Synthesis and Characterization of Al & SiCp Nano Particles by Non-contact Ultrasonic Assisted Method

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Abstract: The present study deals with proper mixing of SiCp nano particle in the aluminum metal matrix in two stages of processing i.e. primary and secondary. During primary processing, the breaking of agglomeration of nano particles take place and these are mixed with liquid aluminum powder using high frequency(35kHz) mechanical vibration. But, during secondary processing, mixing of nano particles along with subsequent cooling take place using high frequency non contact ultrasonic method. The study also reveals that in the liquid metal nano particle were uniformly dispersed and the segregation of the particles near the grain boundaries is due to pushing of the nano particle during grain growth. The study was performed by taking aluminum as matrix and SiCp as reinforcement with weight fraction of 2% and 3% and SiCp particles sizes of 30nm each. Scanning electron microscopy(SEM) and X-ray diffraction(XRD) were conducted for characterization of nano composite material.
Key words: non-contact method, MMNC, SEM, XRD, reinforcement, dispersed

INTRODUCTION

There are different methods have been used for synthesis of aluminum based metal matrix nano composite and several fabrication techniques have been used in recent years to manufacture the nano composite material with specific properties [1-3] including mechanical alloying, vertex process and spray deposition, stir casting, equal channel angular pressing, in situ processing [4-25] etc. Contact & non-contact ultrasonic assisted methods have been adopted for the dispersion of nano ceramic particles in the liquid aluminum and its alloys during the fabrication of metal matrix nano composites (MMNC). But due to improper distribution of nano particles in the metal matrix, solidification process advances which is relatively a cheapest route. Dispersion of the nano particles in the liquid media will require large amount of force to break the bonds in between the particles and the conventional methods are used to cast MMNCs will not be sufficient to deagglomerate the nano particles. Also, when the nano powder is added into the aluminum molten metal, then the viscosity of the nano composite increases significantly. This hinders the mixing process. The lower the particle size, the higher will be the viscosity of the melting and the higher is the volume fraction of particulate, the higher will be the viscosity[25]. Therefore, it is very difficult to uniformly

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disperse nano particles in metal matrix. Yang et al & Padhi et al [25-32] overcame the problem of agglomeration by using ultrasonic waves.

Materials Required

Pure Al- Aluminum is a low density metal which is more advantageous in many applications due to high stiffness to weight ratio. Aluminum has melting point 660°c, density 2.7 gm/cm³ & hardness in Rockwell is 24HRB and its composition in weight percentage are Fe - 0.96, Mg - 0.43, Si - 0.26 and Al - 98.35.

Silicon Carbide- Silicon Carbide has excellent properties such as abrasive, low density, high strength, low thermal expansion, high thermal conductivity, high hardness, high elastic modulus etc.

Experimental Procedure

In this system SiCp was supplied through the feeder with vibrating sieving system in Fig.1. At the same time, the molten metal aluminum was poured through the pipe. Then vibrator starts and it breaks the nano particles and mix with the aluminum molten metal. The vibration was carried out for a period of five minute to ensure complete mixing and subsequent solidification in the mold. The castings were cut in both longitudinal and transverse section for micro structural evaluation.

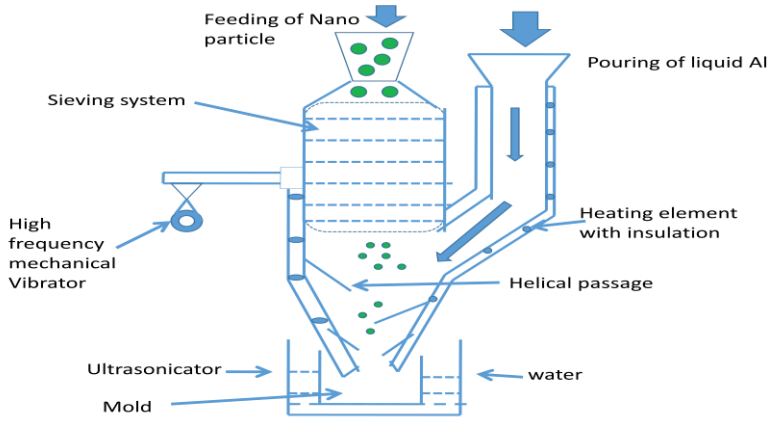

FIGURE 1. Schematic diagram of experimental set up

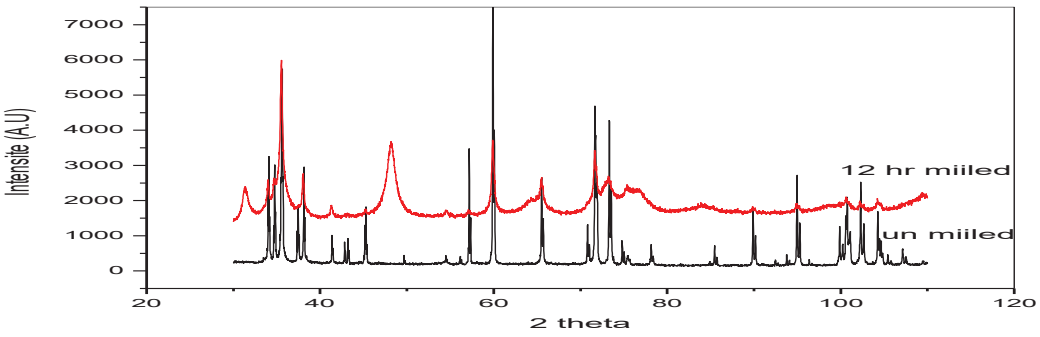

FIGURE 2. X-Ray Diffraction of both milled (12 hrs) and unmilled SiCp powders

Here SiCp nano-particles were used as reinforcements. These were prepared by ball milling. SiCp powder was milled for 12 hour. The nano powders were characterized using high resolution X-Ray Diffractometer (PHILLIPS,

X-PERT-PRO) and High Resolution Transmission Electron Microscope (JEOL, JEM-2100). Figure 3 shows the X-Ray diffract grams of milled and un-milled SiCp powders. Based on the Scherrer's Equation [33] the X-Ray diffraction were analyzed for estimating the crystallite size of SiCp Using this method the estimated crystallite size for SiCp was ~ 12.3 nm. Figure 4 show the HRTEM photograph of SiCp powders. It can be seen from Figure .2 that the particle size of SiCp varied from ~10 nm to ~20 nm.

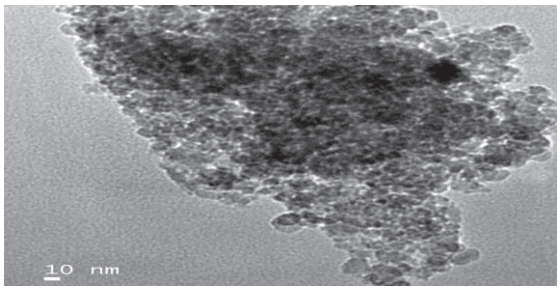

FIGURE 3. High Resolution Transmission Electron Microscopy(HRTEM) photograph of SiCp powders

PROCEDURES FOR CHARACTERIZATION OF CAST INGOT

Specimen preparation for micro structural study

The specimens were cut with low speed abrasive cutter for micro structural analysis. Three different grades (60, 400 and 600 grits) of emery papers were used to polish the specimen surface. Then using alumina fine powder cloth polishing for about 20-30 minute was carried out. The surface was cleaned with alcohol solution. Finally diamond polishing was done and the specimen surface was cleaned with acetone. The specimens were etched with Keller's reagent. The purpose of etching is to optically enhance micro structural features such as grain size and phase feature. The etching was done for 30-40 seconds. Etched specimens were cleaned in acetone using an ultrasonic vibrator and dry hot air. The specimens were cold mounted in order to achieve a uniform flatness of specimen surface and the cold mount base.

Hardness Test

Micro hardness Test: The micro hardness tests were done by using Leica VMHT Auto digital micro hardness test machine which is high degree of automation. The Vickers hardness number (Hv) was determined by measuring the length of the diagonals of the indentation. In the present study the geometry of the indenter was square based pyramidal diamond with face angles of 136^{o}. The test was carried out at 100 gm load applied for 15 s dwell time. The variation in the micro hardness values obtained for a particular specimen was +/– 5Hv and they were averaged.

Vickers's Hardness Test: Vickers bulk hardness was taken using WMW "HECKERT" hardness testing machine (made in Germany). The Vickers hardness tester has a large screen display LCD, used the menu interface type structure and can be in operation panel choose hardness HV .The load applied was 5kg and dwell time was 15s. The variation in the bulk hardness values obtained for a particular specimen was +/– 6Hv and they were averaged.

Results of Micro hardness and Vickers's Hardness Tests

The ingots were cut into smaller samples as shown in Fig. 4(a) and 4(b) show the locations of samples cut out from the ingot of ~ 230 gm for micro hardness (6 samples) and Vickers hardness tests (4samples) respectively. Fig. 4(c) shows the locations of 12 samples cut out from the ingot of ~ 340 gm for micro hardness tests. Vickers hardness samples for 340 gm ingot were cut as per Fig4 (b). Hardness tests were repeated 2-3 times on each sample and the average values are shown in Table 1. The hardness variation in the ingot weighing ~230 gm was not significant indicating that at the scale of the ingot the dispersion was uniform. In the ingot, having a weight of ~340 gm, higher hardness values were observed in the bottom region.

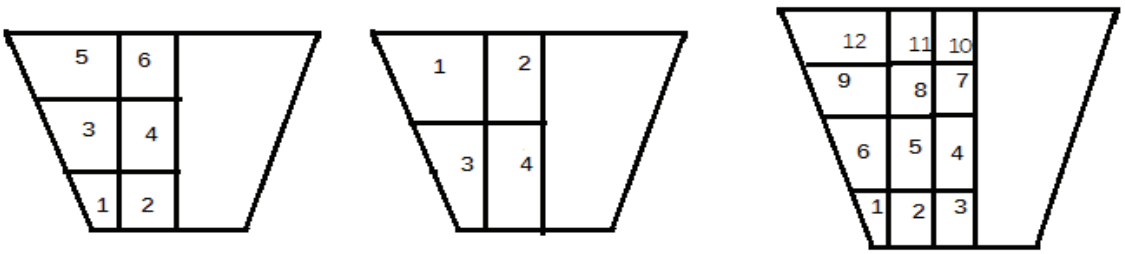

FIGURE 4. Locations of the sample for hardness (a) Cast wt =230 gm for micro hardness (b) Cast wt=230 gm for Vickers's hardness (c) Cast wt=340 gm for micro hardness

TABLE .1 Hardness distributions of Al-SiCp nano composites

Sample nos (Fig.5(a) 2wt %)	Average micro hardness(Hv)	Sample nos (Fig.5(b) 2wt %)	Average Vicker's hardness (Hv)	Sample nos (Fig.5(c) 3wt %)	Average micro hardness (Hv)
1	103.9	1	83	1	136.8
2	101.2	2	87	2	133.2
3	98.4	3	89	3	137
4	102.8	4	82	4	130.1
5	104.2	-	-	5	127.4
6	107.2	-	-	6	121.2
-	-	-	-	7	119.1
-	-	-	-	8	115.4
-	-	-	-	9	114.2
-	-	-	-	10	117.4
-	-	-	-	11	112
-	-	-	-	12	111.3

Specimens Preparation of for TEM study

The specimen was cut by using low speed diamond cutter. The thinning operation was done by placing the specimen over a belt grinder. The slice was then attached to a block using suitable adhesive and subjected to thinning using emery paper (6 grit), such that the thickness of the specimen was reduced below 100 μm. Finally discs of 3mm diameter were punched out of the specimen using mechanical punch (Gaton Model 642). Then pre-thinning of the discs was carried out using a dimple grinder (Gaton Model-656). By dimpling, the centre regions of the discs were thinned to ~20 μm from a thickness of ~100 μm. The pre-thinned discs were finally subjected to ion milling (Gaton Precision Polishing System Model 691). The difference between the top and bottom gun angles of the ion milling machine was 3.5°. The vacuum inside the ion beam milling chamber was 10^{-6} Torr. A beam of 5keV was used for ion milling. As a result of ion milling a hole was formed in the centre and examined under a TEM.

TEM Characterization

For TEM analysis high resolution TEM (JEOL, JEM-2100) was used. The TEM operated at an accelerating voltage of 200 kV. Bright field images of nano-particulates spread in the Al matrix were taken and selected area diffraction patterns were recorded. EDAX(Energy Dispersive X-ray Analysis) was also carried out.

Results of TEM Studies

The Fig.5 shows EDAX of Al-SiCp nano composites confirming the SiCp nano particles in the composites. TEM samples were made from different regions of the cast. From the qualitative observation, it can be inferred that

the SiCp nano-particles have got dispersed uniformly within the ingot. However within a length scale of 1 μm one can observe segregation of the nano particles. It is possible that the nano-particles have segregated in the grain boundary region. Fig.6 shows the TEM micrographs of Al-2 wt% SiCp nano composite taken at different magnifications. Fig. 6(a) shows the SiCp nano particles spread uniformly. They are possibly surrounded by sub-grains. Fig. 6(b) shows the SiCp nano particles segregated along a line, which is possibly a grain boundary.

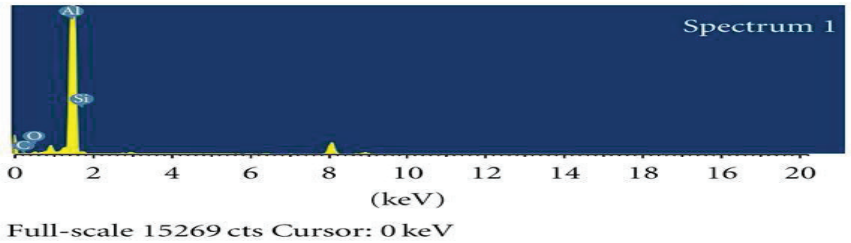

FIGURE 5. EDAX of Al-SiCp nano composite

Optical Microscopy

The study was conducted for the evolution of micro structures of Al and SiCp nano composites and uniform mixing of mixing of nano particles in the Al matrix. Optical microscopy images have been qualitatively analyzed in terms of two main parameters, particles size and homogeneity of the sample. From the Fig.7(a), 7(b) & 7(c) it is clear that the SiCp nano particle is uniform across the ingot & it is uniformly dispersed near the gain boundaries.

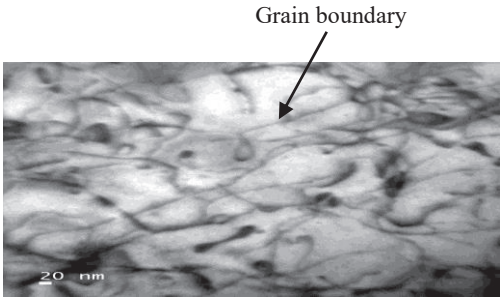

FIGURE 6(a). TEM micrographs of Al-SiCp

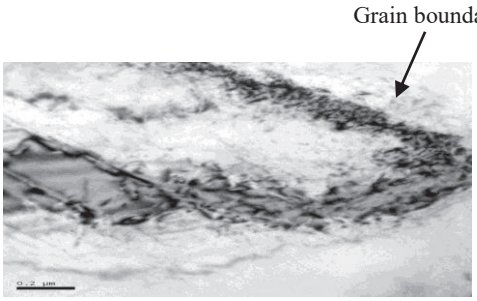

FIGURE 6(b). TEM micrographs of Al-SiCp

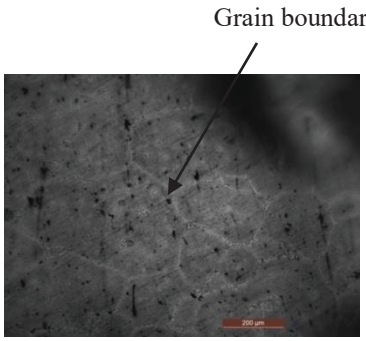

FIGURE 7(a). Al+SiCp 2wt%

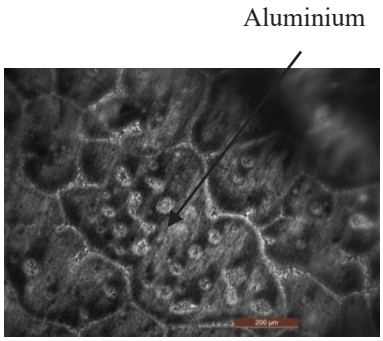

FIGURE 7(b). Al+SiCp 3wt%

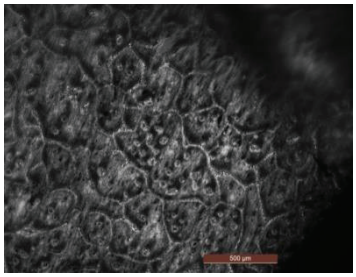

FIGURE 7(c). Al+SiCp 3wt%

FIGURE.7 Optical Microscopy of Sample Al+SiCp(SEM studies)

DISCUSSIONS

From the micro-hardness values taken from different locations of the ingot, it is clear that the distribution of nano-particles is uniform across the ingot. It was found that the liquid metal nano-particles were uniformly dispersed and the segregation of the particles near the grain boundaries is due to its grain growth. By TEM observation from Fig.6(a) and Fig.6(b), the particles are uniformly dispersed due to ultrasonic vibration and cavitations.

From the above discussion, it is clear that the mechanism of uniform distribution of nano particle within liquid metal is subjected to ultrasonic vibration. Nano-particles have a tendency to agglomerate due to Vander Waals forces. Thus during casting of nano-composites, high-intensity ultrasound has been used for mixing, dispersing and deagglomeration the nano particles [25-28]. When sound wave propagates into the liquid, alternating high pressure and low-pressure cycles are generated. During the low-pressure cycles, small bubbles or voids are formed in the liquid. When the bubble reaches a critical size, it can no longer absorb energy. As a result, they collapse violently during the high-pressure cycle. During the collapse of bubbles, high-pressure shock waves are generated and propagate through the liquid at velocities above the speed of sound, which keep the nano particles uniformly dispersed.

CONCLUSION

1. From the micro-hardness values taken from different locations of the ingot, it is clear that the distribution of nano-particles is uniform across the ingot.
2. TEM studies reveal segregation of nano particles near the grain boundaries.
3. From EDAX of Al-SiCp nano composites, the presence of SiCp was confirmed.
4. From Optical microscopy images have been qualitatively analyzed and found that uniform distributions of SiCp nano particles in the metal matrix nano composite.

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