# Synthesis, Characterization and Mechanical Behaviour of $AI_2O_3$ , TiO<sub>2</sub>, and Cu Reinforced AI 7068 Nanocomposites

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**Abstract:** This present research work aims at fabrication of AA7068 metal matrix composite reinforced with a different weight percentage of Al<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub> and Cu (0 w t.%, 2 w t.%, and 4 w t.%) nanopow ders through mechanical alloying of 30 hrs which is produced using powder metallurgy route. The consolidation pressure of 500 MPa was applied for compaction of the composite and sintered at a temperature of 600°C for two hrs in the presence of argon gas flow. An XRD result reveals that there are no intermetallic compounds formed in the milled powder after 30 hr of mechanical alloying. The reinforcement particles were well embedded and uniformly distributed in matrix composites was confirmed by bright-field emission transmission electron microscopy (FETEM) image and selected area diffraction (SAD) ring pattern. From the DSC curve of AA 7068–2.0 wt. % Al2O3, TiO2 and Cu nanocomposite powders after 30 hrs of mechanical alloying. the endothermic peak at 536.85°C corresponds to the melting of aluminium which was followed by a steady-state exothermic reaction at 579.51°C was obtained. The green density and sintered density of prepared nanocomposites were calculated and compared. Brinell hardness test has been conducted and the maximum value of 192 BHN w as obtained by adding a weight percentage of 2 w t. % of Al<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub> and Cu particles.

Keywords: Nanocomposites, Mechanical Alloying, Characterization, Mechanical Behaviour.

## 1. INTRODUCTION

In recent years, hybrid reinforcements with metal matrix nanocomposites are a prime consideration to enhance mechanical properties [1]. Aluminium 7068 alloy is widely used in the automotive, aerospace Industries and ordinance applications due to good mechanical properties compared with other aluminium alloys. Titanium Dioxide (TiO<sub>2</sub>) was used as reinforcement in epoxy-co-polyamide composite and got improvement in tensile strength and elastic modulus in the recent study [2]. The effect of copper (Cu) addition in the aluminium matrix gave on the resultant hardness increment [3]. Alumina (Al<sub>2</sub>O<sub>3</sub>) has excellent hardness, dielectric property, wear resistance and chemical inertness properties. The effect of various reinforcements in aluminum and magnesium metal matrix composites through various synthesis methods are analysed in Table 1.

Various authors have studied the effect of various reinforcements with aluminium metal matrix composites on mechanical properties and its microstructural analysis. However, there is limited work in the effect of hybrid reinforcement with 7068 aluminium alloy on mechanical properties and its microstructural analysis through mechanical alloying. Therefore, the main aim of the present research work is to study and investigate the effect of  $AI_2O_3$ ,  $TiO_2$  and Cu reinforced AI 7068 alloy nanocomposites through mechanical alloying on its mechanical properties and its characterization analysis.

#### 2. EXPERIMENTAL PROCEDURE

### 2.1. Raw Materials

Aluminium alloy 7068 was produced by blending of highly pure (99.5 %) elemental powders and 200-mesh size. The raw materials were purchased from High Purity Laboratory Company (HPLC), India. Table **2** shows the chemical composition to make aluminium alloy 7068. Blending was done at high-energy ball mill at 250 rpm for 2 hr.

## 2.2. Processing of Nanocrystalline Powders

Various weight percentages (0%, 2%, 4%) of  $AI_2O_3$ ,  $TiO_2$  and Cu reinforced AI 7068 nanocomposite powders were prepared using a planetary ball mill. Balls of made up of tungsten carbide and 10 mm diameter each weighing 10 g. Totally 300 g (30 balls) were sealed with 100 g of the AI 7068 -Wt%,  $AI_2O_3$ ,  $TiO_2$  and Cu powder mixture. The speed of the mill is set to 250 rpm and the processing time is set to 30 hours. However, 5 hours of milling is alternated with 20 min. of cooling to avoid a significant temperature rise.

# 2.4. Compaction and Sintering

The milled powders were consolidated into cylindrical pellets of 10 mm diameter 15 mm length using a

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#### SI. Authors **Major Metal** Reinforcements Methods Inferences Ref. No Matrix 1 K. Jhon Joshua AI 7068 Mechanical Alloying AA7068 pow ders was reduced [4] from 3.280 µm to 1.785 µm for a et al. milling period of 40 hours at a speed of 101 rpm. 2 K. Jhon Joshua AA7068 Pow der Metallurgy The wear resistance has been MaO [5] (0%, 1%, 2% and 5%) et al. route improved by adding MgO particles in AA7068 matrix material. 3. J. Lakshmi AA7068 6 vol.% of MoS2 -X vol. Pow der Metallurgy The increased wear resistant. [6] Hybrid % of WC Route hardness and high strength Pathy et al. (X=0,5,10 and 15) Composites M. Madhusudhan AA7068 ZrO2 Stir Casting Techniques Hardness and Tensile strength 4. [7] was increased with increase in et al. Zirconium di oxide particles in w eight percentage of composites 5. V. Sridhar Magnesium AI2O3 Pow der Metallurgy An increase in amount of nano-[8] (0.35, 0.7, and 1.4 et al. Route alumina reinforcement led to a progressive increase in microvolume %) hardness of pure (Mg) magnesium. Ali Hubi Haleem 3, 6, 9, and 12 w t.% of Pow der Metallurgy Improvement in Brinell Hardness 6. Aluminium [9] α- Al2O3 particles Route (BHN) by 89% and compression et al. strength by 54 %. 7. M. Karbalaei Aluminium 12 wt. % of α-Alumina Casting Improvement in hardness and its [10] tensile strength in the Akbari nano-particles nanocomposites with addition of 1.5 and 2.5 vol. % Al2O3 nanoparticles 8. M. Karbalaei A356 AI2O3 Pow der Metallurgy The effect of particle size and the [11] amount of reinforcement is given Akbari Route improvement in mechanical properties and fracture behavior A. Baradesw aran AI 7075 1.5 and 2.5 vol.% Liquid Metallurgy Route Improvement in hardness and its [12] 9 Al2O3 nanoparticles tensile strength in the et al and Graphite nanocomposites TiB2 content increased to 5% 10. Chen et al. Aluminium Zn-Al-Cu-TiB2 Liquid Metallurgy Route [13] (mass fraction), an improvement in hardness and ultimate tensile strength (UTS) was achieved 11. S. Dhanalakshmi AI7075 AI2O3-B4C Stir Casting The maximum tensile strength, [14] et al. micro-hardness and macrohardness of 309 MPa, 140 VHN, and 112 BHN was obtained Abhishek Kumar AI2O3 12. A359 Electromagnetic Stir The tensile strength of the as cast [15] Casting Method composites increases on et al. increasing the weight fraction of AI2O3. Mahallaw i et al. AI2O3 and TiO2 13. A390 Mechanical Stirring Enhancement in the micro-[16] hardness, hardness and wear nanoparticles resistance of the Al2O3/TiO2 nano-dispersed hypereutectic A390 alloys 14. A. Mazahery et al. Aluminium 0.75, 1.5, 2.5, 3.5, and Mechanical Stirring Presence of nano-[17] 5 vol. % nano-Al2O3 AI2O3 reinforcement led to significant improvement in 0.2% yield strength and ultimate tensile stress

#### Table 1: The Effect of Various Reinforcements in Aluminum and Magnesium Metal Matrix Composites

| Table 1. d | contd |
|------------|-------|
|------------|-------|

| SI.<br>No | Authors   | Major Metal<br>Matrix       | Reinforcements   | Methods Inferences  |  | Ref. |
|-----------|---|-----------------------------|--|---|--|------|
| 15.       | Sahin <i>et al</i> .                              | AI 2014                     | AI2O3  | Pow der Metallurgy<br>Route Method  | w der Metallurgy<br>Route Method<br>The w ear rate increased w ith<br>increasing the load and<br>decreased w ith increasing the<br>particle sizes for composites   |      |
| 16.       | S.A. Sajjadi <i>et al</i> .                       | A356<br>Aluminium<br>Alloy  | AI2O3  | Stir and Compo-<br>Casting Processes  | The improvement in yield<br>strength, ultimate tensile strength,<br>compression strength and<br>hardness w ere obtained  | [19] |
| 17.       | S.A. Sajjadi <i>et al</i> .                       | A356<br>Aluminium<br>Alloy  | Micro and nano<br>level Al2O3  | Stirring & Compo-<br>Casting Method   | The amount of porosity in the<br>composites increased with<br>increasing w eight fraction and<br>speed of stirring and decreasing<br>particle size   | [20] |
| 18.       | Sekar <i>et al</i> .                              | A356<br>Aluminium<br>Alloy  | AI2O3  | Combined effect of Stir<br>and Squeeze Casting  | Hardness, Compressive strength<br>and double Shear strength<br>show ed an increase w ith an<br>increase in addition to Al2O3<br>nanoparticles  | [21] |
| 19.       | S. Tahamtan <i>et al</i> .                        | AI/A206                     | Nano and Micro<br>level Al2O3  | Combining ball milling<br>and stir casting<br>technology  | Improvement in tensile properties  | [22] |
| 20.       | K. Umanath <i>et al</i> .                         | AI 6061                     | SiC/Al2O3  | Liquid Metallurgy Route   | The w ear resistance of the 15%<br>hybrid composite w as better than<br>that of the 5% hybrid composite  | [23] |
| 21.       | Mohammed <i>et al</i> .                           | Aluminium                   | Graphene   | High energy ball milling<br>(HEBM) & molecular<br>level mixing (MLM)<br>processes follow ed by<br>spark plasma sintering<br>(SPS) | Improvement of 79, 49 and 44%<br>of yield strength, ultimate<br>strength, and Vickers hardness,<br>respectively, for 1 w t % graphene<br>containing nanocomposite in<br>comparison to the unreinforced<br>AI–4Cu alloy             | [24] |
| 22.       | Avw erosuoghene<br>Moses Okoroa<br><i>et al</i> . | Ti6Al4V                     | 0.5, 1.0 and 1.5 w t%<br>Multi-w alled carbon<br>nanotubes (MWCNT)<br>pow ders | High energy ball milling<br>(HEBM) follow ed by<br>spark plasma sintering<br>(SPS)  | Micro-hardness improved<br>tremendously with an increase in<br>sintering temperatures  |      |
| 23.       | M. K.Akbari <i>et al</i> .                        | A356                        | TiB2 & TiO2  | High Energy Ball Milling  | Improvements in hardness and<br>w ear resistance w ere obtained in<br>A356-1.5 vol.% TiB2 composite  | [26] |
| 24.       | Meysam<br>Toozandehjani<br><i>et al</i> .         | AI                          | AI2O3  | High Energy Ball Milling  | The increase in the milling time<br>resulted in the homogenous<br>dispersion of 5 w t % nanoparticles<br>and improvement in the density,<br>densification, micro-hardness<br>(HV), nano-hardness (HN), and<br>Young's modulus (E). |      |
| 25.       | Yilong Yang <i>et al</i> .                        | AA 2219                     | TiC  | Ultrasonic Solidification   | The average grain sizes of a-AI matrix alloy reduced by 61%  | [28] |
| 26.       | Julia Osten <i>et al</i> .                        | AA 7068                     | 5% TIC   | Mechanical Alloying<br>and Hot Pressing<br>routes   |  | [29] |
| 27.       | Amin Azimi <i>et al</i> .                         | AI 7068                     | TiC  | Mechanical Alloying   | High compressive strength and<br>hardness of 938 MPa and HV 265<br>w ere achieved  | [30] |
| 28.       | R. Taherzadeh<br>Mousavian et. al                 | A356<br>aluminium<br>matrix | SiC nanoparticle   | Mechanical Alloying   | Improvement in tensile strength<br>(UTS), yield strength (YS) and<br>strain  | [31] |

Table 1. contd....

| SI.<br>No | Authors                         | Major Metal<br>Matrix | Reinforcements     | Methods             | Inferences   | Ref. |
|-----------|---------------------------------|-----------------------|--------------------|---------------------|--|------|
| 29.       | K.R. Ramkumar<br><i>et al</i> . | AI 3003<br>alloy      | TiO2 nanoparticles | Mechanical Alloying | Ultimate Tensile strength of 249 ±<br>04 MPa w as obtained w ith 3%<br>TiO2 reinforced AI 3003<br>composites | [32] |
| 30.       | D. eyasimman<br><i>et al</i> .  | AA 6061               | 2 w t.% TiC        | Mechanical Alloying | The Maximum hardness of 1180 MPa was obtained for $2 \text{ w t}$ .% TiC.                                    | [33] |
| 31.       | D. Jeyasimman<br><i>et al</i> . | AA 6061               | 2 w t.% MWCNT      | Mechanical Alloying | anical Alloying The Maximum hardness of 818<br>MPa was obtained for 2 wt.%<br>MWCNTs.                        |      |
| 32.       | D. Jeyasimman<br><i>et al</i> . | AA 6061               | 2 w t.% Al2O3      | Mechanical Alloying | The Maximum hardness of 740<br>MPa was obtained for 2 w t.%<br>Al2O3 .                                       | [35] |

Table 2: Chemical Composition of Aluminium Alloy 7068

| Element | Percentage |  |
|---------|------------|--|
| Si      | 0.12       |  |
| Fe      | 0.15       |  |
| Cu      | 1.80       |  |
| Mn      | 0.10       |  |
| Mg      | 2.50       |  |
| Zn      | 8.00       |  |
| Ti      | 0.10       |  |
| Cr      | 0.05       |  |
| Zr      | 0.15       |  |
| AI      | Balance    |  |

hydraulic press with a capacity of 10 Tons at a compaction pressure of 500 MPa. The green pellets were sintered for 2 hours at 873 K under a reducing atmosphere and presence of the Argon gas flow. The theoretical densities of the samples were calculated using the rule of mixture. The density of sintered pellets was estimated precisely using the Archimedes principle. The estimated error in the density measurements was less than 1%.

# 2.5. Powder Morphology and Hardness Measurement

Nanoparticles within the Al7068 matrix was investigated by scanning electron microscopy (SEM), X-ray diffraction (XRD), transmission electron microscopy (TEM) and differential scanning calorimetry (DSC). The crystallite size and lattice strain of the milled powder samples were investigated by X-ray diffraction analysis on a D/Max Ultima III; XRD machine (Rigaku Corporation, Japan). The samples were continuously exposed to Cu K $\alpha$  radiation ( $\lambda$ =1.5406 Å) at a scanning speed of 2° per min. operating at 30 mA and 40 KV. The scanning range was 20°-80° in steps of 0.02. The crystallite size (*t*) and microstrain ( $\epsilon$ ) were determined using the standard Williamson-Hall analysis [36]. The morphology of the resulting powders and distribution of reinforcement nanoparticles within the AI 7068 matrix was investigated by SEM (TESCAN model VEGA 3 LMU). The structure of mechanically alloyed powder was observed with a JOEL JEM 2100F field emission transmission electron microscope (FETEM).

# 3. RESULT AND DISCUSSION

#### 3.1. Powder Morphology Evolution

The scanning electron microscope images and X-Ray diffraction peaks of the as-received pure aluminium, Al<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub> and Cu powders were shown as Fig. (1a-d). The aluminium powders were irregular in shape and had an average particle size of approximately 75 µm. Major peaks (1 1 1), (0 0 2), (0 2 2), (1 1 3) and (2 2 2) of aluminum with FCC crystal structures were obtained. The corresponding JCPDS Card Number is 98-008-4180. Peaks (2 1 1), (2 1 1), (0 2 1) and (1 1 3) of Al2O3 with FCC crystal structures were obtained. The corresponding JCPDS Card Number is 98-010-3822. Similarly all the eight major peaks (1 1 0), (0 1 1), (0 2 0), (1 1 1), (1 2 0), (1 2 1), (2 2 0), (0 0 2), (1 3 0), (0 3 1) and (1 1 2) of TiO2 with FCC crystal structures were obtained. The corresponding JCPDS Card Number is 98-002-2145. Three major peaks (1 1 1), (0 0 2) and (0 2 2) of Cu with FCC crystal structures were obtained. The corresponding JCPDS Card Number is 98-009-2397. As received pure powders



**Figure 1:** The SEM and XRD images of as received pure powders (a) AI (JCPDS Card Number: 98-008-4180); (b) Al2O3 (JCPDS Card Number: 98-010-3822); (c) TiO2 (JCPDS Card Number: 98-002-2415)and (d) Cu (JCPDS Card Number: 98-009-2397).

morphology and sizes were confirmed by XRD and SEM.

The aluminium 7068 powder was ball milled up to 30 hr after 5 hr ball milling was stopped and taking one-

gram sample out for morphological analysis. Repeated the process up to 30 hr and kept it separate. Then aluminium 7068-2 wt. % (Al<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub> and Cu) powder particles were ball milled up to 30 hr. The mixture

powder was ball milled at 30 hr after 5 hr ball milling was stopped and taking one- gram sample out. Repeated the process up to 30 hr and kept it separate. Similarly, Aluminum 7068 -4 wt.% (Al<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub> and Cu) powder particles mixture was ball milled up to 30 hr. The mixture powder was ball milled up to 30 hr after 5 hr ball milling was stopped and taking one- gram sample out. Repeated the process up to 30 hr and kept it separate. During, ball milling the aluminium powder particle size was reduced. After the 30 hr ball milling, the microparticles changed into nanoparticles. After the 5 & 10 hr of the particle is flattening and fracturing. After 15 hr of the milling, cold welding was predominance. After the 20 hr of the milling, fracturing was dominance. After the 25 hr of the milling, equiaxed particle formation was started. After the 30 hr of the milling, steady state was achieved. The SEM and XRD

images of AI 7068-2 Wt. % AI2O3, TiO2, and Cu nanocomposite powders as a function of the milling time and its morphological changes were shown in Fig. (2). From the XRD images, clear peaks of Aluminum, Al<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub> and Cu were obtained and confirmed the preferred nanocomposite was AI 7068-2 Wt.%AI<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub>, and Cu nanocomposite. In addition, a small Zn peak was obtained because of its weight fraction is more (7.5 Wt. %) compared to other elements in AA 7068. Peaks for Si, Mg, Fe, Ti, Cr and Mn which are related to AI 7068 alloy, were not detectable due to their low volume fraction [37]. These components were expected to dissolve in the Al lattice. Fig. (2), X-ray diffraction images indicate that MA decreased the peak intensities and increased the peak width of Aluminium due to the structural refinement that resulted from the MA milling time.



**Figure 2:** The SEM and XRD images of Al 7068–2 Wt.%Al<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub>, and Cu nanocomposite powders as a function of the milling time after: (a) 5 h; (b) 10 h (Particle flattening and fracturing); (c) 15 h (Cold welding predominance); (d) 20 h (Fracturing dominance); (e) 25 h, (Equi-axed particle formation starts) and (f) 30 h (Steady state).

#### 3.2. Structural Evaluation

Table **3** shows the structural evaluation of AA 7068 - 2wt. % Al<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub> and Cu composite powder for various mechanical alloying times (5hr, 10 hr, 15 hr, 20 hr, 25 hr and 30 hr). The crystallite size was decreased from 48 nm to 28 nm after 5 hr to after 30 hr of mechanical alloying. However, the lattice strain increased from 0.0037 to 0.005 as a function of the milling time due to the severe plastic deformation (SPD) by the high-energy ball mill. The slight decrease in the crystallite size and slight increase in the lattice strain indicate the attainment of steady state milling at 30 h. This was shown in Fig. (**3a**). The Williamson-Hall analysis [36] was adapted to measure crystallite size (*t*) and lattice strain ( $\varepsilon$ ) using the following expression:

$$\beta_{hkl} . \cos \theta_{hkl} = \left[\frac{k\lambda}{t}\right] + 4\varepsilon . \sin \theta_{hkl}$$
(1)

where k is the shape factor (0.9),  $\lambda$  is the wavelength of the X-ray radiation (1.5406),  $\theta_{hkl}$  is the Bragg angle and  $\beta_{hkl}$  is the full-width at half maximum after instrumental broadening correction. The first five AI reflections, peaks (1 1 1), (0 0 2), (0 2 2), (1 1 3) and (2 2 2) were used to construct a linear plot of  $\beta_{hkl} \cos \theta_{hkl}$  against 4 sin  $\theta_{hkl}$ . Crystallite size (t) was obtained from the intercept and the strain ( $\varepsilon$ ) from the slope. The lattice parameter calculated by using interplanar spacing and miller indices. The actual lattice parameter was obtained from the intercept as described by Cullity [37] by constructing a linear plot between the calculated lattice parameter for each Bragg angle on the Y-axis and the corresponding value of  $\cos^2 \theta / \sin \theta$  on the Xaxis. The lattice parameter reported for pure FCC AI at room temperature is 4.0496 Å [37]. The lattice parameter reported for AI 6061-2 wt. % AI<sub>2</sub>O<sub>3</sub> nanocrystalline is 4.0456 Å after milling for 30 h [35]. In this investigation, the lattice parameter for the AI 7068-2 wt. % Al<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub> and Cu nanocrystalline matrix was

Table 3: Structural Characterization of AA 7068 -2wt. % Al<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub> and Cu Composite Powder for Various Ball Milling Times (hr)

| SI. No. | Ball Milling (hrs) | Crystallite Size (nm) | Lattice Strain (ɛ) | Lattice Parameter (a) Å | Unit Cell Volume (V) m <sup>3</sup> |
|---------|--------------------|-----------------------|--------------------|-------------------------|-------------------------------------|
| 1       | 5                  | 48                    | 0.003742           | 4.0490                  | 66.38092                            |
| 2       | 10                 | 43                    | 0.004112           | 4.0480                  | 66.33175                            |
| 3       | 15                 | 37                    | 0.004571           | 4.0462                  | 66.24331                            |
| 4       | 20                 | 34                    | 0.004985           | 4.0448                  | 66.17457                            |
| 5       | 25                 | 31                    | 0.005500           | 4.0425                  | 66.06175                            |
| 6       | 30                 | 28                    | 0.006140           | 4.0410                  | 65.98824                            |



**Figure 3:** (a) Change in crystallite size and lattice strain of AA 7068–2 wt.% Al<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub> and Cu as a function milling time; (b) lattice parameter as function of milling time.

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4.0414 Å after milling for 30 hr. So that the adding reinforcement, reduces lattice parameters values slightly.

Fig. (4a) shows the bright field emission transmitssion electron microscopy (FETEM) image of Al 7068-2 wt. % Al<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub> and Cu after 30 hr. From that image, bright areas represent nanocrystalline aluminium matrix and dark areas represent cu and TiO<sub>2</sub> nanoparticles. In addition, alumina nanoparticles were identified. From Fig. (4a), the observed grain size of the  $\alpha$ -Al matrix is almost equi-axed and at an ultra-fine level. Further, the Al<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub> and Cu nanoparticles were uniformly distributed and embedded in the  $\alpha$ -Al matrix. Fig. (4b) shows the selected area diffraction pattern and confirms the nanocrystalline composite powder.

Fig. (5) Shows the DSC curve of AA 7068–2.0 wt. % Al2O3, TiO2 and Cu nanocomposite powders after 30 hrs of Ball milling. DSC curve of Al 7068-2 wt.% Al2O3, TiO2 and Cu nanocomposites analysed, because The 2 wt.% of reinforcement content in metal matrix gave optimum mechanical properties (Hardness value). Hence, Al 7068-2 wt. % Al2O3, TiO2 and Cu nanocomposites were taken for analysis



**Figure 4:** (a) Bright Field Emission Transmission electron microscopy (FETEM) image of Al 7068-2 wt. % Al<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub> and Cu after 30 hr; (b) Corresponding SAD ring pattern.



Figure 5: The DSC curve of AA 7068–2.0 wt.% Al<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub> and Cu nanocomposite powders after 30 hrs of Ball milling.

The differential scanning calorimetry used to measure enthalpy changes due to changes in the physical and chemical properties of a material as a function of temperature or time. Heat flow is directly proportional to the capacity of the material for the given temperature. The studies were conducted by using 17 mg sample in an aluminium sample holder by purging of pure nitrogen gas at an airflow rate 50 ml/min with a temperature range of 25°C to 600°C through a heating rate of10°C/min. The endothermic peak at 536.85°C corresponds to the melting of aluminium, which was followed by a steady-state exothermic reaction at 579.51°C. The higher calorific value obtained from this study was ( $\Delta$ H) 24.74J/g.

#### 3.3. Density and Hardness Measurement

Table 4: Density and Hardness Measurement

Table 4 shows the density and hardness measurement of prepared nanocomposites. Theoretical density was calculated by the rule of mixture. The green and sintered densities of the prepared nanocomposites were determined to employ the Archimedes principle. The growth of the theoretical, green and sintered

densities (%), significantly increased, due to the very fine particle size and distribution of reinforcements in a soft alloy matrix. Fig. (6a-b) shows the relation between theoretical, green and sintered densities. The density positively correlated with the reinforcement content (0%, 2% and 4%) The densities of these nanocomposites was increased after sintering. The growth of the theoretical density (%) significantly increased, due to the very fine particle size and distribution of reinforcements in a soft alloy matrix. This effect was confirmed by our earlier results (33-35). The Hardness test was conducted by the Brinell Hardness Testing Machine. The experimental error was less than 10% in the hardness measurement. The Brinell hardness number 173.2 BHN, 192 BHN and 184 BHN values were obtained for AI 7068-0 wt.% AI2O3, TiO2 and Cu nanocrystalline, AI 7068-2 wt.% Al<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub> and Cu nanocomposites and AI 7068-4 wt.% Al<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub> and Cu nanocomposites respectively.

The higher amount of reinforcement (More than 2 Wt. %) will lead to particle agglomeration and deteriorate mechanical properties. Al 7068-2 Wt.%

| SI. No | Nanocompositos   | Theoretical<br>Density (g/cc) | Actual Den       | Brinell Hardness |              |
|--------|--|-------------------------------|------------------|------------------|--------------|
|        | Nanocomposites   |                               | Before Sintering | After Sintering  | Number (BHN) |
| 1      | AI 7068-0 w t.% Al <sub>2</sub> O <sub>3</sub> , TiO <sub>2</sub> and copper | 2.85                          | 2.62416          | 2.64138          | 173          |
| 2      | AI 7068-2 w t.% Al_2O_3, TiO_2 and copper                                    | 3.02254                       | 2.73994          | 2.76988          | 192          |
| 3      | AI 7068-4 w t.% Al $_2O_3,\text{TiO}_2\text{and copper}$                     | 3.19508                       | 2.90685          | 2.95462          | 184          |





Figure 6: (a) Theoretical density Vs Green density; (b) Theoretical density Vs Sintered density.

Al2O3, TiO2 and Cu nanocomposites gave more hardness value than Al 7068 -4 Wt.% Al2O3, TiO2 and Cu nanocomposites. Because, the agglomeration of particles will lead to decrease mechanical properties. Furthermore, the addition of reinforcement content gave a reduction in hardness. Fig. (7) shows Brinell hardness number (BHN) of prepared AA 7068 nanocomposites after 30 hrs of MA. The 2 Wt. % of reinforcement content in metal matrix gave optimum mechanical properties and it was confirmed by previous studies [32-34].



**Figure 7:** Brinell hardness number (BHN) of prepared AA 7068 nanocomposites after 30 hrs of MA.

# 4. CONCLUSION

The present study examined AA7068-xwt. %  $AI_2O_3$ ,  $TiO_2$  and Cu (x = 0 wt. %, 2wt. %, and 4wt. %) nanocomposite prepared by powder metallurgy route and synthesis, structural changes, characterization and mechanical behavior.

The powder morphology of prepared nanocomposites was investigated and reported as a function of milling time. An XRD result reveals that there are no intermetallic compounds formed in the milled powder after 30 hr of mechanical alloying. An equiaxed and almost spherical powder morphology was obtained after 30 hr mechanical alloying which was characteristic of the steady state. The reinforcement particles were well embedded and uniformly distributed in Al 7068 metal matrix composites. It was confirmed by bright-field emission transmission electron microscopy (FETEM) image and selected area diffraction (SAD) ring pattern. From the DSC curve of AA 7068–2.0 wt. % Al<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub> and Cu nanocomposite powders after 30 hrs of mechanical alloying, the endothermic peak at 536.85°C corresponds to the melting of aluminium which was followed by a steady-state exothermic reaction at 579.51°C. The higher calorific value of ( $\Delta$ H) 24.74J/g. Density and hardness measurement was analyzed. Al 7068-2 wt. % Al<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub> and Cu nanocomposites gave better hardness value 192 BHN and it was 11% more than unreinforced nanocrystalline.

Densification behavior, wear behavior and tensile and compression strength measurements for the above-prepared nanocomposite materials will be analysed and addressed in future work.

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