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Environmental Effect on Mechanical Properties of Aluminide Matrix Composites

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This paper investigates the validity of the toughness measurement with a variation of the loading rate for distinguishing the fracture mechanism of aluminide intermetallics and their composites. The ductility and fracture toughness of Ni_3Al alloys and their composites are governed by inherent grain boundary brittleness and moisture-induced embrittlement at ambient temperatures. Although B doping is effective in suppressing both factors, remarkable improvement of toughness mainly depends on grain boundary strengthening. The toughness of the alloys is influenced by the dislocation locking mechanism and the extrinsic embrittlement promoted by diffusion of oxygen at intermediate temperatures. Extrinsic embrittlement is the predominant mechanism in determining the toughness at 673 K. Restriction of the dislocation motion is the predominant factor in determining toughness at 873 and 1073 K. The composites reinforced with TiC particles exhibit exceptionally constant toughness at 300 to 900 K.

Keywords environmental effects, fracture, intermetallic compounds, Ni_3Al, reactive hot pressing

1. Introduction

Intermetallics and their matrix composites have been anticipated as novel structural materials in several industries such as aerospace engineering for the last 20 years.[1] As a result of many studies, some alloys and composites with high strength and ductility have been successfully developed.[1] Furthermore, it has been clarified that their mechanical behavior is governed by environmental effects as well as inherent mechanisms connected with dislocation motion and grain boundary cohesion.[2–6] Damage tolerance designs[7] in severe environments need to be developed to establish practical applications. Because the environmental effects are promoted simultaneously with the inherent mechanisms,[8] the predominant factor determining their toughness should be clarified in several environments to develop a strategy for damage tolerance design. The present work focuses on the environmental effects and inherent mechanisms of intermetallics. The fracture mechanisms of Ni_3Al alloys and their composites were analyzed by using loading-rate dependence of fracture toughness as an example of advanced intermetallic-based structural materials.

2. Experimental Procedure

The Ni_3Al matrix composites with ceramic fine particles, including TiC (0.7 μm, Japan New Metals Co., Ltd., Osaka, Japan), TiN (0.7 μm, Japan New Metals Co., Ltd., Osaka, Japan), and α-Al_2O_3 (0.4 μm, Sumitomo Chemical Co., Ltd., Tokyo, Japan), were fabricated by reactive hot pressing.[9] The processing details have been described in a previous paper.[9] Tensile test pieces with a gauge section of 0.5 × 1 × 8 mm were cut by electric discharge machining. The tensile test was performed with a strain rate of 8.3×10^{-4} s^{-1} at 300 to 1473 K in air. The fracture toughness of these materials was measured using rectangular bars (8 × 4 × 35 mm) with a chevron-notch (a_0 = 4 mm) at a loading rate of 10^{-2} to 10 MPa $m^{1/2}$ s^{-1} in air and a silicone oil (SRX310, Toray Dow Corning Silicone Co., Ltd., Tokyo, Japan) bath at 300 K. The toughness measurement at 673 to 1073 K was performed with the chevron-notched bars after isothermal holding for 0.6 and 7.2 ks at the test temperatures in air. The fracture surfaces of the specimens were observed by scanning electron microscopy.

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3. Results and Discussion

3.1 Mechanical Behavior of Ni_3Al Alloys

Figure 1 shows the tensile strength and elongation of the Ni-24 at.% Al-0.1 at.% B alloy at 300 to 1273 K in air. The temperature dependence of their mechanical behavior can be divided into three regions corresponding to different fracture mechanisms, labeled regions I, II, and III in Fig. 1.

3.1.1 Region I (300 to 400 K). The ductility and fracture toughness of these alloys are sensitive to the intrinsic grain boundary brittleness[2,3] and to moisture-induced embrittlement (environmental embrittlement)[4] at 300 to 400 K. Figure 2 shows the fracture toughness of Ni-25 at.% Al and Ni-24 at.% Al alloys with and without 0.1 at.% B doping at 300 K. The alloys without doping exhibit a characteristic loading-rate dependence of fracture toughness in air. This dependence is not observed in the oil bath. Therefore, the mechanical behavior is caused by the environmental effect. Environmental embrittlement is a dynamic phenomenon originating from stress-assisted diffusion of atomic hydrogen (H) formed by surface reaction of moisture in air. The fracture toughness of the alloys measured at 10 MPa $m^{1/2}$ s^{-1} in air agrees with that in the oil bath. Thus,

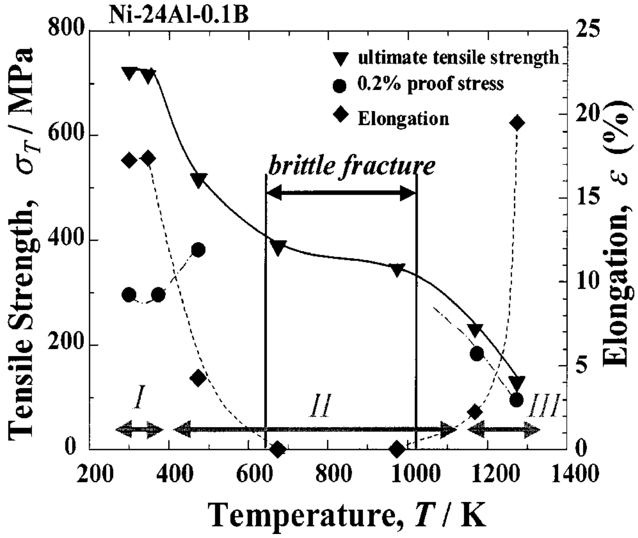

Fig. 1 Tensile strength and elongation in air of Ni-24% Al-0.1 at.% B alloys fabricated by reactive hot pressing

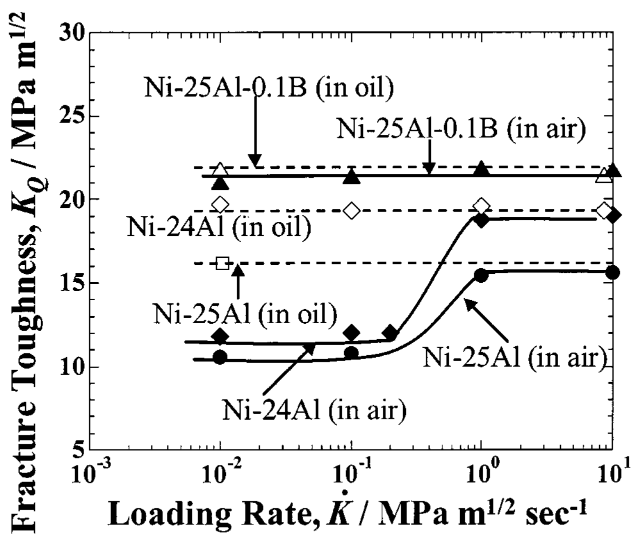

Fig. 2 Loading rate dependence of fracture toughness of Ni-25 at.% Al and 24 at.% Al alloys with and without 0.1 at.% B doping at 300 K in air and in an oil bath

the toughness without the environmental effect can be estimated by the toughness measurement above 10 MPa m$^{1/2}$ s^{-1}.

The environmental embrittlement of Ni-25 at.% Al alloys is eliminated by doping. At the same time, the toughness intrinsically improved to 30% higher than that of the nondoped alloy. However, the Ni-24 at.% Al-0.1 at.% B alloys do not fracture validly in the present condition of toughness measurement due to their high ductility. The fracture toughness of the Ni-24 at.% Al alloys with the doping is much higher than that of the Ni-25 at.% Al-0.1 at.% B alloy. Chen *et al.*, who performed a computer simulation with embedded-atom style potentials, have demonstrated that grain boundary cohesion of Ni_3Al alloys is remarkably enhanced by B doping when Ni atoms substitute into Al sites at grain boundaries.[10] Remarkable improvement

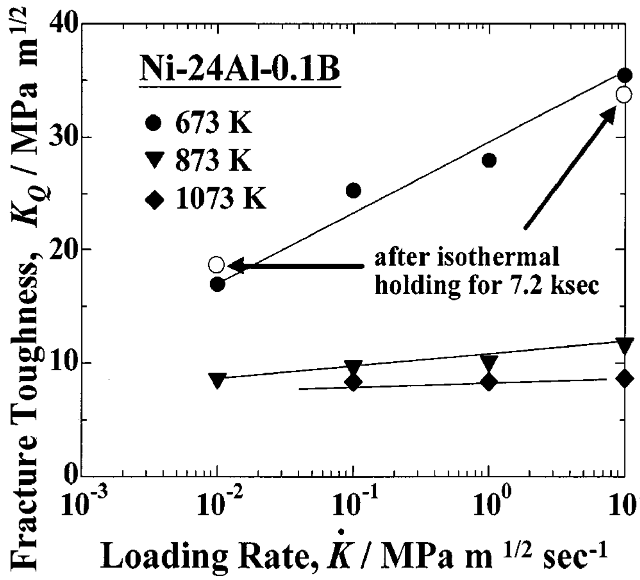

Fig. 3 Loading rate dependence of fracture toughness of Ni-24 at.% Al-0.1 at.% B alloys at 673, 873, and 1073 K in air

of fracture toughness in the alloys with Ni-rich composition seems to be caused by the enhancement of grain boundary cohesion by the B doping. Unfortunately, the grain boundary reinforced by the segregation of B deteriorates under H_2 gas with a partial pressure of 10 Pa.[11,12]

3.1.2 Region II (400 to 1100 K). Ni_3Al alloys are well known to exhibit a yield anomaly caused by the Kear-Wilsdorf mechanism,[1] *i.e.*, locking of cross-slipped dislocations from (111) to (100), in this region. This mechanism concurrently leads to decreased ductility and toughness of the alloys. Furthermore, the environmental embrittlement promoted by diffusion of oxygen is observed at these temperatures.[5,6] Although the reliability of the alloys is known to be governed by these two mechanisms in this region, the kinetics of embrittlement remain unclear.

Figure 3 shows the loading-rate dependence of fracture toughness of the Ni-24 at.% Al-0.1 at.% B alloy at 673, 873, and 1073 K. Embrittlement based on the dislocation locking mechanism does not occur at 673 K because the alloy exhibits high toughness above 30 MPa m$^{1/2}$ at a loading rate of 10 MPa m$^{1/2}$ s^{-1}. However, the toughness at 10^{-2} MPa m$^{1/2}$ sec^{-1} decreases to 50% lower than that at 10 MPa m$^{1/2}$ s^{-1}. The toughness does not vary with isothermal holding for 7.2 ks before testing. These results indicate that the oxygen embrittlement is a dynamic phenomenon promoted in force fields. The loading-rate dependence of fracture toughness is diminished at 873 and 1073 K, as shown in Fig. 3. Hence, the dislocation locking mechanism is the predominant factor governing fracture toughness at these temperatures. The fracture toughness is restricted to a basic value even if the environmental effect is inhibited by alloying techniques such as alloying of Cr.[13] Hence, damage tolerance designs based on the intrinsic low toughness should be considered for the alloys at these temperatures.

3.1.3 Region III (~1100 K). The ductilities of the alloys drastically increase in region III. Dislocations in the alloys can

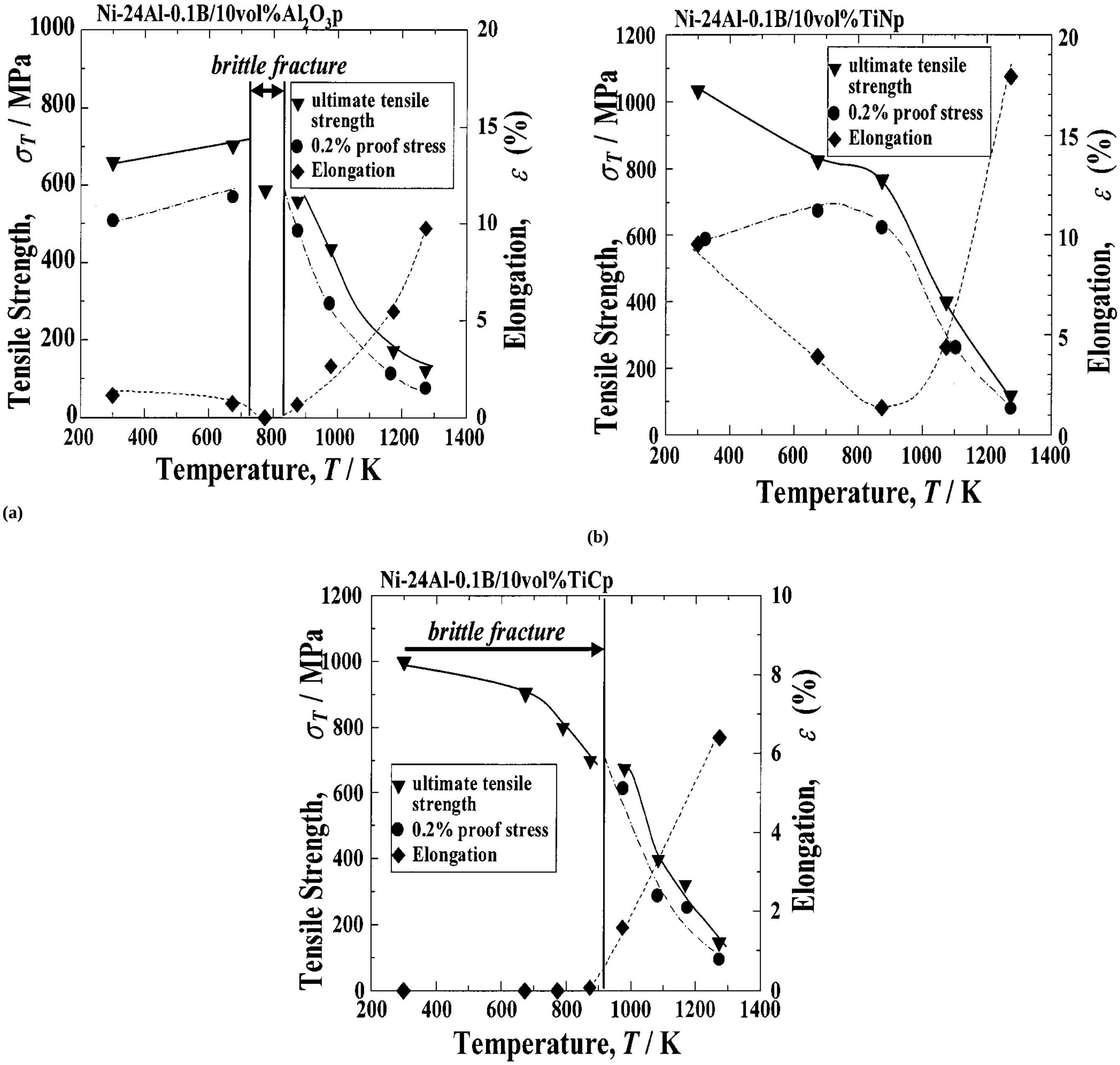

Fig. 4 Tensile strength and elongation of Ni-24 at.% Al-0.1 at.% B matrix composites reinforced with (**a**) α-Al_2O_3, (**b**) TiN, and (**c**) TiC particles in air

slip along the (100) plane in this temperature range. Hence, significant plastic deformation caused by the dislocation motion occurs. Because most materials retain significant strength to approximately 0.5 to 0.6 of their melting point (T_m), $0.7T_m$ is a convenient criterion for selection of structural materials.[14] In the case of Ni_3Al alloys (T_m = 1663 K), the upper limit of their structural applications corresponds to 1273 K.

3.2 Mechanical Behavior of Ni_3Al Matrix Composites

Figure 4(a) to (c) show the results of tensile test in air of the Ni-24 at.% Al-0.1 at.% B matrix composites with 10 vol.% α-Al_2O_3, TiN, and TiC particles, respectively. The composites with α-Al_2O_3 particles are expected to exhibit the typical mechanical behavior of the composites reinforced only by the physical mechanism because the particles have perfect chemical compatibility with the matrix alloys. The 0.2% proof stress is improved to 1.5 times higher than that of the monolithic alloy at ambient temperatures by the effect of the particles. However, the ductility decreases with the addition of reinforcement particles due to the interference of dislocation motion, although the grain boundary brittleness and the moisture-induced embrittlement can be suppressed by the B doping. The ductility decreases

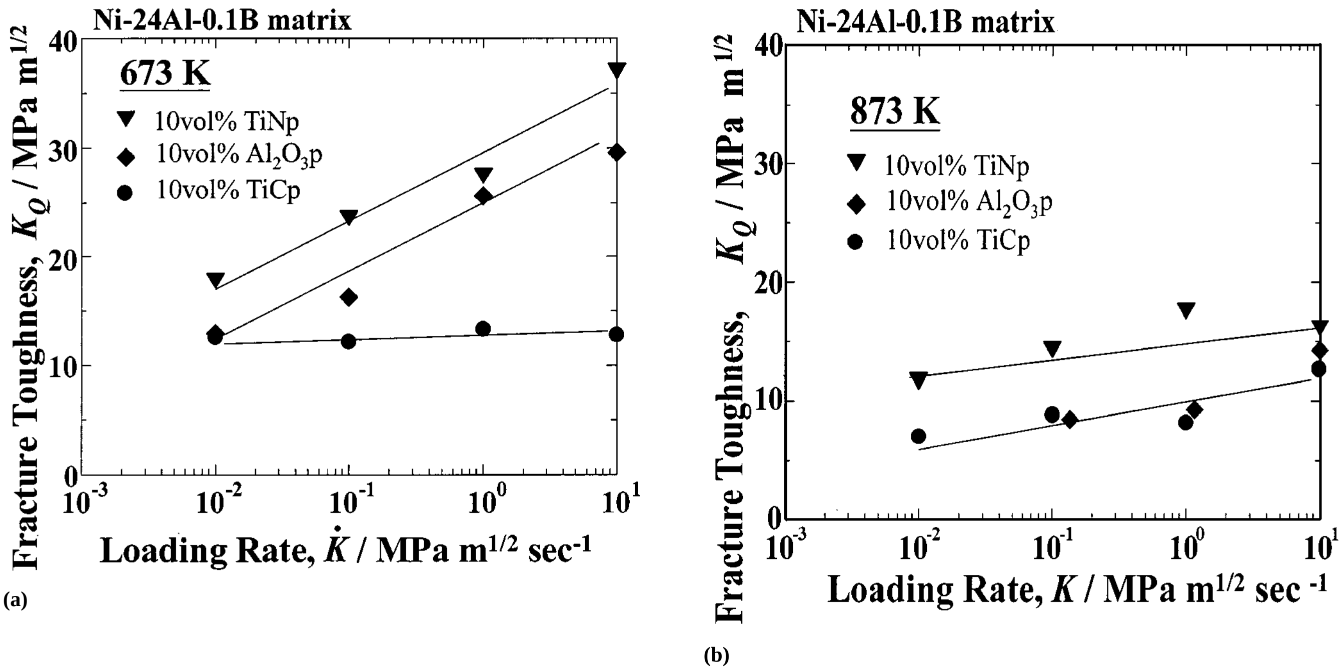

Fig. 5 Loading-rate dependence of fracture toughness of Ni-24 at.% Al-0.1 at.% B matrix composites at (**a**) 673 and (**b**) 873 K in air

with increasing temperature up to ~800 K, corresponding to the yield stress anomaly. The composites fracture in a brittle manner at 773 K. Subsequently, the ductility gradually increases with decrease in the 0.2% proof stress and the ultimate strength above 900 K.

For composites with TiN particles, the tensile strength and ductility are higher than those with α-Al_2O_3, as illustrated in Fig. 4(b). Although the composites do not fracture in a brittle manner, the ductility decreases at intermediate temperatures. On the other hand, the tensile strength of the composite with TiC particles is remarkably higher than those of the other composites. However, the composites reveal brittle fracture below 900 K, as shown in Fig. 4(c). The magnitude of the strengthening effect is quite different, depending on the kind of reinforcement particles. The chemical effect of a small amount of constitutional elements dissolved in the matrix is suggested to contribute to the mechanical behavior of the composites as well as to the physical reinforcing mechanism such as the pinning effect against dislocation motion.

The fracture toughness of the composites, except that of the TiC composites, can be improved by B doping concurrently with suppression of moisture-induced embrittlement as described in a previous paper.[15] The present paper focuses on their toughness in temperature region II. Figure 5(a) and (b) show the fracture toughness of the Ni-24 at.% Al-0.1 at.% B matrix composites with 10 vol.% reinforcement particles at 673 and 873 K, respectively. Their toughness values at a loading rate of 10 MPa $m^{1/2}$ s^{-1} at 673 K are almost the same as those at 300 K. Thus, their toughness does not intrinsically decrease at 673 K. However, the toughness of the TiN and α-Al_2O_3 composites shows significant loading-rate dependence in air. Therefore, the dynamic embrittlement caused by diffusion of oxygen is the predominant mechanism governing their fracture toughness at 673 K. However, the toughness of the composites decreases intrinsically at 873 K, in a manner similar to the monolithic alloy.

The toughness of the TiC composites is exceptionally constant regardless of the loading rate, as shown in Fig. 5(a) and (b). Furthermore, the toughness at these temperatures approximately coincides with that at 300 K. Thus, the toughness of the TiC composites is unrelated to the environmental effect and the characteristic dislocation motion at intermediate temperatures. As a result of the remarkable strengthening with the addition of TiC particles, the dislocation motion should be intrinsically restricted. Hence, the fracture toughness of the TiC composites is constant in the temperature range of 300 to 900 K.

4. Concluding Remarks

The mechanical behavior of Ni_3Al alloys and their matrix composites with ceramic particles has been investigated in several environments. The loading rate dependence of fracture toughness clearly distinguishes the environmental effect from simultaneous intrinsic mechanisms. The results of the present work are summarized below.

- The environmental embrittlement of the alloys at ambient temperatures is inhibited by 0.1 at.% B doping as well as the enhancement of inherent grain boundary cohesion. However, grain boundary strengthening is the predominant toughening mechanism at ambient temperatures.
- The fracture toughness of the alloys is governed by the dynamic embrittlement promoted by the diffusion of oxygen at 673 K. As the temperature increases, the intrinsic brittleness induced by the dislocation locking mechanism becomes the main factor determining their fracture toughness.

- The composite material design by using ceramic particles is effective in improving their strength. The strengthening effect is quite different, depending on the kind of reinforcement. The TiN composites exhibit higher strength and ductility than the α-Al_2O_3 reinforced ones. The TiC composites are inherently brittle concurrently with remarkable strengthening.
- The mechanisms governing the fracture toughness at intermediate temperatures of TiN and α-Al_2O_3 composites are similar to those of the monolithic alloy. However, the fracture toughnesses of the TiC composites do not vary at 300 to 900 K.

Acknowledgments

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