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Effect of Mechanical Alloying and Hot Consolidation Processes on the Microstructure and Mechanical Properties of ODS Steels

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1. Introduction

Oxide dispersion strengthened (ODS) steel is the most promising candidate for a core structural material for next-generation nuclear systems such as a Gen. IV fission and DEMO fusion reactors, mainly due to its excellent high temperature strength and irradiation resistance [1]. Finely dispersed nano-oxide particles with a high number density in the homogeneous grain matrix are essential to achieve superior mechanical properties at high temperatures, and these unique microstructures can be obtained through the mechanical alloying (MA) and hot consolidation processes. Microstructure of ODS steel significantly depends on its powder properties including the particle size, purity, and homogeneity after the MA process. The hot consolidation process is also very important to develop favorable microstructures. These fabrication processes should be carefully controlled to improve the mechanical property at high temperatures.

In the present study, the effects of mechanical alloying conditions and hot consolidation process on the microstructure and mechanical properties of ODS ferritic steels are investigated.

2. Methods and Results

2.1 Experimental procedure

The ODS steels used in this study are Fe-15Cr-1Mo-0.35 Y_2O_3 in wt%. Some minor elements such as Ti and Zr were added to modify the oxide particle distribution. The ODS steels were fabricated by mechanical alloying and a hot isostactic pressing (HIP) process. Metallic raw powders and Y_2O_3 powder were mechanically alloyed by a horizontal ball-mill apparatus(Model: Simoloyer CM-20). Mechanical alloying was carried out under ultra-high purity argon (99.9999%). The mechanical alloying process was performed at impeller rotation speeds of 160, 240rpm for 48, 60hrs with a ball-to-powder weight ratio (BPWR) of 15:1. After the mechanical alloying, the particle distribution was measured by a laser diffraction scattering method using a particle size analyzer. The surface morphology of MA powders was observed using SEM. Chemical compositions and oxygen concentration of MA powders were analyzed by an ICP-AES and KS D 1778 methods, respectively. MA powders were then sieved and charged in a type 304 stainless steel capsule. All powder handling processes for the weighing, collecting, sieving, and charging were conducted in completely controlled high purity argon atmosphere to prevent the oxygen contamination during these processes. Sealed capsules were then degassed at 400^oC under a vacuum degree of 5×10^{-4} torr for 3h. To investigate the effect of HIP temperatures and pressure, HIP was carried out at 950-1150^oC under 100, 190MPa for 3h at a heating rate of 5^oC/min and followed by a furnace cooling. Hot rolling at 1150^oC was carried out in a fixed rolling direction for a plate shape with 65% of a total reduction rate. Detailed conditions for mechanical alloying and HIP process are given in Table 1. The grain morphology and precipitate distributions were observed by the electron microscopy. Tensile test specimens were taken out in the rolling direction of hot rolled ODS steel, and tensile tests were carried out at room temperature and at 700^oC.

Table I. Fabrication processes of ODS steels

	Mechanical alloying				Hot isostatic pressing			
	Milling time (h)	Milling speed (rpm)	BPWR	Atm.	Heating rate (^oC/min.)	Temperature (^oC)	Duration time (h)	Pressure (MPa)
1	48	160	15:1	Ar	5	1150	3	100
2	48	240	15:1	Ar	5	1150	3	100
3	60	240	15:1	Ar	5	1150	3	100
4	48	240	15:1	Ar	5	950	3	100
5	48	240	15:1	Ar	5	1050	3	100
6	48	240	15:1	Ar	5	1150	1.5	100
7	48	240	15:1	Ar	5	1150	3	190

2.2 Effect of different MA process conditions

The surface morphologies of MA powder in various mechanical alloying parameters are shown in Fig. 1. All powders were spherical or flake shapes with a somewhat rough surface. The powders milled at 240rpm for 48 were shown to be relatively uniform, while those milled at 160rpm was appeared to be ununiform in terms of particle size. An irregular flake shape of 160rpm means that the pulverization of the raw powders is not sufficient during the MA process, and a partial agglomeration with stacking occurs. An increase in the milling time promoted to enlarge the MA powder size to be almost twice. Smaller MA powder normally has an advantage of a higher charging density when a capsule fills with MA powders, and thus the milling at 240rpm for 48hrs is considered to be more favorable in this point of view.

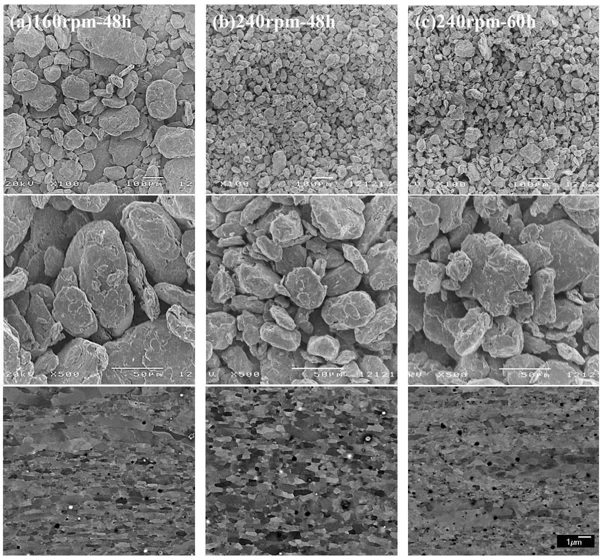

Fig. 1. Surface morphology of MA powders and microstructures of ODS steels in different milling conditions.

SEM images of a grain morphology observation on ODS ferritic steels milled in different milling conditions are shown in Fig. 2. Elongated grains toward the hot rolling direction which is parallel to the horizontal direction in the images were observed. Secondary recrystallization was found to occur during the hot rolling process at 1150 °C because elongated grains were clearly distinguished through an observation of the back-scattered secondary electron image mode. Grain distribution of ODS steel milled at 240rpm for 48h was considerably homogeneous, while low milling speed ODS steel showed the co-existence of fine and huge grains. ODS steel milled for 60h showed a very fine and uniform grain distribution.

2.3 Effect of different HIP process conditions

The grain morphology on ODS ferritic steels consolidated in HIP process conditions were observed. At different HIP temperatures of 950 to 1150°C for 3h, no significant changes were observed in the grain morphologies. In Fig. 3, changes in the tensile properties were shown. Low HIP temperature tended to increase both tensile strength and elongation of ODS steels, but the effects could be negligible. This means that a low consolidation temperature of 950°C is enough to form the fine grains and nano-oxide particles of ODS ferritic steels. However, it is obvious that higher consolidation temperature makes the grains and oxide particles unstable, and the mechanical properties should be deteriorated [2]. The HIP duration time and pressure are also important parameters to determine the properties of ODS ferritic steels. However, a short HIP duration time of 90min and high HIP pressure of 190MPa were insufficient to enhance the tensile strength at high temperature.

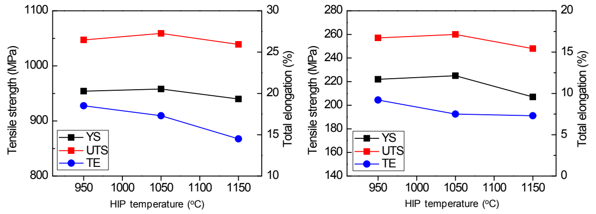

Fig. 2. Tensile property changes in different HIP temperatures.

3. Conclusions

ODS ferritic steels were fabricated under various conditions to investigate the effects of mechanical alloying and hot consolidation process on the microstructures and mechanical properties. A slow milling speed made a heterogeneous grain distribution, and a long milling time resulted in high impurity inclusions in the microstructure. High HIP pressure was not sufficient to densify the ODS ferritic steels. There were no significant effects of HIP temperature from 950 to 1150°C on the grain morphology and tensile strength. On the basis of these results, the fabrication process of 15Cr ODS steels could be optimized to have a favorable microstructure and tensile properties at high temperature.

Acknowledgements

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