Fabrication and Investigation on Properties of Ilmenite ($FeTiO_3$) based Al-Nanocomposite by Stir Casting Process

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Abstract

In the present work Al (99.7) matrix nano composites were fabricated and characterized with the aid of stir casting. Ilmenite($FeTiO_3$) nano particles synthesized by top down approach using high-energy wet ball milling process. Al (99.7) matrix reinforced with the Ilmenite($FeTiO_3$) nano particles at 1 to 5 weight percent were fabricated with the help of stir casting. XRD characterization was carried out on nano particles showing the least particle size. SEM images of cast composites shows the uniform distribution of nano particles in the Al matrix. The results were analyzed and found that the composites reinforced with 5 wt percent nano particles cast at 850°C have homogeneity in micro structures and exhibit's increased mechanical properties like hardness and tensile strength.

Keywords: *Al Matrix, Ilmenite, Stir Casting, High Energy Ball Mill*

1. Introduction

During the past two decades, researches into the production of metal matrix nano composites have been investigated by numerous investigators. Recently developments in aluminum matrix composites (AMCs) are playing important role in the composites areas. AMCs exhibit excellent mechanical properties like low density, high specific strength, high structural efficiency, good wear resistance, excellent thermal and electrical properties. As a result, their applications were expanded into the fields of aerospace, automotive, infrastructure and military industries [1]. AMC's are advanced materials prepared by combining two or more materials in which tailored properties were analyzed. Upon the reinforcement of micro/ nano particles in the AMC's has improved mechanical properties due to the reinforcement of high strength and high modulus particles like BiC, SiC, TiC, Al_2O_3, TiCr, $FeTiO_3$, etc. to strengthen the metal matrix. Among various fabrication techniques for composites stir casting is one of the most economical, simple casting route, used in the present study to fabricate AMC's. Prasad et al. fabricated rice husk ash particulates as reinforcement using stir casting technique and their resuls showed fairly uniform distribution due to the vortex generated with improved mechanical, tribological and damping behavior of the composites [2-4]. Ansary yar *et. al.*, [5] studied the micro structure and mechanical properties of the aluminum alloy matrix composite reinforced with nano particle MgO by vortex method and showed mechanical properties such as hardness and compressive strengths improved. Sajjadi *et. al.*, [6] reported the enhanced mechanical properties of Al-Al_2O_3 micro and nano composites fabricated by stir casting method. Sanjeev Das *et. al.*, [7] investigation on stir casting route has been used for incorporating zircon sand particle of different size and amount in Al-4.5wt% of alloy melts and their findings showed improved abrasive wear characteristics. To the best of the authors knowledge no attempts have been made to study the effect on mechanical properties with the reinforcement of nano sized Ilmenite in the Aluminum matrix. In the present investigation nano-particles Ilmenite was used as reinforcement for the fabrication of Al-Ilmenite nano composites by stir casting technique. Nano particle Ilmenite synthesized by high energy ball mill via top down method. Microstructure and mechanical properties of the base alloy and the composites were studied.

2. Experimental Procedure

2.1. Raw Materials for Experiment

Commercial available Al (99.7%) was taken as matrix material. It is easily castable and low cost. Considering properties like density, chemical reactivity at high temperature and irritability, nano Ilmenite is selected as reinforcement material. Al (99.7%) ingot of 25kg weight was procured from the M/S metal hose, Secunderabad, India. The chemical composition of Al was given in Table 1. The ingot is cut into small pieces, which are used to melt in the crucible. The Ilmenite ($FeTiO_3$) nano particles are used as reinforcement in the aluminum matrix for synthesizing the composites. These Ilmenite particles were collected from beach sand of Visakhapatnam and purified to 99.9% .Density of Ilmenite is 4.72Kg/m^3. The chemical composition of Ilmenite is given in the Table 2.

Table-1. Chemical Composition of Pure Aluminum

Sample	Si	Fe	Ti	V	Cu	Mn	Ga	Zn	Oth imp	Al
%	0.06	0.15	0.003	0.008	0.001	0.003	0.01	0.02	0.02	99.769

Table-2. Chemical Composition of Ilmenite

Constituent	**%**
Titanium	47.60 %
Iron	16.01 %
Oxygen	36.39 %
Total	100 %

2.2. Synthesis of Nano Structural Ilmenite by High Energy Ball Milling

The Ilmenite particles were synthesized to nano size by high energy ball milling. The Ilmenite particles are brought to nano size by top-down approach. 25 grams of Ilmenite(9.8µm) were placed in 80ml tungsten carbide mixing jar together with 5 tungsten carbide milling balls (10mm diameter) and one tungsten carbide milling ball (15mm diameter); gives a ball to powder weight ratio of 10:1 milling of particles takes place at 400 rpm for 48 hours. Methanol was added into the jar to prevent powder sticking to the balls and walls of the jar. After completion of milling hours the powder is taken out of the jar and it is collected in a vacuum container after methanol is allowed to evaporate.

2.3. Characterization of Ilmenite

The milled Ilmenite was characterized with an X-ray diffractometer (model: 2036E201; Rigaku Ultima.IV, Japan). JADE software was used to find the size of the nano particles and also to detect the presence of various particles. The X-ray diffraction measurements were carried out with the help of a goniometer (model: 2036 E201) using Cu k_α radiation (k_α = 1. 54060Å) at an accelerating voltage of 40kv and a current of 40mA. The samples are scanned at the range from 3˚ to 90˚of 20 with a scan rate of 2˚/min. Scanning electron microscopy (Model: SEM-Quanta 400, FEI, nether lands) with EDX energy dispersive X-ray spectroscopy is used for the elemental analysis of certain phases observed in the nano Ilmenite particles.

2.4. Fabrication of Al-Ilmenite Nano Composites

Al (99.7) and nano sized Ilmenite ($FeTiO_3$) are used for the fabrication of composites. The stir casting techniques involved meting weighted amounts of the alloy in a clay-bonded graphite crucible using an electrical resistance furnace. The temperature of the melt was raised to about 750^{o}C and

weighted percentage of nano Ilmenite particles (1, 2, 3, 4, and 5%) were added to the melt. The powder was packed in the aluminum foil as a packet. These packets were added into the molten metal of crucible when the vortex was developed, the packet of nano particles melted and the particles were distributed around the molten alloy. The melt was stirred at 650-700 rpm for 10-15 min using a graphite impeller attachment to a speed variable motor. The temperature was maintained at 850^{o}C throughout the stirring. There is an argon gas supply to the crucible in order to minimize the oxidation of molten aluminum. The melt after reinforcing with the nano particles is then poured into a permanent mold made of mild steel which is preheated and is allowed to cool and get solidify.

2.5. Mechanical Tests

The micro structure was investigated by optical microscopy dispersive X-ray scanning electron microscopy with an EDX energy electroscope. Tensile tests were carried on specimens prepared with a gauge length of 32 mm and a diameter of 6 mm. The schematic diagram of a tensile test specimen is shown in fig. 2a. For each composition, for standard tensile specimens were tested Fig.2b. Hardness of the cast nano composite is studied using Vickers hardness tester (Model: LECO-LV700, USA), the applied load is 2kg and dwell times is 15 Sec. An average of five readings was taken from each hardness value.

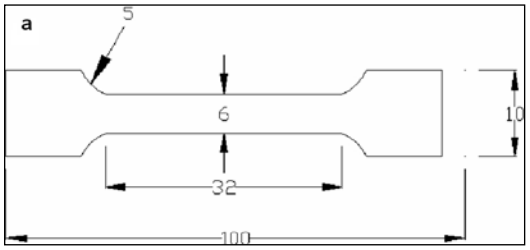

Figure 2a. Schematic Diagram of Tensile Test Specimen

Figure 2b. Fabricated Tensile Test Specimens

3. Results and Discussions

3.1. SEM and XRD Analysis of Synthesized Nano Ilmenite Particles

The SEM image of ball milled $FeTiO_3$ taken at 24 hour is shown in Figure 3a. Figure 3b shows the EDS spectrum of nano Ilmenite particles and the percentage chemical composition of Ball Milled Ilmenite was given in Table 3. Figure 3c shows deconvoluted graph from XRD analysis of nano Ilmenite particles. From the graph it was observed that the intense peak found at 32.8157^{o}(2Th) and corresponding FWHM value (0.1428^{o}2Th) was noted. According to Scherrer formula, the least particle size was found to be 57 nm.

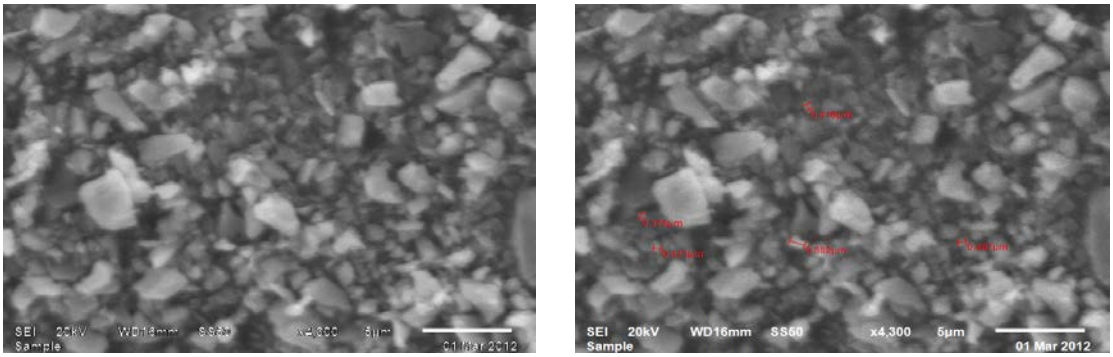

Figure 3a. SEM Images of Nano Ilmenite Particles

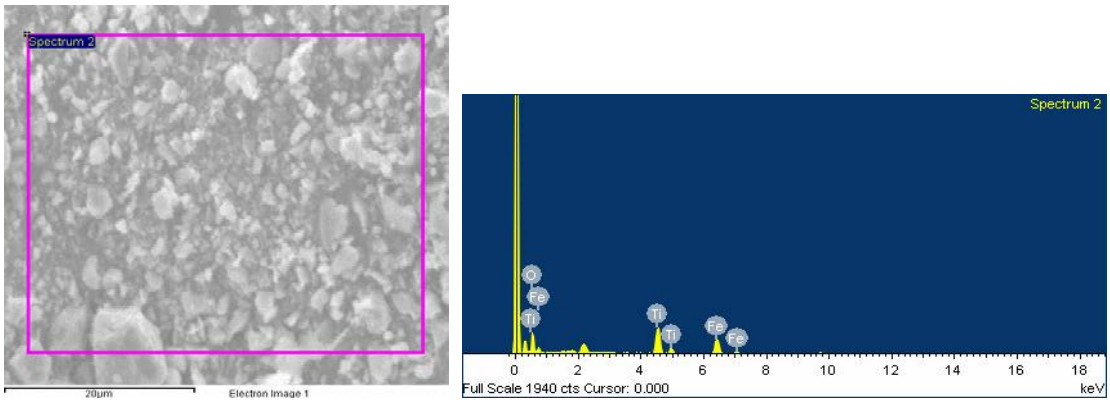

Figure 3b. EDS Spectrum of Nano Ilmenite

Table 3: Results of EDS Spectrum

Element	Weight%	Atomic%	Compd%	Formula
Ti K	47.60	27.96	79.40	TiO_2
Fe L	16.01	8.06	20.60	FeO
O	36.39	63.98		
Totals	100			

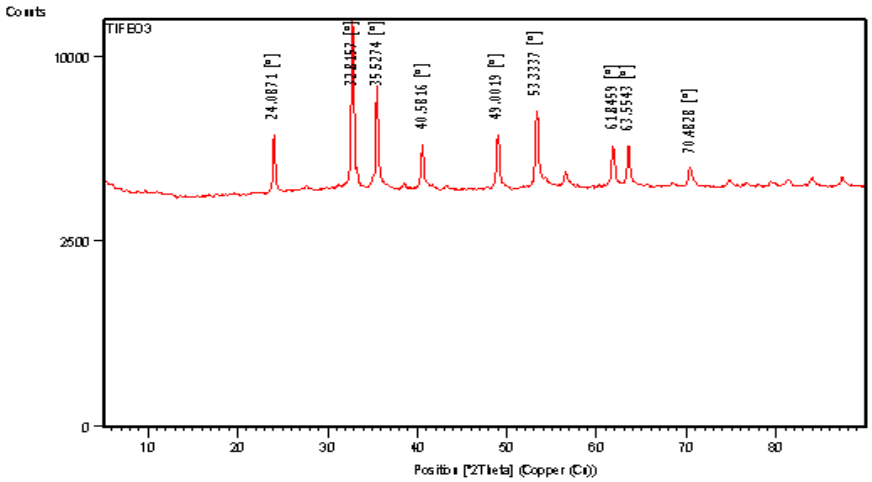

Figure 3c. Deconvoluted Graph of Ilmenite Nano Particles Synthesized by High Energy Ball Mill

3.2. Micro Structures

Samples reinforced with various percentages of nano Ilmenite particles are examined using Scanning Electron Microscope. Figure 4 shows the SEM images of aluminum matrix with addition 5wt% nano particles. Figure 4a shows the image of fabricated composite at 5µm resolution and Figure 4b shows the image at 2µm resolution. From the micrographs it was observed that the nano Ilmenite particles are well distributed in the aluminum matrix though some clusters were formed.

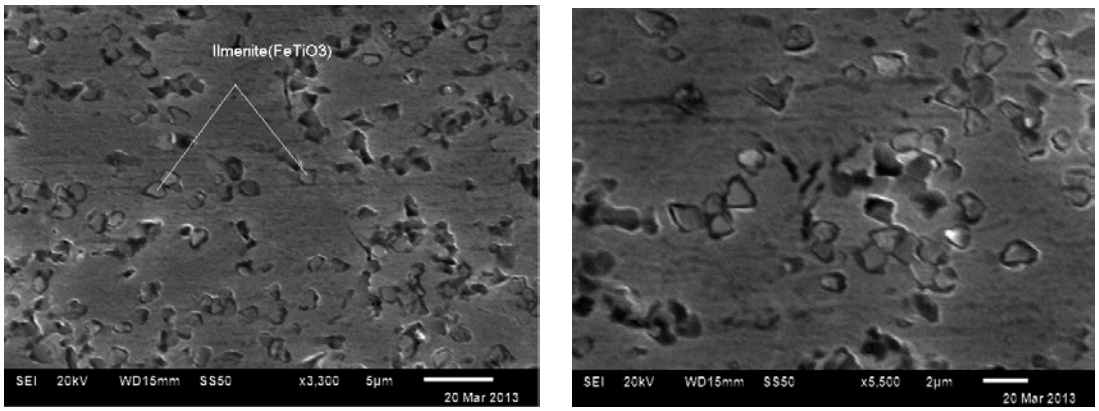

Figure 4. SEM image of 5% reinforcement at a) 20μm b) 2μm

3.3. Mechanical Properties

Figure 5 and Figure 6 shows the tensile strength Vs percentage of reinforcement and percentage of elongation Vs percentage of reinforcement respectively. It could be noted that the strength value increases with the increase of reinforcement of nano Ilmenite particles in the aluminum matrix as the load is distributed uniformly on nano Ilmenite particles in the matrix. Strength of metal matrix nano composite increases with the addition of nano Ilmenite slightly initially, but as the percentage of reinforcement of nano Ilmenite particles increases,the tensile strength tends to increase more with the higher percentages. The increase in mechanical properties of the composites was probably due to presence of hard phase titanium and iron elements. The composite containing 5 weight % Ilmenite show maximum ultimate tensile strength. And the ductility of the metal matrix nano composite slightly decreases with high Ilmenite concentration.

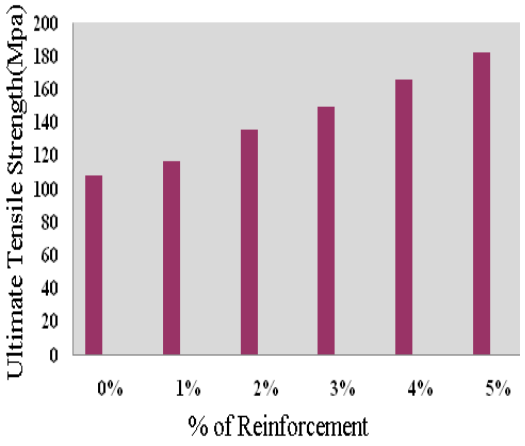

Figure 5. Tensile Strength Vs % of Reinforcement

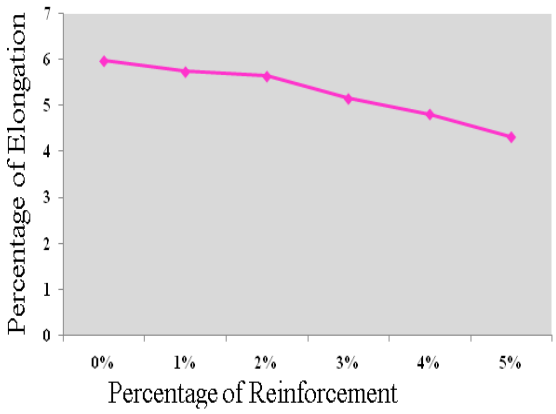

Figure 6. % of Elongation Vs % of Reinforcement

Hardness test was performed using Vickers hardness test. Figure 7 shows the hardness values of different wt% reinforcements. From Figure 7 it could be observed that the hardness value increases with the increase of reinforcement. The increment in the hardness of the composite may be because of nano Ilmenite particles acts as obstacles to the motion of dislocation. It is understood that Ilmenite nano particles are harder than aluminum so its hardness property is rendered to the soft matrix.

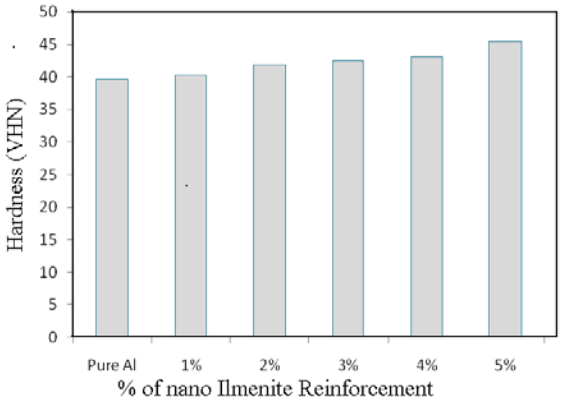

Figure 7. Graph Showing Hardness Results

4. Conclusion

1. High energy ball mill is used to synthesize nano particles using top down approach. SEM and XRD analysis was carried out on the milled powder to get particle size and chemical composition.
2. The stir casting technique is used to fabricate Al/nano Ilmenite particulate composites in which the Ilmenite particles are uniformly distributed through out the matrix alloy.
3. SEM micrographs shows that the nano particles are evenly distributed through some of the clusters were formed but good distribution was achieved.
4. The tensile and hardness values were increasing with the increase in reinforcement. At 5wt% nano Ilmenite reinforcement maximum tensile strength and hardness values has been observed. It can be concluded that with the addition of nano Ilmenite particles in the aluminum matrix, the mechanical properties like hardness and tensile strength hass been increased when compared to the cast aluminum, without much effecting ductility nature of matrix material .

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