OPTOELECTRONICS AND ADVANCED MATERIALS – RAPID COMMUNICATIONS Vol. 7, No. 5-6, May - June 2013, p. 406 - 410

Microstructure and mechanical properties of PM Fe-Cu-Al_2O_3 alloys

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The purpose of this study was to investigate the effect of Cu addition on the microstructure and mechanical properties of PM Fe-Cu-Al_2O_3 alloys produced using hot-pressing method. Copper powder was added to iron powder at 5 wt.%, 10 wt.% and 15 wt.% rates. Production of Fe-Cu-Al_2O_3 alloys was carried out under pressure of 35 MPa, at 750, 800 and 850 °C, and for a sintering time of 4 minutes. Microstructure phase composition, relative density, hardness, and transverse rupture strength of the alloys were investigated. Phase composition and microstructure of the alloys were characterised by optical microscopy, X-ray diffraction and scanning electron microscope (SEM-EDS) techniques. The transverse rupture strength (TRS) of the samples was assessed by means of a three-point bending test. Hardness of Fe-Cu-Al_2O_3 alloys changed between 57 HB and 110 HB depending on the amount of Cu and sintering temperature. Results show that the TRS of the alloys decreased together with the increase in the amount of copper, while it increased with increasing sintering temperature.

(Received February 28, 2013; accepted June 12, 2013)

Keywords: PM, Fe-Cu-Al_2O_3 alloy, Sintering, Microstructure, TRS

1. Introduction

Powder metallurgy (PM) process is more commonly used in manufacturing engineering materials day by day. The developments in powder technology enable manufacturing complex shaped machine parts in high manufacturing speed, high quality, low dimensional tolerance and economically through different pressing techniques. One of the basic advantages of PM manufacturing technique is to be capable of manufacturing materials, which are difficult to be manufactured using other manufacturing methods [1]. PM method consists of the steps of pressing metal powders inside a mould, bonding powder particles with each other, and sintering. This method is also a large process which several materials are manufactured by pressing-sintering or hot pressing. Not only simple cylindrical shafts and bearings but also complex shaped parts such as filters, gears can be conveniently manufactured through powder metallurgy method [2]. Pressing the parts to be manufactured using PM method is usually performed at room temperature. Recently, part manufacturing by hot pressing is made also in environments above room temperature. It is observed that higher density is obtained by less mould-metal powder interface friction, in less pressing pressures by using this method [3].

In this century, Fe-Cu powder materials have been widely used for antifriction and construction materials. These materials are characterised by sufficient strength and plasticity in combination with high corrosion resistance and heat conductivity. While iron provides strength to alloy, copper gains ductility in these materials. These materials are important in ferrous powder metallurgy as diffusion alloyed industrial iron powders have typically a copper content of 1.5% in weight, but compositions as high as 25% of weight of Cu might also be found commercially available [4]. These materials lost the manufacturers' interest due to problems such as excessive shrinkage and losing mechanical properties during manufacturing phase of these materials by liquid phase sintering [5].

The purpose of this study is to investigate the effect of copper addition and sintering temperature on microstructure and mechanical properties of PM Fe-Cu-Al_2O_3 alloys manufactured by hot pressing method. Solid phase sintering was preferred in order to avoid shrinkage problem in the material at the sintering phase.

2. Experimental studies

In order to manufacture PM Fe-Cu-Al_2O_3 alloys, iron powder with 99.9 percent (pct) purity and 6.8 µm particle size, copper powder with 99.9 percent (pct) purity and <63 µm particle size and alumina oxide (Al_2O_3) with 99.9 percent (pct) purity and 1 µm particle size were used as starting materials. Cu powder was added into Fe powders at 5 wt.%, 10 wt.%, and 15 wt.%. Al_2O_3, on the other hand, was added to the matrix in very small quantities (1 wt.%) in order to prevent grain coarsening and fill the pores due to small grain sizes [6]. Iron, copper, and alumina oxide powders were mixed with the addition of 1 wt.% of paraffin wax, at speed of 20 rpm, for 30 minutes using a ∞ shaped rotary mixer. Then, powder samples were hot pressed in graphite moulds for 4 min at 750, 800 and 850 °C, with an applied pressure of 35 MPa using an automatic hot pressing machine. The relative densities of alloys were measured according to Archimedes' principle. Hardness measurements were performed in Brinell scale with a ball diameter of 2.5 mm and a load of 62.5 kg. In order to determine the transverse rupture strength (TRS) of the alloys, three-point bending tests were performed using a Schimatzu universal testing machine. Samples for metallographic examination were prepared using standard polishing techniques. An optical microscopy, scanning electron microscope (SEM) fitted with energy dispersion X-ray spectroscopy (EDS), and X-ray diffractometer (XRD) were used to investigate microstructure and fractured surfaces of samples.

3. Results and discussion

PM Fe-Cu-Al_2O_3 alloy was successfully produced using the hot pressing method together with a 4-minute sintering time at 750, 800 and 850 °C, under pressure of 35 MPa. Fig. 1 illustrates optical microstructures of Fe-Cu-Al_2O_3 alloys sintered at 750 and 850 °C. When we examine the microstructure images, the existence of three different areas is noted. The light-coloured parts are copper, the dark-coloured parts are the pores, and the medium dark coloured parts are iron. Aluminium oxide cannot be seen in the optical images because it is in very small quantities and in the size of a very small grain. Copper is positioned in network and locally in the microstructure. It is seen from the images that as the proportion of copper in samples increase, copper is more spread among the iron grains. Another matter which can be understood from the images is that copper which remains among iron grains cannot fairly be spread among the grains and it rather remains in large lumps. This is because the liquidity of copper is lower due to the non-performance of liquid phase sintering. It is seen that copper is spread better and in thin structures among iron grains because the sintering temperature is higher. It can be easily seen from the microstructure images that there is decrease in the size and quantity of the pores which are found in the microstructure along with the increase in the sintering temperature and the quantity of copper addition. Copper serves as a filler in sealing the pores [7].

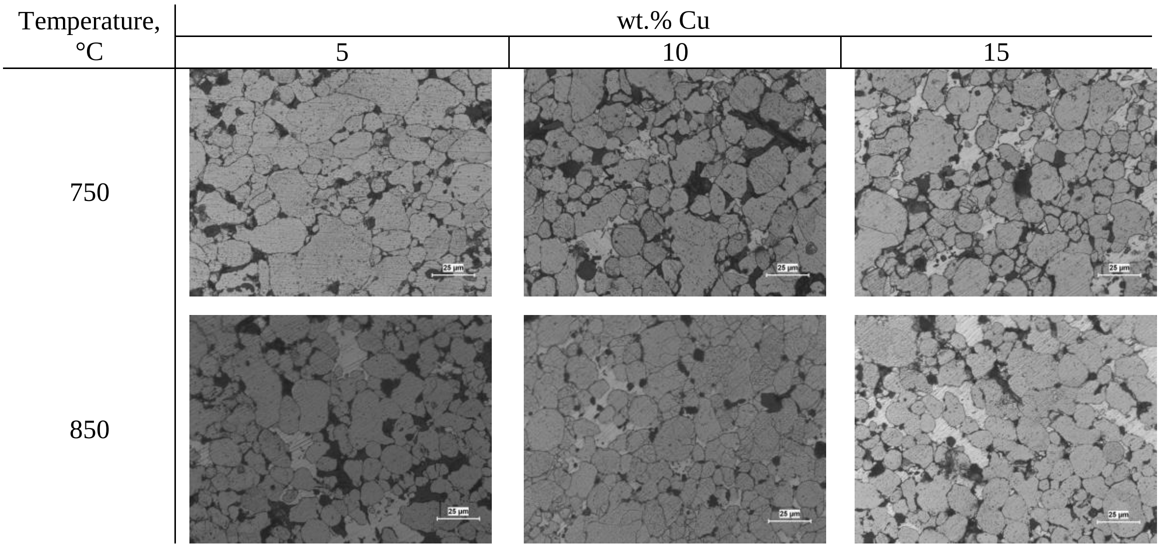

Fig. 1. Optical micrographs of Fe-Cu-Al_2O_3 alloys sintered at 750–850 °C.

Fig. 2 illustrates the XRD graph of 10 wt.% Cu added alloy which is sintered at 750-800-850 °C. Results revealed that seven phases emerged in the alloy. These phases are $Cu_{40}Fe_{60}$, Fe_2O_3, Fe_3O_4, Fe_3Al, Cu_3Al_2, CuO and Al_7Cu_2Fe. It is observed that as sintering temperature increases, there is an increase in the severity of the peaks of oxide phases. This is associated with the fact that iron and copper are more inclined to oxidization in higher temperatures [8].

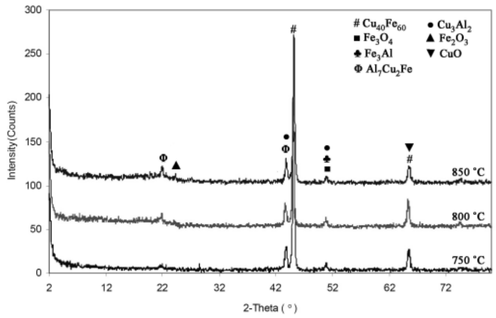

Fig. 2. XRD diffraction patterns of Fe-Cu-Al_2O_3 alloys.

In order to obtain information about the condensability properties of Fe-Cu-Al_2O_3 alloys, the relative density of the alloys was measured. Theoretical and sintered densities were used for calculating relative densities. Theoretical densities of alloys were calculated using following Eq. (1);

$$\frac{1}{\rho_a}=\frac{w_1}{\rho_1}+\frac{w_2}{\rho_2}+\frac{w_3}{\rho_3} \qquad (1)$$

where ρ is density, w is weight fraction, and subscripts a, 1, 2 *and* 3 refer to the alloy and components, respectively [9]. Fig. 3 illustrates the effect of sintering temperatures and copper content on relative densities of Fe-Cu-Al_2O_3 alloys. The relative densities of the alloy increased as copper addition rate increased. A denser structure was provided by filling the large quantities of copper pores. There was an increase in the relative densities as sintering temperature increased. As temperature increased, the movement of solid particles within the alloy increased due to solid state diffusion and thus a dense alloy was obtained [10-12]. The highest relative density was obtained at 15 % of Cu addition and sintering temperature of 850 °C with a value of 96 %.

Hardness values of the alloys were determined by taking mean of five different measurements from each sample. Fig. 4 illustrates the hardness graph of Fe-Cu-Al_2O_3 alloy. As understood from the graph, there were significant increases in the hardness values of the alloys along with the increase in sintering temperature. This results from the higher condensation of the samples along with the temperature increase. The hardness values of samples sintered at 750 °C are very close to each other and 60 HB in average. The hardness values of samples sintered at 800 °C, on the other hand, continued to increase compared to each other but the hardness value of 15 % Cu content sample slightly decreases. The hardness values of the samples in this group are approximately 65-70 HB in average. The hardness values of the samples sintered at sintering temperature of 850 °C have reached up to 110 HB in average. However, the hardness value of 15 % copper content sample remained lower compared to other two samples. This is associated with the high copper ratio in the alloy; and the result is normal and as expected. In addition, the hardness values of Fe-Cu-Al_2O_3 alloys decreased as copper addition ratio increased. This can be associated with the copper's being ductile.

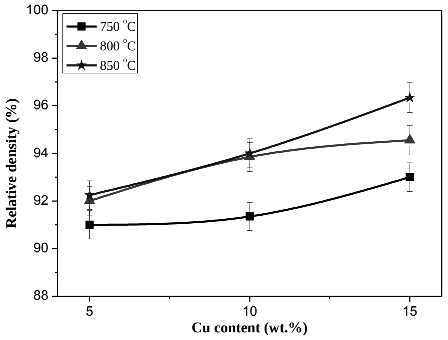

Fig. 3. The variation of relative density as a function of Cu.

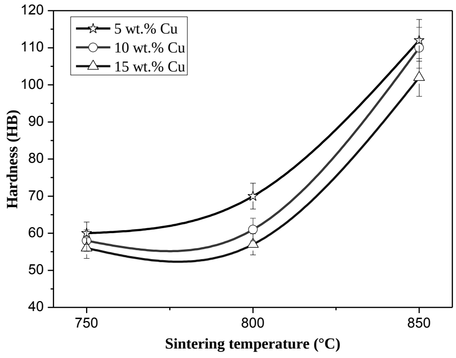

Fig. 4. Hardness of Fe-Cu-Al_2O_3 *alloys.*

Fig. 5 illustrates the bending graphs of Fe-Cu-Al_2O_3 alloys which were sintered at 750, 800 and 850 °C. Examining the TRS graph of the samples sintered at 750 °C in Figure 5a, it is observed that all samples are broken in the same load but their % extension values vary based on the copper ratio in the sample. This result is as expected because the copper is a ductile material and it increased the ductility of the sample. However, there has been no variation in the bending strengths of the samples. The bending load of all of the three samples was approximately 400 MPa with a slight difference. The extensions of the samples at sintering temperature of 750 °C was approximately 4.5 % in 5 % Cu addition, 5.5 % in 10 % Cu addition and 5.7 % in 15 % Cu addition. A significant result obtained here is that if it is desired to increase the extension of the alloy provided that its bending strength remains the same, this can be made by changing the copper ratio of the material.

It can be easily seen in Fig. 5b that there is a slight increase in the bending strengths of the alloys when sintering temperature is increased up to 800 °C. Alloy with 15 % copper ratio has the highest bending strength which is followed by alloys with 10 % and 5% copper ratio. As the copper ratio increases, extension % values increase, as well. 15 % Cu added alloys has the highest extension while 5 % Cu added alloy has the lowest extension.

Fig. 5c illustrates TRS graphic of Fe-Cu-Al_2O_3 alloys sintered at 850 °C. It is understood that increase in sintering temperature caused a significant increase in the bending strength when this graphic is compared to previous graphics. While the highest strength value is 400-450 MPa at sintering temperatures of 750 and 800 °C, it is about 800 MPa at sintering temperature of 850 °C. More bonding has occurred between iron and copper along with the increase in the sintering temperature. And this has resulted in the increase of TRS values. Moreover, the increase in the sintering temperature and the decrease in the amount of pores cause the increase in TRS value [13]. Very significant differences have occurred in the amounts of extension as well. While the amount of extension does not exceed 6 % in low temperatures, this is between 12-20 % in the alloys which are produced at sintering temperature of 850 °C.

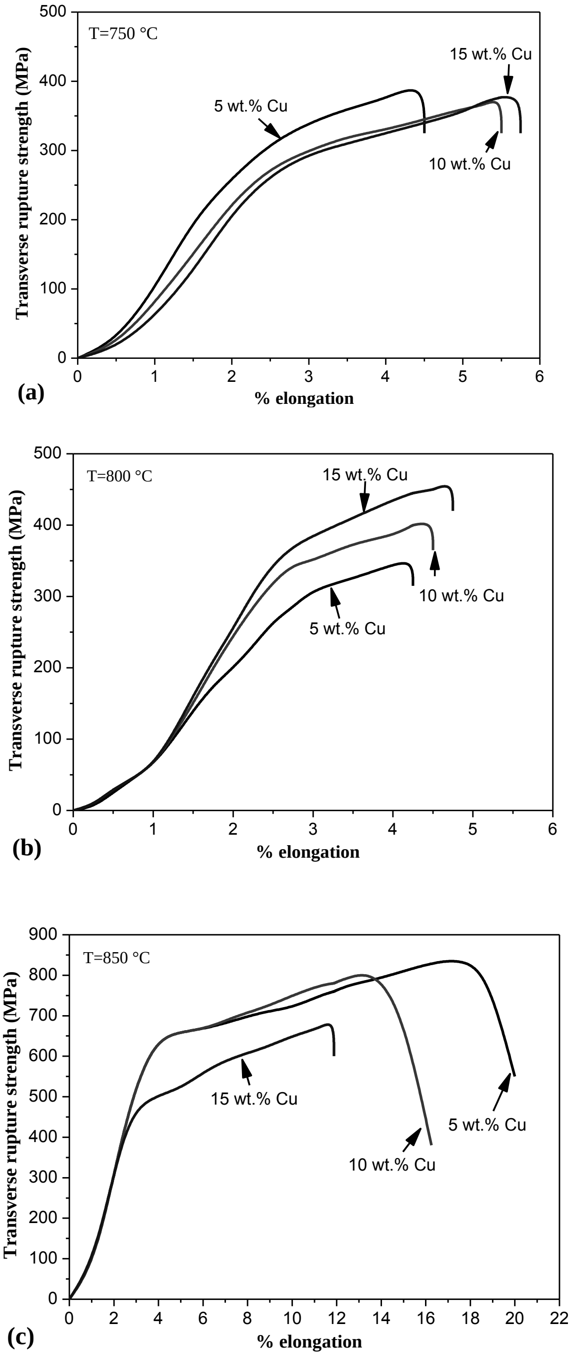

Fig. 5. TRS graphics of Fe-Cu-Al_2O_3 alloys sintered at: (a) 750 °C, (b) 800 °C, and (c) 850 °C.

Fig. 6 illustrates the SEM image of the fractured surface of 15 wt.% Cu added alloy sintered at 850 °C. While ductile fracture which looks like a spongy appearance is observed in some parts of the surface, brittle fracture is observed in some parts. The presence of some cracks on the fractured surface also gives clues about the presence of brittle fracture. Moreover, some metal powder grains have also remained in their place and preserved their integrity and fracture has occurred completely in the grain surface.

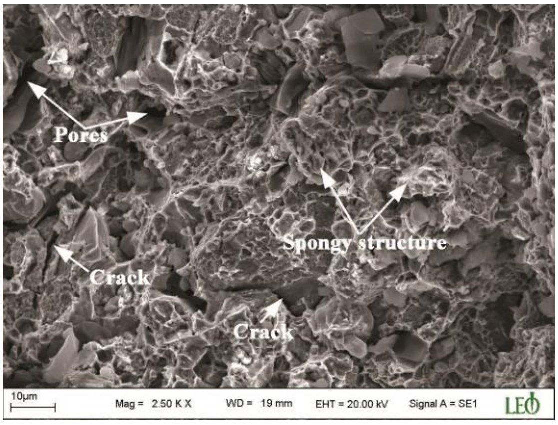

Fig. 6. The SEM image of the fracture surface of the Fe-Cu-Al_2O_3 alloy containing 15 wt.% Cu sintered at 850 °C.

4. Conclusions

PM Fe-Cu-Al_2O_3 alloys were successfully produced using a hot pressing method together with a four-minute sintering at 750, 800 and 850 °C, under pressure of 35 MPa. It can be understood from the microstructure images that copper enters among the iron grains and serves as good filler. Grain coarsening at high sintering temperatures was prevented by a little amount of Al_2O_3 addition. According to XRD analyses, $Cu_{40}Fe_{60}$, Fe_2O_3, Fe_3O_4, Fe_3Al, Cu_3Al_2, CuO, and Al_7Cu_2Fe phases formed in the microstructure. As the relative densities of the alloys increased, their hardness decreased as copper addition increased; and both relative densities and hardness increased as sintering temperature increased. The highest relative density was reached in 10 % Cu addition and at sintering temperature of 850 °C with 96 % value. The highest hardness was obtained at 5 and 10 % Cu rate and at sintering temperature of 850 °C with 110 HB value. TRS values of the alloys increased with the increase of the sintering temperature, and decreased with the increase of copper addition.

Acknowledgments

The study was supported by the Firat University Scientific Research Projects Unit (Project no: FÜBAP–1604).

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