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Synthesis and Consolidation of Cu-Al_2O_3 Composite by Mechanical Alloying

L. Rajesh kumar[1], S.Vidhya[2]

Assistant Professor, Department of Mechanical Engineering, KPR Institute of Engineering and Technology, Arasur, Coimbatore, Tamil Nadu, India.[1]

P.G Scholar, Department of Mechanical Engineering, KPR Institute of Engineering and Technology, Arasur, Coimbatore, Tamil Nadu, India.[2]

ABSTRACT: The microcrystallite copper with alumina reinforcement is studied for particle reduction by mechanical alloying. Mechanical alloying creates a repeated fracture and cold welding by which the particle size gets reduced. To study the reduction process of the milled powders, X-Ray Diffraction (XRD) analysis has been performed. From the intensity peak graphs, the particle size reduction is calculated by usingWilliamson and Hall equation of uniform deformation model. The distribution of alumina is improved with increase in milling. It also helps in improving the wear resisting property of the material which can be used in resistance welding electrodes. The particle size reduction achieved by mechanical alloying process is nearly 2.5nm from its as received sample to 12h sample after milling.

KEYWORDS: Cu-Al_2O_3 Composite, Mechanical Alloying, Ball Mill, XRD Analysis, Grain Size, Lattice Strain.

I. INTRODUCTION

In the recent years, materials science and engineering is the popular field where most of the researches are carried out. Materials science involves studying of materials paradigm (synthesis, structure, properties and performance). A variety of materials have been united with each other to form composites which give intended properties that are different from their individual base materials. It has taken the important position in the field of engineering. The new material have been preferred for many reasons: Examples include materials which are stronger, lighter and less expensive when compared to conventional materials. Composites can be made out of ceramic, polymer and metal materials among them metal materials posses additional advantages like fire resistant, operating at a wide range of temperature, does not absorb moisture, possess better electrical and thermal conductivity. Many different combinations of matrix and reinforcement can be made, among which the work is carried out on a metal matrix with ceramic reinforcement composite. The resulting composite gets combined properties of both the metal and the ceramic. The fabrication process followed for making the composites is powder metallurgy. One of the metals which are suitable to be used for electrical equipments is copper (Cu). To add some more properties like wear resisting, shape capability, strength and stiffness to the copper, aluminium oxide (Al_2O_3) is chosen as a reinforcement material [4]. This combination is found to be used in electrical conductors, lead frames, resistance welding electrodes and also the first wall of International Thermonuclear Experimental Reactor made of stainless steel is proposed to be bonded with the alumina dispersion-strengthened copper (DS Cu) plate for higher heat dissipation [9]. For dispersion of alumina in copper they have to go through a process called mechanical alloying. The process of mechanical alloying is carried out in a high energy ball mill. During mechanical alloying, there is a repeated mechanism of cold welding and fracturing. As a result of repeated cold welding and fracture,the homogeneous distribution of alumina among copper and reduction of the particle size into nano levels is achieved. The powder samples collected after milling were studied for their morphology using X-Ray Diffraction (XRD). The morphology of the particle is significantly related to properties such as compatibility, fluidity, magnetism and to chemical reactivity i.e., oxidation and sintering process [5]. Grain size has a greater impact on the strength of the material. From the XRD peaks, the grain size and the lattice strain of the samples will be calculated with change in milling time [2] [5].

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II. MATERIALS AND METHODS

The fabrication procedure of copper alumina composite through powder metallurgy consists of several steps like mechanical alloying, preheating, compaction and sintering. The copper and alumina powders constitute a framework designed with three different weight ratios. The composition of copper and alumina is showed in table 1.

TABLE.1 Weight Ratio of Composites

Sample No	Weight ratio (%)	
	Copper	Alumina
1	95	5
2	90	10
3	85	15

A method of fabrication where the composites are produced from powdered metals is called powder metallurgy. Powder metallurgy converts about 97% of initial materials into finished products and also the net shape of the product is obtained with less secondary machining process. The mechanical alloying process for Cu-5 wt% Al_2O_3 is started in the planetary ball mill. The planetary ball mill consists of a revolving sun wheel and two rotating milling vials. The speed ratio between the sun wheel and the vial is set as 1:4. Powder which is to be milled is placed in the milling vials along with the stainless steel balls as grinding medium. When the barrel rotates, Steel balls in the barrel are lifted to a certain height and fall down freely, so materials in the barrel are impacted [3].The ball to powder weight ratio is chosen to be 9:1. Toluene ($C_6H_5CH_3$) is used as the process control agent that completely immerses the steel balls and the powder present inside the vials. The process control agent reduces the accumulation of weld powders on the walls of the vials [6]. The milling operation is done for a total time 12 hour. After every 3hour the samples are collected to analyse the particlesize. The distribution of alumina in copper is analysed from the four samples obtained after 3, 6, 9, 12 hours of milling.

Fig. 1 High Energy Ball Mill

The precursor powders from ball mill are collected in a crucible in which powder gets settled at the bottom and the clear toluene liquid is filtered from it. To dry out the powder completely and to make the powder ready for warm compaction, the wet powder is preheated at a temperature of 180°C for duration of 30 minutes. The pre heated powder is transferred to the multiaxial forging die. For warm compaction, the die requires a coat of molybdenum disulphate as lubricator. The die is placed in a universal testing machine for the compaction load. The die compaction pressure is said to be 500MPa for a time of 30 sec. The compacted specimen in its initial are named as green compact. Green density of the specimen is decided by the parameters like compaction pressure and compaction temperature. The green compacts are placed in an electric furnace at a temperature of 550°C for about 3h and it's cooled by natural aging process.

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III. RESULTS AND DISCUSSION

A. Geometric Characterization

The results of morphology are obtained from the Schimadzu XRD 6000 X-Ray Diffractometer. It is used for detection of peaks of crystalline materials and examination of particle size. It depends on the elementary principles of electron beam and x-ray interactions with solid materials. XRD is operated at a scanning speed of 10.0 deg/min**.** X ray diffraction is analysed for the as- received samples and after every 3 hour of milling. Figure 1 show XRD intensity peaks of the initial samples of pure copper and alumina. The strongest peaks of pure copper occur at 2θ= 43.6357°, 50.7560°, 74.4020°. To find the structure of initial alumina, the intensity peak values of alumina and its corresponding 2θ are compared with the peaks of different structures of alumina (η-, γ-, δ-, θ-, β-, κ-, χ, and α-alumina). Peaks are obtained at a range of 10°- 90°.

TABLE 2. Stronge Peaks of Alumina

S.No	2 Theta (2θ°)	Intensity (Counts)
1	14.4218	1184
2	28.1510	862
3	38.2812	772
4	49.0138	546

The strongest peak values of alumina and their corresponding 2θ values are shown in TABLE 2. By comparing these XRD peaks of the below alumina [Fig 2 (b)] with the peaks of different structures of alumina, the peak positions match with the peaks of γ-alumina(gamma- alumina) [10]. Hence the structure of alumina (Al_2O_3) is found to be in gamma phase(γ- phase).

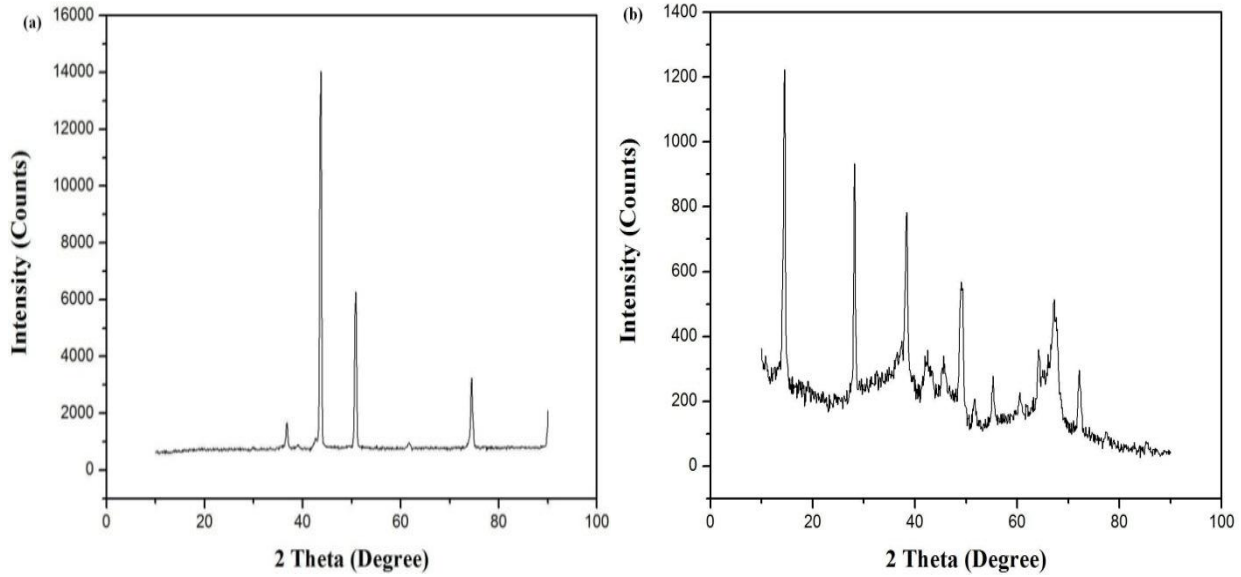

Fig.2 X ray Diffraction of As Received Samples of (a) copper.(b) alumina.

B. X- Ray Diffraction Analysis

Schimadzu XRD 6000 X-Ray Diffractometer is used for measuring the intensities using Cu Kα radiation. The diffractometer is operated with voltage of 40.0 (kV) and current of 30.0 (mA).The XRD spectrum of nanocomposite precursor Cu-Al_2O_3 powder with increasing milling time(3h, 6h, 9h, 12h)is shown in the Fig.3. X-Ray Diffractometer provides various parameters relating to the analysis of crystalline size and lattice strain.

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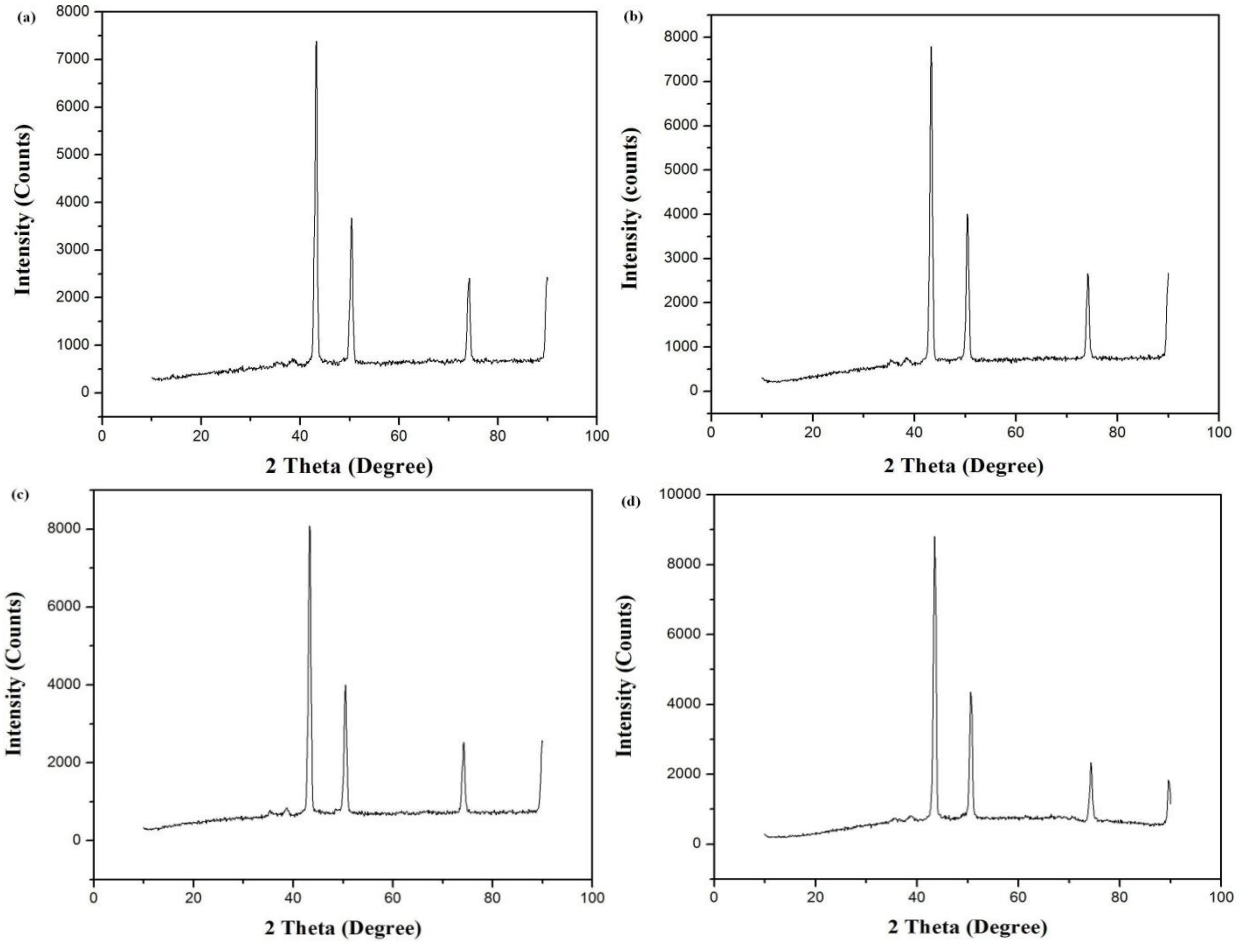

Fig. 3. XRD Peak of 95%Cu- 5%Al_2O_3 after Milling of Every 3 hours (a) 3h (b) 6h (c) 9h (d) 12h.

C. Evaluation of Particle Size

Williamson and Hall proposed a mathematical expression for uniform deformation model, calculating the crystalline size and lattice strain. The equation is

$$\beta cos\theta = \frac{k\lambda}{t} + 4\varepsilon sin\theta \qquad (1)$$

Where, β is the full width at half-maximum (FWHM) given by the XRD analyser, θ is the Bragg angle ($2\theta/2$), K is the shape factor (0.9), λ is the wavelength of the X ray (1.5406). The crystallite size (t) is obtained from the intercept, c=kλ/t. The lattice strain (ε) is obtained from the slope, m=ε [2]. The grain size and lattice strain at every 3 hour is obtained by constructing a linear plot between βcosθ and 4sinθ. The grain size and lattice strain of milled samples are presented in TABLE 3.

TABLE 3. Crystallite Size and Lattice Strain of Cu-Al_2O_3 Composite at Different Milling Time.

S.No	Milling Duration (h)	Grain Size (nm)	Lattice Strain (%)
1	3	1.9126	-10.47
2	6	1.8912	-12.125
3	9	1.7822	-12.29
4	12	1.7343	-14.609

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From TABLE 3. it is evident that the grain size is reduced with increase in period of milling. Generally, broadening of the peaks indicate the decrease in crystalline structure and increase in the lattice strain. The particle size reduction confirms the uniform dispersion of alumina among copper [7]. This can be obtained by the higher period of milling. The grain size and lattice strain of the particles at corresponding milling time is shown in the Fig 4 and Fig 5.

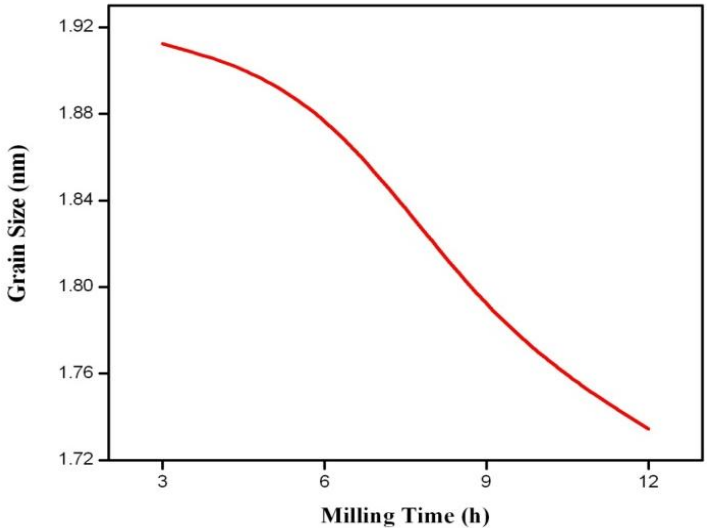

Fig.4. Milling Time vs Grain Size

The above Fig 4. Shows the linear decrease in the grain size from around 1.91nm at 3h of milling to final grain size of 1.73nm after 12 hours of milling. The decrease in grain size between 3-6 hours is lesser whereas the increase in lattice strain is higher and vice versa during next three hours of milling (6-9 hours).

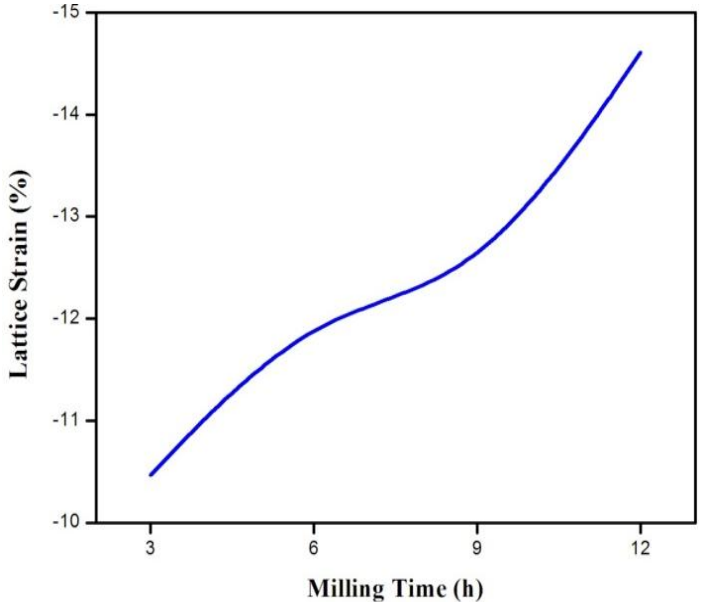

Fig.5. Milling Time vs Lattice Strain

Depending on milling time, the size of the crystallite is greatly reduced with the increase in lattice strain. There is more reduction of grain size at the first 3 h of milling with a trifling increase of lattice strain. An extreme increase in lattice

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strain is found after a severe plastic deformation (after 3 hours). The particle size and distribution of particles has minor impact on the green density of the composite [8]. After certain point of time, the material reaches a steady state beyond which there will not be any further fracture or cold weld. Further milling of materials after reaching a steady state would cause the lattice strain to decrease.

IV. CONCLUSION

The morphology is studied through XRD analysis for the milling of 95% Cu with 5% Al_2O_3 for total duration of 12 hours.The utilised alumina is found to have gamma structure. The main purpose of the milling was to reduce the particle size which was satisfactorily fulfilled. The grain size is reduced from its initial as received value of 2.5315nm to 1.7343nm after 12 hours of milling. After 12 hour of milling, it is noted that the materials have reached a steady state beyond which there is no cold weld or fracture. The reduction of grain size improves the uniformity of the contents in the specimen. With the reduction of grain size, the lattice strain gets increased. The lattice strain of the particles is increased with the increase of milling time. The raise in lattice strain takes place with minor decrease in grain size and vice versa. By decreasing the size of the particle and increasing the lattice strain, the mechanical properties of composite may also be enhanced.

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