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Effects of thermal and thermo-mechanical treatments on Fe-14Cr-ODS steels microstructure.

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INTRODUCTION

The Sodium cooled Fast Reactor (SFR) concept is one option for the next generation of nuclear reactors (GEN IV). However, these reactors should operate up to high irradiation doses (≈ 150 dpa) and with high nominal service temperature (≈ 650°C). New materials for fuel claddings are therefore necessary. Due to their good swelling resistance and their good mechanical properties at high temperature, ferritic/martensitic ODS (for Oxyde Dispersion Strengthened) steels are prospective materials.

ODS steels are classically elaborated by powder metallurgy involving high energy milling of Y_2O_3 and metallic powders. This mechanical alloying is followed by a consolidation by Hot Extrusion (HE) or by Hot Isostatic Pressing (HIP). Yet, ferritic ODS steels with high Chromium content (> 12%), consolidated by hot extrusion, exhibit a strong grain texture, both morphological and crystallographic ({110} fiber texture), which generates an anisotropy of mechanical properties [1, 2, 3]. These alloys show particularly poor mechanical properties in the transverse direction. Because of their high Cr concentration, these steels do not transform to austenite at high temperature and therefore it is not possible to change this texture by phase transformation during a heat treatment, as in lower Chromium containing martensitic/ferritic steels. Therefore, the only way by which a more equiaxed grain microstructure could be achieved is through recrystallization.

Because the oxide dispersion increases dramatically the recrystallization temperature [4, 5], it is necessary to use cold deformation and the associated high dislocation density to achieve recrystallization.

The main goal of this paper is to show that it is possible to obtain recrystallization without a significant degradation of the nanoscale precipitation microstructure, and thus the related mechanical properties.

STRATEGY OF THE STUDY

In this work, samples of ferritic 14Cr-1W-0.3Ti-$0.3Y_2O_3$ ODS steels elaborated at CEA by HE and HIP have been annealed at high temperatures (1300-1400°C). The evolution of the microstructure for each annealed state is characterized by Scanning Electron Microscope (SEM) and Small Angle Scattering (SAS).

In a second stage, HE samples have been cold deformed to different deformation conditions (Fig. 1) and heat treated at temperatures ranging from 850 to 1450°C during 30 min.



Fig. 1. Samples of HE 14Cr-ODS material cold deformed to different conditions.

The morphological and crystallographic anisotropy of the grain microstructure has been tracked by Scanning Electron Microscopy coupled with an Electron BackScatter Diffraction detector (SEM-EBSD) observations and X-ray diffraction texture measurements (XRD). Transmission Electron Microscopy (TEM) observations and SAS measurements have been performed to determine the particle size distribution. These analyses have helped to identify two “extreme” conditions of thermo-mechanical treatment for which the microstructure has been recrystallized.

Finally, these recrystallization conditions were applied on the same ferritic ODS steel, except that it was consolidated by Hot Isostatic Pressing (HIP) instead of extrusion, so that it presented an initially isotropic texture.

RESULTS

Evolution of the microstructure after heat treatment

Heat treatments have been realized on both HE and HIP consolidated ferritic steels to confirm the microstructure stability at very high temperature. Supporting on the work of H. REGLE’s PhD, the two temperatures which have been used are 1300°C and 1400°C, or a temperature lower and a temperature higher than 90% of melting point [6]. No signs of recrystallization have been detected even if precipitation coarsening by Ostwald ripening mechanism was evidenced. Indeed, the mean radius of nanoparticles increases from 1.3 nm in the initial state to 3.4 nm after

heat treatment at 1400°C during 1 hour (multiplicative factor of 2.6) and 4.4 nm after annealing at 1400°C during 3 hours (factor 3.4).

Evolution of microstructure after thermo-mechanical treatments on HE material

Only two cold deformation rates (40% and 70%) will be presented in this paper. The different metallurgical states show an evolution of the microstructure with temperature and cold deformation. Indeed, Vickers hardness decreases with increasing annealing temperature. For example, the Vickers hardness decreases from 475 HV_1 in the initial state to 247 HV_1 after 40% cold deformation followed by 1450°C heat treatment. This loss of hardness is due to recovery / recrystallization that were confirmed by SEM observations. Despite some residual areas of supposedly unrecrystallized grains, the microstructure consists mostly of large equiaxed grains (≈ 10 µm) (Fig. 2). A similar microstructure is obtained for the 70%-1150°C / 30 min sample.

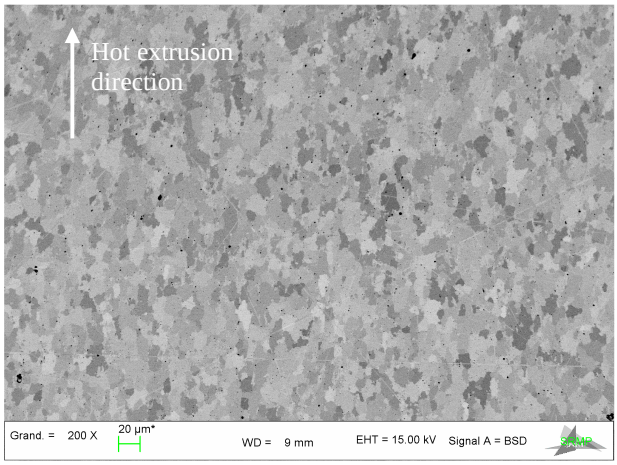


Fig. 2. Microstructure of the 40%-1450°C / 30 min sample.

SAS analyses indicate that the better condition for recrystallization is 70% of cold deformation and heat treatment at 1150°C because no coarsening of the precipitate microstructure has occurred (Fig. 3). This observation shows that there is no strict correlation between the recrystallization process and precipitation coarsening.

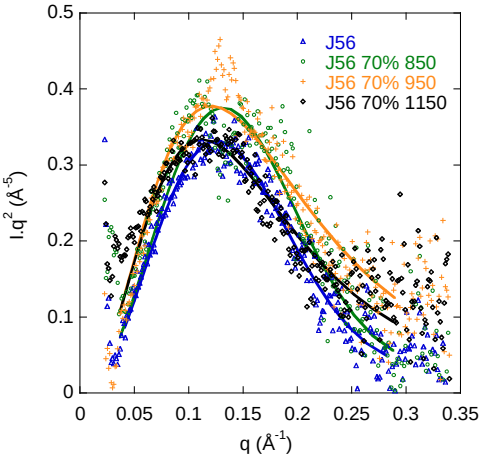


Fig. 3. Kratky's representation of SAS measurements of 70% cold deformed HE ODS steel heat treated at temperatures between 850 and 1150°C compared with the un-deformed and un-heat treated specimen.

Evolution of texture during thermo-mechanical steps

An important point to understand the recrystallization process is to monitor the texture evolution. For this, texture measurements by X-ray diffraction (XRD) and EBSD analyzes have been realized.

As expected, we observe a strong fiber texture with the <110> direction parallel to the extrusion direction in the un-deformed and un-annealed HE sample. After deformation by uniaxial compression, the <110> fiber texture in the extrusion direction is replaced by a <111> fiber texture in this same direction in accordance with what is typically observed in body centered cubic steels [7]. After annealing, this fiber texture <111> is still present with an apparently lower intensity despite the sample recrystallization. In order to illustrate the presence of the fiber texture in the recrystallized areas, EBSD analyses were performed and confirmed the preferential orientation of the recrystallized grains.
Furthermore, as Chou and Bhadeshia conclude [4], the high intensity of <111> fiber appears to facilitate recrystallization.

Evolution of microstructure after thermo-mechanical treatments on HIPed material

Recrystallization tests with the same conditions like on the HE material were carried out. However, the HIPed material does not exhibit the same behavior as the HE material. Indeed, no condition allows obtaining recrystallized microstructure suggesting a shift of the recrystallization temperature to higher temperature, which seems to be confirmed by Vickers hardness measurements.

In conclusion, the Hot Extruded sample recrystallized more easily, even in the absence of nanoparticles coarsening, than the Hot Isostatic Pressing sample.

Hypothesis to explain this difference include a higher stored energy from dislocation density and differences in crystallographic and morphological texture.

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