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Mechanical properties of recrystallized $L1_2$–type $Ni_3(Si, Ti)$ intermetallics

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The mechanical properties of the $Ni_3(Si, Ti)$ alloys undoped and doped with 50 p.p.m. boron, both of which were polycrystalline specimens prepared by recrystallization, were investigated by tensile testing. The yield stress was found to increase with increasing test temperature to a maximum at 800 K, followed by a decrease. The tensile elongation was highest at room temperature and tended to decrease with increasing temperature for both alloys, but was consistently higher in the boron-doped $Ni_3(Si, Ti)$ alloys than in the undoped ones over all the test temperatures. The change in the ultimate tensile stress (UTS) with temperature was similar to that of tensile elongation. The transgranular fracture became dominant as the elongation increased, regardless of the alloys and the testing temperature. Thus, this work again verified that the alloying method proposed by the present authors is useful for improving the grain-boundary cohesion of $L1_2$-type ordered alloys.

1. Introduction

The Ni_3Si alloy is known to increase its strength with increasing temperature [1], and has excellent corrosion and oxidation resistance at ambient and elevated temperatures. However, this alloy suffers from grain-boundary brittleness similar to the Ni_3Al alloy. The addition of boron to improve the ductility of this alloy has been unsuccessful.

In a previous paper [2] in which the metallographic and structural analyses for $L1_2$-type Ni–Si–Ti ternary alloy were performed, it was found that the alloying element, titanium, substitutes for elemental silicon and also was greatly soluble in the Ni_3Si alloy. The mechanical properties of the Ni_3Si alloyed with elemental titanium were investigated in their polycrystalline form [3]. Consequently, some useful results were introduced by this alloying [3]; the yield stress increased with increasing titanium concentration at all testing temperatures. Also, the peak temperature in the yield stress–temperature curve increased with increasing titanium concentration. The most striking result was that the ductility was established by the addition of elemental titanium. The higher elongation values were observed in the alloys consisting of higher titanium and higher nickel concentrations. Further improvement of the tensile elongation was found by the addition of a small amount of boron. The variation of the elongation with temperature showed a peak at intermediate temperatures. The ductility of the Ni_3Si alloys observed in the previous work [3] verified the alloying principle to improve the grain-boundary cohesion of $L1_2$-type ordered alloys which has recently been proposed by the present authors [4–7]; it was demonstrated that a replacement of X (= b sub-group element in the periodic table) with T (= mostly transition metals) in the Ni_3X alloy reduces the difference in the electrochemical bonding nature, resulting in a lowering of the propensity for grain-boundary fracture and improving the ductility in the Ni_3X alloys.

The main purpose of this work was to determine whether the change in the mechanical properties of Ni_3Si alloys on the addition of elemental titanium was due to the microstructural effect or to the alloying effect itself. For this purpose, the $Ni_3(Si, Ti)$ alloys and the $Ni_3(Si, Ti)$ alloys doped with a small amount of boron were prepared in the recrystallized form. Their mechanical properties were observed by tensile test over a wide range of test temperatures, and also correlated with the fractographic observation.

2. Experimental procedure

The alloys were prepared by nonconsumable arc melting in an argon atmosphere. Nickel, 99.9 wt % purity, silicon, 99.999 wt % purity, and titanium sponge, 99.8 wt % purity, were used as starting materials. The nominal and analysed chemical compositions of the alloys used in this work are shown in Table I and the agreement between the two values was fairly good; the selected composition was 79.5 at. % Ni, 11 at. % Si and 9.5 at. % Ti (denoted $Ni_3(Si, Ti)$ alloy) and located in a single-phase region of the $L1_2$ structure as shown in Fig. 1 at which marked strengthening and ductility are expected [3]. Also, to improve the ductility further, the alloy consisting of this composition was doped with 50 p.p.m. boron using an Ni–10 wt % boron master alloy. The alloy buttons with dimensions approximately 15 mm × 15 mm × 80 mm were homogenized

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TABLE I Nominal and analysed values of chemical compositions for the alloys used in this work

Alloy	Nominal (analysed) chemical compositions			
	Ni (at. %)	Si (at. %)	Ti (at. %)	B (wt. %) (p.p.m.)
$Ni_3(Si, Ti)$	79.5	11(10.97)	9.5(9.43)	
$Ni_3(Si, Ti)$ + 50 p.p.m. B	79.5	11(10.93)	9.5(9.44)	50(37)

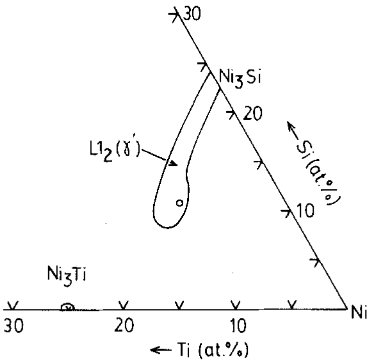

Figure 1 Chemical composition of the present specimen (○) which was located in a phase field of $L1_2$ structure (γ').

at 1323 K for 1 d in vacuum and then rolled at 573 K in air to about 50% reduction. The annealing for recrystallization was done at 1273 K for 5 h in vacuum. After heat treatment, metallographic examination, X-ray analysis and transmission electron microscopic (TEM) observations were performed to characterize the microstructure of these alloys. Thin foil specimens for TEM observation were prepared by electropolishing in a solution of 10% H_2SO_4 and 90% CH_3OH at 273 K. TEM observation was made using a Jeol electron microscope operating at 100 kV.

The tensile specimens with dimensions approximately 1 mm × 2.2 mm × 14 mm gauge length were prepared using a precision wheel cutter and an electro-erosion machine. The faces of the specimens were abraded on SiC paper. The mechanical tests were carried out using an Instron-type testing machine at a nominal strain rate of 1.2×10^{-3} sec^{-1}. The testing temperatures were from 77 to 1073 K. Tests at 77 K were performed with an apparatus suspended in a Dewar vessel filled with liquid nitrogen. The tests at room temperature and at elevated temperatures were conducted in a vacuum better than 1.3×10^{-3} Pa. After tensile testing, the fracture surfaces of the specimens were examined by scanning electron microscopy (SEM).

3. Results and discussion

3.1. Microstructure

Fig. 2 shows the optical microstructures of the $Ni_3(Si, Ti)$ alloy (Fig. 2a) and the boron-doped $Ni_3(Si, Ti)$ alloy (Fig. 2b). Both alloys exhibit apparently single-phase structure with the average grain size of approximate 50 μm.

Figs 3 and 4 show the TEM bright-field images of both alloys. The $Ni_3(Si, Ti)$ alloy again showed no second phases and relatively straight grain boundaries, as seen in a bright-field image (Fig. 3a), and also an indication of the ordered structure; i.e. the ordered spots as indicated in a selected-area diffraction pattern (Fig. 3b). On the other hand, the boron-doped $Ni_3(Si, Ti)$ alloy showed second phases at the grain-boundary planes and/or at triple junctions of grain boundaries (Fig. 4a). Although structural and chemical analyses were not performed for these particles, it is likely that they are boride combined with the constituent atoms. Thus, it appears that the solubility limit of boron in the ternary $Ni_3(Si, Ti)$ alloys is very low, i.e. below 50 p.p.m. (~ 0.03 at. %). This value is extremely low in comparison with that of the Ni_3Al alloy (i.e. between 1.5 and 2.0 at. % [8]).

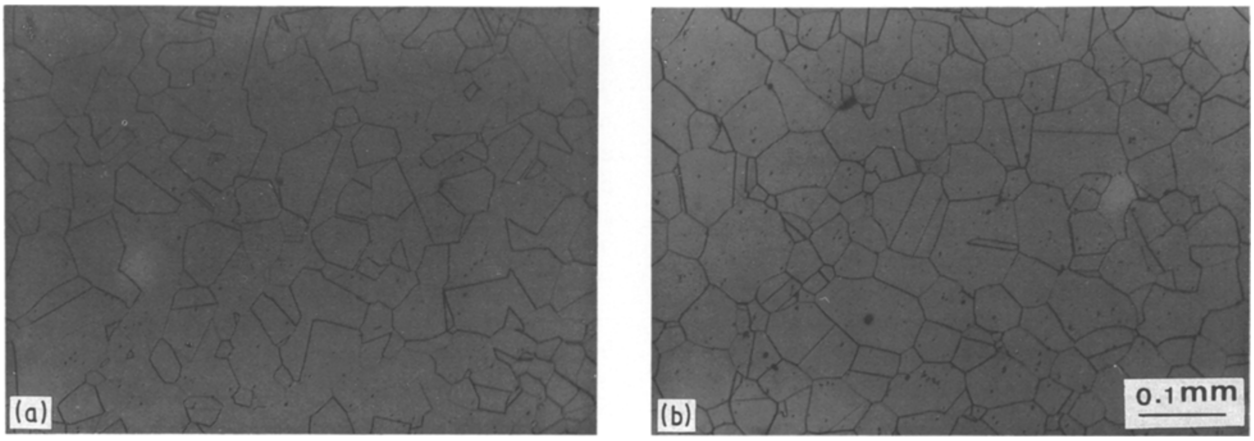

Figure 2 Optical microstructures of (a) the $Ni_3(Si, Ti)$ alloy and (b) the $Ni_3(Si, Ti)$ alloys doped with 50 p.p.m. boron.

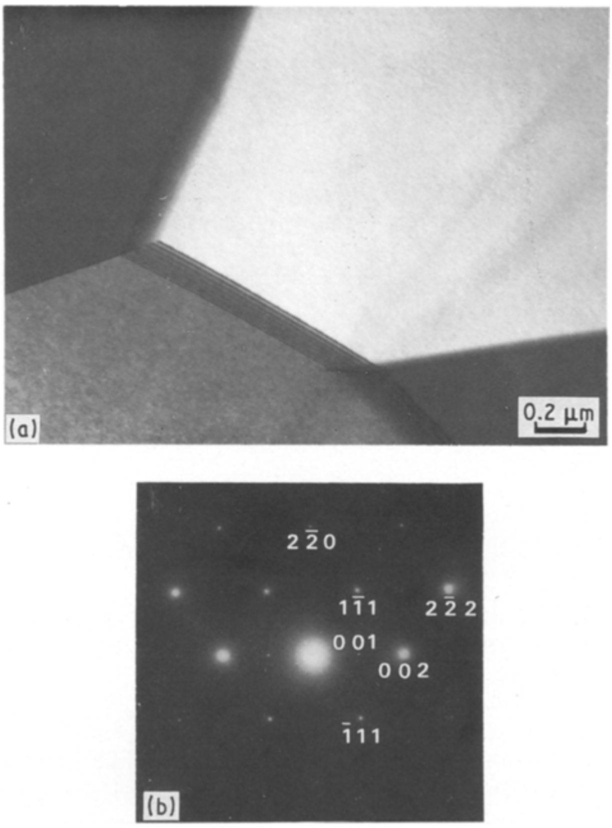

Figure 3 (a) Bright-field image and (b) selected-area diffraction patterns (TEM) for the $Ni_3(Si, Ti)$ alloy.

3.2. Tensile properties

Fig. 5 illustrates the temperature dependence of the yield stress (defined as an offset stress of 0.2% plastic strain) of both alloys. The yield stresses began to increase from 77 K with increasing temperature, reached the maximum (peak) and then tended to decrease with further increasing temperature. At sufficiently high temperatures, the yield stresses were not plotted because these alloys did not show enough plasticity, as described below. It is clearly demonstrated from this figure that (1) the level of the yield stress and its temperature dependence in recrystallized materials is not different from those of the as-cast alloys [3], and (2) both alloys displayed identical yield stress–temperature curves, implying solid-solution strengthening or particle strengthening in the boron-doped $Ni_3(Si, Ti)$ alloy to be negligibly small. The latter result might be due to the fact that the boron content in the solution was too low and also the volume fraction of boride as the second phase was too low.

Fig. 6 illustrates the variations of the elongation (Fig. 6a) and the ultimate tensile stress (UTS) (Fig. 6b) of the two alloys with testing temperature. The tensile elongation was highest at room temperature regardless of the doping with boron. However, the boron-doped $Ni_3(Si, Ti)$ alloys showed consistently higher elongation values. In the case of the $Ni_3(Si, Ti)$ alloy, the elongation decreased monotonically from room temperature with increasing temperature and then disappeared almost at 673 K, whereas in the case of the boron-doped $Ni_3(Si, Ti)$ alloy, the elongation remained almost constant up to 673 K, then decreased rapidly with increasing temperature and eventually disappeared at 973 K. The UTS behaviour was very similar to that of elongation; the UTS was highest at room temperature and decreased with increasing temperature for both alloys, but was higher in the boron-doped $Ni_3(Si, Ti)$ alloys than in the undoped $Ni_3(Si, Ti)$ alloys.

Here, it should be noted that the elongation and the UTS values obtained for recrystallized materials (this work) are basically identical to those obtained for the as-cast materials [3], although the direct comparison is impossible because of a slight difference in alloy composition between the two materials. Therefore, it is suggested here that the increase in ductility of the Ni_3Si alloy on addition of titanium is principally due to the "alloying" effect. The difference is only that the elongation and the UTS values of the recrystallized $Ni_3(Si, Ti)$ alloys were improved, particularly at ambient temperatures. This improvement might be attributed to the finer grain size and equiaxed grain morphology.

Higher elongation values were actually shown in the boron-doped $Ni_3(Si, Ti)$ alloys which contained particles of boride. In the case of the boron-doped Ni_3Al alloy, the formation of the boride caused complete elongation loss [8]. Therefore, it is noted here

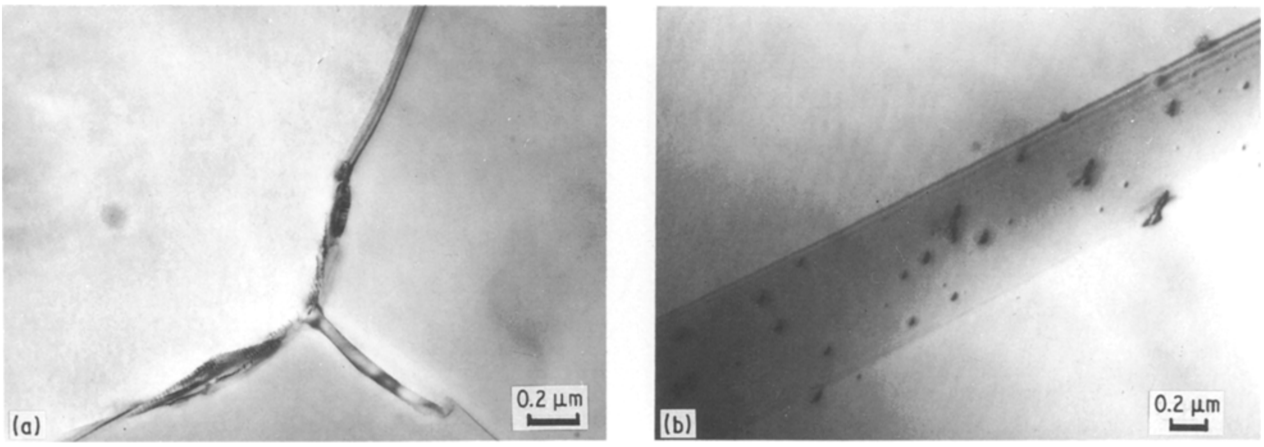

Figure 4 Bright-field images (TEM) for the $Ni_3(Si, Ti)$ alloy doped with 50 p.p.m. boron.

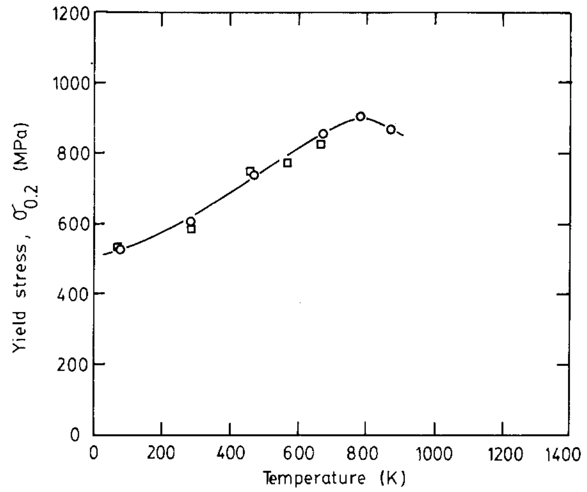

Figure 5 Variations of the 0.2% yield stress of the (□) $Ni_3(Si, Ti)$ alloys and (○) boron-doped $Ni_3(Si, Ti)$ alloys with temperature.

that the free boron dissolved in the matrix is certainly beneficial to the ductility of this alloy through the segregation to the grain boundaries, as in the boron-doped Ni_3Al alloy, and also that the second phases precipitated at the grain boundaries as the boride is not so detrimental as in the boron-doped Ni_3Al alloy.

3.3. Fracture behaviour

Figs 7 and 8 illustrate the variations of the fractographic patterns observed in the $Ni_3(Si, Ti)$ alloys and the boron-doped $Ni_3(Si, Ti)$ alloys with testing temperature, respectively. For the $Ni_3(Si, Ti)$ alloy, the

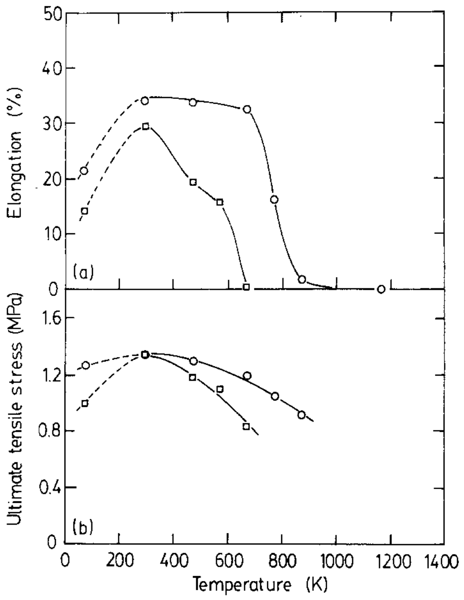

Figure 6 Variations of (a) the elongation and (b) the ultimate tensile stress (UTS) of the (□) $Ni_3(Si, Ti)$ alloys and (○) boron-doped $Ni_3(Si, Ti)$ alloys with temperature.

transgranular fracture with the dimple-like patterns was dominant at room temperature and the intergranular fracture patterns tended to be more dominant with increasing temperature. The fracture pattern at 77 K was a mixture of the transgranular and the intergranular fracture patterns. For the boron-doped $Ni_3(Si, Ti)$ alloys, the transgranular fracture mode was more dominant up to higher temperatures. Thus, the fracture surfaces of the tensile specimens exhibited a variety of fracture patterns, depending on the testing

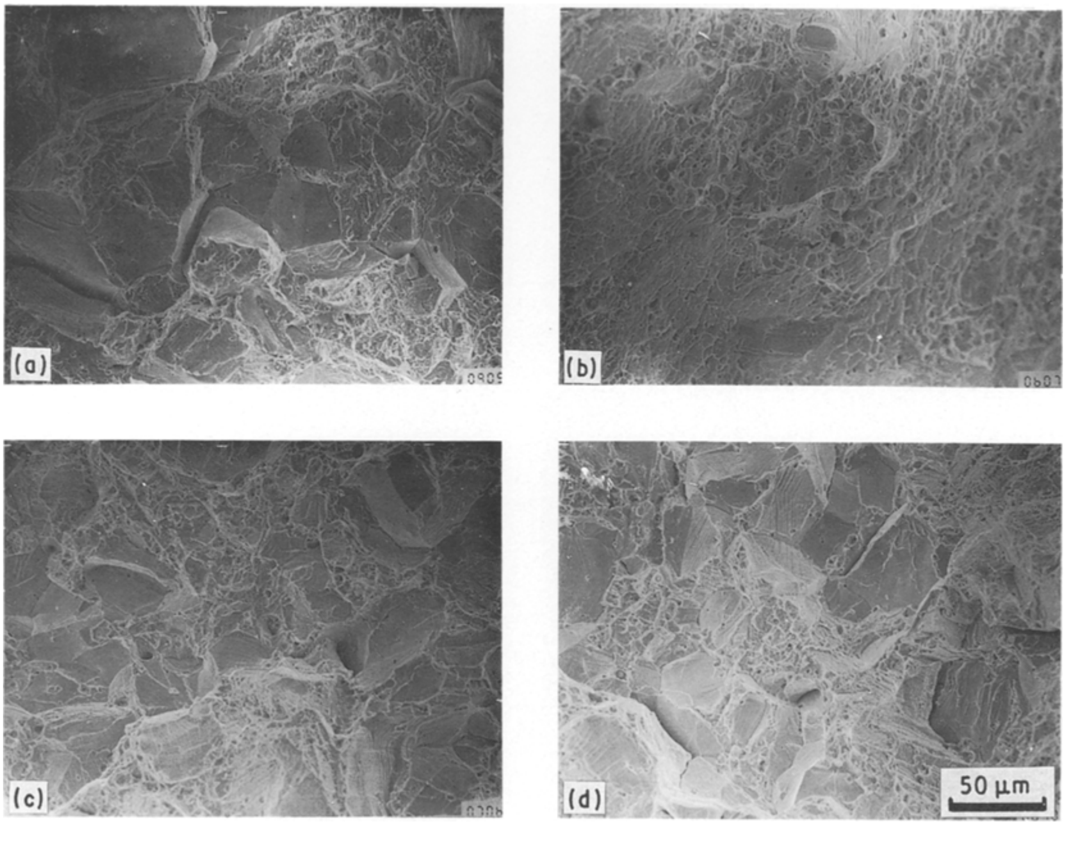

Figure 7 Variation of the fractography of the $Ni_3(Si, Ti)$ alloys with temperature: (a) 77 K, (b) 293 K, (c) 573 K, (d) 673 K.

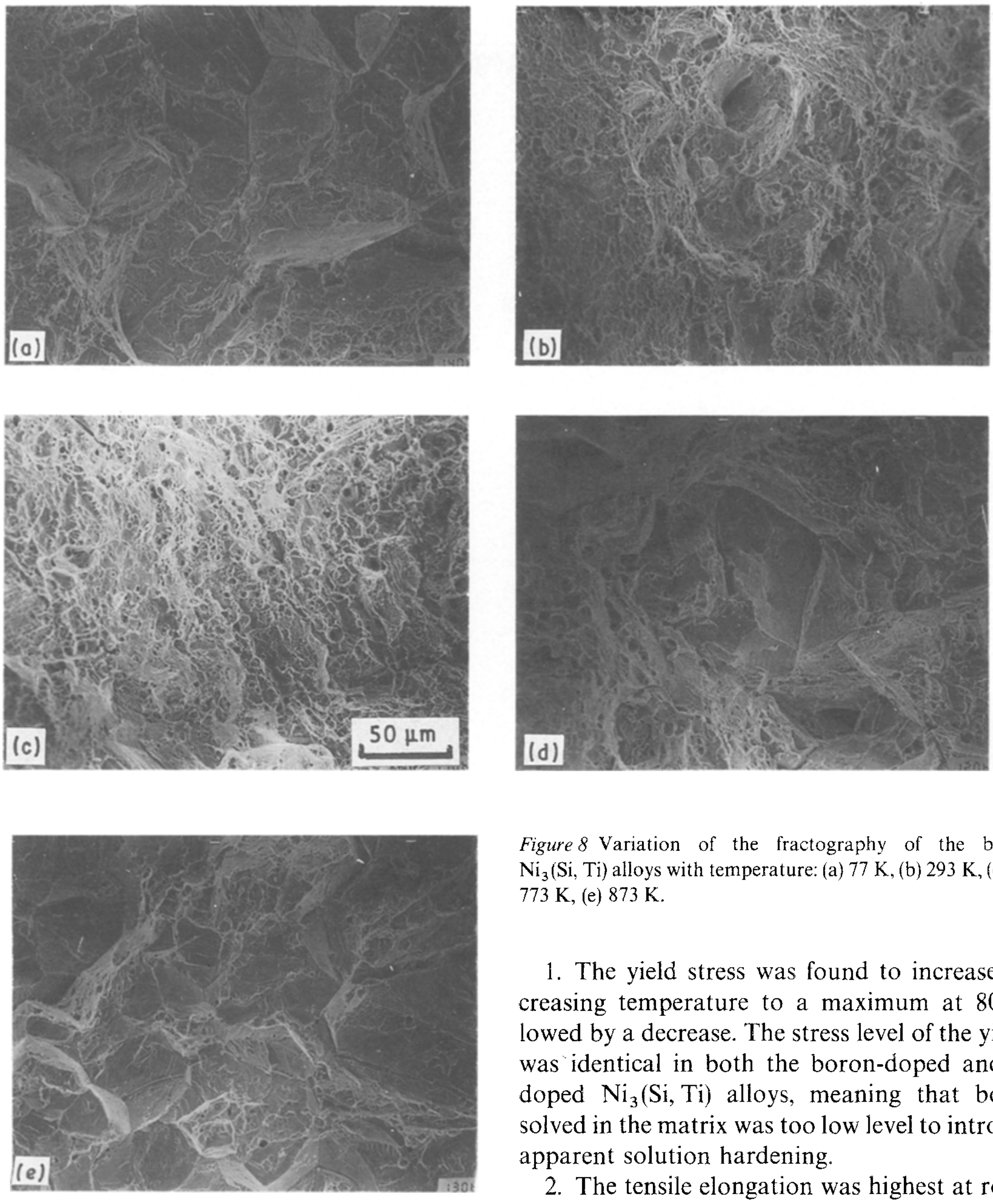

Figure 8 Variation of the fractography of the boron-doped Ni_3(Si, Ti) alloys with temperature: (a) 77 K, (b) 293 K, (c) 673 K, (d) 773 K, (e) 873 K.

temperatures and the alloys. However, the fracture patterns were primarily correlated with the degree of elongation (or the UTS) itself. As the elongation (or the UTS) value increased the fracture pattern changed from intergranular to transgranular.

4. Conclusions

The mechanical properties of the yield stress, elongation, ultimate tensile stress (UTS) and fracture mode in the Ni_3(Si, Ti) alloys undoped and doped with 50 p.p.m. boron were systematically investigated by tensile testing, using the recrystallized specimens. The Ni_3(Si, Ti) alloys were the $L1_2$ single-phase structures with a grain size of approximately 50 µm, whereas the boron-doped Ni_3(Si, Ti) alloys consisted of almost the same grain size but contained the second phases of the particles at grain boundaries. Emphasis was placed on the testing temperature and the effect of boron doping on these mechanical properties. The following results were obtained from the present study.

1. The yield stress was found to increase with increasing temperature to a maximum at 800 K, followed by a decrease. The stress level of the yield stress was identical in both the boron-doped and the undoped Ni_3(Si, Ti) alloys, meaning that boron dissolved in the matrix was too low level to introduce any apparent solution hardening.
2. The tensile elongation was highest at room temperature and tended to decrease with increasing temperature for the two alloys. However, the tensile elongation was consistently higher in the boron-doped Ni_3(Si, Ti) alloys than in the undoped ones over all test temperatures. The change in the UTS with temperature was similar to that of the tensile elongation. This behaviour was basically identical to that reported in as-cast materials of this alloy [3].
3. The elongation and the UTS behaviour observed in these alloys correlated well with the fracture patterns. That is, the transgranular fracture became dominant as the tensile elongation increased, being independent of the alloys and the testing temperatures.
4. The increasing ductility of the Ni_3Si alloy on the addition of elemental titanium again verified that the alloying method proposed by the present authors [4–7] is useful for improving the grain-boundary cohesion of $L1_2$-type ordered alloys.

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