Journal of Nuclear Materials 470 (2016) 139–146

Contents lists available at ScienceDirect

Journal of Nuclear Materials

journal homepage: www.elsevier.com/locate/jnucmat

Evaluation of microstructural parameters of oxide dispersion strengthened steels from X-ray diffraction profiles

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HIGHLIGHTS

- The microstructural parameters of oxide dispersion strengthened steels were obtained.
- The microstructure of irradiated and unirradiated samples was investigated.
- Oxide nanoparticles are modeled as spherical inclusions.
- We considered the influence of dislocations, inclusions and size effects.

ARTICLE INFO

Article history:
Received 30 June 2015
Received in revised form
21 November 2015
Accepted 24 November 2015
Available online 30 November 2015

ABSTRACT

The microstructural parameters of oxide dispersion strengthened (ODS) steels from measured diffraction profiles were evaluated using an approach where the complex oxide nanoparticles ($Y_2Ti_2O_7$ and $Y_4Al_2O_9$) are modeled as spherical inclusions in the steel matrix with coherent or incoherent boundaries. The proposed method enables processing of diffraction data from materials containing spherical inclusions in addition to straight dislocations, and taking into account broadening due to crystallite size and instrumental effects. The parameters of crystallite size distribution modeled by a lognormal distribution function (the parameters m and σ), the strain anisotropy parameter q, the dislocation density ρ, the dislocation arrangement parameter M, the density of oxide nanoparticles ρ_{np} and the nanoparticle radius r_0 were determined for the ODS steel samples. The results obtained are in good agreement with the results of transmission electron microscopy (TEM).

1. Introduction

One of the actual directions of investigations in materials science is the development of structural materials for future fusion reactors [1–3]. These structural materials will be exposed to more aggressive working conditions than in current reactors: higher temperatures and higher fluxes of neutron irradiation from deuterium to tritium fusion. The choice of structural material greatly influences the design of the reactor. Reduced activation ferritic/martensitic steels were chosen as one of the potential structural materials for fusion reactor applications [4], but the operating temperature for these materials is limited to about 550 °C because of poor mechanical properties. As a result, materials used for future reactors must possess improved properties compared to those used for conventional ferritic/martensitic steels. Oxide dispersion strengthened (ODS) steels were proposed as a potential group of advanced materials for future fusion reactors. These steels are characterized by uniformly dispersed oxide nanoparticles in the steel matrix. ODS steels have a number of advantages in comparison with ferritic/martensitic steels. The inclusion of dispersed oxide nanoparticles enables an increase in the operating temperature up to around 700 °C [5], which leads to enhancement of the reactor efficiency. ODS steels possess good creep and corrosion resistance, resistance to such radiation-

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http://dx.doi.org/10.1016/j.jnucmat.2015.11.047
0022-3115/

induced phenomena like void swelling and material embrittlement/hardening. The presence of nanoparticles can provide effective traps for helium atoms and point defects, thus retarding the degradation of steels under neutron irradiation. Oxide nanoparticles impede the dislocation movement during tensile deformation and this effect is governed by the nanoparticle size and the distance between nanoparticles.

One of the current topics for ODS steel research is the investigation of the influence of high radiation doses on the mechanical properties of these steels. The expected irradiation dose in future fusion reactors is about 200 dpa. In real reactors such a dose accumulates over several decades. In the energy spectrum of real reactors there are high-energy ions (hundreds of MeV) – from fission fragments (Xe, Kr) in addition to neutrons. In order to investigate the radiation damage from high doses of this type of irradiation, simulation experiments are carried out using particle accelerators. When high-energy ions are used in simulation experiments using accelerators it is impossible to achieve such high damage doses because of the low current densities (rate of dose accumulation). Therefore, ions with energy from several tens to hundreds of keV are used instead. In this case the current density is high enough to obtain high damage doses during a short period of time. Although high-energy irradiation cannot achieve the required fluences, it does enable one to study various peculiarities of defect formation, such as latent track formation, which influences the microstructure of steels. Such low dose experiments facilitate understanding of the mechanism of damage formation due to ion impact during the earlier stages of reactor operation.

Additional focus of research for ODS steels is the investigation of microstructure [6] because the physical properties are affected by the microstructure of a material (grain size, nanoparticle size, density of oxide nanoparticles, etc.). The study of microstructure modification under irradiation [7] and the thermal stability of nanoparticles at high irradiation doses [8,9] are also of interest. The investigation of ODS steel degradation by fission fragments is an important contribution to the study of stability of ODS steels. The knowledge of the microstructure response to such irradiation can help improve the properties of ODS steels.

One of the most effective techniques for the investigation of the microstructure is high-resolution transmission electron microscopy (HRTEM). Many important properties were studied with the help of HRTEM [10,11] which include the approximate sizes of crystallites, the shape and complex core/shell structure of the oxide nanoparticles and the density of oxide nanoparticles. HRTEM results contributed to the interpretation of the formation mechanisms of nanoparticles in ODS steels [12]. However, the complexity of the technique and difficult sample preparation limits its suitability to routine applications. Another powerful tool for the determination of material structure is X-ray diffractometry. X-ray diffraction methods are indirect but they are experimentally easy and nondestructive. There exists a necessity to have some computational tool for processing diffraction data from ODS steels in order to retrieve microstructural parameters.

In view of X-ray diffraction, oxide nanoparticles in ODS steels will be modeled as spherical inclusions in the steel matrix with coherent or incoherent boundaries. It is required to study X-ray diffraction by materials containing spherical inclusions which influence the peak shape in addition to small crystallite size, straight dislocations and instrumental effects.

There are numerous whole profile fitting procedures for the processing of X-ray diffraction data. One of them is the method of Whole Powder Pattern Fitting which uses phenomenological functions, e.g. the Voigt function to fit experimental diffraction profiles [13,14]. This does not describe the entire intensity range properly, as it only enables the correct fitting in the region around the maxima or the tail regions of the peaks.

The method of Convolutional Multiple Whole Profile Fitting employs microstructurally based profile functions and enables the correct fitting of the entire intensity range [15,16]. It takes into account the broadening due to dislocations, stacking faults, size and instrumental effects by using the convolutional equation. A similar method of Whole Powder Pattern Modeling was developed [17]. However, none of these methods include the presence of spherical inclusions. Therefore, it is necessary to have some computational instrument which would be able to evaluate microstructural parameters from the peak shape due to dislocations, small crystallite size and instrumental effects and additionally take into consideration the effect of spherical inclusions on the peak shape.

In this paper the following is presented. First, the description of the ODS steel samples and X-ray diffraction measurements is discussed. Second, the theoretical background of the proposed approach for the microstructural parameter evaluation is presented. The description of diffraction profile shape due to small crystallite size, dislocations, oxide nanoparticles (on the basis of a model of spherical inclusions) and instrumental effects is discussed. Then the description of the evaluation procedure is given. This is followed by the application of the developed approach for the data fitting of ODS steel samples from which the microstructural parameters are estimated.

2. Experimental

X-ray diffraction experiments were performed on a five-circle SmartLab diffractometer from Rigaku equipped with a 9 kW rotating anode using Cu Kα radiation (λ = 0.154056 nm). X-ray diffraction measurements were carried out in parallel-beam geometry to avoid influence of the sample surface quality on the peak position. A high-resolution setup with the combination of a two-crystal Ge monochromator in the 220 setting, a divergent height limiting slit equal to 5 mm, incident and receiving slits equal to 1.0 mm and a scintillation counter detector was used to achieve sufficient resolution for the measurement with ODS steel samples. The Ge monochromator allows to obtain only Kα_1 radiation without K_b and Kα_2 lines. The diffracted beam monochromator (DBM) was used to decrease fluorescence. This monochromator improves the signal to noise ratio up to 4.6 for samples containing elements which produce this scattering.

XRD analysis was performed on unirradiated and ion-irradiated ODS steel samples. For the investigation we considered two kinds of ODS steels of the following composition:

1. Fe–15Cr–2W–0.2Ti–0.35Y_2O_3 (KP123),
2. Fe–15Cr–4Al–2W–0.35Y_2O_3 (KP4).

In the first case, due to the presence of *Ti* in this kind of ODS steel complex $Y_2Ti_2O_7$ oxide is formed as oxide nanoparticles instead of Y_2O_3 [18]; in the second case, $Y_4Al_2O_9$ oxide nanoparticles are formed due to the presence of *Al* [19].

Table 1
Samples; ions, used for irradiation; ion energies and fluences used for the investigation.

Sample	Ions	Ion energy, MeV	Ion fluence, cm^{-2}
KP123	unirradiated	–	–
KP4-Xe	Xe	167	1.5×10^{14}
KP123-Bi152	Bi	700	1.52×10^{13}
KP123-Bi615, front side	Bi	700	6.15×10^{12}
KP123-Bi615, back side	unirradiated	–	–