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Influence of the fabrication process parameters on microstructures and mechanical properties of 10Cr-1Mo ODS steel

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1. Introduction

In several countries with nuclear power reactors, considerable research and extensive effort for Generation IV future nuclear systems have been made to improve the efficiency, safety, reliability, and proliferation-resistance of nuclear reactors [1]. For this, it is imperative to develop an advanced structural material with both high strength and irradiation resistance at high temperatures under severe neutron exposure environments [2]. Ferritic/martensitic (FM) steels are considered as an attractive candidate material for a structural component of a nuclear system such as a sodium cooled fast reactor (SFR) owing to their excellent neutron radiation resistance to a void swelling, compatibility with various types of coolant, and technological maturity [3-5], but are known to reveal an abrupt loss of their creep and tensile strengths at temperatures above 600 °C [6]. Accordingly, oxide dispersion strengthened (ODS) FM steels have been developed as the most promising core structural material for high- temperature components operating in severe environments such as nuclear fusion and fission systems owing to its excellent elevated temperature strength and radiation resistance stemming from the addition of extremely thermally stable oxide particles dispersed in a ferritic/martensitic matrix [7-9].

To realize the structural components such as plates, sheets and tubes in SFR, the development of manufacturing processes is an essential issue for the ODS FM steel. While the ODS steel has superior radiation resistance and high temperature strength, in comparison with the existing commercial steels, it is difficult for the ODS steel to obtain sufficient workability for the fabrication due to high hardness and low ductility at room temperature, meaning that the manufacturing of the ODS plate including cladding tube can be complicated by the low cold workability. In order to prevent the ODS steel from any damage during the manufacturing process, thus, the introduction of intermediate heat treatments between cold rolling processes is necessary [10].

This study investigates effects of the fabrication process parameters such as the cold working ratio, the intermediate and final heat treatments on the microstructure and mechanical properties of 10Cr-1Mo ODS steel. In an effort to optimize the manufacturing route of the ODS FM steel, the microstructural and mechanical evolutions for the ODS plate manufactured by a control of the fabrication process parameters were evaluated in the present study.

2. Experimental procedure

The work presented herein was focused on ODS FM steel, the chemical compositions of which are given in Table 1.

Table 1. Chemical composition (wt. %) of 10Cr-1Mo ODS FM steel.

Alloy (wt.%)	Fe	Cr	Mo	Mn	V	Ti	C	Y_2O_3
10Cr-1Mo ODS FM steel	Bal.	10	1.2	0.5	0.15	0.25	0.13	0.35

A 10Cr-1Mo ODS steel was prepared by mechanical alloying (MA), hot isostatic pressing (HIP), and a hot extrusion process. Pre-mixed metallic raw powders and yttria powder were mechanically alloyed under a high purity Ar gas (purity in 99.999%) atmosphere. The MA powders were placed in an AISI 304L stainless steel container, sealed after a degassing process, and consolidated by the HIP process at 1150°C under a pressure of 100 MPa for 4 h. Hipped samples were hot-extruded by a 600 ton capacity press for several seconds with a 6.3: 1 extrusion ratio after annealing in the furnace at 1100°C for 2 h. The hot-extruded specimen was annealed at 1150 °C during 4min followed by a slow cooling to obtain a homogenized and softened microstructure, and then was machined to a plate shape with a thickness of 4mm.

Table 2. Intermediate heat treatment conditions of 10Cr-1Mo ODS FM steel.

Specimen	Intermediate heat treatment (IHT) conditions		
	Heat ID	Temp.(°C)	Time (hr)_Cooling type
10Cr-1Mo ODS Steel	IHT 1	1150°C	4min_FC
	IHT 2	780°C	1hr_AC
	IHT 3	1050°C X 780°C	(1hr_AC) X (1hr_AC)

To determine the optimized cold rolling condition, the annealed specimen was cold-rolled with a cross-section reduction ratio of 5~15%. After the cold rolling, various intermediate heat treatments were employed, as given in Table 2. 10Cr-1Mo FM ODS steel was prepared by mechanical alloying (MA), hot isostatic pressing (HIP), and hot extrusion process. Hardness measurements were carried out after the cold rolling process and intermediate heat treatments to evaluate the influences of the intermediate heat treatments on the mechanical properties. The microstructures were observed using SEM, electron back-scatter diffraction (EBSD) and transmission electron microscopy (TEM).

3. Results and Discussions

The workability of steels can be estimated by hardness measurement during the cold rolling followed by the heat treatments. Table 3 shows the results of the difference in hardness with cold working ratio in the range of 5 to 15%.

Table 3. Vickers hardness measurements of the cold-rolled 10Cr-1Mo ODS FM steel.

Reduction Ratio (%)	10Cr-1Mo ODS Steel					
	5%		10%		15%	
	Result	Hv	Result	Hv	Result	Hv
M.P		334		329		331
1st CR	Pass	343	Pass	361	Pass	381
2nd CR	Pass	358	Crack	402	Crack	425
3rd CR	Pass	377				
4th CR	Crack	392				

Our findings revealed that some damages such as cracks were observed at the plate and its hardness value increased up to more than 400 Hv after the ODS plate was cold-rolled with a reduction ratio of over 15%, which is consistent with the previous study [11].

Fig. 1 shows the effect of intermediate heat treatments during the cold rolling on the Vickers microhardness of the specimen. It can be seen that IHT1 was found to lead to a significant hardness decrease in the range of 30 to 40 Hv.

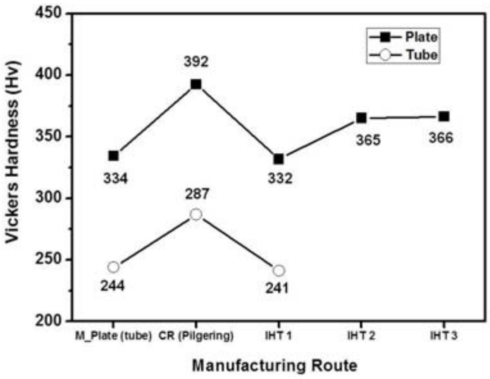

Fig. 1. Effects of intermediate heat treatments during the cold rolling on the Vickers microhardness of 10Cr FM ODS steel.

From these results, it was found that cold rolling with the cross-section reduction ratio of about 15% for each pass and intermediate heat treatment performed in the austenitic region at 1150ºC for 4 min followed by furnace cooling with a rate of 5ºC/min are proper to guarantee safe manufacturing for 10Cr-1Mo ODS steel. Based on the results, ODS plate was fabricated by a combination of rolling passes for reducing the thickness and softening heat treatments allowing a reduction of the material hardness.

A post-mortem analysis was performed to understand the microstructural evolutions in the course of manufacturing, which is essential to optimize the manufacturing route for 10Cr-1Mo ODS steel. Fig. 2 shows an optical micrograph and SEM image of the grain morphology for hot-extruded and homogenized 10Cr-1Mo ODS steel. The hot-extruded specimen, as depicted in Fig.2 (a), consists of martensite with fine equiaxed grains and a small portion of delta-ferrite with elongated grains along the hot rolling direction. Due to the slow cooling subsequent to the homogenization, a softened ferritic structure with a mean grain size varying from 3 to 5 μm was observed in the mother plate, as shown in Fig. 2(b).

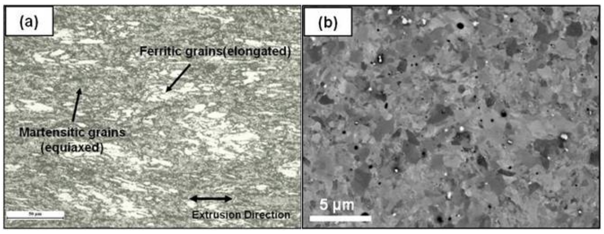

Fig. 2. OM and SEM images showing the grain morphology for (a) hot-extruded and (b) homogenized(mother plate) 10Cr-1Mo FM ODS steels.

In Fig. 3, an EBSD observation using a band contrast map for 10Cr-1Mo ODS steel was conducted to examine grain morphology evolution during the manufacturing process. It can be seen that the grains were relatively refined and elongated in the rolling direction, and a high fraction of red and blue regions representing <100> and <111> textures developed after about 15% cold working, as shown in Fig. 3(a). In Fig. 3(b), a high fraction of large ferrite grains was observed, and the grain sizes were 3-5 times bigger than the cold-rolled grains in Fig. 3(a). While the <111> textures partially remained, the <100> textures were significantly reduced, showing the reduction of the morphological anisotropy induced by cold rolling. Fig. 3(c) shows the grain morphology of the thin plate after final heat treatment which consists of normalizing at 1050ºC for 1 h, and was followed by tempering at 780ºC for 1 h. It was observed that the grain structure is quite homogeneous, indicating that the elongated grain structure resulting from the eighth cold rolling is ultimately replaced by an equiaxed grain. It is suggested that the final heat treatment can be effective at enhancing the mechanical properties through the equiaxed grains and improved isotropy.

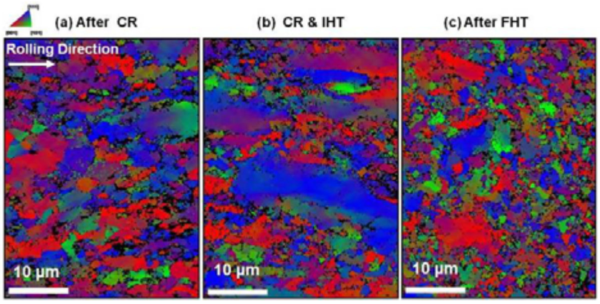

Fig. 3. EBSD images showing microstructure evolutions during the cold rolling and heat treatments for 10Cr-1Mo ODS steel.

Fig. 4 illustrates TEM images showing martensitic (Fig. 4(a)) and ferritic (Fig. 4(b)) structures of the thin plate after cold rolling with a reduction of 75% and a final heat treatment. It was found that the final ODS steel has a typical tempered martensite structure with very fine martensitic grains and a few ferritic grains.

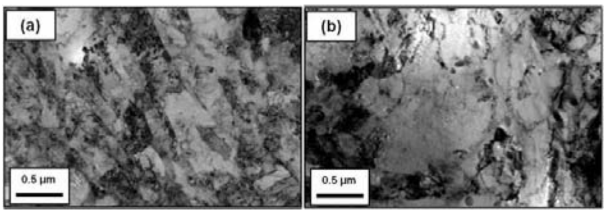

Fig. 4. TEM micrographs showing (a) martensitic and (b) ferritic structures of 10Cr-1Mo ODS steel after the cold rolling with a reduction of 75% and final heat treatment.

In Fig. 5, a TEM bright-field image of a nano-oxide particle distribution for 10Cr-1Mo ODS steel after completion of the manufacturing process is presented. It is clearly shown that fine oxide particles are homogeneously distributed in the matrix and that the sizes are 5-10 nm.

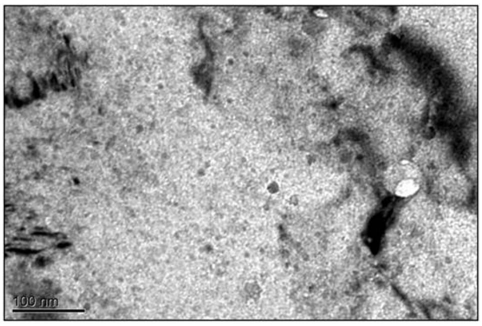

Fig. 5. TEM bright-field image showing nano-oxide particle distribution for 10Cr-1Mo ODS steel after the manufacturing process of thin plate.

Fig. 6 shows the tensile test results of the 10Cr-1Mo ODS steel in the presence of the manufacturing process at both temperatures, together with the corresponding results in the absence of the manufacturing route. The result demonstrates that there is not much difference in the tensile strength and elongation between the specimens for both temperatures. In the case of elevated temperature, the tensile strength value with the manufacturing remained higher while the elongation exhibited an adequate ductility, yet somewhat lower, compared to the values without the manufacturing. These results indicate that that the tensile property of the 10Cr-1Mo ODS steel remains robust and superior even after the manufacturing process.

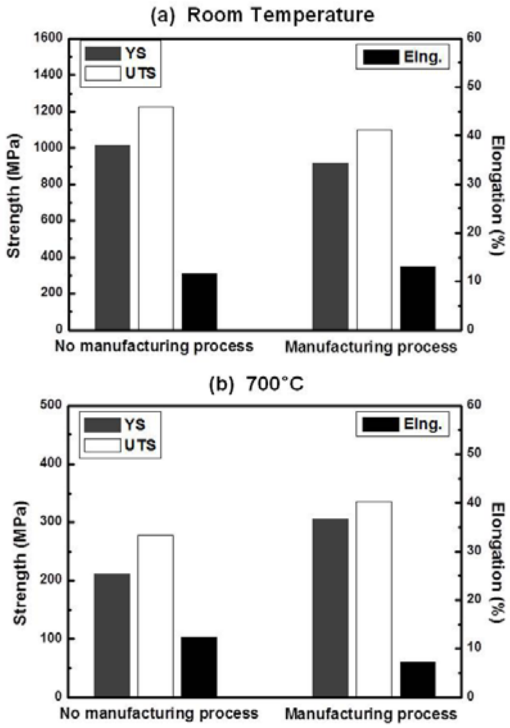

Fig. 6. Tensile properties of 10Cr-1Mo ODS steels in the absence and presence of manufacturing process at (a) RT and (b) 700°C.

4. Conclusions

In the present study, the effect of a cold rolling and intermediate heat treatments on microstructures and mechanical properties of 10Cr-1Mo FM ODS steel were investigated. During the manufacturing route the hardness measurements remained below the critical value of 400 Hv. Intermediate heat treatment with slow cooling led to a softened ferritic structures which can be further cold rolled. The final heat treatment consisting of a normalizing followed by air cooling and a tempering resulted in a homogeneous microstructure, leading to enhancing mechanical properties. While further study is needed, it is believed that these results can be useful in optimizing the manufacturing of advanced ODS steel.

ACKNOWLEDGEMENT

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