Effect of Hot rolling on Microstructure and Mechanical behaviour of B₄C nano particulates reinforced Al6063alloy Composites

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Abstract: In this research composite of aluminum alloy reinforced with nano B_4C particles were fabricated through ultrasonic-cavitation assisted casting process followed by hot-rolling at a temperature of $450^{\circ}C$, The Micro structural analysis showed the presence and uniform distribution of boron carbide nano-particles with good bonding with Al6063 alloy, in case of hot rolling, The B_4C particles have been found to align in the rolling direction. The Grain size analysis of composite indicates that significant grain refinement compared to base matrix alloy. The addition of nano B_4C particles shows improvement in hardness compared to non-reinforced aluminium in before and after hot rolling. The tensile strength of as cast composites was increased with an increase in nano B_4C particle content up to 4 wt. % but beyond 4wt% leads to the decrease in strength of composites, The hot rolled Al6063alloy and its composites show a substantial increase in hardness, tensile strength and ductility those of as cast counterpart.

Keywords: Al6063alloy, Ultrasonic Cavitation assisted casting, hot rolling, Microstructure and Mechanical behaviour

| Date of Submission: 10-04-2021 | Date of Acceptance: 26-04-2021 |
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I. Introduction

Particulate reinforced aluminum alloy composites are becoming one of the most promising materials and have the ability to provide tailored mechanical properties such as low weight, high strength, high specific strength, stiffness and good wear resistance, make them attractive for several interesting applications in the field of aerospace, automotive, defense industries as well as sporting goods and electrical packaging [1-2]. Among all the available aluminum alloys 6000series have numerous advantages including good formability, medium strength, good weldability, corrosion resistance and heat treatable [3]. Depending on applicability and functionality the aluminum matrix is getting strengthened by using hard ceramic reinforcement particles such as SiC, TiC, B_4C , Al_2O_3 , Si_3N_4 , AlB_2 and TiB₂ [4].

The B₄C particles are of great importance among them because of certain distinguishing features including high strength, high stiffness, which possesses lower-density $(2.52g/cm^3)$, extremely more hardness, better chemical stability, and neutron absorption capability[5].From this point of view, composites with B₄C reinforced aluminium alloy matrix composites have been used in a wide range of industrial applications.

The performance of composites can be improved by reducing the size of reinforcement particles from micro to nano level such materials are referred as nano-composites. The several processing techniques are widely used for fabrication of metal matrix nanocomposites such as Sintering process, Powder metallurgy, Mechanical alloying,[6-7]Ball milling process[8],Spray deposition techniques , Squeeze casting, Pressure infiltration, Stir casting and other various casting techniques [9].Uniform distribution of nano-particles in molten metal is a very challenging task due to low wettability, wide surface to volume ratio with the use of traditional stir-casting technique, This process can be used without agglomeration and clustering to disperse the micro-sized particles in molten metal, However Some researchers have been suggested the ultrasonic-cavitaion assisted casting techniques to disperse and distribute the nano reinforcement particles in molten metal which improves the wettability, grain structure and homogeneous distribution of nanoparticles in the matrix alloy[10-11].However the casting defects such as shrinkage cavities, porosities and blow holes are produced during the processing of composites which are very difficult to remove or eliminate at the time of manufacture. In order to conquer these casting defects further the developed composites materials are subjected to secondary forming process, such as rolling, extrusion and forging resulting in reduced porosity, improved bonding among matrix and reinforcement phase and uniform distribution of reinforcements particles

and as cast microstructure is transformed into a deformed structure which contributes to improve the processing properties of the composites. [12-13]. Among various forming methods rolling is the most widely used forming process in industrial applications to manufacture sheets for automotive bodies, construction materials, roofing panels, railtracks, refrigerators, truck frames and other household appliances[14]. Alizadeh, et.al. [15] investigated the effect of the mechanical and wear behaviour of B₄C nanoparticles reinforced Al2024alloy composites synthesized by mechanical-milling followed by extrusion process, observed the significant improvement in mechanical properties and wear resistance by increasing the content of B_4C nano-particles in the Al2024 alloy matrix. Mohamadsharifi, et.al. [16] studied the mechanical and wear behavior of B_4C nano particles reinforced AMMNC fabricated by mechanical alloving. They have reported that the enhancement in tensile strength and wear resistance of developed composites by increasing the content of the B_4C nano-particles in the aluminium matrix alloy. It is observed from the literature review huge amount of work exists on microstructure and mechanical characterization of nanocomposites with aluminum materials, however very limited information is available on effect of hot-rolling process on micro structural and mechanical behavior of Aluminium alloy based nanocomposite. In view of the above, the current research work is aimed to develop a metal matrix composite containing boron carbide nano-particles as reinforcements in Al6063 alloy matrix using an ultrasonic cavitation assisted solidification process followed by hot rolling in order to produce a high strength composite material, the effect of hot-rolling on micro structural and mechanical characteristics of synthesized composite will be studied and compared with that of cast composites.

II. Methodology

2. Materials and Methods

2.1. Materials details

In the present study Al6063alloy used as a matrix material, it is a medium strength alloy with having silicon and magnesium as the major alloying elements, this alloy is commonly referred as an architectural alloy, Al6063 chosen as matrix material for present study due to its excellent characteristics like good formability, higher corrosion resistance good surface finish, The 6xxx-group alloys have widespread applications especially in the structural, automobile and aircraft industry, The elemental composition of Al6063matrix alloy used in the present study as reported in table-1.

Table-1: Chemical Composition of Al6063 alloy

| Elements | Si | Mg | Cu | Mn | Fe | Zn | Cr | Ti | Al |
|-------------|------|------|------|------|------|------|------|------|-----|
| Composition | 0.45 | 0.50 | 0.02 | 0.03 | 0.22 | 0.02 | 0.03 | 0.02 | Bal |

Boron Carbide (B₄C) nanoparticles used as reinforcement material due to its attractive properties,

including lower density, high strength and high hardness, Boron carbide nanoparticle of size 100 nm used as a reinforcing material. The boron carbide has a lower density than Al6063, so after mixing the reinforcement material into the matrix alloy the overall composite density may decrease. Boron carbide is capable of reacting to the aluminum grain boundary and enhancing the mechanical properties of composites. The Common applications of B_4^C includes armor and wear protection, neutron absorbers, bearings, nozzles and turbines. The typical properties of matrix alloy and reinforcement are listed in table-2. The SEM image and EDAX spectra of B_4^C as shown in figure1

| | Table-2: Properties of A16063 and Boron carbide | | | | | | |
|----------|---|------------------------------------|---------------------------------|--|-------------------------------|--|--|
| Material | Density (g/cm ³) | Melting point (^O C) | Thermal conductivity (W/m.k) | Coefficient of thermal expansion(µm/m- ^O C) | Modulus of Elasticity(GPa) | | |
| A16063 | 2.71 | 620 | 210 | 23 | 70 | | |
| B_4C_p | 2.51 | 2450 | 40 | 5 | 450 | | |

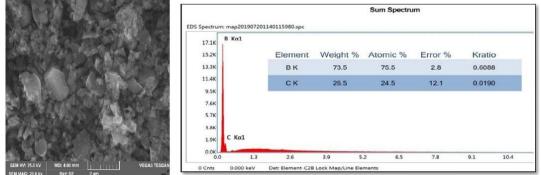


Figure1: SEM image and EDAX spectra of B₄C nano particulates

2.2. Fabrication of Composites and Hot rolling process

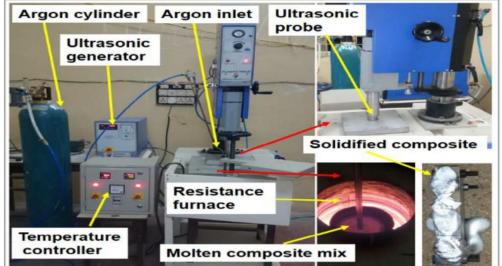


Figure2: Ultrasonic assisted stir casting set up

The Al6063alloy-Al6063-nanoB₄C composites were synthesized using an ultrasonic assisted casting process. The fig.2.Illustrates the experimental set up consisting of an electrical resistance heating furnace with a maximum heating capacity of 1200°C for melting of Al6063 alloy in EN8-steel crucible, ultrasonic processing system, mechanical stirrer and protection gas system, a known quantity of Al6063 alloy was melted in an electrical resistance heating furnace at a heat of 750° C, The preheated B₄C nano-particles were added into the molten melt and stirred with the help of mechanical stirrer for 10mintues to achieve proper mixing of reinforcement particulates. The molten métal is protected from atmospheric air by Argon gas during melting. The proper homogeneous mixing of the nanoparticles in the molten aluminum is the most challenging task in the fabrication of MMNC, due to the formation of clusters of nanoparticles [17]. To overcome this problem after stirring, the ultrasonic probe was used to produce a vibration frequency of 18 to 20 kHz and a maximum output of 4 kW for the melting process. The ultrasonic probe is immersed in molten metal to a depth of around 30 mm which help to break down the nanoparticle clusters through cavitations and acoustic emission. Finally after completing the process crucible was lifted from the resistance heating furnace and the slurry of molten metal was transferred into the pre-heated metallic die. The aluminum metal matrix nano composites containing B C nano particles at proportions of 2wt. %, 4wt. % and 6wt. % were fabricated. As shown in fig2-(a) The cast composites were cut into 40x50x10 mm³ rectangular specimens and heated to a temperature of 450° C for one hour, once the heating was done then the specimens were subjected to a hot rolling process using a hot rolling mill (Model:BHULER&CO.GmbH VRW105/32-100,2007Germany) as shown in fig2-(b).The hot rolling process consisting of four passes in each pass achieved a 20 percent reduction in thickness, fig2-(c) shows the photograph Rolled sheets of Al6063alloy and Al6063- B₄C nano composites.



Fig 3(a): Before hot rolling



Fig 3(b): Hot rolling mill



Fig 3(c): Photograph of hot rolled composites

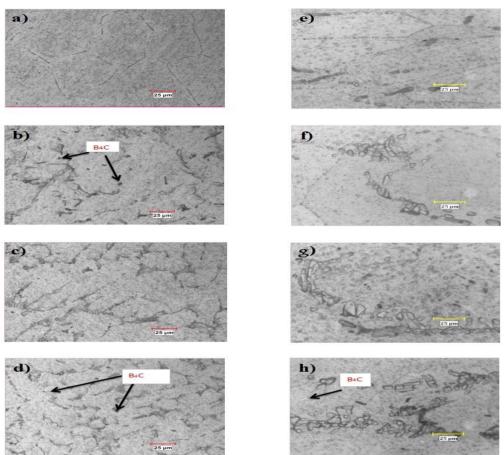
2.3. Microstructure and mechanical properties testing

Metallographic techniques were used to polish the samples, the polished surfaces of samples were etched using Keller's reagent(5ml HNO₃, 3ml HCl, 2ml HF and 190ml H₂O) and their microstructure were examined by an Optical microscope (Make: JSM840a Jeol).Grain size analysis was performed using climax image analyzer as per the standard ASTM-E112 test process. The Vickers Micro hardness test was carried out with a load of 100g for dwell time of 10s on metallographically polished samples by using the Vickers hardness tester. The tensile experiments were carried out both before and after hot rolling samples accordance with the ASTM-E8 standard. The average of three tensile test results to be used for UTS of each specimen, the samples used for tensile testing were cut through wire EDM process in rolling direction (RD).

III. Results and Discussions

3.1 Microstructure analysis

The optical microscopic images of Al6063alloy and Al6063- nB₄C metal matrix composites prior to rolling process as shown in fig.4 (a-d) it was observed from the microstructure where the reinforcement particles have been found along the grain boundaries and also it is observed from the optical images the hard ceramic B₄C particles are uniformly dispersed in the Al6063matrix alloy, it is also observed after hot rolling that most of the reinforcement particles have been aligned in the direction of metal flow, compared to the Al6063 alloy, the composites shows a smaller size of grains due to the presence of B₄C nano-particles which contribute to the grain refining. The existence of boron in B₄C, a well-known grain refiner plays a very important role in the grain refining of the composites. The optical images of Al6063alloy and Al6063-nB₄C composites after hot rolling as shown in fig.4 (e-h). The micro structural changes occurred during rolling. The grain morphology has been modified in the direction of rolling to the elongated grain structure, Nucleation of new grains at the grain boundaries of grains; the microphotograph clearly reveals the minimal porosity in both base alloy and its composites after hot rolling. In addition, the composites show a good bond between the aluminium matrix and B₄C nano-particles which may be attributed to better wettability of particles, uniform distribution and refinement the reinforcement within the alloy matrix leading to the improvement in strength of the composites.



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Figure.4:Optical images of as cast sample a) Al6063alloy b) 2wt% B₄C c) 4wt% B₄C and d) 6wt% B₄C and Hot rolled samples e) Al6063alloy f) 2wt% B₄C g) 4wt% B₄C and h) 6wt% B₄C

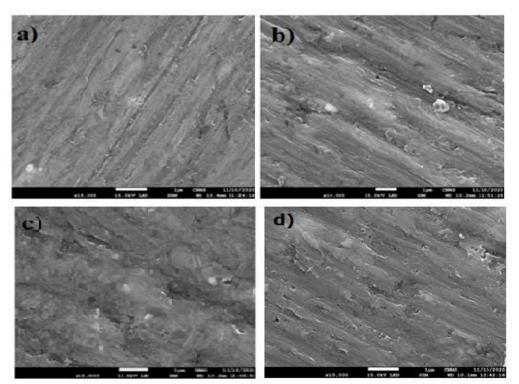


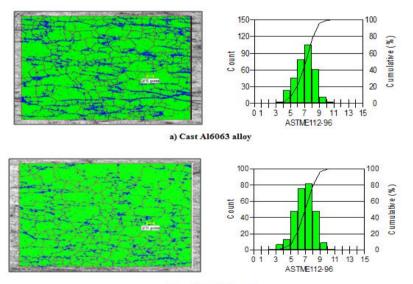
Figure.5: SEM micrographs of hot rolled samples a) Al6063alloy b) $2wt\%B_4C$ c) 4wt% B_4C and d) 6wt% B_4C



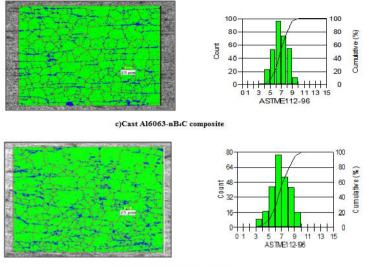
Figure.6: SEM micrographs of hot rolled Al6063-nB₄C composite sample with EDAX Spectrum

3.2 Grain size analysis

The grain size analysis was performed on the Al6063 alloy and Al6063-nano B_4C composites as shown in Fig.7 (a-d) it is observed that before and after rolling condition the average grain size of the composite is substantially reduced compared to base alloy matrix. This may be due to the presence of hard B_4C nano particles, during solidification of cast composites which acts as nucleating sites and extensively refined grain size. However, as compared with as cast composite hot rolled composite shows greater degree of grain refinement, The improved grain structure refining of hot rolled of alloy and its composites can be mainly attributed to the dynamic recrystallization during rolling.[18] The drastic decrease in grain size leads to enhancement in properties such as microhardnes, tensile strength and toughness of the prepared composites.



b)Hotrolled Al6063 alloy



d)Hot rolled Al6063-nB4C composite

Figure.7 :(a-d) Grain size analysis of Al6063alloy and its Composite before and after hot rolling



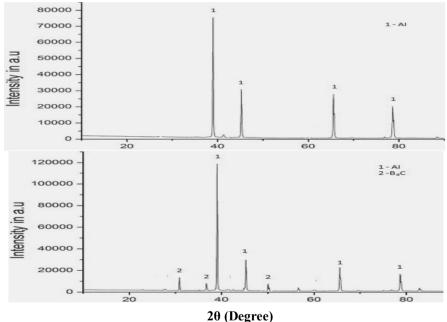


Figure.8:X-Ray Diffraction pattern of hot rolled Al6063-B₄C nano composite.

The XRD pattern of hot rolled Al6063alloy and Al6063-nB₄C composite as shown in fig. The XRD pattern of Al6063 alloy indicates most of the peaks corresponding to pure aluminium. The presence of Aluminium phases with different intensities are confirmed at 39°, 45°, 65° and 79°. The maximum Aluminium phase peak intensity is observed at 39°. In addition, the XRD pattern of Al6063-B₄C composite indicates different phases of Al and B₄C nano-particles The aluminium phases are available at different 20 angle with variable intensities and B₄C particle phases are identified at 31°, 38° and 50° with different intensities.

3.4. Micro hardness study

The influence of boron carbide nano particle on micro hardness of the Al6063matrix alloy and Al6063/nanoB₄C composites before and after rolling conditions are as shown in fig.7, The casted and hot rolled samples subjected to micro hardness studies, the micro hardness of specimens was found and averaged by placing the test indenter at different places to avoid the indenter placing on much hardre B₄C reinforcement particles. The graph shows that micro hardness increases with the increase of addition of B_4C nano-particles to

the Al6063 alloy before and after hot rolled conditions. It was found that the hardness of Al6063/B₄C Composites is increased up to 22.9% after hot rolling. The hardness increase is mainly attributed to the following factors. In general the addition of harder reinforcement particles in an aluminum alloy which enhances the hardness of the matrix [19]. The adding of hard B_4C reinforcement particles to the matrix alloy which may increases the density of dislocation during solidification of cast composites, this in turn increases resistance to plastic deformation resulting in enhanced hardness. The hot rolled composites samples shows the better hardness values in compared to the cast samples due to the following factors. The hot rolled process reduces the porosity content, refines the grain structure and more homogenous distribution of B_4C nano-particles in the hot rolled composites.

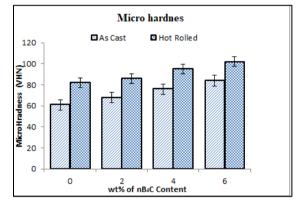


Figure.9: Variation of micro hardness before and after hot rolling

3.5 Ultimate tensile strength analysis

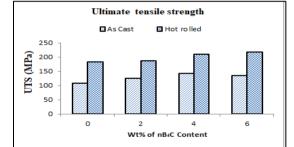


Figure.10: Variations of ultimate tensile strength before and after hot rolling

Figure.8 shows the variations in tensile strength of Al6063 alloy and Al6063-nanob₄c composites before and after hot rolling conditions. The graph shows that an increase in strength of composite with an increase in B_4C nano particle content up to 4wt% but above 4wt% leading to a decrease in tensile strength before rolling process, this may due to agglomeration of nano-particles and content of micro porosities, however due to the application of hot rolling process, significant improvement in the tensile strength of both alloy and its composites after hot rolling process as compared to the cast composite. The hot rolled composite shows a better tensile strength which can be mainly attributed to the effect of microstructure changes in the matrix alloy such as grain refinement, porosity reduction, and uniform distribution of reinforcements in the matrix alloy. The Various factors that contribute to the improvement of the tensile strength of composite including a excellent bonding between the matrix alloy and reinforcement, distribution of hard ceramic particles in the soft ductile matrix alloy and refinement of grain structure. The existence of nano-particles which acts as barrier to the movement of dislocation in the matrix alloy, due to variation in thermal expansion coefficient between the matrix and B4C particles, thermal residual stresses are induced and increased dislocation density, the increased dislocation density enhances the strength of composites. The addition of hard reinforcement particles to soft ductile matrix alloy which also enhances the strength of composites. The reinforcement particles acts as a nucleation sites increase the nucleation rate and decrease the grain growth rate results in smaller grain size composite compared to base metal. According to the Hall-petch equation, smaller the grain size higher the strength, and this may be reason for improvement in strength of composite compared to the base alloy,

3.6 Ductility analysis

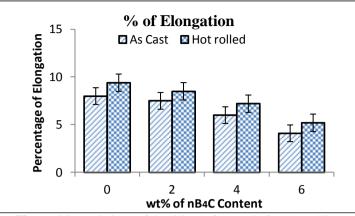
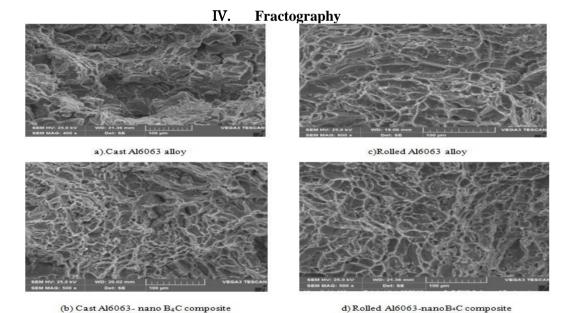


Figure.11: Variations of ductility before and after hot rolling

The variation in ductility of the Al6063 alloy and Al6063-nB₄C composites prior and after hot-rolling process as shown in fig.9 it is observed that the addition of B_4C nano-particles in Al6063alloy matrix tends to decreases the ductility of composites. The decline in the ductility of composites may be due to the presence of hard ceramic B_4C particles in soft ductile matrix alloy, the reinforcement of hard ceramic particles creates phases as well as some secondary intermetallic phases that resist deformation of matrix on application of load and lead to nucleation of cracks at interfacial zones as well as the presence of possible micro porosities contributing to the failure of matrix material [20]. Due to the presence of boron, refinement of grain structure also leads to the ductility of composites. In the alloy matrix, the rolling process strengthens the composite by increasing the hardness which consequently reduces the ductility. The presence of scattered brittle materials and intermetallic compounds that could have formed during the process act as crack nucleation sites leading to early failure of the composite under the application of load.



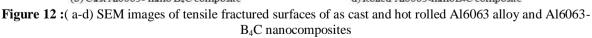


Fig.12 (a-d).shows the SEM images of tensile fracture surfaces of Al6063 and Al6063-nB₄C composites before and after hot rolled condition. Fig. 10(a) and (b) shows the fractography of cast 6063 alloy and its composite shows that the ductile fracture which as a large number of dimples shaped structures and also large voids can be seen in the matrix Al6063 alloy, and the composite containing of B₄C nanoparticles having less ductile fracture, debonding between the nano B₄C Particles and Al 6063 matrix interface are the some of the reasons for the failure of composites also a small voids are observed in the Al6063-nano B₄C composites and also the presence of small dimples indicating the formation of ductile fracture exhibits cleavage fracture due to the brittleness of hard ceramic surrounded soft matrix alloy it is observed that the cracks in the matrix alloy are

large with fracture hard B₄C particles. The SEM analysis of tensile fracture surfaces of rolled Al6063 alloy and Al6063-nB₄C composites as shown in fig (c) and fig (d) it is observed that the sizes of dimples are finer in the matrix alloy and its developed composite when compared with their cast counter parts owing to better plastic deformation as shown in fig (c) and as supported by the results of elongation studies. Further the shape of the dimples is honey comb as shown in figure (d) which suggests the enhanced ductility in the material. Fractograph studies clearly support that an excellent bond between matrix and reinforcement.

V. Conclusions

> Aluminum (6063) alloy based composites reinforced with B_4C nano particles were fabricated by using UCA casting techniques and effectively hot-rolled at a temperature of 450°C.

> The microstructure indicates the uniform distribution of B_4C nano-particles and refined grain structure in the matrix alloy after hot rolling led to increase in the mechanical properties of the composite

 \gg With the addition B₄C nano-particles to the matrix alloy, the micro hardness of Al6063-nB₄C composites has improved before and after rolling.

> The result of the tensile analysis indicates that the tensile strength of as cast composites was increased with an increase in B_4C particle content up to 4 wt % but beyond 4wt% leads to decrease the strength of composites due to more agglomeration of particles and high porosity content but in the hot rolled state the UTS values increased continuously with an increase in B_4Cp content due to less porosity and more homogeneous distribution of B_4Cp in the matrix alloy.

> The ductility of hot rolled composites was improved than that of as- cast composites due to reduced porosity content, homogeneous distribution of particles and microstructure refinement.

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