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# Effect on microstructures, hardness and SDAS of primary Mg$_2$Si/Al-Si eutectic phases of centrifugally cast functionally graded Al-(Mg$_2$Si)p in-situ composites

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# Effect on microstructures, hardness and SDAS of primary Mg₂Si/Al-Si eutectic phases of centrifugally cast functionally graded Al-(Mg₂Si)p in-situ composites

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**Abstract**. In the present study, the centrifugal casting route was employed to synthesize the Al-(Mg₂Si)p functionally graded in-situ composites at 1200 rpm rotating speed of mold. The formation of primary Mg₂Si particles/Al-Si eutectic phases by the in-situ chemical reaction has been investigated. In the Al-Mg-Si alloys system, in-situ formed Mg₂Si particles are lately introduced a new aspect, because of due to lighter density (1.99 gm/cm$^3$) than Al-matrix (2.27 gm/cm$^3$) the Mg₂Si particles are more capable of migrating into inner zone of cast tubes due to centrifugal force. The X-Ray diffraction (XRD) technique was carried out to confirm the presence of in-situ formed Mg₂Si/Si particles in the cast composites. Optical microscopy analysis was carried out to reveals distribution, size and volume fraction of Mg₂Si particles of cast cylindrical tubes. Scanning electron microscopy (SEM) analysis was also carried out to reveal the morphological characteristics and some intermetallics (π & β phases) present into the cast FGM tubes. The Vickers hardness test was carried out along the radial direction of tubes. Resultant, the hardness value is remarkably improved by 20% to 30% at the inner zone due to high volume percent of reinforced primary Mg₂Si hard particles. The objective of this investigation was to observe the effect on microstructures, hardness and secondary dendrites arm spacing (SDAS) of Mg₂Si particles/Al-Si eutectic phases of fabricated FGMs tube and analyzed their gradient properties.

**Key words**: Mg₂Si and Al-Si eutectic phase, FGMs, SDAS, Hardness, and Microstructures.

## 1. Introduction

Functionally graded materials can be considered as new class of advanced composite materials. Recently, the various researchers have been developed the composite structures depending on the design prospects and requirements [1]. Now days, the Al-Si-Mg casting alloys are increasingly being used in the fabrication of engine components in the automotive industry as an alternative material for cast iron. The in-situ Al-Mg₂Si composites have great potential applications in the automotive and aerospace industries due to their excellent properties, e.g., high hardness, low thermal expansion coefficient; low density, high elastic modulus, high strength and superior wear resistance properties.

Functionally graded materials (FGM) are of practical interest because of the gradation of physical, mechanical and chemical properties can be achieved across volume depending on design.

In Al-base alloys the several reinforcing phases e.g.$Al_2O_3$, $TiB_2$, SiC, $Al_3Ti$, etc. can be used but the segregation of Si and $Mg_2Si$ primary crystal particles seems to be of great interest in FGMs. Moreover, functionally graded materials (FGM) are a group of advanced materials in which composition and microstructures, as well as specific properties, vary continuously along the thickness. Thus, in such types of materials the reasonable features such as mechanical, physical and wear resistance properties can be achieved. Besides that, in FGMs, the thermal stability may also obtain, so a wide range of engineering applications especially in automotive engine components can be used where thermal fluctuation may cause damage the cylinder liners and pistons [2-6]. There are several fabrication techniques of in-situ FGMs composite include control mold, chemical vapor deposition(CVD), pressure vapor deposition(PVD), the plasma spray technique, and powder metallurgy techniques can be used, but centrifugal casting method is more economical for fabrication of cylindrical components(engine block liners). However, among these fabrication processes, centrifugal castings have been lately introduced a new concept to produce the in-situ metal matrix composites (PMCs) tubes with gradient properties [7-8].

In Al-base alloys the several reinforcing phases e.g.$Al_2O_3$, $TiB_2$, SiC, and $Al_3Ti$, etc. can be used but due to light density (1.99 g/cm$^3$) and high strength of $Mg_2Si$ primary particles seems to be of great interest in FGMs. Moreover, most of the authors are fabricated the PMMCs with several particulate reinforcements have larger densities (SiC, $TiB_2$, etc.) than the Al matrix. Thus, with few PMMC systems, the reinforcements having lighter densities may be used with a concentration towards the inside area of the tube through centrifugal casting [3, 9]. In general, as a major reinforcing phase (e.g., SiC, $TiB_2$, $ZrB_2$, $Mg_2Si$ and other ceramic phases), to the reduction in weight, the $Mg_2Si$ is the most effective reinforcing phase for engine block applications. In situ formed $Mg_2Si$ phase in the matrix are the new aspect, because of it is more capable of migrating into inner surfaces of cylindrical cast tubes due to their lighter density (1.99 gm/cm$^3$) than other phases and Al-matrix (2.27 gm/cm$^3$) using centrifugal casting. Furthermore, there is very limited information about the centrifugal casting for producing the FGMs, especially as in-situ composites, by centrifugal casting. The present work especially shows a practical way to fabricate an FGM with $Mg_2Si$ reinforcements. Centrifugal casting can be used to fabricate both (i) reinforced towards inside and (ii) reinforced towards the outside of the tube specimen depending on the densities of reinforcements. Farahany et al. [10] have investigated some intermetallics formation during solidification of Al- 13 Mg-7Si-2Cu in-situ composite by gravity casting. They have also reported, the formation of primary $Mg_2Si$ begins during solidification followed by eutectic Al- $Mg_2Si$, $Al_5FeSi$ and simultaneous precipitation of $Al_5Cu_2Mg_8Si_6$ and $Al_2Cu$ complex intermetallic phases. Shabastari et al. [11] studied the effect on the morphological changes in $Mg_2Si$ particles of Al- 25%$Mg_2Si$ composites processed by slope casting and semi-solid techniques. They have also observed the particle size refinement due to increased nucleation rate and fragmentation of particles by fluid shearing stress in slope casting. The spheroidization of α-Al and $Mg_2Si$ particles were observed in semi-solid processing.

E.Georgatis, et al. [12] have adopted the modified investment casting technique and studied the effect of microstructure, heat treatment and consequent mechanical properties of Al- 11% $Mg_2Si$-Si in-situ composite. They have been found that fine dendritic arm spacing to improve the aging performance.

However, the effect of particles distribution into the core with varying processing conditions, and compositions are not reported yet, which are very crucial for automotive engine blocks and liners.

In the present investigation, a stringent work is made to correlate secondary dendrites arm spacing (SDAS) and its effect on microstructures, hardness properties and grain refinements mechanism of fabricated Al-Mg$_2$Si in-situ composites by centrifugal casting. Generally, at the inner surface of cylinder liners of an automotive engine is required more strength and wear resistance properties. So in this context, the hard Mg$_2$Si particles with light density than matrix have more capable of migrating into the inner region of tubes due to centrifugal force. However, due to segregation of Mg$_2$Si particles along the radial direction of the components are achieved the gradient properties and have the great potential for automotive engine blocks and liners.

## 2. Experimental procedure

### 2.1 Materials and method

Industrially pure commercial A356 (Al-0.3Mg-7.3Si) alloy was used as matrix materials to produce the Al-Mg$_2$Sip functionally graded in-situ composites. The required amount of Mg-turnings about 20 % of additional pure Mg turning (wrapped in the Al foil) was added to the molten matrix materials to fabricate the master alloys ingots. Here, the 20%Mg turning was used to recover the melting loss of Mg. In this work, centrifugal casting technique has been adopted for manufacturing of axi-symmetric cylindrical tubes with the gradient structure. At first, three ingots of A356+0.30%Mg, A356+10%Mg, and A356+20%Mg master alloy were prepared with the addition of pure Mg tunings in required proportions. After that, the pure A356 ingots along with master alloy were melted at 800°C in the electrical resistance furnace with MgCl$_2$.6H$_2$O and KCl salts as a flux. By the in-situ reaction, the Mg$_2$Si particles are formed in the molten alloy, and the molten composite was then transferred to a preheated stir casting melting furnace attached to vertical centrifugal casting machine (Figure 1) and was stirred for 10 min with a graphite stirrer for homogeneous mixing.
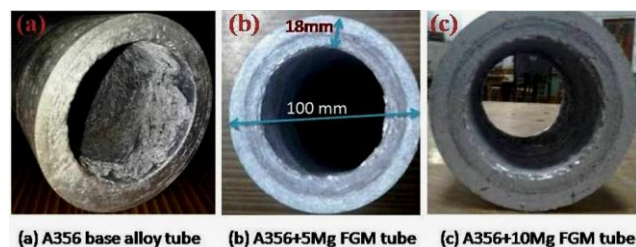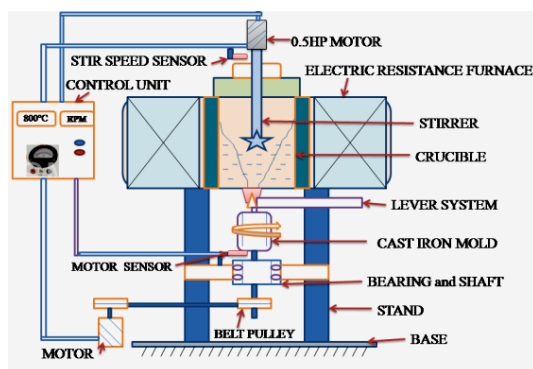


**Figure 1.** Centrifugal casting machine Set-Up.        **Figure 2.** Centrifugally cast alloy and FGM tubes.

The melt was then poured the bottom into a rotating cast iron mold at a constant rotational speed of 1200 rpm. The mold was preheated to 250°C before pouring. The composite is poured into rotating cast iron mold at 1200 rpm. Finally, the three cylindrical casting tubes were made as (a) A356 base alloy (b) A356+Mg (c) A356+10Mg with gradient properties. The tubes of dimensions 100 mm in the outer diameter, 18 mm in thickness and 150 mm in length were centrifugally cast as shown in Figure 2. The chemical composition of the base alloy and composites (Table 1) were analyzed with an Optical Emission Spectrometer (Foundry Master).
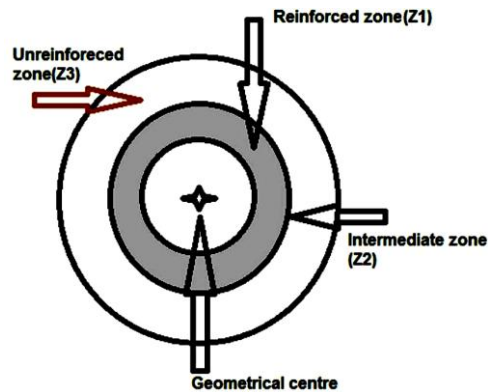
**Figure.3** Cross-section of cast tube showing different zone: inner ($z_1$), middle ($z_2$) and outer ($z_3$).

For the identification of $Mg_2Si/Si$, and α-Al phases in the casting, the XRD analysis was carried out using CuKα radiation of wavelength 1.541836Å with Ni filter. Optical micrographs were taken to reveals the microstructures of the centrifugally cast A356 alloy and of FGMs, in the different zones at inner ($z_1$), intermediate ($z_2$) and outer ($z_3$). The hardness of the base alloy and composites was estimated by Vickers Hardness Tester (Leica) at 5 Kgf loads along three zone $z_1$, $z_2$ and $z_3$ for each composite as shown in Figure 3.

**Table.1** Chemical composition of as-cast base alloy and FGMs

| Alloy and composites | Si | Mg | Cu | Fe | Al |
|---|---|---|---|---|---|
| **A356 Alloy** | 6.50 | 0.30 | 0.60 | 1.40 | bal. |
| **A356+5.0Mg composite** | 5.48 | 5.13 | 0.63 | 0.81 | bal. |
| **A356+10.0Mg Composite** | 5.54 | 10.50 | 0.36 | 0.44 | bal. |

2.2 Determination of secondary dendrite arm spacing (SDAS) and solidification time

The image analyzer was used to evaluate the SDAS by identifying and measuring aligned groups of each dendrite cells on the screen. The SDAS is then calculated using equation as SDAS=L/nM, where L is the length of the line, M is the magnification, and n is the number of dendrite cells. The solidification times ($t_E$) have been calculated after knowing the SDAS from the given equation as follows:

$$t_E = \{SDAS/A\}^3$$

Where 'A' is a material constant, for the type Al- Si7-Mg aluminum alloy, the value of 'A' as 11.7 [13].

## 3. Results and Discussion

*3.1 Microstructural and Hardness analysis*

The microstructures of A356 base alloy, 356-5Mg and A356+10Mg FGMs tube composite using the vertical centrifugal machine is shown in Figures 4-6(a), (b) and (c) at zone $z_1$, $z_2$ and $z_3$ respectively. Graded distribution of particles have seen in A356+5Mg and A356+10Mg FGMs tubes at three different locations from the inner($z_1$) (a), middle($z_2$) (b) and (c) periphery($z_3$) of 18 mm thick ring of 100 outer diameters casting as Figure.5-6. In contrast, for A356 alloy eutectic Al-Si, α-Al dendrites cells and grains structures have been observed refer Figure.4 (a-c). However, in FGM the outer periphery of the casting showed Figure. 5(c) has the low concentration of $Mg_2Sip$ than the interior of the casting with a sharp interface separating the particle-rich and particle free zone and the inner area

with the higher concentration of particles Figure. 5(a). Similarly, as Figure.6 a-c shows the particles segregation from inner to periphery of cast tube for A356+10Mg FGMs.
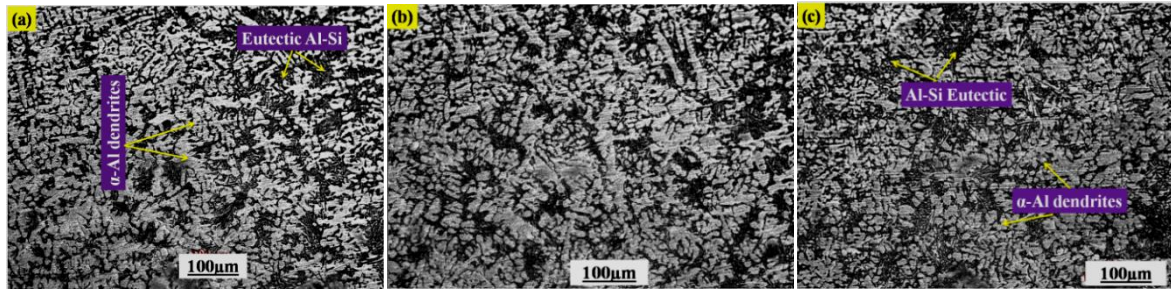


**Figure.4**: (a-c) Microstructures of A356 base alloy tube at (a) inner zone $z_1$, (b) middle zone $z_2$ and (c) outer zone $z_3$ respectively.
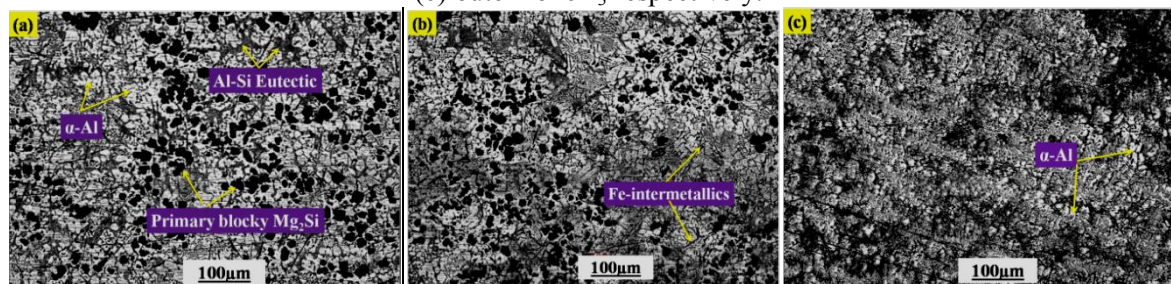


**Figure 5:** (a-c) Microstructures of A356+5Mg FGM tube at (a) inner zone $z_1$, (b) middle zone $z_2$ and (c) outer zone $z_3$ respectively.
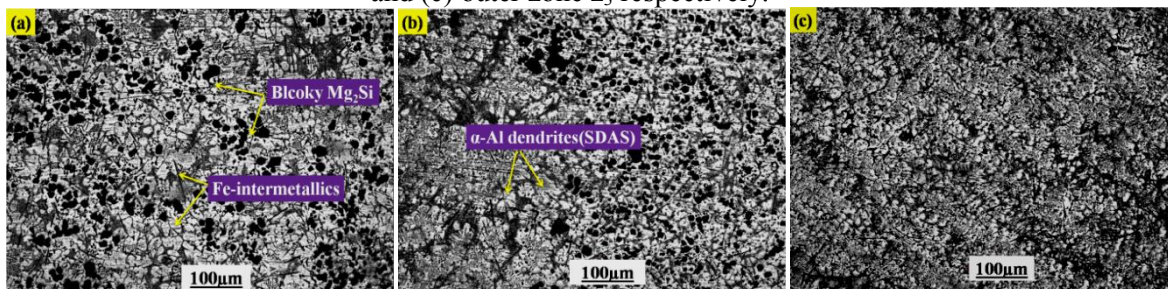


**Figure. 6**: (a-c) Microstructures of A356+10Mg FGM tube at (a) inner zone $z_1$, (b) middle zone $z_2$ and (c) outer zone $z_3$ respectively.

The segregation of particles was seen because of action of centrifugal force and depending on the density of particles. The lighter dense particles are moving toward inner zone and Al-grains (high density) toward the periphery due to centrifugal forces. The size distribution and volume percentages of $Mg_2Sip$ evaluated using the image-J analyzer software shows (Figure 8. a-c) for A356+5Mg FGM and A356+10Mg FGMs. The outer periphery of the cylinder contained less vol. % $Mg_2Sip$ gradually increases towards the core of tubes respectively. The $Mg_2Si$ particles (blocky shape), Al-Si eutectic phase and intermetallic phase (Needle like shape) have been observed in the microstructural examination Figures5-6(a& b). Intermetallics phases were not detected in XRD patterns as shown in Figures 7 because of very low vol.% of the π & β phases were observed under SEM analysis.

The Vickers hardness values evaluated along the radial directions of the composite FGM tubes fabricated are shown in Figure 10. The same trends were observed in each alloy and composites; the maximum hardness at the inner zone and gradually decreasing, then increasing to some extent near the outer zone. In the base alloy, the graphs show the reverse trend, the value of hardness increasing towards the outer zone due to fast chilling effect and finer SDAS. Although, in inner areas, the SDAS

are coarser which lower the hardness due to the high volume fraction of $Mg_2Si$ particles hardness values are increases. These effects on hardness could explain and possible by observing the SDAS and solidification time as shown in Figure 9. However, in the base alloy at inner zone hardness values are decreases due to coarser SDAS but towards outer periphery the hardness of base alloy increases due to finer SDAS.

*3.2 XRD Analysis and phase distribution observation*

The XRD analysis was carried out to confirm the phases formed in alloy and FGM composites as shown in Figure 7. So clearly we can see the peak intensity of $Mg_2Si$ particles and eutectic Si as well as α-Al at 2θ angles. When Magnesium amount increases the peak intensity was higher in case of A356+10Mg FGM and lower with A356+5Mg FGM composites. The microstructure of centrifugal cast A356+10%Mg tube Figure 6 (a)-(c)) showed the very interesting distribution of the particles. A small portion of $Mg_2Sip$ is segregated in the core $z_1$ of the tube and gradually decreases toward the periphery ($z_3$) along which shows the gradient distribution of particles. Due to gradient distribution of particles along the thickness of the tube, the hardness values increases and gradually varies along the thickness this may depend on coarsening and de-coarsening of secondary dendrite arm spacing.
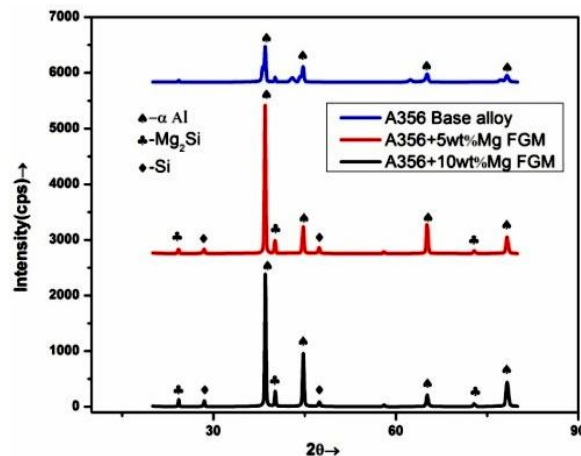


**Figure.7** XRD analysis graphs of base alloy and FGMs showing the phases formed.

Chirita et al.[14] have found the same trend and valid with this observation in the case of composites, they have also explained due to rapid chilling on the surface causes some primary $Mg_2Si$ particles are entrapped in the surfaces of tubes and due to finer SDAS the hardness increase near the outer zone. These increments in hardness values are more prominent in A356+5Mg and A356+10Mg FGMs. During the solidification processes, most of the primaries $Mg_2Si$ are formed, and the majority of these particles are moved to the inner zone due to centrifugal force. After that, at the time of semi-solid state the particles movement become slow by centrifugal force.

For evidence, refer to Figure 8 due to the high volume fraction of blocky $Mg_2Si$ particles are showed maxima hardness values around 125-130HV in case of A356+10Mg FGM than A356+5Mg FGMs and gradually decreased to some extent toward periphery as shown in Figure 10.But in case of base alloy due to the absence of $Mg_2Si$ particles, Vickers hardness values are minimum at inner 80 HV and gradually increases toward periphery around 90 HV to 100 HV due the finer dendrites cell. These finer dendrites cells are formed due to rapid chilling effect near the mold wall.
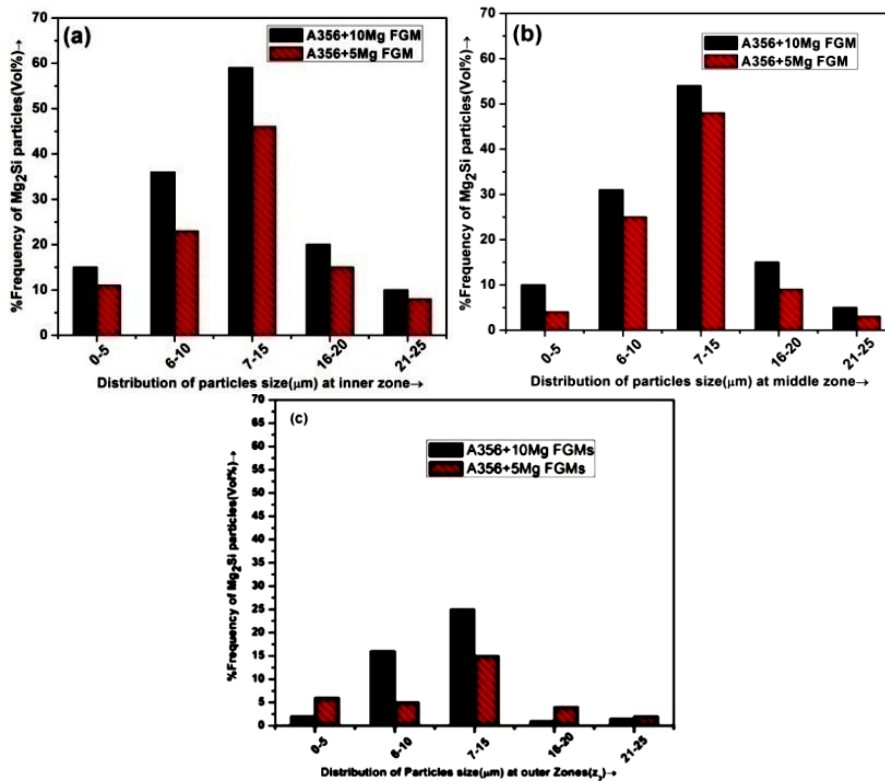
**Figure.8**. %Frequency and size distribution of Mg$_2$Si particles in three zones of A356+5 %Mg and A356+10Mg FGM composite at (a) inner (z$_1$), (b) middle (z$_2$) and (c) outer(z$_3$) zones.
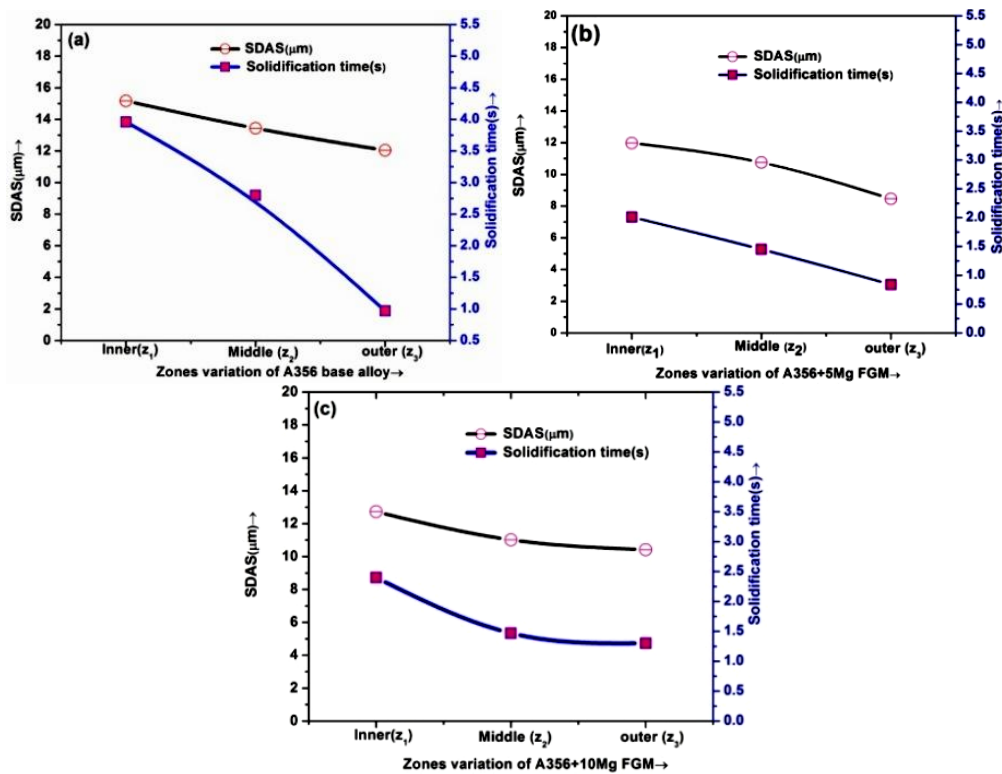


**Figure.9**. SDAS Vs Solidification time graphs for different zones of base alloy and FG composites (a) A356 alloy (b) A356+5%Mg and (c) A356+10%Mg respectively.
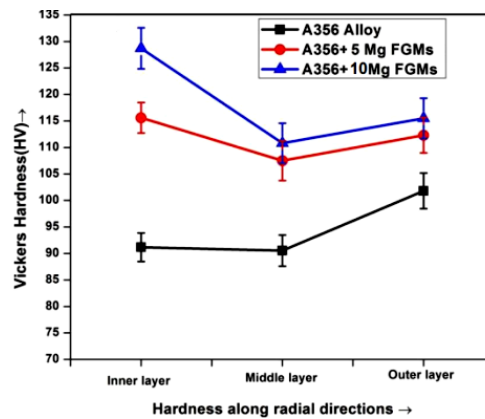
**Figure.10.** Vickers hardness graphs for different zones of base alloy and FG composites; A356 alloy, A356+5%Mg and A356+10%Mg respectively.

SEM micrographs as shown in Figurev11 for FG-composites of A356+5Mg and A356+10Mg shows the primary $Mg_2Si$ particles, and other phases form during solidification like α-Al, β & π phases and Chinese script like morphologies. These constituents of intermetallic phases, the strength and SDAS affected and finally, the microstructures and graded behavior of composites have changed. Sometimes these phases acted as the obstacle to dislocation movements and enhanced the mechanical strength. Thus, the Al-$Mg_2Si$ functionally graded composite cylinders liner prepared for automotive engine block replacing the existing cast iron liners have been successfully agreed to the benefit of weight saving as well as high hardness and strength. By using the Al-$Mg_2Si$ in-situ functionally graded composite provide the significant saving in the weight and new aspect in case of automotive industries is possible. The effect of different Mg content on the in-situ A356- $Mg_2Si$ composite functionally graded material has been synthesized by in-situ centrifugal casting.
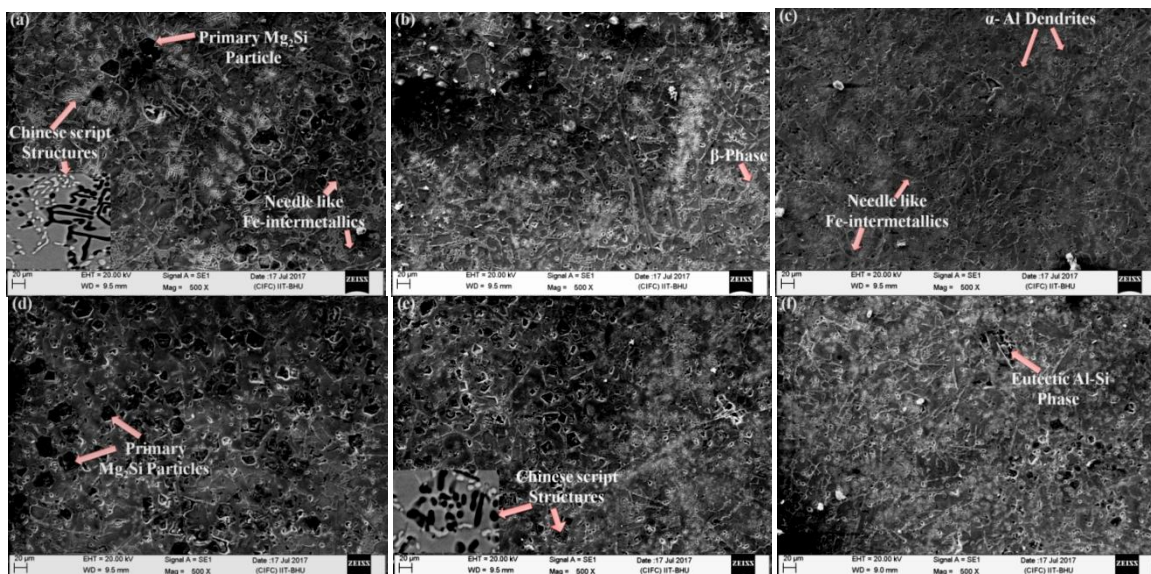


**Figure 11.** SEM graphs for different zones o f FG composites ;(a),(b) &(c) for A356+5%Mg and (d),e) &(f) for A356+10%Mg respectively.

## 4. Conclusions

The effect of solidification time and SDAS along the radial direction of centrifugally cast tubes on microstructure and hardness properties of Al-Mg$_2$Si in-situ composite was investigated. From the observation of results, the following conclusions can be made:

The microstructure of centrifugally cast Al-Mg$_2$Si FGM was analyzed and found that, the most of the particles are migrated into inner zone of tubes and some of the Mg$_2$Si particles are stick to the surface and middle zone which formed the gradient structure of casting. The Mg$_2$Si volume fraction and size were also evaluated from the surface to core of FGMs. In both A356+5Mg and A356+10Mg FGM tubes, the volume fraction was maximum in the inner zone about 47 to 57% and size range about 20-30 μm was observed. In the SEM analysis, the Mg$_2$Si particles (blocky shape), Al-Si eutectic phase and Fe-intermetallics phase (Needle like shape) and some π & β phases were observed which affected the gradient properties of the composites. Intermetallic phases were not detected in XRD patterns because of very low vol.% of the are present. As expected, the hardness is maximum in the inner layer of cast tubes which varies from 110 HV to 120 HV with 5wt.% Mg alloys. When Mg content is increased to 10 Wt% Mg then hardness value reaches up to 130 HV in the inner core of the cast FGM tubes. Hence hardness of in-situ FGMs increased by 25% to 30% as compared to base alloys.

Apart from these, the hardness values have correlated with secondary dendrites arm spacing (SDAS) cells from inner to the outer periphery of cast base alloy and FGM tubes. Resultant, because of rapid chilling effect to the surface of tubes, the finer SDAS was observed hence the hardness of base alloy increases at the outer zone but at intermediate layer, hardness was decreased due to coarser dendrites cells. Although, in the case of composites coarse dendrite can not affect the hardness values due to the presence of hard Mg$_2$Si particles. Hence, the hardness will increase at the inner zone and gradually decreases towards surfaces. Further in respect of scope and applications, such types of functionally graded materials are most capable in the casting industries especially in automotive manufacturing sectors for engine blocks and liners.

**Highlights:**

- The Al-Mg$_2$Si in-situ composites were developed by centrifugal casting method.
- Secondary dendrites arm spacing (SDAS) effect was correlated with solidification time.
- In the case of A356 base alloy casting, the hardness values are affected due to coarser dendrites cells into inner zone of tubes.
- In the case of FGM tubes, the SDAS effect has seen on the size and volume fraction of Mg$_2$Si particles.

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