INVESTIGATION ON MECHANICAL PROPERTIES OF A356 NANOCOMPOSITES FABRICATED BY ULTRASONIC ASSISTED CAVITATION

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Abstract: *This paper presents mechanical properties of A356 matrix nanocomposites fabricated by reinforcing nano silicon carbide (SiC) particles at 0.1 to 0.5 weight percent with the aid of ultrasonic cavitation. SiC nanoparticles synthesized by bottom-up and top-down approach are used in this work as reinforcements and change in mechanical properties are observed. For comparision, A356 matrix composite reinforced with microparticles at 0.5 wt% by stir casting also prepared. Till now, SiC nanoparticles produced by bottom up approach were used as reinforcements in A356 alloy by so many researchers to improve mechanical properties. But in this work, nanoparticles produced by high energy ball milling (Top-down approach) are also used as reinforcements. It is observed that there is reduction in tensile properties compared to the composites prepared by reinforcing nanoparticles via bottom up approach but significant increase compared to the composites prepared by reinforcing same amount of microparticles by stir casting. The reasons may be due to the non-uniformity in size of particles and contamination while milling nanoparticles. However, the change in properties is less and use of nanoparticles via top down approach is admissible when compared to the cost of nanoparticles synthesized from bottom up approach or chemical routes.*

Keywords: *Ultrasonic Cavitation, Nanocomposites, Metal Matrix, A356 Alloy, SiC, Nano*

1. Introduction

There are two approaches to the synthesis of nanomaterials and the fabrication of nanostructures; viz top-down and bottom up. Top down approach involves the breaking down of the bulk material into nano sized structures or particles. These techniques are an extension of those that have been used for producing micron-sized particles. An example of such a technique is high energy ball milling. The difficulty with top-down approaches is ensuring all the particles are broken down to the required particle size. Furthermore, longer milling times will result in more milling impurities, which together with any milling aids, if used, can be difficult to remove. However, by minimizing the milling time and using the purest, most ductile metal powders available, a thin coating of the milling tools by the respective powder material can be obtained which reduces Fe contamination tremendously. Atmospheric contamination can also be minimized or eliminated by sealing the vial with a flexible 'O' ring after the powder has been loaded in an inert gas glove box.

The biggest problem with top down approach is the imperfection of surface structure and significant crystallographic damage to the processed patterns. But this approach leads to the bulk production of nanomaterial. Regardless of the defects issued by top down approach, they will continue to play an important role in the synthesis of nanostructures e.g. in the synthesis of nano composites etc. Bottom up approach promises a better chance to obtain nanostructures with less defects, more homogeneous chemical composition. But production rates of nano powders through bottom down approach are very less i.e. few grams per day.

Casting, as a liquid phase process, is well known for its capability to produce products with complex shapes. It will be desirable to produce as-cast light weight components of MMNCs with good reinforcement distribution and structural integrity. However, nano-sized

ceramic particles present difficult problems: it is extremely difficult to disperse them uniformly in liquid metals because of their poor wettability in metal matrix and their large surface-to volume ratio, which easily induces agglomeration and clustering [1].

In order to achieve a uniform dispersion and distribution of nanoparticles in aluminum matrix nanocomposites, G.I.Eskin et al. [2], Yong Yang et al. [3], Yong Yang et al.[4], Yong Yang, Xiaochun Li et al [5] developed an innovative technique that combined solidification processes with ultrasonic cavitation based dispersion of nanoparticles in metal melts. It was reported that ultrasonic cavitation can produce transient (in the order of nanoseconds) micro "hot spots" that can have temperatures of about 5000°C, pressures above 1000 atm, and heating and cooling rates above 10^{10} K/s [6]. Transient cavitations could produce an implosive impact strong enough to break up the clustered fine particles and disperse them more uniformly in liquids.

It is envisioned that strong microscale transient cavitations, along with macroscopic streaming, might effectively disperse & distribute nanoparticles into melts and also enhance wettability, thus making the production of as-cast high performance light weight MMNCs feasible.

By utilizing the ultrasonic cavitation based solidification processing method, A356 alloy reinforced by SiC nanoparticles have been fabricated successfully [4, 5, 7, 8] and reported that there is an increase in mechanical properties. But in all the above works, A356 Metal Matrix Nanocomposites (MMNCs) were prepared under different experimental conditions and process parameters i.e weight of aluminium melt, processing temperature, Power rating of ultrasonic transducer, nano powder feeding method, time of sonication etc. but size of silicon carbide (average size≤30nm) and mode of synthesis (i.e bottom up approach) is same in all the above works.

In the present work, except matrix material all the remaining process parameters and experimental conditions are different including silicon carbide size and the mode of its synthesis (i.e Top down approach as well as bottom up approach.)

In this paper, microstructures and tensile stress of SiC reinforced A356 matrix nano-composites has been investigated.

2. Experiment Setup and Procedure

2.1. Materials for experiments

Aluminum alloy A356 was selected as matrix material. Because it is readily castable, widely studied and used. To select a suitable reinforcement and matrix for the aluminum nanocomposite, important factors such as density, wettability and chemical reactivity at high temperatures should be considered. Silicon carbide is selected as reinforcement material because of its good wettability, nearly identical density and the big difference of the thermal expansion coefficients with aluminum alloys.

A356 alloy was procured from M/S. Sri Krishna Enterprises, Secunderabad, India. The composition analysis of raw material along with other results such as hardness, density is presented in Table.1 & 2. These values are obtained by using optical emission spectrometer (SHIMADZU-JAPAN, Model: TDA-7000) & single pan balance respectively at Chennai Mettex Laboratories, Chennai, India.

Table 2: Density & Hardness of A356 alloy (not heat treated)

Density (gm/cc)	2.662
Hardness (Brinell, 10mm ball dia & 500 Kg load)	57

The SiC nano particles are used as reinforcement in the aluminum matrix for synthesizing the composites. The nano particles were procured from Hefei Kaier Nanometer Technology & Development Co., Ltd, China**.** These were synthesized by bottom up approach i.e Chemical Vapor Deposition with an average particle size ≤ 50nm.

Table 1: Composition analysis (Wt %) of A356 alloy

Designation	Si	Cu	Mn	Mg	Zn	Ti	Fe	Ni	Pb	Cr	V	Co	Al
A356	6.515	0.013	0.082	0.278	0.030	0.128	0.371	0.020	0.0004	0.014	0.006	0.004	92.5

Another nano sized SiC ceramic particles used in this study were prepared by high energy ball milling. Green SiC micro powder of 9.3 µm & 125 µm were obtained from M/S. Silcarb Industries ltd., Bangalore, India. 9.3 µm SiC powder was milled in high energy planetary ball mill (model Fritsch Pulverissette 6, Insmart systems, Hyderabad, India) to produce nanopowder of 34.179nm and 125 µm SiC powder was used to produce micro composites.
25 grams of green silicon carbide powder (9.3 µm) were placed in 80 ml tungsten carbide mixing jar together with 5 tungsten carbide milling balls (10mm diameter) and one tungsten carbide milling ball (15mm diameter); giving a ball-to-powder weight ratio of 10:1. The jars were agitated using a high energy planetary ball mill at 20 rpm for 18 hours. 2 ml of methanol was added as a process control agent (PCA) in order to prevent powders sticking to the balls and the jar walls. High energy ball mill and the mixing jar loaded SiC and tungsten carbide balls are shown in Fig.1 (a) & (b).

Fig.1: (a) Planetary High Energy Ball Mill
(b) Mixing Jar loaded with milling balls and SiC

Powder

XRD analysis was done to find size of nanoparticle and also to detect the presence of different elements in the SiC after milling. XRD work was carried out on a RIGAKU ULTIMA-IV, Japan System. The X-ray diffractograms were taken using Cu Kα radiation. The powder is packed on a sample holder of size 20mm x 20mm rectangular cavity having depth of 2 mm. the samples are scanned at a scanning speed of 2 degree per minute (2θ) in the range of 10 to 80^0, the measurement is done at an applied voltage of 40kV and current of 40mA. Figure.2. (a) & (b) shows convoluted and deconvoluted graphs from XRD analysis of SiC particles. From deconvoluted graph (Fig.2 (b)), the intense peak found at 35.601^0 (2θ) and corresponding FWHM value (0.297^0) noted. According to Scherrer formula [9], the average crystallite size found as 34 nm.

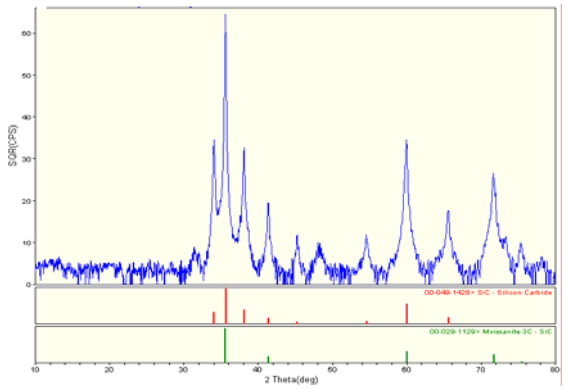

Fig.2: (a) Convoluted Graph for SiC Nano particles Synthesized by High Energy Ball Milling.

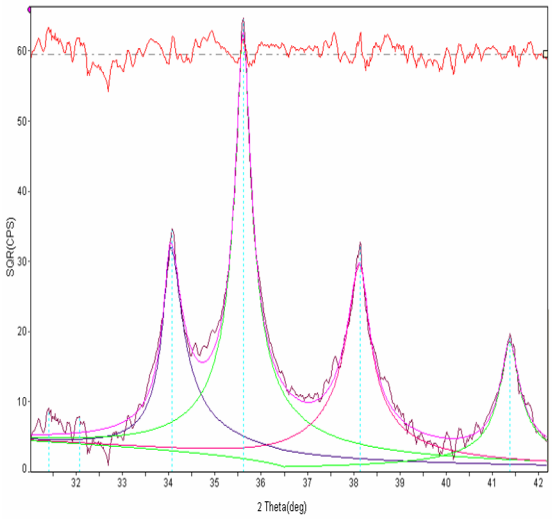

Fig.2: (b) Deconvoluted Graph for SiC Nano particles Synthesized by High Energy Ball Milling.

2.2. Experimental Set Up

The experimental nanomanufacturing setup is shown in Fig. 3.(a) & (b), including furnace, ultrasonic probe & Transducer, ultrasonic generator, temperature controller, and inert gas protection nozzles. In this process, an electric resistance heating unit was used to melt the A356 in an EN8 steel melting pot of size 110 mm diameter by 150 mm height with 1.0 kg capacity. Nanosized SiC particles were fed into melts during the ultrasonic processing. The aluminum melt pool was protected by argon gas. One temperature probe was used to monitor the processing temperature.

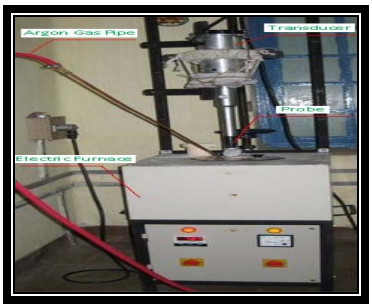

Fig.3. (a) Electric Furnace with ultrasonic transducer

b) Ultrasonic Generator

2.3. Production of Metal matrix micro and nanocomposites:

The processing temperature was controlled at 700°C approximately 100°C above the alloy melting point. An ultrasonic probe (Hangzhou Success Ultrasonic Equipment Co., Ltd, Zhejiang Province, China.) with 20 kHz, 1KW output was used to generate adequate processing power inside the crucible. Ultrasonic probe is of 31 mm in diameter and 102 mm in length specially made for aluminum melt which can withstand high processing temperature with minimum ultrasonic cavitation induced erosion. An ultrasonic power of 1kW from the transducer was found to generate adequate non-linear effects inside the crucible[10]. Preheated nanosized SiC particles were added into melts from the top of the crucible during the process.

Though density of SiC nanoparticles is nearer to the density of aluminium 356 alloy, because of the surface tension of the melt, the nanoparticles tend to float on the top of the melt surface at first before ultrasonic cavitation helped to mix them into the melts. So before inserting the probe into the melt, stirring was done using metallurgical stirrer at a speed of 750rpm for 3 minutes. This is an effort to mitigate higher times of sonication.

Aluminium 356 matrix nanocomposites with various weight percentages of nanosized SiC were fabricated, including 0.1, 0.2, 0.3, 0.4 and 0.5 wt. % by reinforcing nano particles synthesized from bottom up as well as top down approach separately. Melts were processed for 6 minutes for every 0.1 wt% and it was increased by 6 minutes for every 0.1 wt% increment. Sonication time is limited by trial and error method by looking at good dispersion of reinforcements in the matrix.For comparision, A356 matrix micro composites with 0.5 wt% of 125 μm SiC (supplied by Silcarb industries, Bangalore, India) by stir casting (stirring at 700^0C at 750 rpm for three minutes) were prepared separately.

When nanosized SiC particles were added in the Al alloy melts, the viscosity of the molten Al alloy significantly increased. Thus, after efficient ultrasonic processing, a higher melt temperature of 750°C was used to ensure the flowability inside a mould. The melt with the reinforced nano particles then poured to a permanent mould made of Mild steel. After pouring is over the melt was allowed to cool and solidify in the mould. The permanent mould for tensile test specimen is shown in fig.4 below.


Fig.4: Tensile Specimen Permanent Mould

2.4. Performing Experiments

In each casting, three standard tensile specimens [11] were made with a gauge length of 32mm and a diameter of 6 mm. schematic diagram of tensile test specimen is shown in Fig.5.

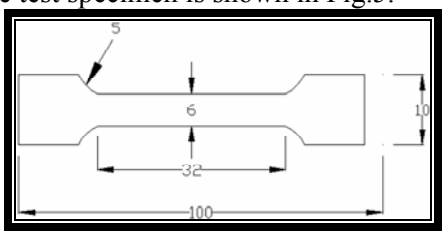

Fig.5: Schematic Diagram of Tensile Test Specimen [11]

To be comparable with industrial standard, castings for tensile testing were T6 heat treated. T6 stands for Precipitation heat treatment at 155^0C for 6 hours following solution heat treatment at 540^0C for 5 hours and quenching. The tensile properties of the nanocomposite & microcomposite specimens were tested with an INSTRAN machine according to the standard of ASTM E8. The hardness of the heat treated samples was measured using a UHL Vickers micro hardness measuring machine by applying a load of 50gms. The load was applied for 20 seconds. In order to eliminate possible segregation effect a minimum of three hardness readings were taken for each specimen at different locations of the test samples. Before each hardness test series, the specimens, which are of 50mm thickness and 10x10 mm cross section were metallographically polished with a 240 grit SiC paper until the oxide layer was removed and the opposite sides were perfectly parallel.

For metallographic examination, specimens of 10x10x10 mm were prepared by grinding through 1x0, 2x0, 3x0, 4x0 quality emery papers followed by polishing with 6µm diamond paste and etched by Keller's reagent. The microstructures were obtained by viewing the samples at different magnification levels on SEM (Model: HITACHI make with field emission gun).

3. EXPERIMENT RESULTS:

3.1. Micro Structures:

Samples reinforced with SiC nanoparticles from top down as well as bottom up approach with ultrasonic processing at 1.0kW power were examined with the SEM, and typical microstructures are shown in Fig.4. Figure 6(a) shows the aluminum matrix with addition of 0.5 wt % nano particles of bottom up approach. Fig 6(b) shows a typical SEM image of sample with 0.5 wt% nano particles of top down approach. Fig 6(c) shows optical microscope image of sample with 0.5 wt% microparticles. The nanoparticles were well dispersed in the A356 matrix, although some microclusters remained in the matrix. It is believed that high intensity ultrasonic waves generated strong cavitation and acoustic streaming effects.

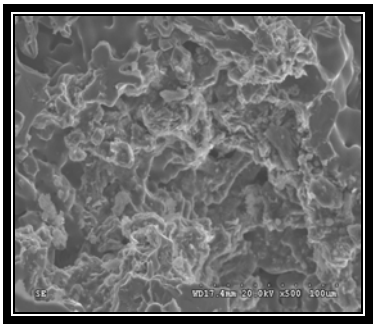

Fig.6: (a) SEM Image of A356 matrix reinforced with 0.5wt % of nano SiC of Bottom up approach

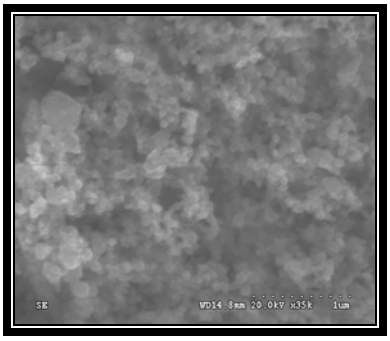

Fig.6: (b) SEM Image of A356 matrix reinforced with 0.5wt % of nano SiC of Top-Down approach

Fig.6: (c) Optical microstructure of A356 matrix reinforced with 0.5Wt% of micro SiC particles (125µm) at 200x magnification.

3.2. Mechanical Properties

The tensile test results and hardness results are presented in Table.3, Where the ultimate tensile strength, yield strength and hardness of micro and nanocomposites are compared with that of A356 alloy.

It can be observed that with only 0.5wt% nano sized SiC made by high energy ball milling, the ultimate tensile strength and yield strength of the nanocomposites improved by 37% and 48.3% respectively. But ductility has been decreased. This can be understood from the decrease in elongation percentage to 2.58%.

Where as with the addition of 0.5 wt % nano sized SiC made by bottom up approach, the ultimate strength and yield strength of the nanocomposites improved by 60.6% and 55.26% respectively. Ductility in this case has not been decreased and retained within the permissible range i.e 4 to 6 %. The improvement in mechanical properties for the above cases is significantly higher than that of aluminum alloy composite even with maximum percentage of micro particle reinforcement i.e 0.5wt%. Whereas for microcomposite, the elongation has not changed significantly. It may be due to small wt% of microparticles reinforced in the composite. Mechanical properties and stress Vs strain curves are (pure A356, 0.5wt%micro SiC composite,0.1 to 0.5wt% nano SiC composite) shown in Fig.7 & 8.

Hardness value change due to variation of reinforcement ratio also is given in Table.3. improvement in hardness with only 0.5wt% high energy ball milled particle reinforced composites is more compared to bottom up approach particle reinforced composites. In the former case it is observed as 66.67% and 55.55% in the later case.

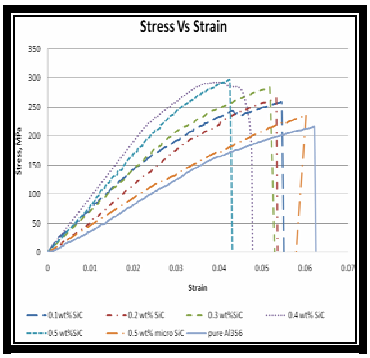

Fig.7: Stress Vs Strain for Nano & Micro Composites (SiC Particles synthesized by Top Down appraoch)

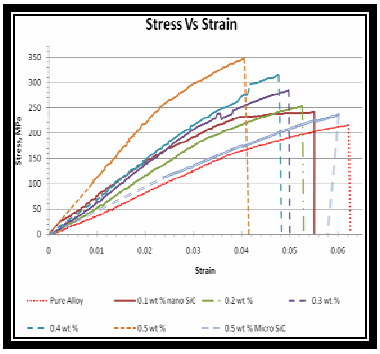

Fig.8: Stress Vs Strain for Nano & Micro Composites (SiC Particles synthesized by Bottom up approach)

Table.3: Mechanical Properties of specimens

Sl.No	Specimen	Nanocomposite reinforced with SiC particles made from high energy ball milling				Nanocomposite reinforced with SiC particles made from Bottom up approach			
		Yield Stress (MPa)	Ultimate Stress (MPa)	% Elongation	Hardness (HV)	Yield Stress (MPa)	Ultimate Stress (MPa)	% Elongation	Hardness (HV)
1	Pure A356 alloy	173.25	216.11	6.25	54	173.25	216.11	6.25	54
2	A356+0.1Wt% nano SiC	183	258	5.5	61	190	240	5.5	59
3	A356+0.2Wt% nano SiC	204.42	265	5.73	68	207	253	5.28	65
4	A356+0.3Wt% nano SiC	217.53	283	5.28	74	227	284	5	71
5	A356+0.4Wt% nano SiC	235	292	3.04	82	248	313	4.83	77
6	A356+0.5Wt% nano SiC	257	296	2.58	90	269	347	4.14	84
7	A356+0.5Wt% micro SiC	178	236	5.58	58	178	236	5.58	58

4.Conclusions

In this study, hardness, tensile strength of A356 reinforced with different weight percentage (0.1-0.5wt%) of SiC nanoparticles was examined and compared with pure alloy and 0.5 wt% micro SiC particle reinforced alloy.

With the increase in reinforcement ratio, tensile strength, hardness of nano SiC reinforced composites were increased with no significant change in ductility. Where as for microcomposite, slight increase in strength, hardness and decrease in ductility were observed. Usually there will be significant change in properties of microcomposites when reinforcements are more than 10% by weight. But for the sake of comparision with the nanocomposites, micro composites were prepared by stir casting.

By SEM Microstructures, it can be observed that reinforcements are well dispersed in aluminium matrix. Though many researchers used different power capacities of trasnducers and various sonication times, this investigation proves that 1 kW power ultrasonic transducer with 6 minutes sonication time for 0.1 wt % of reinforcements is sufficient to disperse nanomaterials in 500grams of aluminium melt.

Irrespective of the synthesis technique followed for the manufacturing of SiC nanoparticles, composites showed significant improvement in mechanical properties. But when compared to high energy ball milled particles, CVD synthesized particles shown a uniform increase in tensile properties with the retention of ductility as reinforcement percentage increases. More specifically, the rate of increase in yield strength is not in proportionate with that of ultimate tensile strength and hardness. Decrease in ductility and non uniformity in increase of tensile properties in the former case may be due to uneven size of particles and contamination if any while milling of nanoparticles.

It may be concluded that the use of nanoparticles by top down approach also be permissible to use in the fabrication of metal matrix nanocomposites. How ever it is advised to use nanoparticles produced by bottom up approach for the fabrication of nanocomposites in view of ductility retention with the uniform increase of tensile properties.

Scope For Future Work

In the present work, only the nanoparticles were added upto 0.5 wt% only in steps of 0.1% because of the difficulties experienced in feeding the nanoparticles due to their higher surface to volume ratio and of higher milling times for nanoparticles. The same work may be extended for higher weight percentages of nanoparticles by inventing good feeding techniques. Tribological behaviour and machinability of nanocomposites in untouched in this work. It may be tried as future work.

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