhomogeneities more than 20 nm increased. These results are considered due to the dislocations clustering around precipitates.

Acknowledgement
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T12 - P215

CHARACTERISATION OF IRRADIATION-INDUCED PRECIPITATES IN REACTOR PRESSURE VESSEL STEELS

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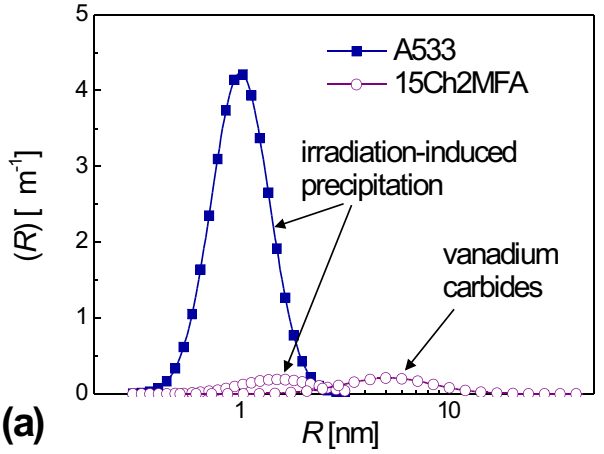
Embrittlement of steels exposed to high doses of neutron irradiation essentially reduces the lifetime of reactor pressure vessels (RPV). The degradation of mechanical properties is closely related to microstructure changes, which involve the creation of lattice defects and subsequent diffusion processes leading - besides other effects - to the creation of ultra-fine precipitates. Formation of these precipitates depends on steel composition, however the role of different solute atoms in this process is not yet fully understood.

We have studied neutron-irradiated RPV steels of different compositions by *Small-Angle Neutron Scattering (SANS)*. Although the information contents of SANS data is too low to describe the complex microstructure of steels in detail, this method has substantial advantage of yielding integral characteristics of the precipitates like volume fraction, mean size or even size distribution averaged over macroscopic sample volumes. These statistically representative parameters of microstructure can be thus directly compared to mechanical characteristics like yield strength. Moreover, magnetic interaction of neutrons with inhomogeneities in ferromagnetic iron matrix permits to separate nuclear and magnetic scattering cross-sections and obtain thus additional parameter, which helps to assess possible chemical composition of the precipitates.

Significant difference (factor of 10) has been observed between volume fractions of small precipitates created in Cu-rich steel of western type (A533B) and V-rich VVER-440 steel (15Ch2MFA) irradiated under similar conditions (Fig.1a, see [1] for details of composition and irradiation conditions). The volume fractions are evaluated under assumption of zero magnetization of the precipitates in magnetically saturated Fe matrix, which is not necessarily satisfied in Fe-rich precipitates. The lower volume fractions in the low-Cu steel 15Ch2MFA can thus signify lower magnetic contrast due to lower concentration of sol-



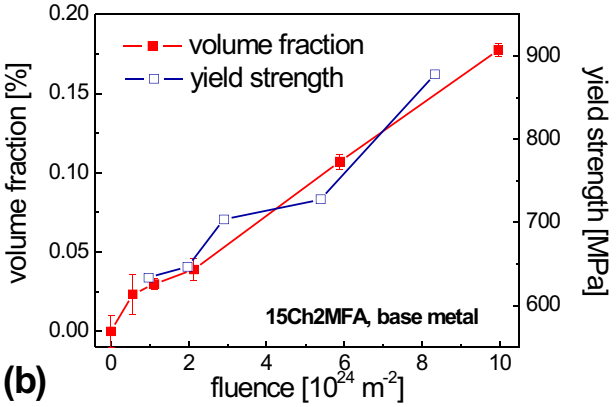


Fig1. (a) Volume-weighted distribution of precipitate radii as obtained by SANS. (b) Increase of total volume fraction of small precipitates and yield strength (from [2]) in 15Ch2MFA steel in dependence on fluence.

ute atoms in exchange of iron. This is in agreement with the models in which copper strongly accelerates nucleation of well-formed Cu-rich precipitates through radiation-enhanced diffusion [3]. On the other hand, the left peak for low-Cu steel comes probably from dilute solute atmospheres with high iron contents and consequently lower scattering contrast. Obviously, the right-hand peak at ~7.5 nm corresponds to vanadium-carbides, which are not present in the A533B steel.

As for the dependence of precipitation on fluence, we have observed continuous increase of volume fractions (Fig 1.b) in VVER-440 steels, which is in correlation with growing yield strength measured earlier [2]. The discussion of SANS results presented in this contribution is supported by comparison with the results of other techniques (transmission electron microscopy and positron annihilation [1], as well as atom probe tomography [2]).

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T12 - P216

SMALL ANGLE X-RAY SCATTERING (SAXS) ON DEFORMED PVDF – FOILS

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PVDF is an polymer witch shows a piezoelectric effect, can be used as matrix in photovoltaic cells and act as a pyroelectric transducer. For that the nanostructure of this material is very interesting and, because of the change in the electrical properties during deformation, studies on the nanostructure of the material are necessary to understand the difference between native and stressed samples. Small angle X – ray scattering is a good method to study the nanostructure of polymers and is very sensitive for changes. We applied tensile stress to thin PVDF foils with a different degree of crystallinity and studied the change in the structure while tensile stress was applied. For the change in the nanostructure, scanning three dimensional SAXS methods have been applied. For this purpose an uniaxially stretched foil was scanned over the deformed region of the specimen. Stretched foils visibly show 3 areas: undeformed regions are transparent, then some milkiness appears upon deformation and the area of plastic flow again is transparent. In the non deformed stage the samples show an isotropic orientation of the lamella, in the milky area randomly orientated crazes are formed and in the area of plastic flow a fibrous structure appears, showing an lamellar – angle of 60° with respect to the stress direction. The distance of the lamella packages also changes from 11.0 nm to 6.5 nm upon stretching, which is a sign that the deformation is largely caused by microscopic shearing.

from the interface distribution function, calculated from the second derivation of $g_1(x)$. Lamellar thickness was determined by the position of the first maximum of $g(x)$ and long period from the position of the first minimum. Both SAXS long periodicity peak as well as crystal diffraction peaks appear simultaneously at 144°C and within a short temperature interval they reach their maximum values. The results from Gaussian fitting of Lorentz corrected SAXS peak is presented in Figure 1, and the parameters, determined from the analysis of the $g_1(x)$, in Figure 2. These results were compared with DSC cooling trace.

The following conclusions could be drown from the above results:

1) Non-isothermal crystallization of the VAE copolymer from the melt leads to the appearance of SAXS and WAXS peaks at the same temperature, revealing that the

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THE NEW SANS APPARATUS OF THE STRUCTURE POWDER DIFFRACTOMETER SPODI AT FRM-II

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The *S*tructure *P*owder *DI*ffractometer SPODI, currently being tested and calibrated at the new neutron source FRM-II at Garching near Munich, is going to be equipped with a Small-Angle Neutron-Scattering (SANS) apparatus. This combination of a powder diffractometer and a SANS apparatus is feasible due to the unique features of the instrument SPODI [1,2]. A neutron guide with a length of 14.5 m coated with supermirrors ($m_h = 2$, $m_v = 3$) results in a high neutron intensity at the monochromator as well as in a low divergence. After wavelength selection with an uncertainty of ~ 0.01 at the Ge (551) waferstack monochromator, another 5 m long flight path (4 m in vacuum) can be used to collimate and focus the neutron beam with very low vertical divergence in comparison to other structure powder diffractometers.

Based on these unique geometrical specifications extensive Monte Carlo simulations of various instrument configurations for the wide-angle part [3] and the small-angle part of the instrument SPODI [4] were performed using the software package McStas [5]. With the Ge(551) monochromator currently installed wavelengths between 1.1 Å and 2.6 Å can be selected with small tilting of the crystals while the standard wavelength will be 1.5469 Å. Using pin-hole slits with a diameter between 1 and 6 mm, it was found that scattering vectors Q=4 sin( )/ between 0.0029 $Å^{-1}$ and 4 $Å^{-1}$ can be achieved, as depicted in figure 1.

Since thermal neutrons are scattered, the resulting angles are smaller than at conventional SANS machines using cold neutrons. Therefore a much smaller detector area, but also smaller pixel sizes are needed. The SANS apparatus at SPODI is going to utilize an image plate scanner by MAR Research. This scanner with an active area of 345 mm diameter is equipped with a Fuji BAS-ND image plate and has a cycle time of 108 s at 100 µm minimum pixel size and maximum image diameter. The usage of thermal neutrons also reduces the scattering of neutrons by air between the sample and the detector. Therefore, at the current status no vacuum system for the flight path is planned, but could be retrofitted, if necessary.

Since neutron image plates are very sensitive to -rays, the detector is placed in a housing made of polyethylene and lead. The frontside of the detector is covered with a 1 mm thick lead foil to absorb soft -rays. Hard -rays are not absorbed by this thin foil, but these do not contribute to a detector signal, since the image plate is not sensitive to photons with energies above 300 keV [6, 7]. The main shielding has a modular conception and can be adjusted if the -spectrum in the experimental hall make this step necessary.

The detector and the housing, weighing 350 to 550 kg (depending on the composition of the shielding), are moved by a combination of three translation stages. This arrangement permits the detector to be positioned over the sample table resulting in a minimum sample-detector distance of about 0.1 m. On the other side, a maximum distance of 3 m with the regular set-up, with an optional extension even 5 m, can be reached. Figure 2 shows an overview of the SANS set-up with the detector being in a position that corresponds to the minimum sample-detector distance.

Due to the features described above, the SANS apparatus of SPODI has several applications. Firstly, it will be

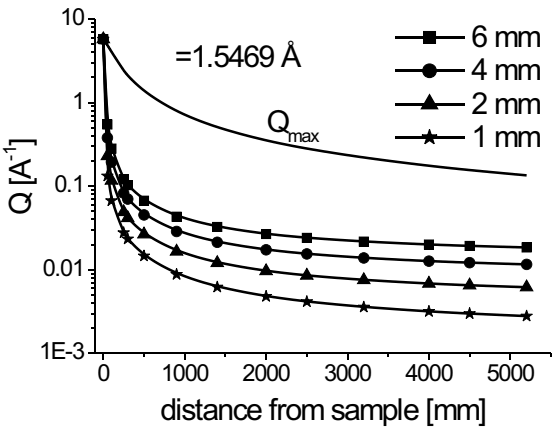

Fig. 1: Maximum and minimum scattering vectors Q as a function of sample-detector distance and diameter of pin-hole slits obtained by Monte Carlo simulations with the program McStas [5].

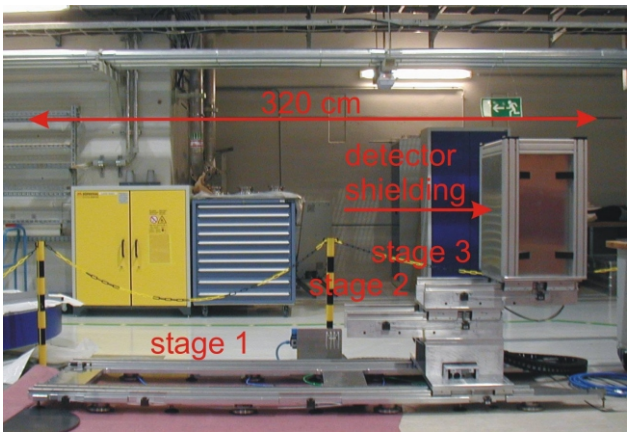

Fig. 2: Photograph of the SANS apparatus with the detector being at the minimum sample-detector distance of about 0.1 m. Note the three translation stages.

used to obtain SANS data of samples investigated by wide-angle scattering at SPODI. These SANS experiments can be performed simultaneously or sequentially to the powder diffraction yielding information about particle sizes, size distributions and morphologies of particles or pores without the necessity to change the samples´ environment or orientation. This renders possible an extensive characterisation of a sample in one step. Furthermore, at small sample-detector distances a determination of full Debye-Scherrer cones is possible. On the one hand, this data can be used for a fast pre-characterisation of a sample before a detailed measurement of the diffraction pattern with high resolution is started. On the other hand, the diffraction pattern in forward direction allows for a texture analysis of the samples. At last, the signal from the image plate scanner can be utilized to align and calibrate the instrument SPODI itself.

To our best knowledge, the instrument SPODI is the first thermal powder diffractometer being equipped with a SANS apparatus, significantly enlarging the range of achievable scattering vectors Q and resulting in unique possibilities for neutron diffraction experiments.

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T12 - P218

SAXS INVESTIGATION OF SINTERED NIOBIUM POWDER SURFACE STRUCTURAL INHOMOGENEITIES

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This report presents a method for investigating of solid porous surface inhomogeneities. The method proposed in this report was used to investigate the surface of compact niobium Samples obtained by high temperature vacuum sintering of niobium powder. The technique described in [1] was used the small-angle X-ray scattering (SAXS). The range Of scattering angles is to 2 up to 360 angular minutes. With the collimation process used and the radiation chosen, a resolution is achieved which allows to detect pores with dimensions from 0,1 to 50 nm. The SAXS measurements were treated according to special program which included the background curve substraction of the the SAXS diffractometr through the use of the 5-point cubic interpolation technique in the region of every experimental point.

The following characteristic pore dimensions were obtained by SAXS: volume $V = 10^6 nm^3$; area $S = 10^4 nm^2$; dimension l = 20nm. The pores, of which two dimensions exceed the third one (lamella), are approximated by cylinders. The corve of the SAXS data scattering invariant has some peak values. This indicates the polymodality of the surface inhomogeneities system. It should be noted that surface inhomogeneities displays the fractal nature. The approximation procedure of the cylinder shapes is known in the fractal theory as the Swartz area paradox [2].

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