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Intermediate temperature embrittlement in high-purity Ni and binary Ni(Bi) alloy

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The tensile ductility of high-purity Ni and binary Ni(Bi) alloy were studied in the temperature range of 400–850 °C. Ni(Bi) alloy shows evident intermediate temperature embrittlement with a minimum ductility between 700 and 750 °C, while high-purity Ni does not. It is concluded that this embrittlement must be an impurity effect. On the basis of quantitative estimates, the embrittlement is interpreted as being caused by thermo-induced non-equilibrium grain-boundary segregation of Bi.

Keywords: Ni alloys; Grain-boundary segregation; Tensile test; Embrittlement; Bismuth

Ni-based superalloys are advanced structural materials that are mainly used in aircraft engines and also extensively used in the chemical, petrochemical and electrical industries [1–3]. Many experiments [4–10] have demonstrated that these alloys retain an embrittling behavior in the intermediate temperature range (between 500 and 900 °C), which means that the maximum elongation and reduction in area in tensile tests at intermediate temperature are much lower than those at low ($\leqslant$500 °C) and high temperatures ($\geqslant$900 °C). Nowadays, the development of more efficient and longer life engines requires the development of more reliable Ni-based superalloys. Since service temperatures of superalloys generally lie in the corresponding temperature range [1–10], intermediate temperature embrittlement (ITE) has become an urgent problem to be solved.

Ni-based superalloys can be classified into wrought, cast and powder metallurgy alloys according to the manufacturing route, and into solution-strengthened and age-hardened alloys according to the strengthening mechanism. On the one hand, ITE exists not only in wrought superalloys [4,8,10], but also in cast [5] and powder metallurgy [7] ones. On the other hand, ITE exists in both solution-strengthened [4,10] and age-hardened [5,7,8] superalloys. Therefore, ITE is a general feature of Ni-based superalloys that is independent of the manufacturing route and strengthening mechanism.

At present, there are several possible explanations for ITE [4–10], which are quite different from each other. Researchers have drawn conflicting conclusions even for the same superalloy, e.g. Hastelloy X [10,11]. This lack of consensus necessitates a convincing interpretation of this general feature. The present work was thus carried out to clarify the mechanism of ITE by high-purity Ni and binary Ni(Bi) alloys. The experimental results and theoretical analyses should provide a base from which to improve service lives and reliabilities of Ni-based superalloys.

Tensile samples of high-purity Ni (the total concentration of impurities is less than 1 wt. ppm) were cut directly from the as-delivered sheets by a spark erosion machine. The reduced cross-section is $2\,\mathrm{mm} \times 2\,\mathrm{mm}$ and the gauge length is 6 mm. Samples were heat-treated at 1000 °C for 0.5 h in an evacuated quartz capsule (6×10^{-3} Pa). Afterwards, the quartz capsule was broken and the samples were immediately quenched in iced water. An Ni(Bi) alloy of 25 wt. ppm Bi was produced by vacuum induction melting, mixing high-purity Ni with the correct amount of high-purity Bi. The ingot was then deformed into a bar of 4 mm diameter and checked to ensure that it was free of cracks. The bar was heat-treated for 455 °C/1 h + 600 °C/1 h + 1000 °C/0.5 h, then immediately quenched in iced water.

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Tensile samples with dimensions of 2 mm diameter and 6 mm gauge length were cut from the quenched bar. Tensile tests were performed in air using an INSTRON-1195 machine with a strain rate of $10^{-2}\ s^{-1}$ in the temperature range of 400–850 °C. All samples were held at test temperatures for 45 min prior to mechanical testing. The elongation at fracture was used to indicate the ductility. To obtain reliable data, results of three or six independent samples were averaged in the case of high-purity Ni or Ni(Bi) alloy, respectively. After tensile testing, the average grain sizes of samples were measured by means of optical microscopy. Furthermore, the microstructure of the Ni(Bi) alloy after heat treatment was observed by transmission electron micrography (TEM; Libra 200FE), with particular emphasis on the grain boundaries (GBs).

Figure 1 presents the key result of this study, the tensile ductility of high-purity Ni and binary Ni(Bi) alloy (a), as well as representative stress–strain curves (b) obtained during testing the Ni(Bi) alloy. It is clear from Figure 1a that the ductility of high-purity Ni increases with increasing temperature and then decreases slightly above 800 °C. By contrast, the ductility of the Ni(Bi) alloy first drops significantly up to 700 °C, then increases rapidly over 750 °C. Thus, the Ni(Bi) alloy reveals clear ITE with maximum embrittlement between 700 and 750 °C, while the high-purity Ni does not. The stress–strain curves of the Ni(Bi) alloy in Figure 1b confirm the existence of ITE and indicate that the ultimate strength decreases with increasing test temperature.

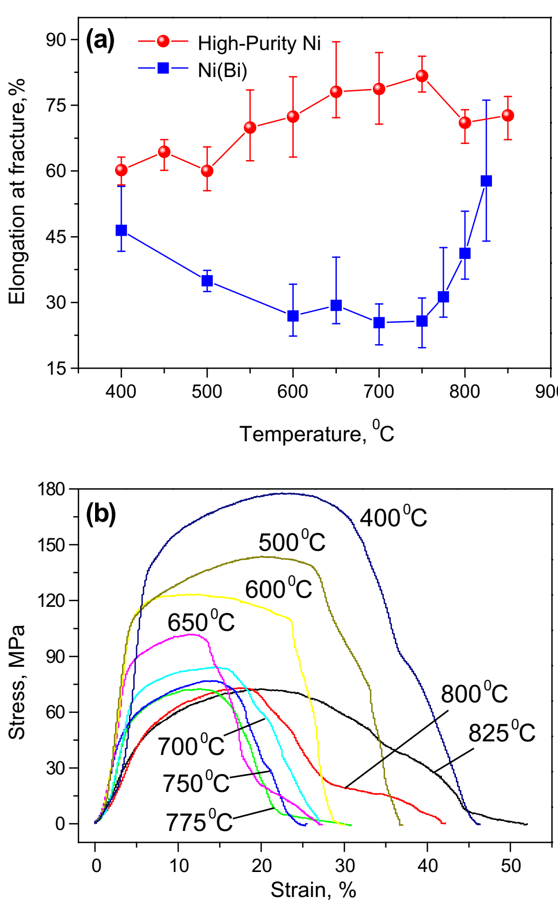

Figure 1. (a) Elongations of high-purity Ni and Ni(Bi) alloy at different temperatures and (b) representative stress–strain curves of Ni(Bi) alloy.

Figure 2 shows the optical microscopy of samples after tensile testing at (a) 400 °C, (b) 750 °C and (c) 825 °C, as well as a TEM micrograph of a sample heat-treated at (d) 750 °C for 45 min. It can be clearly seen from Figure 2a–c that no dynamic recrystallization occurs in the Ni(Bi) alloy during the tensile test. The average grain size for all samples was determined to be 630.8 μm. From the TEM image in Figure 2d, it is clear that no precipitates exist at GBs.

To date, several interpretations of ITE have been proposed, namely GB shearing or sliding [4], decohesion of the glide plane [5], gas phase embrittlement [6], intergranular precipitates [7], dynamic strain aging [8] and GB segregation of impurities [9,10]. The model study of this work demonstrates that ITE must be an impurity effect. In this case, the first three interpretations are not valid. Intergranular precipitates certainly represent a kind of impurity effect. However, the TEM image in Figure 2d verifies that with the very small Bi content considered no intergranular precipitates have formed after the heat treatments used in the mechanical tests. Thus, the interpretation of intergranular precipitates must be also excluded. Dynamic strain aging is another kind of impurity effect, but is characterized by a serrated flow in the stress–strain curve [12]. According to Figure 1b, however, no serrated flows are observed in stress–strain curves of Ni(Bi) alloy over the whole range of temperatures. Hence, the observed ITE cannot be related to dynamic strain aging either. It is noteworthy that fracture surfaces of commercially pure Ni and Ni-based superalloys in intermediate temperature range have frequently been reported to be intergranular

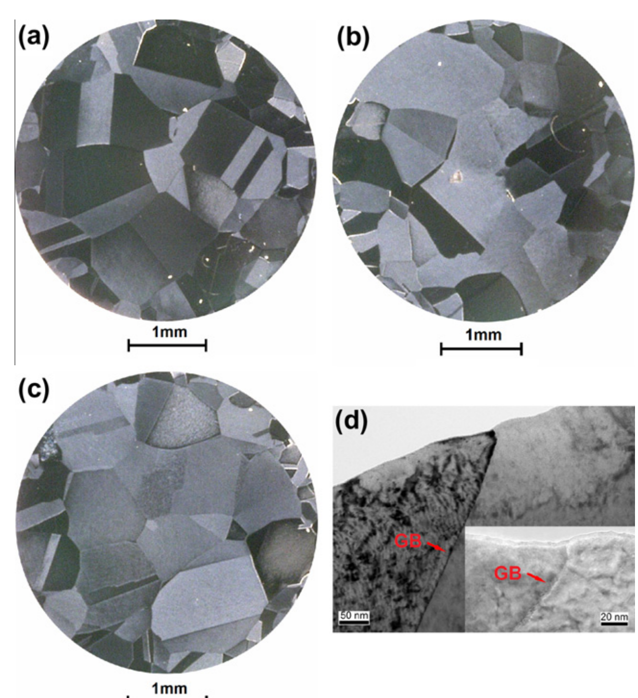

Figure 2. Optical microscopy images of samples after tension at (a) 400 °C, (b) 750 °C and (c) 825 °C, as well as (d) a TEM image of samples heat-treated at 750 °C for 45 min.