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High temperature fatigue behaviour of intermetallics

K BHANU SANKARA RAO

Mechanical Metallurgy Division, Indira Gandhi Centre for Atomic Research, Kalpakkam 603 102, India
e-mail: bhanu@igcar.ernet.in

Abstract. There would be considerable benefits in developing new structural materials where high use temperatures and strength coupled with low density are minimum capabilities. Nickel and titanium aluminides exhibit considerable potential for near-term application in various branches of modern industry due to the number of property advantages they possess including low density, high melting temperature, high thermal conductivity, and excellent environmental resistance, and their amenability for significant improvment in creep and fatigue resistance through alloying. Reliability of intermetallics when used as engineering materials has not yet been fully established. Ductility and fracture toughness at room and intermediate temperatures continue to be lower than the desired values for production implementation. In this paper, progress made towards improving strain-controlled fatigue resistance of nickel and titanium aluminides is outlined. The effects of manufacturing processes and micro alloying on low cycle fatigue behaviour of NiAl are addressed. The effects of microstructure, temperature of testing, section thickness, brittle to ductile transition temperature, mean stress and environment on fatigue behaviour of same γ-TiAl alloys are discussed.

Keywords. Nickel aluminides; titanium aluminides; low cycle fatigue; micro-alloying.

1. Introduction

Ordered intermetallic alloys have undergone extensive development over the past two decades due to their potential as structural materials in gas turbine engines for future fighter and commercial aircraft (Anton & Shah 1990; Noebe *et al* 1993; Darolia 2000). Of the many intermetallic alloys under initial consideration, nickel aluminides, and γ-titanium aluminides are the few systems that have emerged as promising candidates for further development. This is due to the number of advantages they have in properties including low density, high melting temperature, high thermal conductivity, and excellent environmental resistance, and their potential for significant improvment in creep resistance through alloying (Darolia 1991; Lerch & Noebe 1993; Miracle 1993; Recina 2000). However, the B2 nickel aluminides and γ-titanium aluminides are challenged by lack of ambient temperature ductility and toughness. These deficiencies severely limit possible applications and impose limits on the fabrication and forming of these materials. The lack of ductility at room and intermediate temperatures has been attributed to an inherently low cleavage stress, which is partly attributed to a low mobile dislocation density, and the inability to initiate a sufficient number of independent slip systems to satisfy the Von-Mises criterion for generalized plasticity of a polycrystalline aggregate (Noebe *et al* 1993). At elevated temperatures and moderate strain rates, tensile ductility is no longer a serious limitation. Nickel and titanium aluminides undergo a dramatic brittle-to-ductile transition at elevated temperatures with attendant increases in tensile elongation, fracture strength and fracture toughness. Several studies have suggested that thermally activated deformation processes, such as localized dislocation climb, provide the additional deformation mechanisms necessary to maintain grain-boundary compatibility, thus contributing to the sharp transition from brittle to ductile behaviour. Some nickel aluminide and γ-titanium aluminide-based alloys are currently under further development to overcome these ductility and toughness problems and bring the candidate materials closer to applications in advanced aero-engines, automotive parts and land-based gas turbines. Low fracture toughness, high-brittle to ductile transition temperature (BDTT), and severe strain rate dependence continue to be of concern when intermetallics are considered as replacements for ductile, high toughness Ni-base superalloys. In general, other problems in implementing aluminides in actual applications include the difficulty in controlling and reproducing the required microstructure during the manufacturing process (Recina 2000). Relatively small variations in chemical composition, as well as actual casting and heat treatment procedures may have considerable effect on the microstructure and, thus, on the resulting properties (Recina & Karlson 1999). Closer control of the microstructure in γ-titanium aluminide leading to lower scatter in mechanical properties is sometimes possible by forging and by introducing grain-refining agents when casting (Christodoulou *et al* 1988; Kumar & Whittenberger 1992). Since intermetallics are intended for use in conditions where they undergo not only mechanical loading, but also change in temperature, their isothermal and thermomechanical fatigue behaviour is of particular interest (Christ *et al* 2001). In this paper, progress towards understanding the low cycle fatigue behaviour of intermetallics is reviewed. Special attention has been paid to describe the effects of processing route, micro alloying, microstructure and test temperature on low cycle fatigue behaviour of B2 NiAl and γ-titanium aluminides.

2. Effects of manufacturing processes on strain controlled low cycle fatigue behaviour of polycrystalline NiAl

The effects of processing route on strain-controlled LCF behaviour of B2 NiAl have been examined in detail at 1000 K (Lerch & Noebe 1994; Bhanu Sankara Rao *et al*1995). The NiAl samples were produced by three different processing routes: hot isostatic pressing (HP) of pre-alloyed powders, extrusion of pre-alloyed powders (PE), and extrusion of vacuum induction-melted ingots (CE). The average chemical composition and grain size of NiAl obtained at the end of different processing routes are given in table 1. The HP alloy has larger grains and larger variation in grain size compared to the fully recrystallized extruded materials, CE and PE. The hot isostatically pressed (HP) material contains prior particle boundaries that are composed of narrow stringers of fine Al_2O_3 particles. The CE material consists of internal voids, which are aligned parallel to the extrusion axis. Despite the 12:1 reduction ratio during extrusion, the CE alloy possesses residual porosity, owing to the presence of entrapped gas. The PE alloy is fully consolidated with fewer defects than the CE or HP material.

The effect of processing route on strain-controlled low cycle fatigue (LCF) life of binary NiAl at 1000 K is depicted in figure 1 (Bhanu Sankara Rao *et al* 1995). The LCF behaviour of

Table 1. The chemical composition and grain size of NiAl processed through different manufacturing processes.

Fabrication method	Grain size (μm)	Composition (at.%)					
		Ni	Al	O	C	S	N
HP-hipped powder (Heat P1418)	70 ± 14	$50{\cdot}5^*$	$49{\cdot}5^*$	0·028	0·014	$< 0{\cdot}002$	0·0006
PE-extruded powder (Heat P2098)	12 ± 3	$50{\cdot}2^*$	$49{\cdot}7^*$	0·025	0·017	$< 0{\cdot}0017$	0·0009
CE case + extruded	18 ± 3	50^{**}	50^{**}	0·011	0·029	$< 0{\cdot}002$	$< 0{\cdot}003$

* Determined by wet chemical analysis methods, $\pm 0{\cdot}2$ at. % within a 95% confidence interval
** Nominal composition

the CE material determined at room temperature (below the BDTT) is also included in figure 1 for comparison with the high temperature data. CE and PE alloys exhibit similar lives under identical strain ranges. The HP alloy displays shorter lives than the extruded materials, with an increasing difference in life at low strain ranges. Detailed understanding of the various factors responsible for the shorter life of HP material has been developed on the basis of the cyclic stress response behaviour, and prevailing deformation, damage and fracture modes at elevated temperature. Typically, the alloy under all processing conditions exhibits relatively little cyclic hardening or softening at 1000 K. However, the HP alloy displays a higher cyclic stress response over the entire life range compared to the extruded alloys as shown in figure 2. In the

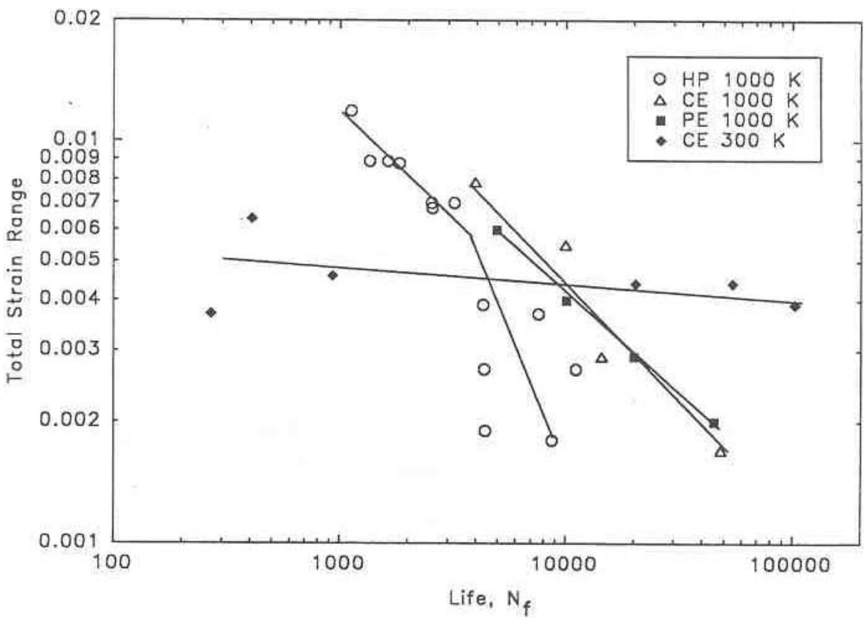

Figure 1. Effect of processing route on strain controlled LCF behaviour of binary NiAl (Bhanu Sankara Rao *et al* 1995).

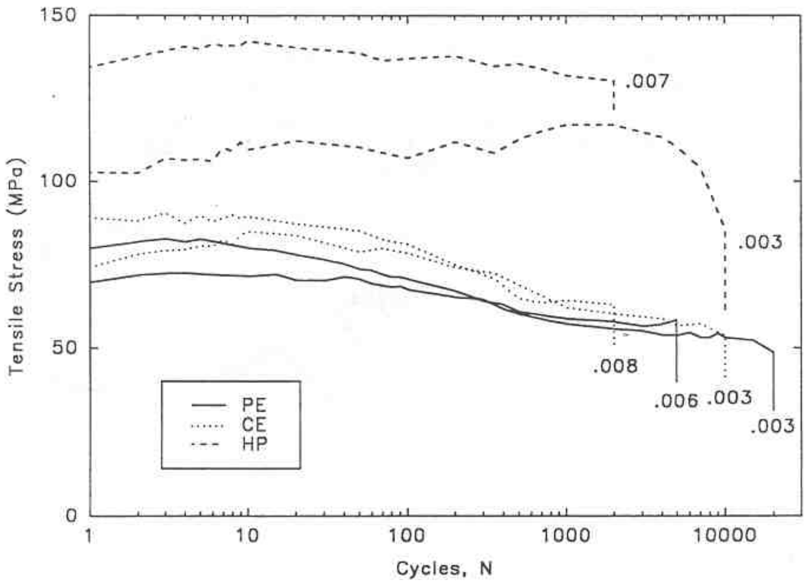

Figure 2. Effect of processing route on cyclic stress response of NiAl at 1000 K (Bhanu Sankara Rao *et al* 1995).

HP alloy, the deformation substructure is composed primarily of dislocation tangles, loops and dipoles, while in contrast the substructure in CE and PE materials consists of dislocation cells. The HP alloy shows lower tendency to form dislocation cells or sub grains suggesting that dislocation cross slip and climb are inhibited. Since the HP alloy has a significantly higher BDTT than the other two alloys, recovery processes are expected to be fewer, leading to higher dislocation density and a more homogeneous dislocation structure in the HP material. The differences in cyclic stress response of the three materials results primarily from the variation in substructure.

All the three alloys exhibit intergranular crack initiation and propagation prior to the onset of transgranular cleavage overload failure. The HP alloy has much smaller zone of slow and stable intergranular crack growth compared to the extruded alloys. The high response stresses in the HP alloy could lead to large stress concentration at the crack tip, which would account for increased crack growth rates and hence reduced number of cycles in crack propagation stage. Higher response stresses would also act to reduce the critical crack size for final fracture reducing the number of cycles to failure. The relatively fast and unstable transgranular cleavage sets in much earlier in the HP alloy. In the case of HP alloy, samples cycled at low strain ranges exhibit much shorter lives than would be expected by extrapolation from high strain range regime of the strain-life plot, figure 1. This type of discontinuity in strain-life plots has also been observed in several nickel-base superalloys (Lerch & Gerold 1985; Valsan *et al* 1989), aluminum alloys (Sanders & Starke 1976), magnesium alloys (Wareing *et al* 1973) and dual phase-steels (Mediratta *et al* 1986). The two-slope behaviour in strain-life plots has been correlated with: (a) the differences in crack initiation and propagation modes at low and high strain ranges, (b) the differences in deformation mechanisms, and (c) the synergistic interaction between fatigue and various time- and temperature-dependent damage processes. The discontinuity in the strain-life curve in the HP alloy occurs as a consequence of the operation of different damage mechanisms in the high and low life regimes (Bhanu

Sankara Rao *et al* 1994, 1995). In the low life regime (high strain ranges), there is no major interaction between fatigue and other time-dependent processes such as creep and oxidation, and the life is dictated by fatigue crack initiation and growth. In the long life regime (low strain ranges), creep and environmentally activated damage micro mechanisms combine with fatigue damage and lead to drastic reduction in life. All the creep cavities on grain boundaries of the HP binary alloy are rounded in shape (r-type) with no evidence of triple-point wedge cracking. Creep cavities in the HP condition are believed to be associated with the alumina particles that are present in the grain boundaries (Bhanu Sankara Rao *et al* 1995). It is well-established that the secondary phases in the grain boundary can provide suitable nucleation sites for intergranular cavitation (Raj 1978; Bhanu Sankara Rao 1989; Rodriguez & Bhanu Sankara Rao 1993). These isolated cavities grow by the diffusional transport of vacancies or by deformation of the matrix material and, therefore, do not necessarily require grain boundary sliding. Same alumna particles are located at the grain boundaries in the extruded NiAl, which reduce the amount of creep cavities in this material, and therefore, the life of the extruded alloy at low strain ranges is not affected like in the HP alloy and does not display the bilinear fatigue life behaviour. These results indicate that the oxide particles in the HP samples are a major source of concern. These significant contributed to the large amount of scatter observed in life at low strain ranges (figure 1). It has been suggested that strict control over cleanliness of the powder needs to be exercised in order to improve the fatigue life in alloys processed by the HP route.

Comparative evaluation of fatigue life of NiAl and the lives of various turbine disk and blade alloys at temperatures of approximately 1000 K are made in figures 3 and 4. Binary NiAl exhibited superior fatigue life when compared to most superalloys on the plastic strain basis (figure 3), but was inferior to most superalloys on the stress basis (figure 4). The poor performance of binary NiAl on a stress range basis is due to its low yield strength compared to the superalloys. In view of this attempts have been made to improve the strength of the alloy by alloying with nitrogen as well as zirconium.

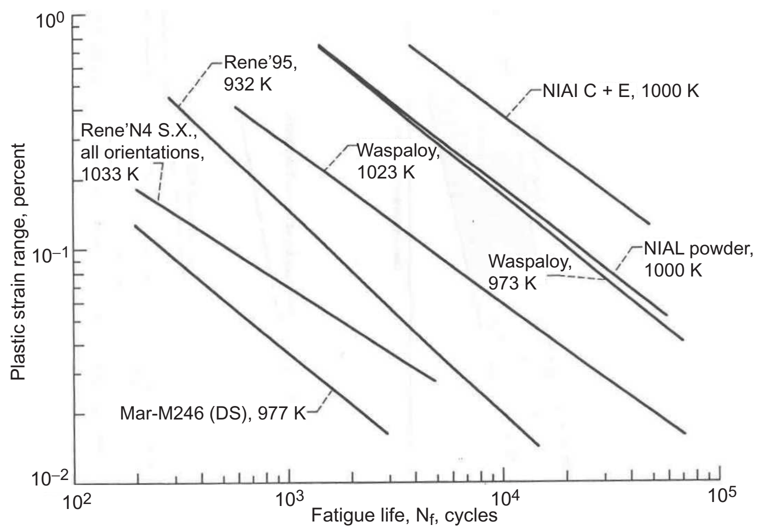

Figure 3. Fatigue life of NiAl compared to Ni-based superalloys on plastic strain range basis at nominally 1000 K (Lerch & Noebe 1993).

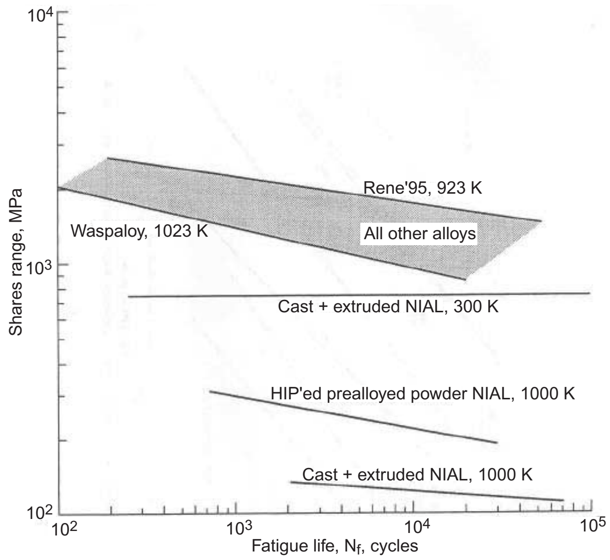

Figure 4. Fatigue life of NiAl compared to Ni-based superalloys on stress range basis at nominally 1000 K (Lerch & Noebe 1993).

3. Micro alloying effects on the high temperature fatigue behaviour of NiAl

The effects of the addition of 0·1 at% of nitrogen and zirconium on fatigue resistance of NiAl at 1000 K are depicted in figure 5 (Bhanu Sankara Rao *et al* 1994; Noebe *et al* 1995). The total strain fatigue resistance of NiAl (N) is higher than that of binary NiAl in the range of strain

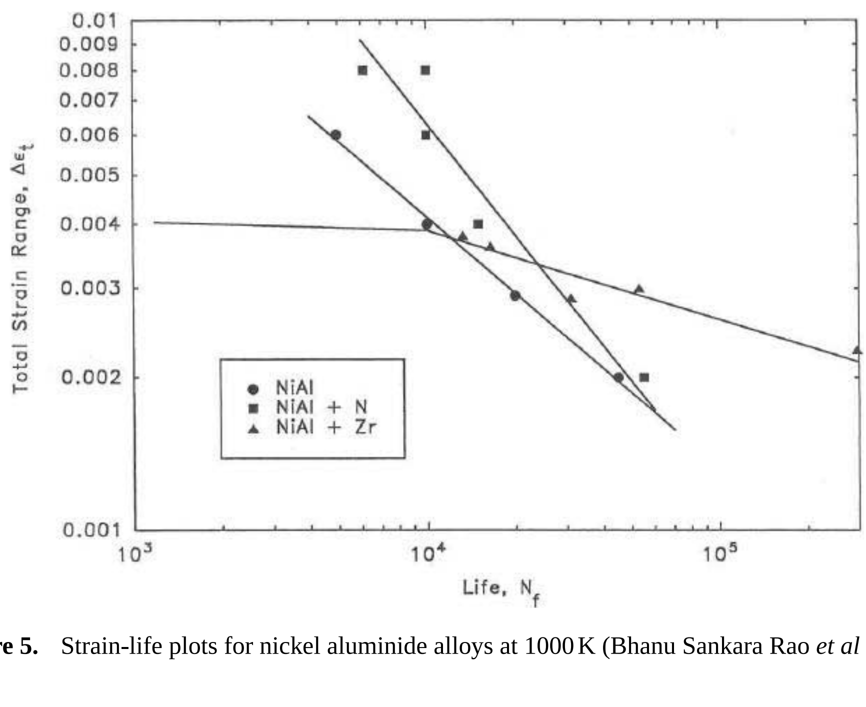

Figure 5. Strain-life plots for nickel aluminide alloys at 1000 K (Bhanu Sankara Rao *et al* 1994).

amplitudes examined. Zirconium additions are even more beneficial for fatigue life at total strain ranges below 0·38%. On increasing the strain from 0·38% to 0·40%, the Zr-doped alloy shows a drastic reduction in life. There are clear differences in the stress response behaviours of binary, nitrogen-doped and Zr-doped alloys (figure 6). Zirconium-doped alloy displayed much larger response stresses than the other two alloys. NiAl (Zr) exhibits a short period of cyclic hardening and attains a maximum stress in the very early stages of cyclic life. Beyond the maximum stress value, a gradual softening takes place prior to the regime of nearly stable stress response. Also the initial hardening period is prolonged with decreasing strain range. Conversely, NiAl and NiAl (N) experience cyclic softening initially. This is followed by a period of stable stress response that continues until the tensile stress decreases rapidly due to the formation of microcracks and their subsequent growth, which immediately precede failure. The LCF test temperature is slightly below the BDTT of NiAl (Zr) (1100 K) but much higher than the BDTTs of NiAl (600 K) and NiAl (N) ($\sim$ 620 K). The LCF behaviour of these alloys is significantly influenced by the relation between the test temperature and their respective BDTTs. Since the BDTT of NiAl (Zr) is higher than test temperature, substructural recovery is not significant and this leads to more homogenous distribution of dislocations within the grains. Additionally, Zr atoms are very effective in pinning dislocations below the BDTT and increasing the viscous drag force on dislocations at higher temperatures. Consequently, the mutual interactions between dislocations and solid solution hardening due to Zr atoms result in higher cyclic stress response. Because the BDTT of the binary and N-doped alloys is much lower than the test temperature, significant recovery is able to occur during testing resulting in lower response stresses. The marginal improvement in cyclic stress response of the N-doped alloy over the binary NiAl has been associated with dislocation pinning by the finely dispersed AlN particles in the former alloy.

The two stages in the strain-life plot (figure 5) of NiAl (Zr) occur as a consequence of the change in fracture behaviour from slow and stable intergranular crack growth at strain ranges $\leq$ 0·38% to brittle cleavage-dominated overload fracture at high strain ranges. At strain ranges above 0·38%, the peak tensile stress reaches the monotonic cleavage fracture stress of the Zr-doped alloy in less than 100 cycles, enabling fast crack growth by transgranular cleavage. At strain ranges less than 0·38%, the peak tensile stresses remain at much lower level than the cleavage fracture stress. In general, fatigue lives are governed by the ductility of

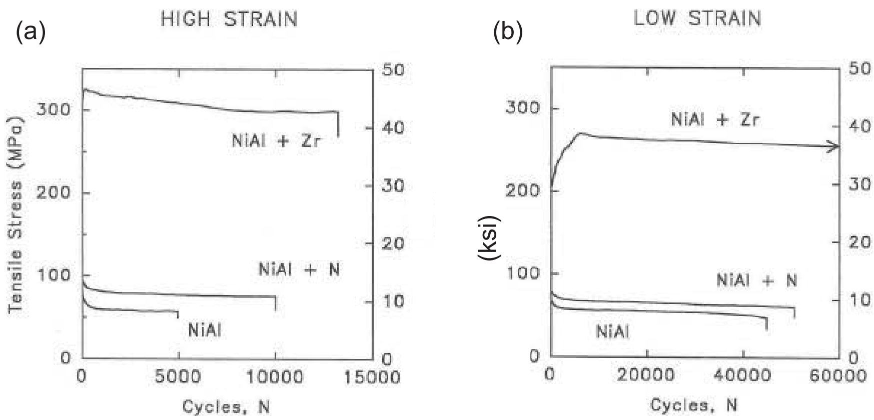

Figure 6. Typical stress response curves for NiAl alloys at high **(a)** and low **(b)** strain ranges (Bhanu Sankara Rao *et al* 1994).

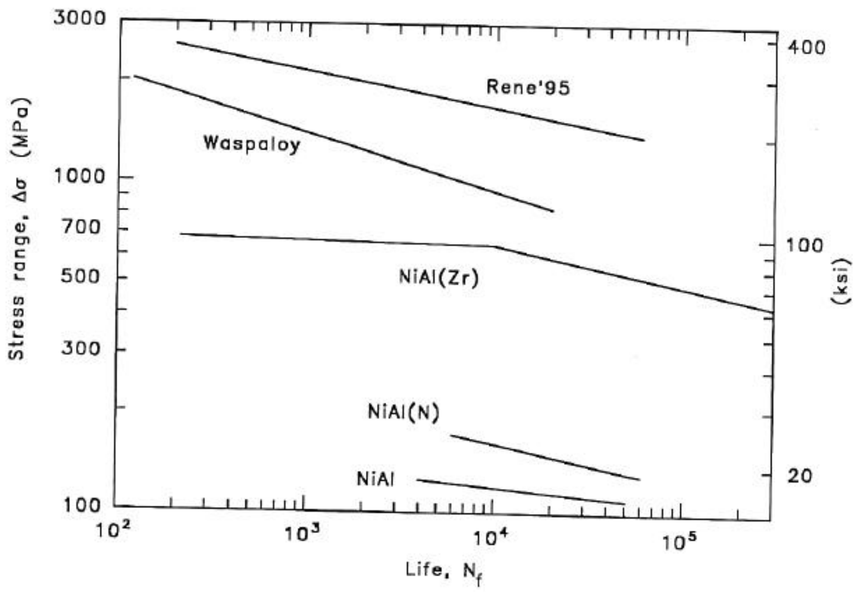

Figure 7. Comparison of fatigue lives of superalloys and NiAl alloys on stress range basis (Bhanu Sankara Rao *et al* 1994).

the material at high strains and by its strength at low strains. The longer lives of NiAl (Zr) at low strain ranges result from its basic capacity to resist the applied strains on the basis of high strength. Both NiAl and NiAl (N) have shorter lives at low strain ranges due to the synergistic interaction between fatigue and creep. A higher slope of the strain range-life plot of NiAl (N) reflects this interaction. In order to improve the fatigue resistance of the N-doped alloy, grain boundaries have to be strengthened to reduce grain boundary sliding and the associated intergranular wedge cracking that is observed in these two alloys. Since Zr segregates to the grain boundaries in NiAl and apparently strengthens the boundary regions preventing grain boundary sliding, NiAl (Zr) does not suffer from this problem. It has been found that even extremely small Zr addition (approximately 0·1 at%) significantly improves the fatigue life on a stress range basis; approaching that of superalloys (figure 7).

4. Effects of microstructure on low cycle fatigue behaviour of the γ-based titanium aluminides

The interest in using light γ-TiAl alloys in aircraft jet engines and land-based gas turbines has been increasing steadily. Many successful engine tests of parts utilizing the unique mechanical properties of γ-TiAl, the low density leading to high specific stiffness, excellent creep and oxidation resistance, strength retention at elevated temperatures and burn resistance, have been completed. The possibility of production of the components through casting and forging processes has been studied. The major problems associated with the use of γ-TiAl in gas turbines are the once regarding close control of composition, control and reproduction of microstructure at the processing stage. It has been found that the microstructure exerts large influence on LCF resistance of Ti–48Al–2W–0·5Si alloy (Recina *et al* 1998). A duplex (equal amounts of γ and α phases) fine-grained structure has superior LCF resistance with smaller

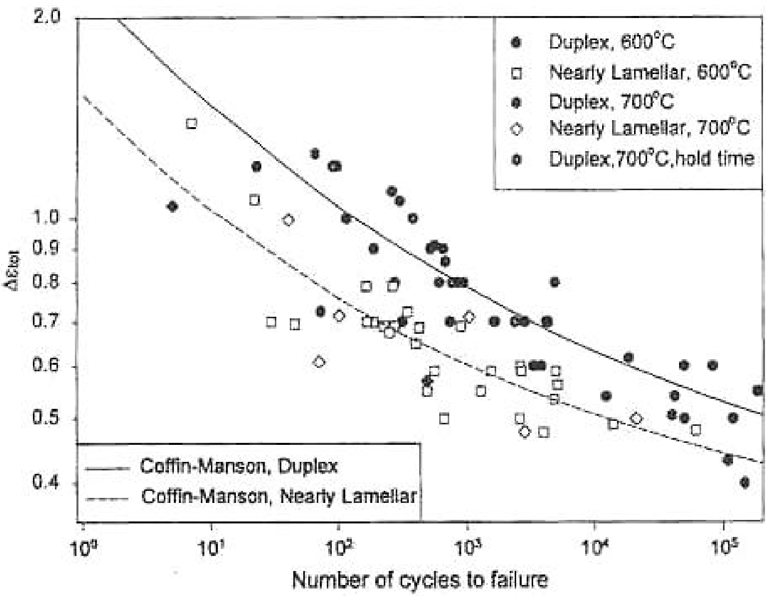

Figure 8. Strain-life plots at various temperatures with 300s hold time in tension for γ-TiAl. Closed symbols represent materials with duplex structure; Open symbols represent the nearly lamellar structure (Recina *et al* 1998).

scatter in life in comparison with a coarse grained lamellar structure consisting of alternate laths of γ and α_2 phases (figure 8). The α_2 phase occurs as a result of low aluminum content in the alloy. The increase in temperature or the imposition of hold time seems to have lesser influence than the microstructure. The variation in lifetime is found to have correlation with the amount of inelastic strain developed during the fatigue cycle. The duplex material exhibits longer life due to larger isotropic hardening and smaller Bauschinger effect, which lead to smaller inelastic strains and damage in each cycle. Investment cast γ-TiAl shows similar fatigue resistance as IN 738LC superalloy at elevated temperatures.

5. Effects of temperature and environment on LCF behaviour of near γ-TiAl

Near γ-TiAl (Ti–47Al–2Nb–2Mn (at %) with 0·8% vol. TiB_2) has been developed to replace heavier alloys in the hottest section of compressor in jet engines (Christ *et al* 2001). The effects of temperature and environment on LCF behaviour were evaluated in the duplex state of microstructure comprising equiaxed γ grains and γ/α_2 lamellar grains. In the isothermal tests performed between 773 (500°C) and 1023 (750°C), fatigue behaviour is found to be strongly affected by temperature as a consequence of change in the cyclic stress response at $\sim$ 923 K (600°C). Below this transition temperature, the alloy cyclically hardens while the saturation stress response is quickly established at higher temperatures. The cyclic life is strongly affected by the environment (figure 9). In vacuum, the number of cycles to failure rapidly decreas with increasing temperature. The alloy exhibits prolonged cyclic life in air at higher temperatures. Environmental effects are more pronounced at temperatures below the BDTT (Maier *et al* 2002), resulting in the unusual inverse effect of temperature on fatigue life for tests conducted in air environment (figure 10). Such anomalous behaviour

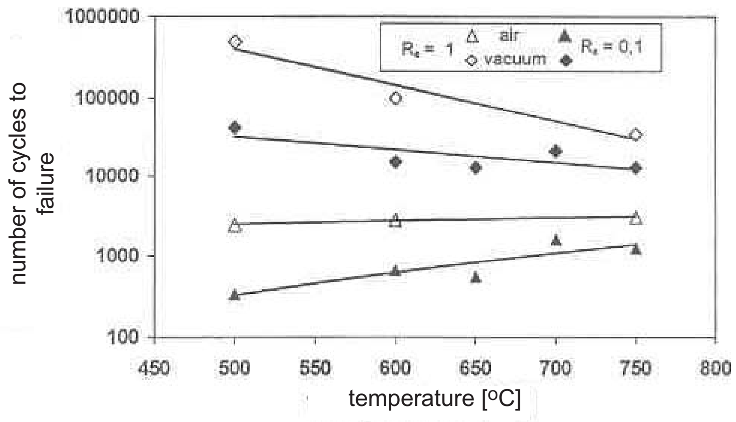

Figure 9. Influence of temperature on cyclic life of γ-TiAl for fatigue tests performed at a strain range of 0·56% with $R_\varepsilon = -1$ and 0·1 in air and vacuum.

has also been reported in fatigue crack growth tests on pure TiAl and a TiNb particulate-reinforced alloy, where properties are reported to be worse at 873 K than at 1073 K. Hydrogen embrittlement contributes to damage evolution in titanium alloys at intermediate temperatures ($\sim$ 573–773K) due to the interaction of water vapour with material freshly exposed at the crack tip during cyclic plastic deformation (Gregory 1994). It is argued that this is also a viable mechanism for titanium aluminides (Morbu *et al* 1997), and below BDTT the corrosive effects of water vapor appears to be more prominent than above the BDTT. This does explain the unusual inverse effect of temperature on fatigue life seen in tests conducted in air.

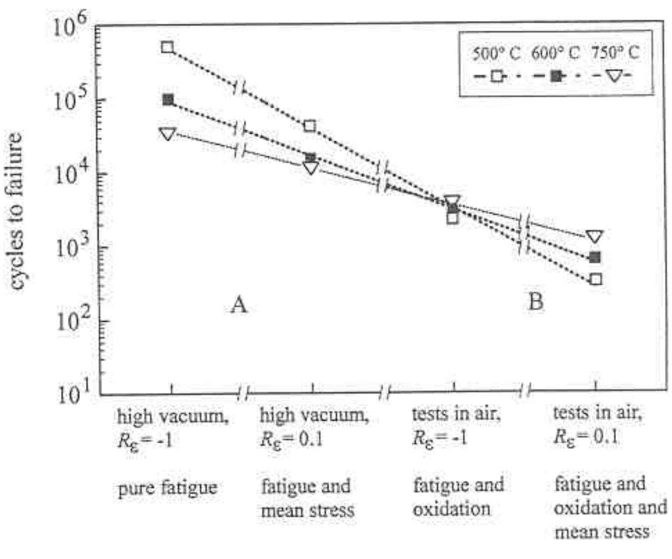

Figure 10. Influence of test conditions on LCF life of γ-TiAl in tests conducted at a total strain range of 0·56% and $\dot{\varepsilon} = 3 \times 10^{-3}\text{s}^{-1}$. The figure shows the schematic representation of various damage mechanisms contributing to deterioration in life at various temperatures (Maier *et al* 2002).

6. High temperature fatigue behaviour of γ-TiAl sheet material

Though, it a being guess that the first commercial application of γ-TiAl may arise in the form of fabricated sheet components, the fatigue properties of sheet material have been scarcely examined. Very recently, studies on γ-TiAl sheet material have been conducted (Schallow & Christ 2002) in the framework of a European Union project DOLSIG that aims at the development and manufacture of an exhaust cone. The sheet material studied has the designation gamma-MET and was produced via an ingot route. Batches of 1·5, 1·0 and 0·7 mm thickness showing a near-gamma microstructure in the primary annealed condition was investigated. The main emphasis in these investigations was placed on a comparison of LCF test results on a γ-TiAl bulk material with those obtained on sheet material, both tested over the temperature range, 773 to 1023 K. Since the bulk material data was obtained at R-values of 0 and -1 to account for the effects of mean stress, it is necessary to design and optimize the anti-buckling device to enable the testing of the sheet material under compressive stresses. The important findings are summarized below. The sheet material shows higher cyclic strength compared to bulk material tested under identical conditions; the increased strength has mainly attributed to a smaller grain size and differences in microstructure. The cyclic stress response of the sheet material is characterized by stable stress response from the beginning till the onset of failure, figure 11. This behaviour is independent of temperature and strain amplitude. At $R = 0{\cdot}1$, the sheet material exhibits pronounced cyclic hardening below BDTT which decreases with increasing temperature. In contrast, the cyclic stress response of the bulk material is characterized by cyclic hardening irrespective of the R-value. Both the types show a cyclic relaxation behaviour leading to mean stress reduction when tested at $R = 0{\cdot}1$, (figure 12). In case of the sheet material, the relaxation behaviour is more pronounced and strongly temperature-dependent. Sheet material with imposed mean stress experiences a reduction in strength throughout cyclic life. Both the materials show increase in life at higher temperatures. The effect of R-ratio on fatigue life of sheet material is shown in figure 13. The R-ratio of 0·1 points out the existence of tensile mean stress in the cycle. At 773 K (500°C), cyclic life at $R + 0{\cdot}1$ is reduced to less than half of the value

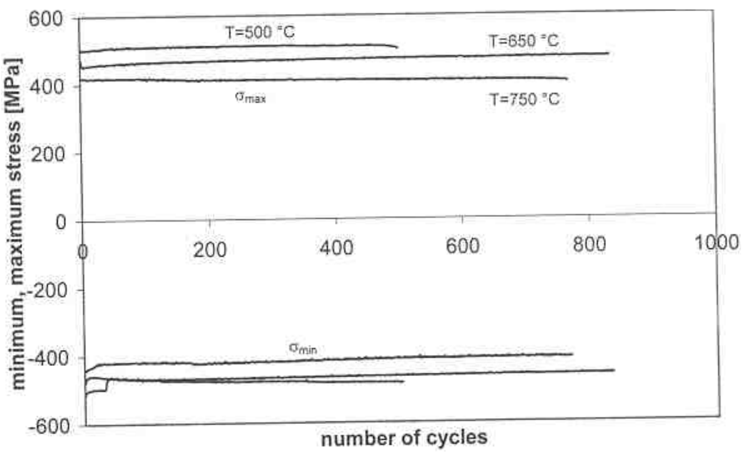

Figure 11. Cyclic stress response curves of γ-TiAl sheet material at a strain range of 0·70%. $R = -1$ (Schallow & Christ 2002).

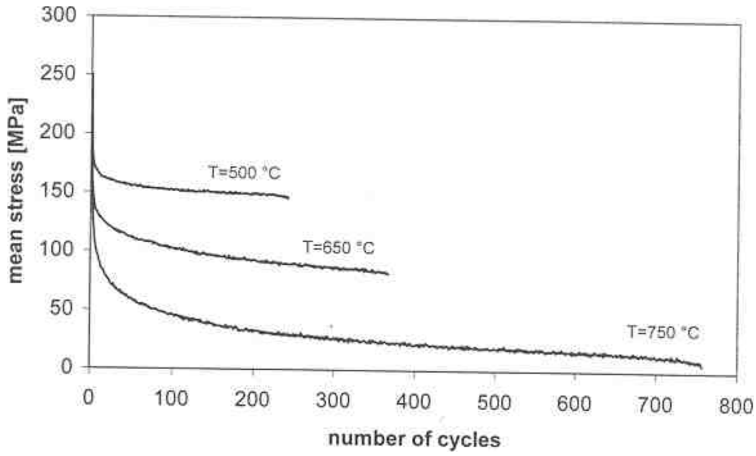

Figure 12. The variation of mean stress during cyclic loading of γ-TiAl at a strain range of 0·70% and $R = 0{\cdot}1$ in the temperature range 773–1023 K (500–750°C) for sheet material (Schallow & Christ 2002).

under symmetrical conditions ($R = -1$). However, the detrimental effect of imposed mean stress decreases with increasing temperature as can be seen from the converging course of the curves.

7. Conclusions

Very extensive research programmes have been pursued worldwide in order to develop intermetallic alloys for potential structural applications in gas-turbine engines. Excellent progress has been achieved in improving resistance to creep, fatigue and oxidation, and in processing and design methodology. However, ductility and fracture toughness at room and intermediate

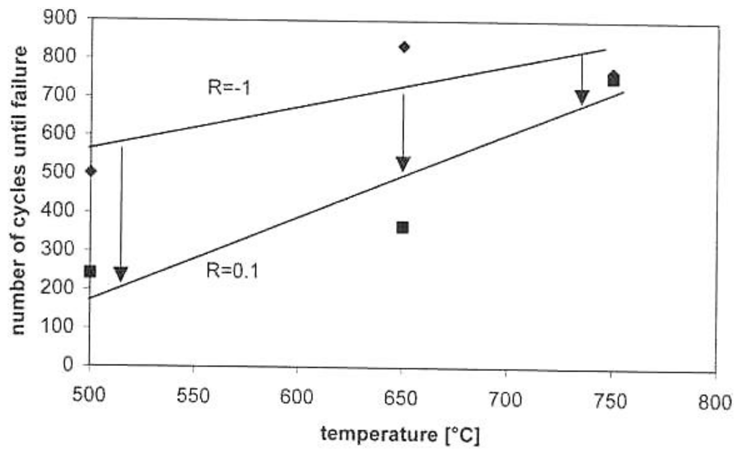

Figure 13. Effect of strain ratio R (mean stress) on cyclic life of γ-TiAl of sheet material (Schallow & Christ 2002).

temperatures continue to be lower than the desired values. The desired balance between high temperature creep, fatigue and creep-fatigue interaction resistance and fracture toughness has not yet been achieved in intermetallics. A low defect tolerance exists in intermetallic alloy components due to low fracture toughness. Darolia (2000) made an excellent review of ductility and fracture toughness issues related to implementation of NiAl in gas-turbine engines. Some γ-TiAl based intermetallic alloys are currently under further development to overcome the aforementioned problems and bring these candidate materials closer to applications in advanced aero-engines, automotive parts and land-based gas turbines. Nickel based superalloys, which intermetallics are supposed to replace, possess an excellent combination of creep strength, fatigue strength, oxidation resistance and fracture toughness that are unlikely to be surpassed. Consequently, use of new intermetallic-based alloy components will be very selective and gradual, and there may not be a rapid and widespread displacement of nickel-based superalloys in the near future.

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