Intermediate-temperature brittleness of a ferritic 17Cr stainless steel

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A ferritic 17Cr stainless steel was tensilely tested at high temperatures and an intermediatetemperature brittleness, which showed a low reduction of area, was observed. The brittleness occurred near 810 to 970 K. The intermediate-temperature brittleness shifted to higher temperatures at higher tensile strain rates.

1. Introduction

Stresses concentrate at triple points and ledges of grain boundaries during grain boundary sliding. It is reported that the stress concentration makes voids form on grain boundaries, and that intermediate-temperature brittleness occurs [1-4]. Intermediate-temperature brittleness has been reported for steels and non-ferrous metals [5–9], but this brittleness has not yet been reported in ferritic chromium stainless steels. In this study, the authors try to detect this phenomenon in a ferritic 17Cr stainless steel.

2. Materials and experimental procedures

Electrolytic iron and chromium were melted in a vacuum high-frequency induction melting furnace, and cast as an ingot. The ingot was homogeneously annealed at 1223 K for 6 h. The chemical composition of the ingot is shown in Table I. Hot forging and hot cross-section rolling were performed at 1123 K. Bars were then drawn at room temperature to 2.0 mm diameter. The drawn wire was cut into 50 mm lengths and annealed. Annealing temperatures and times, and mean grain diameters measured by the intercept method, of the annealed ferritic 17Cr stainless steel are shown in Table II. All tensile wire specimens were heated at 1573 K for 1 h and quenched in water, then heated to the tensile test temperature and kept at this temperature for 30 min; a tensile test was then performed in an Instron-type tensile test machine which could apply a constant conventional strain rate.

3. Experimental results and discussion

Fig. 1 shows true stress-true strain curves of specimens of a mean grain diameter d = 0.6 mm tensilely tested at the indicated temperatures and tensile strain rates, where the tensile strain rate is the conventional

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one $\dot{\epsilon} = (l - l_0)/l_0 \times t$, where l_0 is the initial gauge length and l the gauge length after tensile deformation in the tensile deformation time t. As seen in Fig. 1, the elongation to fracture at a constant strain rate decreases and shows a minimum, then increases with the test temperature. The temperature of the minimum elongation to fracture shifts to a higher value at higher tensile strain rates.

Fig. 2 shows the relation between the reduction of area and the tensile test temperature. The reduction of area is obtained from $(F_0 - F)/F_0 = \phi$ where F_0 is the initial cross-sectional area of the specimen and F the cross-sectional area after fracture. The minimum of the reduction of area in Fig. 2 corresponds to the intermediate-temperature brittleness.

Fig. 3 shows the relation between the reciprocal of the temperature of the minimum reduction of area $T_{\rm min}$ and the logarithm of the tensile strain rate. There is an Arrhenius relation between them, and the two lines cross at 923 K. At higher temperatures the activation energy is 712 kJ mol⁻¹, and it is 310 kJ mol⁻¹ at low temperatures. The activation energy for the volume diffusion of chromium in the Fe–6.8 wt % Cr alloy is 310 kJ mol⁻¹ [10]. The low-temperature linear part seems to correspond to this volume diffusion. At higher temperatures the tensile strain rates are faster than $10^{-3} \sec^{-1}$, so that diffusion cannot control the deformation.

Fig. 4 shows the relation between the reduction of area and the tensile test temperature for specimens having different average grain sizes. The temperature showing the minimum reduction of area is almost the same on different grain-size specimens when the tensile strain rate is constant.

The strain-rate and temperature dependencies of high-temperature deformation can be expressed by the Zener-Holloman parameter $Z = \varepsilon \exp(Q/RT)$ where Q is the activation energy for high-temperature deformation, R the gas constant and T the deformation



Figure 1 True stress-true strain curves of ferritic 17Cr stainless steel elongated at high temperatures at various tensile strain rates, $\dot{\epsilon}$ (sec⁻¹): (a) 1.43 × 10⁻¹, (b) 1.43 × 10⁻², (c) 1.43 × 10⁻³, (d) 1.43 × 10⁻⁴, (e) 1.43 × 10⁻⁵.

Figure 1 Continued. 500 773K 400 823K True stress (MPa) 300 848K 873K 200 898K 100 923K 973K 0 5 10 25 30 15 20 (d) True strain (%) 400 773K True stress (MPa) 300 823K 838K 200 873K 100 0 5 10 15 20 (e) True strain (%) 40 Reduction of area, ϕ (%) 30 20 7 1573K 1h Test temp. X O 30min 10 Figure 2 Relation between reduction of area and tensile test temperature at various tensile t strain rates. Specimen grain size: d = 0.60 mm. $\dot{\epsilon}$ (sec⁻¹): (\bullet) 1.43 × 10⁻⁵, (\bigcirc) 1.43 × 10⁻⁴, (\triangle) 1.43 × 10⁻³, (\blacktriangle) 1.43 × 10⁻², (\Box) 1.43 670 770 870 1170 1270 970 1070

 $\times 10^{-1}$.

Test temperature (K)

TABLE I Chemical composition of specimen (wt %)

Cr	С	S	Р	Si	Mn
16.70	0.008	0.006	0.003	0.01	0.02

TABLE II Heat treatment and grain size

Heat treatment	Mean grain diameter (mm)		
1573 K, 1.5 h annealing	0.60		
1373 K, 1.0 h annealing	0.16		
1273 K, 1.0 h annealing	0.06		



temperature. Fig. 5 shows the relations between the Zener-Holloman parameter and the reduction of area. All the experimental data lie on one curve. The intermediate-temperature brittleness appears near $Z = 10^{14}$ to 10^{15} at lower temperatures and $Z = 10^{36}$ to 10^{38} at higher temperatures.

According to previous research, there are three causes of intermediate-temperature brittleness: (i) impurity segregation and precipitation on grain boundaries [11, 12], (ii) void formation on and near grain boundaries [1–4], and (iii) deformation in grains [9]. In high-nickel alloys their small amount of sulphur makes the alloys brittle, but in iron-chromium

Figure 3 Arrhenius plot of temperature of minimum reduction of area against tensile strain rate for d = 0.60 mm; $\dot{\varepsilon} = A \exp(-Q/RT_{\min}).$



Figure 4 Influence of grain size on intermediate-temperature brittleness. Strain rate: $1.43 \times 10^{-3} \text{ sec}^{-1}$. d (mm): (•) 0.06, (□) 0.16, (▲) 0.60.

Figure 5 Relation between Holloman parameter and reduction of area; grain size d = 0.60 mm. (a) Low-temperature range (T < 923 K), (b) high-temperature range (T > 923 K). $\dot{\varepsilon}$ (sec⁻¹): (\bigcirc) 1.43×10^{-5} , (\blacksquare) 1.43×10^{-4} , (\triangle) 1.43 $\times 10^{-3}$, (**A**) 1.43×10^{-2} , (**D**) 1.43 $\times 10^{-1}$.

Reduction of area, ϕ (%)





Figure 6 Optical microstructure of specimen fractured at 820 K at strain rate $\dot{\varepsilon} = 1.43 \times 10^{-3} \text{ sec}^{-1}$; grain size $d = 160 \,\mu\text{m}$.

alloys it generally does not influence the intermediatetemperature brittleness.

Voids on grain boundaries are observed with an optical microscope in specimens fractured at intermediate temperatures, as seen in Fig. 6. If flow stresses have a reverse temperature dependency in which the flow stress increases with deformation temperature in the intermediate-temperature brittleness range, there is a possibility that grain deformation leads to brittleness [9], but no reverse temperature dependency of the flow stress is seen in Fig. 1. Void formation on grain boundaries might therefore be responsible for the intermediate-temperature brittleness.

4. Conclusion

Intermediate-temperature brittleness is observed in a ferritic 17Cr stainless steel. This brittleness temperature shifts to a higher value under higher strain rates.

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