

Microstructural Studies and Material Characterization of Alumina Nanoparticulate Reinforced Functionally Graded Al-12Si (wt.%) alloy, produced using Centrifuge Casting Technique

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Research Article

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Abstract

The current research work discusses the concept of reinforcing alumina nanoparticulate to Al-12Si (wt.%) alloy using a unique casting technique known as centrifuge casting system established in-house at our advanced manufacturing centre. The resulting cast solid cylindrical specimen will be functionally graded along the length of the specimen containing higher percentage of primary silicon and alumina nanoparticulate at the top region than the bottom region, with a gradual transition in between the two regions. The microstructure of the functionally graded Al-12Si (wt.%) alloy, reinforced with 0.5, 1 and 1.5wt.% of alumina nanoparticulate was analysed using "Scanning Electron Microscope (SEM)" and the elemental composition of the cast specimens were obtained using "Energy-dispersive X-ray analysis (EDX)". The outcomes of the EDX analysis confirmed the presence of alumina nanoparticulate in the cast specimens. Further, the mechanical properties of the cast Al-12Si (wt.%) alloy, reinforced with alumina nanoparticulate were studied, and it was found that the specimen with the addition of alumina nanoparticulate showed enhanced mechanical properties when compared with the Al-12Si (wt.%) alloy, without alumina nanoparticulate addition, cast under identical conditions.

1. Introduction

"Functionally Graded Materials" (FGMs) are composites with two components, characterised by a compositional gradient from one component to another. FGMs have gained prominence during the last two decades because of the ability of the material to achieve a large gradation of physical and/or chemical properties in a single component [1] [2]. The microstructure of the FGMs show continuous change in the morphology, configuration and the properties can be tailored in a defined direction [3] [4].

Ghanaraja et al. [5] has worked on the different processing methods available for fabricating metal matrix composites reinforced with alumina nanoparticulate. They have studied numerous processing methods such as deformation processing, powder metallurgy and different solidification processing methods. Further, they have reported that, amongst the different processing techniques available, stircasting method has proved to be one of the economical techniques available for the synthesis of composites, where the particles are reinforced into molten alloy with the help of stirring and the obtained slurry is cast to get composite specimens.

According to Iman et al. [6], amongst all the metal matrix composites, aluminium-silicon alloy-based composites are increasingly being used due to their light weight, high specific stiffness, and outstanding wear resistance. Various researchers [7] [8] have reported that the as-cast properties of Al-Si alloys may be improved by reinforcing the matrix with nano-sized particulate. By reinforcing alumina (Al_2O_3) nanoparticulate to the Al-Si alloy, cast in the semi-solid state with the help of mechanical stirring, the strength and the hardness of these alloys may be enhanced. The authors have reported that, this observation may be due to the formation of smaller and equiaxed grain structure from the dendritic columnar structure, obtained from semi-solid processing conditions. Further, the semi-solid casting conditions results in increased viscosity and may improve the wettability of the ceramic particle and

physically arrest the reinforcement material. The alumina nanoparticulate possess suitable properties like relatively higher thermal conductivity and coefficient of thermal expansion that are compatible with the Al-Si alloy system.

Aluminum based Metal Matrix Composites (MMCs) are commonly used for several engineering applications. Aluminum oxide/Alumina (Al_2O_3) [9], Silicon Carbide (SiC) [10], Titanium Di-Boride (TiB₂) [11], Boron Carbide (B_4C) [12] and Titanium Carbide (TiC) [13] ceramic particles are commonly used reinforcements to aluminum and magnesium in particulate form to produce MMCs at a relatively reduced production cost with improved mechanical properties. Aluminum Metal Matrix Composites (AMMCs) may be tailor-made to attain high specific strength and stiffness, with good electrical and thermal conductivity [14], [15]. This has attracted many engineers and researchers to use AMMCs for numerous applications in aerospace and automotive segments [16].

The literature review shows minimal information regarding the studies on enhancing the mechanical properties of Al-12Si (wt.%) alloy-based nanocomposites cast using centrifuge casting technique and most of the research work carried out by earlier researchers' emphases on centrifugal casting and stir casting methods. In the current paper, centrifuge casting method has been used to produce alumina nanoparticulate reinforced Al-12Si (wt.%) alloy specimens. Further, the microstructural behaviour and mechanical properties of these specimens have been reported and discussed.

2. Experimental Procedure

The centrifuge casting machine used in the present research work is shown in Fig. 1.

During the process of centrifuge casting, the molten metal is emptied into the static mould and the machine arm turns around a vertical axis. The uniqueness of centrifuge casting machine is that the effect of centrifugal force is not applied instantly as in the case of conventional casting techniques since the mould takes a short duration to reach the casting speed [17]. Excellent mould filling and microstructural control may be achieved by using centrifuge casting technique, resulting in improved mechanical properties [18] [19].

A charge of 250gms of Al-12Si (wt.%) alloy was melted in a resistant furnace, heated to 800°C temperature and maintained at that temperature for 30 minutes. Further, upon obtaining the liquid state, degassing of the melt was carried out to remove the gases, using hexachloroethane degasser tablets [20]. Subsequently, the temperature of the melt was brought down to the semi-solid state (600–650°C) [21] and later, alumina nanoparticulate, preheated to 300°C, enclosed in small packets of aluminum foil was directly immersed in molten metal with continuous stirring for one minute, using powered stir casting equipment at a constant stirring speed of 1000 rpm. The melt containing Al-12Si (wt.%) alloy reinforced with alumina nanoparticulate was transferred into the metal mould suspended at one end of the arm and the arm was rotated at 300rpm [22]. For each sample, 250gms of material was cast to get a solid cylinder-shaped specimen of 40mm diameter and 50mm altitude and cast specimens with 0.5, 1 and

1.5wt.% addition of alumina nanoparticulate were prepared using centrifuge casting technique. Further, the cast specimens were observed at different regions using "SEM (TESCAN-VEGA3 LMU)" for microstructural studies and the specimens were also tested for ultimate tensile strength and hardness using "electronic tensometer" and "Brinell hardness testing machine (model BV-120, which confirms to IS Specification 1754)" respectively. Further, the specimen was cast with the addition of 2wt.% of alumina nanoparticulate and it was observed that the mechanical properties decreased when compared with the specimens containing 0.5, 1 and 1.5wt.% of alumina nanoparticulate addition, cast under identical conditions. The decrease in mechanical properties may be due to the presence of porosity, alumina nanoparticulate agglomeration and development of cavities [23] [24]. Hence, specimens with the addition of 0.5, 1 and 1.5wt.% of alumina nanoparticulate was selected for the study of microstructure and mechanical properties.

In the current research work, alumina nanoparticulate of average particle size 40nm has been used as particulate reinforcement and it has been obtained from "Nano Labs Pvt Ltd". LM6 grade aluminum alloy, procured from Fenfe Metallurgicals has been used in the present research work as base alloy and its chemical composition is given in Table 1.

Alumina nanoparticulate with average particle size of 40nm is used as reinforcement to LM6 aluminum alloy and the properties of alumina nanoparticulate are given in Table 2.

Table 1
Chemical composition of LM6 alloy

Elements	Si	Mg	Sn	Ti	Mn	Fe	Ni	Cu	Zn	Pb	Al
Percentage	12	0.1	0.05	0.2	0.5	0.6	0.1	0.1	0.1	0.1	Remaining

Table 2 – Properties of alumina nanoparticulate

Test item	Purity	Color	Particle size	Phase type	Surface specific area	Density
Composition	99.9%	White	30-50nm	Alpha	12-18m ² g ⁻¹	3.9gcm ⁻³

Figure 2(a) shows the TEM image of alumina nanoparticulate and Fig. 2(b) shows the TEM image of cast Al-12Si (wt.%) alloy with the addition of 1.5wt.% of alumina nanoparticulate. It may be seen from the figure that the alumina nanoparticulate are dispersed as tiny clusters and they seem to be approximately sphere-shaped. The average crystalline size of alumina nanoparticulate was found to be 40nm.

The pictorial representation and the actual specimen cut for metallographic examination is shown in Fig. 3. Metallographic studies were carried out on the sectioned surface, which was grinded initially and later polished using different abrasive papers. Further polishing was carried out using fine silicon carbide (SiC) powder and later with 15micron diamond paste to obtain a scratch free surface. The obtained specimens were etched using Kellers reagent to develop the microstructure [25]. SEM (TESCAN-VEGA3 LMU) was used to observe the microstructure of the cast specimens and later the tensile strength and

hardness were measured with the help of electronic tensometer and Brinell hardness testing machine respectively. A load of 15.625kgs was applied for 15 seconds during hardness testing. All the trials were performed on three alike specimens and the average test results are presented in the form of graphs.

3. Results And Discussion

The SEM microstructural images and the results of EDX analysis of cast Al-12Si (wt.%) alloy without addition of alumina nanoparticulate and the same alloy with addition of different contents of alumina nanoparticulate at the top region and bottom region of the specimen have been presented and discussed.

The SEM images of Al-12Si (wt.%) alloy at different regions without the addition of alumina nanoparticulate, cast using centrifuge casting technique are shown in Fig. 4.

From Fig. 4, it may be seen that the microstructural features of Functionally Graded (FG) cast Al-12Si (wt.%) alloy show a variation of structure from top region to bottom region of the casting with eutectic structure sandwiched between the hypoeutectic and hypereutectic structures. The microstructures show a discontinuous dispersal of needle-shaped silicon particles in the eutectic aluminum matrix at the bottom region of the casting. The top region of the casting shows needle shaped eutectic phase of silicon and large primary crystals of silicon, which indicates a high silicon hypereutectic microstructure. This may be due to the collective effects of rapid cooling and centrifugal action. Rapid cooling of the alloy at the bottom region suppresses nucleation of primary silicon and results in near hypoeutectic structure.

The SEM images of Al-12Si (wt.%) alloy at the top region and bottom region with 0.5, 1 and 1.5wt.% addition of alumina nanoparticulate are shown in Fig. 5(a), (b) and (c) respectively. The results of EDX analysis at the top region and bottom region with 1.5wt.% addition of alumina nanoparticulate are shown in Fig. 5(d).

3.1 Microstructural Analysis

It may be seen from the Figs. 5(a)-(c), that the microstructure of the Al-12%Si alloy comprises of three different phases i.e., primary silicon elements, α -Al in equiaxed arrangement and binary Al-Si eutectic structure. The structure of the primary silicon is found to be polygonal in shape, with tiny primary needle alike silicon particles. It may also be observed that the top region of the cast specimen shows higher presence of primary silicon as compared with the bottom region of the cast specimen. This observation has been found true for all the specimens with different content of alumina nanoparticulate addition.

The structure of the eutectic silicon may depend on growth conditions, whereas the structure of primary silicon may be controlled by enhancing the quantity of nuclei through enhancing the rate of nucleation. Alumina nanoparticulate act as nucleating agent and the reinforcement of alumina nanoparticulate increases the quantity of primary silicon particles during the process of solidification. This observation has been found true for all the specimens with different content of alumina nanoparticulate addition.

Further, Amirkhanlou S and Niroumand B [26] have reported that addition of nanoparticulate reinforcement with the assistance of stirring may help to achieve thermal consistency in the semi-solid slurry creating low gradients of temperature that may improve the nucleation barrier in the bulk liquid. In addition, the primary dendrites are split due to the process of mechanical stirring and the stirring process may also help in breakdown of the secondary and tertiary dendrite arms. This occurrence is the primary reason for obtaining equiaxed grain development in cast specimens.

From Fig. 5(d), it may be seen that at all the three different content of alumina nanoparticulate addition, the presence of primary Si and alumina nanoparticulate is confirmed in the Al-Si matrix and it is found to be distributed across all the three regions of the cast specimen. Further, it may be observed that the top region of the cast specimen shows an existence of higher weight percentage of primary Si and alumina nanoparticulate as compared to the other two regions of the cast specimen.

The existence of higher content of alumina nanoparticulate at the top region of the cast specimen may be attributed to two factors namely (i) relatively higher rate of solidification caused by centrifuging action of the mold, resulting in limited time available for the movement of nanoparticulate in the casting and (ii) inherent nature of the particulates which are nano in size resulting in posing difficulty for dispersion.

3.2 Tensile properties

The tensile test results of cast Al-12Si (wt.%) alloy without alumina nanoparticulate addition and cast Al-12Si (wt.%) alloy with different content of alumina nanoparticulate addition has been shown in Fig. 6(a)-(c). The tensile test specimen measurements are shown in Fig. 6(d).

From Fig. 6(a), an increase in the values of yield strength and tensile strength may be observed with increase in the addition of alumina nanoparticulate. Further, by increasing the addition of alumina nanoparticulate beyond 1.5wt.%, a decrease in the values of tensile strength and yield strength may be observed. From Fig. 6(b), a decrease in the value of ductility may be observed with increase in the addition of alumina nanoparticulate and this observation may be owing to void creation during tensile elongation and assorted dissemination of reinforced particulate [24]. The relationship between stress and strain for the cast Al-12Si (wt.%) alloy with different contents of alumina nanoparticulate addition has been shown in Fig. 6(c).

Further, it may be seen from Fig. 6(a), that the tensile strength of cast Al-12Si (wt.%) alloy at the top region of the cast specimen is 151.8MPa and the tensile strength of Al-12Si (wt.%) alloy at the top region of the cast specimen with 1.5wt.% of alumina nanoparticulate addition is 212.7MPa. A 40% improvement in the value of tensile strength may be observed for the specimen with 1.5wt.% of alumina nanoparticulate addition as compared to the cast Al-12Si (wt.%) alloy specimen without nanoparticulate addition.

The observed increment in tensile strength due to alumina nanoparticulate addition may be owing to (i) Orowan strengthening or dispersion hardening [27], (ii) the combined effect of rise in grain boundary area resulting from grain refinement [28], (iii) the robust thermal stress at the Al-12Si (wt.%)/Al₂O₃ boundary

due to the difference in coefficient of thermal expansion between the reinforcement and the matrix and (iv) the actual transfer of tensile load to the huge quantity of closely bonded solid nano-alumina particulate [29].

3.3 Hardness

The "Brinell Hardness Number" (BHN) of cast Al-12Si (wt.%) alloy and Al-12Si (wt.%) alloy

with different contents of alumina nanoparticulate addition, at the top region and bottom region has been presented in Fig. 7.

From Fig. 7, it may be observed that the Al-12Si (wt.%) alloy specimens with different contents of alumina nanoparticulate addition showed increased hardness, both at the top region and bottom region, as compared to the Al-12Si (wt.%) alloy specimen without any alumina nanoparticulate addition. It may also be observed that for all the three different contents of alumina nanoparticulate addition, there is a marginal increase in the value of hardness at the top region of the cast specimen with reference to the bottom region of the cast specimen.

Further, the specimen with 1.5wt.% of alumina nanoparticulate addition showed an increase of 30.76% at the top region and 21.1% at the bottom region, as compared to the Al-12Si (wt.%) alloy specimen without any alumina nanoparticulate addition. This observed increase in the hardness value may be due to the existence of higher content of primary silicon and alumina nanoparticulate at the top region as compared to the bottom region of the cast specimen (confirmed by EDX analysis), collective effects of various strengthening mechanisms and stirring process employed during casting [30].

The strengthening mechanism for metal matrix nanocomposites (MMNCs) may also be due to the presence of higher dislocation density in MMNCs developed as a result of the difference in the coefficient of thermal expansion between the matrix and the alumina nanoparticulate reinforcement [31]. Further, the alumina nano dispersions may restrict the movement of dislocations. This may also be the contributing factor for the observed increase in the mechanical properties of MMNCs [32].

4. Conclusion

- Nanoparticulate reinforced functionally graded Al-12Si (wt.%) alloy composites can be successfully produced using centrifuge casting technique.
- The improvement in tensile strength observed in the present research work may be due to the nanosized particle reinforcement, transfer of tensile load to alumina nanoparticulate and grain size refinement of aluminum matrix.
- The hardness of the nanoparticulate reinforced Al-12Si (wt.%) alloy composites may increase with the particulate content in the matrix. Further, the observed higher hardness may be owing to the restriction of the dislocation motion, caused by the reinforcement of alumina nanoparticulate.

• The Al-12Si (wt.%) alloy composite with 1.5wt% of alumina nanoparticulate addition, cast at 600°C (semi solid-state temperature) using mechanical stirring at 1000rpm provided the best mechanical properties within the scope of the investigation.

Declarations

Ethics approval and consent to participate – Informed consent was obtained from all individual participants included in the study.

Consent for publication - The participants have consented to the submission of the research work to the journal.

Availability of data and materials – Data sharing not applicable to this article as no datasets were generated or analysed during the current study.

Competing interests - The authors have no conflicts of interest to declare that are relevant to the content of this article.

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Authors' contributions - All authors contributed to the study conception and design. Material preparation, data collection and analysis were performed by Dr Chethan K S, Dr Kiran Aithal S, Dr Ramesh Babu N, Mr Manjunath H N and Mr Pavan K N. The first draft of the manuscript was written by Dr Chethan K S and all authors commented on previous versions of the manuscript. All authors read and approved the final manuscript.

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Disclosure of potential conflicts of interest

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All authors certify that they have no affiliations with or involvement in any organization or entity with any financial interest or non-financial interest in the subject matter or materials discussed in this manuscript.

The authors have no financial or proprietary interests in any material discussed in this article.

Research involving Human Participants and/or Animals - Not applicable

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Figures

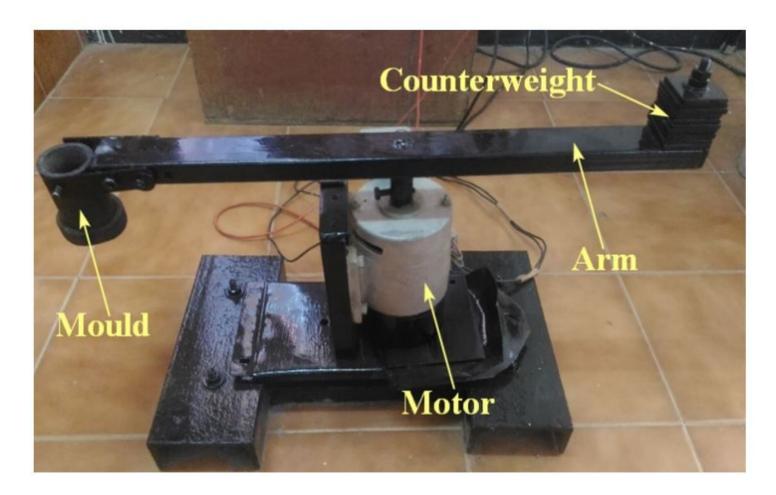
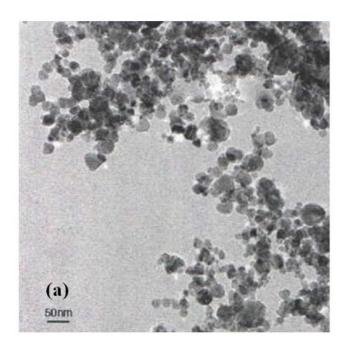


Figure 1

Centrifuge casting machine



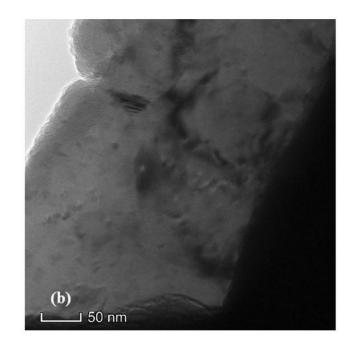
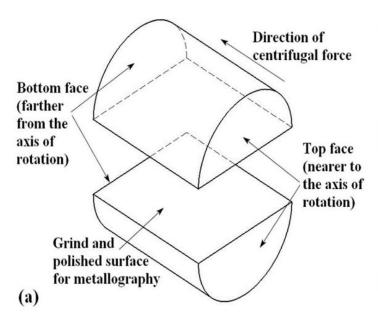


Figure 2

TEM image of (a) alumina nanoparticulate and (b) Al-12Si (wt.%) alloy with the addition of 1.5wt.% of alumina nanoparticulate.



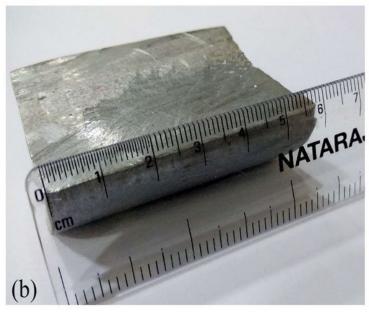


Figure 3

Specimen cut for metallographic examination, (a) Pictorial depiction and (b) Actual specimen.

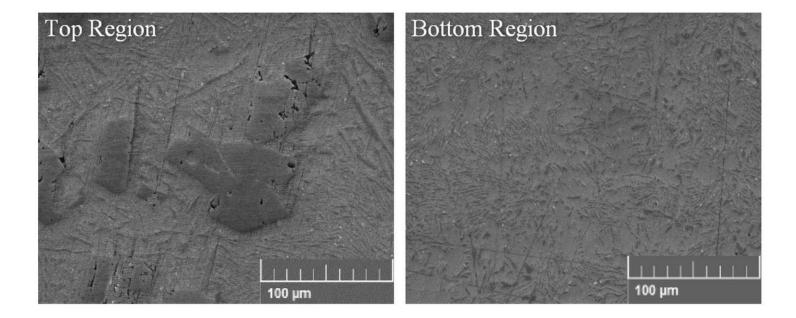


Figure 4

SEM image of Al-12Si (wt.%) alloy at the top region and bottom region, without addition of alumina nanoparticulate.

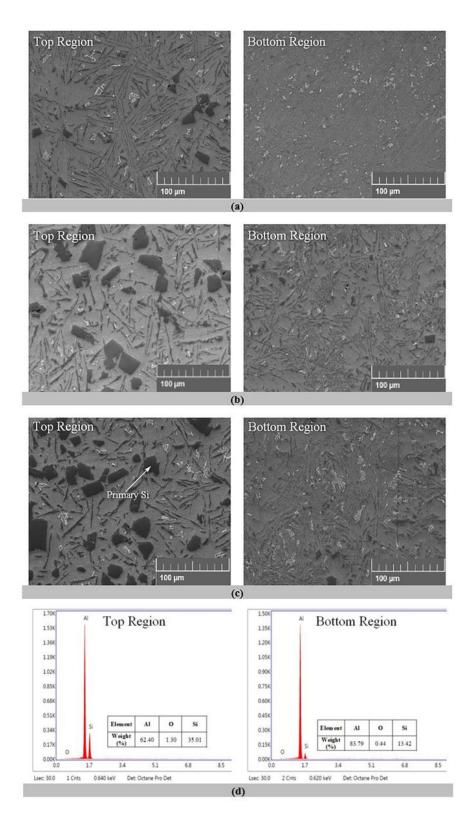


Figure 5

SEM images of Al-12Si (wt.%) alloy at the top region and bottom region with (a) 0.5wt.% addition of alumina nanoparticulate (b) 1wt.% addition of alumina nanoparticulate (c) 1.5wt.% addition of alumina nanoparticulate and (d) EDX results of Al-12Si (wt.%) alloy with 1.5wt.% addition of alumina nanoparticulate.

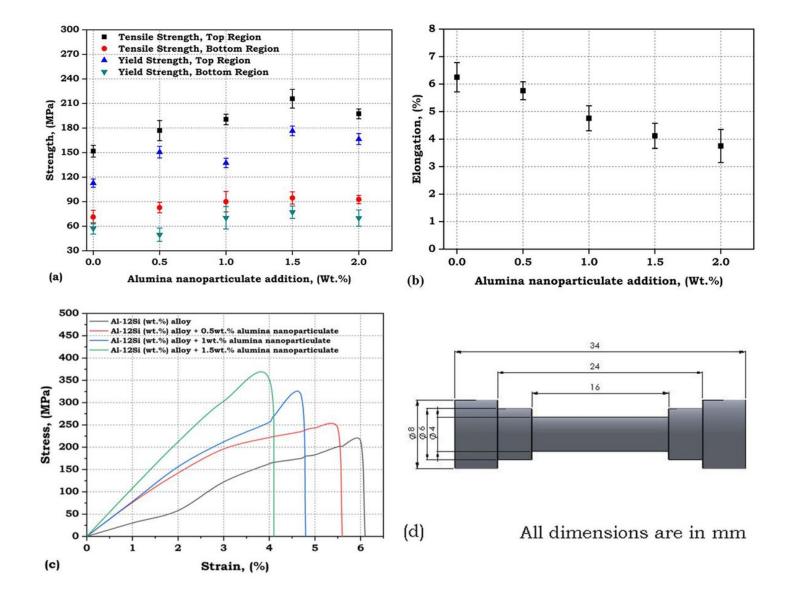
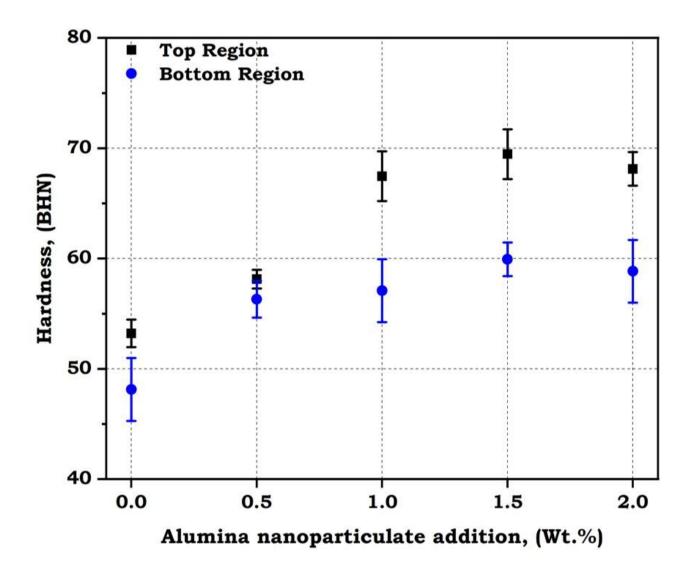


Figure 6

Tensile characteristics of cast Al-12Si (wt.%) alloy with different contents of alumina nanoparticulate addition (a) variation of yield strength and tensile strength (b) elongation (c) stress-strain curve (d) tensile test specimen details.



Hardness (BHN) of cast Al-12Si (wt.%) alloy with different contents of alumina nanoparticulate addition, at the top region and bottom region.

Figure 7