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EFFECT OF WEIGHT OF REINFORCEMENT AND COATING THICKNESS ON THE HARDNESS OF STIR CAST AL7075-NICKEL COATED DURALUMIN POWDER MMC

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In the present work, it is experimented to reinforce duralumin powder (3 to 7 wt %) into Al7075 matrix by stir casting technique. Since matrix and reinforcement both have almost similar melting temperatures, the least expensive additive manufacturing metallurgical route seems to be the best fit. In this study, an effort was made to produce the Al7075 matrix composite reinforced with duralumin by a novel stir casting method by coating duralumin powders with nickel which has high temperature melting point compared to reinforcement material. Nickel has good wettability and avoids undesirable chemical reactions between the reinforcement and matrix at higher temperatures, acting as a protector for both the duralumin and matrix. Since, aging kinetics of duralumin (Al2024) and Al7075 are different, both positively respond to heat treatment in a single stretch for property alteration. During stir casting, even though duralumin melts along with the matrix, it will be under the solid protection barrier (coat) of nickel, avoiding dissolution with the Al7075 matrix. To verify the presence of reinforcement duralumin in the matrix and to decide the soundness of the casting produced by stir casting, confirmation tests are made like microstructures with EDS and micro hardness distribution. The microstructure analysis of the composite showed an even distribution of nickel coated duralumin in the matrix when the coating thickness of a nickel is greater than 8 μm . The hardness test analysis has shown an improvement in the hardness with the increase in the weight % of the reinforcement. Improvement in the hardness of composites is due to an increase in dislocation number, which shows higher resistance to plastic deformation [2]. Statistical analysis has shown that the coating of reinforcement does not have any significant effect on the mechanical properties. The regression equation is fit to determine the hardness of the composite involving the factors within the range of values considered for this study.

Keywords: stir cast, coating, composites

1 INTRODUCTION

Metal Matrix composite, particularly Al7075 alloy matrix composite has a wide range of applications due to the versatile nature of the matrix to accommodate a variety of reinforcements and the possibility of matrix property alteration through heat treatment. Conventional nonferrous alloys have limitations for achieving desired strength, stiffness, and density target combinations. "Metal matrix composites" (MMCs) are produced to achieve a desirable combination of mechanical properties [1]–[2]. The Al7075 alloy is the aerospace industry's most widely used alloy. The Al7075 alloy belongs to the wrought alloy category and is characterized by its enhanced properties such as specific strength, higher toughness, and hardness [3]. They are generally utilized in the hardened (T6) condition. They are used in applications like aircraft fittings, gears and shafts, fuse parts, meter shafts and gears, missile parts, regulating valve parts, worm gears, and keys [4]. Duralumin (Al2024) has an excellent combination of machining characteristics, fatigue resistance, and tensile strength among other Al-Cu alloys [5]. It is widely used in aircraft structures, specifically fuselage structures and wings under tension [6]. It is also used in reciprocating and rotating parts like pistons, brake rotors, drive shafts, and other structural elements that require lightweight and high strength materials [7]. According to various studies, adding different reinforcements improves the mechanical properties of Al7075 matrix composites significantly [8]–[10]. Compared to other Al alloys, if strength is the most important consideration, then Al7075 is probably the better choice [11]. Shear strength, ultimate tensile strength, fatigue strength, yield strength, and hardness of Al7075 are comparable with those of duralumin alloy. Duralumin is the superior choice when both workability and high strength are required [12]–[13]. Surfaces of reinforcement can be coated with metallic or non-metallic compounds to enhance wet ability, mechanical properties, and to evade any undesirable chemical reaction between the reinforcement and matrix at higher temperatures [14]–[16]. It is also found that coating of reinforcements has shown a significant improvement in the MMC's mechanical properties [17]–[19]. Stir casting is beneficial for producing particulate-reinforced MMC's due to its ease of production and lower cost [20]. Compared to the costly additive manufacturing metallurgical route, stir casting is the best option when lower melting point matrix material is involved [21].

In this study, the MMC's are fabricated by using Al7075 as a matrix and nickel coated duralumin as reinforcement material. The mechanical characterization and microstructure analysis have been carried out to assess its applicability. Since there is limited work reported on the production and characterization of composites with matrix

and reinforcements having similar melting points, the present work even finds its novelty in statistical analysis to decide the quality of the composite casting. It is seen that the weight % of reinforcement is the major contributing factor to the composite's hardness, whereas the contribution of a thickness of the Ni coating on hardness was the minimum.

2 MATERIALS AND METHODS:

2.1 Chemical composition

The chemical composition of the cast product in the as received condition is determined using spectroscopy analysis. The details of the composition are given in Tables 1 and 2. It may be seen that the as received material has nearly the same chemical composition as that of the standard Al7075[3] and duralumin[6]material respectively.

Table 1: Chemical composition (wt.%) of as received Al7075

Material	Cr	Fe	Cu	Mn	Zn	Ti	Si	Mg	Al
wt.% (As received)	0.21	0.43	1.6	0.139	5.4	0.15	0.27	2.38	Balance
wt.% (Standard)	0.18 - 0.28	Max 0.5	1.2 - 2	Max 0.3	5.1 - 6.1	Max 0.2	Max 0.4	2.1 -2.9	Balance

Table 2: composition (wt.%) of as received duralumin powder

Material	Cr	Fe	Cu	Mn	Zn	Ti	Si	Mg	Al
wt.% (As received)	0.07	0.41	4.25	0.59	0.19	0.11	0.42	1.48	Balance
wt.% (Standard)	Max 0.1	Max 0.5	3.8 - 4.9	0.3 - 0.9	Max 0.25	Max 0.15	Max 0.5	1.2 - 1.8	Balance

2.2 Coating

The electro less nickel plating method was used to coat nickel on duralumin powder (reinforcement) with a coating thickness of 3, 5, 8, 9, and 10 μm . Electro less nickel plating (EN) was an autocatalytic reaction that deposits a uniform layer of nickel-phosphorus or nickel-boron alloy on the surface of a solid material or substrate, such as plastic or metal. The process includes dipping the substrate into a plating solution bath, where a reduction agent such as hydrated sodium hypophosphite ($\text{NaPO}_2\text{H}_2 \cdot \text{H}_2\text{O}$) reacts to deposit the nickel alloy with the ions of the material [14].

2.3 Stir casting

The Al7075 rod pieces were placed into the furnace and the temperature was increased to 750 $^\circ\text{C}$ and the uniform temperature was maintained for the liquid melt. Hexachloroethane- (C_2Cl_6) tablets (to degas) and alkaline powder (to remove slag) were added to melt. The liquid melt was mechanically stirred at 400 rpm to form a vortex, and then nickel coated duralumin powder (preheated at 300 $^\circ\text{C}$ / 3 h) was added to it. The stirring was continued for 10 minutes, and then the liquid melt was poured into cast iron preheated rectangular and pin moulds (500 $^\circ\text{C}$ for 1 h) and allowed to solidify to obtain castings for hardness and tensile test respectively.

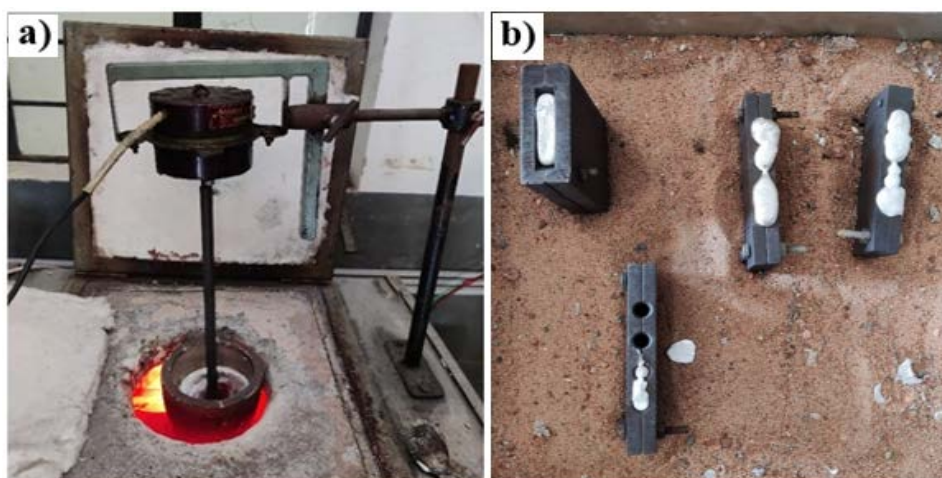


Fig.1: (a) Stir casting setup (b) Molten Al7075-Ni coated duralumin powder composite poured into preheated moulds

2.4 Hardness measurement

The cast samples from rectangular moulds were cut to the 3x5x3 cm dimensions to prepare samples for hardness testing. The hardness samples were polished using emery paper to obtain microscopically flat surfaces and etched

in Keller's reagent (190 mL H₂O + 5 mL HNO₃ (65%) + 3 mL HCl (32%) + 2 mL HF (40%)) for 15 sec to enhance the contrast on the specimen surface. Vicker's hardness test was conducted using MATZUSAWA MICRO VICKERS HARDNESS TESTER, MODEL-MMT X 7A with 200gmf load and 15 sec dwell time to study whether the reinforcement has been distributed uniformly throughout the composite and the effect of the reinforcement on the hardness of the composite in as cast conditions using ASTM 18-02 procedure.

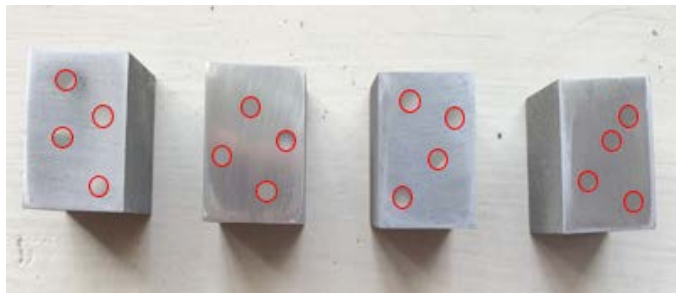


Fig.2: Hardness measurement samples of dimension 3x5x3 cm with location of hardness testing

2.5 Microstructure

The Scanning Electron Microscope (SEM) is used to obtain the microstructure of all the samples. Energy Dispersive X-ray Spectroscopy (EDS) is used to confirm the presence of reinforcements in the composites. The samples were polished using emery papers of different grades (80, 200, 400, 600, 800, 1000, and 2000 grit) and etched in Keller's reagent for 15 sec and washed with jet of water to enhance the contrast on the specimen surface. SEM and EDS were conducted using ZEISS EVO MA18.

2.6 Design of Experiments

The independent variables, or control factors, selected for the test are the amount of reinforcement and coating thickness. The dependent variable for the study is hardness. The control factors are varied at three different levels as given in Table 3. The values selected for the control factors are based on the literature review. The total number of experiments was conducted as per the full factorial approach. The full factorial approach is used to carry out the experiments. The total number of trials is obtained based on the formula, $LF = 3^2 = 9$ no. of trials[22].

Table 3: Details of control factors with different levels

Factors/Levels	1	2	3
Reinforcement (wt %)	3	5	7
Coating Thickness (µm)	8	9	10

The obtained results are subjected to Analysis of Variance (ANOVA) to determine the relative contribution of each factor to the hardness variation. A regression equation is generated for predicting the hardness involving the factors within the range of values considered for this study.

3 RESULTS AND DISCUSSION

3.1 Microstructure analysis

Microstructure analysis was carried out on uncoated and nickel coated duralumin powder to check whether the coating has taken place correctly or not. SEM images and Energy Dispersive X-ray Spectroscopy (EDS) reports of uncoated and nickel-coated duralumin powder (Figs. 3(a), 3(b), 4(a), and 4(b)) clearly demonstrate the evidence for nickel coating on duralumin powder.

Microstructure analysis was done on as cast Al7075 - 5 wt.% nickel coated duralumin powder MMC's with a coating thickness of 3, 5, 8,9, and 10 µm to check the presence of reinforcement in composites. The SEM images of as cast Al7075-5 wt.% nickel coated duralumin powder MMCs with 3 and 5 µm coating on the reinforcements are shown in Figs. 5(a) and 6(a). Since Al7075 & duralumin powder both have similar melting temperatures, the shallow (3 and 5 µm) Ni coated reinforcement (Al2024) might have dissolved during processing. This shows that the Ni coat thickness is not sufficient to protect the reinforcement from mixing with the base metal at casting temperature. The high temperature barrier (Ni cover around the reinforcement) is unable to bear the pressure of the molten reinforcement within it. The barrier cover might have burst and the reinforcement melt mixed with the base melt while stirring. The corresponding EDS (Fig.5(b) and Fig. 6(b)) show no indication of Ni in the selected areas. Since the quantity of Ni dissolved is very small (trace), it is not reflected even in the base metal during the EDS.

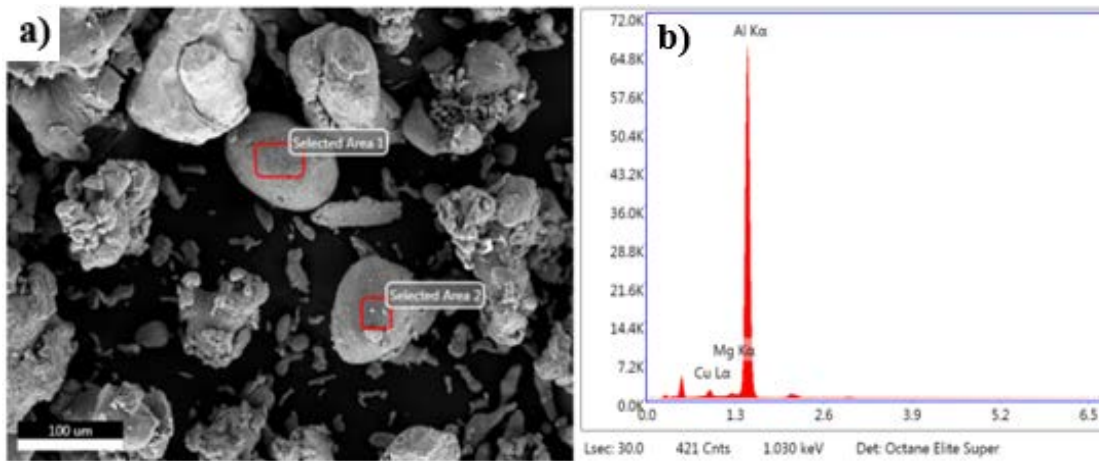


Fig.3:
(a) SEM of duralumin powder
(b) EDS of selected area 1

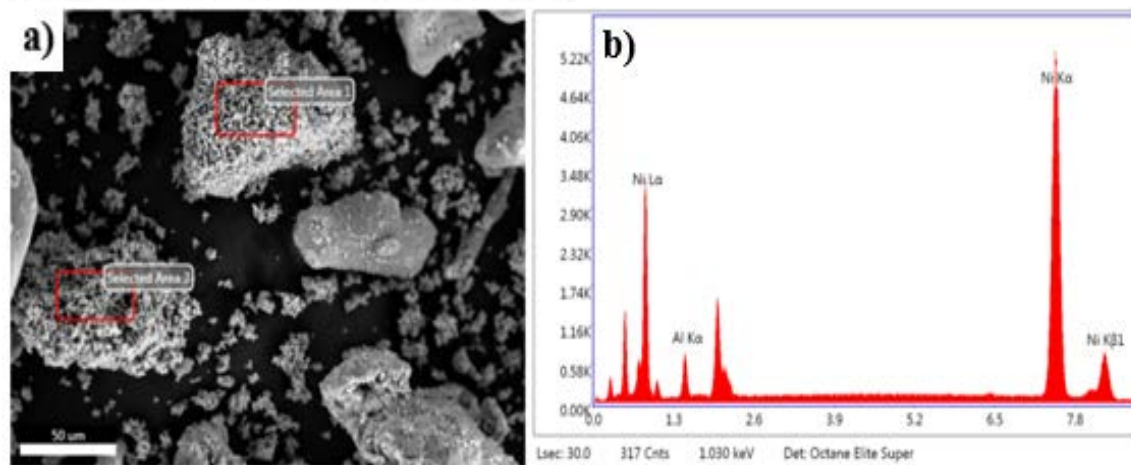


Fig. 4:
(a) SEM of nickel coated duralumin powder
(b) EDS of selected area 1

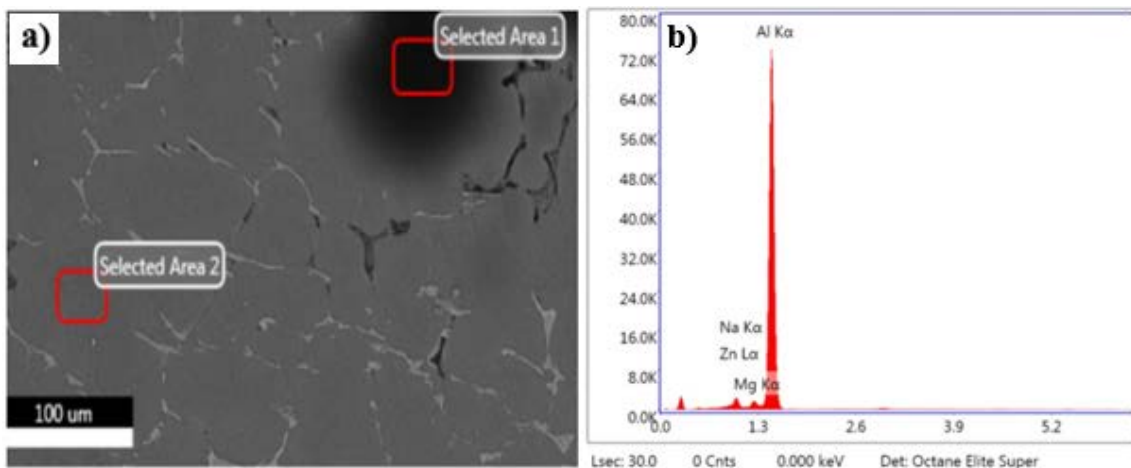


Fig.5:
(a) SEM image of as cast Al7075 - 5 wt.% nickel coated duralumin powder MMC's with coating thickness of 3 µm
(b) EDS of selected area 1

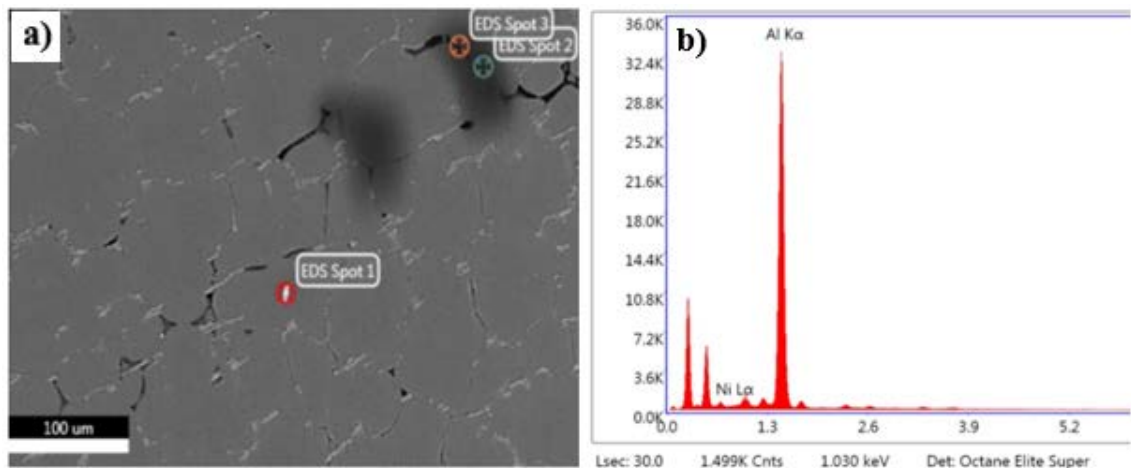


Fig.6:
(a) SEM image of as cast Al7075 - 5 wt.% nickel coated duralumin powder MMC's with coating thickness of 5 µm
(b) EDS of selected spot 1

The SEM images in Figs. 7(a), 8(a), and 9(a) depict the reinforcement spots (spectrum 1) as nickel coated duralumin with coating layer thicknesses of 8, 9, and 10 μm , respectively. The corresponding EDS (Fig. 7(b), 8(b), and 9(b)) shows the existence of Ni at spectrum 1. Among the range of coating thickness (3 - 10 μm) selected, 8 μm is the minimum thickness to be considered to bear the presence of the molten duralumin present. Duralumin and Al7075 (base metal) both have the same melting temperature. When the base metal is in molten state, duralumin is also in molten state. To prevent the mixing of duralumin with the base metal, high melting temperature nickel coating is given. When the base metal and reinforcement are in molten state, solid cover of nickel serves as interface between these two so that identity of the reinforcement is not lost in the composite.

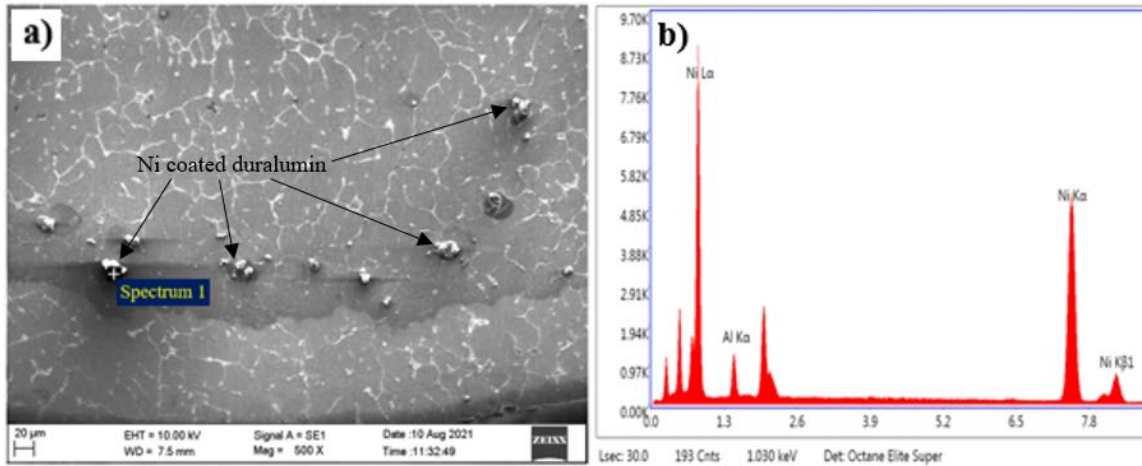


Fig.7: (a) SEM image of as cast Al7075 - 5 wt.% nickel coated duralumin powder MMC's with coating thickness of 8 μm (b) EDS of selected spot

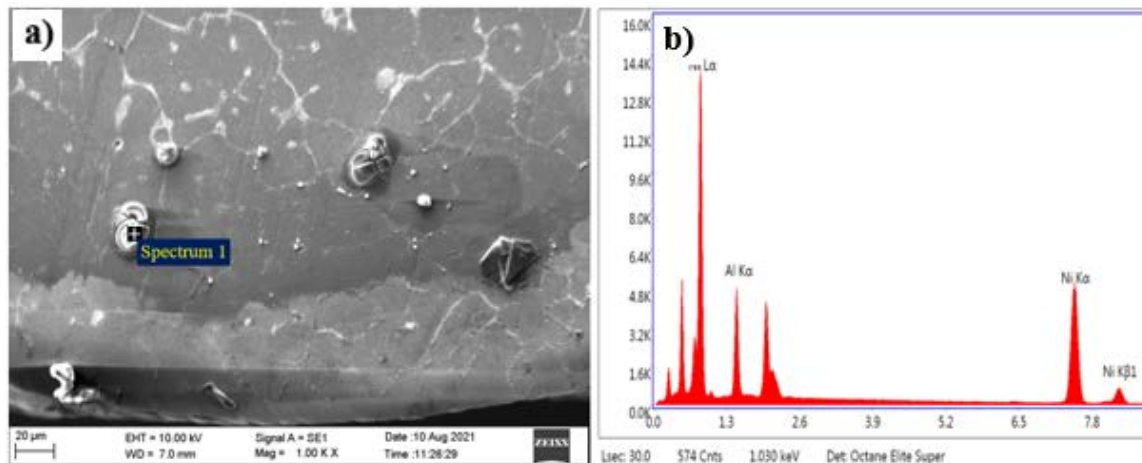


Fig.8: (a) SEM image of as cast Al7075 - 5 wt.% nickel coated duralumin powder MMC's with coating thickness of 9 μm (b) EDS of selected spot

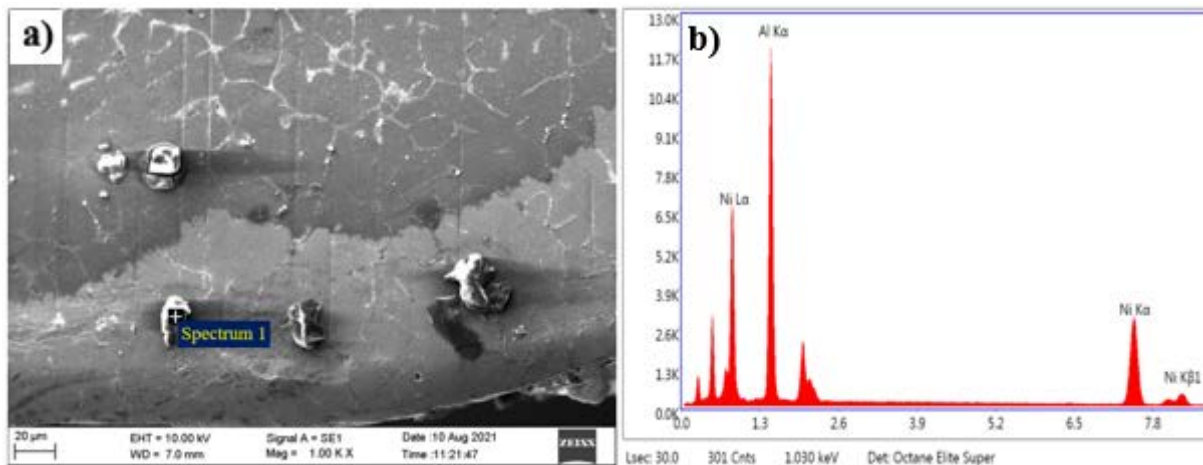


Fig.9: (a) SEM image of as cast Al7075 - 5 wt.% nickel coated duralumin powder MMC's with coating thickness of 10 µm (b) EDS of selected spot

3.2 Hardness test

Fig. 10 gives the hardness test results in graph form for different samples under various conditions. Vickers's hardness test is conducted to understand the reinforcement effect on the hardness of the composite. As cast Al7075-nickel coated duralumin powder composites are subjected to the Vickers's hardness test (HV). Fig. 10 shows that 7 wt.% reinforced composite has higher hardness as compared to 3 and 5 wt.% reinforced composites, and composites with a 10 µm coating of nickel on reinforcements have better hardness than composites with an 8 and 9 µm coating of nickel on reinforcements. From Fig. 10, it is clear that the value of hardness increases with increasing the reinforcement percentage in the matrix alloy (Al7075).

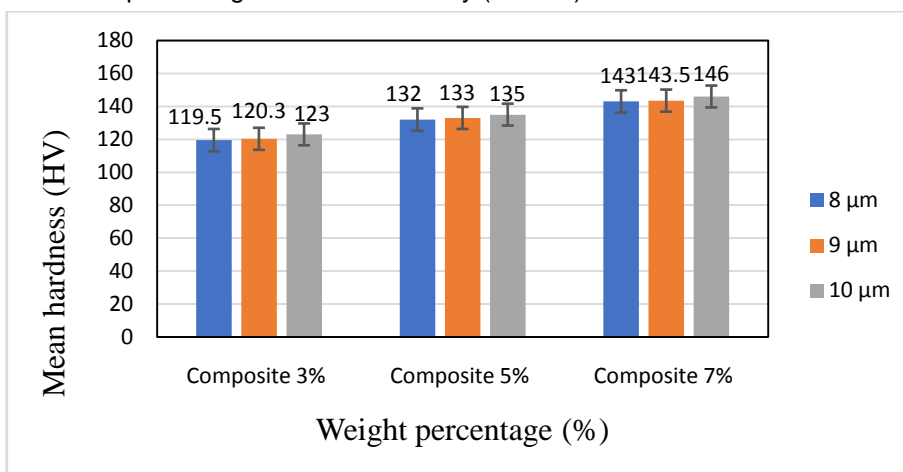


Fig.10: As cast hardness of Al7075- 3, 5, 7 wt.% nickel coated duralumin powder MMC's with coating thickness of 8, 9, and 10 µm

In the aluminum alloy matrix, during the solidification process, there is a thermal mismatch between reinforcement and matrix phase due to a difference in the thermal expansion coefficient, which increases dislocation densities to raise the bulk hardness and UTS. The microstructure and mechanical properties of the MMC's are altered due to a mismatch between the matrix and reinforcement strain and accordingly internal stress developed in the system[2]. Improvement in the hardness of composites is due to an increase in dislocation number, which shows higher resistance to plastic deformation[2]. Adding reinforcement during solidification reduces matrix grain size, improving the mechanical properties of the composite[23]–[25]. There was a substantial improvement in the hardness of composites as the wt.% of duralumin was increased, but there was a minimal enhancement in the hardness of composites as the coating thickness was increased. Therefore, to find the contribution of coating thickness and wt.% of reinforcements on the hardness of the composites, Analysis of Variance (ANOVA) was conducted.

3.3 Statistical analysis

The results obtained for hardness under various conditions are subjected to statistical analysis using Minitab software. An Analysis of Variance (ANOVA) was conducted to determine the amount of relative contribution of each factor on the hardness of the test samples. ANOVA results for hardness are given in Table 4.

Table 4: ANOVA result for hardness

Source	DF	Seq SS	Adj SS	MS	F	P	% contribution
Coating thickness	2	16.38	16.38	8.19	177.57	<0.001	1.98
wt. %	2	810.91	810.91	405.45	8792.99	<0.001	97.99
Error	4	0.18	0.18	0.05			
Total	8	827.47					

From Table 4, it may be seen that wt. % is the major contributing factor to the variation of hardness under the range of values considered for this study. Variation in coating thickness does not have much contribution to the hardness for the range of values considered for the study. In the as cast condition, there is a nominal improvement in the mechanical properties of the composite material. The effect of variation in coating thickness is also found to be minimum. However, considerable improvement is expected in the mechanical properties when the same composite is subjected to age hardening treatment where the kinetics of age hardening is different in Al7075 matrix

as well as duraluminium reinforcement. The difference in the aging kinetics may impose misfit strain at the interface to improve bond strength and related properties.

The regression equation is fit to predict the hardness of the samples involving the parameters within values considered for this study. The regression equation is given in equation 1.

$$\text{Hardness} = 89.5 + 5.81 (A) + 1.58 (B) \quad (1)$$

Where, A–Reinforcement (wt. %), B- Coating thickness (μm)

$$R\text{-Sq} = 99.7\% \quad R\text{-Sq}(\text{adj}) = 99.6\% \quad (2)$$

The high R square value obtained for the regression equation is evident for the good fit, and the same equation may be used to predict the hardness of the samples involving the parameters within the range of values considered for this study.

4 CONCLUSIONS:

The composites can be manufactured by stir casting successfully with 3, 5, and 7 wt.% Ni coated duralumin powder. The EDS report confirms the Ni coating on the reinforcement powder. 3 and 5 μm Ni coat thickness on the reinforcement is unable to bear the molten pressure of the reinforcement, leading to dissolving in the melt matrix during stir cast processing. 8, 9, and 10 μm thickness Ni coating on the powder, during the processing of the composites, can provide a protective cover for the reinforcement powder to prevent dissolution with the matrix. The uniformity in microhardness distribution in the cast composite shows that the casting obtained is sound without reinforcement clusters in the matrix. As the wt.% of reinforcement increases in the matrix, the bulk hardness of the composites also increases. The ANOVA technique is conveniently applied to determine the amount of relative contribution of reinforcement quantity and coating thickness on the hardness of the test samples. It is seen that the weight % of reinforcement is the major contributing factor on the composite's hardness, whereas the contribution of a thickness of the Ni coating on hardness was minimum. However, the coating thickness may play a vital role when the aging treatment will be carried out on the composite. The high R square value (99.6), confirms that the regression equation produced may be utilized to determine the hardness values for various wt.% of reinforcements (between 3 to 7) and coating thickness combinations (between 3 to 10 μm).

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