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Precipitation behavior of oxide particles in mechanically alloyed powder of oxide-dispersion-strengthened steel

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Abstract

The precipitation behavior of oxide particles in mechanically alloyed powder of oxide-dispersion-strengthened steel was studied using chemical extraction method and X-ray diffraction analysis. In particular, the temperature and time dependences of the precipitation behavior of Y–Ti–O oxide particles were investigated. The results suggest more appropriate hot extrusion conditions to finely disperse oxide particles, to improve, and to guarantee the creep property of oxide-dispersion-strengthened steel.

Keywords: Mechanical alloying; Oxide-dispersion-strengthened steel; Precipitation behavior; Creep property; Microstructural observation

1. Introduction

Oxide-dispersion-strengthened steels have attracted attention for advanced nuclear power plants applications such as fast and fusion reactors, because of their superior high temperature mechanical properties. 9Cr oxide-dispersion-strengthened steel (9Cr–2W–0.2Ti–0.34Y_2O_3) developed by Japan Atomic Energy Agency has superior creep property compared with conventional 9Cr heat resistant steels. The creep rupture strength (1000 h at 973 K) of 9Cr oxide-dispersion-strengthened steel is about three times greater than that of conventional 9Cr heat resistant steels [1–4], as shown in Fig. 1. The 9Cr oxide-dispersion-strengthened steel enormously contributes to practical applications of fast reactors and fusion reactors [5–7]. This steel has a high density of small Y–Ti–O oxide particles dispersed in the matrix, as shown in Fig. 2(a). These particles improve the creep property by pinning movable dislocations at elevated temperatures.

Mechanical alloying and powder metallurgy processes are applied to finely disperse these small oxide particles in the matrix, because oxide particles aggregate together and coarsen during conventional casting processes. The production of oxide-dispersion-strengthened steel involves many processes, such as mechanical alloying, degassing, canning, hot extrusion, and heat treatments.

In the procedures, the hot extrusion process strongly affects precipitation behavior of oxide particles and their dispersion. Precipitation behaviors of oxide particles in mechanically alloyed powder are studied in this investigation.

2. Experimental procedure

The material processing procedures for the preparation of oxide-dispersion-strengthened steel are schematically shown in Fig. 3. The chemical composition,

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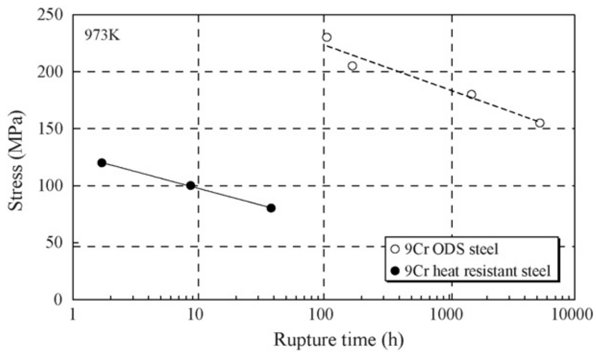

Fig. 1. Improved creep property of 9Cr oxide-dispersion-strengthened steel.

Table 1
Chemical composition of 9Cr oxide-dispersion-strengthened steel

	wt%
Fe	88.3
C	0.14
Cr	8.67
W	1.96
Ti	0.23
Y	0.27
O	0.18
N	≈0.040
Al	–
Si	0.048

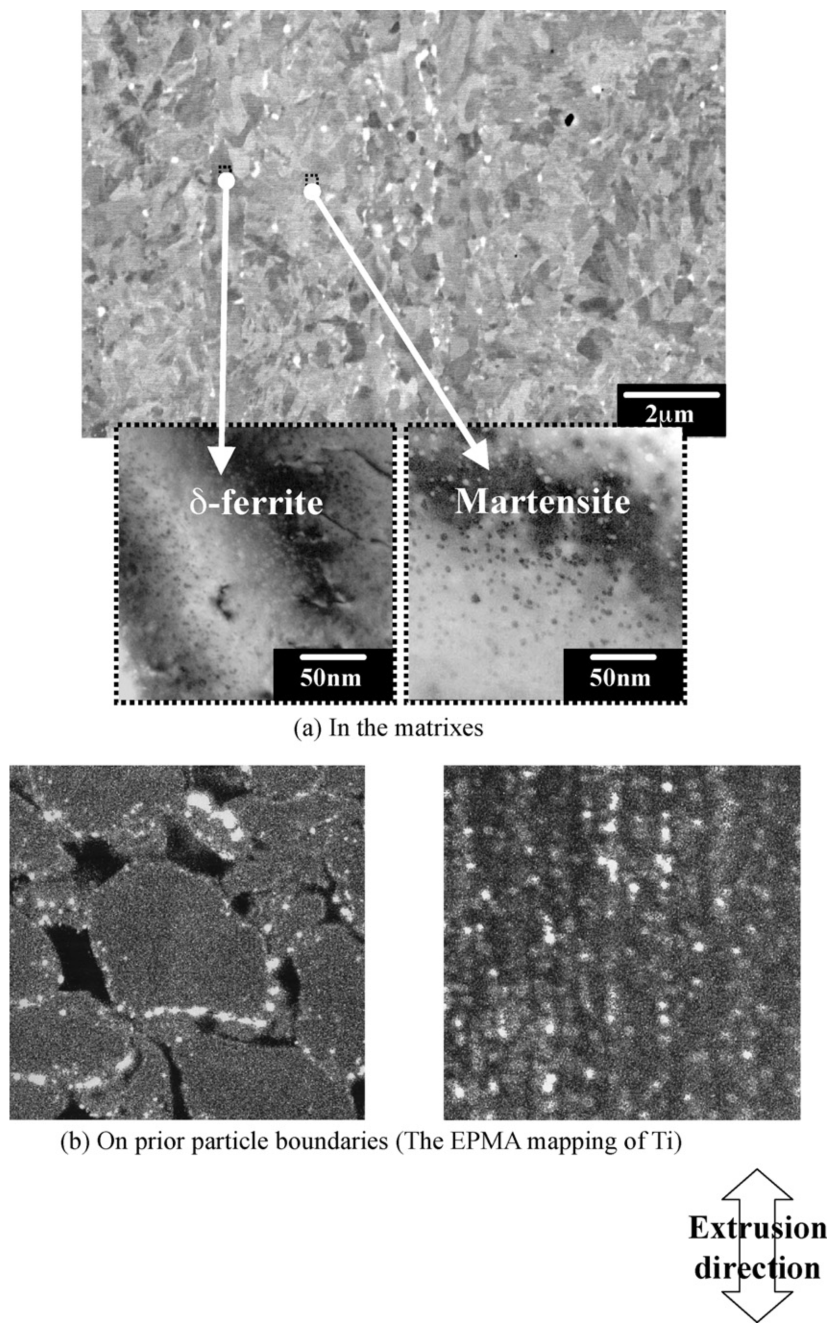

Fig. 2. Microstructures of 9Cr oxide-dispersion-strengthened steel.

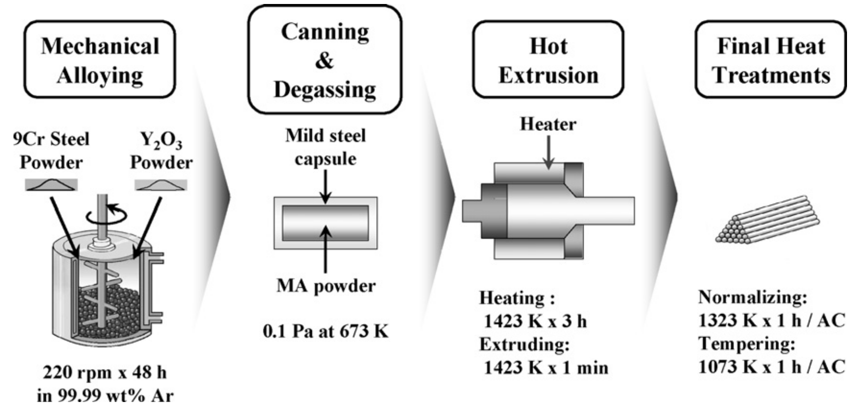

Fig. 3. Material processing procedures for the preparation of 9Cr oxide-dispersion-strengthened steel.

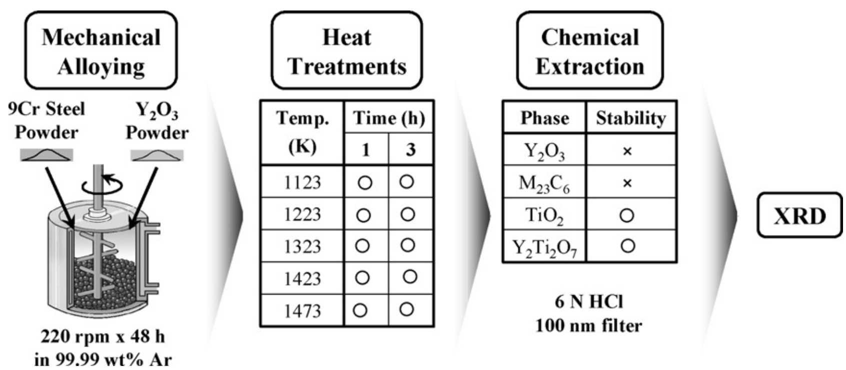

Fig. 4. Experimental procedures.

as shown in Table 1, is prepared by mechanical alloying of argon-gas-atomized pre-alloyed metal powder with Y_2O_3 powder in a high-energy attritor in an argon atmosphere for 48 h at 220 rpm. Mechanically alloyed powder is then degassed 2 h at 673 K in vacuum (0.1 Pa), after which it is canned in mild steel and hot extruded at 1423 K into bar. Normalizing and tempering conditions are 1323 K × 1 h/AC (air cooling) and 1073 K × 1 h/AC, respectively. Fig. 4 schematically shows experimental procedures. Mechanically alloyed powders were heat treated over the range from 1123 K to 1473 K. The holding times during the heat treatment were 1 and 3 h at each temperature. The chemical extraction was conducted using a 6 N HCl solution. In this condition, Y_2O_3 and $M_{23}C_6$ are unstable and dissolve, but TiO_2 and $Y_2Ti_2O_7$ are stable. Y_2O_3 particles are not observed by all the TEM (transmission electron microscope) observations of 9Cr oxide-dispersion-strengthened steel [1–4]. Noises caused by $M_{23}C_6$ can be rejected in XRD (X-ray diffraction) analysis. The pore size of filter used for the vacuum percolation was 100 nm. The weights of extracted residues were measured with an accuracy of ±0.1 mg. Extracted residues were analyzed by XRD with Cu Kα radiation source at 40 kV and 20 mA.

3. Result and discussion

Fig. 5 shows the weight percentages of oxide particles. They increased with increasing temperature, but no time dependence was observed at all the temperatures. At temperatures above 1423 K, the weight percentage was probably saturated.

The phases of oxide particles analyzed by XRD are shown in Fig. 6. Anatase type TiO_2 particles were observed at all the temperatures. They were very stable at elevated temperatures, because they had no temperature and time dependences. In the results of SEM and TEM observations on 9Cr oxide-dispersion-strengthened steel microstructures (Fig. 2), TiO_2 particles did not contribute to the improvement of the creep property, because their particle sizes were too large. Most of them did not precipitate in the matrix but on prior particle boundaries, as shown in Fig. 2(b) [8].

On the other hand, $Yi_2Ti_2O_7$ particles were only observed at temperature above 1323 K. They had temperature and time dependences. Each of peak intensities increased with increasing time. Since the weight percentages of the oxide particles had no time dependences, the XRD analysis suggests that $Yi_2Ti_2O_7$ particles aggregated together and their sizes became large by

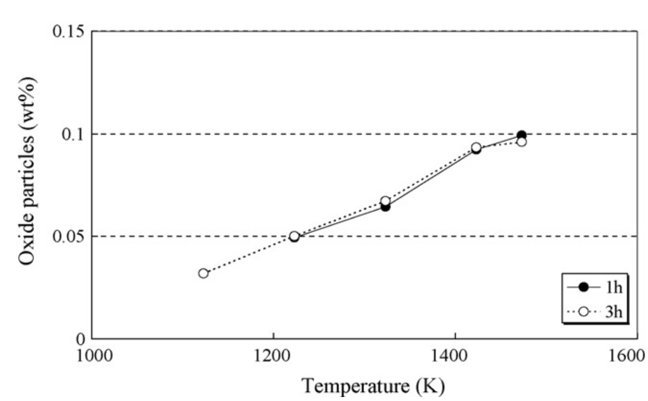

Fig. 5. Weight percentages of oxide particles in mechanically alloyed powders after the heat treatments.

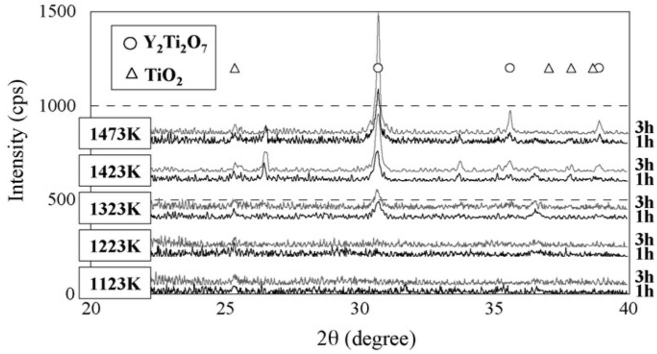

Fig. 6. Phases of oxide particles in mechanically alloyed powders after the heat treatments.

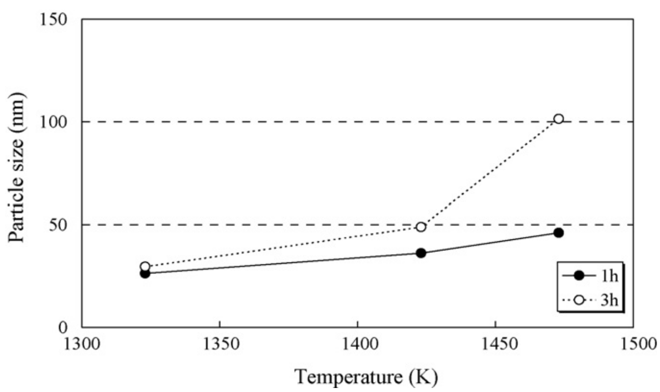

Fig. 7. Particle sizes of $Y_2Ti_2O_7$.

holding at elevated temperatures. These changes in particle size are shown in Fig. 7. The results were calculated using the Scherrer formula. These particle sizes had temperature and time dependences. The calculated sizes were relatively larger than the sizes observed by TEM and SANS (small-angle neutron scattering) analysis. At the conditions of the chemical extraction method and the vacuum percolation, small oxide particles were not observed. The threshold strength values calculated from the results are underestimated, because observed particle sizes are relatively large. These calculated values are estimated on the side of prudence. Thus, calculated results give available information to determine more appropriate hot extrusion conditions.

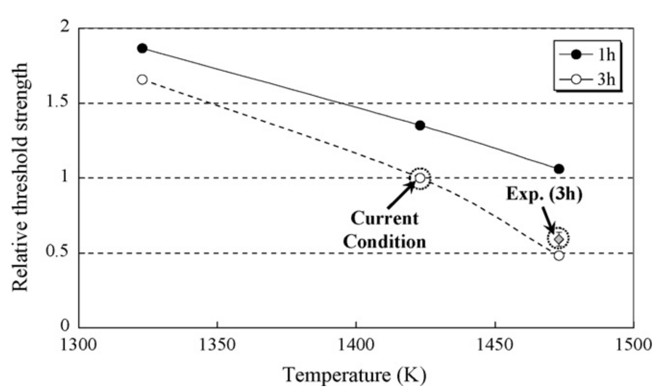

Fig. 8. Estimation of hot extrusion condition effect on threshold strength.

Table 2
Appropriate hot extrusion conditions for improving the creep property of 9Cr oxide-dispersion-strengthened steel

Condition	Creep strength	Workability	Sinterability
1423 K × 1 h	↑	→	→
1473 K × 1 h	→	↑	↑

Estimated threshold strengths are shown in Fig. 8 calculated by the Orowan/Srolovitz mechanism [9,10]. An experimental result is also plotted. The experimental result was obtained from the data of the creep rupture strength (1000 h at 973 K), because the creep property is strongly affected by the threshold strength. In the figure, little difference was observed between estimated and experimental results. The little difference confirmed that threshold strength values estimated by the chemical extraction results give available information of more appropriate hot extrusion conditions. From the results, lower temperature conditions are favorable to fine dispersion of $Y_2Ti_2O_7$ particles. However, these conditions degrade workability and sinterability. Lower temperature conditions should be determined by considering both workability and sinterability. Based on these considerations, Table 2 gives available hot extrusion conditions for the current material processing.

4. Summary

In this work, precipitation behaviors of oxide particles in mechanically alloyed powders were studied by using the electrolytic extraction method in order to improve and guarantee the creep property of 9Cr oxide-dispersion-strengthened steel. The temperature and time dependence of precipitation behaviors of oxide particles were studied.

- The formation of $Y_2Ti_2O_7$ was observed over 1323 K.
- The weight percentage of $Y_2Ti_2O_7$ had no time dependence.
- The size of $Y_2Ti_2O_7$ increased with increasing time and temperature.
- Calculated relative threshold strength indicated a way to modify the hot extrusion condition.

From the result of this work, more appropriate hot extrusion conditions were determined for the current material processing. The hot extrusion conditions will be applied and investigated in a future work.

References

[1] S. Ukai, M. Fujiwara, J. Nucl. Sci. Technol. 39 (2002) 778–788.
[2] S. Ukai, T. Kaito, S. Ohtsuka, T. Narita, M. Fujiwara, T. Kobayashi, ISIJ Int. 43 (2003) 2038–2045.
[3] S. Ohtsuka, S. Ukai, M. Fujiwara, T. Kaito, T. Narita, J. Nucl. Mater. 329–333 (2004) 372–376.
[4] S. Ohtsuka, S. Ukai, M. Fujiwara, T. Kaito, T. Narita, Mater. Trans. 46 (2005) 487–492.
[5] R.L. Klueh, J.P. Shingledecker, R.W. Swindeman, D.T. Hoelzer, J. Nucl. Mater. 341 (2005) 103–114.

[6] M.J. Alinger, G.R. Odette, D.T. Hoelzer, J. Nucl. Mater. 329–333 (2004) 382–386.
[7] H. Kishimoto, M.J. Alinger, G.R. Odette, T. Yamamoto, J. Nucl. Mater. 329–333 (2004) 369–371.
[8] H. Sakasegawa, S. Ohtsuka, S. Ukai, H. Tanigawa, M. Fujiwara, H. Ogiwara, A. Kohyama, Fus. Eng. Des. 81 (2006) 1013–1018.
[9] J.W. Martin, Micromechanisms in Particle-Hardened Alloys, Cambridge University, 1980.
[10] A.J.E. Foremann, M.J. Makin, Phil. Mag. A14 (1966) 911–924.