Effect of Tungsten on Mechanical Properties of High-Purity 60 mass%Cr–Fe Alloys

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The effect of the tungsten addition on the mechanical properties of high-purity 60 mass%Cr–Fe alloy was investigated. High-purity 60 mass%Cr–Fe–4 mass%W alloy ingots were prepared by melting in a newly designed high-frequency induction furnace equipped with a cold copper crucible. The purity of the ingots was higher than 99.98 mass% after the analysis of 21 elements. The deformability at temperatures of 1273 to 1573 K and the tensile properties between 293 K and 1073 K were measured by Gleeble test and tensile test. It was found that the tensile strength of 60 mass%Cr–Fe–4 mass%W alloy is higher than that of 60 mass%Cr–Fe alloy, and the addition of tungsten is effective in improving tensile properties of a 60 mass%Cr–Fe alloy.

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1. Introduction

A high-chromium iron alloy has desirable properties such as high strength, corrosion resistance and oxidation resistance at elevated temperatures.¹⁾ However, the ductility and the deformability of Cr–Fe alloys decrease with the increment of chromium content.²⁾ In commercial Fe–Cr alloys, the maximum Cr content is 30 mass% to keep good ductility.

Recently, Abiko and co-researchers reported that the ductility of Cr–Fe alloys containing 50 or 60 mass%Cr was remarkably improved by purification. Abiko and Kato reported that high-purity 50%Cr–Fe alloy has high strength with high deformability and several desirable physical properties for high-temperature service such as low thermal expansion coefficient and high thermal conductivity in comparison with type 304 stainless steel.³⁾ Kano *et al.* reported that the tensile strength and the hot ductility of high-purity 60%Cr–Fe alloy were higher than that of high-purity 50%Cr–Fe alloy.⁴⁾ On the other hand, purification generally causes a decrease in strength at elevated temperatures and a decrease in recrystallization temperature due to the reduction of impurities. Isozaki *et al.* found that the addition of tungsten to high purity 50 mass%Cr–Fe alloys was effective in improving strength and ductility at high temperatures.^{5,6)} Kako *et al.* reported that the amount of twinning increased with the addition of tungsten.⁷⁾

The purposes of the present research are to prepare a high-purity 60 mass%Cr–Fe–4 mass%W alloy and to investigate the effect of the tungsten addition to high-purity 60 mass%Cr–Fe alloy on the high temperature mechanical properties such as tensile strength and elongation.

2. Experimental Procedure

2.1 Preparation of high-purity 60 mass%Cr–Fe–4 mass%W alloy ingots

Two ingots of high-purity 60Cr–Fe–4W alloy were prepared from chromium of 99.98 mass% purity, electrolytic iron of 99.995 mass% purity, and tungsten of 99.999 mass% purity by melting under an argon atmosphere of 4×10^4 Pa, in

a newly designed high-frequency induction furnace equipped with a water-cooled copper crucible.⁸⁾ Before the melting, the chamber was baked and the raw materials were heated in an ultra-high vacuum of 2×10^{-7} Pa to remove gaseous impurities adsorbed on their surfaces.

Table 1 shows the chemical composition of two ingots referred to as 60Cr–Fe–4W (I) and 60Cr–Fe–4W (II). The chemical composition of high-purity 60 mass%Cr–Fe alloy used by Kano *et al.* is also shown in the table.⁴⁾ The total amounts of gaseous impurities such as carbon, nitrogen, oxygen, and sulfur in 60Cr–Fe–4W (I) and 60Cr–Fe–4W (II) are 53.5 and 49.3 mass ppm, respectively. Though the addition of tungsten to 60Cr–Fe alloy sometimes causes the increase in the detection limit of chemical analysis for several impurity elements, the total purity of the 60Cr–Fe–4W ingots can be considered to be higher than 99.98 mass% and almost the same as that of 60Cr–Fe alloy.

2.2 Preparation of specimens

The ingots were hot-forged and hot-rolled after heating at 1473 K in a high purity argon atmosphere. The microstructures of the ingots was destroyed by hot-forging to obtain uniform grain size. The rods 15 mm square formed by hot-forging were hot-rolled to rods 7 mm in diameter. For Gleeble test, specimens 6.4 mm in diameter and 100 mm in length were made from the rods by machining. They were annealed at 1523 K for 900 s under a vacuum of 3×10^{-2} Pa and quenched into oil cooled to 273 K. For tensile test, specimens 20 mm in gauge length and 3 mm in diameter were machined from the same rods. They were annealed at 1273 K for 1.8 ks under a vacuum of 3×10^{-2} Pa and quenched into oil cooled to 273 K.

2.3 Gleeble test

The high-temperature tensile strength and the reduction in area were measured by a Gleeble unit,⁹⁾ which is a high-temperature testing machine. The Gleeble test was carried out at a strain rate of 10^{-1} s at temperatures between 1273 K and 1573 K. The microstructure of the fractured specimens was also observed with an optical microscope.

Table 1 Chemical composition of high-purity Cr-Fe and Cr-W-Fe ingots.

Ingot	Element (mass%)		Gaseous impurity (mass ppm)					Metallic impurity (mass ppm)			
	Cr	W	C	N	O	S	total	Al	B	Co	Cu
60Cr-4W-Fe (I)	59.8	4.13	12.9	7.3	27.0	6.3	54	<10	0.62	<20	3.0
60Cr-4W-Fe (II)	61.0	3.40	11.9	7.6	23.6	6.2	49	<10	0.54	< 1	2.0
60Cr-Fe (Kano <i>et al.</i> ⁴⁾)	60.8	—	14.7	5.3	32.6	5.3	58	3.0	0.54	0.6	5.7

Ingot	Metallic impurity (mass ppm)										
	Ga	Mn	Mo	Nb	Ni	P	Si	Sn	Ti	V	Zn
60Cr-4W-Fe (I)	4.1	<1	<20	<20	<15	6	31	5.6	<3	<20	<15
60Cr-4W-Fe (II)	4.3	<1	< 3	< 3	<15	5.8	36	6.1	<3	< 1	<15
60Cr-Fe (Kano <i>et al.</i> ⁴⁾)	3.8	2.96	1.2	0.24	8.3	5.2	20	4.8	2.5	1.19	0.6

2.4 Tensile test

In order to clarify the mechanical properties such as yield stress, tensile strength, elongation, and reduction in area, tensile test was performed at an initial strain rate of $4.2 \times 10^{-4} \text{ s}^{-1}$ at temperatures between 293 K and 1073 K under a high vacuum of $6 \times 10^{-4} \text{ Pa}$. The microstructure of the fractured specimens was observed with an optical microscope.

3. Results and Discussion

3.1 Effect of W on high temperature strength and hot ductility

Figure 1 shows the tensile strength and the reduction in area of 60Cr-Fe-4W (I) after Gleeble testing at temperatures between 1273 K and 1573 K, together with the data of 50Cr-Fe and 60Cr-Fe.^{3,4)} The data of 50Cr-Fe-5W and 50Cr-Fe-8W alloys⁵⁾ were also shown in the same Figure. It was found from the present investigation that the tensile strength of 60Cr-Fe increases by the addition of tungsten at temperatures between 1273 K and 1573 K. The reduction in area of 50Cr-Fe increases by the addition of tungsten; however, the

reduction in area of 50Cr-Fe-8W is lower than that of 50Cr-Fe-5W. This means that the alloy has the most suitable content of tungsten for the highest reduction in area. On the other hand, the reduction in area of 60Cr-Fe-4W (I) is lower than that of 60Cr-Fe. The addition of 4 mass%W may be unsuitable in 60Cr-Fe alloy to obtain the highest reduction in area at high temperature.

Macrostructures of the specimens of 60Cr-Fe-4W (I) fractured by Gleeble testing at temperatures between 1273 K and 1573 K, and microstructures of the fractured parts are shown in Fig. 2 and Fig. 3, respectively. The ductility of 60Cr-Fe-4W (I) increases with increasing temperature. Small recrystallized grains are observed in the specimen tested at 1473 K and 1573 K, but no recrystallized grain at 1373 K. Kano *et al.* reported that small recrystallized grains in 60Cr-Fe were recognized in the specimen tested at 1373 K.⁴⁾ It means that tungsten addition increases the recrystallization temperature. Figures 4 and 5 show the microstructures of the fractured parts at 1573 K of 60Cr-Fe-4W (I) and 60Cr-Fe, respectively. At the necking part, the wavy microstructures along the elongated direction are observed in Figs. 3(b), (c), (d)

Fig. 1 Tensile strength and reduction in area of Cr-Fe-W and Cr-Fe alloys measured by Gleeble testing.

and Fig. 4(b). The size of the recrystallized grains of 60Cr-Fe-4W (I) is smaller than that of 60Cr-Fe. From these microstructure observation, it can be emphasized that the addition of tungsten is effective in the increase of the recrystallization temperature which causes the increase of the tensile strength of 60Cr-Fe alloy at high temperature.

3.2 Effect of W on tensile properties and microstructure between 293 K and 1073 K

Figure 6 shows the stress-strain curves of 60Cr-Fe-4W (II) at temperatures between 293 K and 1073 K. Figure 7 shows

Fig. 2 Photographs of the specimens of 60Cr-Fe-4W (I) fractured by Gleeble testing at (a) 1273 K, (b) 1373 K, (c) 1473 K and (d) 1573 K.

Fig. 3 Microstructures of the specimens of 60Cr-Fe 4W (I) fractured by Gleeble testing at (a) 1273 K, (b) 1373 K, (c) 1473 K and (d) 1573 K.

Fig. 4 Microstructures of the fracture part of 60Cr-Fe-4W (I) after Gleeble testing at 1573 K (a) and of a high magnification photograph (b).

Fig. 5 Microstructures of the fracture part of 60Cr-Fe after Gleeble testing at 1573 K (a) and the high-magnification photograph (b).

Fig. 6 Stress-strain curves of 60Cr-Fe-4W(II) alloy at the temperatures between 293 K and 1073 K.

the tensile strength and the yield stress, the elongation and the reduction in area of 60Cr-Fe-4W (II) and 60Cr-Fe as a function of temperature between 293 K and 1073 K. The data of 60Cr-Fe are also plotted by open symbols.⁴⁾ During the test at 293 K, a few audible sounds happened at the end of the elastic deformation; the sounds are caused by twinning. Indeed, deformation twins are observed at many grains in the microstructure of the specimen tested at 293 K as shown in Fig. 8.

Serrations in the stress-strain curves are observed between 673 K and 823 K in Fig. 6. Figure 9 shows the microstructures of the fractured specimens tested at 673 K and 773 K. The traces of deformation twins are observed in each specimen. The amount of the deformation twins at 773 K is larger than that at 673 K. The same phenomena in 60Cr-Fe was also

Fig. 7 Tensile properties dependence of 60Cr-Fe-4W (II) and 60Cr-Fe alloys in the temperature range between 293 K and 1073 K. (TS: tensile strength, YS: yield stress, EL: elongation, RA: reduction in area).

Fig. 8 Microstructures of the fracture part of 60Cr-Fe-4W (II) after tensile testing at 293 K (a) and the high-magnification photograph (b).

Fig. 9 Microstructures of the fracture part of 60Cr-Fe-4W (II) after tensile testing at (a) 673 K and (b) 773 K, and of high-magnification photographs (a') and (b').

reported by Kano *et al.*⁴⁾ It is thought that the deformation mechanism of 60Cr-Fe-4W between 673 K and 773 K is the same as that of 60Cr-Fe; that is, the serration at 673 K is due to deformation twinning and dislocation slip which associates with the Portevin-Le Chatelier effect, but the serrations at 723 K and 773 K are due to deformation twinning.

In the stress-strain curves between 873 K and 1073 K in Fig. 6, the nominal stress gradually decreases until fracture

after the stress drop at the end of the elastic deformation. Isozaki *et al.* and Kano *et al.* discussed the deformation mechanism of high-purity 50Cr-Fe and 60Cr-Fe between 873 K and 1073 K, respectively,^{4,5)} that is, the stress drop after yield is due to grain boundary sliding. The gradual stress decreases after the stress drop is due to uniform dislocation slip through the specimen. The deformation mechanism of 60Cr-Fe-4W between 873 K and 1073 K is thought to be the same as those of 50Cr-Fe and 60Cr-Fe.

By the addition of tungsten to 60Cr-Fe, the tensile strength and the yield increase at temperatures between 293 K and 1073 K. Tungsten in 60Cr-Fe alloy gives no effect on the elongation and the reduction in area between 293 K and 873 K, however, addition of tungsten to 60Cr-Fe is very effective for the elongation and the reduction in area at the temperature between 923 K and 1073 K, as seen in Fig. 6 and Fig. 7. Indeed, the elongation of 60Cr-Fe-4W is nearly 90% at 1073 K. There are no continuity between the two values of tensile strength and reduction in area measured by tensile test at 1073 K (Fig. 7) and by Gleeble test at 1273 K (Fig. 1). This discontinuity is thought to be due to the difference in strain rate between the two kinds of testing. Compared with Gleeble test which is a testing at high strain rate, tensile strength of the alloy is low and reduction in area of the alloy is high by tensile test. It is thought that a deformation of 60Cr-Fe-4W alloy is greatly influence by strain rate.

Figure 10 shows the microstructures of fractured specimens of 60Cr-Fe-4W (II) and 60Cr-Fe at 1073 K. Both specimens were fractured in the intergranular mode. There are many cavities near grain boundaries, especially at the fractured part on comparison between these two microstructures, however, the amount of cavities along grain boundaries considerably decreases by the addition of tungsten. Isozaki *et al.* suggested that in 50Cr-Fe the decrease in the amount of cavities is due to on increase of the grain-boundary cohesion by the addition of tungsten.⁶⁾ On the same way, it is thought that the addition

Fig. 10 Microstructures of (a) 60Cr-Fe-4W (II) and (b) 60Cr-Fe alloys fractured at 1073 K, and these high-magnification photographs (a') and (b').

of tungsten to 60Cr-Fe also increases the grain-boundary cohesion of 60Cr-Fe and decreases the amount of cavities along grain boundaries, thus improving the tensile properties.

4. Conclusions

High-purity 60 mass%Cr-Fe-4 mass%W alloys of 99.98% purity were prepared from high purity chromium, electrolytic iron, and tungsten using a cold-copper-crucible furnace newly designed, and the effect of W on their high-temperature mechanical properties was investigated.

The main results are as follows:

- (1) The addition of tungsten to 60Cr-Fe increases the tensile strength in the temperature range between 293 K and 1573 K.
- (2) The addition of tungsten to 60Cr-Fe increases the elongation and the reduction in area in the temperature range between 923 K and 1073 K in the tensile test.
- (3) The addition of tungsten is effective in restricting the nucleation of cavities during high temperature deformation, which is due to an increase in the grain boundary cohesion.

Thus we conclude that the addition of tungsten is quite useful for improving tensile properties of 60 mass%Cr-Fe alloy.

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