Journal of Nuclear Materials 459 (2015) 205–216

Contents lists available at ScienceDirect

Journal of Nuclear Materials

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

Effects of cryomilling on the microstructures and high temperature mechanical properties of oxide dispersion strengthened steel

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ARTICLE INFO

Article history:
Received 1 October 2014
Accepted 14 January 2015
Available online 21 January 2015

ABSTRACT

The effects of cryomilling on the microstructures and high temperature mechanical properties of oxide dispersion-strengthened (ODS) steel were examined. Cryomilling was newly tried on this ODS steel to control oxides, grains, and dislocation microstructures. Fe–14Cr–3W–0.4Ti (wt.%) alloy powder and 0.3 wt.%Y_2O_3 powder were mixed and were mechanically alloyed (MA) through ball milling at each of room temperature (RT) and −150 °C and then hot isostatic pressing (HIP), hot rolling, and annealing processes were implemented to manufacture two types of ODS ferritic steel, K1 (RT) and K4 (−150 °C). Oxide particles were shown to be finer and more uniformly distributed in K4 (5–10 nm size distribution) than in K1 (average size 30 nm). The two alloys were subjected to high temperature compression (RT ~ 900 °C) tests. K4 represented higher yield strength under all temperature conditions. However, K4 showed rapid strength decreases at high temperatures exceeding 700 °C and showed similar levels of strengths to K1 at 900 °C. This is considered attributable to the fact that although cryomilling increased the number density of oxide particles, it simultaneously reduced grain sizes too much, so that grain boundary weakening at high temperatures could not be sufficiently prevented.

1. Introduction

The advanced oxide dispersion-strengthened (ODS) steel is a material consisting of the Fe matrix in which several-nano to several tens of nano-sized oxide particles are uniformly dispersed at high number densities, which is attracting great attention as a structural material for generation IV reactors and fusion reactors because its resistance to high temperature creep and neutron irradiation damage is quite excellent [1–5]. In addition, because of its excellent high-temperature mechanical properties and its characteristics, such as excellent corrosion and oxidation resistance, it is expected to be applied as a material for the heat-resistant components of various gas turbines and engines for which the enhancement of heat resistance and durability limits is required. That is, currently, studies to develop advanced ODS steel are being actively conducted because the possibility of demand for this material is shown not only in the areas of future-type nuclear power and nuclear fusion systems, but also in various other industrial fields such as automobiles, aviation, power generation, and munitions [6].

In general, ODS steel is manufactured by mixing raw material powder with Y_2O_3 powder, mechanically alloying (MA) the mixture through ball milling, and consolidating it through hot extrusion or hot isostatic pressing (HIP) [1]. Here, the MA and consolidating processes are the main processes that determine the properties of ODS steel. Through the MA, Y_2O_3 powder is dissociated into the matrix and alloyed with the raw material powder. Thereafter, in the consolidating process, it meets the alloy element (Ti) in the matrix to form nano-oxide particles ($Y_2Ti_2O_7$, Y_2TiO_5) [7–9]. These oxide particles are thermally and mechanically very stable to suppress the movements of dislocations and grain boundaries (to improve high-temperature strength and creep properties) under high-temperature stress. In particular, these particles, smaller than 10 nm in size, exist coherent with the matrix to reduce misfit strain with matrix, thereby maintaining the alloy stably at high temperatures and increasing resistance to void swelling and helium embrittlement due to neutron irradiation [10–13].

However, dispersing Y_2O_3 uniformly in the matrix through milling is a quite complicated and difficult process that very sensitively reacts not only to the properties of raw material powder, but also to milling conditions. If the MA process is not optimized, Y_2O_3 will not be uniformly distributed to show a rapid growth of grains at

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http://dx.doi.org/10.1016/j.jnucmat.2015.01.032
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Table 1
The chemical compositions of K1 and K4 alloys (wt.%).

	Fe	Cr	W	Ti	C	N
K1	Bal.	13.88	2.97	0.42	0.013	0.026
K4	Bal.	13.76	3.21	0.45	0.066	0.091

some areas during consolidation, eventually leading to the formation of non-uniform bimodal microstructures where coarse and fine grains coexist [14]. In addition, MA is a process implemented for a long time, and great amounts of heat are generated in progress due to friction between balls and powder, vibrations, and plastic deformation. Therefore, if the generated heat is not sufficiently discharged over time, milling effects will decrease so that the quality of alloyed powder is deteriorated, eventually leading to the occurrence of the abovementioned phenomena, which may adversely affect the final properties [15]. Despite the fact that cooling conditions (milling temperature) during MA change the microstructures of ODS powder and the alloy and thus may considerably affect high-temperature mechanical properties, which are among the most important properties of ODS steel, no studies on this matter have been reported thus far.

In particular, if MA is implemented under the cooling conditions of cryogenic temperatures, high-impact energy will be delivered to the powder so that grains and oxide particles can become finer and evener in size, and oxide particles can be distributed uniformly, leading to more-excellent strengthening effects [16,17]. However, due to increases in brittle fractures, it may become easier to have inflows of impurities from the milling media, or the interfaces between powder particles may become unstable. In addition, as diffusion is suppressed, the solubility of alloy elements in matrix may become lower, leading to unexpected effects on the high-temperature mechanical properties of ODS steel.

Therefore, the present study was intended to examine the high-temperature mechanical (compression) properties of ODS steels (RT milling material: K1, −150 °C milling material: K4) manufactured through MA under the cooling conditions of RT and −150 °C, which are extremely contrasting temperatures, and to analyze the effects of microstructure factors on the mechanical properties to figure out the correlations between cryomilling and high-temperature deformation/fracture behavior.

2. Experimental procedure

The raw material powder used in the present study was an alloy powder with a chemical composition of Fe–14Cr–3 W–0.4Ti (wt.%) manufactured through gas-atomization into globular shapes with particle sizes in a range of 50–100 μm. This raw material powder was mixed with 0.3 wt.%Y_2O_3 powder with particle sizes in a range of 30–50 nm, and the mixture was put into a milling chamber to perform mechanical alloying (MA). The MA was implemented for 40 h in a highly pure Ar (99.999 wt.%) gas atmosphere at each of RT and −150 °C temperature conditions (±5 °C temperature deviation). At this time, ball milling was done in dry states, the ball-to-powder weight ratio was 25, the milling container and the grinding media used were made of AISI 52-100 hardened steel, and the rotation speed was set to 100 rpm. Liquid nitrogen was used as a refrigerant for the −150 °C temperature condition, and the liquid nitrogen was made to flow outside the milling chamber so that it would not come into direct contact with the powder. Temperatures were monitored using a thermocouple connected to the inner wall of the milling chamber. Thereafter, the mechanically alloyed powder was put into a mild steel can made of SUS316, and 1 Pa level vacuum state was created in the can at a temperature of 400 °C, the can was sealed, and the powder was consolidated through HIP at 1100 °C. Finally, the material was hot rolled at 1150 °C with a thickness reduction ratio of 50% and annealed at 1100 °C to

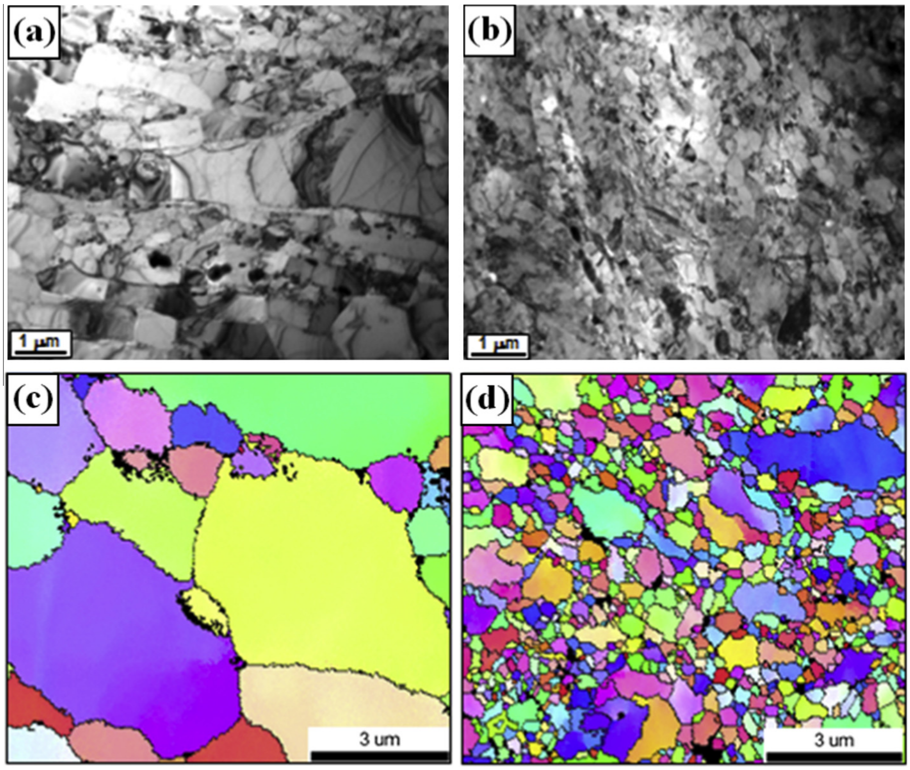

Fig. 1. TEM images and EBSD maps showing the microstructure of (a) and (c) K1 alloy and (b) and (d) K4 alloy [17].