

# Grain refinement and wear studies of nickel aluminide reinforced Al Composites

J. Abuthakir, G. Venkatesh

In the present study 1.5 wt% Ni particles were added to AA6061 alloy melt during stir casting and nickel aluminide reinforced Al Metal Matrix Composites (MMCs) were synthesized in the as cast, solutionized and T6 conditions. Base alloy was also synthesized in similar conditions. Base alloy and composite samples were characterized using X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Energy dispersive spectroscopy (EDAX) and Transmission Electron Microscopy (TEM).  $Al_3Ni_2$  and AlNi intermetallic phases were observed during characterization. T6 composite exhibited a grain refinement of  $38\mu m$ . Hardness of the T6 composite showed a 50% increase compared to base alloy. T6 composite showed 90% reduction in wear loss compared as cast base alloy.

**KEYWORDS:** AA6061; 1.5 WT% NI PARTICLES; INTERMETALLIC PHASES; GRAIN REFINEMENT; HARDNESS; WEAR LOSS.

## INTRODUCTION

Recent studies show that in situ intermetallic-reinforced Al MMCs—where reinforcement phases are generated internally through chemical reactions within the matrix—exhibit superior wear performance compared to MMCs reinforced with externally added (ex situ) ceramic particles, which often suffer from weaker interfacial bonding and non-uniform distribution. Intermetallics reinforced Al MMCs are being prepared through various routes such as Friction Stir Processing (FSP), Powder Metallurgy as well as various casting routes [1, 2, 3, 4]. The Synthesis of intermetallic reinforced Al MMCs through solid state reactions between metallic particle additives and Al alloy have been attracting wide research attention [5]. Literature review reveals that AlNi,  $Al_3Ni$ ,  $Al_3Ni_2$ ,  $Ni_3Al$ , TiAl and  $TiAl_3$  are the aluminide intermetallic compounds widely reinforced in Al MMCs [1]. Amongst these reinforcements, nickel aluminide (AlNi,  $Al_3Ni$ ,  $Al_3Ni_2$  and  $Ni_3Al$ ) intermetallic reinforcements have been widely preferred because it imparts higher hardness to the matrix compared to other intermetallic reinforcements [5, 6, 7]. Intermetallic reinforced Al composites have been widely investigated for wear-based applications using various Al alloys added with Ni/Ti particles [8, 9, 10]. However, very limited attempts have been reported in the synthesis of nickel aluminide reinforced AA6061 Al MMCs for wear-based applications. Literature studies also revealed that

**J. Abuthakir**

Assistant Professor, Hindusthan Institute of Technology,  
Coimbatore, India - 641032

**G. Venkatesh**

Assistant Professor, PSG College of Technology,  
Coimbatore, India - 641004

fewer investigations were carried out on wear behavior of nickel aluminide reinforced Al MMCs containing Ni particles in the range of 0-5 wt% wherein superior wear resistance was reported [5].

Hence the aim of the present investigation is to synthesis Al composites reinforced with nickel aluminides intermetallics in the as-cast, solutionized as well as T6 conditions using AA6061 alloy containing 1.5 wt% Ni particles prepared via stir casting. The investigation also aims to compare grain refinement, hardness and wear behavior of composite with the base alloy in the as-cast, solutionized and T6 conditions.

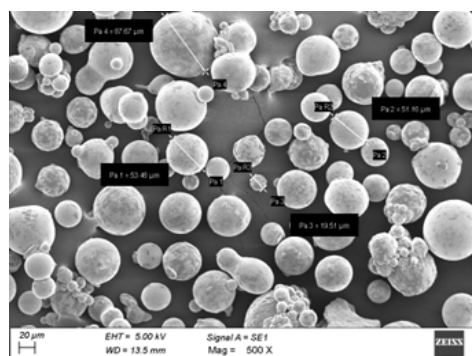
### MATERIALS AND EXPERIMENTATION

AA6061 alloy was used as the base material and its chemical composition is shown in table 1. Ni particles of average size 50 $\mu$  (figure 1) were added to the AA6061 alloy melt in a furnace maintained at 750°C. Stirring was carried out at 200 rpm for 10 minutes. As cast composite and base alloy was solutionized at 550°C for 1 hour and further subjected to T6 treatment (550°C for 1 hour and peak aged at

165°C). XRD (Shimadzu), SEM (ZEISS), EDAX (TEAM) and TEM (JEOL) analysis were carried out to study nickel aluminides formation and distribution in the AA6061 matrix. XRD analysis was carried out in the scanning range of 10° to 90° at a scan step time of 1 second in steps of 0.0530°. Optical metallographic examination was carried out for base alloy and composite in all the conditions as per ASTM E3-01 standard and the etchant used was 10% HF solution. Vickers hardness test (Mitutoyo) was carried out at a 100g load for composite samples and base alloy in the as-cast, solutionized and T6 conditions as per ASTM E92 standard. Wear studies on the base alloy and composites were carried out using a DUCOM pin-on-disc wear tester as per ASTM G99 standard. Counter pin material used in the wear test was E11 hardened steel disc. Wear test was performed for base alloy and composite at sliding distance of 500m, load of 20N and sliding speed of 2m/s in the as-cast, solutionized and T6 conditions. Worn out surface morphology of base alloy and composite samples were analysed using SEM (ZEISS/JEOL).

**Tab.1** - Chemical composition (weight %) of base alloy in the as-cast condition.

Elements	Ni	Si	Mg	Fe	Cu	Mn	Al
Weight percentage	-	0.77	0.85	0.415	0.279	0.082	Remainder

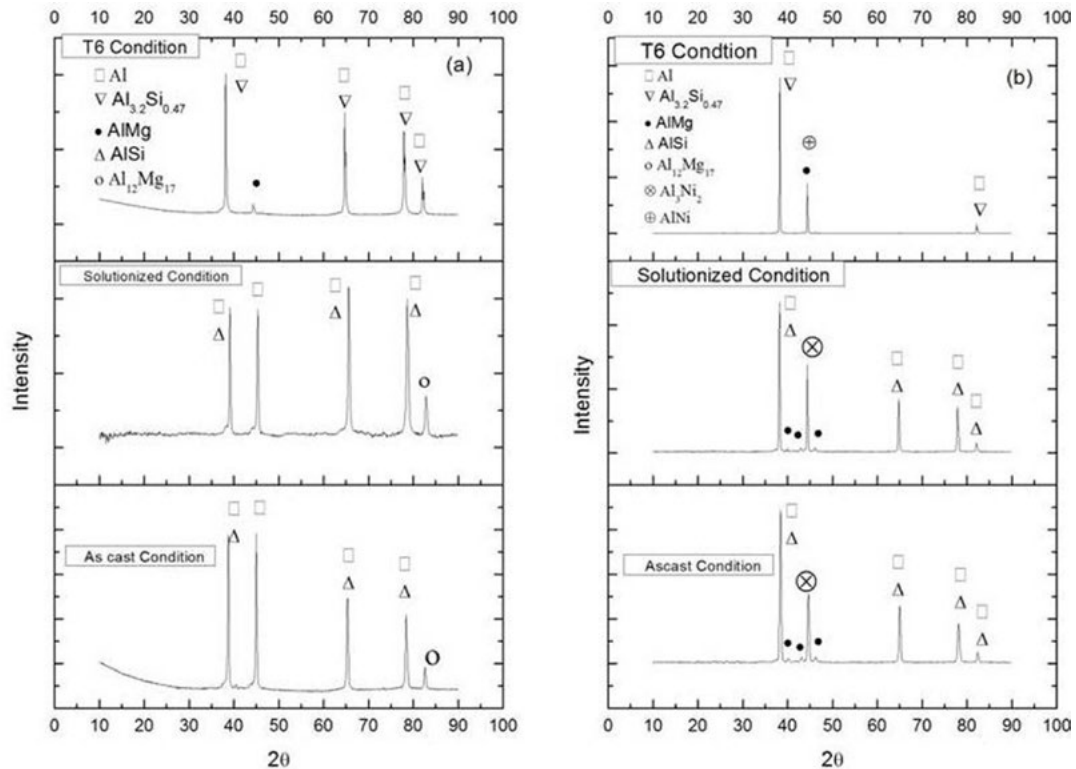


**Fig.1** - Ni particles used in the investigation.

### X-RAY DIFFRACTION STUDIES OF BASE ALLOY AND COMPOSITE

XRD results of the base alloy and composite samples are shown in figure 2. Base alloy exhibited peaks of Al,

Al<sub>12</sub>Mg<sub>17</sub> and Al-Si phases in the as-cast condition. Similar phases were shown by base alloy in the solutionized condition. Al, Al<sub>3,2</sub>Si<sub>0,47</sub> and AlMg phases were observed in peak aged (T6) base alloy.



**Fig.2** - XRD results of (a) base alloy and (b) AA6061-1.5 wt% Ni composite in the as-cast, solutionized and peak aged (T6) conditions.

AA6061-1.5 wt% Ni composite showed the presence of Al and AlMg phases in the as-cast, solutionized and T6 conditions. The composite however exhibited peaks of AlSi and  $\text{Al}_3\text{Ni}_2$  phases in the as-cast and as solutionized condition whereas it showed  $\text{Al}_{3.2}\text{Si}_{0.47}$  and AlNi phases in the T6 condition. Al exhibits rapid diffusion into Ni at temperatures above 600°C leading to the formation of intermetallic layers such as NiAl and  $\text{NiAl}_3$  at the Al-Ni interface. These reactions are thermodynamically favourable and are reported to progress quickly even at relatively short holding times.

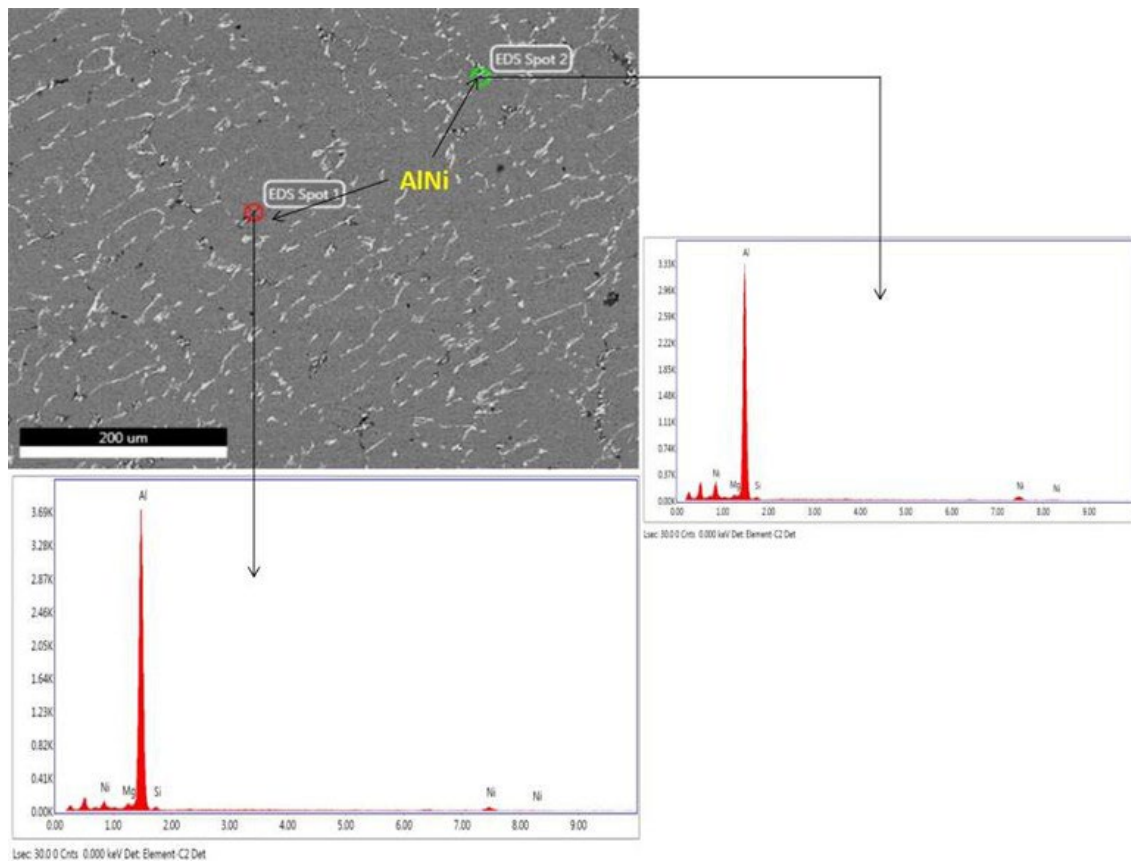
As the reaction layer grows, it progressively consumes the Ni particle, and the resulting intermetallic compound becomes brittle, breaking into fine, fragmented phases during stirring and solidification. These fine intermetallic fragments are subsequently distributed throughout the matrix leaving no discrete Ni particles in the final composite microstructure. Similar Al peaks were reported by Satishkumar et al. [11] in the investigation of ZrC re-

inforced AA6061 Al composite. Naeem et al. [5] observed nickel aluminide intermetallic phase formation in Ni particles added Al-Zn-Mg-Cu alloy at  $2\theta$  of 45° with no mapping of Al peaks due to low intensity of peak.

Formation of intermetallic phases during solutionized and T6 conditions, which consumed part of the Al matrix, reducing the relative intensity of Al peaks. Al peak near 45° overlaps with strong intermetallic peaks ( $\text{Al}_3\text{Ni}_2$  and AlNi), causing apparent disappearance. Comparison of the obtained XRD results with ICDD database allowed distinguishing between the  $\text{Al}_3\text{Ni}_2$  and AlNi peaks.

### SEM-EDAX INVESTIGATION OF BASE ALLOY AND COMPOSITE

SEM-EDAX image of AA6061-1.5 Ni wt% composite is shown in figure 3. Presence of Al, Si, Mg and Ni elements were predominantly observed, and Ni element was found to be distributed in the matrix at the grain boundary networks. This confirmed that the AlNi intermetallic phase formed in the T6 composite was distributed in the matrix.



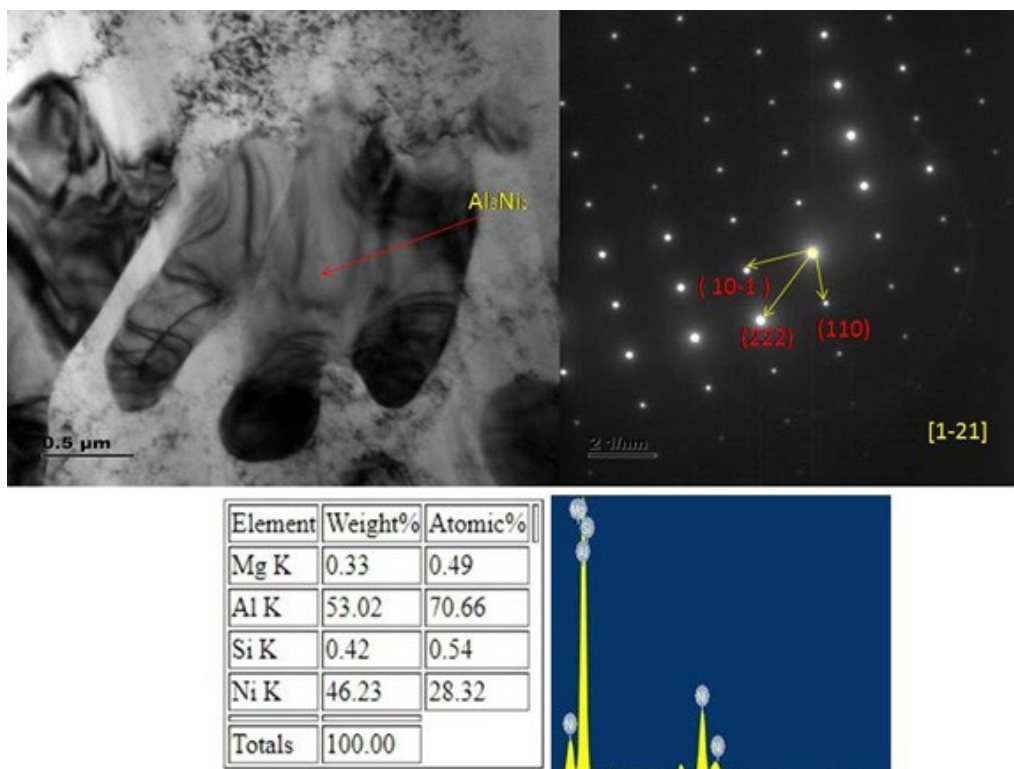
**Fig.3** - EDAX (spot) analysis of AA6061 + 1.5 Wt% Ni composite in the peak aged (T6) condition.

As-cast and solutionized composites also showed the presence of similar elements and had distribution of Ni element in the matrix at the eutectic networks which confirmed the distribution of  $Al_3Ni_2$  intermetallic phase in the matrix. Base alloy exhibited the presence of Al, Si and Mg elements in the as-cast, solutionized and T6 condition.

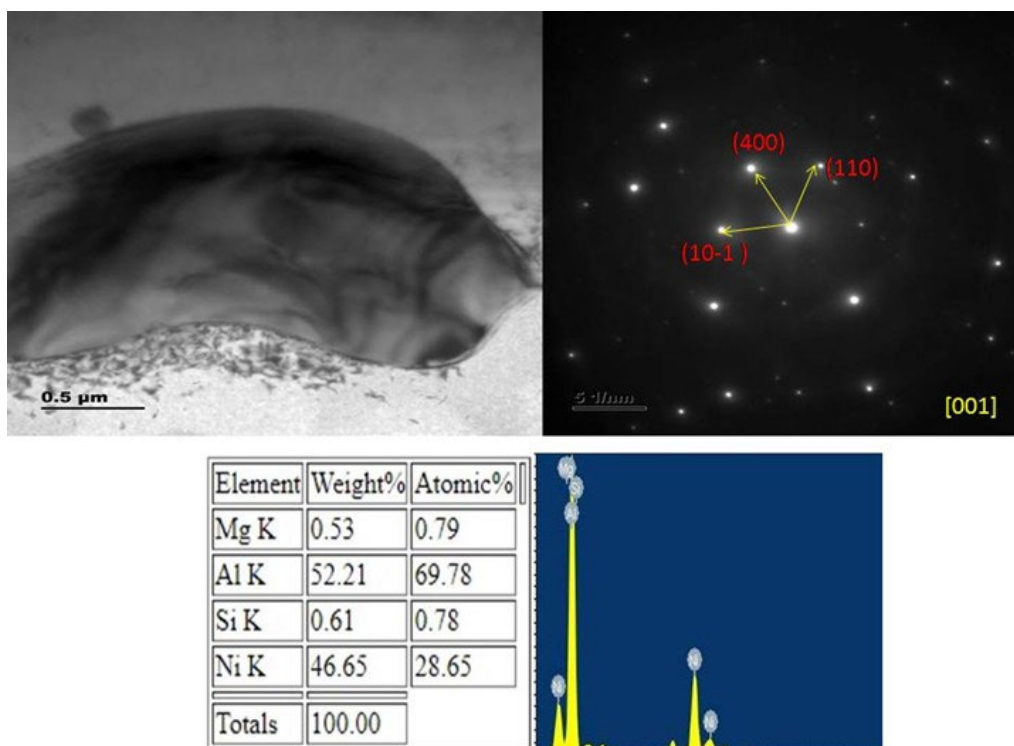
#### TEM INVESTIGATION OF COMPOSITE SAMPLES

Bright field image of AA6061-1.5 wt% composite (figure 4) showed the formation of  $Al_3Ni_2$  intermetallic phase in the as-cast condition. SAD pattern of the as-cast composite was revealed (110) and (10-1) planes of  $Al_3Ni_2$  intermetallic phase belonging to cubic system. AlMg plane (222) was also observed in the SAD pattern. Zone axis of the SAD pattern was found to be [1-21]. Morphology of the  $Al_3Ni_2$  intermetallic phase was lenticular having a size of  $2\mu m$ . Solutionized composite showed (10-1) and (110) planes of  $Al_3Ni_2$  intermetallic phase. Al-Mg phase was also observed

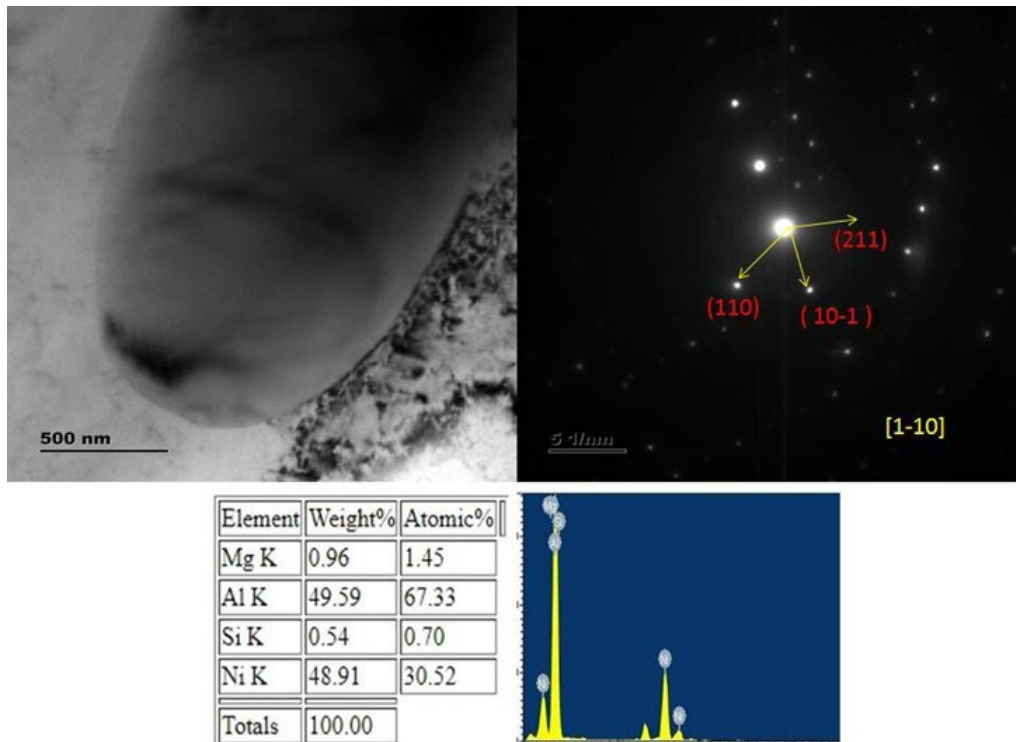
with (400) plane. Zone axis of the solutionized composite was found to be [001].  $Al_3Ni_2$  intermetallic phase formed in the solutionized composite exhibited elongated elliptical morphology having a size of  $2.5\mu m$  as shown in figure 5. T6 composite showed (110) and (10-1) planes of AlNi intermetallic phase. AlMg phase was also observed in the SAD pattern of T6 composite in (211) plane. T6 composite exhibited zone axis at [1-10]. Elliptical morphology of AlNi intermetallic phase was observed in bright field image of T6 composite (figure 6). Size of the AlNi intermetallic phase in the T6 composite was calculated as  $2.3\mu m$ .



**Fig.4** - (a) Bright field image (b) SAD pattern and (c) EDAX of the AA6061-1.5 wt% Ni composite in the as-cast condition.



**Fig.5** - (a) Bright field image (b) SAD pattern and (c) EDAX of the AA6061-1.5 wt% Ni composite in the solutionized condition.

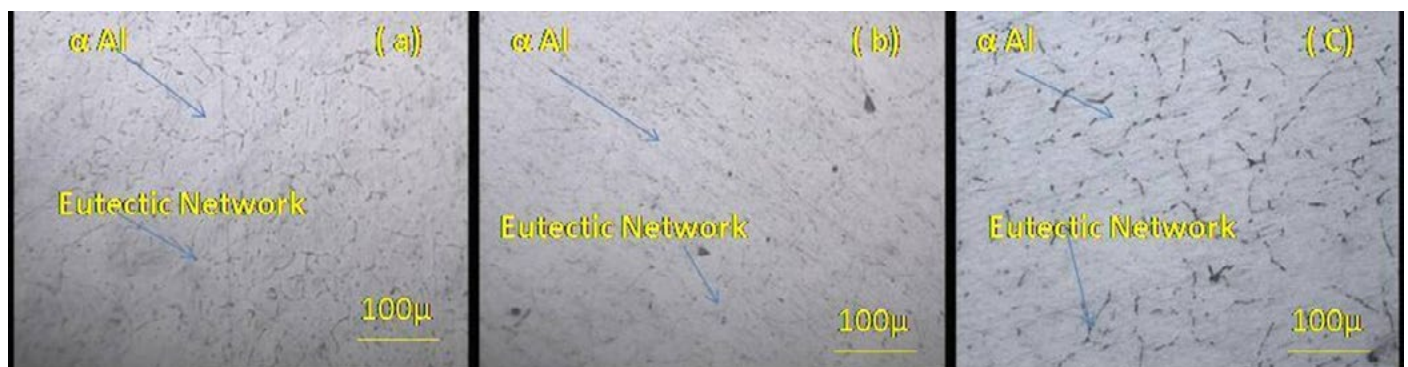


**Fig.6** - (a) Bright field image (b) SAD pattern and (c) EDAX of the AA6061-1.5 wt% Ni composite in the T6 condition.

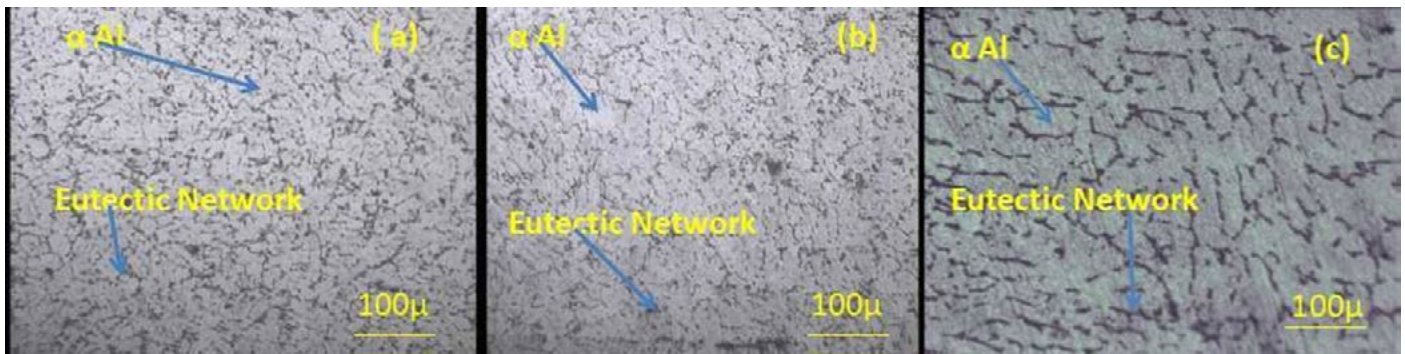
In as-cast and solutionized conditions, thermodynamics and rapid solid-state reaction favor the formation of  $Al_3Ni_2$ . During T6 (solution + artificial aging), prolonged exposure at elevated temperatures promotes further diffusion and transformation of  $Al_3Ni_2 \rightarrow AlNi$ , a more stable phase at higher temperature/time conditions. Based on indexing of the diffraction spots, the patterns identified corresponded to the zone axes of [1-21], [001] and [1-10] in the case of as-cast, solutionized and T6 composites respectively.

#### METALLOGRAPHIC EXAMINATION OF BASE ALLOY AND COMPOSITE

Microstructure of the base alloy and composite showed  $\alpha$ - phase in the as-cast, solutionized and T6 condition as shown in figure 7 and 8. Grain size of base alloy was found to be  $84\mu m$  in the as cast condition whereas grain size was  $72\mu m$  and  $65\mu m$  in the solutionized and T6 condition respectively. Increase in grain refinement was observed in the base alloy in the T6 condition and solutionized condition compared to as-cast condition due to the distribution of  $AlMg$  and  $Al_{12}Mg_{17}$  intermetallic phases observed in the XRD studies.



**Fig.7** - Microstructure of base alloy in the (a) as-cast (b) solutionized and (c) T6 condition.



**Fig.8** - Microstructure of AA6061-1.5 wt% Ni composite in the (a) as-cast (b) solutionized and (c) T6 condition.

Composites showed an increase in grain refinement compared to base alloy in the as-cast, solutionized and T6 condition respectively due to the heterogeneous nucleation sites provided by the addition of Ni particles [5]. AA 6061-1.5 wt% composite in the T6 condition showed a significant reduction in grain size ( $38\mu\text{m}$ ) compared to solutionized composite ( $47\mu\text{m}$ ) and as-cast composite ( $56\mu\text{m}$ ). This can be attributed to the distribution of equilibrium AlNi intermetallic precipitate formed of size  $2300\text{nm}$  in the T6 composite [3].

### MICROHARDNESS STUDIES OF BASE ALLOY AND COMPOSITE

Base alloy in the T6 condition found to give a hardness of 117 VHN compared to hardness of as-cast base alloy (93 VHN) and solutionized base alloy (101 VHN) due to precipitation hardening effect induced by AlMg equilibrium precipitate intermetallic phase formed in the T6 condition. Composite showed higher hardness than base alloy in the as-cast, solutionized and T6 condition respectively. Hardness values of base alloy and composite are shown in table 2.

**Tab.2** - Hardness of base alloy and composite in different conditions.

	As-cast condition	Solutionized condition	T6 condition
Base alloy	93 VHN	101 VHN	117 VHN
AA6061-1.5 wt% Ni Composite	118VHN	134	153 VHN

Composite in the peak aged condition showed a higher hardness of 153 VHN compared to composite in other conditions due to the precipitation strengthening imparted by AlNi equilibrium intermetallic precipitate phase formed [5] and excellent interfacial bonding between the matrix and intermetallic reinforcement [4, 6]. Moreover, composite exhibited peak ageing at  $165^\circ\text{C}$  for an ageing time of 4 hours compared to one of 8 hours for base alloy. Formation of AlNi intermetallic phase had resulted in the reduction of peak ageing time. Similar peak ageing results

were observed by Chen et al. [13] while studying peak ageing behaviour of AA6061 alloy reinforced with  $\text{Y}_2\text{O}_3$  and TiC at ageing temperature of  $160^\circ$ .

### WEAR STUDIES OF BASE ALLOY AND COMPOSITE

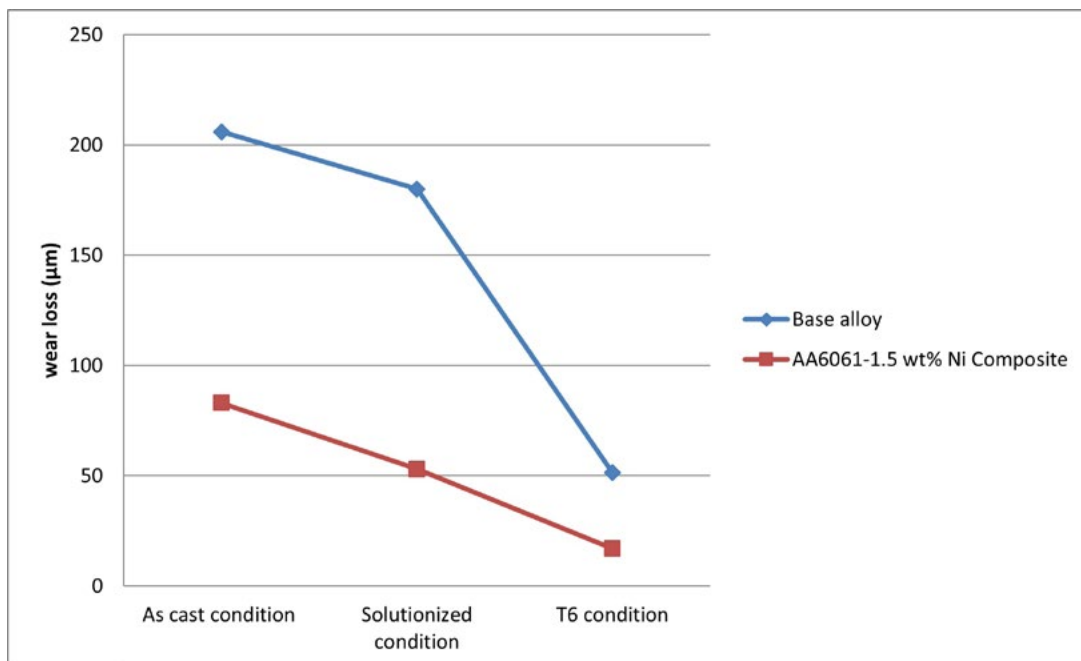
Wear loss of base alloy in T6 condition was found to be  $51.5\mu\text{m}$  in the tested condition which was 75% less than wear loss of base alloy in the as-cast condition. Increase in hardness of T6 base alloy had resulted in improved behavior compared to wear loss of base alloy in other conditions. Composites exhibited lower wear loss than base

alloy due to the higher hardness imparted by nickel aluminate phase formation [9, 12]. Peak aged (T6) composite showed a wear loss of 17µm compared to wear loss of solutionized composites (53µm) and as-cast composites (82µm) respectively. Wear results are shown in table 3 as

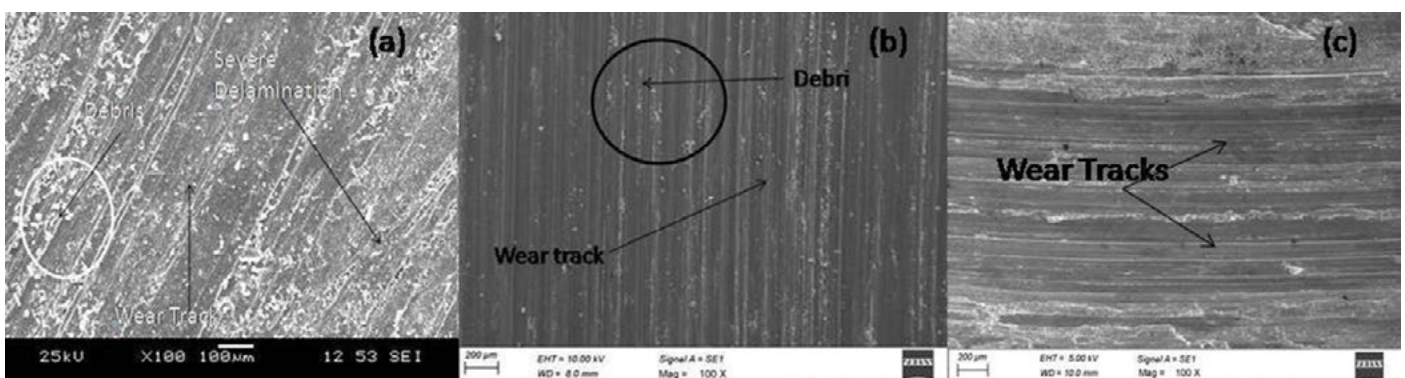
well as in figure 9. Higher hardness of T6 composite compared to other composite in as cast and solutionized conditions had led to superior wear resistance of T6 composite [8]. Worn out surface of the tested samples is shown in figure 10 and 11.

**Tab.3** - Wear results of base alloy and composite samples in the tested conditions.

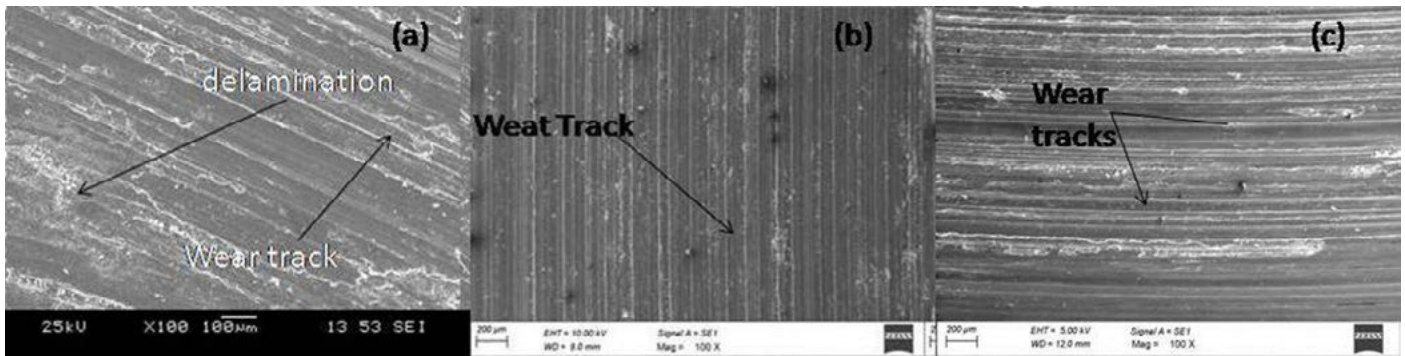
	As-cast condition	Solutionized condition	T6 condition
<b>Base alloy</b>	206µm	180µm	51.5µm
<b>AA6061-1.5 wt% Ni Composite</b>	83µm	53µm	17µm



**Fig.9** - Wear loss results of base alloy and composite samples.



**Fig.10** - Worn out surface of base alloy in the (a) as-cast, (b) solutionized and (c) T6 condition.



**Fig.11** - Worn out surface of AA6061-1.5 wt% Ni composite in the (a) as-cast, (b) solutionized and (c) T6 condition.

Base alloy showed a transition of adhesive wear behavior from severe delamination in the as-cast condition to presence of numerous wear tracks along with debris. Wear tracks were predominantly observed in the composite samples. Mild wear tracks were observed in the T6 composite. Sreenivasan et al. [10] observed delamination wear in 5, 10 and 15 wt% TiB<sub>2</sub> reinforced AA6061 alloy.

## CONCLUSION

Al<sub>3</sub>Ni<sub>2</sub> intermetallic phase was observed in the composite in the as-cast and solutionized conditions having a size of

2µm and 2.5µm respectively. AlNi intermetallic phase was observed in the composite in the T6 conditions having a size of 2.3µm. AlNi intermetallic phase formation and its distribution in the T6 composite had resulted in a higher grain refinement and higher hardness which led to superior wear resistance of T6 composite compared to other composites and base alloy. Composites predominantly showed wear tracks whereas base alloy exhibited severe wear tracks along with debris.

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