Consolidation and Characterization of Copper Alumina Composites through Powder Metallurgy

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

E. Krishnaram [3]
UG Scholar
Department of Mechanical engineering, KPR Institute of Engineering and Technology, Arasur, Coimbatore, Tamil Nadu, India

H. Mukund Vijay[2]
UG Scholar
Department of Mechanical engineering, KPR Institute of Engineering and Technology, Arasur, Coimbatore, Tamil Nadu, India

E. Nanda Kumar[4]
UG Scholar
Department of Mechanical engineering, KPR Institute of Engineering and Technology, Arasur, Coimbatore, Tamil Nadu, India

Abstract:- Microcrystalline copper (Cu) with alumina (Al_2O_3) reinforcement powder (Cu- 5 wt% Al_2O_3) is ball milled for studying the reduction of particle size at different intervals of time. After mechanical alloying, the precursor is heated and then compacted at a pressure of 175MPa. The obtained green compact is sintered at 950°C for about 1 hour. To study the powder morphology, X-Ray Diffraction (XRD) analysis have been done. From the results of XRD, the particle size reduction is calculated by using Williamson and Hall equation of uniform deformation model. The particle size reduction from 3h to 12h is 452.3 nm to 141.5 nm. A graph comparing the milling time and the particle size is plotted showing the greater reduction of size between the third and sixth hour of milling whereas a graph comparing the milling time and lattice strain shows a lesser raise of strain during same milling period. The powders were also characterized by scanning electron microscopy (SEM) to identify the dominant process during milling.

Keyword — ***Powder Metallurgy, Copper alumina composite,Ball milling, particle size reduction, XRD and SEM.***

I. INTRODUCTION

The morphological properties of materials plays major role in determining their importance in application. This paper is also specifies such similar works for determining the properties by using composites manufactured by two different methods [1]. Composites are bonded materials to a matrix that have similar characteristics. Composites are of three types as metal matrix composites, ceramic matrix composites and polymer matrix composites based on types of materials involved. Each material will have its own unique property [2]. One of the metals which is suitable to be used for electrical equipments is copper(Cu).To add some more properties like wear resistance, corrosion resistance, shape capability, strength and stiffness to the copper, aluminum oxide (Al_2O_3) is chosen as a reinforcement, forming a metal matrix composite material [3] [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 is proposed to be bonded with alumina dispersion-strengthened copper (DS Cu) plate. Wear tests would not necessarily tell whether the coating was unsuitable for icebreaker application. For a dispersion strengthened copper alumina powders they have to go through a high energy ball milling for homogeneous distribution and reducing the particle size into nano levels [7]. The deterioration of materials causes higher maintenance costs, early system failures, or an overall shortened service life. The load applied to the contacting asperities is so high that they deform and adhere to each other forming micro-joints. The motion of the rubbing counter bodies result in rupture of the micro-joints [8]. In the present work copper and alumina powders were milled in a high energy ball mill and the powder samples were collected to study the morphology using X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM). The morphology of the particle is significantly related to physical properties such as compatibility, fluidity, magnetism and to chemical reactivity such as oxidation and sintering process [2]. 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.

II. EXPERIMENTAL PROCEDURE

A method of fabrication where the composites are produced from powdered metals is called powder metallurgy. By this method the near net shape of the final specimen can be obtained with negligible amount of initial powder wastages (about 3%) and hence eliminating the machining process. The Cu-Al_2O_3 composite is to be prepared for the composition of 95%wt copper and 5%wt of alumina.

A. Ball milling

Cu-5 wt% $Al_2O_3$is milled in the planetary ball milling. 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 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. The ball to powder weight ratio is 9:1. Toluene ($C_6H_5CH_3$) is used as the process control agent which completely immerses the steel balls and the powder present inside the jar. The process control agent

reduces the accumulation of weld powders on the walls of the vials. The milling is operated for a total time 12 hour. After every 3hour the samples are collected to analyze the particles. The distribution of alumina in copper is analyzed from the four samples obtained after every three hour.

B. Preheating and compaction

The precursor powders from high energy ball milling are collected in a crucible where powder gets settled at the bottom and the clear toluene liquid is separated from it. The die is placed in a universal testing machine for the application of load. The die compaction pressure is said to be 200MPa for a time of 30 sec.

C. Sintering

The compacted specimen in its initial 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 a electric furnace at a temperature of 950°C for about 1h and it's cooled by natural aging process.

III. RESULTS AND DISCUSSION

A. Geometric characterization

XRD analysis was done on the powder particles I order to determine their particle size. Schimadzu XRD 6000 X-Ray Diffractometer 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 10.0000 deg/min**.** X ray diffraction is analyzed for the as- received samples and after every 3 hour of milling. Figure 1show 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 2θare 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°.The strongest peak values which must coincide with the peak values of different structures of alumina and the2θ value of the same is shown in TABLE I.

B. X- Ray Diffraction Analysis

By comparing these XRD peaks of the above alumina with the peaks of different structures of alumina, the peak positions match with the peaks of γ-alumina(gamma- alumina).Hence the structure of alumina (Al_2O_3) is found to be in gamma phase(γ- phase).The XRD spectrum of nanocomposite precursor Cu-Al_2O_3 powder with growing milling time(3h, 6h, 9h, 12h). X-Ray Diffractometer provides various parameters relating to the analysis of crystalline size. The intensity peaks against 2Theta (2θ) of XRD at different milling time is shown in the Fig. 2.

C. Evaluation of Particle Size

Williamson and Hall proposed a mathematical expression for 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, c=kλ/t. The lattice strain (ε) is obtained from the slope m, m=ε. 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 II.

TABLE I. 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

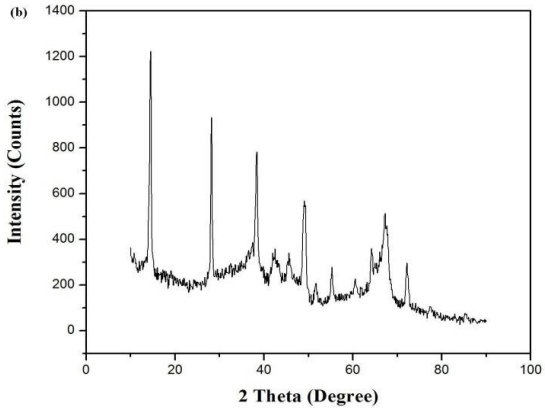
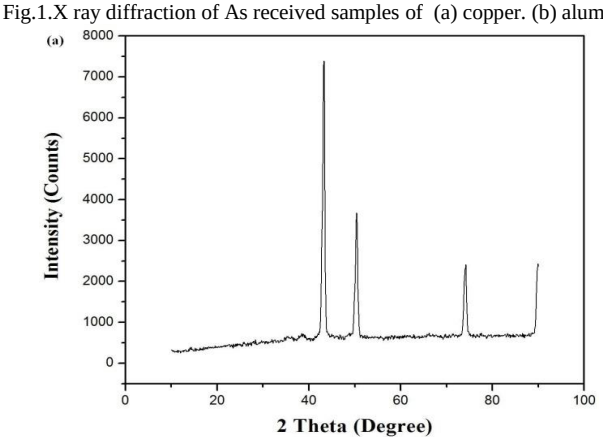
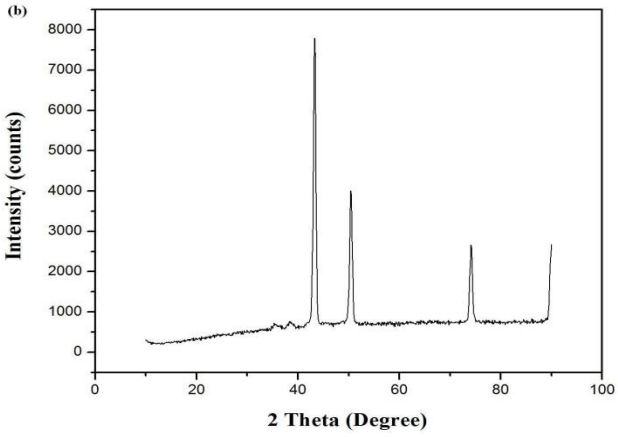


Fig.1.X ray diffraction of As received samples of (a) copper. (b) alumina.





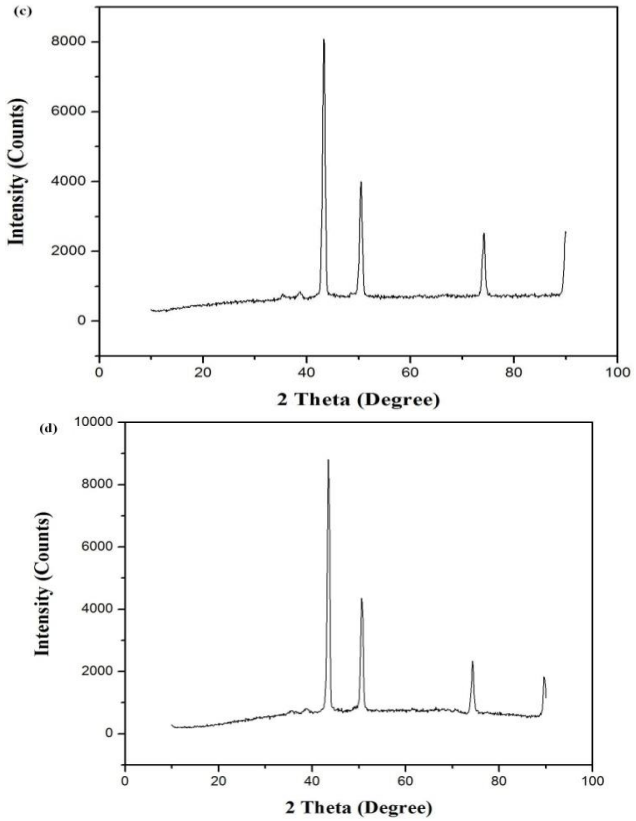


Fig. 2.XRD peak of 95%Cu- 5%Al_2O_3 after milling of every 3 hours (a) 3h (b) 6h (c) 9h (d) 12h.

Table II. Crystallite Size and Lattice Strain of the Cu-Al_2O_3Composite at Different Milling Time.

S.No	Milling Duration (h)	Grain Size (nm)	Lattice Strain (%)
1	3	452.3	-10.47
2	6	285.4	-12.13
3	9	326.9	-12.29
4	12	65.8	-14.61
5	15	141.5	-13.78

From table II 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. 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 3 and Fig 4.

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 and between the 3h-6h of milling where the increase of lattice strain is trifling. The extreme increase in lattice strain is found after a severe plastic deformation. Further milling of materials after reaching a steady state would cause the lattice strain to decrease.

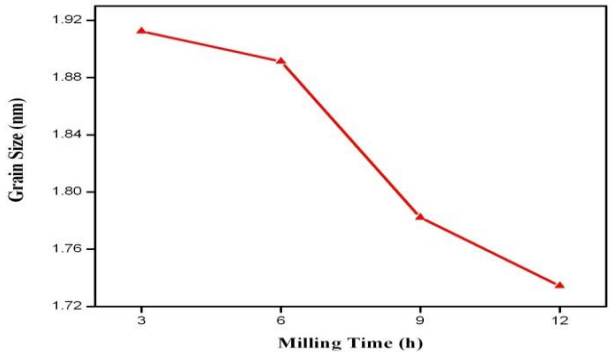


Fig.3. Milling Time vs Grain Size

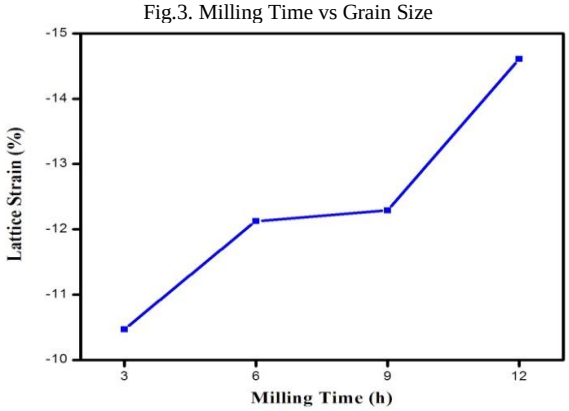


Fig.4. Milling Time vs Lattice Strain

IV. SEM ANALYSIS

The scanning electron microscope (SEM) uses a focused beam of high-energy electrons to generate a variety of signals at the surface of solid specimens. SEM reveals information like exterior morphology (texture), chemical composition, crystalline structure and orientation of materials making up the sample. The collected sample for every 3 hour is analyzed for its distribution by scanning electron microscope. SEM images of copper alumina powders revealed that the powders has undergone two major process such as fracture and cold welding. During fracture the strain is induced on the powder surface thus causing an irregularity on the surface. This was repeated at the time of milling and as a result the powders lost its actual sizer. During cold welding the powder particles tend to stick with each other at the spots where there were irregularities due to which the particle size increased. These two were the dominant process during milling cycles. After 3, 6 and 12 hours of milling fracture dominated thus reducing the particle size. In the meanwhile after 9 and 15 hours of milling cold welding dominated and as a result particle size had increased. As the particle size could not be reduced further after 15 hours of milling due to attainment of saturation, the milling cycles were stopped. Figure 5 (a) and 5 (b) shows the SEM images of the milled powders.

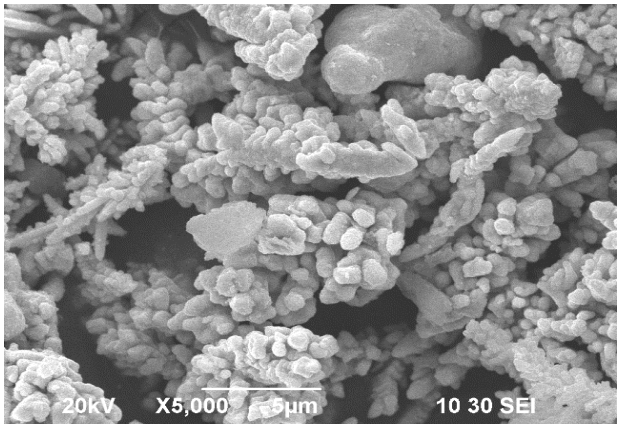


Fig. 5 (a) SEM image after 3 hours of Milling

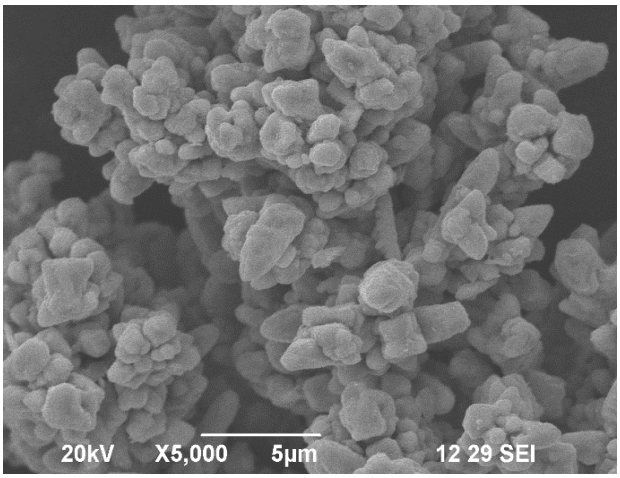


Fig 5 (b) SEM image after 12 hours of milling

V. CONCLUSIONS

Copper-Alumina powders were successfully milled in high energy ball mill and their morphology was characterized. From the milling of 95%Cu with 5%Al_2O_3 for total duration of 15 hours, the morphology of the powders were studied through XRD and SEM analysis. The results of the analysis are listed below: 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 452.3 nm to 141.5 nm after 15 hours of milling, but a lowest particle size of 65.8 nm was achieved after 12 hours of milling. 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 of 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 is also enhanced. From SEM it could be concluded that the dominant process like cold welding and fracture occurred alternatively. During the 12 hours of milling the reduction in particle size is purely due to fracture and the increase in particle size after 15 hours of milling was due to cold welding.

REFERENCES

[1] D. Jeyasimman , S. Sivasankaran , K. Sivaprasad , R. Narayanasamy, R.S. Kambali “An investigation of the synthesis, consolidation and mechanical behavior of Al 6061 nanocomposites reinforced by TiC viamechanical alloying” Materials and Design 57 (2014) 394–404.

[2] S. Sivasankaran, K. Sivaprasad, R. Narayanasamy, Vijay Kumar Iyer“Synthesis, structure and sinterability of 6061 AA100−x–x wt.% TiO2 composites prepared by high-energy ball milling”Journal of Alloys and Compounds 491 (2010) 712–721.

[3] Mina Bastwros, Gap-Yong Kim, Can Zhu, Kun Zhang, Shiren Wang, Xiaoduan Tang,Xinwei Wang “Effect of ball milling on graphene reinforced Al6061 compositefabricated by semi-solid sintering” Composites: Part B 60 (2014) 111–118.

[4] K. Dash, B.C. Ray, D. Chaira“Synthesis and characterization of copper-alumina metal matrix composite by conventional and spark plasma sintering” Journal of Alloys and Compounds (2010)

[5] Linnan Du, Georg Mauer, Robert Vaben “Study on the effects of wet ball milling and boron nitrideadditive on Li-N-H hydrogen storage system” Energy Procedia 29 (2012) 147 – 155.

[6] Y.Y.Li, T.L.Ngai, D.T. Zhang, Y.Long, W.Xia “ Effect of die wall lubrication on warm compaction powder metallurgy”, Journal of Materials Processing Technology129(2002) 354-358.

[7] S.Sivasankaran, K. Sivaprasad, R.Narayanasamy, P.V.Satyanarayana,“X-ray peak broadening analysis of AA 6061100−x−x wt.% Al2O3nanocomposite prepared by mechanical alloying” Material Characterization 62(2011) 661-672.

[8] C. Machio, H.K. Chikwanda, and S. Chikosha “Effect of process control agent (PCA) on the characteristics of mechanicallyalloyed Ti-Mg powders” The Journal of The Southern African Institute of Mining and Metallurgy, VOLUME 111 MARCH 2011.