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Particle size effects in mechanically alloyed 9Cr ODS steel powder

H. Sakasegawa [a,*], S. Ohtsuka [a], S. Ukai [b], H. Tanigawa [c], M. Fujiwara [d], H. Ogiwara [c], A. Kohyama [e]

[a] *Oarai Research and Development Center, Japan Atomic Energy Agency, 4002 Narita, Oarai, Higashi-Ibaraki, Ibaraki 311-1393, Japan*
[b] *Department of Materials Science and Engineering, Hokkaido University, Kita 8 Nishi 5, Kita-ku, Sapporo-shi, Hokkaido 060-0808, Japan*
[c] *Naka Fusion Institute, Japan Atomic Energy Agency, 2-4 Shirakata, Tokai-mura, Ibaraki-ken 319-1195, Japan*
[d] *Kobelco Research Institute Inc., 1-5-5 Takatsudai, Nishi-ku, Kobe-shi, Hyogo-ken 651-2271, Japan*
[e] *Institute of Advanced Energy, Kyoto University, Gokasho, Uji-shi, Kyoto-fu 611-0011, Japan*

Abstract

The superior creep properties of 9Cr ODS (Oxide Dispersion Strengthened) steels result from small oxide particles dispersed in the matrix. Thus many investigations have studied the creep properties by focusing on the oxide particle distribution, but these results are not sufficient to clarify the creep mechanism and to develop material processing procedures for mass production. Potential microstructural features affecting the creep properties should be studied. The 9Cr ODS steel has many potential microstructural features, for example, PPB (Prior Particle Boundary) pores, precipitates, and two matrix phases. In particular, PPB pores link up and develop into large creep cavities. Therefore, particle size classification of mechanically alloying (MA) produced powders was performed to control PPB. Contrary to our expectations, the particle size classification did not only affect PPB creep cavities, but also other microstructural features. MA powder particle size effects on microstructural features and the creep properties were studied.

1. Introduction

The 9Cr Reduced Activation Ferritic/martensitic steels (RAFs) are leading candidates for blanket and first wall structures of fusion reactors, because of their high thermal conductivity, low thermal expansion coefficient, and low void swelling. In order to develop more attractive RAFs with improved creep properties, oxide dispersion strengthening is the most effective approach.

The 9Cr Oxide Dispersion Strengthened (ODS) steel has superior creep properties, because very small Y–Ti–O complex oxide particles dispersed in the matrix are obstacles to moving dislocations. Thus many studies have attempted to clarify the creep mechanism by focusing on oxide particles and dislocations using TEM (Transmission Electron Microscopy) observations. However, these results are insufficient to clarify the creep mechanism and to develop material processing procedures for mass production [1–6].

* Corresponding author. Tel.: +81 29 267 4141; fax: +81 29 267 7173.
E-mail address: sakasegawa.hideo@jaea.go.jp (H. Sakasegawa).

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The production of the 9Cr ODS steel involves many processes, including mechanical alloying (MA), degassing, canning, hot extrusion, and heat treatment. These procedures cause complicated structures, as shown in Fig. 1. The 9Cr ODS steel has many microstructural features which potentially affect the creep properties. These include pores on Prior Particle Boundaries (PPB) aligned parallel with the extrusion direction, intra/intergranular precipitates, oxide inclusions, and two matrix phases (tempered martensite matrix and δ-ferrite matrix), along with oxide particles and dislocations. These microstructural features are summarized in Table 1.

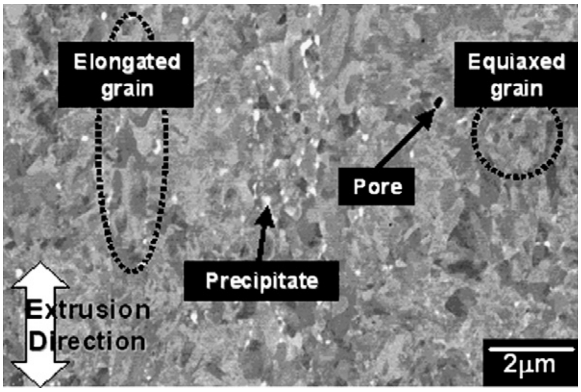

Fig. 1. Microstructures of the 9Cr ODS steel (NT).

In previous work [7], a potential microstructural feature, PPB, was studied using SEM (Scanning Electron Microscope), as shown in Fig. 2. PPB pores linked up and developed into large creep cavities. MA powder particle size classification was performed to control PPB and to study the PPB effect on the creep property. However, our recent studies on potential microstructural features reveal that MA powder particle size classification does not only affect PPB creep cavities, but also other microstructural features. MA powder particle size is suggested to be an important parameter for both clarifying the creep mechanism and developing material processing procedures for mass production.

In this work, MA powder particle size effects on microstructural features and the creep properties were studied.

2. Experimental procedure

The 9Cr ODS steel chemical composition, shown in Table 2, was prepared by mechanical alloying of argon-gas-atomized pre-alloyed metal powder along with Y_2O_3 powder in a high-energy attritor in an argon atmosphere for 48 h at 220 rpm. After that, MA powder particle size classification was performed. MA powders were divided into the large powder (>90 μm) and the small powder (<45 μm). These classified MA powders were then degassed

Table 1
Microstructural features of the 9Cr ODS steel

No.	Micro-metric		Nano-metric	Isotropy	Reports
1	PPB (MA powder particle surface)	–	–	○	[7]
2		Cavity	–	○	[7]
3		Precipitate	–	○	–
4	Elongated grain (δ-ferrite)	Matrix	–	–	[2,8,9]
5			Oxide particle	–	[2,8,9]
6			Dislocation	–	–
7			Precipitate	–	–
8		Boundary	–	○	–
9			Precipitate	○	–
10	Equiaxed grain	Matrix	–	–	[2,8,9]
11	(Tempered martensite)		Oxide particle	–	[2,8,9]
12			Dislocation	–	–
13			Precipitate	–	–
14		Boundary	–	–	–
15			Precipitate	–	–
16	Boundary	–	–	○	–
17		Precipitate	–	○	–
18	Creep cavity	–	–	○	[7]

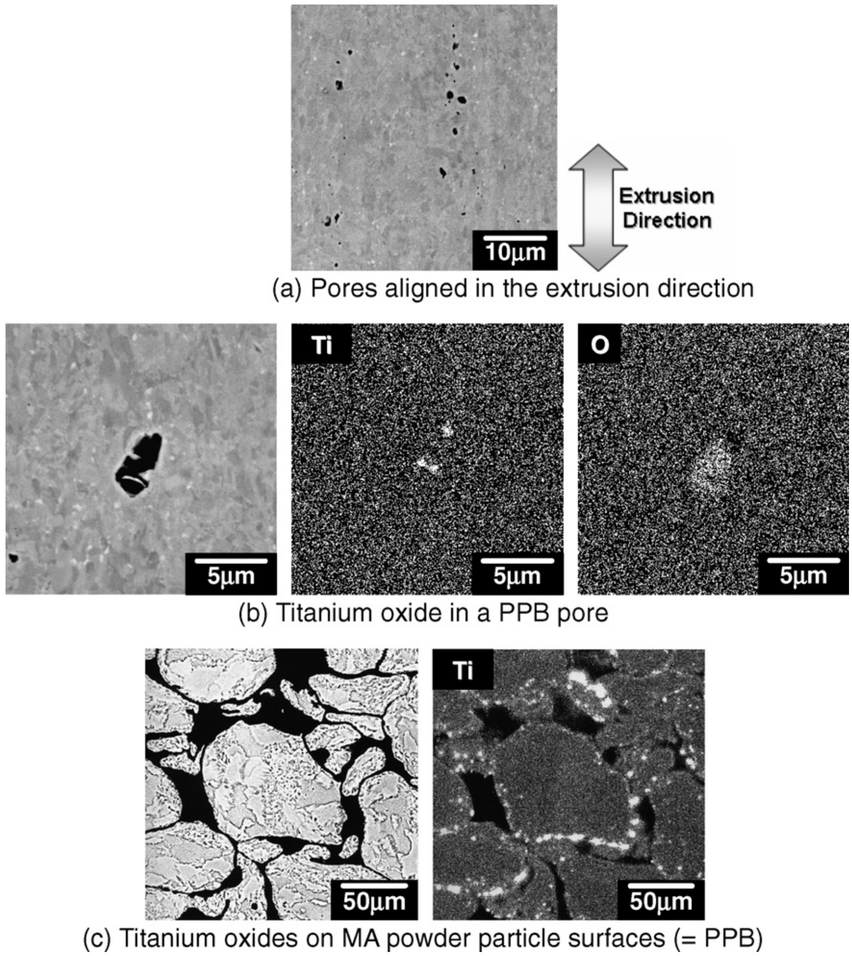

Fig. 2. Titanium oxides in PPB pores.

Table 2
Chemical compositions (wt%)

Element	Raw	Small	Large
Fe	88.3	88.3	88.2
C	0.14	0.15	0.14
Cr	8.67	8.88	9.07
W	1.96	1.89	1.94
Ti	0.23	0.22	0.22
Y	0.27	0.26	0.27
O	***0.18***	***0.20***	***0.17***
N	≈0.040	0.050	0.041
Al	–	0.017	0.017
Si	0.048	0.051	0.052

2 h at 400 °C in vacuum (0.1 Pa), after which they were canned in mild steel and hot extruded at 1200 °C into bar. Normalizing and tempering conditions were 1050 °C × 1 h/AC (Air Cooling) and 800 °C × 1 h/AC, respectively. Creep rupture tests were conducted at 700 °C in air. The gage length of the specimen used was 20 mm and the diameter of the circular cross section was 4 mm. The creep property data were for specimens taken in the extrusion direction.

3. Results and discussion

3.1. Properties of classified MA powders

The properties of classified MA powders are shown in Table 2 and in Fig. 3. No differences in phases were observed, but the oxygen content was decreased by using the larger size MA powder. The decrease in the oxygen content improves creep properties of the 9Cr ODS steels [8,9]. MA powder particle size classification is suggested to be an effective process to improve and to guarantee the creep properties.

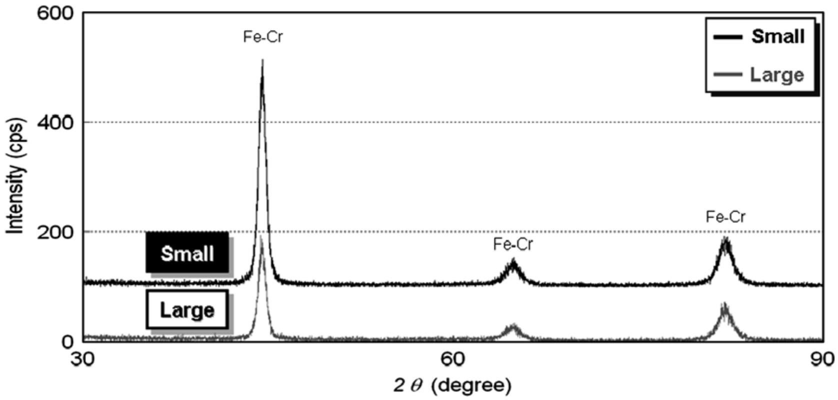

Fig. 3. Phases in classified MA powders.

3.2. PPB pores and creep cavities

Fig. 4 shows the effects of MA powder particle size classification on PPB pores and creep cavities. No differences in PPB pores were seen before the tests, but pronounced differences in creep cavities were observed after the tests. Creep cavities were formed on PPB. The effect of PPB creep cavities

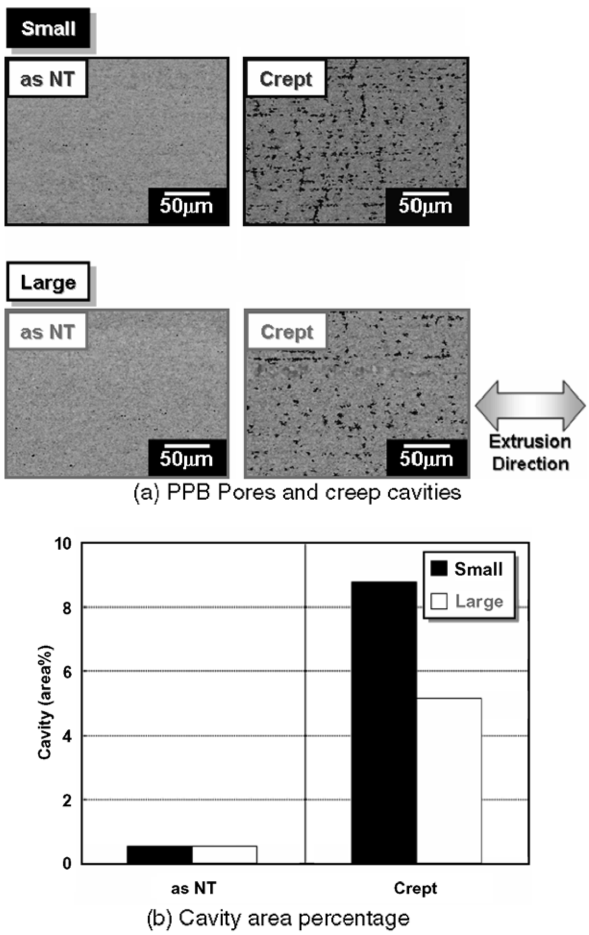

Fig. 4. Effects of particle size classification on creep cavities.

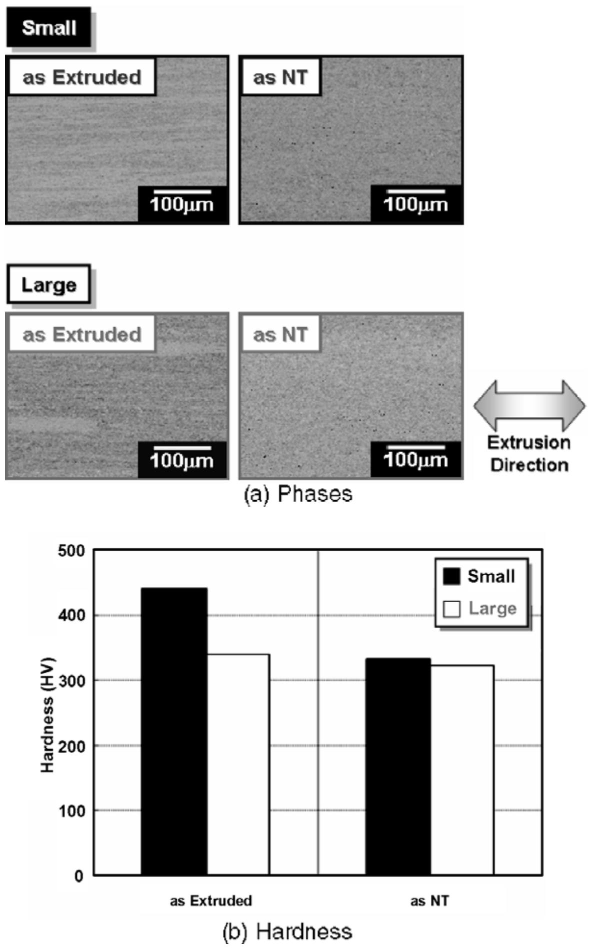

Fig. 5. Effects of particle size classification on matrixes.

on creep rupture propagation was studied in previous work [7].

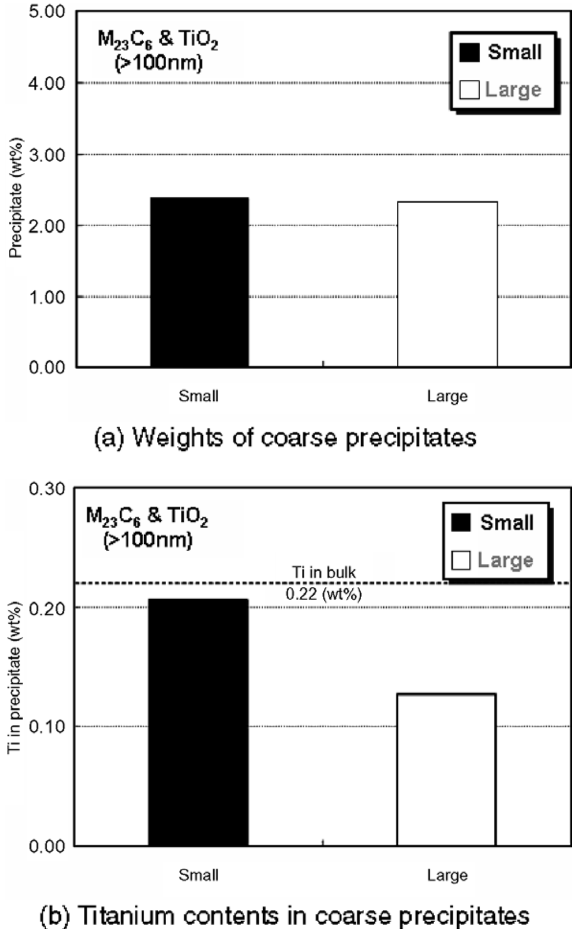

Fig. 6. Effects of particle size classification on precipitates.

3.3. Matrixes and precipitates

Fig. 5 shows the effects of MA powder particle size classification on the material matrix. The result of Vickers hardness tests suggests that little martensitic transformation took place when using the large MA powder, because a decrease in hardness was not produced by tempering.

This phenomenon caused by using the large MA powder can be interpreted by the titanium oxide morphologies on PPB, as shown in Fig. 2. Fig. 6 shows the result of precipitate analysis based on electrolytic extraction. Precipitates were $M_{23}C_6$ and TiO_2 obtained using a 100 nm pore size filter. These coarse precipitates cannot contribute to the dispersion hardening. No difference in precipitate weights was observed, but the titanium content in precipitates was reduced by using the large MA powder. The titanium content decrease in precipitates indicates that TiO_2 on PPB decreased and the titanium content in the matrix increased. The increase in the matrix titanium content leads to the conclusion that the limited martensitic transformation took place in the specimen consolidated using the large MA powder, because titanium is a ferrite-forming element.

Minor differences in hardness and grain size were observed between the two specimens. These results suggest that there was minor difference in the oxide particle dispersions between these specimens [3]. The hot extrusion temperature in this work (1200 °C) was probably so high that the difference in the titanium contents could not cause the difference in the oxide particle dispersions. The distribution of Y–Ti–O complex oxide particles is sensitive

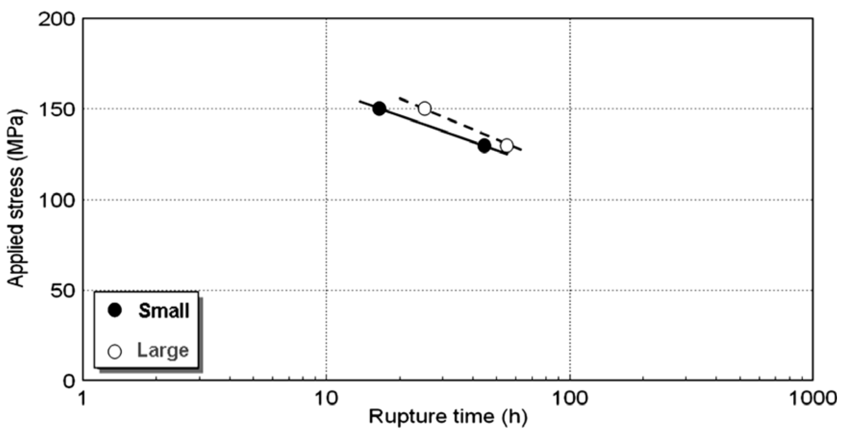

Fig. 7. Creep rupture test results at 700 °C.

to the hot extrusion condition. Detailed examinations should be performed in a future study on the hot extrusion condition using the large MA powders.

4. Creep properties

Fig. 7 shows the result of creep rupture tests conducted at 700 °C. The direction of applied stress is parallel to the extrusion direction. The creep rupture time increased when using the large MA powder. The improvement probably results from the decrease in PPB creep cavities when using the large powder, because only a minor difference in hardness was observed. In the case of cladding tubes for Fast Breeder Reactor, the direction of applied stress (hoop stress) is perpendicular to the extrusion direction. It seems that the creep rupture time difference between the two directions of stress would be larger than the result of this work, because PPB creep cavities are aligned in the direction perpendicular to the hoop stress.

5. Summary

In this work, MA powder particle size effects in the 9Cr ODS steel were studied.

Effects of using the large MA powder can be summarized as follows:

- The oxygen content decreases.
- The titanium content in the matrix increases.
- The martensitic transformation is inhibited.
- Creep cavities are reduced.
- The creep rupture time increases.

From the results, MA powder particle size is shown to be an important parameter for improving and guarantying the creep properties of the 9Cr ODS steel.

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