Microstructural evolution and mechanical properties of ODS steel produced by cryogenic milling.

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1. Introduction

Oxide dispersion strengthened (ODS) steel has great advantage at creep resistance as well as irradiation resistance. Recent progress shows ultra-fine grained microstructure with high density of nanoclusters (NCs, here after) [1-5]. They exhibited superior creep resistance compared with conventional ferritic/martensitic steels. Generally, ODS steel is produced by high energy mechanical alloying (MA) process in order to make fine distribution of Y_2O_3 powder throughout ferritic or ferritic/martensitic matrix [6, 7]. During the step, dissolution or morphization of Y_2O_3 powder into the matrix occurs. Since the amount of lattice defects or strains generated by MA has a major role in oxide particle dissolution, proper control of MA parameters not only may help the dissolution process but also can improve the other microstructural properties, which results in better mechanical properties [8].

MA is a long-term continuous operation producing a lot of heat. If the heat cannot be discharged in time, it will directly affect the efficiency of milling and quality of resulting powder. The dissolution or amorphization process during MA is believed to be dependent on temperature but still no information is available yet. Since diffusion processes are involved, the formation of expected or unexpected alloy phase can be affected by the temperature of powder and milling media. For example, the degree of solid solubility and the formation of amorphous or nanocrystalline phase may be different according to temperature.

Present work is performed to clarify aforementioned issues and design new manufacturing process for advanced ODS steel. To facilitate the experiments, we choose to put very excess amount of Y_2O_3 (15 wt.%) into Fe-14Cr-3W pre-alloyed powder instead of 0.3 wt.% Y_2O_3 which is normal content in this kind of system. This is because Y_2O_3 peak cannot be detected by XRD in the martial which contain such a low amount of Y_2O_3. The larger amount of Y_2O_3 content permits to study the dissolution kinetics and the oxide precipitation by XRD study.

2. Methods and Results

2.1 Materials and experiment procedures

ODS alloys with a nominal composition of Fe–14Cr–3W–0.4Ti–$0.25Y_2O_3$ (wt.%) was processed at Korea Institute of Materials Science by mechanical alloying of a mixture of pre-alloyed metallic powder (powder size between 45 and 150 μm) and Y_2O_3 powder (powder size between 17 and 31 nm) in a static argon environment. Mechanical alloying processes were conducted at room temperature, -90, and -180°C.

The mechanically alloyed powder was filled into a mild steel can that was evacuated to a vacuum of ~1 Pa at a temperature of 400°C, sealed, and hot extruded at 850°C. The processing is described in detail elsewhere [9].

In order to investigate the powder morphology and to characterize the size and distribution of the particles before and after mechanical alloying, JEOL-7001F was used for scanning electron microscoping. Also, the structural evolution during MA was examined by using X-ray diffractometer, Rigaku-2500 machine with a Cu-Kα radiation. Very slow scanning rate (0.003°/sec) in the range from 20 to 90° was employed to determine dissolution or amorphization behavior of pre-alloyed and Y_2O_3 powder.

2.2 Powder evolution

As mechanical alloying temperature decreases, resulting powder particle size decreased. This is due to the fact that cold welding stage was avoided under cryogenic milling conditions. As well as particle size, grain size was also decreased with milling temperature decrease. In order to investigate the fracture mechanism, the fracture surface of a sample tensile tested at 900 °C and a strain rate of $10^{-3}s^{-1}$ was examined. Fig. 1 shows the region where the FIB sample was taken and a STEM image of the sample. Relatively large cavitation at grain boundaries, which is commonly produced by GBS, was not observed. The smooth surface and round shaped grains shown in Fig. 1(c) confirm the occurrence of grain boundary decohesion. Both the diffraction pattern and EDX in Fig. 1(c) and Fig. 2, respectively, show that the surface grains are inherent matrix grains and not a metal oxide formed at high temperature. Also, they are not considered to be incipient melting particles, because incipient melting normally occurs above 1300 °C for steel. Fig. 1(d) is a TEM image of a sliced surface of the sample fractured at 700 °C. Compared with the image of Fig. 1(c), the morphology of very sharp tearing fracture is noticed, which indicates extensive dimple formation.

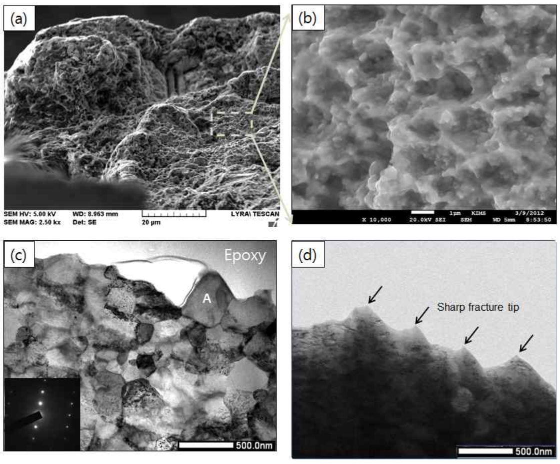

Fig. 1. The fracture surface of ODS sample (a, b) tensile tested at 900 °C; (c) TEM image of the fracture surface sliced parallel to tensile direction shows very smooth grain boundary lines. The inset is the diffraction pattern of grain ‘A’; (d) TEM image of the fracture surface of the sample tensile tested at 700 °C.

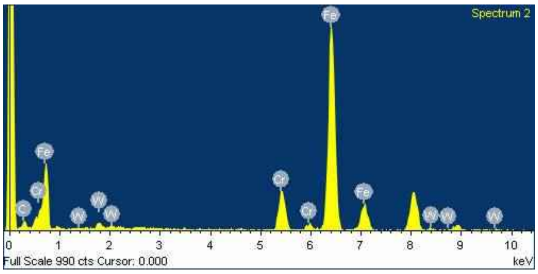

Fig. 7. EDX spectrum obtained at the grain ‘A’ in Fig. 2(c). No peak of oxide compound is detected.

2.3 Deformation mechanisms

Closer views of microcracks showing their coalescence and propagation were revealed by SEM micrographs. These microcracks are believed to initiate from coarse precipitations or micropores and to preferably propagate following the aggregate boundaries looking clear and thick. These observations indicate that the ductility of the sample alloy, and possibly the fracture toughness, depends on the local strain level where the microcracking initiates. In general, the fracture behavior of materials produced by powder metallurgy strongly depends on the pore, oxide/carbide distributions, and/or grain shape. A remedy for increasing ductility of such alloy will be more complete degassing.

3. Conclusions

To improve the properties of advanced ODS alloy, the effectiveness of cryomilling on the milling behaviour of Y_2O_3 and Fe-alloy powder was investigated. It was found that resistance to dislocation emission across obstacles did not drop abruptly with temperature rise. TEM observation of the fracture surface of a sliced sample confirmed the occurrence of grain boundary decohesion at 900 °C and a strain rate of $10^{-3}s^{-1}$.

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