

Physical metallurgy of nickel aluminides

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Abstract. A description of the important physical metallurgy aspects of Ni_3Al and NiAl encompassing structure, crystallographic defects, slip systems and phase stability has been presented in this article. The microstructures generated in the two alloys by conventional as well as novel processing techniques have been discussed. The effect of alloying additions on the microstructure has been enumerated. Besides description of the aforementioned physical metallurgy aspects, an important purpose of this review is to focus on the reasons of brittleness in the two alloys and means of alleviating this problem primarily by alloying. The effect of alloying on the slip behaviour has also been described.

Keywords. Intermetallic compound; microstructure; Ni_3Al ; NiAl .

1. Introduction

Intermetallic compounds have emerged as materials with vast potential for application in a wide range of technologically important areas (Darolia 1991). The enormous potential of intermetallics especially aluminides stems from their many attractive properties, such as high oxidation and corrosion resistance and relatively low densities, combined with their ability to retain strength and stiffness at elevated temperatures (Busso & McClintock 1994; Jayram & Miller 1994). A bulk of the investigations in intermetallics has been done on the aluminides in the Ti-Al and the Ni-Al systems (Field *et al* 1991a). The two aluminides of significance in the Ni-Al system (figure 1) are Ni_3Al and NiAl . Ni_3Al has received considerable attention as a potential structural alloy. Ni_3Al acts as a strengthening phase in most superalloys (Dey *et al* 1993). Because of its low density, high melting temperature, good thermal conductivity and excellent oxidation resistance NiAl has emerged as a possible high temperature structural material. It has several advantages over superalloys (Dey & Sekhar 1997). As is the case with other aluminides, NiAl and Ni_3Al have certain drawbacks, the most important being the inadequate ductility at ambient temperature and lack of good high temperature creep resistance. Several attempts have been made to alleviate the problem of room temperature brittleness in Ni_3Al and NiAl through modification of slip systems, grain refinement, use of single crystals and microalloying with boron (Lahrman *et al* 1991; Field *et al* 1991b). In the case of Ni_3Al , single crystals have been found to be ductile (Pope & Ezz 1984). The level of research effort in the case of Ni_3Al has been substantially more than in case of other aluminides and commercialization of Ni_3Al alloys for selected applications should occur very

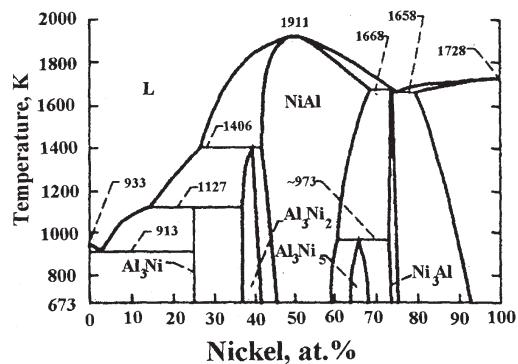


Figure 1. Ni-Al phase diagram (Singleton *et al* 1986).

soon. The problem of lack of ductility in NiAl has been addressed (Noebe *et al* 1993). In a recent study it has been demonstrated that this intermetallic compound shows large ductility under certain conditions of impurity and imperfection content, heat treatment and surface perfection (Levit *et al* 1996). In the past decade several alloying additions to NiAl have been attempted to improve its ductility (Darolie *et al* 1992).

Intermetallics can be fabricated both by conventional melting and casting route as well as the powder metallurgy route. Studies have shown that in addition to conventional casting and powder metallurgy fabrication techniques, micropyretic synthesis also known as combustion synthesis or self propagating high temperature synthesis (SHS) can also be used successfully to obtain near net shapes in these alloys (Moore & Feng 1995; Dey & Sekhar 1998). Micropyretic synthesis offers several advantages over other techniques of synthesis, the most important ones being the possibility of direct net shape fabrication and enormous saving in energy spent on fabrication and processing (Dey & Sekhar 1998).

In this article, the physical metallurgy of Ni_3Al and NiAl has been described. Besides information on crystal structure and ordering behaviour, considerable importance has been given to the microstructures developing in these two alloys under different processing conditions and after different alloying additions. Since lack of ductility has been a major problem in these intermetallic compounds, ample emphasis has been given on the examination of slip systems operating in these two intermetallic compounds and reasons for brittleness has been ascertained. Means of ductilizing these two intermetallic compounds have been described. In every section of this paper, the pertinent aspects of Ni_3Al has been discussed first. This is followed by discussions on NiAl.

2. Crystal structure

2.1 Structure

The crystal structure of Ni_3Al is ordered cubic and its space group is $Pm\bar{3}m$. Strukturbericht designation for this structure is $L1_2$ (Pearsons 1992). Figure 2 shows the unit cell of Ni_3Al which can be visualized as an ordered cubic structure with the Al atoms occupying corners of the cube and the Ni atoms located at the cube faces in the lattice.

The crystal structure of NiAl is primitive cubic CsCl structure. Its space group is $Pm\bar{3}m$. The Strukturbericht designation for this structure is $B2$ (Pearson 1992). It can be described in terms of two inter penetrating cubic cells where Al atoms occupy one sublattice and the Ni atoms occupy the second sublattice (Barrett & Massalski 1980). This unit cell is shown

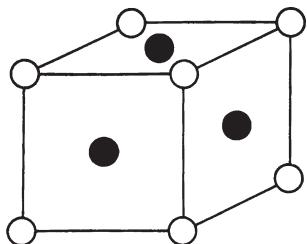


Figure 2. Unit cell of Ni_3Al ; Ni atoms (filled circles) are on face centres and Al atoms (open circles) at corners.

in figure 3. Due to the inter penetration of the two unit cells the symmetry is lowered from a body centred cubic (A2) to simple cubic (B2). Further ordering in this lattice is possible on addition of alloying elements like titanium. Such additions transform the structure of NiAl from B2 to DO_3 (Darolia 1991).

2.2 Lattice parameter

The lattice parameter of Ni_3Al is 0.357 nm (Stoloff & Liu 1997). It has been observed that in comparison to the addition of substitutional solute elements like Si, Fe, Cr, Ti and V, addition of interstitial solutes like boron and carbon has a much stronger influence on the lattice parameter of this alloy and its strengthening (Stoloff & Liu 1997). The lattice parameter has been found to depend not only on the nature and the amount of solutes added but also on solidification and heat treatment conditions.

The lattice parameter of NiAl is 0.2887 nm (Hughes *et al* 1971; Taylor & Doyle 1972). Since this intermetallic compound can exist over a range of compositions (figure 1), the parameter of this intermetallic compound has been shown to be strongly dependent on the stoichiometry within this wide single-phase region (Taylor & Doyle 1972). A maximum in the lattice parameter occurs at the stoichiometric composition. Though some studies had shown a lattice parameter maximum at slightly Al-rich compositions, these observations were probably due to inadequate control over the chemistry of the alloy and inaccurate methods of determination of the composition (Fraser *et al* 1973).

In addition to the lattice parameter, the variation in density as a function of composition has been examined in the case of NiAl . It has been observed that the density of NiAl increases linearly with composition with increasing Ni content. A change in slope of this curve has been noticed at the stoichiometric composition which has a density of $\sim 5.90 \text{ g cm}^{-3}$ (Taylor & Doyle 1972). The composition dependence of lattice parameter as well as density has been used in the case of NiAl to understand the nature of lattice defects particularly point defects present in this intermetallic compound. On the Ni-rich side, an increase in density and decrease in lattice parameter with increasing Ni content has been observed since Ni atoms are smaller

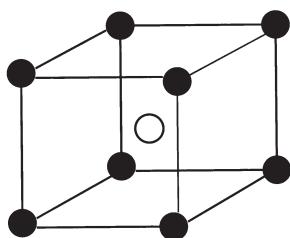


Figure 3. Unit cell of NiAl ; Al atoms (open circles) are on body centres and Ni atoms (filled circles) at corners.

than Al. In case of the Al-rich alloys the decrease in density and the lattice parameter with Al content is more rapid than otherwise expected by replacement of Ni atoms by Al indicating the presence of vacancies in Ni sites (Noebe *et al* 1993). Unlike in NiAl, no systematic study on the variation of density as a function of stoichiometry seems to have been carried out in case of Ni₃Al.

3. Phase stability

The intermetallic compounds examined in this study are ordered phases at ambient temperatures. It is important to ascertain the extent of ordering at higher temperatures because many physical and mechanical properties are governed by the extent of order in the alloy. It has been shown in many studies that Ni₃Al is ordered to very near its melting point (Stockinger & Neumann 1970). Cahn *et al* (1987) have shown that the ordering temperature of Ni₃Al is 1723 K. Antiphase domain boundaries have been very often seen in this alloy and are more or less uniform in size except for microstructures generated by rapid solidification (Stoloff & Liu 1997). Studies have shown that microsegregation occurs in rapidly solidified Ni₃Al and is responsible for the non-uniform domain size (Stoloff & Liu 1997). The aluminium rich regions in microstructures where segregation has occurred have been found to have a coarser domain size. In these regions the ordering temperature is above the solidus. In alloys that freeze from the disordered state, fine domains have been observed. As far as the value of the APB energy is concerned, it can be changed by addition of solutes. The value of the APB energy has been found to lie in the range of 85 – 140 mJ/m² as evaluated by transmission electron microscopy (TEM) examination (Veyssiére 1984).

NiAl has one of the largest negative heats of formation among the intermetallic compounds with B2 structure. Its heat of formation has been found to depend on composition, the largest value being that of the stoichiometric composition indicating excellent stability for this composition (Kubaschewski 1958). Consistent with the strong Ni–Al nearest neighbour bonds, it has been observed that significant order existed in NiAl upto the melting temperature and no irrefutable proof has been obtained so far to suggest that an order-disorder transformation occurs in this alloy (Noebe *et al* 1997). The most common parameter that has been used for establishing the degree of order in this intermetallic compound is the degree of intrinsic disorder α' . This is defined as the ratio of the number of disordered atoms of one kind to the total number of lattice sites in a stoichiometric crystal (Noebe *et al* 1993). The value of this parameter at around 1273 K, by using Wagner–Schoottky theory and assuming anti-site defects are present on both sides of the stoichiometric composition, has been found to be quite small and lies in the range of 4×10^{-4} to 9×10^{-5} (Noebe *et al* 1997). A very low value of this parameter is indicative of the fact that very few atoms are out of position at 1273 K. NiAl is very difficult to disorder because of the wide composition range over which the B2 structure is stable. It has not been possible to suppress ordering in NiAl under conditions, which generate cooling rates of the order of 10^8 K/sec (Sumiyama *et al* 1989). The other attractive aspect of NiAl is that it can not be disordered even by heavy ion bombardment at very low temperatures (15 K) (Mori *et al* 1984). This exceptional radiation stability makes this intermetallic compound a candidate for nuclear applications.

4. Elastic constants and other physical properties

The elastic constants have been determined for Ni₃Al (Pope & Ezz 1984). The shear anisotropy of Ni₃Al has been found to be very close to that of NiAl (Stoloff & Liu 1997). Young's

modulus (E) of this intermetallic compound at room temperature is very close to that of nickel. However, the rate of decrease of E with temperature is about half that of nickel.

In case of NiAl the elastic properties of single crystals have been found to be in good agreement with that of polycrystals (Wasilewski 1966). It has been observed in this intermetallic compound that the Young's modulus is very sensitive to heat treatment and other processing conditions but not to composition. The room temperature Young's modulus of this alloy has been found to be 193 GN m^{-2} (Rusovic & Warlimont 1979). This has been found to decrease almost linearly with temperature. In the case of very Ni-rich compositions, anomalous temperature dependence of the elastic properties for both single crystal and polycrystalline material has been observed. In Ni-rich compositions, the B2 structure transforms martensitically to an ordered tetragonal $L1_0$ phase in a reversible manner and this is responsible for the aforementioned anomalies (Rusovic & Warlimont 1977).

Some other properties like thermal expansion coefficient, electrical and thermal resistivity and Seebeck coefficient as a function of temperature in case of high purity Ni_3Al have been determined (Stoloff & Liu 1997). These properties have also been estimated in the case of NiAl (Noebe *et al* 1993). Thermal expansion behaviour of NiAl has been found to be strongly dependent on temperature (Wasilewski 1967). Low temperature resistivity of NiAl has been determined in the temperature range of 4.2 to 298 K as a function of stoichiometry. It has been observed in these studies that a sharp minimum occurs in the resistivity at the stoichiometric composition. The minimum has been explained by considering electron scattering from constitutional point defects (Jacobi *et al* 1970). Table 1 shows some of the physical properties of the two intermetallic compounds.

5. Crystal defects

5.1 Point defects

The nature of point defects present in Ni_3Al has been found to be different from that in NiAl. Ni_3Al does not have the stoichiometry constitutional vacancies of the type present in highly ordered intermetallics like NiAl having the AB stoichiometry (Stoloff & Liu 1997). This is indicative of the fact that hardening resulting out of addition of ternary alloying additions or by the change in Al content in Ni_3Al originates due to intrinsic factors such as size and modulus misfit (Stoloff & Liu 1997).

NiAl belongs to the category of intermetallics with a B2 structure where A atoms sit on β sites and vacancies on α sites (Neumann *et al* 1976). At the stoichiometric composition,

Table 1. Some physical properties of Ni_3Al and NiAl.

Properties	Ni_3Al	NiAl
Electrical resistivity ($10^{-8}\Omega \text{ m}$)	32.59	8 to 10
Thermal conductivity (W/m.K)	28.85	76
Thermal expansion coefficient ($10^{-6}/\text{K}^{-1}$)	12.5	13.2
Lattice parameter (nm)	0.357	0.2887
Young's modulus (GPa)	168	188
Specific heat (J/g.K)	0.54	0.64
Melting point (K)	1668	1955
Bonding	Covalent/metallic	Covalent/metallic

materials having this kind of defect structure have a triple defect structure because three defects must exist simultaneously, two vacancies on the α sites and one A atom on a β -site. The occupancy of Al sublattice is always full. Hence when there is excess Ni, it occupies the Al sublattice with no significant composition dependent vacancy concentration occurring in either sublattice. However, when Al is in excess, vacant sites or constitutional vacancies are formed in the Ni sublattice. It is not known whether Al can substitute Ni on the Ni sublattice in case of Al rich compositions. It has been shown in many studies that antistucture defects on the Ni sublattice are energetically unfavourable and are very unlikely. A high concentration of thermal vacancies can be quenched in NiAl in addition to the constitutional vacancies (Parthasarathi & Fraser 1984). The thermal vacancies form individually in disordered materials. However, for an ordered material like NiAl, either a vacancy must form on each sublattice in a balanced manner or two vacancies form on one sublattice with an associated antistucture defect on other sublattice. It has been speculated that where as the constitutional vacancies are accounted for by the latter mechanism the thermal vacancies arise by the former mechanism (Fan & Collins 1990). When samples are rapidly quenched from the melt or after further annealing of samples quenched rapidly from higher temperatures, the thermal vacancies tend to agglomerate and form voids (Locci *et al* 1989). The constitutional vacancies, however, become incorporated within the NiAl lattice in an ordered fashion.

5.2 Line defects

Deformation in Ni_3Al occurs by $\langle 110 \rangle \{111\}$ slip at low temperatures and by $\langle 110 \rangle \{100\}$ slip at high temperatures. The motion of $1/2\langle 110 \rangle \{111\}$ dislocations due to slip would create an antiphase domain boundary. The energy of this boundary will be high enough to make this slip mode improbable. The passage of a $\langle 110 \rangle \{111\}$ dislocation on the other hand does not leave any such fault behind and therefore, this type of dislocation is able to move in this material with ease.

The highly ordered nature of NiAl does not favour slip vectors, which result in destruction of order (Noebe *et al* 1993). The three translation vectors which do not destroy the order in the lattice are $a_o \langle 100 \rangle$, $a_o \langle 110 \rangle$ and $a_o \langle 111 \rangle$, where a_o is the lattice parameter of NiAl. The first slip vector is rather rarely observed in NiAl. This type of dislocation also has the lowest energy (Lloyd & Loretto 1970). It has been seen that the energy of the $a_o \langle 110 \rangle$ dislocations on $\{110\}$ planes is sufficiently low so that dissociation into two $a_o \langle 100 \rangle$ dislocations is not expected. The dislocations near the $a_o \langle 110 \rangle \{100\}$ edge orientation are, however, expected to dissociate into two $a_o \langle 100 \rangle \{100\}$ dislocations of appropriate line directions. Several types of dislocation loops, which are essentially edge in character and lie in $\{001\}$ planes with $a_o \langle 100 \rangle$ burgers vector are commonly observed in the substructure of NiAl. One such loop is a square loop with rounded corners with segments parallel to $a_o \langle 100 \rangle$. The other type of loop is also square in shape but has very sharp corners and has edges parallel to $a_o \langle 110 \rangle$ (Tisone *et al* 1968).

The dislocation mobility in this material can be ascertained by ascertaining the core structure of the various dislocations, which can be established only by theoretical calculations (Farkas *et al* 1991). The problem of potential slip systems in this material has been addressed by Farkas *et al* (1991) using embedded atom potentials. Their calculations indicated that $\langle 100 \rangle$, $\langle 110 \rangle$, and $\langle 111 \rangle$ dislocations all possess components of a non-planar core configuration and a large stress would be necessary to impart mobility to these dislocations.

5.3 Planar defects

The anti-phase boundaries in case of Ni_3Al are relatively high-energy faults and result from disturbances of unlike nearest neighbours in the slip plane (Stoloff & Liu 1997). The superlat-

tice intrinsic stacking faults (SISF) result due to the change in stacking sequence but without any nearest neighbour violations. These are low energy faults having energies in the range of $5 - 15 \text{ mJ/m}^2$ as compared to the value of 225 mJ/m^2 for the complex stacking fault (CSF). The CSF are produced by a shear of the type $1/6\langle\bar{1}\bar{1}2\rangle$. This fault vector in $L1_2$ structures is expected to have higher energies than the APB's and is identical to a stacking fault in the fcc structure. Ni_3Al also has superlattice extrinsic stacking fault (SESF) which is similar to the SISF except for the fact it does not result in first nearest neighbour violations. However, it changes the stacking sequence (Fu & Yoo 1991). It has been shown that SISF's do not contribute significantly to the plastic strain during deformation. These, however, may act as obstacles to superlattice dislocation motion.

Planar faults induced by deformation have not been encountered in NiAl. Theoretical calculations have shown that the most probable planar fault in this alloy is $1/2a_0\langle111\rangle$ APBs on $\{110\}$ and $\{112\}$ planes (Loretto & Wasilweski 1971). The energy associated with such faults has been found to be quite high. The TEM study of Veyssiére & Noebe (1992) has indicated that the separation between the partials should be of the order of 1.5 nm which corresponds to the APB energy of 500 to 750 mJ m^{-2} in the $\{110\}$ and $\{112\}$ planes respectively. These values are in good agreement with that determined by first principle total energy calculations of Fu & Yoo (1991) and LMTO calculations of Hong & Freeman (1991).

Though it has not been possible to see deformation induced faults in NiAl, in Al rich alloys planar faults have been observed which form parallel to the $\langle001\rangle$ planes. These faults occur by the nucleation of excess vacancies and Al atoms on the $\{100\}$ planes (Ball 1969). These could be related to the omega like defects seen in this material that are considered to act as sites of nucleation for these faults (Ball 1969).

6. Operative slip systems

Considerable amount of effort has been expended by numerous investigators to determine experimentally operative slip systems in near stoichiometric Ni_3Al single crystals and polycrystalline alloys and justify slip behaviour on a theoretical basis. Motion of superdislocations comprising two unit dislocations of type $1/2\langle110\rangle$ on $\{111\}$ at low temperatures causes slip in Ni_3Al . $\langle110\rangle\{100\}$ slip occurs at higher temperatures (Pope & Ezz 1984). Since the superpartials are further able to dissociate, the total energy of a super dislocation depends on the length of the total burgers vector as well as on the energies of any of the faults present (Takeuchi & Kuramoto 1973). This intermetallic compound shows an anomaly in its flow behaviour. Its critical resolved shear stress for $\langle111\rangle\langle101\rangle$ slip has been found to increase with increasing temperature (Pope & Ezz 1984). Several models have been proposed to explain this unusual behaviour (Pope & Ezz 1984). Out of these models, the cross slip model has been found to be most satisfactory. This model proposes that with increasing temperature the $1/2\langle101\rangle\langle111\rangle$ screw dislocations cross-slip from $\{111\}$ planes where they are mobile to $\{010\}$ planes where they are immobile. This leads to an increase in the flow stress with increasing temperature (Pope & Ezz 1984).

In case of single crystals of NiAl, it is generally believed that it exhibits two significantly different types of slip behaviour depending on whether the specimens are being tested in the soft directions or the hard direction. The soft orientations are non- $\langle001\rangle$ loading directions where the $b = \langle100\rangle$ slip predominates (Kim 1990). In case of single crystal specimen undergoing testing in the soft directions, the dominant slip vector is either $\langle001\rangle$ or a combination of $\langle111\rangle$, $\langle110\rangle$ and $\langle100\rangle$ (Loretto & Wasilweski 1971). The hard test orientations are those close to $\langle001\rangle$ as the $\langle001\rangle$ burgers vectors have a zero or near zero resolved shear stress. In

the case of hard oriented single crystals like the one with {001} orientation, the resolved shear stress for $\langle 100 \rangle$ slip approaches zero. Therefore the deformation of single crystals with this orientation requires considerably higher stress and the deformation process is quite different as compared to single crystals with soft orientation. The mechanism of deformation has been found to depend on the temperature of deformation. At temperatures as low as 77 K (liquid nitrogen temperature), the slip takes place on the {123}, {112} or the {110} plane with the slip direction being $\langle 111 \rangle$ (Pascoe & Newey 1968). In the temperature range of ambient to 600 K, uniform deformation by $\langle 111 \rangle$ slip on {112} and {110} planes and kinking has been seen. At temperatures higher than 600 K tensile ductility can be observed in single crystals with {001} orientation with deformation occurring by the movement of the {110} dislocations, {001} dislocations or a combination of glide and climb of both {001} and {011} type dislocations. In (001) single crystals of NiAl crept at elevated temperatures, both {001} and {011} type dislocations have been observed (Field *et al* 1991a).

In the case of polycrystalline NiAl, deformation has been found to mostly occur by the {001} slip. Though in some cases like in extruded specimen burgers vectors other than {001} has been encountered, these probably originated due to interaction between gliding $a_0\langle 001 \rangle$ dislocations (Zaluzec & Fraser 1974). Dislocations with {011} slip vectors have been observed in bicrystals of NiAl having a special orientation. The very limited observation of {011} slip appears to be an exception in NiAl, which occurs only under very specific conditions, and it can be concluded that deformation of polycrystalline NiAl occurs by the movement of the {001} dislocations (Noebe *et al* 1993).

6.1 Effect of alloying additions on the slip characteristics

Unlike NiAl, Ni_3Al exhibits $\{111\}\langle 110 \rangle$ slip and has sufficient number of slip systems for showing extensive plastic deformation. The single crystals of this intermetallic compound have been found to show extensive ductility (Stoloff & Liu 1997). However, ductility in polycrystals has been found to be rather low. The reason for poor ductility in polycrystals is grain boundary embrittlement (Stoloff & Liu 1997). The grain boundary embrittlement was thought to be caused by the segregation of impurities like sulphur, phosphorus and oxygen to the boundary. However, this behaviour was also seen even in ultra pure specimens where these impurities were not present (Liu *et al* 1985). This was indicative of the fact that the grain boundaries are inherently brittle in this material. The brittleness of grain boundaries seems to be due to the following important differences between the Ni and Al atoms, which constitute Ni_3Al : difference in ordering energy and electronegativity, valency difference and atomic size difference (Takasugi *et al* 1985). These factors reduce the grain boundary cohesive strength. The other factor, which makes the grain boundary week, is the presence of moisture in the environment. The hydrogen in the moisture gets liberated in atomic form when moisture reacts with Al in the alloy. This atomic hydrogen makes the grain boundaries brittle (Liu *et al* 1992). Boron was found to alleviate this problem substantially. Microalloying with 0.1 wt% B resulted in tensile ductility higher than 50% in air. It was observed that B segregates to the grain boundary (Aoki & Izumi 1979) and reduced the grain boundary embrittlement by two mechanisms. It increased the grain boundary cohesive strength and secondly it allowed slip transfer across the grain boundary. It is believed that strong segregation of B to the grain boundaries blocks the diffusion of hydrogen along the grain boundaries. More studies are required to better understand this phenomenon (Takasugi *et al* 1985; Liu 1992).

The flow behaviour of NiAl is effected by a variety of factors, important ones being temperature, stoichiometry, alloying additions and grain size (Bowman *et al* 1992). In case of NiAl it was initially thought that contamination of the grain boundaries by impurities causes

intergranular fracture in this material. It is now believed that grain boundary incompatibility due to a lack of sufficient number of independent slip systems is primarily responsible for intergranular failure in NiAl (Noebe *et al* 1993).

Besides $\langle 001 \rangle$ slip, which is the most frequently encountered slip in NiAl, $\langle 110 \rangle\{011\}$ dislocations have been frequently observed in extruded NiAl (Cotton *et al* 1991). However, it has been seen that these dislocations occur as a result of dissociation reaction and not as freshly nucleated glessile dislocations and hence their contribution to increasing ductility is insignificant (Cotton *et al* 1991). The $\langle 110 \rangle\{110\}$ slip systems are independent of the more commonly encountered $\langle 100 \rangle\{110\}$ slip and operation of the former alone supplies only two independent slip systems. The von Mises criterion for polycrystalline ductility (Dieter 1988) will be met by simultaneous operation of both families of slip systems (Noebe *et al* 1993). Alloying appears to be an attractive route for modifying the slip systems in the alloy and for imparting ductility to the material in due process. In order to impart ductility it will be necessary to activate more number of slip systems and make $\langle 111 \rangle$ slip easier. For this to be possible the ordering energy of NiAl has to be reduced. This can be done by alloying with elements like Cr, Mn and V (Hong & Freeman 1991). Theoretical calculations have shown that up to 70% reduction in APB energy is possible by alloying (Hong & Freeman 1991). However, in order to realize this a very large addition of these alloying additions has to be made which will be far in excess of their solubility in this intermetallic compound. NiAl modified with these alloying additions appeared to show $\langle 111 \rangle$ slip as observed by some workers (Law & Blackburn 1987). However, this observation has not been confirmed by other studies nor has any marked improvement in tensile ductility been seen in this intermetallic compound after alloying. The only noticeable change has been in the area of fracture mode where a transition from intergranular cleavage to transgranular cleavage was observed (Law & Blackburn 1987).

Dey and others in their studies (Dey & Sekhar 1997; Dey & Sekhar 1999; Dey *et al* 2000), have examined the effect of minor alloying additions on the micropyretic synthesis of NiAl. These alloying additions have been made with a view to improve the mechanical properties of NiAl. Besides Cr, Mn and V, the other alloying additions that have been tried by Dey & Sekhar (1997) are Ti, Nb and B. The choice of Cr, Mn and V were governed by aforementioned factors. The selection of Ti, Nb and B was done due to the following reasons. Ti additions have been done to NiAl to improve ductility of the alloy and some success has been achieved in improving the fracture toughness of the intermetallic compound. In the continuous endeavour of ductilising intermetallic phases with B2 structure, its was noticed that it was possible to stabilize the B2 structure in Nb_3Al which otherwise has a A15 structure by addition of Ti (Kohmoto *et al* 1993). It is well known that Ti containing B2 structures are quite ductile (Kohmoto *et al* 1993). With this point in view, ternary Nb-Al-Ti alloys have been developed and these have been found to show considerable ductility at ambient temperatures (Kohmoto *et al* 1993). $\text{Nb}_{45}\text{Al}_{15}\text{Ti}_{40}$ is one such alloy in the Nb-Al-Ti system. It is interesting to note that where as NiAl has a B2 structure and is extremely brittle at room temperature under most testing conditions, the alloy $\text{Nb}_{45}\text{Al}_{15}\text{Ti}_{40}$ with a similar structure is ductile. The alloys of the $\text{Nb}_{45}\text{Al}_{15}\text{Ti}_{40}$ type have poor oxidation resistance due to the large amount of Nb present in it. In order to develop intermetallic phases having B2 structure, which are ductile as well as oxidation resistant Dey & Sekhar (1999) have tried alloying of NiAl with Nb in addition to Ti. Dey & Sekhar (1999) have observed that addition of Nb leads to increase in fracture toughness of the alloy which seems to be due to an increase in the ductility of the alloy. Similar results have been obtained by Kayseer *et al* (1991) in case of NiAl alloyed with Ti and Nb. The deformed microstructure of those alloys, which showed improvement in ductility, was examined in order to find out the nature of the dislocations contributing to deformation

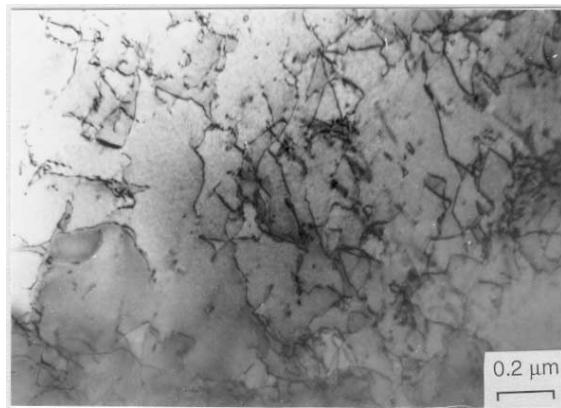


Figure 4. TEM micrograph showing the dislocation arrangement in a deformed specimen of $\text{Ni}_{49.25}\text{Al}_{49.5}\text{Nb}_{0.75}\text{Ti}_{0.5}$.

and their arrangement. Figure 4 shows the dislocation arrangement in the microstructure of $\text{Ni}_{49.25}\text{Al}_{49.5}\text{Nb}_{.75}\text{Ti}_{.5}$ alloy subjected to 4% compressive deformation (Dey & Sekhar 1999). The dislocation arrangement appeared to be heterogeneous in such microstructures and it was possible to see areas, which had much higher dislocation density as compared to others. Line direction analysis showed that the dislocations were lying either on $\{100\}$ or $\{110\}$ type of planes. Elongated prismatic loops could also be seen and these were mostly lying on the $\{100\}$ slip planes indicating that $\{100\}$ slip was active. The dislocation arrangement in the deformed NiAl with Nb and Ti was quite similar to that in the unalloyed NiAl. No substantial activity of the $\langle 111 \rangle$ dislocations could be seen in any of the alloys. The alloying additions made in this study particularly Nb and Ti are good choices for lowering the ordering energy of NiAl, thereby making $\langle 111 \rangle$ slip easier. The occurrence of $\langle 111 \rangle$ slip would lead to the fulfillment of the Von Mises criterion for ductility (Dieter 1988) which requires five independent slip systems to be active in order for the material to show noticeable amount of ductility. The observations made in the studies carried out by Dey & Sekhar (1997, 1999) and Sekhar *et al* (1996) as well as in some other studies (Darolia 1991; Dollar *et al* 1993) indicate that $\langle 111 \rangle$ slip does not occur in NiAl despite the alloying additions. The improvement in ductility after alloying seems to result from the enhanced mobility of the same type of dislocations, which are present in unalloyed NiAl. Darolia *et al* (1992) in their comparative study of deformed microstructures of binary NiAl and NiAl alloyed with Fe, Ga and Mo have observed similar type of dislocations and did not encounter any significant $\langle 111 \rangle$ dislocation activity. These authors have attributed the observed increase in dislocation mobility and ductility of NiAl alloys with the addition of small amounts of Fe, Ga and Mo to the following factors; gettering or trapping of interstitial elements, dislocation core structure interactions, slip system modification, electronic effects and stoichiometric effects. The dislocation core structure interactions and electronic structure modifications on alloying seem to be the most probable cause for the enhancement of dislocation mobility and increase in ductility out of these factors. In this regard, the observations made in the study carried out by Dey & Sekhar (1997) are similar to those of Darolia *et al* (1992) on microalloyed NiAl. An increase in the value of the fracture toughness has been observed in the case of NiAl with Fe, Cr and V addition and it has been attributed to the increase in the mobility of the same type of dislocations due to the aforementioned reasons (Dey & Sekhar 1997). However, no change in the arrangement of dislocations has been observed in the unalloyed NiAl and that containing Fe, Cr and V.

Addition of B and Ti in the alloy Fe_3Al has lead to the improvement of high temperature strength and refinement of grain size (Noebe *et al* 1993). In an analogous manner, it was

hoped that addition of Ti and B to NiAl would improve the mechanical properties by forming borides or improving the grain boundary strength (George & Liu 1990). Dey *et al* (2000) have examined the effect of Ti and B additions on the process of micropyretic synthesis of NiAl and its microstructure.

7. Typical microstructures and effect of alloying and processing conditions on microstructure

Various types of microstructures have been observed in Ni_3Al depending upon the processing route. The most common microstructure is the more or less equiaxed microstructure seen in the cast and wrought condition. Such microstructures have been found to contain a very few antiphase domain boundaries (Stoloff & Liu 1997). In the microstructure produced by rapid solidification, it has been observed that the grain size is rather small, there is extension of solid solubility and an increase in homogeneity. Dey *et al* (1994) have examined the formation of Ni_3Al in a laser alloying process. The Ni_3Al phase formed by laser treatment of a piece of nickel on which Al and Mo powders were pasted. The microstructure was found to comprise cells of the Ni_3Al phase. A gradient in the degree of order could be observed within the cells of the Ni_3Al phase. Rapid solidification resulted in the development of a partially ordered alloy with very fine domains in the central portion of the cells. The alloy solidified by direct ordering into larger domains in the central regions of the cells.

The microstructure produced in Ni_3Al after combustion synthesis has also been examined (Moore & Feng 1995). However, this examination has been done mostly at optical microscopy level. No detailed study at TEM level seems to have been done on the microstructures of Ni_3Al synthesized by this technique.

Interstitial alloying additions like that of boron, to the extent of about 2000 ppm, have not been found to bring any change in the microstructure of Ni_3Al . Precipitation of borides has not been noticed at the grain boundaries with this level of boron in the alloy. Boron is known to segregate to the grain boundaries. Substitutional alloying additions have various solubilities in the alloy and their occupancy of the lattice also differs. Elements like Co, Cu, Pd, Pt and Sc occupy the Ni sites while Al sites are occupied by elements like Si, Zn, Ti, V, Mn, Zr and Ta. There are some elements, which can occupy either site, like Fe, Cr, Mo and W. Many of these alloying additions influence the critical resolved shear stress for slip on different slip systems.

If alloying additions exceed the solubility limits, multiphase alloys result. Multicomponent alloys have been developed where elements like Cr has been added to Ni_3Al to reduce environmental embrittlement in oxidizing atmosphere at elevated temperatures. For solid solution strengthening at high temperatures alloying additions like Zr and Hf has been made. It is beneficial to have about 10 vol% of the disordered γ phase in the alloy by suitable alloying additions. This results in lower environmental embrittlement in oxidizing atmosphere and improved creep properties at elevated temperatures. Many of the multicomponent alloys are meant for use in the cast condition. In this condition the alloy has a coarse grained microstructure. Some of the alloys are used in the wrought condition and often have a fine-grained microstructure. Though such a microstructure has excellent ambient temperature strength, it suffers from poor creep resistance due to grain boundary sliding. Dispersion strengthened Ni_3Al with TiB_2 , Al_2O_3 , ThO_2 or Y_2O_3 as dispersoids have also been prepared (Stoloff & Liu 1997). Their properties are not very well known. Attempts to develop composites with Ni_3Al as the base have not been very successful.

Analogous to Ni_3Al , attempts have been made in the case of NiAl to modify the microstructure and bring about improvement in ductility and toughness. These efforts include refinement

of grain size and development of multiphase alloys. The microstructure of micropyretically synthesized NiAl with alloying addition has been examined by Dey & Sekhar (1997) before and after preheating of the specimens. Dey & Sekhar (1997, 1999, 2000) have synthesized this intermetallic compound with preheat and composition as variables. The alloying additions that have been chosen are Fe, Cr, V, Nb, Ti and B. The solubility of Cr in NiAl is known to be low (1.5 wt% Cr) (Noebe *et al* 1993; Dey & Sekhar 1997). The addition Fe in the study carried out by Dey & Sekhar (1997) was well within solubility limits in NiAl (Noebe *et al* 1993). The solubility of V is about 5 at% when substituted for Al and nil when substituted for Ni (Noebe *et al* 1993). In the studies carried out by Dey & Sekhar (1997, 1998), the microstructures of the alloys containing small additions of Cr, V and Fe were identical and comprised equiaxed grains. The grain interior as well as the grain boundary did not reveal any second phase when observed under the optical microscope. The small alloying additions did not bring about a very noticeable change in the grain size. Pores of different sizes could be seen in the microstructure of all the alloys in addition to the grains. Micropyretic synthesis is a process in which alloying occurs at a local level at a very rapid rate. In a process of this nature, the homogeneity of the resulting microstructure in terms of phase content and composition is important. Energy dispersive spectroscopy (EDS) point count analysis at the center of the grains as well as at the boundary of NiAl synthesized micropyretically by Dey & Sekhar (1997) after additions of alloying elements did not reveal any noticeable difference in the composition of the two regions (Dey & Sekhar 1997). The study carried out by Dey & Sekhar (1997) has shown that the compositional and microstructural homogeneity of the micropyretically synthesized product is unusually good. Since V substituted for Al, it should not normally precipitate out although formation of a V bearing grain boundary precipitate phase was observed in the case of the preheated specimen (Dey & Sekhar 1997).

Some studies have shown that Ni_3Al can have small amount of tetragonality when present in certain Ni based alloys (Dey *et al* 1993). Tetragonality in the B2 lattice can also develop as a result of alloying. This aspect has been examined by Dey & Sekhar (1997) in micropyretically synthesized NiAl, where, however, selected area electron diffraction (SAD) carried out in the TEM and X-ray diffraction did not reveal the presence of tetragonality.

The solubility of Ti in equiatomic NiAl is 10 at.%. In the study carried out by Dey *et al* (2000) involving NiAl with Ti and B addition, the amount of Ti added was less than required for the formation of the Ni_2AlTi phase. It should be noted that only a part of the Ti is going to the NiAl lattice. Substantial part of it is consumed in the formation of the TiB_2 phase. B is likely to occupy the interstitial positions in the NiAl lattice because of the size of the B atom (Dey *et al* 2000). However, when B is being added along with Ti, it will try to form TiB_2 rather than alloy with NiAl. This was found to happen in the study carried out by Dey *et al* (2000). In their study involving micropyretically synthesized NiAl with Ti and B addition, the unpreheated specimen showed a multiphase microstructure comprising a matrix of the NiAl phase and the grains of this phase did not have equiaxed morphology as seen in the case of unalloyed NiAl. Instead the grains had a spherical morphology and at the junction of these grains a mixture of phases appeared to be present. The effect of addition of Ti and B was to add additional enthalpy by the formation of the TiB_2 phase. It could be inferred that due to higher heat input, the microstructure changed to the spherical or the dendritic form as compared to the equiaxed microstructure seen in the binary NiAl. The grains were free from any kind of mottling. These observations were suggestive of the fact that the alloying additions could be accommodated in NiAl lattice as substitutional solid solution elements.

If the limit of solubility of Ti is exceeded in NiAl containing Ti, the formation of the Ni_2AlTi phase is expected. The structure of the NiAl and the Ni_2AlTi phases are very similar.

Ni_2AlTi is also known as the Heusler or B' phase. It has a DO_3 structure and forms by further ordering on the Al sites of the CsCl structure by Ti (Darolia 1991). The solubility of Nb in NiAl is 5 at.% (Dey & Sekhar 1999). Besides NiAl with the B2 structure, the other binary, ternary and quaternary phases expected in alloys containing both Ti and Nb are the following; Nb_2Ni , NbNi , $\text{Al}_{4.6}\text{Ni}_{23.4}\text{Nb}_{24}$, AlNbNi , AlNbNi_2 , $\text{Al}_{5}\text{Nb}_{5}\text{Ni}_3$, AlNb_2 besides a large number of phases in the NiTi system (Pearsons 1992). It has been apprehended that addition of Nb to NiAl is likely to lead to the formation of sigma phase (Nb_2Al) in the alloys and formation of this phase can severely impair the properties of the alloy (Dey & Sekhar 1999). These authors have carried out extensive microstructural investigations in NiAl with Ti and Nb addition and have used x-ray diffraction and TEM for the examination of these alloys. It has been observed in their studies on these alloys that in alloys containing smaller amounts of Ti and Nb the main phase to form is the B2 NiAl . As the amount of Ti and Nb was increased ($\text{Ni}_{29.25}\text{Al}_{49.5}\text{Nb}_{0.75}\text{Ti}_{0.5}$) the AlNbNi phase was found to form which has a hexagonal structure having the lattice parameters $a = 0.500$ nm and $c = .8093$ nm (Pearsons 1992). This was followed by the appearance of the Nb_2Al phase in alloys containing still higher amounts of Ti and Nb as in the alloy $\text{Ni}_{45}\text{Al}_{45}\text{Nb}_5\text{Ti}_5$. In an alloy having the composition $\text{Ni}_{25}\text{Al}_{32}\text{Nb}_{23}\text{Ti}_{20}$, it was possible to see the Ni_2AlTi phase which originates as result of further ordering of the B2 lattice to a DO_3 lattice (Dey & Sekhar 1999). Finally the B2 NbAlTi phase could be seen in $\text{Ni}_{14}\text{Al}_{24}\text{Nb}_{33}\text{Ti}_{29}$ alloy which had very large amount of Nb and Ti.

It has been shown in recent studies that the extent of preheat can have profound influence on the microstructure and mechanical properties of the micropyretically synthesized alloys (Dey & Sekhar 1997; Dey *et al* 2000). The extent of preheat has not only been found to influence the morphology of the grains but these have also been found to influence the nature of the phases present (Dey & Sekhar 1997; Dey *et al* 2000). A detailed account of how the extent of preheat influences the microstructure in case of NiAl with Fe, Cr, V, Ti and B addition has also been given in the same study. An increasing amounts of preheat transformed the grain morphology from equiaxed to dendritic in the alloys containing Cr, Fe and V. In addition to the morphology of the grains, preheating also influenced the extent of segregation of the alloying additions and the phase formation at the grain boundaries. In the case of specimen made with preheat, the specimens cool down more slowly allowing enough time for segregation and formation of the second phase particles of phases like V_2FeAl at the grain boundaries in specimens containing Cr, Fe and V as compared to a fully homogeneous microstructure in the unpreheated specimen (Dey & Sekhar 1997). The extent of preheat had a major influence on the microstructure and the nature of phases present in the micropyretically synthesized $\text{Ni}_{45}\text{Al}_{45}\text{Ti}_8\text{B}_2$ alloy. In the preheated specimens of this alloy, the formation of the TiB phase could be seen in addition to that of TiB_2 .

Attempts have been made to synthesize composites containing NiAl as base and particulate or fiber-like or semicontinuous reinforcements in order to make tough alloys. NiAl with 9 at% Mo and having an eutectic microstructure showed a much improved fracture toughness once the microstructure was altered by directional solidification to comprise an eutectic composite consisting of ~ 11 vol% of 1 μm diameter rods in a single crystal NiAl matrix (Noebe *et al* 1997).

8. Concluding remarks

Understanding of the structure, crystallographic defects, alloying and deformation behaviour of Ni_3Al has reached a state of considerable maturity, making the application and commercialization of this intermetallic compound a distinct possibility in the very near future. Though

these aspects have also been examined in case of NiAl, the amount of research carried out in case of this intermetallic compound is less in comparison. The microstructures generated in these alloys by various processing routes have been examined in considerable detail. The high strength combined with low-density make these intermetallics very attractive for many technological applications. However, the poor ductility and impact strength particularly in case of NiAl is a formidable problem. This issue has been addressed by looking into the possible slip systems in NiAl and ascertaining means of enhancing their number in order to promote ductility. One major means that has been considered for this purpose is alloying. More extensive research needs to be done to address this problem. The environment has been found to influence the phenomenon of embrittlement in Ni₃Al. Alloying has been found to play a major role in alleviating this shortcoming.

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