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Microstructures and Mechanical Properties of NiAl-(Cr) and TiAl-(Cr) Intermetallic Alloys

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PACS.81.40.-z – Treatment of Materials and its Effects on Microstructures and Properties

Abstract. — The oxidation resistant light weight aluminides: NiAl and TiAl with ordered B2 and L1₀ super lattices show promising mechanical properties for high temperature applications. The ductility and strength properties at room and higher temperatures are strongly dependent upon the degree of ordering of the intermetallic compounds and of the microstructural parameters of the multiphase alloys, such as NiAl-(Cr) and TiAl-(Cr,Si) The presented paper describes and discusses the influence of second phases on the mechanical properties of these intermetallics with special emphasis of structural defects — antistructure atoms — and the dislocation mobility.

1. Introduction

The high melting point aluminides TiAl and NiAl have been studied extensively because of their great potential as structural materials in the aerospace industry and for engine parts. The attractive properties of these intermetallics are the low density, good creep strength and high temperature oxidation resistance. Among several other intermetallics TiAl and NiAl base alloys with additions of transition metals, such as chromium, molybdenum and vanadium exhibit considerable ductility at room or medium temperature [1-4]. Their reduced density may reproduce thirty percent reduction in weight of engine components (turbine blades, compressor discs, valves, etc.) and the relatively high thermal conductivity provides improved cooling efficiency. Numerous investigations on mechanical and physical properties and microstructural characterizations including high resolution electron microscopy (HREM) and atom probe field ion microscopy (APFIM) have been performed in the last decades [5-13]. The present paper describes and discusses the microstructural related properties of newly developed advanced NiAl-(Cr) and TiAl-(Cr) alloys for structural applications at high temperatures.

2. Physical and Mechanical Properties of the Monolithic Aluminides

Structural applications of titanium and nickel aluminides in jet propulsion and combustion engines require an optimum balance of their physical and mechanical properties including hot gas corrosion resistance. Another important criterion is the ductility or fracture toughness as a measure of the damage tolerance [3]. One of the objective of material scientists is to

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improve the properties by macro or micro alloying and by proper materials processing in order to achieve high temperature strength and excellent creep resistance with sufficient ductility. As introduction to the specific properties of selected NiAl and TiAl base alloys, the main physical and mechanical properties of the pure stoichiometric phases Ti₃Al, TiAl and NiAl are presented in Table I. The maximum service temperature of TiAl base alloys is about 800 °C and for special uses up to 1000 °C. The latter requires oxidation resistant coatings and improved creep behaviour by strengthening the material with fibres or particles. For certain applications small amounts of boron and silicon as micro alloying additions have been used [14,15].

Two phase TiAl/Ti₃Al alloys of the basic composition Ti₅₂Al₄₈ with lamellar or duplex microstructure behave quite ductile at room temperature (2% elongation to failure and \( K_{IC} \) 20 MPa m\(^{1/2} \)). The DBTT of NiAl is between 400 and 600 °C depending upon the stoichiometry, the amount of impurities and microstructural parameters (grain size, second phase etc...). Another important criterion for structural application is the elastic stiffness as a function of the temperature. NiAl
exhibits higher elastic moduli of about 180 ± 5 GPa at room temperature. For polycrystalline TiAl base alloys the elastic moduli was determined to be 175 ± 3 GPa [16–19].

The thermal conductivity is also an important property which influences the design of engine parts operating at elevated temperature. A high thermal conductivity and low thermal expansion coefficients cause low thermal gradients and reduce the amount of thermally induced stresses. The thermal conductivity of NiAl is of the order of 70–80 W m⁻¹ K⁻¹ over a large temperature range of 20–1100 °C and is quite high compared to that of Ni base super alloys of about 8 W m⁻¹ K⁻¹ at room temperature.

The thermal conductivity of γ-TiAl increases with increasing temperature from 19 ± 2 W m⁻¹ K⁻¹ at room temperature to 35 ± 2 W m⁻¹ K⁻¹ at 600 °C, respectively. A high thermal conductivity and lower thermal capacity lead to a short heating up period in order to achieve proper operation temperatures of the engine parts. The considered aluminides possess the ordered b.c.c. lattice structure B2 (NiAl) and the tetragonally distorted face centred cubic structure L1₀ (TiAl). The modified two phase NiAl base alloys contain large amount of chromium (max. 28 at%). These hypo-eutectic alloys are falling on the quasi binary section of the ternary Ni-Al-Cr system, shown in Figure 1a. The basic composition of the TiAl alloy is about Ti₅₂Al₁₆±₁Cr₁₃±₅Si₀₂. See Figure 1b. Both aluminides NiAl and TiAl show dislocation glide on ordinary slip systems which are restricted in their operational meaning fold. However, superdislocations will be activated with much larger shear stresses, but in general less than 5 slip systems are active and therefore the von Mises criterion is not fulfilled. The predominant slip system in NiAl at room temperature is (100){011} which provides only three independent slip systems. This explains the lack of ductility. At higher temperature above the DBTT, the (100){011} slip system is shifted to (111){011} [20,21]. The favourable slip system in the f.c.c. lattice of TiAl is α/2[110] (111). Although super dislocations of the type α/2[101], α/2[011], and α/2[112] on the (111) plane are mobile. In addition, deformation twinning occurs on {111} planes in (112) directions in γ-TiAl base alloys with lower stacking fault energy. Chromium and manganese are known to reduce the complex stacking fault energy [22–24].

3. Materials Preparation and Microstructural Features

The materials were prepared by vacuum induction melting (NiAl-Cr) and arc scull melting (TiAl-Cr). The ingots were deformed at high temperatures (1150 to 1250 °C) by extrusion to rods with a reduction in cross section of about 10:1. The specimens for microstructural studies in the SEM, TEM and APFIM and for mechanical tests (elastic modulus measurements, tensile or compression tests and KIC measurements) were cut from the extruded bars. Figures 2a, b show optical micrographs from the deformed Ti₅₂Al₄₀Cr₁₃Si₀₂ (a) and (NiAl)₇₃Cr₂₇ (b) samples. Large amount of deformation leads to a typical breakup of the two phase lamellar microstructure in the as cast state. A fine-grained duplex type of microstructure have been developed by dynamic recrystallisation during hot extrusion. TEM investigations reveal more details of the lath type of microstructure of the Ti₅₂Al₄₀Cr₁₃Si₀₂ alloy. The thinner Ti₃Al lamellae are oriented to the thicker TiAl matrix lamellae.

The relation mutualship is as follow: (111)γ // (0001)α₂ and [110]γ // [1120]α₂ [12, 25]. This is also confirmed by high resolution field ion microscopy (FIM), shown in Figure 3a. The image illustrates the interphase boundaries of γ and α₂ on an atomic scale. The TEM image in Figure 3b shows that in the deformed state the constituents exhibit deformation twins primarily inside the TiAl lamellae. The glide is transfered to the Ti₃Al lamellae by pure dislocation slip at higher deformation temperatures of 650 °C. This refers to the DBTT of the monolithic α₂ phase. The microstructural parameters were determined by SEM and TEM and are displayed in Table II.
The formation of deformation twins in the \( \gamma \) lamellae is due to the dissociation of the \( a/2\{112\} \) superdislocation on the \( \{111\} \) slip planes, which is identical with the twin plane [25]. Some amounts of chromium dissolved in \( \gamma \)-TiAl lead to a decrease of the complex stacking fault energy ECSF from 70 mJ/m\(^2\) to < 14 mJ/m\(^2\). Consequently the antiphase boundary and the stacking fault energies are reduced and therefore twinning and the formation of APB will more easily occur. The presence of a higher APB density in chromium alloyed TiAl have been clearly shown by [26]. Similar results on the improvement of the ductility in \( \gamma \)-TiAl base alloys were obtained for \( Ti_{50}Al_{48}Mn_{2} \) investigated by Huang and Hall [27]. Misfit dislocations were also observed at the interphases in the NiAl-Cr alloy. Emission of mobile \( a\{100\}\{001\} \) dislocations from the interphases either in the chromium rich phase or in the NiAl matrix phase improve the ductility of this material. The TEM image in Figure 4 shows clearly the presence of \( a\{100\} \) dislocations at the interphases of the chromium rich solid solution and the NiAl matrix after 2% plastic deformation in compression at room temperature [28].
Fig. 2. — Optical micrographs showing the microstructures of the extruded TiAl-Cr (a) and NiAl-Cr (b) samples (longitudinal section). The TiAl-Cr material possesses dynamically recrystallized γ-grains and heavily deformed α2 and γ-lamellae. The asymmetrically deformed NiAl-Cr rod exhibit a dynamically recrystallized NiAl matrix with aligned α-Cr(NiAl) crystals.

Fig. 3 — FIM image (a) illustrates γ - α2 - γ-interphases on the atomic scale; the TEM bright field image (b) showing shear processes of γ-TiAl matrix by [112](111) twinning and slip transfer to the Ti₅Al lamellae.

Fig. 4. — TEM bright field image reveals α(100) misfit dislocations at interphases of Cr rich particles in the NiAl matrix.
Table II — Microstructural parameters of two phase TiAl/Ti₃Al and NiAl–Cr.

<table>
<thead>
<tr>
<th>Alloy composition</th>
<th>Ti₅₂Al₄₈</th>
<th>Ti₅₂Al₄₆Cr₁₅Si₀₂</th>
<th>(NiAl)₇₃Cr₂₇</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean lamellae thickness (Ti₃Al) and mean particle diameter (Cr) in the as cast state [nm]</td>
<td>500-2000</td>
<td>200</td>
<td>1200 ± 300</td>
</tr>
<tr>
<td>Mean lamellae thickness (in the as cast state [nm])</td>
<td>2000</td>
<td>500</td>
<td>–</td>
</tr>
<tr>
<td>Interlamellar/interparticle separation distance [nm] (as cast)</td>
<td>2000</td>
<td>500</td>
<td>100</td>
</tr>
<tr>
<td>Thickness of TiAl twins [nm] (deformed)</td>
<td>100</td>
<td>100</td>
<td>–</td>
</tr>
<tr>
<td>Average grain size [μm] (in the extruded state)</td>
<td>–</td>
<td>0.5</td>
<td>5/0.5</td>
</tr>
<tr>
<td>Chromium content in the second phases (Ti₃Al) and Cr(Ni,Al) [at%]</td>
<td>–</td>
<td>12</td>
<td>99.5</td>
</tr>
<tr>
<td>Chromium content in the matrix phases (Ti₃Al, NiAl) [at%]</td>
<td>–</td>
<td>2.5</td>
<td>~1</td>
</tr>
<tr>
<td>Volume fraction of the second phases Ti₃Al or Cr(Ni,Al)</td>
<td>10-15</td>
<td>15-20</td>
<td>~30</td>
</tr>
</tbody>
</table>

Table III. — $K_{IC}$ values (MPa m$^{1/2}$) of NiAl–Cr and TiAl(Ti₃Al) alloys.

<table>
<thead>
<tr>
<th></th>
<th>25 °C</th>
<th>500 °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>NiAl</td>
<td>5-6</td>
<td>~20*</td>
</tr>
<tr>
<td>(NiAl)₇₃Cr₂₇</td>
<td>11-12</td>
<td>33-35</td>
</tr>
<tr>
<td>Ti₄₅Al₅₅</td>
<td>5-8</td>
<td>16-18</td>
</tr>
<tr>
<td>Ti₅₂Al₄₆Cr₁₅Si₀₂</td>
<td>32-34</td>
<td>40-42</td>
</tr>
</tbody>
</table>

*BDTT $\approx$ 683 K.

4. Mechanical Properties

4.1. Compression and Tensile Test. — The mechanical properties of the TiAl and NiAl base alloys with different Cr contents were investigated in greater detail. The flow stress and ductility depend strongly upon the stoichiometric composition of the matrix phases. This effect was investigated thoroughly on the NiAl-Cr material by Schäfer et al. [29]. The results are shown in Figure 5 where the flow stress and ductility in compression is plotted versus the
Al/Ni ratio. It is clearly seen that in hypo- and hyperstoichiometric NiAl-Cr compositions the flow stress increases strongly, whereas the ductility curve displays the opposite tendency. The stoichiometric composition shows a minimum flow stress of about 260 MPa and maximum in ductility of about 2.5% plastic strain. The deviation from the stoichiometric composition influences the properties strongly due to structural defects, such as Ni antistructure atoms in Ni rich solid solutions and vacancies in the Ni sublattice in Al rich compounds. Cr additions increases the plastic strain and also the achieved maximum ductility. The effect of Cr on the flow stress and plastic strain in compression is displayed in Figures 7a, b. It is clearly seen that the flow stress (a) increases from 260 MPa to about 650 MPa, respectively. The plastic strain (b) in compression shows a maximum value of $\varepsilon_{pl} \approx 30\%$ at the 27 at% chromium. This correlates well with the interparticle separation between the chromium particles of 0.1 $\mu$m in the NiAl matrix. Another important factor is the mobility of interphase dislocations which contribute to the enhanced plasticity of this material. The modified Ti$_{52}$Al$_{46}$Cr$_1$Si$_{0.2}$ alloy shows relatively high ductility at room temperature in tension tests of about 4% plastic strain in the as cast state with lamellar microstructure and 2% plastic strain in the extruded state with fine grained duplex microstructure. The flow stress of the as cast material ranges from 400 to 450 MPa at room temperature up to 800 °C.

The extruded samples exceed flow stresses of 550 to 600 MPa in the temperature range from 20 up to 500 °C. At 1000 °C the flow stresses of the more temperature resistant lamellar microstructure is about 150 MPa whereas the fine grained duplex microstructure holds the flow stress of 300 MPa at 800 °C. Beyond 800 °C a steep decrease of the flow stress occurs, due to grain boundary sliding mechanisms, (Fig. 6). This leads to superplastic properties in the high temperature regime of about 950 to 1100 °C [30, 31]. The plastic strain in tension of the as cast or extruded TiAl base alloy is about 15% at the operation temperature of 650 °C. This is sufficient enough for achieving a high damage tolerance. This value is lower than the flow stresses of improved TiAl base alloys at similar temperatures. With increasing temperature the flow stress is slightly decreasing and shows at 1100 °C still 150 MPa. From the flow stress versus temperature curves of the aluminides it is clearly seen that NiAl-Cr alloys exhibit better strength properties and a higher temperature oxidation resistance than TiAl base alloys do. The ductility of (NiAl)$_{73}$Cr$_{27}$ increases sharply above the DBTT limit of 450 °C from 2% plastic strain at 600 °C to 50% plastic strain at 800 °C, respectively [32]. In contrast to the investigated TiAl base alloy the extruded (NiAl)$_{73}$Cr$_{27}$ material with fine grained microstructure exhibits a flow stress of 350 MPa at 600 °C in tension tests, see Figure 7.
Fig. 6. — Yield stress (a) and compression strain (b) in dependence on Cr concentration of NiAl-Cr alloys.

Fig. 7 — Comparison of flow stresses of as cast and extruded TiAl base alloys in dependence on the test temperature.

4.2. Fracture Toughness. — The investigated two phase aluminides exhibit a higher fracture toughness than the monolithic intermetallic compounds. The critical stress intensity factors determined in 4-point bending tests at different test temperatures (RT and 500 °C) are a measure for the fracture toughness and are displayed in Table III. In comparison the $K_{IC}$ values of the monolithic NiAl (stoichiometric composition) and of Ti$_{45}$Al$_{55}$ (monophase γ) are also shown. The relatively high $K_{IC}$ values of the two phase intermetallic alloys show clearly the influence of the second phases which improve the fracture toughness [32]. The transition from brittle to ductile behaviour is caused by the activation of dislocation emission and enhanced dislocation mobility at higher temperature including the activation of secondary slip systems which have been discussed in Section 2. From Table I it can be clearly seen that the monolithic intermetallic compounds do not meet the criterion of $K_{IC} > 20$ MPa m$^{1/2}$ for materials which will be used as rotating or oscillating engine parts. However, the modified intermetallic alloys exceed this critical value at elevated operation temperature and are close to the lower boundary condition of the fracture toughness.
5. Conclusions

Newly developed two phase intermetallic NiAl-Cr and TiAl-(Cr) alloys exhibit superior mechanical properties and excellent oxidation resistance at medium and higher temperatures. From the results of the present investigations the following conclusions are drawn:

- The modified Ti_{52}Al_{46}Cr_{12}Si_{0.2} alloy consists of t.f.c \gamma and complex hexagonal \alpha_2 phases in optimum volume portions.

- At the present stage of the development this light weight material exhibits sufficient strength and ductility at room temperature up to 800 °C and shows potential applications as motor components, such as outlet valves, connecting rods, spring rings, and piston bolts.

- Gamma titanium aluminides can be strengthened by a fine dispersion of hard particles, such as Ti_{5}Si_{3} and Ti_{2}B, or by discontinuous SiC-fibres. The reinforced metal matrix composites based on Ti_{52}Al_{46}Cr_{12}Si_{0.2} will achieve higher creep strength and are suitable for high temperature applications in jet engines (compressor, disks and blades, and low pressure turbine blades).

- The ordered b.c.c. NiAl base alloy with 27 at% Cr exhibits large volume fractions of chromium particles finely dispersed throughout the NiAl matrix phase. This material shows high ductility in tension above 600 °C, good strength and creep properties up to 1100 °C. The decrease in the brittle-ductile transition (DBT) temperature from about 680 K to somewhat lower temperatures will be achieved by grain refinement and by the use of nearly interstitial free chromium as alloying element.

- NiAl-Cr alloys are considered to be used as oxidation resistant inlays in combustion chambers of stationary gas turbines and as nozzle segments in jet engines.

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