

**THE EFFECT OF ALLOYING ELEMENTS ON
WEAR BEHAVIOUR OF Ni-Al**

by

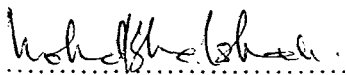
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of the requirements for the degree of
Master of Science**

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Saya isytiharkan bahawa kandungan yang dibentangkan di dalam tesis ini adalah hasil kerja saya sendiri dan telah dijalankan di Universiti Sains Malaysia kecuali dimaklumkan sebaliknya. Tesis ini juga tidak pernah disertakan untuk ijazah yang lain sebelum ini.

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ABBREVIATION

GaAs	Gallium Arsenide
Ni-Al	Nickel aluminide (50 at. % Al and 50 at. % Ni)
Ni₃Al	Nickel aluminide (25 at. % Al and 75 at. % Ni)
Ni₂Al₃	Nickel aluminide (60 at. % Al and 40 at. % Ni)
Ni₅Al₃	Nickel aluminide (37.5 at. % Al and 62.5 at. % Ni)
Rpm	Rotation per minute

ABSTRACT

Considerable amount of research has been performed on nickel aluminide (NiAl) over the last decade, with an exponential increase in effort occurring over the last few years. The enormous potential of NiAl systems mainly from its many attractive properties, such as high oxidation and corrosion resistance and relatively low densities, combined with the ability to retain strength and stiffness at elevated temperature. Furthermore, NiAl has emerged as potential high temperature structural material because of its high melting temperature. The main objective of this research is to synthesis Ni-Al by reaction sintering of partial mechanically alloyed powder and to evaluate the wear behaviour of the prepared materials. The use of mechanical alloying (MA) process to fabricate fine-structured NiAl has received increasing attention in the last few years. MA is a high-energy ball milling process to produce materials with homogenous microstructure and novel properties such as good thermal conductivity and chemical stability. The present study aims to synthesis Ni-Al by reaction sintering of partial mechanically alloyed powder and to evaluate the wear behaviour of the prepared materials. However, the influence of compaction pressure, fabrication method and sintering atmosphere also considered. The mixed powders were mechanically alloyed in a high-energy planetary ball mill for 2 and 4 hours followed by compaction at 200, 250, 300, 350, 400 and 450 MPa. Then, reactive sintering was done in argon and normal atmospheres. Instead of trying to alter the inherent properties of Ni-Al, an alternative approach to improve the ductility and toughness of Ni-Al is by modifying the microstructure of the material. The idea is to improve the mechanical behaviour of Ni-Al through extrinsic toughening mechanisms. Micro alloying additions of B, Cr, Fe, and Mo at 0.1 at. % have been studied in this research. These elements react as ductile reinforcing agents and may strengthen Ni-Al at high temperature. It is found that, Ni-Al with B additions milled for 2 hours, compacted at 250 MPa and sintered in an argon atmosphere showed the best density, microstructure and hardness results. In addition, this sample also has a highest wear resistance compared with other samples.

ABSTRAK

Banyak penyelidikan telah dijalankan ke atas NiAl sejak sedekad yang lalu dan usaha terhadap kajiannya meningkat sejak beberapa tahun yang lepas. NiAl mempunyai potensi besar yang disumbangkan oleh sifat-sifatnya yang menarik seperti rintangan pengoksidaan dan kakisan yang tinggi dan ketumpatan yang rendah. Sifat-sifat ini apabila digabungkan mempunyai keupayaan untuk menahan kekuatan dan kekakuan pada suhu yang tinggi. Selain daripada itu, NiAl berpotensi untuk digunakan sebagai bahan struktur pada suhu tinggi disebabkan oleh takat leburnya yang tinggi dan konduktiviti terma yang baik. Tujuan utama penyelidikan adalah untuk sintesis Ni-Al melalui persinteran reaktif dan mengkaji kelakuan haus sampel yang disediakan. Proses pengalioan mekanikal yang digunakan untuk menyediakan struktur Ni-Al yang halus mendapat perhatian yang tinggi dalam beberapa tahun yang lalu. Pengalioan mekanikal adalah satu proses pengisaran bebola bertenaga tinggi untuk penghasilan bahan dengan mikrostruktur yang seragam dan ciri-ciri yang baru seperti konduktiviti terma dan kestabilan kimia yang baik. Kajian ini bertujuan untuk sintesis Ni-Al melalui persinteran reaktif dan mengkaji kelakuan haus sampel yang disediakan. Kesan tekanan pemadatan, kaedah fabrikasi dan atmosfera persinteran juga dilakukan kajian. Campuran serbuk ini dilakukan pengalioan mekanikal di dalam pengisar bebola (planetary ball mill) selama dua dan empat jam diikuti oleh proses pemadatan pada 200, 250, 300, 350, 400 dan 450 MPa. Kemudian, persinteran reaktif dijalankan di dalam atmosfera biasa dan gas argon. Selain daripada mencuba untuk mengubah sifat-sifat semulajadi Ni-Al, satu pendekatan lain telah diambil untuk memperbaiki kemuluran dan kekuatan melalui pengubahsuaian mikrostruktur bahan ini. Tujuannya adalah untuk memperbaiki sifat mekanik Ni-Al melalui mekanisme penguatan ekstrinsik. Di dalam kajian ini, penambahan unsur-unsur pengalioan mikro seperti B, Cr, Fe dan Mo pada 0.1 at. % telah dikaji. Elemen-elemen ini bertindak sebagai agen meningkatkan kemuluran dan boleh menguatkan Ni-Al pada suhu yang tinggi. Keputusan menunjukkan Ni-Al dengan penambahan unsur B yang melalui proses pengisaran selama dua jam diikuti dengan proses pemadatan sebanyak 250 MPa dan disinter dalam atmosfera gas argon memberikan hasil dengan sifat ketumpatan, mikrostruktur dan kekuatan yang terbaik. Selain daripada itu, sampel ini juga menunjukkan kadar rintangan haus yang paling tinggi jika dibandingkan dengan sampel-sampel yang lain.

CHAPTER 1

INTRODUCTION

1.1 An overview of study

Intermetallic compounds have been known for a very long time, but their impact on the metallurgical community has been relatively insignificant, however, that is now changing. Intermetallic compounds are a unique class of material, consisting of ordered alloy phases formed between two or more metallic elements where the different atomic species occupy specific sites in the crystal lattice.

As with many intermetallic alloys, nickel aluminide (NiAl) was originally studied as a potential structural material because of its high melting temperature, hardness, and chemical stability. The surface science community became intrigued with the surface properties and catalytic behaviors of NiAl. The present work was motivated by the fact that NiAl is one of the few intermetallic systems known to have stable, well defined surface structure on the atomic level.

NiAl has a high melting point, excellent thermal stability and serendipitous lattice match that of GaAs compounds, the electronics industry began to take a serious look at the possibility of using NiAl as interconnect in electronic components (Sands, 1988; Chambers and Loebis, 1990; Joo et al., 1992). In addition, research on NiAl focused on its possible use as a high temperature structural material. The renewed driving forces for this

general application have been generic government aero propulsion programs (Stephen, 1988; Doychak, 1992) and industrial development of NiAl as a turbine engine material (Darolia, 1991; Darolia et al., 1992).

However, because of its poor creep resistance at elevated temperatures, NiAl has not been widely used in structural applications, although it is used as coating on super alloys for oxidation resistance. Another deterrent to the use of NiAl in structural applications is its lack of room temperature ductility and toughness. The lack of ductility at low temperatures is attributed to an insufficient number of slip system due to the operation of $\langle 100 \rangle \{hkl\}$ slip, which provides only three independent slip systems (Stoloff and Sikka, 1996).

Consequently, strain compatibility between grains cannot be achieved and hence, the room temperature tensile ductility of NiAl is almost nil. In a single crystal NiAl, ductility and toughness are controlled by the formation of cleavage micro crack on slip bands and their subsequent instability. It was found that the presence of moisture and oxygen causes these failures.

In this research work, NiAl is fabricated by powder metallurgy route because this method offers advantages such as economical production of complex parts. The parameter must be controlled carefully to ensure that the good results obtained.

1.2 Objectives

There are several objectives in this research, which includes:

- (a) To study the fabrication method of NiAl. A mold was used during sintering to avoid from crack. There are two types of mold; stainless steel mold and graphite mold.
- (b) To characterize the fabricated NiAl. This includes the green density, bulk density, porosity, shape, and size of the particles and others.
- (c) To study the wear behaviour of NiAl. This is a very important area due to the fact that NiAl is widely used in high temperature rotating parts applications and subjected to a wear conditions. This will be carried out with stainless steel as the wear medium at room temperature.
- (d) To improve the hardness of NiAl at room temperature. Micro alloying element (B, Cr, Mo, and Fe) has been used in trying to solve this problem. These elements act as grain refinement agents. However, the percentage of the alloying element must be controlled to produce a good result.

1.3 Project Approaches

Early investigations were exploratory in nature, designed to determine whether NiAl held promise as a high temperature refractory compound (Wachtell, 1952). These were followed by studies in the early 1960s that concentrated on the effects of processing and other metallurgical variables on mechanical behavior (Wood et al., 1960). By the mid-1960s, NiAl was identified as a possible leading edge material for a super alloy turbine vane. However, no solution was found for the low temperature brittleness of this compound, and by the end of the 1960s, government and industrial interest in NiAl had faded. At this point, research shifted primarily to universities; between 1970 and the mid 1980s, there was a very small, but steady effort to investigate the oxidation behavior, mechanical properties, and deformation mechanisms of NiAl. Then, during the mid to late 1980s, research on NiAl exploded on several fronts. However, the current researchers face the same problems that hindered the widespread acceptance of NiAl as a structural material in the early 1960s: namely, its poor creep resistance and inadequate low temperature toughness and ductility.

This research focuses on solving the problem of NiAl brittleness at room temperature. Although there are many researches working on this aspect, there are still different views on NiAl's properties. In this work, methods that can improve NiAl properties are explored, and that means the work must be controlled very well, starting from the sample preparation until the final analysis. Although there are many studies on the effect of milling time, this present study is mainly about getting a homogenous mixture among the raw material. Compaction can be done easily although some additives are needed

because of the homogenization of powder mixture. The powder metallurgy route has been followed in order to produce a denser intermetallic compact. Reaction sintering was done in an argon atmosphere in order to avoid from oxidation. To minimize cracks, graphite mold is used.

Micro alloying with some additives are added in an attempt to improve the properties of NiAl by grain refinement. The refractory metal group like Cr, Mo, W, Re and V exhibit very little solubility in NiAl and form no ternary intermetallic phases, with the exception of V in Ni- rich alloys. Instead, these elements form pseudo binary eutectic systems with stoichiometric NiAl and therefore, may both strengthen NiAl at high temperatures and act as ductile reinforcing agents at low temperature.

The current work explores the physical and mechanical metallurgy of Ni-Al based materials, concentrating on the effects of processing, alloying, and micro structural modification on the mechanical behaviour.

CHAPTER 2

LITERATURE REVIEW

2.1 The Definition of Intermetallics

Intermetallic is the short and summarized designation for the intermetallic phases and compounds which result from the combination of various metals and form a tremendously numerous and manifold class of materials. According to a simple definition by Schulze (1967) and Girgis (1983), intermetallics are compounds of metals whose crystal structures are different from those of the constituent metals and thus, intermetallic phases and ordered alloys are included. In addition, Callister (1996) has defined intermetallic as a compound of two metals that has a distinct chemical formula. On a phase diagram, it appears as an intermediate phase that exists over a very narrow range of compositions.

During the last ten years, intermetallics have received enormous interest in materials science and technology with respect to applications at high temperature and a new class of structural materials is expected to be developed on the basis of intermetallics. Various materials developments are under way in various parts of the world, particularly the USA, Japan, and Germany. These activities are the main subject of the latest papers and other literatures.

However, it is worth mentioning that besides the intermetallics with outstanding high temperature properties, there are other intermetallics with outstanding physical

properties. These have given rise to developments of new functional materials much earlier (mid 1980s) and which will be addressed.

2.2 General Considerations

2.2.1 Bonding, Crystal Structure and Phase Stability

Intermetallic is formed because the bond between the respective different atoms is stronger than that between similar atoms (Cahn and Kramer, 1992). Accordingly, intermetallics form particular crystal structures and the atoms are distributed in an orderly fashion.

The crystal structure of an intermetallic material is determined by the strength and character of the bonding, which depends on the particular electronic configuration. The relation between structure type and atomic properties of the constituent's atoms is not a simple one and thus various criterions have been used in correlating structure types and phase types to predict the crystal structure for a given phase or phase group. Likewise, the intermetallics cannot be expected to show similar metallic bonding to the individual's metals. Table 2.1 in the next page shows the crystalline structures in the Ni-Al system.

Table 2.1: List of crystalline structures in the Ni-Al system (Cahn and Haasen, 1996)

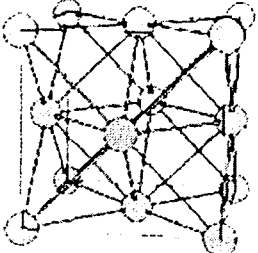
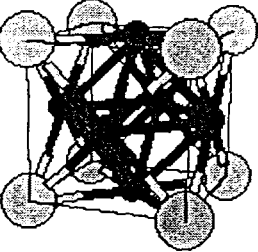
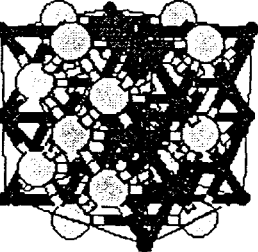
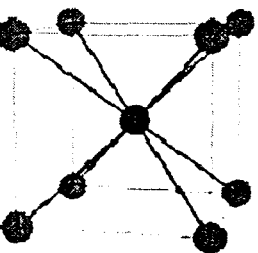
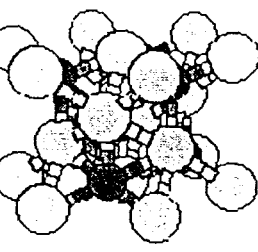
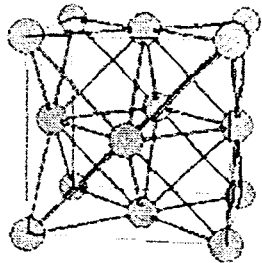
Graphic structure	Composition	Lattice Parameters
	Ni	$a = 0.352 \text{ nm}$
	Al Ni ₃	$a = 0.357 \text{ nm}$
	Al ₃ Ni ₅	$a = 0.744 \text{ nm}$ $b = 0.668 \text{ nm}$ $c = 0.744 \text{ nm}$
	Al Ni	$a = 0.288 \text{ nm}$
	Al ₃ Ni ₂	$a = 0.4036 \text{ nm}$ $c = 0.490 \text{ nm}$

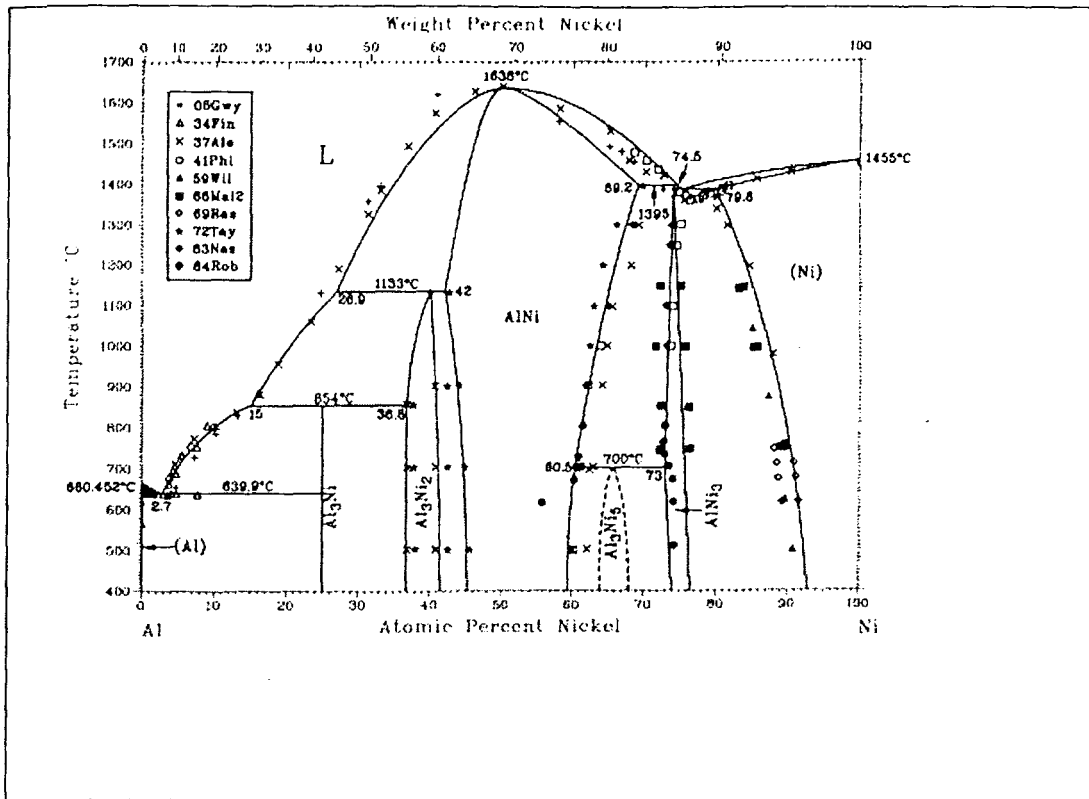
Table 2.1: List of crystalline structures in the Ni-Al system (Cahn and Haasen, 1996) Continued.

Graphic structure	Composition	Lattice parameters
	Al	$a = 0.405 \text{ nm}$

2.2.2 Basic properties of NiAl

As shown in the phase diagram in Figure 2.1, the phase NiAl has an extended range of homogeneity and melts congruently at about 1640°C for the stoichiometric composition with 50 at. % Al. This melting point is higher than those of the constituent elements, which indicate a strong bonding between Ni and Al and corresponding high phase stability with a strong tendency for atomic ordering.

The density of NiAl is relatively low at 5.9 g/cm^3 for the stoichiometric composition compared with conventional Ni-base alloys and it increases with decreasing Al content (Harmouche and Wolfenden, 1987). It is noted that the density change per unit of Al content is larger for Al-rich NiAl than for Ni-rich NiAl because of the difference in the defect structure.



The phase diagram in Figure 2.1 shows the intermetallic NiAl group. This group can be divided into 5 phases. The major phase is NiAl followed by Ni₃Al, NiAl₃, Ni₂Al₃, and Ni₅Al₃. NiAl has a melting temperature of 1638⁰C. It is the highest melting point in this group. That is why NiAl is widely used in high temperature applications such as in the aerospace industry. Table 2.2 listed the characteristics of the NiAl group.

Table 2.2: Special points of the Al-Ni phase diagram (Nash and Murray, 1991).

Reaction	Composition of the Respective phases, at. % Ni			Temperature, °C	Reaction type
$L \rightleftharpoons Al$		0		660	Melting
$L \rightleftharpoons Al_3Ni + Al$	2.7	25	0.11	640	Eutectic
$L + Al_3Ni_2 \rightleftharpoons Al_3Ni$	15	36.8	25	854	Peritectic
$L + AlNi \rightleftharpoons Al_3Ni$	26.9	42	40	1133	Peritectic
$L \rightleftharpoons AlNi$		50		1638	Congruent
$AlNi + AlNi_3 \rightleftharpoons Al_3Ni_5$	60.5	73	66	700	Peritectoid
$L + AlNi \rightleftharpoons AlNi_3$	74.5	69.2	73.75	1395	Peritectic
$L \rightleftharpoons Ni + AlNi_3$	75	79.8	74	1385	Eutectic
$L \rightleftharpoons Ni$		100		1455	Melting

The melting points of the pure metals are 660⁰C for Al and 1455⁰C for Ni. The Al-rich eutectic point (2.7 at. % Ni at 640⁰C) is based on thermal analysis data from Figure 2.1. From Table 2.2, NiAl shows the highest melting point at 1638⁰C and the reaction type is congruent. That means, there are no compositional alterations.

2.3 Processing and Fabrication

2.3.1 General and Previous Study

Compared to most intermetallics, the processing of NiAl is relatively easy in spite of the high melting temperature and limited room temperature ductility. Processing is greatly facilitated by a wide single-phase field, congruent melting point, single-phase microstructure, and high ductility above the brittle to ductile transition temperature. Therefore, NiAl can be fabricated into polycrystalline, single crystal, or composite form via a variety of processing routes. Numerous conventional processing techniques have been employed, including powder metallurgy (PM), casting and extrusion, directional solidification and some less orthodox techniques such as mechanical alloying (MA) and reaction synthesis. Even though NiAl has low fracture toughness at room temperature, machining complex geometries from the as fabricated material is possible with standard techniques such as grinding, abrasive machining, and electro discharge machining (EDM).

There are many research carried out in the mid 1980s until now in areas related to nickel aluminides. German, Rabin and Bose (1988) studied the reactive sintering of nickel aluminides to near full density. A novel processing route termed reactive sintering is used in their work for the fabrication of near full density nickel aluminides. The process involved the formation of a compound from its elemental constituents through an exothermic reaction. The liquid formed during the reaction causes rapid densification. As a result, densities in excess of 95 % of theoretical are possible through appropriate

selection of particle sizes, composition, heating rate, sintering temperature, and sintering atmosphere.

Hwang and Lu (1992) studied the reaction sintering of 0.1 at. % B doped nickel aluminides. The sintering temperature was at 500⁰C to 750⁰C and a tube furnace was used under argon or vacuum atmosphere. Results showed that the highest sintered density is obtained by using compacts of fine Ni and Al powders. The addition of 0.1 wt. % B lowered the sintered density of nickel aluminides from approximately 96 % for undoped material to about 93 % of theoretical density for the B doped material.

Other notable research work done included the work of Golberg and Shevakin (1994) that both noted that ferum element exhibits a large solubility in B2 intermetallic compound, NiAl and even forms an isostructural aluminide FeAl. To date, a particular emphasis has been put on Fe effects on poor room temperature ductility and limited high temperature strength of NiAl.

Later, Ronald and Michael (1996) reported on established alloy design, especially for alloying NiAl. They found out that the refractory metal elements (Cr, Mo, and Re) exhibit very little solubility in NiAl and form no ternary intermetallic phases. Instead, these elements form pseudo binary eutectic systems with stoichiometric NiAl and therefore, may both strengthen NiAl at high temperatures and act as ductile reinforcing agents at low temperatures. Alloying addition must be controlled in certain percents (0.1-

0.2 at. %) to ensure consistently and significantly increase the room temperature tensile ductility of soft orientation, single crystal NiAl.

Further works included the findings of Piezonka and Molinari (1997) on dimensional changes occurring during reactive sintering of Ni-Al-Mo elemental powder mixtures. Sintering treatment was conducted on powder compacts in a dilatometer at various heating rate. Sintering was performed in a horizontal Netzsch 402E dilatometer under vacuum better than 10^{-3} Pa. The holding time at the maximum sintering temperature of 750°C was 15 minutes. The results showed that heating rate strongly affects reaction sintering of Ni-Al-Mo compacts hence, giving effect to the density of the sintered products. The reaction between elemental powders is very sensitive to the heating rate, especially when aluminium rich compacts are considered.

Later on, Ryu, Shim and Hong (1997) studied the reactive processing and mechanical properties of ZrO_2/NiAl intermetallic matrix composite. Mixtures of Ni and Al powders with reinforcing particles of partially stabilized 10% ZrO_2 have been ball milled and hot pressed at 980°C and 30 MPa. As a result, 10% ZrO_2/NiAl composite fully densified above 99% was successfully synthesized by the hot pressing of ball- milled powders of Ni, Al and ZrO_2 at 980°C and followed by hot extrusion at 1200°C . Meanwhile, Kiyotaka, Toshiki and Masayuki (1997) studied the microstructure and mechanical properties of NiAl intermetallic compound synthesized by reactive sintering under pressure. The powder mixture was cold pressed into cylindrical green compacts in a metal mould, up to a maximum compacting pressure of 1400 MPa. The compacts were sintered in air under

several levels of pressure up to 360MPa and at several temperatures up to a eutectic temperature of 913K. The pressure was applied by using a pseudo – HIP (pseudo-Hot-Isostatic-Pressing). Figure 2.2 below shows the relationship between pressure and hardness. Plainly, as the pressure increases, the hardness also increases.

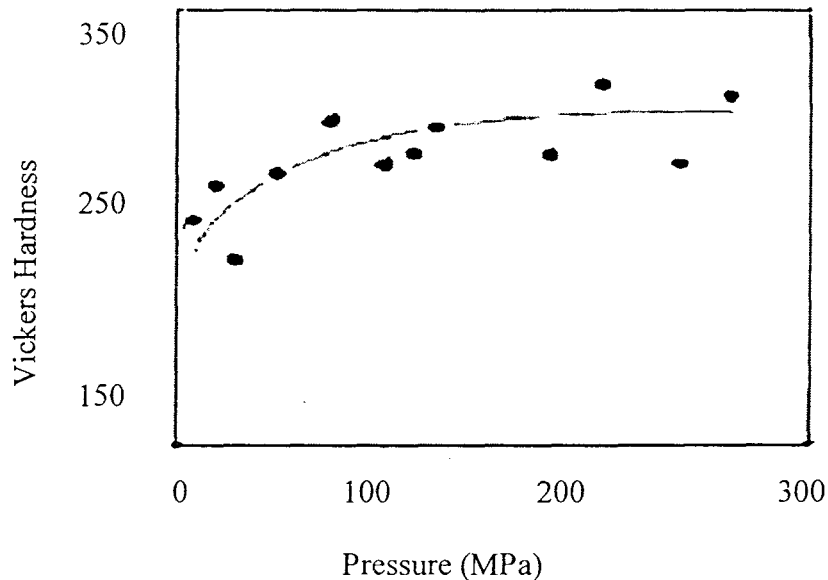


Figure 2.2: Effects of the sintering pressure on the hardness of NiAl, (Kiyotaka, Toshiki and Masayuki, 1997)

Jin and Stephenson (1998) then studied the sliding wear behavior of reactively hot pressed nickel aluminides. The influence of load and intermetallic stoichiometry on wear rate was done using a block on ring test method with 440C as the counter face material. Results showed that wear rate increases linearly with load and decreases as the nickel content increases. Figure 2.3 in the next page illustrated the formation of such a wear debris particle.



(a) Initial asperity contact



(b) Junction failure



(c) Transfer of particle



(d) Secondary contact



(e) Merging of two particles



(f) Transfer particle just
before detachment



(g) Transfer particle large enough
to support total load



(h) Compressed transfer particle



(i) Further compression and shear
due to sliding

Figure 2.3: Mechanism by which composite wear debris may form (Sasada, 1984)

Afterwards, Yang and Sun (2002) worked on the critical temperature in the initiation of plastic strain in nearly dislocation-free crystals of NiAl. The plastic deformation in nearly dislocation-free NiAl single crystals loaded along $\langle 001 \rangle$ initiated at a well-defined temperature. This was revealed by the anisothermal creep test. This test consists of

subjecting a specimen to a constant load or stresses while the temperature not constant; deformation or strain is measured and plotted as a function of elapsed time. All the results of nearly dislocation-free NiAl single crystals showed that the plastic strain initiated abruptly with increasing temperature. A typical deformation curve is shown in Figure 2.4 below, where the plastic displacement is plotted against the sample temperature for a test under 475 MPa and at a heating rate of 1.30 K/h. The plastic displacement remains at zero, with rising temperature, up to about 708 K at which there is an abrupt onset of plastic strain.

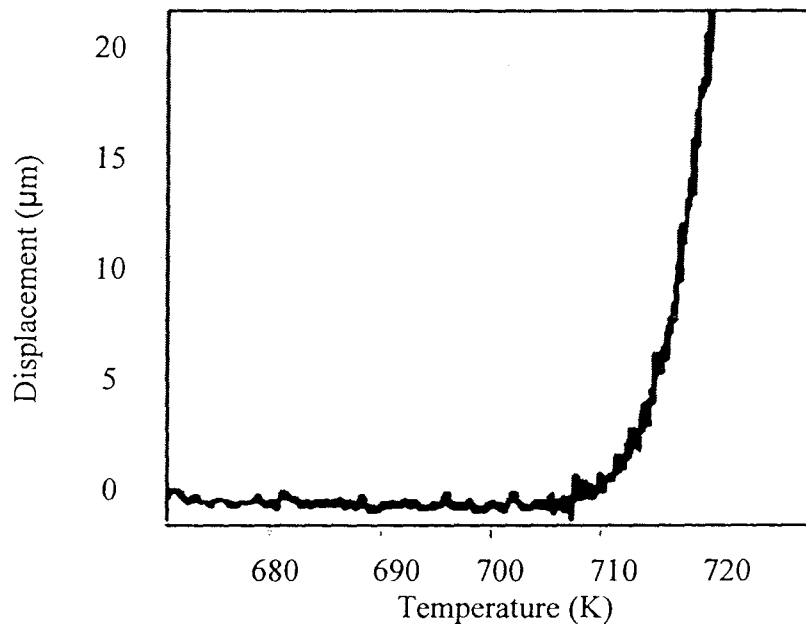


Figure 2.4: Plastic displacement versus temperature under a stress of 475 MPa and heated at a rate of 1.3 K/h. The plastic strain initiated at approximately 708K (Yang and Sun, 2002)

Recently, Kovalev and Barskaya (2003) studied the effect of alloying on electronic structure, strength, and ductility characteristics of NiAl. The effect of ferum, chromium, cobalt, molybdenum, boron, and lanthanum doping on mechanical properties of NiAl was investigated. Alloying has a beneficial effect on the decrease of the ductile-brittle transition temperature and micro mechanism of fracture. The doping of nickel aluminide by Fe (2 at. %), Mo (2 at. %), Co (2 at. %) was studied by valence band XPS and plasmon loss electron microscopy. The produced powders were compacted by hydrostatic pressing ($P=500\text{MPa}$) and sintered at $900\text{-}1400^{\circ}\text{C}$. Figure 2.5 showed the results of the test of mechanical properties of alloys based on the NiAl in the tension, compression, and impact toughness. Figure 2.5 also exhibits the strongest effect of alloying on the brittle ductile transition temperature of NiAl exhibited in the tensile tests.

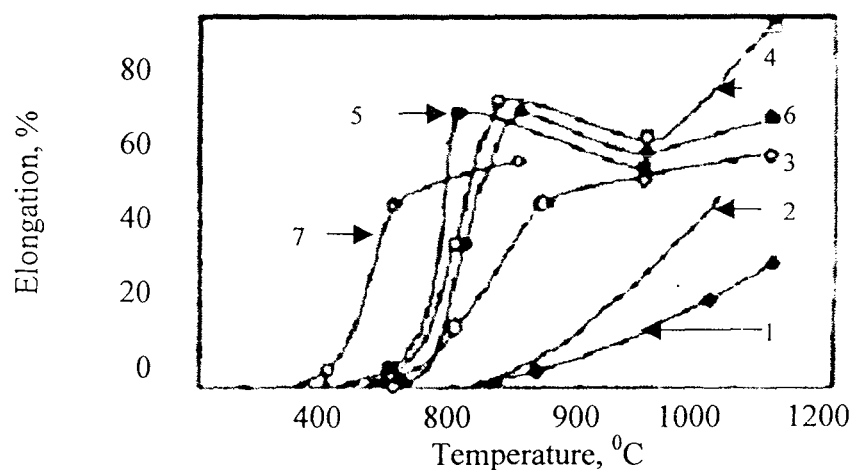


Figure 2.5: Elongation of NiAl alloys under testing at $400\text{-}1200^{\circ}\text{C}$. (1) casted NiAl; (2) extruded NiAl; (3) NiAl (Mo); (4) NiAl (W); (5) NiAl (Fe); (6) NiAl (Cr); (7) NiAl (Co, B, La) (Kovalev, Barskaya and Wainstein, 2003)

2.3.2 Powder Metallurgy

Among the various metal working technologies, powder metallurgy is the most diverse manufacturing approach. One attraction of powder metallurgy (PM) is the ability to fabricate high quality, complex parts to close tolerances in an economical manner. In essence, PM converts metal powders with specific attributes of size, shape, and packing into a strong, precise, high performance shape. Key steps include the shaping or compaction of the powder and then subsequent thermal bonding of the particles by sintering. The process can be effectively automated with low relative energy consumption, high material utilization and low capital costs. These characteristics make PM well aligned with the current concerns about productivity, energy, and raw material.

Consequently, PM process has become and replacing traditional metal forming operations. Further, powder metallurgy is a flexible manufacturing process capable of delivering a wide range of new materials, microstructures, and properties. That creates several unique and nice applications for PM such as wear resistant composites.

2.3.2.1 Definitions

A few terms must be understood before discussing about powder metallurgy. First, powder is defined as finely divided solid, smaller than 1 mm in its maximum dimension (German, 1994). In most cases, the powder will be metallic although in many instances they are combined with other phases such as ceramics or polymers. An important characteristic of the powder is its relatively high surface area to volume ratio. The particles severally exhibit behaviour that is intermediate between that of a solid and a

liquid. Powders will flow under gravity to fill containers or die cavities, so in this sense they behave like liquids. They are compressible like a gas. Nevertheless, the compression of a metal powder is essentially irreversible, similar to the plastic deformation of a metal. Thus, a metal powder is easily shaped, with the desirable behavior of a solid after processing.

Powder metallurgy is the study of the processing of metal powders, including the fabrication, characterization and conversion of metal powders into useful engineering components. The processing sequence involves the application of basic laws of heat, work, and deformation to the powder. It is the processing, which changes the shape, properties, and structure of a powder into a final product. The three main steps in powder metallurgy process are illustrated in Figure 2.6.

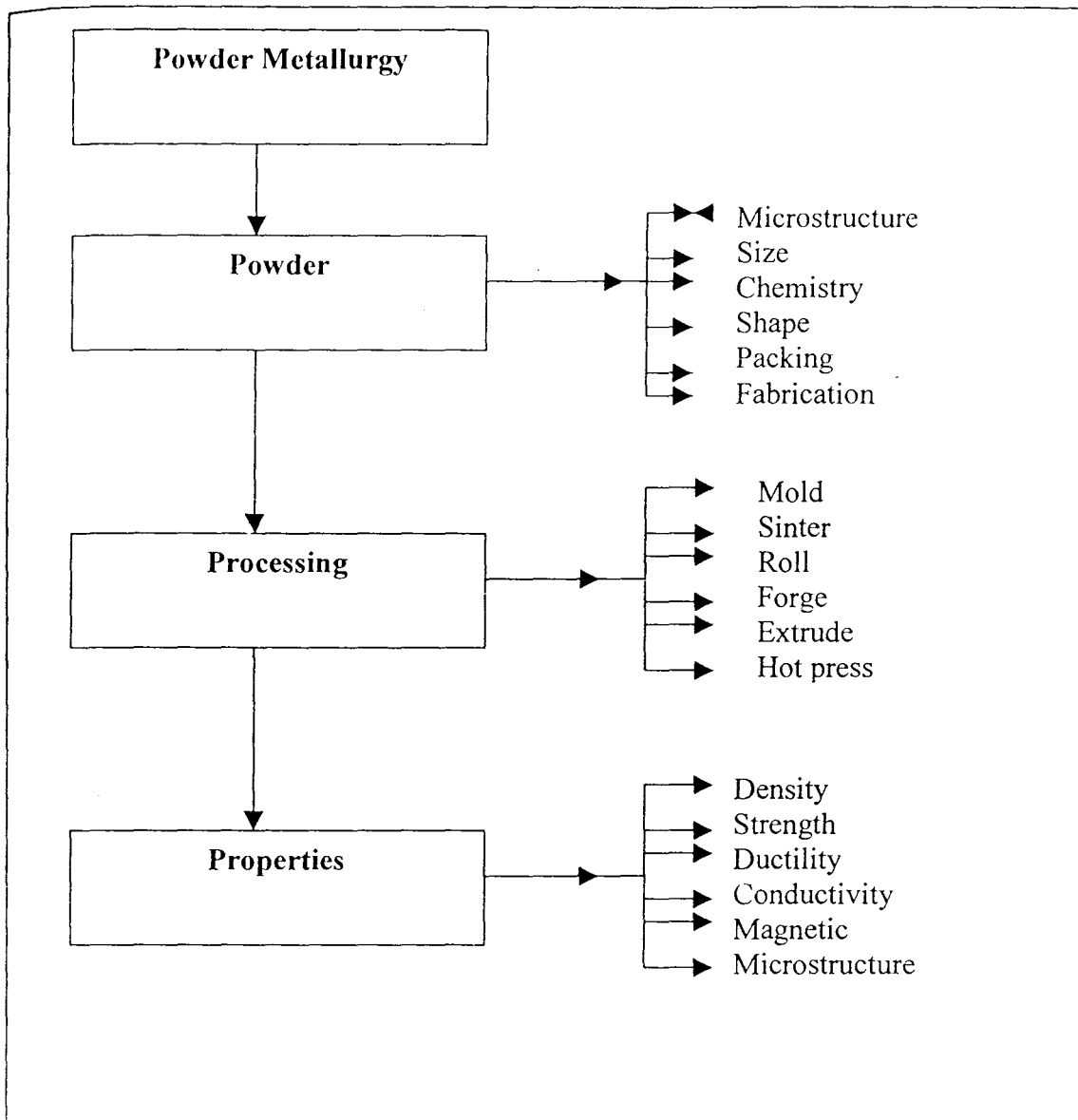


Figure 2.6: The conceptual flow for powder metallurgy from the powder through the processing to the final product (German, 1994)

2.3.3 Reactive Sintering

Pieczonka (1997) defined reactive sintering as a pressureless powder-processing route, which is used to produce intermetallic compounds from elemental powders. The process is a self-propagating or combustion synthesis. Indeed, the sintering process is sustained by the highly exothermic formation reactions of intermetallic compounds, which usually involve the formation of a liquid phase.

In this respect, reactive sintering of aluminides from elemental powders is similar to transient liquid phase sintering. The liquid appearing in the system rapidly spreads throughout the porous structure, engulfs the elemental powders and assists the formation of solid phases behind the advancing liquid interface. As the inter diffusion of components in the liquid phase is a comparatively fast process, it must be extremely carefully controlled.

The reactively sintered products (properties and characteristics) are determined by the starting raw materials (composition, powder purity, particle size, etc) and processing parameters (compaction pressure, degassing, heating rate, furnace temperature, etc). Pressureless densification of reaction-sintered materials is influenced by the wettability of the solid phase by the liquid and its capillary action.

The application of an external pressure during or after alloy formation may certainly improve the process and reduce the porosity, which in reactively sintered materials may occur for several reasons (German, 1994):

- (a) Original porosity of the reactants (usually about 30 %)
- (b) Molar volume reduction
- (c) Different diffusion flow of reactants, especially in intermetallic phases
- (d) Impurity generated porosity
- (e) Porosity from thermal migration

The heat from the furnace reactions (solid state) between the components until the reaction synthesis becomes self-propagating. Heating rate is an important parameter in reactive sintering as it influences the amount of heat generated by the exothermic reaction and, thereby, the amount of liquid which forms in the system. The higher the heating rate, the larger the rate of heat releases. On the other hand, the heating rate has to be limited in view of possible combustion reactions. The amount of liquid should be sufficiently high to densify the structure by capillary action and promote inter particle bonding. Too much liquid may cause shape loss in the sintered specimens and trap gases during the reaction in the compact. Therefore, an appropriate heating rate has to be selected in order to balance the need for liquid phase densification, inter particle bonding and sufficient degassing.

2.3.4 Single Crystals

The melt processing of stoichiometric single crystal is, in principal, relatively easy because of the congruent melting temperature and high ordering energy of NiAl. The high melting point however, push the conventional furnaces and crucible materials to their limit. Because the melting point of NiAl is 1955 K (Watson and Darolia, 1993), the required furnace temperatures is nearly 300K higher than those used to produce superalloys.

In spite of the high temperatures involved, NiAl single crystals have been successfully produced by a Bridgman melt processing technique, originally in small quantities used primarily in slip system studies (McDonnel, 1967). The commercial development of NiAl single crystals is presently being led by General Electric Aircraft Engines (GEAE) in an attempt to achieve the high temperature creep strength necessary to compete with current super alloys. Using a modified Bridgman process, ingots measuring 10.2 cm by 2.5 cm by 3.8 cm have been routinely produced from binary NiAl as well as from more highly alloyed compositions (Darolia, 1992). These ingots were to characterize the physical and mechanical properties of single crystal NiAl and NiAl alloys in an effort to identify a suitable composition for turbine blade applications.

Although these techniques are adequate for producing ingots for characterization, the fabrication of actual single crystal turbine blades is more involved. Starting with single crystal ingots and using a combination of electro discharge machining (EDM), electrochemical machining and grinding process, GEAE has successfully fabricated small