THE INFLUENCE OF HEAT TREATMENT ON MECHANICAL AND MICROSTRUCTURE PROPERTIES OF INTERMETALLIC NICKEL ALUMINIDE, Ni_3Al

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ABSTRACT: *Intermetallic Nickel Aluminides have been used in automotive industry with its superior characteristics such as lightweight, good resistance in high temperature and corrosion, and high oxidation. Mechanical studies including hardness carried out to confirm this Intermetallic Nickel Aluminides suitable for automotive wheel hub. The samples were heat treated for 400°C, 500°C and 600°C and observed Scanning electron microscope for their microstructural behaviour. X-ray diffraction was used to study the crystal structure of the alloy and grain size were calculated. The highest hardness value is the Ni_3Al sample at 600°C which is 213.38 HV at 0.5 kgf. Meanwhile the lowest value is at non-heat treated sample with the value of 56.66 HV at 2.0 kgf load force. Similar trend also observed for Ni_3Al grain size is at the largest at 600°C with 45.462 nm and the smallest during non-heat treated at 22.725 nm. This shows that temperature affects the materials characteristics.*

Index Terms: Nickel Aluminides, hardness, grain size, Intermetallic compound (IMC).

1. INTRODUCTION

Nickel Aluminides has been used as pumps, aluminium casting equipment, high temperature parts for aircraft and dies [1]. Currently intermetallic alloys are used for gaining heat-proof coatings in electrospark deposition (ESD) industries [2]. Furthermore, Ni_3Al has a good catalytic activity [3], ductile, hard, fair conductor and good resistance against corrosion [4]. Aluminium alone is already fairly ductile, lightweight, non-magnetic and highly malleable material. Meanwhile, Nickel is a metal which conductive to electricity and heat, able to resist corrosion at high temperature and malleable. Therefore, combining these two material would form an intermetallic compound that contains a highly ordered crystal structure. Intermetallic compounds possess of metallic bonds with ionic or covalent bond which results in crystal lattice [5]. Certain intermetallics able to sustain this order up until their melting point which makes them stable even at high temperature[i].

In manufacturing engineering, heat treatment had been widely used to improve the mechanical and physical properties of candidate materials [6]. Heat treatment processes includes tempering, hardening, normalizing and annealing [7]. In this research, annealing is used where it is a process of heat the material, hold at a certain temperature and let it cool to a room temperature. This process helps to eliminate the inner stress of material and improve its microstructure. Vickers hardness is used to detect the changes to the mechanical property that may occur. Besides that, X-ray diffraction (XRD) and Scanning Electron Microscope (SEM) observation is done in order to identify the modifications that take place to the material's crystallite structure. Changes in annealing temperature can cause modifications to the crystallize size [8]. Crystallite size information can be obtained from the XRD peaks and insert into Scherrer equation [9] to find crystallite size.

$$L = \frac{K\lambda}{\beta \cos\theta} \qquad (1)$$

where,
L = Scherrer average crystal size (nm)
K = crystalline shape constant (0.9)
λ = X-ray wavelength (0.1541 nm)
β = value of Full Width Half Maximum (FWHM)
θ = value of 2θ (must be in radian)

2. MATERIALS AND METHODOLOGY

A. *Sample Preparation*

The samples were cut into small pieces of 20 mm × 10 mm × 6 mm by using wire cut and placed into furnace for heat treatment process. When the annealing process is done, the samples were ground and polished. Grinding process was carried out starting from rough grinding with 240 grit abrasive paper followed by fine grinding with 1200 grit abrasive paper. Meanwhile, polishing process starts with 6μ polishing pad and 6μ DIAMAT Polycrystalline Diamond followed by a finer polishing pad and DIAMAT Polycrystalline Diamond which are 3μ and 1μ. Polishing process will be done until the samples obtain mirror like surface.

B. *Heat Treatment*

Heat treatment is one of the processes that is used to modify the structure of certain materials to obtain advantages from the materials property that is influenced by the temperature applied [10]. The annealing process can be carried out by using furnace with the maximum capability of 1250°C. Three different temperatures was chosen which are 400°C, 500°C and 600°C. Temperature 500°C is critical as it is the temperature when collision occurs in automotive accidents [11]. Therefore, 400°C and 600°C were only the upper and lower value of the temperature selected. The samples were heated at this temperature, maintaining the heat for 1 hour as soaking time and let it cool to the room temperature.

C. *X-Ray Diffraction*

The crystalline phases of each samples were determined by using X-ray diffractometery (XRD) with CuKα target. This method is to obtain the lattice parameter and crystal structure. Nickel Aluminide samples were studied to observe any changes between non-heat treated and heat treated samples. Grain size can be obtained from the XRD peaks and apply into Scherrer equation.

D. *Microstructure*

Samples were etching before it is viewed through Scanning Electron Microscope (SEM). Referring to ASTM E407 Standard Practice for Microetching Metals and Alloys [12], Nickel Aluminide samples were etched using a mixture of 5 ml Acetic acid, 10 ml of Nitric acid and 85 ml of distilled water. Electroetching method were used where 32 V and 0.03 A were applied for 60 s by using graphite rod to replace platinum wires. Sputter coating is then performed to the samples before observation under SEM.

E. *Hardness*

Vickers Hardness were used in this research as the samples are limited and Vickers indentation require only small space. The experiment were performed referring to ASTM E92 Standard Test Method for Vickers Hardness of Metallic Materials [13]. The load force chosen were 0.5 kgf, 1.0 kgf, 1.5 kgf and 2.0 kgf. Nine indentations were applied to each samples and the hardness results were obtaining from the average of the indentations.

3. RESULTS AND DISCUSSION

A. *X-Ray Diffraction*

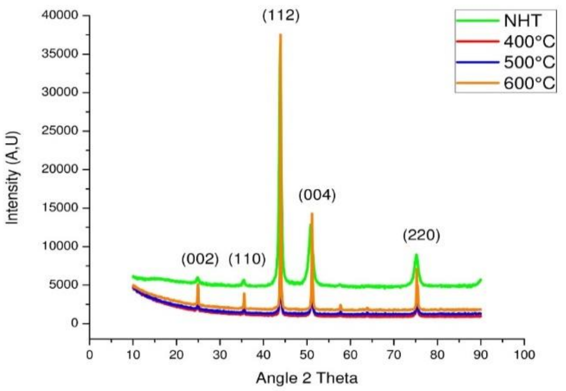

Figure 1 XRD graph for different temperature

The crystal structure of Nickel Aluminide samples with different temperature were place by XRD as shown in figure 1. The main peaks for non-heat treated samples at 2θ were at 24.38, 35.45, 43.87, 50.99 and 75.17 with the diffractions of (0 0 2), (1 1 0), (1 1 2), (0 0 4) and (2 2 0). The other three temperatures have peaks with similar 2θ values with the non-heat treated samples. As the temperature increase, the peaks become sharper.

Heat treatment able to increase the peaks sharpness as the heat energy tend to affects the recrystallization of the material[14, 15]. As the temperature rise, the intensity of all peaks increase steadily and the peaks become more sharper [15]. This is due to the crystal structure that tend to become more stiff with increasing annealing temperature [8].

Table 1 Grain size of Ni_3Al observed from XRD

Annealing Temperature (°C)	Grain Size (nm)
Non-heat treated	22.7250
400	45.4596
500	45.4602
600	45.4620

Results shown in table 1 shows the grain size of each sample that is calculated using Scherrer equation. The data needed to calculate grain size are the 2 theta and FWHM which can be extract from the XRD highest peak. In XRD, the three highest peak is the most intense peak but in this research only one highest peak is used for each sample to determine grain size. For non-heat treated samples the grain size is only 22.725 nm. As for 400°C, 500°C and 600°C, all three only has a slight difference of grain size results with the increment of 0.0006 nm and 0.0018 nm from one sample to another. Generally, the results show the grain size tend to increase as the annealing temperature increase.

Based on Nazarian-Samani et. al (2009) [15], Ni_3Al tend to increase in crystallite size as the temperature increase up to 1000°C. Kumar *et. al* (2011) [4] also stated that heat treatment cause the enlargement of grain size which also increase the hardness value.

Hosseini, S.N. [16] did heat treatment to intermetallic CoAl and found out that the grain size tend to increase as the annealing temperature increase. This is due to the activation energy and grain growth exponents that affects the diffusion of two atoms at high temperature. As the temperature rise, the activation energy for grain growth will increase. These energy are grain boundary energy where it acts as the driving force of grain boundaries movement for grain growth [17] . This statement mostly refers to materials like minerals and ceramics. Since Nickel Aluminides characteristics are close to ceramics, they have similar behaviour.

One of the factors that influence grain growth is temperature where this process involves the movement of high angle grain boundaries as well as kinetic energy which are highly affected due to temperature dependence of boundary movement. Driving force for growth of grain is normally small, major grain growth is often found at high temperature [18].

Table 2 Hardness value for Ni_3Al samples for different temperature

	Load Force (kgf)			
Annealing Conditions	0.5	1.0	1.5	2.0
Non-heat treated	108.32	89.09	70.38	56.66
400°C	143.52	115.12	98.11	86.42
500°C	181.14	137.81	111.51	98.00
600°C	213.38	184.82	151.91	134.93

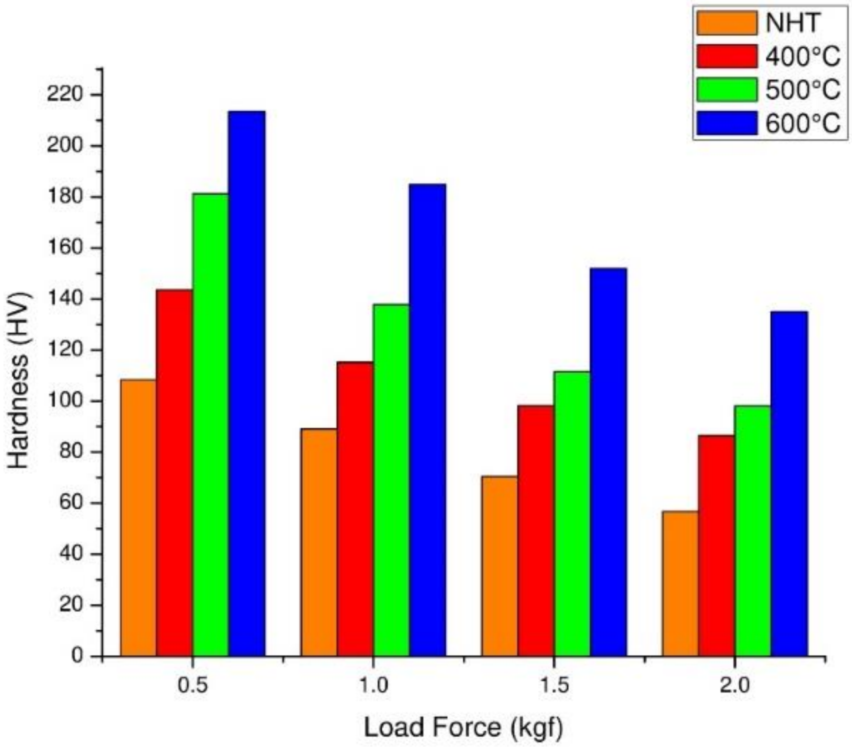

Figure *2* Bar chart of hardness based on annealing condition and load force

B. *Hardness of intermetallic Ni_3Al*

Based on table 2, the values of hardness decrease as the load force increase. Each hardness values are differing around 11 % to 34 % from one another for all four temperatures. Moreover, the value of hardness tends to rise gradually as the temperature of the samples increase for all load force. Non-heat treated Ni_3Al sample differs around average of 19.42 % for each load force from 0.5 kgf to 2.0 kgf. Vertically, the hardness varies about 20.13 % for each annealing conditions. Overall for every sample, the percentage difference from each load to another are below 35 %.

The bar chart trends show the hardness values increase as the temperature of the annealed sample increase. Besides that, the chart also shows as the load force increase the hardness value decrease gradually. For 0.5 kgf load, the maximum hardness value was obtaining by 600°C sample with 213.38 HV whereas the minimum value for hardness was from the non-heat treated sample with hardness value of 108.32 HV. The increment of the maximum and minimum hardness value for 0.5 kgf is 49.34%. As for 1.0 kgf, 1.5 kgf and 2.0 kgf the percentage of increment between the highest and the lowest hardness value are 51.80 %, 53.67 % and 58.00 % respectively. Kumar and Anand (2011) [4] indicated that as the annealing temperature increase, the hardness value also increase.

Meanwhile, the hardness value tends to decrease as the load force applied increase. For high annealed samples the penetration depth from the indenter increase due to the effect of decreasing thermal loading influence. Therefore, the specimens tend to deform.

Specifically, hardness results follow the behaviour of annealing from the indenter that was indented into the alloy samples. The increase values of hardness are due to the dendrites structure that decomposed slowly as the annealing temperature arise. Therefore, at higher annealing temperature samples tend to give higher hardness value as well as increasing the grain size [19].

Other opinions such as from Cinca et. al [5] stated that intermetallics such as FeAl has increasing results for surface hardness as the temperature increase. This is due to the formation on Al_2O_3 and Fe_2O_3 layer that influence the material's properties. It is reported that some intermetallics such as Ni_3Al, FeAl and Fe_3Al will increase in strength as the temperature increase [20]. Especially Ni_3Al, it is resistance to high temperatures and the formation of Al_2O_3 on the material's surface which is suitable for various types of environments and applications.

C. *Microstructure*

Figures 3 (a) to (d) above are the microstructure of nickel aluminide under 10,000X magnification. SEM observation is applied to study any changes to the microstructure of the sample. The samples were ground, polished, etched using the suitable chemicals as listed in ASTM and observed under 2500X, 3000X and 10000X magnification. There is no much of a change in the grain structure of each samples although with application of heat as high as 600°C. This shows that the even at high temperature of impact, the Ni_3Al microstructure does not affected much.

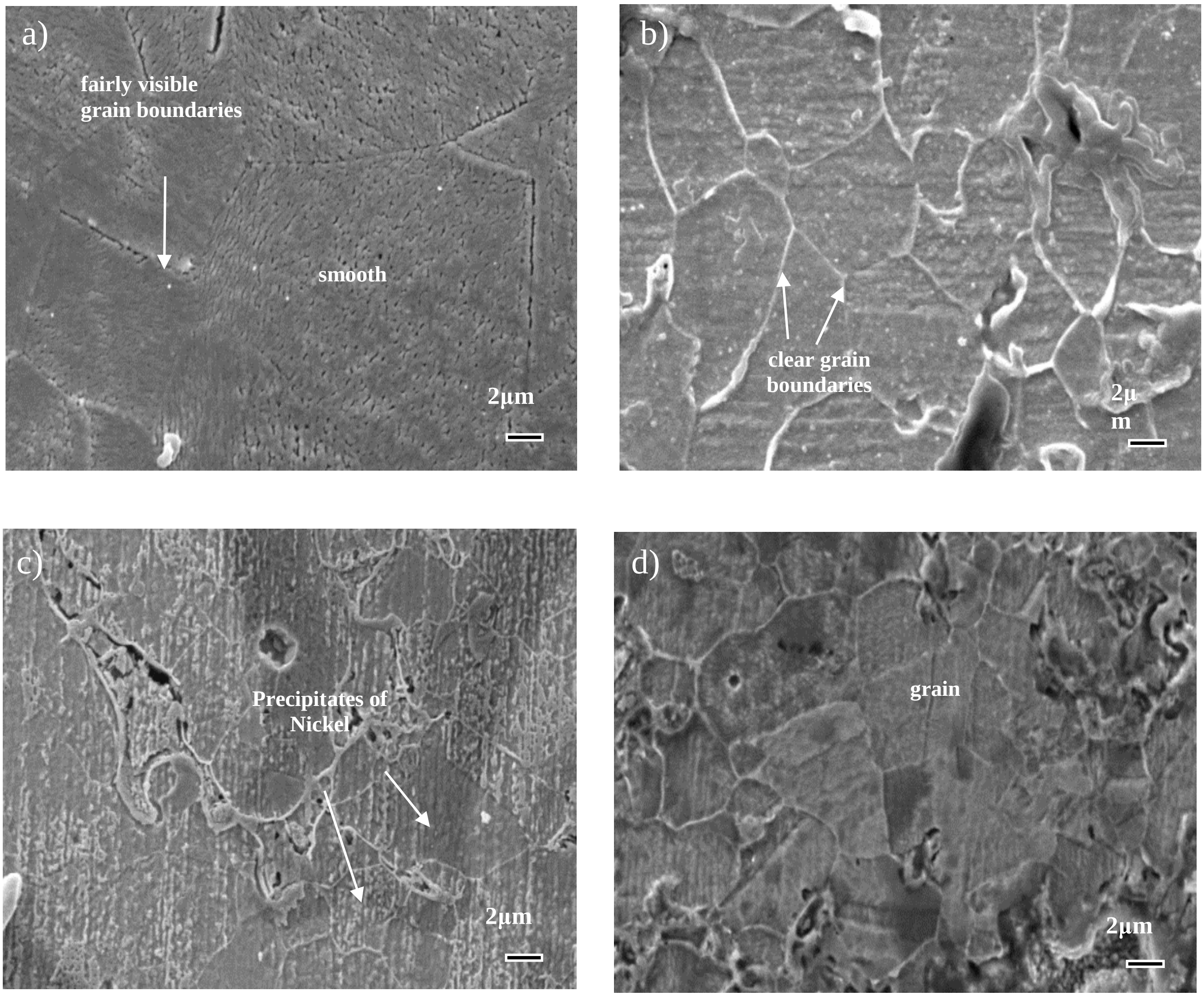

Figure 3 Ni_3Al for a) non heat treated sample, b) 400°C, c) 500°C, d) 600°C

IV. CONCLUSION

The Nickel Alminides samples were heated at 400, 500 and 600°C. When hardness test was performed, the results shows that the hardness value increase as the annealing temperature increase and decrease as the load force applied increase. During hardness at 0.5kgf load force, the hardness value starts from 108.32 HV for non-heat treated sample to 143.52 HV for 400°C, 181.14 HV for 500°C and 213.38 HV for 600°C. As the load force increase, the value of hardness for non-heat treated sample which begins from 108.32 HV at 0.5 kgf, 89.09 HV at 1.0 kgf, 70.38 HV at 1.5 kgf and 56.66 HV at 2.0 kgf. The same goes for three other annealing temperature. From the X-ray diffraction experiment, result the results confirms the metal's crystallography. Moreover, the grain size increase as the temperature of the samples increase due to the intermetallics property. The grains size of non-heat treated sample is 22.73 nm, 400°C is 45.4596 nm, 500°C is 45.4602 nm and 600°C is 45.4620 nm. These results shows that Ni_3Al can be the possible future material for wheel parts. For further research, impact test and fatigue test can be done to study the material's characteristics further.

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