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In situ characterization of microstructural instabilities: Recovery, recrystallization and abnormal growth in nanoreinforced steel powder

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Abstract—An in situ X-ray diffraction experiment was set up to study the microstructural evolution of a nanostructured oxide dispersion-strengthened ferritic steel produced by high-energy ball milling. Dislocation density and grain growth between 20 and 1100 °C were quantified by coupling-modified Williamson–Hall and Warren–Averbach methods. During the early stages of heating, recovery through the rearrangement of dislocations increases the coherent domain size from 23 to about 60 nm. Once the annealing temperature reaches 800 °C, recrystallization starts. Using a specific analysis of 2-D detector signal, it has been possible to grasp the occurrence of abnormal growth leading to bimodal grain size distribution with both ultrafine grains and coarser micronic grains. The grain growth kinetics upon heating were determined for both populations and separately quantified. Ultrafine grains exhibit a continuous moderate growth rate, leading to continuous recrystallization, whereas specific grains experience a rapid abnormal growth up to their final size after a short incubation time.

Keywords: ODS steel; X-ray diffraction; Recovery; Recrystallization; Growth modelling

1. Introduction

Oxide dispersion-strengthened (ODS) ferritic steels are being intensely investigated for high-temperature nuclear applications due to their excellent creep properties and swelling resistance [1]. These assets are obtained thanks to a fine-grained body-centred cubic microstructure reinforced by a dense and homogeneous precipitation of yttrium nanooxides. The classical processing route of this material is based on powder metallurgy involving high-energy ball milling in order to obtain a homogeneous distribution of yttrium in the ferritic powder. This first step is followed by hot isostatic pressing [2–4] and hot extrusion [5]. In the most recent processing routes, the material is also regularly consolidated by field-assisted sintering [6,7]. Plastic deformation during mechanical alloying introduces a high dislocation density and deeply modifies the grain microstructure, which results in nanosized elongated grains [8]. Given the high deformation level and the small grain size, the driving force for microstructural instabilities is particularly high. Yttrium in solid solution precipitates rapidly from 600 °C under annealing. The Zener pinning force is thus also especially elevated due to the dense population of small yttrium precipitates. Having extremely high driving and retarding forces that oppose each other creates an important microstructural instability factor. The presence of an important concentration of particular pinning points, such as triple and quadruple grain boundary junctions, induced by the small grain size, will maximize the Zener force. Indeed, the smaller the grain size, the higher the number of particular points on which pinning is more efficient [9–12]. Therefore, the probability of triggering unstable phenomena like abnormal grain growth is very high.

When the material is extruded, abnormal grains elongate and are responsible for the large anisotropy of the material microstructure. This anisotropy induces detrimental mechanical properties [5] and particularly poor transverse creep strength [13], which is a key property in fuel cladding applications. Indeed, as internal pressure increases in the tube cladding with the accumulation of gas fission products, the major stress component is applied in the transverse direction. Therefore, the material faces a critical risk

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of failure and the control of the microstructure is a key issue to ensure that it can safely fulfil the role of first barrier against the release of radioactive elements. Despite its indisputable technological impact, microstructural instability, such as abnormal grain growth, of industrial nanostructured metallic materials remains only partially explored. The scarcity of such studies is primarily due to the difficulties in following the mechanisms leading to microstructural evolution: recovery, recrystallization and grain growth can be concomitant and are unpropitious to quantify in a time-resolved manner. Yet, for powder metallurgy, kinetic studies of the microstructural evolution starting from the as-milled powder and during subsequent annealing are essential to detect the beginning of critical phenomena like abnormal grain growth.

Because of its non-destructive character, X-ray diffraction (XRD) has proved to be an appropriate method for describing dislocation density and crystallite size [14–17], which are suitable microstructural features for monitoring phenomena such as recovery, recrystallization and grain growth. Nevertheless, the instability and rapid evolution of the ferritic microstructure requires the use of fast and precise in situ characterization methods. Kinetic studies have been successfully carried out by combining synchrotron XRD with fast 2-D detectors to obtain timely, well-resolved diffraction peaks [18,19]. No time-resolved study of recovery, recrystallization and further grain growth upon heating of a nanostructured ODS steel had been reported to date. In the present work, we focus on the evolution of the crystallite size and the dislocation density upon annealing. First, we report the methodology of the in situ XRD measurements and the adaptation of existing methods to improve data analysis. Since X-ray peak broadening is only sensitive to crystallite sizes below 1 μm, it is expected that this in situ investigation will deliver important information on the recovery and early stages of recrystallization of the ODS steel. These results are discussed in terms of the classical microstructural mechanisms of recovery and recrystallization. Using a special algorithm that captures individual spots of high intensity, a qualitative description of abnormal grain growth is also presented.

2. Materials, experimental set-up and methods

2.1. Material

A high-chromium ferritic steel powder was produced by ingot gas atomization by Aubert & Duval. The powder particles were then mechanically alloyed with submicronic yttria powder (Y_2O_3) by Plansee SE using a high-energy attritor. The powder is representative of common industrial nanocrystalline powder widely used to process nanostructured materials. Milling conditions and microscopic evaluation of the as-milled powder are respectively reported in Refs. [8,20]. Using a focused ion beam to produce a suitable thin foil from powder particles, the nanostructure was investigated by transmission electron microscopy and automatic crystallographic orientation mapping. Most of the grains were highly deformed and the amount of geometrically necessary dislocations was estimated from the kernel average misorientation to be over 10^{16} m^{-2}. The mean grain size observed was close to 100 nm [8], a result also confirmed by scanning electron microscopy (SEM)–electron backscattered diffraction investigations [20]. The chemical composition of the milled powder was measured by electron probe microanalysis and is reported in Table 1. In this particular alloy, yttrium, titanium and oxygen are expected to form nanoparticles during hot processing [1,3,4].

2.2. Synchrotron X-ray diffraction

2.2.1. Experimental set-up: an in situ 2-D detection study

Various XRD experiments have shown the benefits of using the synchrotron source to study recrystallization kinetics [18,17]. This study, based on line profile analysis of in situ acquired high-resolution XRD peaks, was performed at the DIFFABS beamline of the synchrotron SOLEIL. The incident energy of photons was selected to be 19 keV ($\lambda = 0.0653$ nm). This energy allows the optimization of the penetration depth, the number of available peaks and the peak width, each of these values being essential for the precision of the results. At this energy, the penetration depth of the beam into the powder was around 25 μm for the (211) planes. Eleven X-ray peaks can be detected in a 2θ range from 15 to 67°. Samples were tilted to 15° with respect to the axis parallel to the incident beam in order to minimize the geometrical spread of the beam onto the powder. Given these parameters, the four most intense diffraction peaks, (110), (200), (211) and (220), were located in a 2θ range at 18.5°, 26.3°, 32.3° and 37.5°, respectively. In situ X-ray diffractograms of these four peaks were recorded independently by a last-generation 2-D detector XPAD S140 composed of chips containing 120×80 pixels with dimensions of 130 μm × 130 μm (Fig. 1).

The resulting 1-D diffractograms were obtained from a radial 2θ range of 7° and an azimuthal ψ range of 6°. Each peak was detected in a single detection sequence, leading to four different detection phases in order to centre the 2θ domain around each peak's central position. An acquisition time of 2 s per peak was chosen, providing good statistics (peak intensity of more than 1 million counted photons, giving a signal to noise ratio of more than 500 for the silicon reference sample; for the ODS samples, the resulting maximum intensity over background ratio was 85). Taking into account the XPAD displacement time from one peak position to the next, the acquisition time of the four peaks took 15 s, with an additional 10 s for returning to the initial position. The 2-D diffraction rings were then integrated over the whole ψ range for a classic measurement and over a specific ψ range to separate the ultrafine grain and coarse grain signals when an abnormal microstructure was obtained. This step was carefully performed by customized routines in order to eliminate the reconstruction artefacts that can arise during evaluation [21].

Table 1. Composition of the as-milled powder measured by electron probe microanalysis.

Element	Fe	Cr	W	Y	O	Ti	Si
Powder (wt.%)	Bal.	14.6 ± 0.1	0.99 ± 0.02	0.16 ± 0.02	0.15 ± 0.01	0.32 ± 0.02	0.19 ± 0.01
Powder (at.%)	Bal.	15.1 ± 0.1	0.30 ± 0.02	0.98 ± 0.02	0.51 ± 0.01	0.35 ± 0.02	0.35 ± 0.01