Experimental report

27/06/2016

Proposal: **1-01-146** **Council:** 10/2014

Title: Spatial distribution of nanoparticles in ODS steel tubes

Research area: Materials

This proposal is a new proposal

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Samples: Fe-9Cr and Fe-14Cr with 0.3wt% Y2O3

Instrument	Requested days	Allocated days	From	To
D22	1	1	23/06/2015	24/06/2015

Abstract:

The improved properties of oxide-dispersion strengthened (ODS) ferritic/martensitic steels are directly connected with a uniform distribution of their nanoparticles. The spatial distribution of Y2O3 particles in circumferential direction of extruded cladding tubes should be investigated by small-angle neutron scattering.

Spatial distribution of nanoparticles in ODS steel tubes

1. Introduction

The application of small-angle neutron scattering (SANS) for the purpose of the characterization of oxide nanoparticles in ~1 mm thick compact samples of ODS alloys is well established [1-4]. The objectives of the present set of experiments were, first, to apply SANS to the case of thin-walled tube material in order to characterize the oxide particles, and second, to derive a non-destructive testing method for circumferential and axial uniformity of tube quality.

2. Experiments

Two pieces of 30 mm length from ODS 9%Cr tube and 14%Cr tube were investigated. The measured dimensions are given in Table 1. The thickness of the tube wall and its uniformity in circumferential direction were measured by means of the ultrasonic pulse-echo technique. Each of the received pieces was cut into two segments of 180° for the purpose of SANS measurements.

Table 1: Measured tube dimensions

Cr	d_o (mm)	d_i (mm)	t (mm)	variation of t
9	10.75	9.67	0.54	within 1% of t
14	10.77	9.67	0.55	within 1% of t

The SANS experiments were performed at the instrument D22. The major instrument parameters were:

- wavelength 0.6 nm,
- sample-detector distance 2 m, in selected cases additionally 11.2 m,
- collimation lengths of 2 m and 11.2 m,
- aperture 10 mm x 1 mm,
- no external magnetic field because of the curvature of the samples,
- six measuring positions at 45°, 90° and 135° for both segments of both tubes.

Absolute calibration was done by means of a direct beam measurement [5]. The data reduction was performed using the software package GRASP [6]. The transformation from scattering curves into particle-size distributions was performed using a Monte Carlo code [7] developed on the basis of an algorithm proposed by Martelli et al. [8].

Results

The scattered intensities as displayed by the neutron detector are shown in Figs. 1(a) and (b) for ODS Fe-9%Cr and ODS Fe-14%Cr, respectively. While ODS Fe-9%Cr exhibits an isotropic azimuthal distribution of intensities, the distribution obtained for ODS Fe-14%Cr shows a pronounced anisotropy. This difference is applicable to all tested positions of the materials. Therefore, it reflects a characteristic difference of the microstructures of the two alloys. The measured total scattering cross sections as a function of scattering vector Q are shown in Fig. 2(a) for three circumferential positions of the ODS Fe-9%Cr tube and of the ODS Fe-14Cr tube in Figs. 2(b). The fitted total scattering cross sections and the reconstructed size distributions in terms of number density of scatterers per size increment or volume fraction of scatterers per size increment are shown in Fig. 3 for both tubes. The scatter bands indicated in Figs. 3(b) and (c) only reflect the stability of the fit (upon variations of the starting conditions for the MC code), but not possible systematic measuring errors. As the scattering curves agree for each of the positions investigated, the size distributions also agree from position to position within measuring errors. The mean radius of scatterers determined on the basis of the number-density-based size distribution is (2.22 ± 0.04) nm and (1.43 ± 0.11) nm for ODS Fe-9%Cr and ODS Fe-14%Cr, respectively. As measurements in a saturation magnetic field were impossible because of the curved specimen geometry, the size distributions can only be given in arbitrary units. Moreover, information on the structure and chemical nature of the scatterers cannot be obtained. However, from the size distributions in relative units, the yttria input in the tube materials, the available experience about ODS alloys and a comparison with bar materials, it can be concluded that the scatterers must be oxides, mainly Y-Ti-bearing oxides.

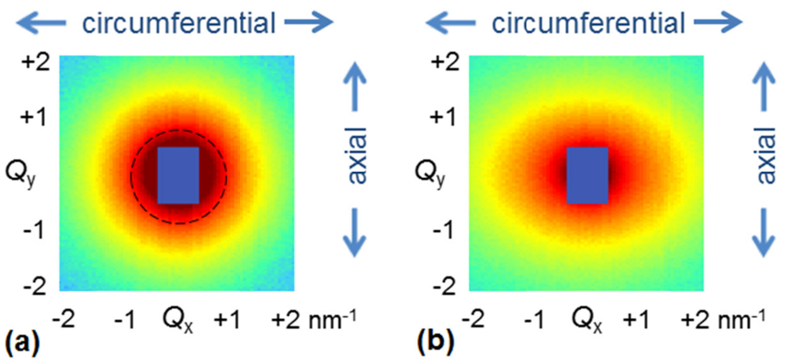


Figure 1: Measured scattering intensities in terms of detector counts, (a) for ODS Fe-9%Cr tube and (b) for ODS Fe-14%Cr tube.

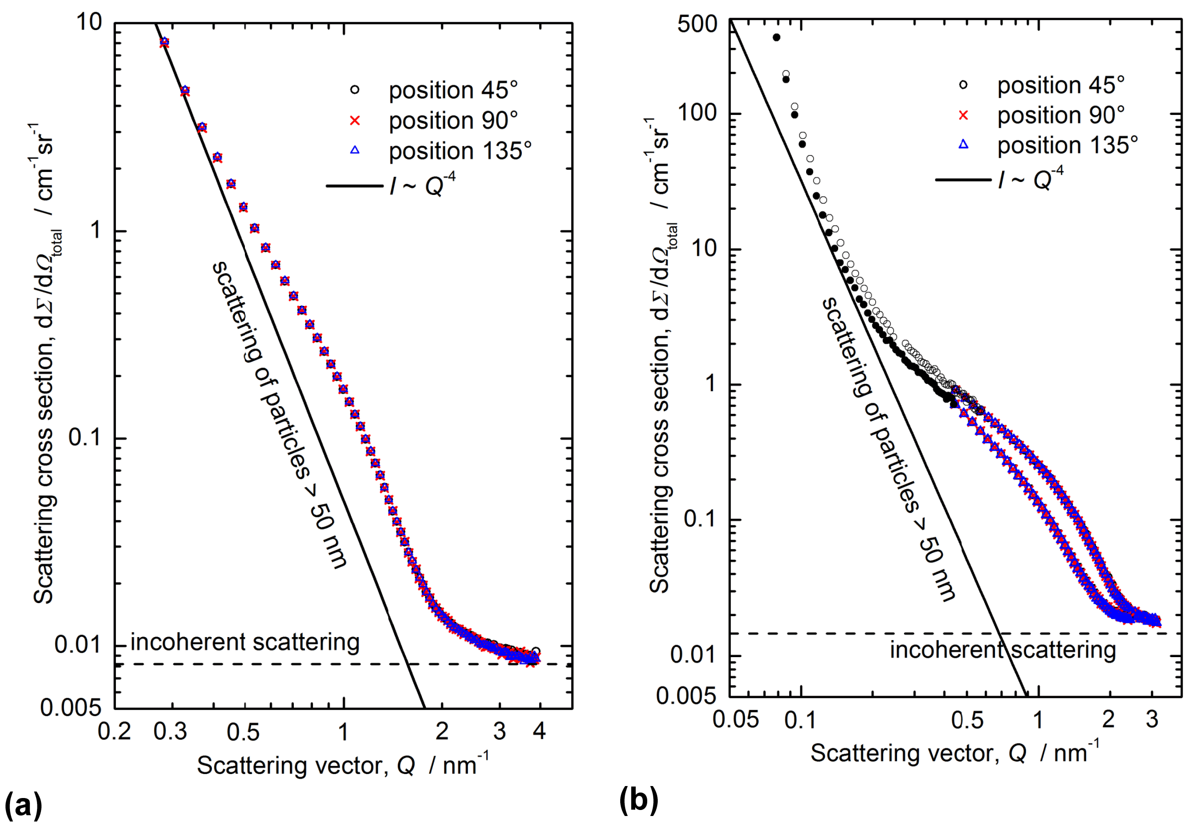
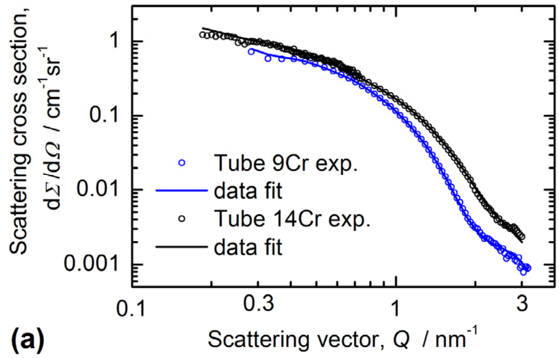


Figure 2: Measured total scattering cross sections as a function of Q obtained for different circum-ferential positions, (a) ODS Fe-9%Cr tube (intensities azimuthal averaged), and (b) ODS Fe-14%Cr tube (Upper curve: intensity in horizontal direction (circumferential), lower curve: vertical direction (axial).



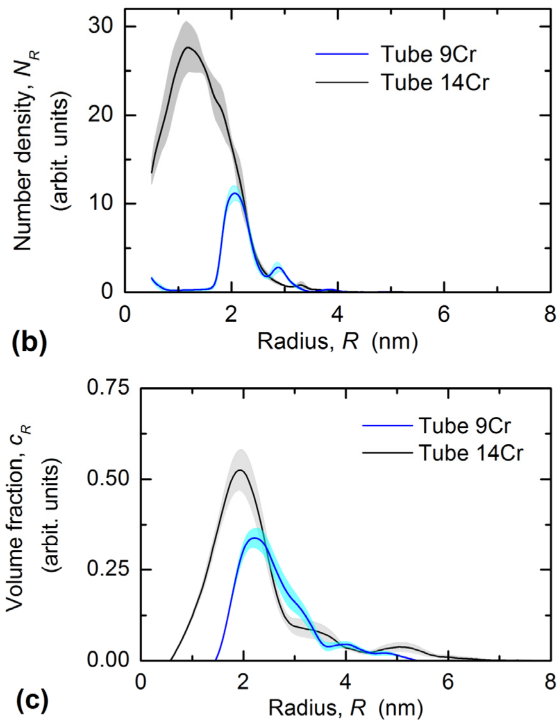


Figure 3: (a) Fitted total scattering cross sections, (b) size distributions in terms of number density of scatterers per size increment, (c)) size distributions in terms of volume fraction of scatterers per size increment.

Summary

(1) Oxide particles in the size range between 0.5 and 5 nm (radius) were observed for both tube materials. The mean size tends to be smaller for the 14Cr tube.
(2) Perfect uniformity in circumferential direction was found for both tube materials and for both tube thickness as well as nano-oxide particle size and density.
(3) SANS intensities measured for 9Cr tube and 14Cr tube exhibit perfect isotropy and pronounced anisotropy, respectively, between circumferential and axial directions. Anisotropy is most probably due to background scattering caused by grain boundaries.

In summary, the application of SANS to the characterization of oxide nanoparticles can be extended from flat samples to thin-walled tubes with the limitation that the size distributions of particles can be derived only in relative units. The method is suitable for a quality check with respect to tube uniformity in circumferential and axial directions as well as anisotropy.

References

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