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Regular

Synthesis of *Al-Cu* Nanoparticles by Mechanical Alloying

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ABSTRACT : Nanocrystalline powder of *Al*-4wt. %*Cu* alloy has been prepared by mechanical alloying using a planetary ball mill. Stearic acid was used as a process control agent the alloy nanoparticles were studied by means of scanning electron microscopy (SEM) and energy dispersive X-ray analysis (EDXA). The SEM results revealed that the *Al-Cu* crystallite particle size decreased to 39-96 nm when milling time was 18 hours. The EDXA result confirmed that the nanoparticles produced were binary alloy of *Al-Cu*.

KEYWORDS : Nanoparticles; Mechanical alloying; X-ray analysis; *Al-Cu* alloy; Stearic Acid

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1 INTRODUCTION

One of the main area in nanotechnology is nanoparticle production. Some successful production of nanoparticle of metals in particular *Al* based alloys have been reported [1, 2]. A good combination of high strength and ductility of *Al* alloys have given the materials a wide spectrum of possible advanced application [3]. In particular *Al-Cu* alloys have been one of the common materials used for interconnects in modern integrated circuit technology [4].

A variety of methods were used to produce the metal nanoparticles such as laser ablation [5], sputtering [6], and mechanical alloying (MA) [1-3, 7-9]. Among the several approaches proposed, the used of MA has received a great attention. Alloying through solid state reactions of elemental powders, flexible method [7], low temperature processing and relatively inexpensive equipment [8] and capability of production on industrial scale are some of the important advantages of MA method.

Generally, to avoid adhesion of powder to the ball and vials during MA processing, certain materials which is called as process control agent must be added to the powder. The application of various process control agents has been reported, for example ethanol [1], stearic acid and ethyl acetate [2]. Amongst them, stearic acid is one of the most effective and very often used process control agents [10]. In this paper we report the use of mechanical alloying process and stearic acid to synthesize *Al-Cu* alloy nanoparticles.

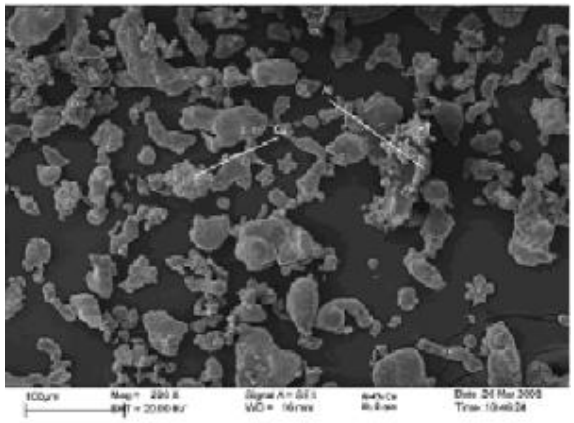

FIGURE 1: The SEM micrograph of *Al-Cu* powder mixture before ball milling

2 MATERIAL AND METHOD

Aluminium and copper powders of 99.9% purity with initial particle sizes of about 60 and 45 μm respectively were used. The powders were mixed to form a mixture with nominal composition of *Al*-4wt%*Cu*. A cylindrical stainless steel container and stainless steel balls were employed for milling the mixture under argon atmosphere using a Fritsch Pulverisette - 5 planetary type ball mill. The container volume was 250 ml and the balls size were 12 mm in diameter. The ball to powder weight ratio was fixed to 20 : 1. Stearic acid of about 2 wt.% of the powder mixture was used

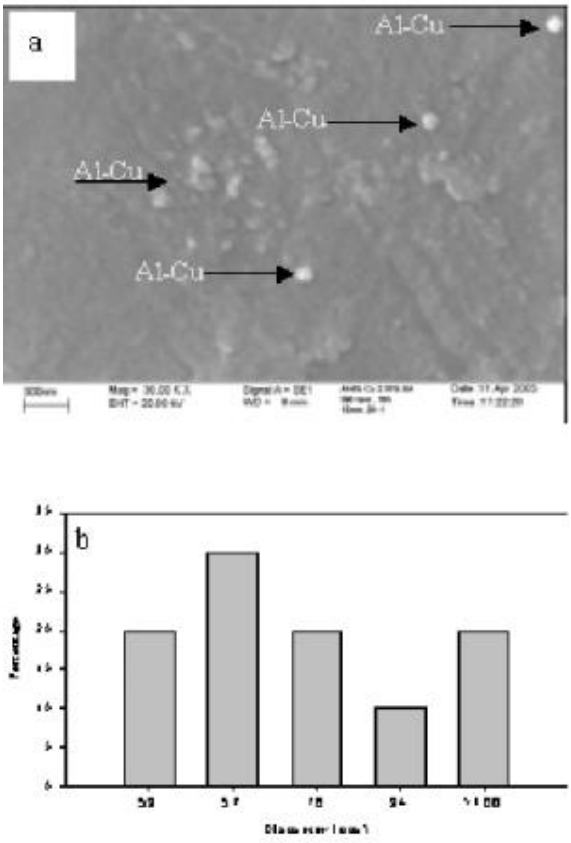

FIGURE 2: (a) The SEM micrograph of *Al*-4% wt. *Cu* powder ball-milled for 16 hours. (b) Histogram of particles size distribution estimated from the SEM image in Fig. 2a.

as a process control agent. The milling times were 16 hours and 18 hours, interrupted for 30 minutes for every 4 hours in order to minimize excessive temperature rise and to limit adherence of the powder to the container walls. A scanning electron microscope (SEM) Model INCA M. 7353 manufactured by Oxford Instrument was used to investigate the microstructure of the alloyed powder while the energy dispersive X-ray analysis was performed to determined its chemical content.

TABLE 1: The percentage of atomic concentration obtained from the EDXA pattern.

Element	**wt. (%)**	**σ wt. (%)**
C	17.35	0.51
O	2.15	0.14
Al	75.31	0.08
Cu	5.19	0.07

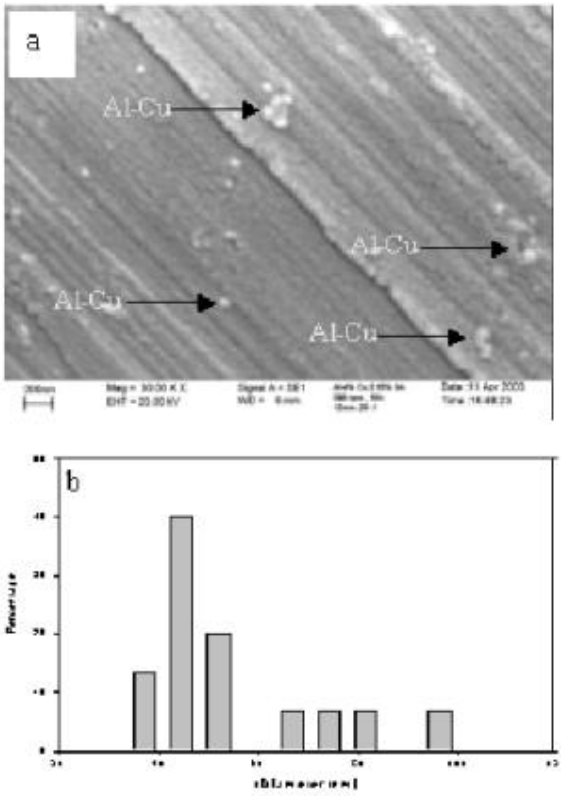

FIGURE 3: (a) The SEM micrograph of *Al*- 4% wt. *Cu* powder ball-milled for 18 hours. (b) Histogram of particles size distribution estimated from the SEM image in Fig. 3a.

3 RESULT AND DISCUSSIONS

3.1 Scanning electron microscopy

The size of powders in the mixture before ball milling was studied using SEM. Fig. 1 shows that the SEM image of a mixture of as- received *Al* and *Cu* powders. The maximum particles size of *Al* and *Cu* were about 60 μm and 45 μm respectively.

During ball milling, the particle size of elemental powder gradually decreased [9] as a result of deformation induced by collisions between the balls and powder particles. The decrease of particle size with the milling time as shown in Figs. 2 and 3 are in accordance with the result reported by Lu et al. [2]. Histograms in Fig. 2b and 3b revealed the distribution of the powders size in nanometers estimated from SEM micrographs of the mixtures which were ball-milled for 16 and 18 hours respectively. The particles produced after the milling can be considered as nanoparticles as their size are about 100 nm or less. The final size of the powders were in the range of 57 $\sim$ 113 nm and 39 $\sim$ 96 nm for milling times of 16 and 18 hours respectively.

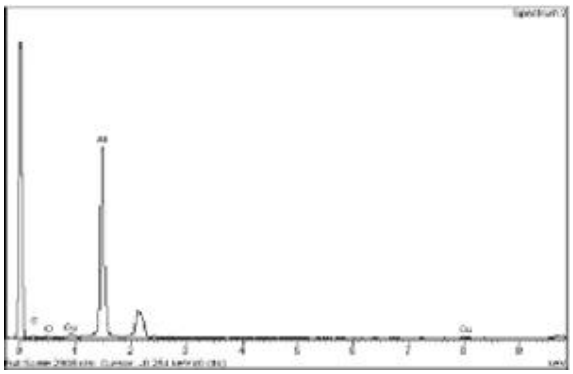

FIGURE 4: The powder energy dispersive X-ray spectrum of mechanically alloyed particles for 18 hours milling time.

3.2 Energy dispersive X-ray analysis

Fig. 4 shows the energy dispersive X-ray spectrum of particle after ball milling. *Al* and Cu were detected in the particle that infers mechanical alloying has taken place between *Al* and Cu particles to form particles of binary alloy of *Al-Cu*. The chemical composition of alloyed nanoparticles obtained by energy dispersive X-ray analysis is presented in Tab. 1. The existence of C and O in the sample is believed to be from the original materials [10] such as residual stearic acid (CH3 (CH2)16CO2H) which was used as a processing control agent. Besides C and O, there are no other impurities detected in the particles (Fig. 4). This indicates that the ball milling procedure does not introduce any spurious impurity [9].

4 CONCLUSIONS

In summary, ball-milling has successfully produced *Al-Cu* alloy particles with size in the range of 57-113 and 39-96 nm for milling time of 16 and 18 hours respectively. The particles size decreases with the increase in milling time. The energy dispersive X-ray analysis result has confirmed that the nanoparticles produced were particles of binary alloy of *Al-Cu*.

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Φips

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