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VARIATION OF MECHANICAL PROPERTIES OF COPPER COMPACTS PROCESSED IN HIGH ENERGY BALL MILLING

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ABSTRACT

A pure copper powders were high energy milled for 1, 3, and 5 hours in addition to non-milled copper. Each case was divided into two groups to be sintered at 825º and 900º C. Mechanical properties of copper consolidated compacts were characterized to determine the effects of milling time and sintering temperature on modulus of elasticity, yield strength, ultimate strength, hardness, and surface resistance to mechanical wear. Powders which milled for one and three hours show enhancements in all properties (except wear resistance) when compared to non-milled powder. Put, powders which milled for five hours show even near or lower values compared to non-milled powder. Specimens sintered at 825º C have higher values of a specific property when compared to specimens sintered at 900º C, regarding their milling is the same. Wear resistance for non-milled specimen is better of the all, and specimens sintered at 900º C have higher resistance to mechanical wear more than those sintered at 825º C.

Keywords: copper compacts, planetary ball milling, powder metallurgy, sintering, milling time.
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1. INTRODUCTION

In global production of metallic powders, copper and copper alloys powders come secondly after iron and iron alloys powder, and are the first of non-ferrous powders in producing various types of products exploiting powder metallurgy technology [1]. Copper compacts were produced either as pure, or a matrix for composite materials reinforced by ceramics, oxides, carbon nanotubes, etc. [2-4]. Mechanical and structural properties of sintered parts like amount of fracture energy absorption, yield strength, modulus of elasticity (Modulus values diverse from about 0.1 to 0.95 of the modulus of full dense copper), hardness, Poisson's ratio [5-7], are affected mainly by process parameters, and they are generally a function of resulting density which proportionate directly to process parameters (time and temperature of sintering, compacting stress, etc.). Also, according to porosity proportions, the density of sintered products may vary from intentionally designed to be highly porous materials as the needs of applications until reaching a near fully dense parts [7,8]. Type of compacting technique makes a difference, i.e. single press, double press, isostatic press. Sintering using traditional electrical furnaces (with/without protection gas) makes procedure easy and less expensive but gives more chance to grain growth because of prolonged time, while a more complicated sintering techniques like spark plasma may shorten time to about 10 minutes [9]. Many mathematical models about sintering and compacting were predicted to approach their thermal and mechanical situations in the preparation processes [10,11]. Another factor that alters the properties of consolidated products is size and shape of powder particles, i.e. micro, sub-micro, nano; and spherical, irregular, dendritic, etc. and mainly affects mechanical properties, and behavior mechanics [8].

2. MATERIALS AND PRACTICAL WORK

Copper powder (MERCK-Germany) with mean particle size < 63μm is used, particles shape is dendritic (produced by electrolytic deposition). Chemical specifications shown in table.1

Table 1 chemical specifications of copper powder (Merck KGaA [12]).

Assay (complexometric)	**Substances insoluble in nitric acid**	**P (phosphorus)**	**Ag (Silver)**	**As (Arsenic)**	**Fe (Iron)**	**Mn (Manganese)**	**Pb (Lead)**	**Sb (Antimony)**	**Sn (Tin)**
≥ 99.7 %	≤ 0.02 %	≤ 0.001 %	≤ 0.002 %	≤ 0.0005 %	≤ 0.005 %	≤ 0.001 %	≤ 0.01 %	≤ 0.001 %	≤ 0.01 %

Powder is divided into four groups and processed by planetary ball milling machine for 1, 3, and 5 hours, in addition to non-milled group (0 hr.). Stainless steel balls are used with balls-to-powder ratio (BPR) = balls/powder weight = 4.34:1, and frequency of planetary ball milling device = 250 rpm in ambient temperature and sealed jars but no special protection atmosphere. Powder is then compacted using a double plungers compacting die with general shape shown in figure 1 by 174 MPa compacting stress in hydraulic compressing machine (figure 2-a). Samples of compression test (figure 2-c,d) have about 8mm diameter and 11mm height, and 2.75mm height of samples for hardness test (figure 2-b). Each group of compacted copper is divided into two sub-group for sintering. One is sintered at 825º and other is sintered at 900º C. Sintering is done using electrical resistance tube furnace in protected atmosphere by argon flow. Purging with argon gas starts with furnace running from room temperature. After reaching specified temperature sintering is performed for 50 minutes.

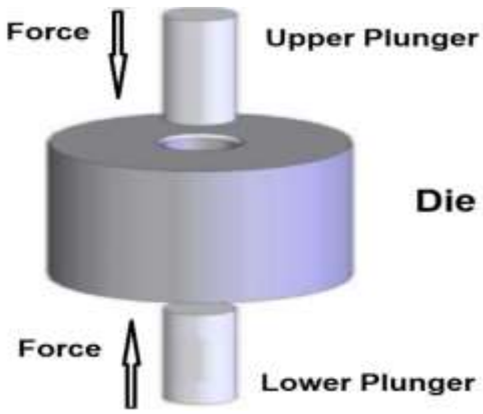

Figure 1 schematic of double plungers compacting die.

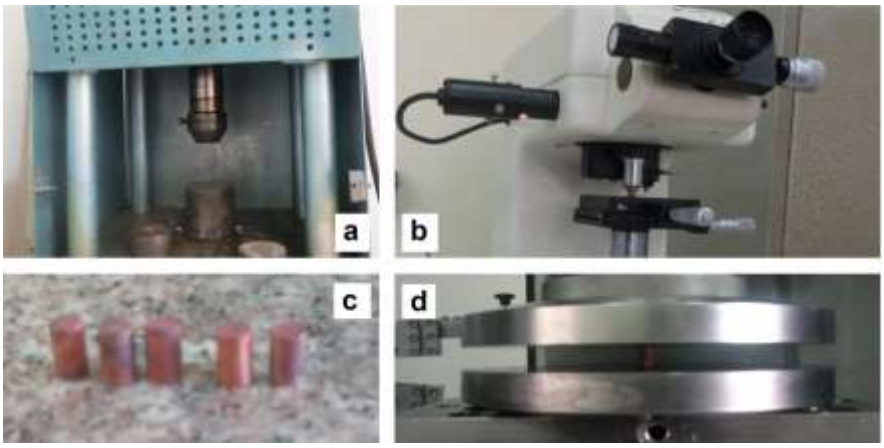

Figure 2 hydraulic press machine(a), Vickers hardness tester(b), consolidated copper compacts(c) and, compression test(d).

3. RESULTS AND DISCUSSIONS

After sintering, a series of mechanical tests were conducted to evaluate response to change milling time and sintering temperature. Compression test is used to determine yield and ultimate strength at compressive stresses, and also modulus of elasticity. Surface hardness is determined by Vickers hardness. Finally, wear test is performed to all samples in same test conditions to find which is more resistant by compare between them depending on weight losses.

Figures 3 and 4 show the effect of milling time and sintering temperature on compressive yield strength and ultimate compression strength; respectively. Generally they have the same behaviors; first the yield and ultimate strength start to increase with increasing milling time, for both sintering temperatures, and then begin to fall down. This can be explained in light of the following; the less time high energy milling well mixes and homogenizes structures, also producing some extent of particle refining and leads to direct enhancement in mechanical response. It is well known that more increase in milling time will lead to agglomeration effects instead of refining and produce much structurally defected grain as a result for prolonged heavy mechanical stresses. In both figures the lower sintering temperature (825° C) gives greater value of property (yield and ultimate) than higher temperature (900° C), and this difference seems to be a constant feature for all milling periods. This difference can be explained in view of grain growth and softening effect of high temperature heating. By sintering in temperature more than 0.6-0.7 Tm (here is 0.81 and 0.865 of Tm), a multiple physical and chemical processes are activated by virtue of diffusion including releasing residual stresses, recrystallization, grain growth, etc. and these processes relate directly to sintering temperature and sintering time, so, higher temperature means much grain growth, softening, and reduces mechanical strength.

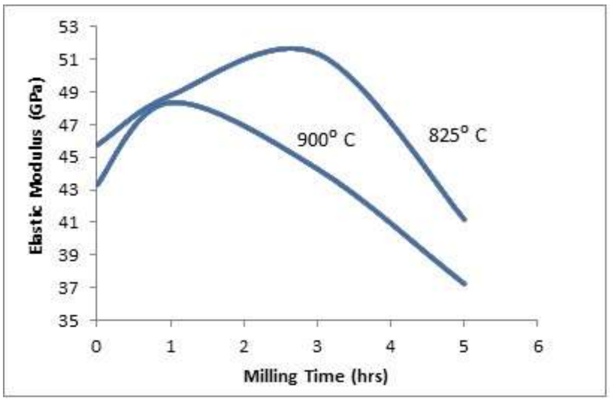

Figure 3 elastic modulus as a function of milling time, for two sintering temperatures

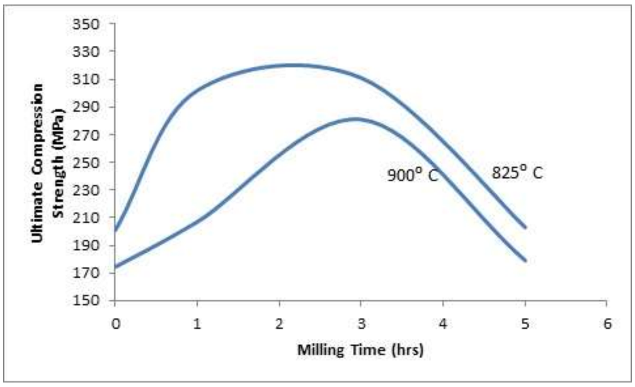

Figure 4 ultimate compression strength as a function of milling time, for two sintering temperatures.

The same explanation can be done to surface hardness (figure 5), the curves of relation between hardness and milling time have the same behaviors, a little difference in the end, at longer time of milling where there is an increase in hardness for both sintering temperatures, here, the increasing in surface hardness without increasing in strength refers to limited surface change not on whole specimen body. It is expected that extended milling time promote a little bit oxidation in sintering, it is thought to be little and limited to a surface because of protective environment. Also the same difference between 825º and 900º C sintering curves is occurring for same reasons.

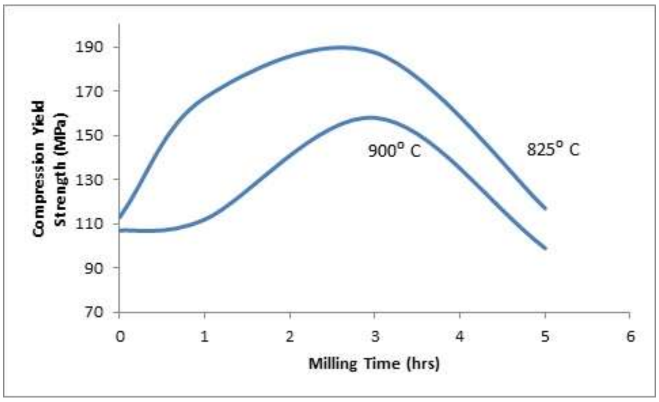

Figure 5 compressive yield strength as a function of milling time, for two sintering temperatures.

Because the modulus of elasticity is directly affected by yield strength, the modulus of consolidated copper (figure 6) follows yield strength in the shape of relation with time of milling but indicating a little falling in values at longer milling time, the reason is back to increase in ductility. Ductility related to temperature and time of sintering, by fixing these two variables the other factor affecting amount of recrystallization and grain growth is amount of residual stresses and stored energy induced through plastic deformation in milling. This means that specimens milled for longer time will gain much plastic deformation, and consequently have a bigger chance to complete recrystallization in lower temperatures and shorter times, and gain a more softening effect and higher ductility in general, and stills the same variance between curves due to different temperatures of sintering.

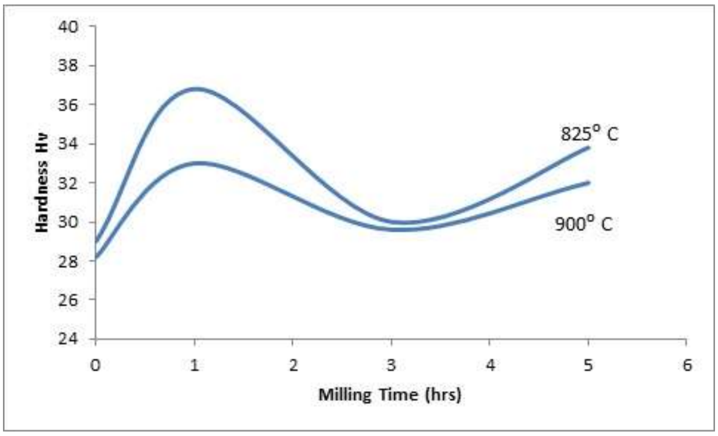

Figure 6 Vickers hardness as a function of milling time, for two sintering temperatures.

In opposite to discussed properties, the wear resistance relation with sintering temperatures gives another variation (figure 7), where samples sintered at 900º C have higher resistance to surface mechanical wear and lesser losses in weight compared to 825º C sintered parts, although they have same behaviors as a consequence to milling time. Lower ductility of 825º C samples is lowering toughness and rising ability to grabbing surface protrusions.

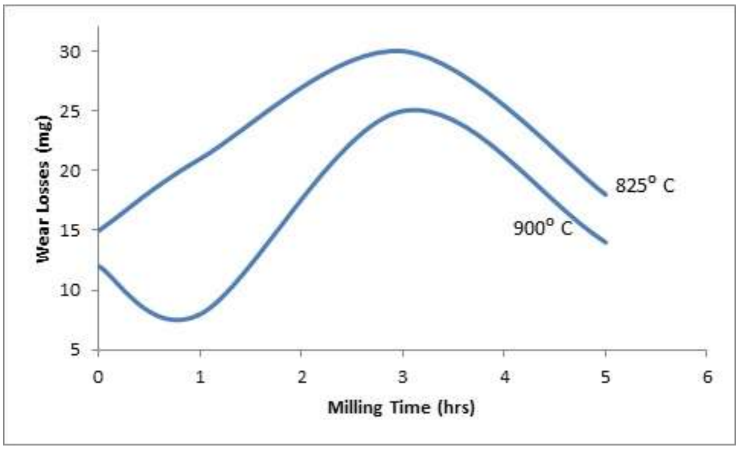

Figure 7 wear losses in weight as a function of milling time, for two sintering temperatures.

4. CONCLUSIONS

We deduce in this study that the mechanical properties of sintered particulate copper (modulus of elasticity, yield strength, ultimate strength, hardness and wear resistance) depend on the extent to which they plastically deformed in milling (expressed by milling time), in addition, they depend on their sintering temperatures.

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