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# Influence of thermal histories on intermediate temperature embrittlement of an Fe–17Cr alloy

DONG-SHENG SUN
*Metallic Materials Central Laboratory, Shandong Polytechnic University, 33 Jing Shi Road, Jinan, People's Republic of China*

T. YAMANE, K. HIRAO
*Department of Materials Science and Engineering, Osaka University, 2-1 Yamadaoka, Suita, Osaka 565, Japan*

Intermediate temperature embrittlement is found in an Fe–17Cr alloy. The embrittlement is due to void formation on and near grain boundaries during tensile deformation. On the other hand, the segregation of phosphorus and sulphur impurity atoms to grain boundaries enhances intermediate temperature embrittlement.

## 1. Introduction

It is reported that a ferritic Fe–Cr steel has no intermediate temperature embrittlement [1]. We have, however, found it during tensile tests at constant strain rates [2]. Previously [3–5], two causes of the intermediate temperature embrittlement have been reported. One is the formation of voids and cavities. The voids and cavities do not migrate with grain boundaries. The other is the segregation of impurity atoms to grain boundaries. We have reported the effect of void formation on the intermediate temperature embrittlement of the Fe–17Cr alloy[2]. In this paper, in order to know the effect of the impurity atom segregation to grain boundaries on the intermediate temperature embrittlement, two thermal histories are given for the Fe–17Cr alloy. One gives the impurity atom segregation to grain boundaries, and another does not. The intermediate temperature embrittlement is then measured by tensile tests.

## 2. Experimental procedure

Electrolytic iron and chromium were melted and cast in a vacuum ($10^{-4}$ torr). The chemical composition of the ingot is shown in Table I. The ingot was homogenized at 1223 K for $6 \times 3600$ s and hot-forged, then section rolled to a bar 10 mm in diameter. The Fe–17Cr bar was drawn to 2.0 mm in diameter at room temperature. The wire was cut into length of about 50 mm as tensile specimens. The tensile specimens were given two kinds of thermal histories. One is that the specimens were heated at 1573 K for 3600 s for homogenization, and quenched in water, then heated to tensile test temperatures for 1800 s (heat treatment a). Another is that the specimens were heated at 1573 K for 3600 s for homogenization, and quenched in water, then heated at 1123 K for 1800 s for the impurity atom segregation to grain boundaries. After the segregation treatment, the specimens were kept at tensile test temperature for 1800 s (heat treatment b), tensile tests were then performed at constant strain rates.

## 3. Experimental results and discussion

Fig. 1 shows the relation between the fracture reduction of area $[(A_0-A)/A_0] \times 100$ and tensile test temperatures, where $A_0$ is the initial cross-section area of specimens and $A$ the fracture cross section area. The specimens were given heat treatment a, and their average grain size measured by intercept method, is 0.6 mm. The intermediate temperature embrittlement is observed at the temperature range of 750 to 1050 K, and it shifts to a higher temperature at a higher strain rate. Fig. 2 shows the fracture reduction of area of the specimens of the grain boundary segregation heat treatment b. These specimens also have intermediate temperature embrittlement. The embrittlement temperatures are, however lower than those of the specimens of heat treatment a, and the shift of the embrittlement to a higher temperature by a faster tensile strain rate, is not clear, compared with Fig. 1.

Fig. 3a and b shows the fracture surface of the specimen tensile tested at 873 K and the strain rate of $1.43 \times 10^{-4}$ s$^{-1}$ after heat treatment a. It is easily understood that the ductile fracture occurs from the magnified fractography under this tensile condition. The fracture surface of the specimens tensile tested at 773 K and the strain rate of $1.43 \times 10^{-3}$ s$^{-1}$ after heat treatment b, is seen in Fig. 4a and b. This tensile test condition is in the range of the intermediate temperature embrittlement. 70% of the fracure surface is a cleavage fracture area where voids are seen.

The same fracture surface in Figs 3 and 4, shows spatter by argon ions, and qualitative amounts of spattered phosphorus and sulphur ions are measured by an ion mass analyser. The spattered areas are all

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TABLE I Chemical composition of Fe–17Cr alloy (mass %)

| Cr | C | S | P | Si | Mn | Fe |
|---|---|---|---|---|---|---|
| 16.70 | 0.008 | 0.006 | 0.003 | 0.01 | 0.02 | balance |

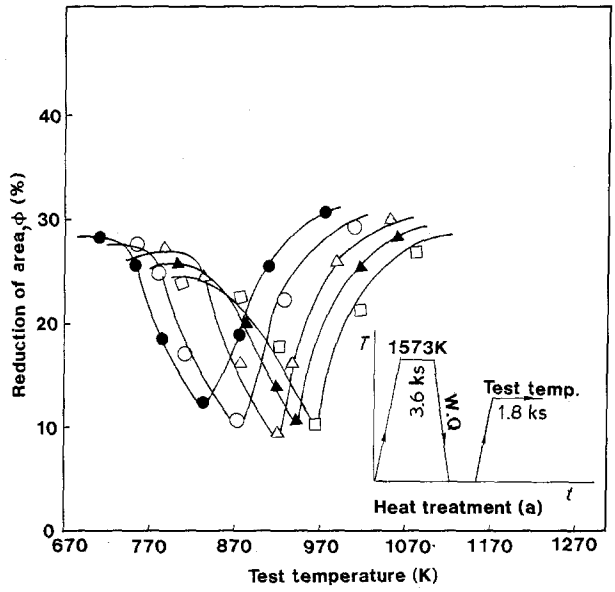


*Figure 1* Changes in fracture reduction of area of specimens after heat treatment a. (● $\dot{\varepsilon}=1.43\times10^{-5}\,s^{-1}$, ○ $\dot{\varepsilon}=1.43\times10^{-4}\,s^{-1}$, △ $\dot{\varepsilon}=1.43\times10^{-3}\,s^{-1}$, ▲ $\dot{\varepsilon}=1.43\times10^{-2}\,s^{-1}$, □ $\dot{\varepsilon}=1.43\times10^{-1}\,s^{-1}$, $d=0.60$ mm.)

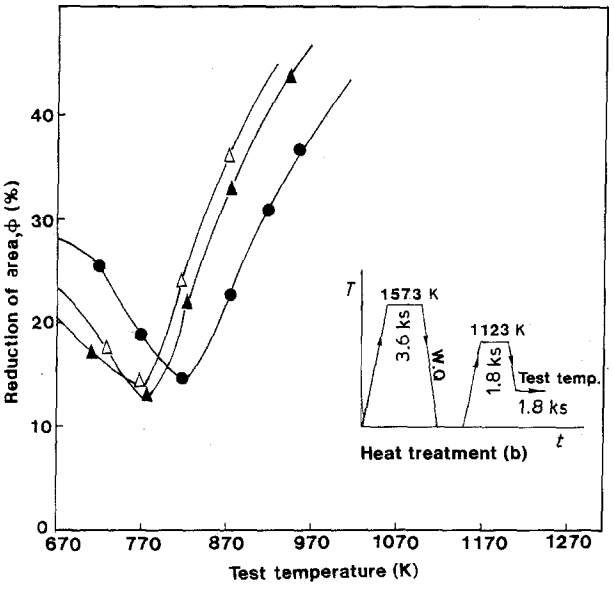


*Figure 2* Changes in fracture reduction of area of specimens after heat treatment b. (△ $\dot{\varepsilon}=1.43\times10^{-3}\,s^{-1}$, ▲ $\dot{\varepsilon}=1.43\times10^{-2}\,s^{-1}$, ● $\dot{\varepsilon}=1.43\times10^{-1}\,s^{-1}$, $d=0.60$ mm).

fracture surface of the specimens. Fig. 5 shows qualitative phosphorus and sulphur contents in the specimens fractured. The phosphorus and sulphur contents near the fracture surface of the specimen shown in Fig. 3, are lower than those of the specimen shown in Fig. 4. The fracture surfaces of the intermediate temperature embrittled specimens are estimated to consist of more grain boundaries than those of non-embrittled specimens. Heat treatment b is considered to enhance the phosphorus and sulphur segregation to grain boundaries. The phosphorus and sulphur contents from fractured surfaces of all specimens fractured at non-embrittlement temperatures, are very low and they do not segregate on fracture surfaces.

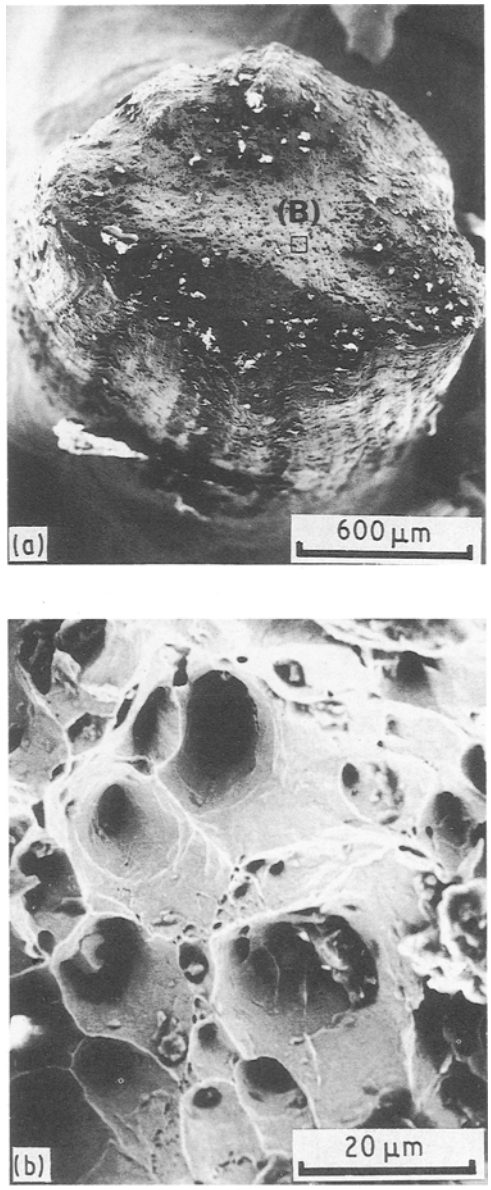


*Figure 3* SEM fractography of specimen tensile tested at 773 K and strain rate $1.43\times10^{-4}\,s^{-1}$ (non-embrittlement condition) after heat treatment a.

As a result, the segregations of phosphorus and sulphur to grain boundaries are considered to enhance the intermediate temperature embrittlement in the Fe–17Cr alloy.

According to the thermodynamical calculation by Swalin [6], the segregation concentration of an impurity at grain boundaries, is expressed by

$$X^{b}_{imp} = \frac{X_{imp}\exp(Q/kT)}{(1 - X_{imp}) + X_{imp}\exp(Q/kT)}$$

where $X^{b}_{imp}$ is the impurity atom concentration at grain boundaries, $X_{imp}$ the concentration of impurity atom in the specimen, $Q$ the binding energy between boundaries and a impurity atom, $T$ the temper-

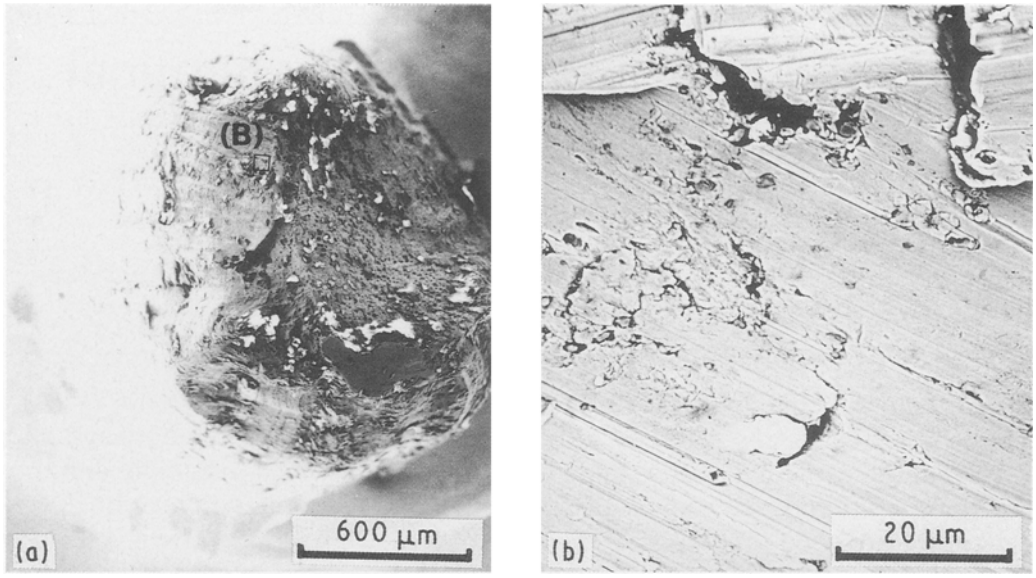


*Figure 4* SEM fractography of specimen tensile tested at 773 K and strain rate $1.43 \times 10^{-3}\,s^{-1}$ (embrittlement condition) after heat treatment b.

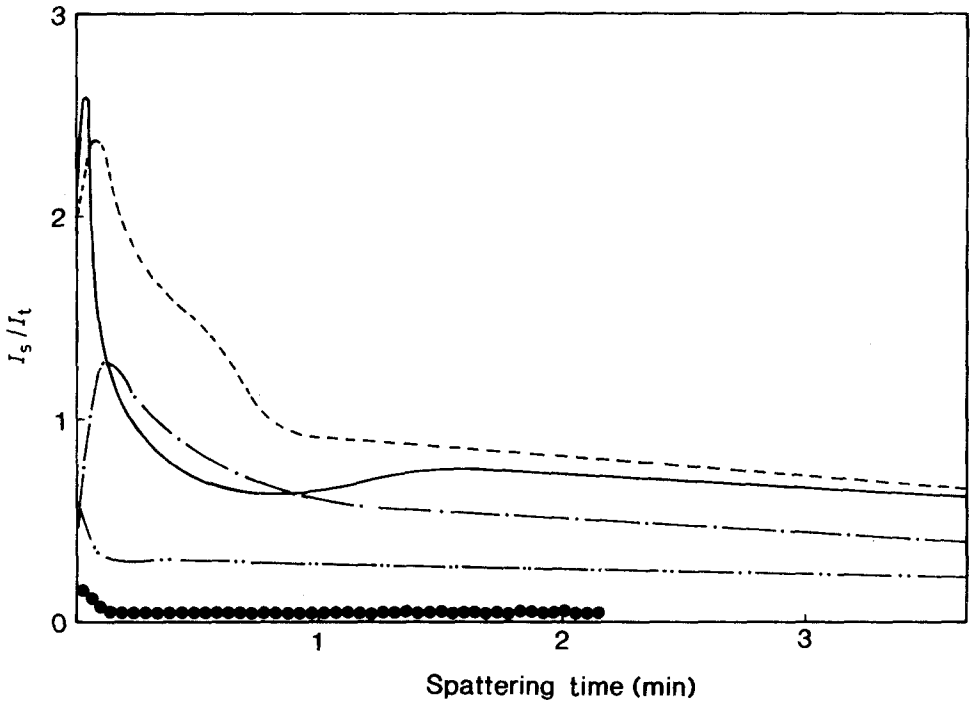


*Figure 5* Phosphorus and sulphur distributions from fracture surfaces in depth. Heat treatment a: $\dot{\varepsilon} = 1.43 \times 10^{-3}\,s^{-1}$, $T = 923$ K (—·— sulphur, ···— phosphorus). Heat treatment b: $\dot{\varepsilon} = 1.43 \times 10^{-3}\,s^{-1}$, $T = 773$ K (– – – sulphur, —— phosphorus). Heat treatments a and b: non-embrittlement temperature (● sulphur and phosphorus).

ature, and $k$ the Boltzman constant. Assuming $Q = 0.1$–$0.5$ eV, $T = 1123$ K (segregation treatment temperature), then $X^{b}_{imp}$ at 1123 K is about 3 to 300 times $X_{imp}$, depending upon $Q$. It is easily imagined that phosphorus and sulphur segregate to grain boundaries and enhance the intermediate temperature embrittleness. On the other hand, when $T = 1573$ K (solution treatment temperature), and the other values are equal as mentioned above, the difference in $X^{b}_{imp}$ and $X_{imp}$ is small and there is almost no segregation of impurity atoms at grain boundaries.

## 4. Conclusion

The impurity segregations of phosphorus and sulphur to grain boundaries enhance the intermediate temperature embrittlement.

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