Effect of Dispersoids on Tensile Deformation of Fe–20Cr ODS Alloys

Yozo KAWASAKI, Yuji IKEDA, Toshiharu KOBAYASHI and Hideshi SUMIYOSHI

National Research Institute for Metals, Sengen, Tsukuba, Ibaraki-ken, 305 Japan.

(Received on February 13, 1996; accepted in final form on July 15, 1996)

Tensile deformation properties of nine kinds of mechanically alloyed Fe–20Cr-based oxide dispersion strengthened (ODS) steels were examined over a temperature range from 300 to 1073 K. One of the specimens used was an alloy without any addition of oxide and the others were eight kinds of ODS alloys with the addition of Y_2O_3 , Al_2O_3 , SiO or ZrO_2 . All of the alloys had fine grain structures. With the addition of Y_2O_3 the 0.2% yield stress of Fe–20Cr alloys increased all over the temperature range, and the work-hardening following the yield at 300 and 673 K also increased considerably. But at higher temperatures than 673 K, the increment of work hardening due to the oxide dispersion was small. Fracture elongation at 673 K was found to be about 5%, while those at 873 and 1073 K were more than 17%. The dispersion strengthening due to the other oxides, Al_2O_3 , SiO and ZrO_2 were very small except that of ZrO_2 at 1073 K.

KEY WORDS: Fe–20Cr ODS alloys; Y_2O_3 ; Al_2O_3 ; SiO and ZrO_2 oxides; tensile deformation properties.

1. Introduction

One of authors found that the addition of rare earth metals (REM) into austenitic steels and Y_2O_3 into superalloys caused trapping of sulfur atoms at the interfaces between matrices and oxide particles, and that these trapping suppressed the segregation of sulfur on the specimen surface and in consequence suppressed the spalling of Al_2O_3 coating layer.²⁾ In the previous work,²⁾ the effect of REM and Y_2O_3 addition on the materials properties was examined in mechanically alloyed (MA) Fe–20Cr ODS ferritic steels, and it was found that, in contrast with austenitic steels and superalloys, rapid segregation of sulfur occurred on the surfaces of ODS ferritic alloys but the spalling of Al_2O_3 coating films being little. This result shows one of the excellent characteristics of ferritic MA ODS.

The MA 956 ferritic steel with the composition of Fe–20Cr–4.5Al–0.5Ti–0.5 Y_2O_3 , has various interesting properties, particularly the excellent resistance against oxidation and hot corrosion, and hence is used for combustion chamber.³⁾ The tensile strength of this steel at high temperatures, 1385 K for example, increases with increasing strain rate, while the ductility in low strain rates is small.⁴⁾ The ODS ferritic steel is also promising for the fuel cladding of FBR (Fast Breeder Reactor) in Japan, because of their good resistance to swelling and of excellent strength at elevated temperatures. Okuda *et al.* found that Fe–13Cr MA ODS (Y_2O_3) ferritic steel was strengthened further by adding Titanium and Molybdenum and that swelling was suppressed by adding Titanium.⁵⁾

The excellent properties of MA 956 and Fe–13Cr MA ODS have been found to connect closely with the dispersion of Y_2O_3 fine particles. However, the roles of

other oxide particles on the materials properties of ferritic MA ODS have not been systematically investigated yet. This work is aimed to study the effects of various dispersoids on the tensile properties of Fe–20Cr-based ODS alloys. For this purpose the same mechanically alloyed specimens as those prepared for the previous work²⁾ were used; an Fe–20Cr steel without addition of oxide and eight kinds of Fe–20Cr-based ODS alloys containing Y_2O_3 , Al_2O_3 , SiO or ZrO_2 . Those are listed in Table 1.

2. Experimental Procedure

The chemical compositions of the nine kinds of Fe–20Cr-based alloys are shown in Table 1, where the symbol FC–00 means Fe–20Cr alloy without addition of oxide, and FC–3Y for example, is the alloy with addition of nominal 0.3% Y_2O_3 ; FC–YA is the alloy only to which two kinds of oxides, nominal 0.5% Y_2O_3 and 0.3% Al_2O_3 are added. The amount of each oxide was calculated from the analytically determined amount of metal element making oxide, and the residual amount of oxygen, $re(O)$, is the rest value in subtracting the amount of oxygen in the form of oxides from the analytical total amount of oxygen, $an(O)$. Table 1 shows that the amount of residual oxygen in the alloys containing Y_2O_3 is much higher than that in FC–00 alloy, and is much lower in the other alloys. Y_2O_3 in FC–YA alloy may form some complex oxides like $3Y_2O_3-5Al_2O_3$ ⁶⁾ and also complex oxides of Fe and/or Cr may be formed in FC–00 alloy with 0.34%O and strengthen it. However, complex oxides were not taken into account for the calculation of the amount of oxides given in Table 1.

The alloys were manufactured by INCO Ltd. Raw powder of the constituent elements for each alloy was

Table 1. Analytical chemical compositions of Fe-20Cr ODS alloys. (mass%)

Alloy	Cr	Y(Y ₂ O ₃)	Al(Al ₂ O ₃)	Si(SiO)	Zr(ZrO ₂)	an(O)	re(O)*	vol% of oxide
FC-00	19.08					0.34	0.34	
FC-3Y	19.21	0.23 (0.29)				0.61	0.55	0.7
FC-5Y	19.31	0.30 (0.38)				0.67	0.59	1.0
FC-8Y	18.91	0.45 (0.63)				0.79	0.61	1.6
FC-11Y	18.94	0.77 (0.98)				0.74	0.53	2.5
FC-YA	19.12	0.55 (0.70)	0.16 (0.30)			0.78	0.49	2.6
FC-5A	18.95		0.28 (0.53)			0.40	0.15	1.0
EC-6S	20.70			0.40 (0.63)		0.51	0.29	3.2
FC-12Z	19.28				0.57 (0.77)	0.52	0.32	1.7

Impurity; Cu; 0.03 / Mn; 0.07 / P; 0.003 / S; 0.006 / C; 0.04 / N; 0.30.

* re(O) = an(O) - oxide(O).

Table 2. Grain sizes of Fe-20Cr ODS alloys.

Alloy	Cross sec. μm (st. dev.)	Longi. sec. μm (st. dev.)	Grain aspect ratio; d_1/d_c
FC-00	4.3 (0.4)	5.6 (0.4)	1.3
FC-3Y	5.1 (0.9)	6.4 (0.6)	1.25
FC-5Y	4.4 (0.3)	6.6 (0.7)	1.5
FC-8Y	4.1 (0.3)	8.1 (0.9)	1.97
FC-11Y	4.4 (0.4)	5.7 (0.4)	1.3
FC-YA*	4.0 (0.7)	—	—
FC-5A	8.7 (0.8)	9.2 (0.8)	1.05
FC-6S	10.3 (1.0)	12.5 (1.0)	1.21
FC-12Z*	3.8 (0.8)	—	—

* TEM photographs.

mechanically alloyed by an attritor for 86.4 ks (24 h), put in an evacuated iron can and extruded at 1338 K in a form of plate included iron sheath with 50 mm width and 13 mm thickness. The plate was first heat treated at 1473 K for 7.2 ks (2 h) in Ar atmosphere. The specimens for tensile test were made by cutting from the plate parallel to extrusion direction followed by shaping with an NC turning lathe. Each specimen had a shape of 50 mm total length, with 8 mm phi thick screws on both tops to pull, 4 mm phi × 16 mm gauge length part, and 5 mm radius shoulder parts. The specimens were polished finally with emery papers of #2000, and heat-treated again at 1473 K for 7.2 ks (2 h) in Ar atmosphere.

Tensile tests were carried out at 300, 673, 873 and 1073 K with a strain rate $5 \times 10^{-3} \text{ s}^{-1}$. The temperature 1073 K corresponds to 60% the melting point 1786 K of Fe-20Cr alloy. Formation of fiber textures in the alloys due to the extrusion was examined with an X-ray diffractometer (Rigaku-RAD-IIB), while the crystalline microstructures were observed with optical microscopy after etching the alloys at 300 K in a solution of 30 g NH₄F, 50 cc HNO₃ and 20 cc water. Distribution of oxide particles and dislocation structures were observed with transmission electron microscopy (TEM) employing JEOL-2000; thin plates were cut perpendicular to the tensile direction from fractured specimens, and thinned electrolytically at 260 K in an etchant solution of ethanol and 10% perchloric acid.

Fig. 1. X-ray diffraction patterns of the transversal section plane of various alloys.

Fig. 2. TEM microstructures of FC-11Y and -5A alloys.

3. Results and Discussion

3.1. Microstructures

Table 2 shows the averaged grain sizes that were measured lengthwise and breadthwise from those optical micrographs, as shown in Fig. 4, which were taken on the longitudinal side sections of fractured specimens. The grain sizes of FC-YA and -12Z alloy specimens, however, were estimated from their TEM photographs because it was difficult to etch either chemically or by ion-sputtering.⁷⁾ The grain sizes of both FC-00 and Y₂O₃ containing alloys were 4–5 micron meters and those of

both FC-5A and -6S alloys were 8–10 micron meters. The aspect ratio of grain was at most only 2; this might be caused by the reason that the extrusion was carried out at a temperature range that recrystallization did not occur.

X-ray diffraction patterns for the transversal cross-sections of the specimens are shown in Fig. 1. The highest and next highest peaks were 110 and 220, respectively, having the ratio of about 1:0.12, and other 200 and 211 peaks were very low. This shows that all of the textures in the nine alloys have strong preferred orientation for 110.

Fig. 3. Tensile stress vs. elongation curves, (a) FC-00, -3Y and -5Y alloys at 300 and 673 K, (b) at 873 and 1073 K, (c) FC-8Y, -11Y and -YA alloys at 300 and 673 K, (d) at 873 and 1073 K, (e) FC-5A, -6S and -12Z alloys at 300 and 673 K, (f) at 873 and 1073 K.

Fig. 4. Optical microstructures of the longitudinal side section of various alloy specimens fractured at 300 K.

Fig. 5. Optical microstructures of the longitudinal side section of specimens, FC-00, -3Y and -12Z alloys, fractured at 673, 873 or 1073 K.

Microstructural distribution of dispersoids in FC-11Y and -5A alloys are shown in Figs. 2(a) and 2(b), where the mean radii of Y_2O_3 and Al_2O_3 are 50 and 95 nm, respectively. The radius of Y_2O_3 in other alloys containing Y_2O_3 agreed roughly with that shown in Figs. 2(a). The radius of SiO in FC-6S alloy was about 93 nm, being similar with that of Al_2O_3 in Fig. 2(b). The radius of ZrO_2 in FC-12Z alloy was approximately 40 nm. The volume fraction in percentage of Y_2O_3 in FC-11Y alloy was 2.5 and the average spacing between the oxide particles was about 5 times as large as the average radius. In all of the eight alloys with oxide particles, the dispersion of the particles was found to be non-uniform, as shown in Figs. 2(a) and 2(b) for the two alloys. This may show the shortage of time even in the mechanical alloying for 86.4 ks (24 h) by the attritor.

3.2. Tensile Deformation

Figures 3(a)–3(f) show tensile stress vs. elongation curves of the alloys at various temperatures, where the tensile stress is defined as the load per area of initial cross-section and the elongation is defined as also the length per the initial gauge-length. At 300 K the tensile stresses of FC-00, -5Y, -11Y, -YA and -6S alloys increased monotonously with the increase in elongation until fracture, while those of the other alloys decreased in large elongation and then fractured. The fracture elongation of FC-11Y alloy was relatively small; this

was confirmed by repeating the tensile test. At 673 K the tensile stresses of alloys except FC-6S and -12Z monotonously increased until fracture with increasing elongation, while those of FC-6S and -12Z alloys decreased in large elongation showing relatively large fracture elongation. At both 873 and 1073 K the tensile stresses in all of the alloys increased at the beginning, reached maxima, and then decreased until fracture, showing large fracture elongation.

Figures 4(a)–4(f) show the optical micrographs of longitudinal-side section of various alloy specimens fractured at 300 K. At this temperature, all of the specimens except for FC-12Z showed transgranular fracture perpendicular to the tensile direction with small elongation. Figure 5 shows the photographs of longitudinal-side section of FC-00, -3Y and -12Z alloy specimens fractured at 673, 873 or 1073 K. At 673 K, they fractured along the plane with an angle of 45 degree to the tensile direction; this will corresponds to the fracture with small elongation. While at 873 and 1073 K they fractured with forming cups and cones, corresponding to large fracture elongation as shown in Fig. 8.

Figures 6(a) and 6(b) show 0.2% yield stresses vs.

(a)

(b)

Fig. 6. 0.2% yield stress vs. inverse of temperature (K), (a) FC-00, -3Y, -5Y, -8Y and -11Y alloys, (b) FC-00, -YA, -5A, -6S and -12Z alloys.

(a)

(b)

Fig. 7. Difference between maximal tensile stress and 0.2% yield stress vs. temperature, (a) FC-00, -3Y, -5Y, -8Y and -11Y alloys, (b) FC-00, -YA, -5A, -6S and -12Z alloys.

inverse of temperature (K). Y_2O_3 containing alloys were stronger than FC-00 over the temperature range. The increase of 0.2% yield stresses, however, was not proportional to Y_2O_3 content, presumably because of non-uniform dispersion of Y_2O_3 particles. Other oxide particles, in contrast with Y_2O_3 , did not strengthen FC-20Cr alloy over the temperature range except ZrO_2 at 1073 K, but 0.2% yield stress of FC-00 at 300 K was even a little higher than those of FC-5A, -6S and -12Z. This is probably because that high residual oxygen content in FC-00 alloy strengthen itself, by forming some kind of oxide particle. At about 1073 K, each stress-elongation curves showed a steep slope at the 0.2% yield stress. However, the role of oxide particles on the slope is little effective, because the curve for FC-00 alloy also showed a similar steep slope; instead, it is supposedly attributable to vacancy defects which take important part in the deformation at so high temperature as 1073 K. If this is true, the gradient of the slope have a relation with the inverse of temperature in thermal activation process.

Figures 7(a) and 7(b) show the difference between maximal tensile stress and 0.2% yield stress vs. temperature; this difference is a measure for work-hardening. The values of the difference at 300 K for FC-5Y and -8Y alloys were about 300 MPa and much larger than the value 120 MPa for FC-00 alloy. This fact shows that Y_2O_3 particles play an important role in

work-hardening.

Figures 8(a) and 8(b) show the fracture elongation of the alloys at various temperatures. The curves for the alloys except FC-5Y, -11Y and -YA seem to have minima around 673 K, and these minima accorded to the fracture along the planes with an angle of 45 degree to the tensile direction. This fracture with minimal elongation seems to be due to the so-called 748 K (475°C) brittleness of FC-20Cr alloy at 748 K, causing from spinodal decomposition. In passing, Read and Murakami⁸⁾ have recently investigated on aging behavior of MA 956 using APFIM technique, and found that Cr concentration fluctuation took place on a microscopical scale of 10 nm in accompany with the increase of the hardness of the alloy.

Figure 9 shows TEM microstructures of alloy fractured at various temperatures. In Fig. 9(a), very dense dislocations along complex slip lines were observed and corresponded to the high strength of FC-3Y. At 300 K, highly dense dislocations were also formed in the other alloys. At 673 K, in Fig. 9(b), dislocation arrangements with high density were formed while substructures were not observed. At 873, in Fig. 9(c), indistinct dislocation substructures were formed and the average size was about 0.3 micrometer. At 1073 K, in Fig. 9(d), the substructures were clearly formed and the average size was 0.9 micrometer. The temperature dependence of dislocation arrangements in all of the alloys examined was similar with what shown in Figs. 9(a)–9(d), though the figures are for various alloys.

4. Conclusions

Tensile properties of Fe-20Cr alloy and Fe-20Cr-based oxide dispersion alloys with Y_2O_3 , Al_2O_3 , SiO or ZrO_2 oxides were examined over a temperature range from 300 to 1073 K. The alloys were manufactured by the method of mechanical alloying. Following results were obtained with respect to the effects of oxide dispersoids on tensile properties of the alloys.

(1) 0.2% yield stress of Fe-20Cr-based alloys is enhanced by Y_2O_3 particles over the testing temperature range. The enhancement due to the particles at high temperatures is small, presumably because the effect of original fine grain size prevails.

(2) Y_2O_3 particles cause the large work-hardening of the Fe-20Cr-based alloys at 300 K.

(3) Fracture elongations of the alloys except FC-6S and -12Z, at 300 and 673 K are about 5%, and seem to show minima at about 673 K. The fracture elongations of the alloys except FC-8Y, at 873 and 1073 K are larger than 17%.

(4) Strengthening effect of Al_2O_3 , SiO and ZrO_2 oxide particles on the Fe-20Cr-based alloys is generally little, except that of ZrO_2 at 1073 K.

Acknowledgments

The authors are very thankful to Dr. S. Yamamoto for setting up NC lathe several times, and to Mr. H. Tanaka for etching the alloy specimens by ion sputtering. They are also indebted to Dr. K. Yagisawa for his valuable discussion on the manuscript.

(a)

(b)

Fig. 8. Fracture elongation at various temperatures, (a) FC-00, -3Y, -5Y, -8Y and -11Y alloys, (b) FC-00, -YA, -5A, 6S and -12Z alloys.

Fig. 9. TEM microstructures in the transversal cross section of fractured specimens, (a) FC-3Y alloy at 300 K, (b) FC-YA at 673 K, (c) FC-00 at 873 K and (d) FC-5Y at 1073 K.

REFERENCES

- 1) Y. Ikeda, M. Tosa, K. Yoshihara and K. Nii: *ISIJ Int.*, **29** (1989), 966.
- 2) Y. Ikeda, H. Sumiyoshi and Y. Ishiwata: *ISIJ Int.*, **35** (1995), 1109.
- 3) INCO Alloy Intern. Inc.: Product Handbook, Publication No. IAI-38, (1988), 27.
- 4) J. D. Whittenberger: *Metall. Trans.*, **9A** (1978), 101.
- 5) T. Okuda, S. S. Nomura, M. Nakanishi, M. Fujiwara and H. Takahashi: Proc. 1st Japan Int. SAMPE Sym., Chiba Japan, 28, Dec., 1 (1989), 1616.
- 6) H. S. Ubhi, T. A. Hughes and J. Nutting: *Frontiers of High Temperature Materials*, ed. by J. S. Benjamin, Inco MAP, New York, (1981), 79.
- 7) H. Tanaka, F. Abe, K. Yagi, E. Nishikawa and T. Sugita: *CAMP-ISIJ*, **8** (1995), No. 6, S366.
- 8) H. Read and H. Murakami: *Appl. Surf. Sci.*, **94/95** (1996), 334.