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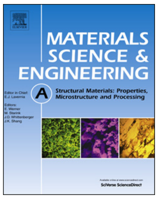

Effects of fabrication processing on the microstructure and mechanical properties of oxide dispersion strengthening steels

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ABSTRACT

Two types of oxide dispersion strengthening (ODS) steels (i.e. 12Cr-ODS-NIFS and 12Cr-ODS-IMR) with similar chemical compositions were produced through different fabrication processing. A systematic comparison was performed to investigate the effects of different fabrication processing on microstructure and mechanical properties of ODS steels. Compared to 12Cr-ODS-NIFS, the 12Cr-ODS-IMR steel exhibited significantly enhanced mechanical properties, such as higher Vickers hardness, tensile strength and creep strength. To understand the microstructural reasons for the excellent mechanical properties of ODS steels, systematic microstructure characterizations were carried out through a combined study of X-ray diffraction, electron backscattered diffraction, and transmission electron microscopy. It was found that both steels had similar grain structures comprising fine and heavily deformed grains smaller than ~1 μm and elongated coarse grains larger than ~10 μm, both of which possessed similar precipitates including coarse TiC_xO_{1-x} particles and homogenously distributed Y_2TiO_5 nanoparticles with diameters of several nanometers. 12Cr-ODS-IMR had a finer grain size, larger aspect ratio of grain length to width, and higher density of dislocations, which should be derived from the additional cold rolling and responsible for its better mechanical properties compared with 12Cr-ODS-NIFS.

1. Introduction

Reduced activation martensitic/ferritic (RAFM) steels are the primary candidates for the fusion blanket structural materials [1], but their creep strength decreases significantly at temperature over ~823 K [2]. Through introducing nano-scale oxide particles into the iron matrix, oxide dispersion strengthening (ODS) steels can be used at higher temperature (~923–973 K) [3,4]. The fine dispersed particles can retard the motion of dislocations and grain boundaries, stabilize the microstructure at high temperature and contribute to the outstanding tensile and creep strength [3–6]. In addition, the interfaces between the high-density nanoparticles and matrix can act as trap sinks for irradiation-induced defects and nucleation sites for helium bubbles, resulting in a high resistance to radiation hardening and swelling [7]. Therefore, ODS steels have recently attracted increasing interests due to the potential applications in fusion blanket system and advanced nuclear fission reactors [3–22].

A key factor for developing ODS steels is controlling their microstructure to improve the mechanical properties under critical service environment with high temperature and intense neutron irradiation. In contrast to RAFM steels produced by traditional melting methods, ODS steels are fabricated by powder metallurgy methods, involving mechanical alloying followed by sintering to obtain micro-scale grain structures, high-density dislocations, and homogeneously distributed oxide particles [8]. Hot extrusion (HE) and HIPing are the most common compaction methods. HE can induce a suitable ductility and low ductile–brittle transition temperatures (DBTT) but anisotropic elongated and textured grains [3,10,13–15]. Other thermal-mechanical processing, e.g. hot forging [3,16,17], hot rolling [3,17,18] and cold rolling [4,10,18], can also be applied on ODS steels to improve properties for different demands. Hot extrusion combining with hot forging can decrease further the porosity of ODS steels and obtain a homogeneous microstructure, thereby improving the ductile properties. Cold rolling can induce a high accumulation of stored energy in materials and promote the recrystallization for achieving a higher deformation ratio and higher strength.

The manufacturing processes are crucial in defining the final microstructure of ODS steels (e.g. grain size, dislocation density, precipitates, oxide particles, and etc) and therefore significantly

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affect the mechanical properties [8–18]. Thus, selecting suitable fabrication procedure is of essential importance for optimizing the microstructures of ODS steels to improve their mechanical properties so that they can meet all the critical requirements for the nuclear applications. In the past decades several ODS steels were developed for the potential applications in fusion/fission reactors [3–10]. However, these ODS steels had different chemical compositions and were fabricated through different manufacturing processes, rendering it impossible to conduct a systematic and comparative study. Up to date, our knowledge of the effects of processing routes on the microstructure and mechanical properties of ODS steels is still very incomplete and even limited.

To clarify the effects of processing routes on the microstructure and mechanical properties of ODS steels, we fabricated two 12Cr-ODS steels (i.e. 12Cr-ODS-NIFS and 12Cr-ODS-IMR) with the almost same chemical compositions and the same procedures of mechanical alloy, hot extrusion, and hot forging. The obvious difference of processing routes existed in the final cold rolling, which was conducted in 12Cr-ODS-IMR while absent in 12Cr-ODS-NIFS. A systematic comparison of the microstructure and mechanical properties of both ODS steels was performed to evaluate the effects of different processing routes, which will provide an important guidance to develop advanced ODS steels.

2. Experimental procedures

12Cr-ODS-IMR steel was produced in Institute for Materials Research (IMR) under a framework of Japan Science and Technology (JST) project after the nuclear accident in 2011. While 12Cr-ODS-NIFS was manufactured in National Institute for Fusion Science (NIFS) to make reference ODS steels for joint research by Japanese Universities [17]. The chemical compositions (in wt%) were 12.01Cr, 1.91W, 0.20Y, 0.31Ti, 0.06O, 0.033C, and balance Fe for 12Cr-ODS-IMR, and 11.65Cr, 1.90W, 0.18Y, 0.29Ti, 0.083O, 0.035C, and balance Fe for 12Cr-ODS-NIFS, respectively. The detailed fabrication routes for both steels are represented in Fig. 1. At first, the argon gas atomized powders with particles size of <150 μm were mechanically alloyed (MA) for both steels. Following the MA process, the MA powders after sealing and degassing were consolidated by hot extrusion at $\sim$1423 K with an extrusion ratio of 6:4. Then the extruded bars were hot-forged into plates. For 12Cr-ODS-IMR, an additional cold rolling with the deformation level up to 40% was performed. Finally, 12Cr-ODS-IMR was annealed at 1323 K for 1 h and then cooled down by air cooling, while 12Cr-ODS-NIFS was annealed at 1473 K for 1 h followed by air cooling.

The mechanical properties of both ODS steels including Vickers hardness, tensile and creep properties were systematically measured. To investigate the microstructural stability of both ODS steels, a series of annealing heat treatments were carried out at 973–1473 K for 1 h. All the hardness was measured at room temperature (RT) with a load of 1000 g and a loading time of 15 s. SSJ samples for tensile and creep property tests were machined along the extrusion direction (ED) with a gauge section of 5 mm $(L) \times$ 1.2 mm $(W) \times$ 0.25 mm (T). The tensile properties were tested from RT to 973 K with an initial strain rate of 6.7×10^{-4}/s. The 0.2% proof strength was measured as the yield strength. The uniaxial creep tests up to rupture were performed at 973 K and 873 K with different stresses.

The samples for EBSD analysis and SEM observations were carefully prepared and the details can be found in Ref. [5]. EBSD analysis were performed to investigate grain morphology, grain size and misorientation angle. SEM was performed at 15 kV to

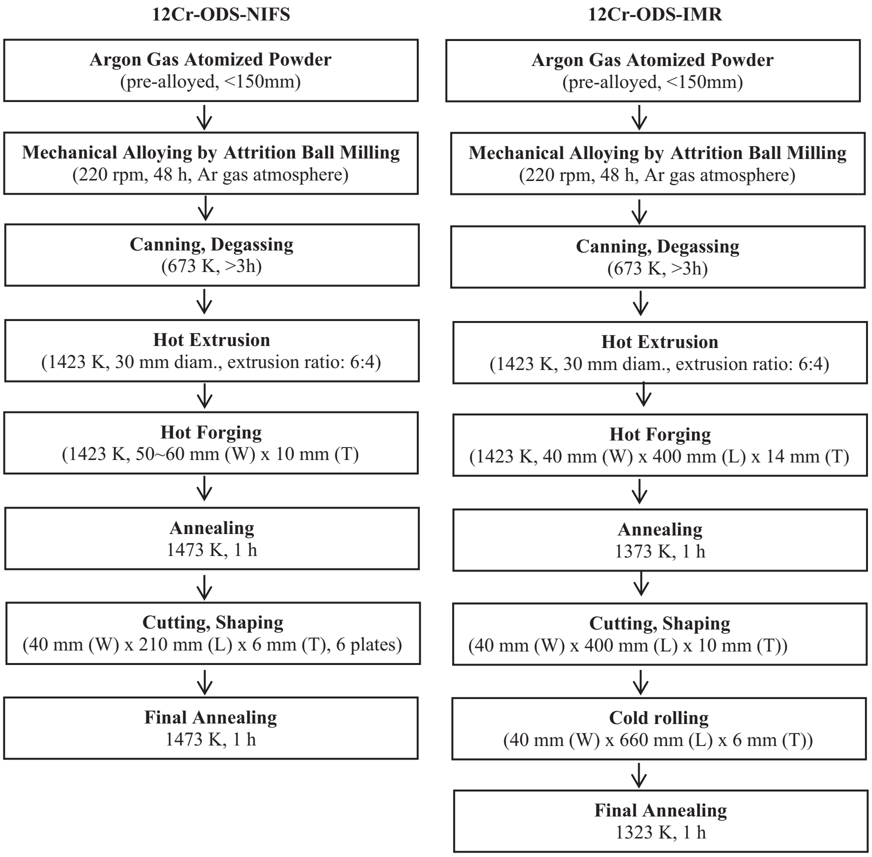

Fig. 1. Manufacturing processes for 12Cr-ODS-IMR and 12Cr-ODS-NIFS plates.