SYNTHESIS OF NANO Al_2O_3 DISPERSION - STRENGTHENED Cu MATRIX COMPOSITE MATERIALS BY MECHANOCHEMICAL PROCESS

Nguyen Duc Duy[1,2], Tran Van Dung[1], Nguyen Dang Thuy[1], Ho Ky Thanh[1,3*]
[1]Ha Noi University of Science and Technology
[2]College of Mechanics and Metallurgy
[3]College of Technology - TNU

SUMMARY

Mechanochemical method was used to synthesize nano Al_2O_3 dispersion - strengthened Cu matrix composite materials. Nanocomposite powders of Cu - Al_2O_3 were produced by milling at room temperature in attritor mill using mixtures of CuO, Al and Cu powder ingredients. The nanocomposite powders Cu - Al_2O_3 were cold pressed into briquettes and then conventionally sintered at various temperatures (from 700°C to 900°C) and time (from 1 to 3 hours). The results were analyzed by x-ray diffraction (XRD) and scanning electron microscope (SEM) with energy dispersive spectrometer (EDS) showed that nano - sized Al_2O_3 ultrafine particles of about (50 ÷ 100) nm was formed and uniformly dispersed into Cu matrix. The study results showed that the effect of pressed - sintered parameters on microstructure and properties of composite Cu - nano Al_2O_3 dispersed such as microstructure and density, hardness. Also, the results demonstrated that nano Al_2O_3 can be able to synthesize by a mechanochemical process in attritor balls milling.

Keywords: *mechanochemical, Cu-Al_2O_3, dispersion, X-ray diffraction, SEM, microstructure*

INTRODUCTION

Composites are materials consisting of two or more components with different properties and distinct boundaries between them. Intensive research over the last two decades has led to the emergence of composites as a new class of engineering materials that provide enhanced combination of high temperature strength and acceptable levels of ductility, toughness, fatigue resistance required for a variety of applications. Metal matrix composites (MMCs) are one of the groups of such type of materials. Copper based metal matrix composites are being used in many industrial applications such as contact supports, electrode materials for lead wires, spot welding and others [1].

Dispersion strengthened Cu - Al_2O_3 composite materials are extensively used as materials for products which require high-strength and electrical properties, such as electrode materials for lead wires and spot welding, relay blades, contact supports that require high strength at a high temperature, wear-resistance for electrical discharge as well as electrical properties and bearing materials for industry [2]. Studies on the synthesis and characterization of nano-scale alumina dispersed copper metal matrix composites have been attracting scientific interest in recent years, since nanostructure-type materials are expected to have special physical and mechanical properties. In the copper- alumina system, the nano-scale Al_2O_3 particulate dispersion provide unique characteristics, such as high thermal and electrical conductivities, as well as high strength and excellent resistance to high temperature annealing. The main requirement for structure of dispersion strengthened materials is homogeneous distribution of very fine oxide paricles (dispersoids) in the copper matrix [3,4]. In nanocrystalline materials, the main role of the dispersoids is to limit grain growth at elevated temperatures and to attain a very small grain size, resulting in high strength due to the fine-grain strengthening mechanism [5].

In this study, Cu - Al_2O_3 nanocomposites have been fabricated by mechanochemical

[*] *Tel: 0984 194198, Email: hkythanh@tnut.edu.vn*

process, wherein the alumina contents were adjusted represents 5 vol.%. Some of mechanical and physical properties such as microstructure and density, hardness of Cu-Al_2O_3 composite was the object of this paper. Also, the results demonstrated that nano Al_2O_3 can be able to synthesize by a mechanochemical process in attritor balls milling.

EXPERIMENTAL PROCEDURE

The starting materials used in this work were CuO (purity ≥ 99.0%, average size about 40÷50 μm), Cu (purity ≥ 99.7%, average size about 40÷50 μm) and Al (purity ≥ 99.7%, average size about 40÷50 μm) powders. For in-situ powder composite, mixed powder Cu-CuO-Al was milled in range of 16 hours in argon atmosphere in the attritor ball mill at 720 rpm speed to creat mixture powder Cu-20vol.% Al_2O_3 nanocomposites. Then, we add Cu powder into the Cu-20vol.%Al_2O_3 mixture was milled for 3 hours in the drum ball mill at 300 rpm speed to produce homogeneous mixture of Cu-5vol.%Al_2O_3 powder.

Then, the nanocomposite powders were cold pressed into the compaction mold at pressures 200 to 400 MPa. The compact samples were conventionally sintered at various temperatures (from 700^{o}C to 900^{o}C) and time (from 1 to 3 hours). The compact samples were characterized for phase analysis by X-ray diffraction (XRD) and scanning electron microscope (SEM) with energy dispersive spectrometer (EDS). Sintered density of the compact samples was determined by Archimedes method and microhardness of the compact samples was measured by Vickers hardness tester.

RESULTS AND DISCUSSION

Results after milling

Assumptions about the impact stages of the strain energy in the milling process is given as follows:

Stage 1: The first stage of the process of milling: the effect of shear stress, the metal powder particles slide on the contact surfaces, the phenomenon and results may occur as follows:

1. There is the phenomenon of cold welding between the Cu particles together to form larger particles of Cu;

2. On the surfaces, slip occurs between Cu and Al have process to creat Cu[Al];

3. On the contact surfaces between CuO and Al, reaction will occur:

$$CuO + Al \rightarrow Cu + Al_2O_3$$

Stage 2: Stage milling stability: three original constituents Cu, CuO and Al along with Al_2O_3, Cu[Al] and the newly formed Cu will combine to form a particle with Cu base. Figure 1 shows the grain surface of the powder mixture after milling 16h. Under the impact of intense plastic deformation in the milling chamber, the contact surfaces are formed within a particle, the reaction occurs. This reaction process can be referred to as inter oxidation reactions and including the reaction between Cu[Al] and CuO. It is the process of diffusion of Al replacing the oxygen atoms in the crystal lattice of CuO. This diffusion process, essentially a reaction is oxygen atom positions in the crystal lattice of Cu with Al_2O_3 phase.

Stage 3: Destroyed stage of Cu-Al_2O_3 powder particles combination: prolonged deformation process born dislocation leading to the destruction of the particle composite materials Cu-Al_2O_3.

With its above mode, after milling powder particle size was significantly reduced. SEM imaging results (Figure 1) shows that the average size of the original powder particles is 40 - 50 µm, 16h after grinding, can be seen clearly that the particle size of nanometer-sized powder achieved.

Microstructure and phase analysis

The Cu-20vol.%Al_2O_3 nanocomposites was prepared by the mechanochemical in the attritor ball milling with phase transformations of the precursors. The preparation is described in detail in our works [6]. Obtained by in-situ Al_2O_3 particles are ultrafine - about 50÷100 nm in diameter.

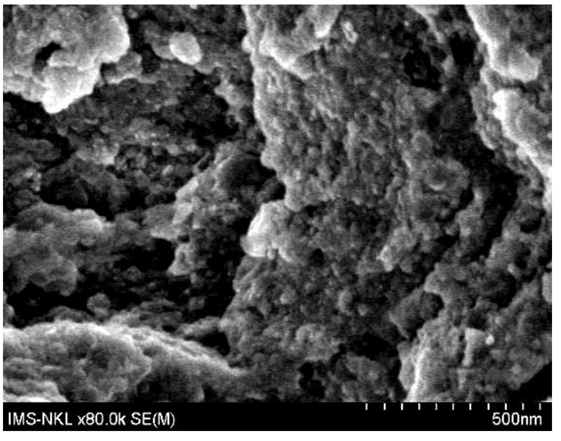

Figure 1. *SEM images of the powder sample after milling 16h*

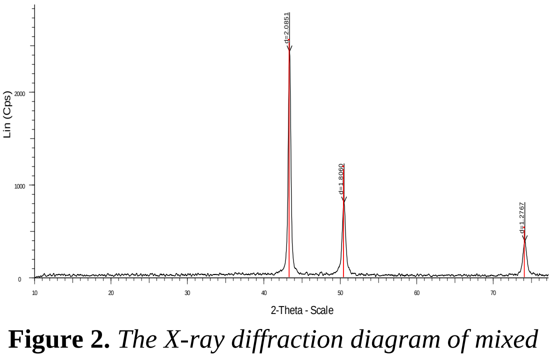

Figure 2. *The X-ray diffraction diagram of mixed powder material samples, Cu-5vol.%Al_2O_3*

The Figure 2 shows the results of X-ray diffraction diagram analysis of mixed powder material samples Cu-5vol.%Al2O3, that only the appearance of the peak of Cu phase, the peak of Al2O3 phase difficult to determine, due to concentration and small size.

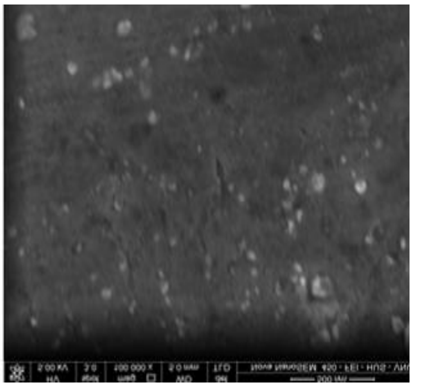

Figure 3. *SEM images of the sample Cu-5vol.%Al_2O_3 sintered at 800^{o}C, 2 h*

With the results of the analysis SEM as shown in Figure 3, Al_2O_3 phase created and dispersed homogenous in the Cu matrix. But here also to note further, in the process of milling, the grain of the constituents will be smaller, leading to the intensity of the peaks are reduced, and the width of the peak increases.

Figure 4 shows images of SEM-EDS of the sample sintered Cu-5vol.%Al_2O_3. The SEM image indicates fairly homogeneous distribution of Al_2O_3 in Cu matrix. EDS image scan indicates that uniform distribution of Cu, Al and O elements all over surface. The level of copper is much higher than that of aluminum and oxygen. The EDS results revealed locations with a relatively high concentration of Al and O elements. These locations were thought as being "Al_2O_3-rich" which may represent the presence of a third phase of $CuAlO_2$ at interface between alumina particles and copper crystallites matrix.

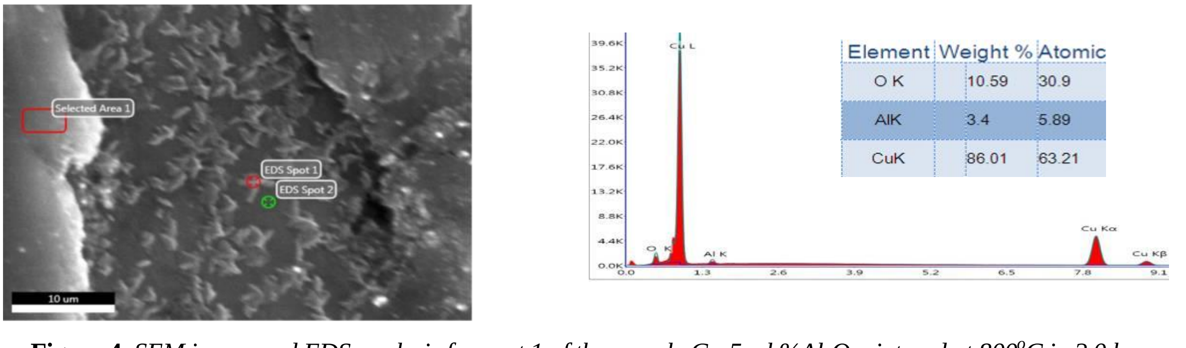

Figure 4. *SEM image and EDS analysis for spot 1 of the sample Cu-5vol.%Al_2O_3 sintered at 800^{o}C in 2,0 h*

Table 1. *Microhardness and Relative density of composite Cu-Al_2O_3*

Composites	Relative density (%)	Microhardness (HV)
Cu (pure)	95.2	58.9
Cu-5vol.%Al_2O_3	80	75
Cu-20vol.%Al_2O_3	75	120

Physical and mechanical properties

Density is very important information of materials, especial in metal and powder metallurgy engineering. The density is much related to the mechanical properties, friction... of material. We usually use Archimedes rule to estimate the density.

Hardness is ability of materials to prevent some deformation such as elastic deformation, and bent by reacting with other objects. The higher hardness, the harder material to be deformed.

The results of relative density and microhardness of the samples of Cu-Al_2O_3 with different alumina content, sintered temperature at 800^{o}C are showed in the table 1.

The result density and hardness of the material Cu-Al_2O_3 shows that, when Al_2O_3 dispersed in the Cu matrix, the hardness of Cu-Al_2O_3 material much larger than sample blocks are pressed sintered from pure Cu powder, with relative density of about 95%, particle size less than 1 µm crystal, hardness of about 60 HV. This demonstrates that Al_2O_3 dispersed phase is very effective hardening. Thereby, it can be stated effective hardening of Al_2O_3 phase in Cu-Al_2O_3 composite materials, the hardness of the sample microstructure Cu-Al_2O_3 nanocomposite materials was quite high compared to pure Cu. The study results showed that the effect of pressed - sintered parameters on microstructure and properties such as microstructure and density, hardness of composite copper matrix - nano Al_2O_3 dispersed [6].

CONCLUSIONS

Composite copper matrix - nano Al_2O_3 dispersion were produced by a mechanochemical methods. The results analysis such as micro-structural and some technological characteristics of the sample Cu-Al_2O_3 materials to draw the following conclusions:

- The process of manufacturing nano composite Cu-Al_2O_3 by mechanical methods which have already been set up as perfectly reasonable.

- Experimentally is possible to synthesize nano-Al_2O_3 dispersed phase in the Cu matrix with nano-sized ultrafine particles of about 50÷100 nm.

- Hardness of Cu-Al_2O_3 materials increased significantly compared to pure Cu material. Confirm the effectiveness of hardening Cu with dispersed Al_2O_3 phase.

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TÓM TẮT

TỔNG HỢP VẬT LIỆU TỔ HỢP NỀN Cu - NANO Al_2O_3 PHÂN TÁN BẰNG PHƯƠNG PHÁP CƠ HÓA

Nguyễn Đức Duy[1;2], Trần Văn Dũng[1], Nguyễn Đặng Thủy[1], Hồ Ký Thanh[1;3*]

[1]Trường Đại học Bách khoa Hà Nội, [2]Trường Cao đẳng Cơ khí Luyện kim Thái Nguyên

[3]Trường Đại học Kỹ thuật Công nghiệp – ĐH Thái Nguyên

Vật liệu tổ hợp nền Cu - nano Al_2O_3 phân tán đã được tổng hợp bằng phương pháp cơ hóa kết hợp. Hỗn hợp bột vật liệu tổ hợp Cu - nano Al_2O_3 tổng hợp bằng quá trình nghiền trộn hỗn hợp thành phần vật liệu bột CuO, Al và Cu trong máy nghiền bi kiểu cánh khuấy. Hỗn hợp bột vật liệu tổ hợp Cu - nano Al_2O_3 được ép đóng bánh và sau đó thiêu kết ở nhiệt độ khác nhau (từ 700^oC đến 900^oC) và thời gian (từ 1 đến 3 giờ). Kết quả phân tích nhiễu xạ tia X (XRD) và hiển vi điện tử quét độ phân giải cao (SEM, EDS) cho thấy rằng các hạt Al_2O_3 siêu mịn với kích thước khoảng (50 ÷ 100) nm được hình thành và phân tán đồng đều trong nền Cu. Kết quả nghiên cứu cũng cho thấy ảnh hưởng của các thông số công nghệ trong quá trình ép - thiêu kết đến cấu trúc và tính chất của vật liệu tổ hợp Cu - nano Al_2O_3 như: tổ chức tế vi, mật độ và độ cứng. Ngoài ra, kết quả nghiên cứu cũng chứng minh rằng nano Al_2O_3 có thể tổng hợp bằng phương pháp cơ hóa trong máy nghiền bi kiểu cánh khuấy.

Từ khóa: *cơ- hóa, Cu-Al_2O_3, phân tán, nhiễu xạ tia X, hiển vi điện tử quét, cấu trúc tế vi*

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Phản biện khoa học: *PGS.TS Ngô Như Khoa - Trường Đại học Kỹ thuật Công nghiệp - ĐHTN*

[*] *Tel: 0984 194198, Email: hkythanh@tnut.edu.vn*