



A Study on Ductilization of Titanium Aluminides at Elevated Temperatures

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The effect of slight exposures in air on the ductility of TiAl and Super α_2 was studied. It was found that embrittlement occurred only in the low-temperature range where they fracture by transgranular cleavage with low ductilities. The ductility decreased to 0.3 % from the original values of 1 and 2.2 % for TiAl and Super α_2 , respectively. The minimum ductility was obtained after a short-term oxidation. Numerous steps, probably due to twins and slips, were observed at the bent surface. The ratio of cracked to uncracked steps was large in severely embrittled specimens. It was concluded that the formation of cracked steps due to a thin oxide film has brought about the premature fracture.

Keywords: Ductility, TiAl, Transgranular cleavage, Oxidation.

INTRODUCTION

Titanium alloys are widely used in aircraft engines due to their low density with high strength [1-5]. Current alloy development has increased their usable temperatures up to 873 K. But, these conventional alloys are considered to be facing with limitations in the temperature capabilities not only from high temperature strength but also from oxidation resistance. Oxidation resistant titanium aluminides offer an opportunity to increase the service temperature of this light weight material.

Super α_2 is a sophisticated Ti_3Al -type aluminide with an improved resistance to oxidation and creep. γ -TiAl is another interesting aluminide, having a lower density and higher temperature capabilities than α_2 -type aluminides. Major concerns about these aluminides are relatively low ductility and low toughness at and below intermediate temperatures.

Further characterizations will be required for these high-temperature materials to be understood their potentials and limitations in applications. Poryadchenko *et al.* [6] have reported that pre-exposure in air has brought about enhanced surface cracking and premature fracture in the tensile tests of Super α_2 at room temperature. In the present paper, this sort of embrittlement problem was extended to include another aluminide of TiAl and also to cover a wide range of deformation temperatures.

EXPERIMENTAL

About 1 kg of a pancake-shaped ingot was button-arc melted. The chemical composition was Ti-33.4Al-0.084O (wt

%). Four-point bend specimens were sampled from the as-cast ingot such that the longitudinal axis of bend coupons lay parallel to the circumference of the ingot. The specimens of 3 mm $\times$ 4 mm $\times$ 40 mm were bent at a nominal strain rate of $1.5 \times 10^{-4} \text{ s}^{-1}$ in air at 973 K. A tension side of the bend specimens was polished before pre-exposure in air for metallographic observations.

Forged Super α_2 bars with a composition of Ti-13.8Al-20.0Nb-2.63V-2.1Mo-0.046O (wt %) were β -solution treated at 1473 K for 1.8 ks. They were subsequently stabilized at 1033 K for 65 ks followed by cooling in air. Tensile tests were done using the specimens of 25 mm in gauge length and 4 mm in diameter at a strain rate of $1.2 \times 10^{-4} \text{ s}^{-1}$. Several specimens of 6 mm in diameter were tested also in vacuum.

Microstructures of these materials are shown in Fig. 1. TiAl has a lamellar structure of α_2 in γ -phase matrix with a grain size of several hundred μm . Super α_2 consists of a coarse Widmanstätten α_2 surrounded continuously by β -phase with mixed grains of about 1 to 2 mm and 0.3 to 0.6 mm in size.

RESULTS AND DISCUSSION

Brittle to ductile transition: The temperature dependence of fracture strains of Super α_2 and TiAl is shown in Fig. 2. Ductilization of Super α_2 occurred at much lower temperatures than that of TiAl, which will be favourable point as a structural material. TiAl still behaved in a brittle manner at 873 K.

The deformation behaviour of Ti-48at %Al, essentially the same as for the present test material, has been reported by several authors [7,8]. They observed a high density of primarily

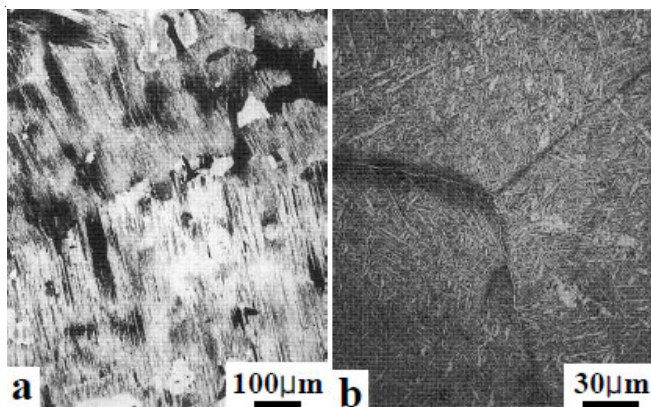


Fig. 1. Microstructures of test materials; (a) TiAl, (b) Super α_2

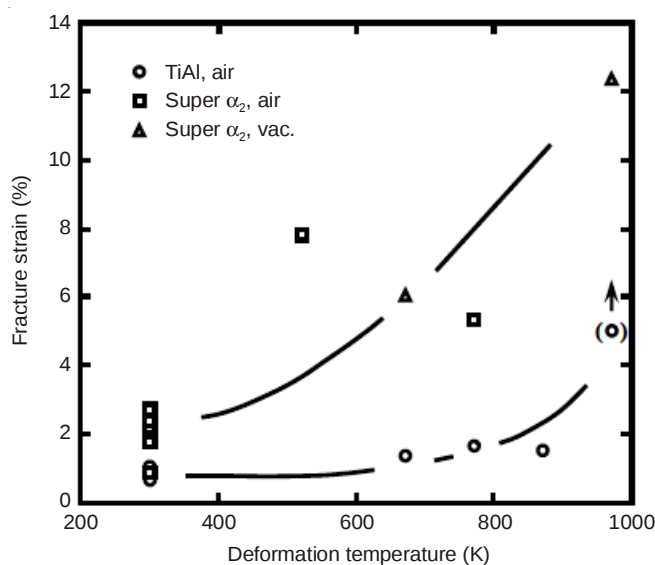


Fig. 2. Temperature dependence of fracture strains of TiAl and Super α_2

64 $1/2[\bar{1}10]$ screw dislocations and twins in the specimens
 65 deformed at room temperature and elevated temperatures up
 66 to 1033 K. The predominant deformation modes remained the
 67 same, but only the extent of twinning and slip became large
 68 at elevated temperatures. For Super α_2 , a high degree of slip
 69 planarity was observed at room temperature. But, for higher
 70 temperature creep, cross-slip between the basal, prismatic and
 71 pyramidal planes becomes easy and dislocation distributions
 72 became homogeneous [9].

73 The fracture appearance has changed from transgranular
 74 cleavage to intergranular fracture with the increase in deformation
 75 temperatures. Fig. 3 shows fracture surface of Super α_2
 76 deformed at 673 K and 973 K. Intergranular fracture occurred
 77 at 973 K, while transgranular cleavage was observed at room
 78 temperature. A mixed fracture-mode was observed at the intermediate
 79 temperature of 673 K. These specimens were tensile
 80 tested in vacuum. This transition of the fracture mode was
 81 observed only when tested in vacuum. Oxidation-affected
 82 surface crack worked as notches in air and changed the fracture
 83 mode at 973 K from the intergranular to transgranular fracture.
 84 This sort of atmospheric effect has previously been reported
 85 in $\text{Ti}_3\text{Al-Nb}$ [10,11].

86 Fig. 4 shows the bending surface of the TiAl specimen
 87 fractured at 773 K, observed by an optical microscope. The

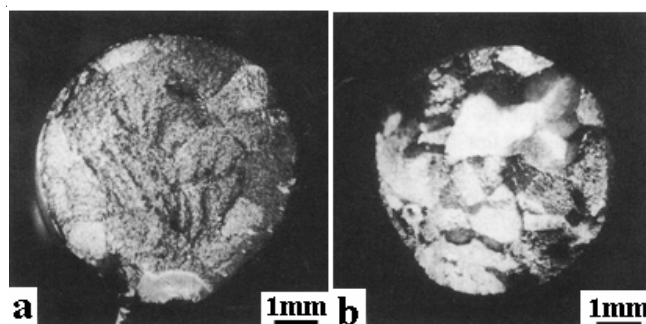


Fig. 3. Fracture surface appearance of Super α_2 tensile-tested in vacuum at (a) 673 K, mostly transgranular and (b) 973 K, intergranular

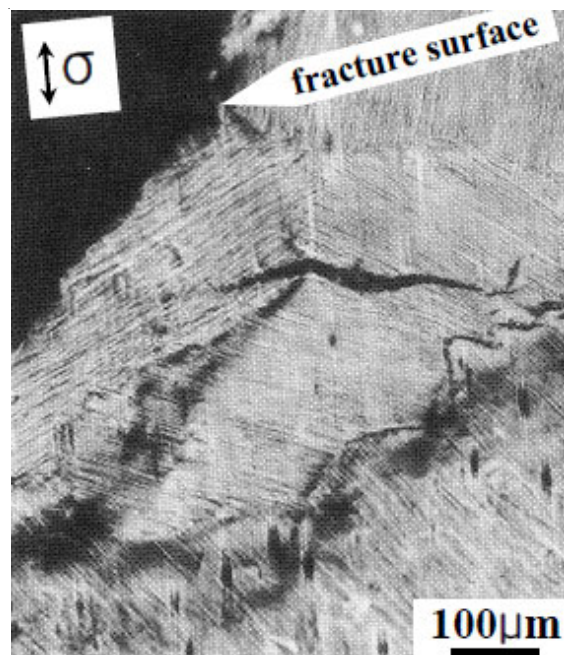


Fig. 4. Thermally etched surface of TiAl bent at 773 K. Both transgranular cleavage cracks and grain boundary cracks are observed

88 lamellar structure was revealed by thermal etching during the
 89 tensile test in hot air. Transgranular cracks crossing the grain
 90 boundary and intergranular cracks were found to be formed
 91 at intermediate deformation temperatures.

92 Fig. 5 is a schematic illustration of the temperature dependence
 93 of cleavage and intergranular fracture stresses. This figure has been
 94 used in explaining ductile-brittle transitions. At lower temperatures
 95 strain hardening associated with a small extent of yielding makes
 96 the applied stress reach to the cleavage fracture stress. At elevated
 97 temperatures the decrease in yield stress allows relatively extensive
 98 yielding before fracture. The grain-boundary cohesive strength
 99 becomes lower than the transgranular strength at elevated
 100 temperatures and, consequently, intergranular fracture appears
 101 at these temperatures.

102 **Deformation-temperature dependence of embrittlement**
 103 **of TiAl by air-exposure:** The TiAl bend coupons were exposed
 104 in air at 1073 K for 1.8 ks. Bend tests of the virgin and oxidized
 105 materials were done in air over the temperature range between
 106 300 and 973 K. As shown in Fig. 6, pre-oxidation degraded
 107 the ductility only in the low-ductility temperature range where
 108 transgranular cleavage fracture dominates. The pre-exposure
 109 effect disappeared at 773 K, where the mixed fracture mode

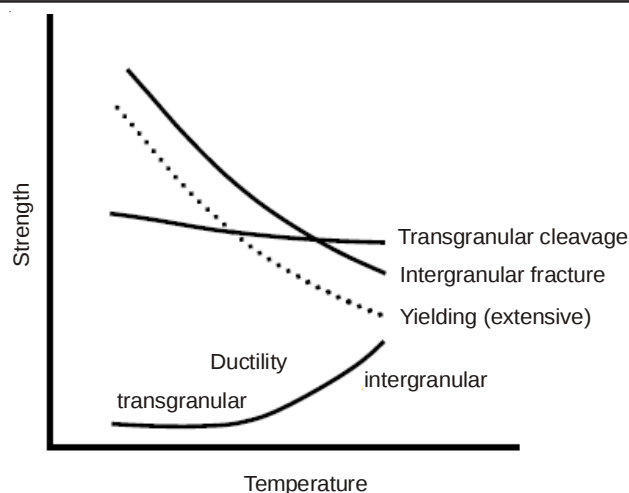


Fig. 5. Schematic illustrations of temperature dependences of cleavage and intergranular fracture stresses.

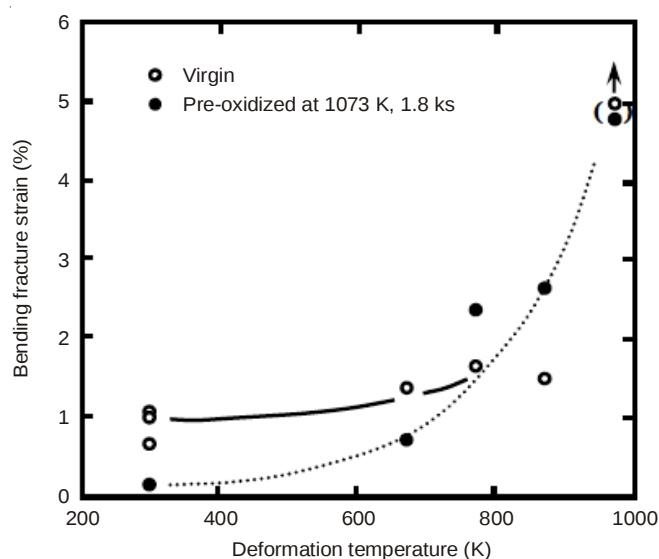


Fig. 6. Effect of pre-exposure at 1073 K for 1.8 ks in air on the bending-fracture strain of TiAl

110 appeared as shown in Fig. 4. A few tensile tests done at 973 K
 111 for Super α_2 showed little pre-exposure effects, confirming
 112 the above conclusion.

113 Exposure at 1073 K for 1.8 ks formed only a thin oxide
 114 film of about 1 μm in thickness. Numerous surface cracks
 115 running normal to the bend axes, which is shown in Fig. 10,
 116 were found for all the test temperatures of the pre-oxidized
 117 specimens. Spalling of oxide scales, as shown in Fig. 7, were
 118 found in the specimens deformed at 773 K or lower. The oxide
 119 film is considered to become virtually plastic above 873 K.
 120 Although the strain to fracture increased at higher deformation
 121 temperatures, plasticity of the oxide films has prevented
 122 the spalling of oxide films. However, the surface cracks were
 123 observed even at these elevated-temperatures. Therefore, the
 124 enhanced plasticity of oxide films cannot be indebted to the
 125 insensitiveness to embrittlement at higher deformation tempe-
 126 ratures.

127 **Effect of exposure conditions on embrittlement at room**
 128 **temperature:** Fig. 8 shows how the tensile elongation decreased
 129 with the oxidation time and temperature. The oxidation time

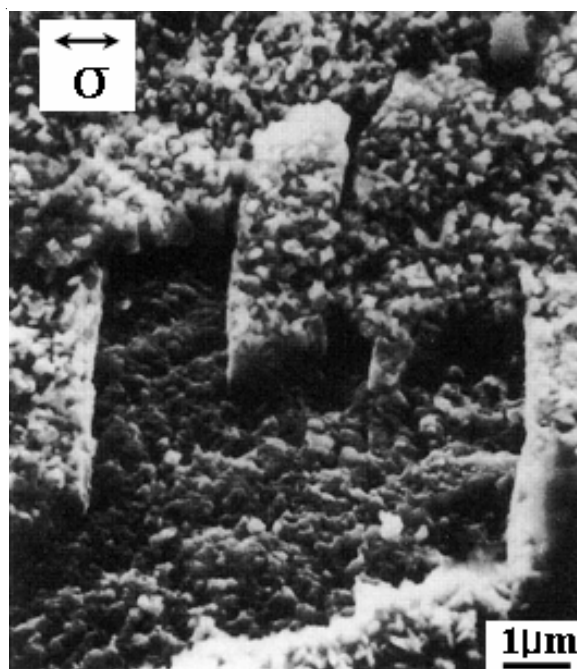


Fig. 7. Spalling of oxide scale by the bend test at room temperature of TiAl pre-exposed at 1073 K for 1.8 ks

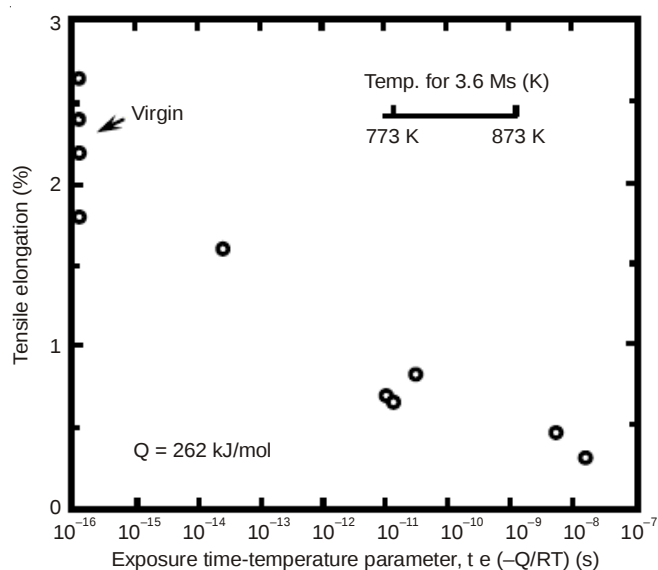


Fig. 8. Decrease in tensile elongation of Super α_2 as a function of exposure time-temperature parameter

130 was varied from 0.9 ks to 1080 ks at 813 K and 973 K. Here,
 131 the activation energy for oxidation of Ti_3Al [12], 262 kJ/mol,
 132 was used for the Q -value in $\exp(-Q/RT)$. The elongation severely
 133 and continuously decreased to 0.3 % from the original value
 134 of about 2 to 2.5 % with the increase of the parameter.

135 The similar tests were done using TiAl. Variations of the
 136 bending fracture strain at room temperature with pre-exposure
 137 temperatures between 773 and 1373 K are shown in Fig. 9.
 138 The exposure time was 1.8 ks. Embrittlement became severe
 139 at higher exposure temperatures and is likely to converge to
 140 0.3 %. Also shown in the figure is the oxide-film thickness
 141 measured by SEM. It is shown that a very thin oxide film of
 142 less than 0.1 μm was enough to embrittle the room temperature
 143 ductility of TiAl. Higher-temperature exposures will produce

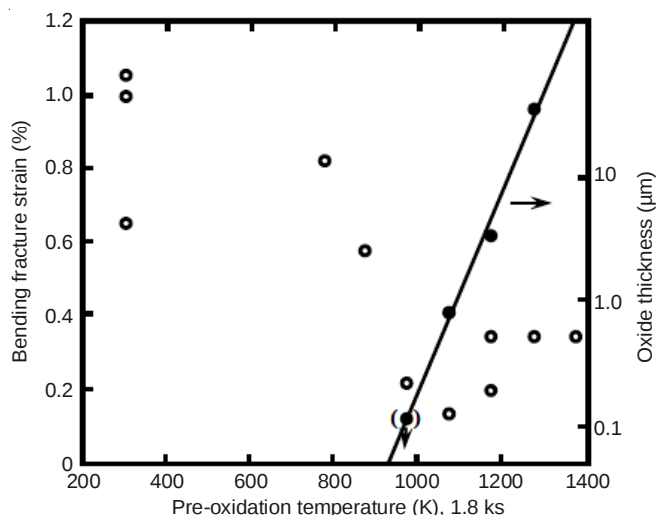


Fig. 9. Isochronic embrittlement and oxide-scale growth curves of TiAl (1.8 ks)

not only a thicker oxide film but also a deeper oxygen penetration. However, the fracture strain at 973 and 1173 K were almost the same. From these findings the embrittlement is considered to be related to the surface film effect rather than the semi-bulk effect due to oxygen penetrations. Dowling and Donlon [13] have observed the same degree of embrittlement in on extruded Ti-48Al-IV-0.2C (at %) as in the present work, but they reported that its cause was the formation of a brittle surface layer with a new phase having a Ti:Al ratio of 2:1.

Numerous lines were observed through SEM on the bent surface. Fig. 10(a) shows the surface of the virgin material fractured at a strain of 1 %. These lines are considered as steps produced by twinning and slip. Some of them, particularly near the fracture face, opened their mouths and formed surface cracks running normal to the bend axis. When interrupted at 0.4 % bend strain, only a few cracks were observed. However, much more cracks were observed on the bent surface of pre-oxidized samples which fractured at the same strain, as shown in Fig. 10(b). The ratio of cracked to uncracked steps increased with the increase of exposure temperatures. Almost the same observations were made in Super α_2 . The depth of the surface cracks was at most 20 to 30 μm for Super α_2 , while less than 10 μm in depth was observed for TiAl. But, these are still greater than the thickness of oxide scales.

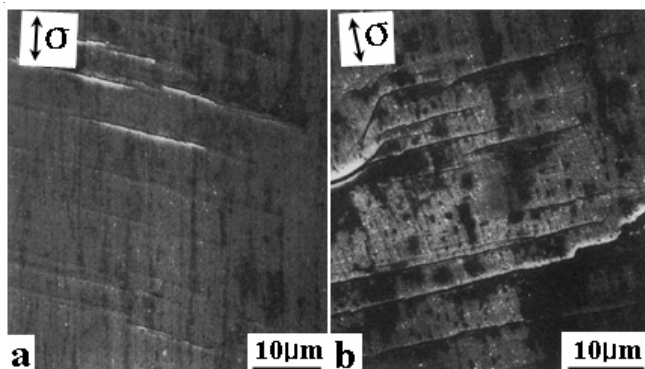


Fig. 10. SEM observation of the bent surface of TiAl fractured at room temperature; (a) virgin, $\epsilon_f = 1\%$, (b) pre-oxidized at 873 K, $\epsilon_f = 0.4\%$

Conclusion

Ductilization was observed in TiAl and Super α_2 at elevated temperatures. The fracture mode has changed from transgranular cleavage to intergranular fracture with the increase in temperature. Embrittlement by a slight oxidation in air was observed only in the low-temperature range where they fracture by transgranular cleavage. Severe embrittlement was observed after a mild oxidation in both aluminides. Numerous steps, probably due to twins and slips, were observed at the bent surface. The ratio of cracked to uncracked steps was large in the severely embrittled specimens. Formation of the cracked steps due to a thin oxide film is considered to have brought about the premature fracture.

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