A Novel Intermetallic Nickel Aluminide (Ni₃Al) as an Alternative Automotive Body Material

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Abstract - Investigation on Intermetallic Nickel Aluminides (Ni_3Al) was carried to determine the suitability of this material to replace the existing automotive body. The purpose is to produce vehicles which are lighter, more fuel efficient and cause less pollution. One key technical design strategy for improving vehicles efficiency is the light weighting. Attractive properties of Ni_3Al including low density (~ $6g/cm^3$) resulting lightweight, high oxidation and corrosion resistance, combined with their ability to retain strength and stiffness at elevated temperatures lead to its selection as a candidate alternative material. The prime focus will be on to obtain the mechanical properties such as hardness which was tested using Vickers Micro hardness Tester. XRD was used to determine the crystal structure of the designed alloy. Microstructural properties of these alloys were examined using optical microscopy and Scanning Electron Microscopy (SEM). SEM equipped with EDX used to do compositional analysis. Heat treatment (annealing) and Tafel extrapolation tests were carried out for thermal and corrosion properties of the intermetallic nickel aluminides respectively.

Index terms: *Alternating material; Intermetallic Aluminides; Mechanical; Automotive, Light-weight*

1. Introduction

Providing better performance with reasonable cost to the end user has been the main aspiration to most of the car manufacturer in today's era. The requirement for a better performance to the end user are detailed as follow; tomorrow's car will handle better, lighter, more fuel efficient and cause less pollution. One of the convenient design strategy used in enhancing the vehicle efficiency is via light weighting. As a known fact that light weighting is not only enhances fuel efficiency, but also reduces the emissions from automobile and improves the performance of driving. The major drawback in implementing light weighting is that the manufacturer has to pay more on the cost for it. It was proves that intermetallic aluminides has appeared to be as one of the material having high potential for a wide range of technological application in some of the essential areas. This enormous potential of intermetallics especially aluminides was contributed from their own mechanical properties such as high resistance to oxidation and corrosion [1]. It also has relatively low density which adds up to the ability to sustain strength and stiffness at elevated temperature [2]. In addition, there were voluminous research on intermetallics has been carried out to investigate the potential properties in aluminides especially the Ni-Al systems [3-6]. Due to the potential of having high resistance towards corrosion and good mechanical

properties, enable nickel to be as one of the materials used in structural and high temperature application for many years. Some of the examples of the nickel properties are listed as followed: hard, malleable, ductile, fairly ferromagnetic, fair conductor of heat and electricity which is not affected by air or atmospheric water, therefore constitute as a very good characteristic against corrosion [7]. Aluminum on the other hand is very light, nonmagnetic, highly malleable and fairly ductile [8]. Having discussed both characteristics of the aluminum and nickel, it shows that by merging these elements in a specific manner will give an opportunity to form intermetallic compounds which are having highly ordered crystal structure. By having such structure in the compound, it enhances the mechanical properties of the compound; increases the strength and hardness, be lighter, and having high melting point temperature [9]. In addition, this intermetallic compound possesses high resistance towards corrosion. In considerations of the above, the aim of the present work is to alternate automotive body material with intermetallic Nickel Aluminides (Ni_3Al) through mechanical and thermal characteristics.

2. Experimental procedure

Preparation of Ni-Al system alloys

In current research work, the Ni_3Al alloy material was prepared by casting elemental nickel (>99.92%) and aluminum (>99.79%) in proper proportions with a high frequency vacuum induction furnace. In order to obtain the accurate composition of the atomic percentage of Ni_3Al system alloy, slightly higher aluminum content has been added as aluminum melt much faster than nickel. Through some specific calculation, the amount of nickel and aluminum used is set to 1760.8g and 310.3g respectively.

The raw material of Ni and Al metal was cut into smaller cubes which then were weighted and the separated according to its composition before it undergone casting process. It worth noting, that the aim of cutting these metals into smaller cubes will actually enhance the mixing process during casting progression. Once the metal has been cut into smaller cubes, the metal will undergone cleaning process before actually can be send for casting where during the process the metals will be clean using a specific chemical. The melting process of the Ni-Al alloy will be carried out using the high frequency induction furnace

(INDUCTOTHERM). The furnace should work in high frequency condition as a known fact that nickel melting temperature is as high as 1455°C. Nickel cubes which have been cut earlier will be placed at the silicon carbide (SiC) crucible. The crucible was line with mixture of the silica sand and sodium silicate. CO_2 used to harden the lining. The crucible then place inside the furnace chamber and was closed. After nearly 30 minute, the cover was opened and the half molten nickel was stir to enhance proper melting. Next aluminum cubes was inserted slowly one by one to make the aluminum cubes diffuse into the molten nickel fully. Since aluminum melting temperature is at 660°C, the aluminum cubes will straight away melt when it is being inserted inside the crucible. The reaction between high melting temperatures elements with lower melting temperatures elements will cause fire spark. The fully liquefied Ni-Al material was maintained at high temperature for a couple of minutes. After convinced the molten of both elements have melt thoroughly, the crucible is being removed from the furnace chamber and the hot molten mixture of nickel and aluminum was poured inside the mold. The process should be done quickly, since nickel has poor fluidity and the risk of not full filling the cavity is large. The mold containing the solidified alloy was left for cooling for a day at room temperature. Finally the mold was break down and the produced alloyed were removed from the sand.

Most of the researchers distinguish annealing as one of the best heat treatment to be applied at metal alloys to release the residual stress on the material due to casting [4]. Three different temperature rates were applied during the heat treatment process and were tested on the design alloy in this research work where the temperature rates can be used as 2 hours at 300, 500 and 700°C. The reason above temperature selected, as it is believed that the temperature during a collision can rise up to 500°C, thus how the metal behavior at that temperature is analyzed. 300 and 700 °C was set as the higher and lower specification for above condition. In [3], 1 hour soaking time used to carry out the annealing on alloy material, and in the current research soaking time is increase to 2 hours to get further effect of the annealing process. During the heat treatment progression, carbolite furnace was being used followed by furnace cool.

X-ray analysis of Ni_3Al alloys were conducted using diffractometer with CuKα target. X-ray diffractometery was utilized to determine the crystal structures of Ni-Al system and the corresponding lattice parameters [10]. The heat treated and non-heat treated specimens of the designed alloys were also studied by X-ray diffraction to estimate the changes in phases with heat treatment.
The optical microscope is used to study the microstructure of the alloy. Electrochemical etching was done, referring to ASTM E407 practice. The solution for the etching was prepared by adding 10 ml of hydrofluoric acid (HF) into 10 ml of distil water.

Then 20 ml ethanol was added slowly into the solution. The prepared specimen had been etched for 10 seconds. Then the etched surface has been examined on optical microscope. For higher magnification microstructure image, scanning electron microscope (SEM) by Zeiss, model EVO 50 was used. The composition of the Ni_3Al alloy was confirmed using energy dispersive X-ray spectroscopy (EDX). The hardness properties of the Ni_3Al alloys were studied using Mitutoyo's HM-221 Mirohardness tester to estimate the changes in hardness under two conditions, as with and without heat treatment. ASTM E92 was referred to justify the important requirement needed during conducting the test. The specimen was fabricated in dimensions of 5.5 mm in thickness, 15 mm in width and 15 mm in length, referring to [11]. The testing conducted using a 50g load and a 15 s dwell time. At least, five measurements were carried out under the same conditions, and the mean values were calculated.

For the corrosion resistance testing purpose, Tafel Extrapolation was used [12]. With this technique, it is possible to measure extremely low corrosion rates. This technique uses data obtained from cathodic or anodic polarization measurements. The metal sample, Ni_3Al is termed as the working electrode and cathodic current is supplied to it by means of an auxiliary electrode composed of some inert material. In the experiment graphite rod was used as counter electrode, and Ag-AgCl was used as reference electrode. Electrolyte made from 96.5% water and 3.5% NaCl was used referred to ASTM G44. This composition resembles the percentage of salt contain in sea water. The samples will emersed in the electrolyte and using the scan rate of 0.5, Tafel graph was plotted using the Gamry DC corrosion Techniques software. Current is measured by means of an ammeter A and the potential of the working electrode is measured with respect to a reference electrode by a potentio-meter-electrometer circuit. For the sequent, current is increased by reducing the value of the variable resistance R. the potential and current at various settings are simultaneously measured. To measure the corrosion rate, the formula was obtained from ASTM G 102 as shown below:-

$$icorr = \frac{\text{Icorr}}{A} \tag{1}$$

Where: i_{corr} = corrosion density, μA/cm^2
I_{corr} = total anode current μA
A = exposed specimen area, cm^2

Corrosion Rate (CR):

$$CR = K1 \frac{\text{icorr}}{\rho} Ew \tag{2}$$

Where: CR = corrosion penetration rate (mm/yr)
K_1 = 3.27 x 10^{-3}, mm g/μA cm yr
ρ = density (g/cm^3)
Ew = equivalent weight

3. Results and discussion

3.1 Stoichiometry in the Ni$_3$Al alloy

The Ni$_3$Al alloy used in this project solely was prepared by casting commercially pure nickel (>99.95%) and aluminum (>99.75%) in the corresponding stoichiometric ratios at around 1600 °C. The melting temperature T$_m$ of the nickel is high (~1455°C), while that is of aluminum much lower (~660°C). The furnace temperature therefore must be set to beyond the T$_m$ of nickel. Since nickel will melt only at 1455°C, aluminum cannot be placed together since aluminum will melt at 660°C and start vaporized beyond that. Thus nickel will be melt first and then aluminum inserted in the liquid nickel. It is evident that exact Ni$_3$Al $_{stoichiometry}$ could not be produced due of losses cause by vaporization, the resulting composition will thus appear to be deficient in Al content. The starting compositions of Ni$_3$Al therefore deliberately selected to slightly richer in Al, to compensate for these anticipated losses of Al. The nickel content used to be 71.5 -73% where the aluminum content is to be 27-28.5%.

3.2 X-Ray Diffraction (XRD) Analysis

The X-ray diffraction pattern confirms the presence of the Ni$_3$Al phase which is shown in Figure 1 below. The peaks were found at 36.3°, 43.9°, 51.3° and 75.7°. When further anneal the sample, super-lattice reflection of the γ'- Ni$_3$Al phase can be seen. An additional peak was found when the sample further anneals after 500°C, where it was identified to be the (100) at 25.9°. This further indicates the establishment of the ordered structure. The crystal structure of Ni$_3$Al is ordered cubic and Strukturbericht designation for this structure is L1$_{2.}$ [5]. The broad reflections observed in Figure 1 reveal that the bulk material was crystalline essentially in the form of a very fine-grained structure.

3.3 Microstructure of Ni$_3$Al alloy

The microstructure of Ni$_3$Al alloy can be clearly seen in Figure 2 below observed using the optical microscope. The microstructure of Ni$_3$Al alloy usually consists of a Ni solid solution matrix and typically cuboidal Ni$_3$Al precipitates [13]. The grain structures are visible resulting from the proper etching. When alloy solidifies during casting, the atoms which are randomly distributed in the liquid state, arrange themselves in a crystalline array. This ordering usually begins at many points in the liquid, and as these block of crystals or grains meet, there is mismatch at their boundary. When metal has solidified and cooled, there will be numerous regions of mismatch between each grain. These regions are called grain boundaries. The grains are mostly equiaxed (having equal dimensions in all direction) and nanocrystalline (grain size below 50 nm) [14] and uniformly distributed in the microstructure. The number

and size of grains development depend mainly on the rate which nucleation (initial stage of crystal formation) takes place. In this alloy, the crystal nucleation rate is considerably high, thus number in a unit volume are large consequently result grain with smaller size [15]. However, due to the complexity of the microstructure, it is difficult to precisely determine the average grain size.

For higher magnification, the microstructure observed using SEM. At magnification of 1000 X, along with grain boundaries, a large network in ordered L1$_2$ Ni$_3$Al phase visible and it was famously known as dendrites arm. For the non-heat treated samples of the Ni$_3$Al alloy shows large present amount of dendrites as shown in Figure 3 (a). These dendrites formed due to the significant difference in melting temperature of nickel and aluminum. Nickel will cool and solidifies faster than aluminum thus develops a multi-branching tree like shape. The aluminum which cools slower compare to nickel will fill the space between these branches. After the alloy annealed at 300, 500 and 700°C, the amount and size of dendrites in the alloy decreases slightly as shown in Figure 3 (b), (c) and (d) respectively . This shows the heat treatment for Ni$_3$Al do effect the micro-structural changes. Heat subjected to the alloy slowly brake down the dendrite arm structure of larger nickel containing phase. It can be observed where the large network start to decay at 300°C and the network become thinner when the temperature increased to 700°C. A higher annealing temperature is required to reduce the amount of dendrite fully. This dendrite arms sometimes possible to raise brittle behavior to the material [16].

During heat treatment, the actual cooling rate of specimens is not high enough the restrain the precipitation of the Ni$_3$Al phase. Precipitation of a phase occurs from supersaturated solid solutions. This is because of the specimen placed inside the furnace with vacuum condition during cooling. Therefore, precipitation is observed within the grains and along the grain boundaries. As noted, grain boundaries are high energy areas, so precipitation frequently begins at the grain boundaries. From the SEM and optical images, it can be observable that the shape of the Ni$_3$Al precipitates within the grains is bamboo-like. Each part of the bamboo is a small Ni$_3$Al needle with little different shape with from other needle. The precipitation of Ni$_3$Al phase leads to high misfit strain in the alloy matrix thus result the needle shape precipitates [17]. The Ni content near the pinpoint of the needle shape precipitates is usually higher than any other region.

3.4 Energy Dispersive X-ray (EDX) Analysis

After confirm the present of Ni$_3$Al phase inside the sample, the alloy were further subjected to SEM/EDX analysis to confirm the composition of the designed alloy. Figure 4 shows the EDX spectra collected for

sample with and without heat treatment. What can be observed is that while the relative of Ni and Al peaks was observed and confirmed their composition and stoichiometry for as prepared and annealed at 300, 500 and 700°C are remained more or less constant. The EDX analysis shows the ratio of Ni to Al is 3:1 respectively. There were some other elements also evident in the alloys which are carbon and oxygen. All elements present were tabulated in Table 1.

The oxygen atomic weight content decreases from 6.2 to 4.21% when the sample further heat treated from as-cast condition up to 700°C. Carbon content also decreases with increasing annealing temperature, where the atomic weight initially around 35.53 decreases to 21.35%.

Table 1: Chemical composition (at. %) of Ni_3Al alloy with different heat treatment conditions

Element	Ni	Al	C	O
nht	43.46	14.81	35.53	6.20
300°C	47.05	15.57	32.43	4.95
500°C	53.93	17.52	23.86	4.69
700°C	56.12	18.32	21.35	4.21

3.5   Vickers Micro Hardness result of Ni_3Al

Hardness value was measured for Ni_3Al alloy using Vickers micro hardness tester at 5 different places. The test was also used to estimate the changes in hardness with and without heat treatment. The results are shown in Table 2. Only result of average hardness value with indention load of 0.01 kgf was tabulated for comparison purpose. The test also carried out with different indention load and the graph is presented in Figure 5. For Ni_3Al alloy, it can be clearly seen that the hardness value which is in initially around 450.6 HV (without heat treatment) increase up to nearly 569.9 HV under the same load (0.01 kgf) after receiving heat treatment at 700°C.

Table 2: Hardness test of Ni_3Al under 0.01 kgf indention load

Conditions	Vickers Hardness (HV)
Non Heat Treated (NHT)	450.6
Annealed 300°C	471.0
Annealed 500°C	507.5
Annealed 700°C	569.4

As far as individual hardness curve of the alloy specimens are concerned, it can be seen that the hardness are prominently higher especially when the indention load decreases. Upon raising the indention load beyond 0.3 kgf, it is interesting to see all the hardness curves of alloy specimens coincide with one another as shown in Figure 3.5. As for the decrease in

hardness with increasing load, it can be interpreted as the consequence of a reduced influence of the thermal load. Since for high annealed specimens, the penetration depth increases and thus it gain more significance in the effective volume subject to deformation. In other words, the resulting hardness tends to reflect the annealing behavior provided that the indenter advances deeply into the alloy regime.

The percentage in increases of hardness value was 26.36%. Ni_3Al result considerably high percentages increases in hardness properties since it associate with dendrites structure which decayed very slightly with the rise of annealing temperature. Presents of precipitates at higher annealing temperature impart more hardness on the alloy surface. Heat treatment also increases the size of the grain and this condition also rise the hardness value of the alloy [3]. The reason for the increment is where larger grain size result material with more compacted density. Nevertheless, Heat treatment at temperature higher than 800°C can reduces the level of residual stresses of the alloy by breaking the dendritic structure and causing more homogeneous distribution phases. A reduced level of residual stresses after heat treatment will also result less hardness to the alloy material since the alloy was attributed to softening [18].

3.6   Corrosion studies by Tafel Extrapolation analysis

The chemical behavior in this research solely depends on the corrosion rate of the Ni_3Al alloy material. The corrosion rates determine using Tafel method whereas the value of i_{corr} was obtained from the Tafel graph as shown in the Figure 6. 0.5 M NaCl was use as corrosive medium due to its large popularity usage in corrosion studies [19]. This corresponds to the NaCl concentration in the sea water. Faraday's Law was used along with the penetration rate method to calculate the corrosion rate. The penetration rate or corrosion rate (CR) was measure using Equation 1 and 2. The corrosion potentials and current densities were determined by the intersection of extrapolated cathodic and anodic Tafel curve portions. The i_{corr} values first obtain through the Tafel scan by interpolating the graph. Then, the gain i_{corr} value will directly apply in the equation 2.

The corrosion properties were determined from the corrosion rate equation where the value was tabulated in Table 3. From the table itself it can be clearly seen that Ni_3Al alloy annealed at 700°C had the lowest corrosive rate value which is 7.24×10^{-4} mm per year. The increment was followed by sample annealed at 500°C, 300°C and nht with value of 7.24×10^{-3}, 8.46×10^{-3} and 3.62×10^{-2} respectively. The alloys which were heat treated was classified as outstanding against corrosion and the one without heat treatment was classified as excellent according to typical ferrous and nickel –based alloys corrosion resistance table [20]. The table illustrate material having corrosion rate lower than

0.02 and corrosion rate between 0.02-0.1 mm/year classified as outstanding and excellent respectively. This shows that, basically this alloy having good corrosion resistance properties. When it further treated, the properties further getting better.

Table 3: Corrosion rate of the Ni_3Al material

Alloy Compound	NHT (mm/yr)	300°C (mm/yr)	500°C mm/yr)	700°C (mm/yr)
Ni_3Al	3.62×10^{-2}	8.46×10^{-3}	7.24×10^{-3}	7.12×10^{-4}

The most stable configuration of an alloy is particularly its crystal lattice, grain boundaries are high-energy areas and more active chemically. Thus, grain boundaries are usually attacked slightly more rapidly than grain faces when expose to a corrosive condition [20]. As described before, annealing makes the grain size grow bigger and eliminate the number of grain boundaries. Thus it does confirm the reason why Ni_3Al annealed at 700°C having the lowest corrosion rate value.

Alloy cannot run away from formation of dendrite structures which form during casting. The as-cast material seems to expose to localized corrosion in the region where aluminum and nickel dendrites form in higher rate. When the sample further annealed, the corrosion rate improved since this dendrites start to decay and thus localized corrosion can basically reduced or prevented. Through annealing the residual stress inside the material was released. This reduces the active region inside the sample where corrosion can also easily attack this region. Other than that, the impurities content in the compound like oxygen and carbon do affect the corrosion rate. The higher content of carbon and oxygen inside the alloy, result a lower value of corrosion rate.

3.7 Comparative of current material properties with Ni_3Al

The research was done mainly to investigate whether Ni_3Al can alternate current or existing automotive body material. From the entire test conducted, basic properties needed was indentified which further will be a milestone for a design of better automotive body material in future. This section compares Ni_3Al non-heat treated and annealed specimens with current automotive body material in terms of weight, hardness, corrosion property, and cost. Basically the main structure or body in white for the existing material was structured using high strength steel (HSS) and aluminum (luxury vehicles). Thus comparison will be done between Ni_3Al, HSS and aluminum.

Comparison on weight was done by referring the density of the material. Steel having density of 7.85 g/cm^3, and aluminum 2.70 g/cm^3 Using Archimedes principle, the density of Ni_3Al was indentify to be 6.67 g/cm^3. This show Ni_3Al has lower weight than steel but still higher weight value compare to aluminum. Ni_3Al is still applicable in alternating steel since it imparting lighter weight on the vehicles. The hardness of HSS is around 400 HV [21] and aluminum is around 107 HV [22]. It was cut and clear here the harness properties of Ni_3Al are far better than aluminum and slightly better than steel even before it was heat treated which was 450.6 HV. This makes Ni_3Al as a better wear and scratch resistance material and useful for dent and chipping resistance by stone for an automotive body.

Steel is well know of is rusty problem, it easily get corroded when exposed to corrosive environment. Aluminum is good against corrosion. Ni_3Al shows very good corrosion properties which will be an advantageous in automotive industry as badly rusted vehicles loses much of its value. Comparison in terms of cost is cannot be done exactly due to the fluctuating value of metals price in market. As known factor steel is much cheaper than aluminum and nickel. So by combining nickel and aluminum it is anticipated that the cost can be reduce and draw level to steel cost. Increasing in demand for nickel and aluminum after proven suitable in structural application will increase production to manufacture this metal and thus will reduce the cost much lower.

4. Conclusion

The study on the mechanical and chemical properties of Nickel Aluminides (Ni_3Al), had been examined in an over range of annealing temperatures. The X-ray diffraction analysis confirms the crystallographic nature of the alloy and EDX confirms the stoichiometry of the alloy at different annealing conditions. The increase in hardness value with respect to annealing temperature also shows that this Ni_3Al alloy is suitable for high strength and good candidate material for alternating automotive body. All the basic characteristic shows that Ni_3Al have potential to replace neither steel nor aluminum as an automotive body material in future. Further testing such as tensile, fatigue, creep and more sophisticated testing using modeling, simulation and crash test should be carry out to really relish this material in the industry.

Acknowledgement

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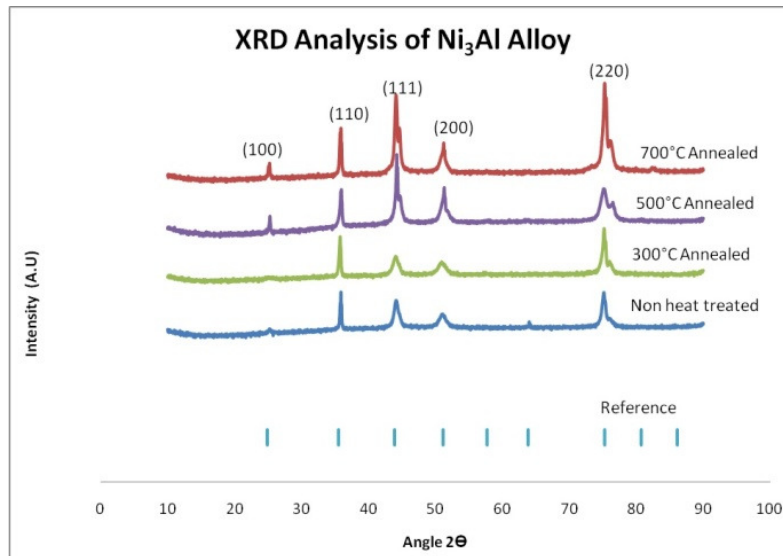
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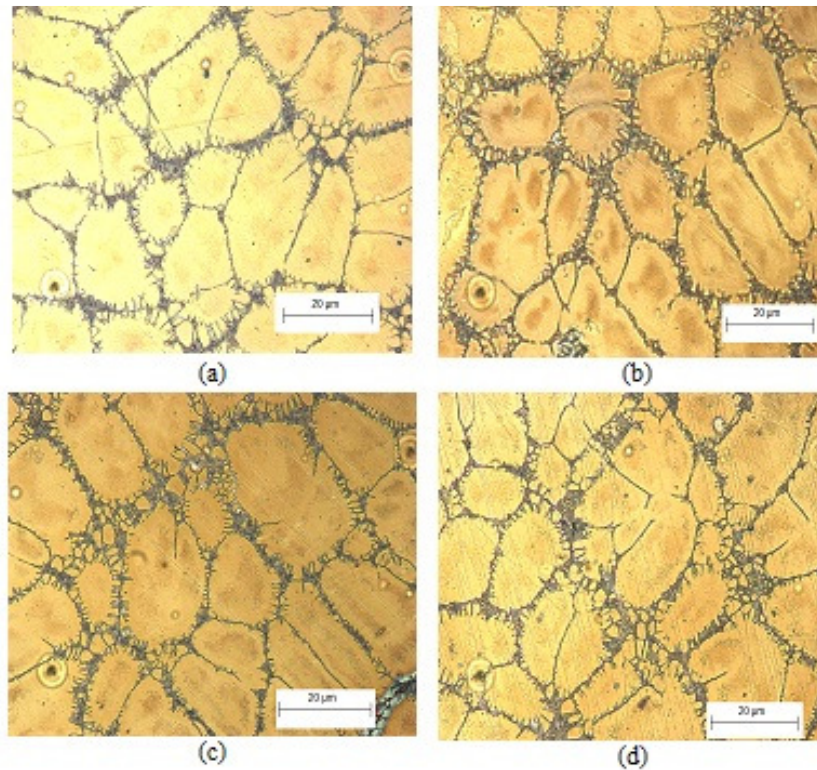**Figure 1:** XRD pattern of Ni$_3$Al alloy for different heat treatment

Figure 2: 20 X magnification, scale 20 µm/cm shows microstructure behavior of Ni$_3$Al alloy annealed at different temperature (a) non heat treated, annealed at (b) 300°C, (c) 500°C, (d) 700°C.
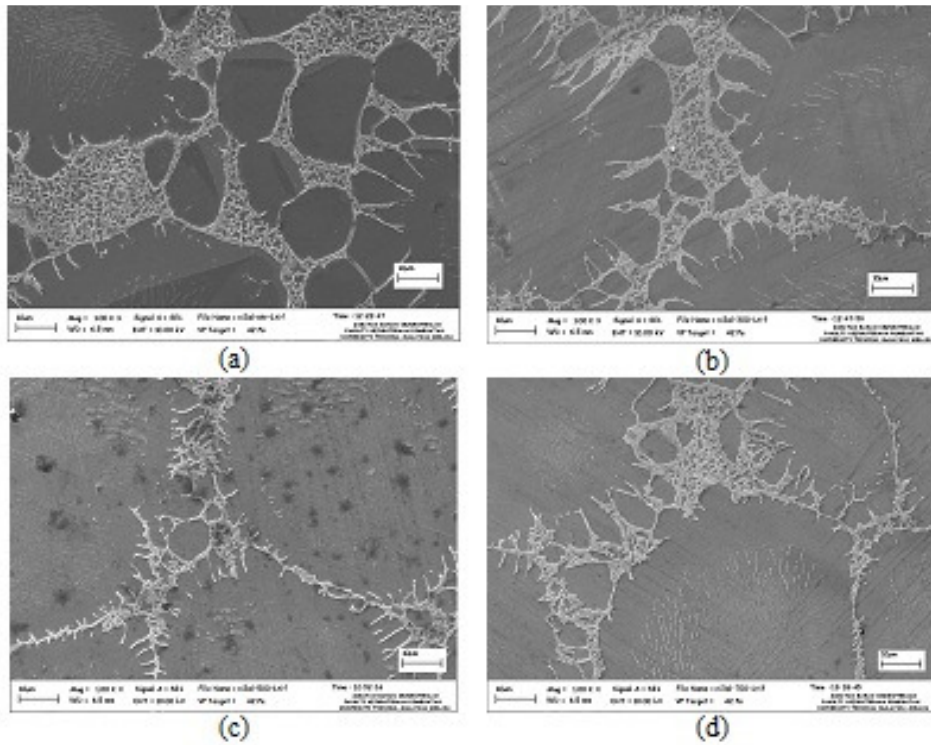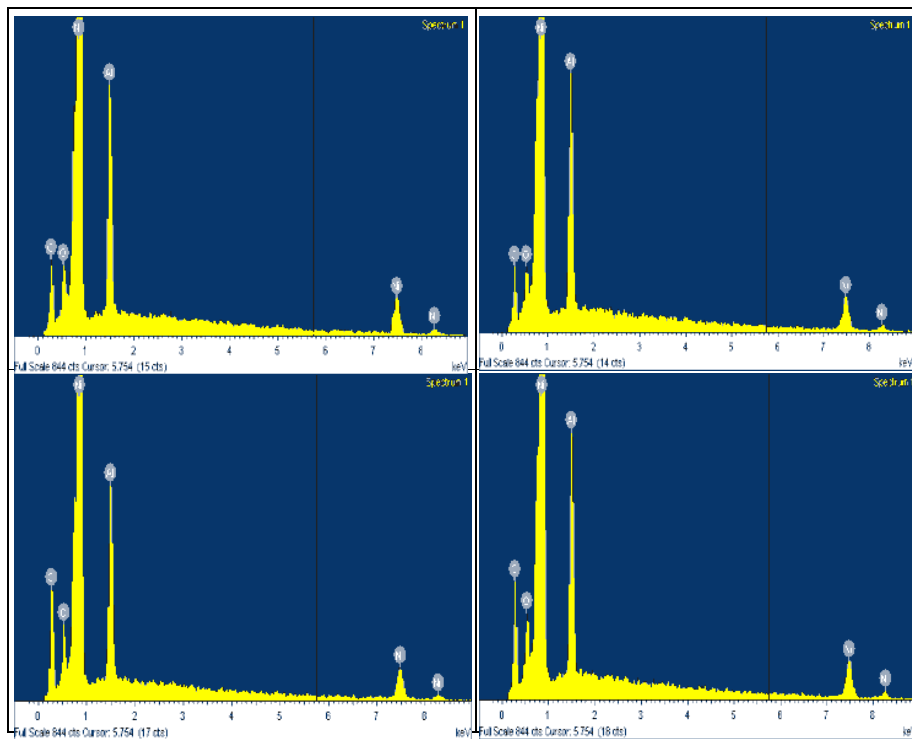
Figure 3: 1000X magnification, scale 10 µm/cm shows microstructure behavior of Ni$_3$Al alloy annealed at different temperature (a) non heat treated, annealed at (b) 300°C, (c) 500°C, (d) 700°C.

Figure 4: SEM/EDX spectra collected for Ni$_3$Al alloy (a) non heat treated, (b) 300°C annealed, (c) 500°C annealed, (d) 700°C annealed.
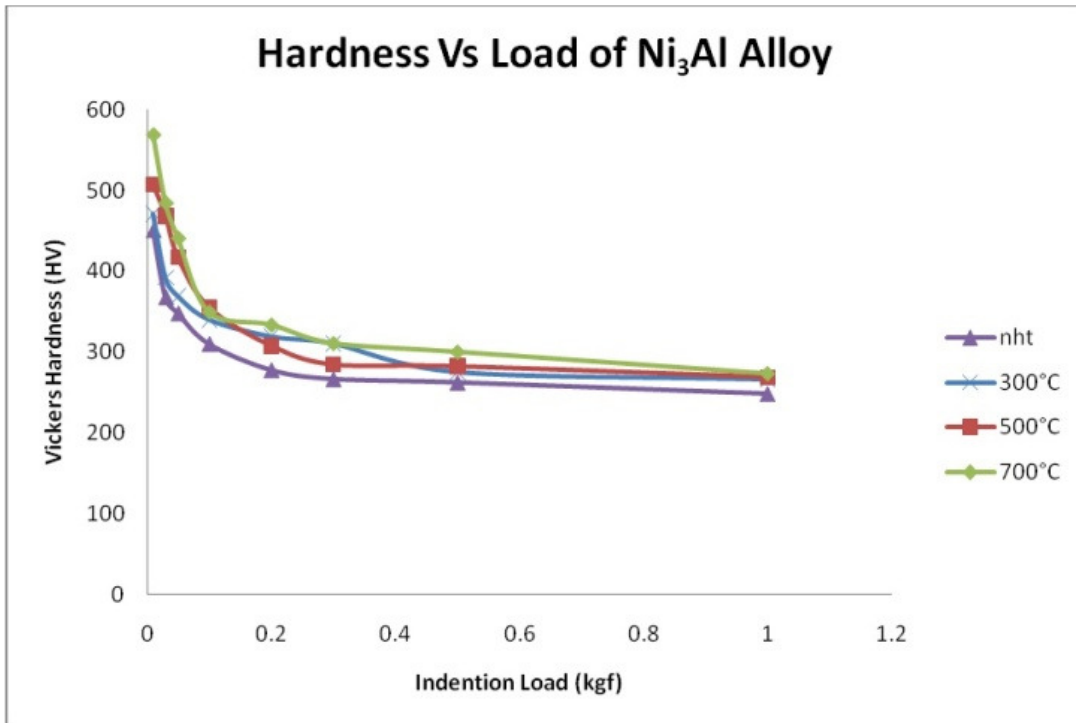
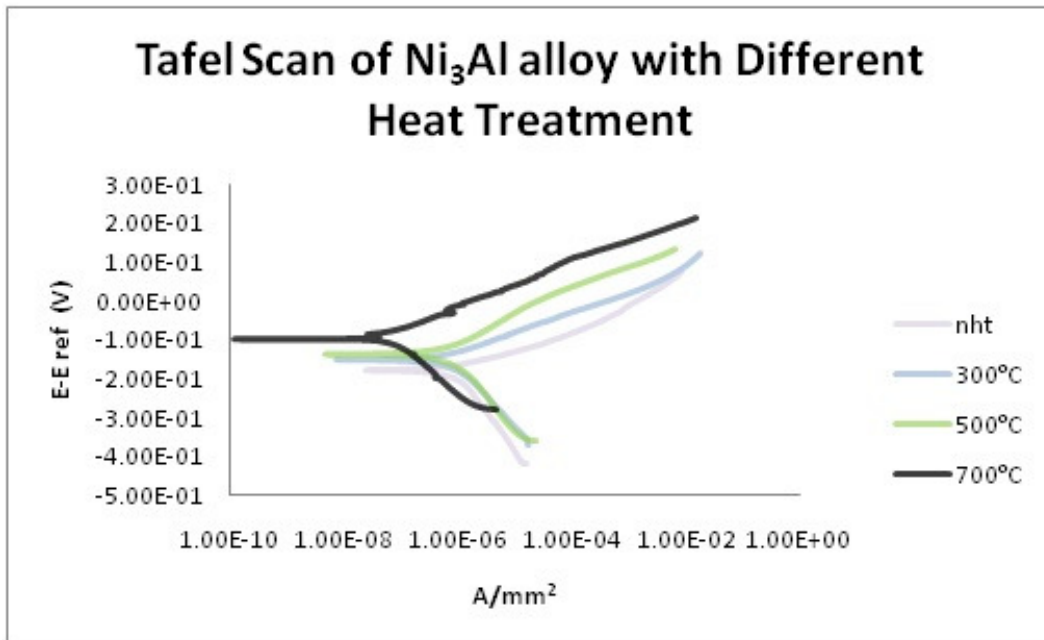**Figure 5:** Graph of hardness vs. load of Ni₃Al

Figure 6: Tafel Extrapolation graph of Ni₃Al with different heat treatment