International Journal of Mechanical Engineering and Technology (IJMET)
Volume 9, Issue 10, October 2018, pp. 529–535, Article ID: IJMET_09_10_056
Available online at http://www.iaeme.com/ijmet/issues.asp?JType=IJMET&VType=9&IType=10
ISSN Print: 0976-6340 and ISSN Online: 0976-6359

IMPACT OF POWDER METALLURGY ON STRUCTURAL EVOLUTION IN PURE COPPER POWDER

Nabeel Alawi Kadhim
Department of Materials Engineering, University of Kufa, Iraq

Heider Yasser Thamir* and Amar J. Albaaji
Department of Materials Engineering, University of Al-Qadisiyah, Iraq
*Corresponding author

ABSTRACT

The effects of powder metallurgy variables on structural evolution in pure copper were studied here. Powders of pure copper were exposed to various periods (1, 3, and 5 hours) of high energy milling, in addition to non-milled case. Sintering in protected environment was accomplished. The consolidated parts then were characterized by XRD, EDS, and SEM to evaluate structural changes. Inspections revealed that a three dimensional treelike powder converted by milling to a rounded, flakey, granular, and other shapes of denser copper particles. Some agglomeration was noted. By increasing time of milling, agglomeration was increased too in first and mid stages, during last stage refinement was a dominant. EDS tests showed that both after milling and after sintering a high purity of products was recorded. Consolidated parts revealed a narrow grain size distribution for non-milled starting material, also, a wider size distribution for milled parts in addition to smaller grains sizes related to recrystallization, with some grain growth. By XRD, some extent of amorphous structure or loss in crystallinity was observed related to prolonged extensive plastic deformation, and it proportionated inversely to temperature of sintering. And this was in opposite to observations about grain growth and increased crystallinity in parts sintered in higher temperatures.

Keywords: copper, powder metallurgy, microstructure, particle shape, agglomeration, crystallinity, grain growth.

Cite this Article: Nabeel Alawi Kadhim, Heider Yasser Thamir and Amar J. Albaaji, Impact of Powder Metallurgy on Structural Evolution in Pure Copper Powder, International Journal of Mechanical Engineering and Technology, 9(10), 2018, pp. 529–535.
http://www.iaeme.com/IJMET/issues.asp?JType=IJMET&VType=9&IType=10

1. INTRODUCTION

High energy milling has effects of disintegrating particles volumes; by applying high impact energy, sever plastic deformation, introducing high stored energy, etc. [1]. These effects depend not only on milling variables (frequency, time, B/P ratio, and balls density and hardness) but also on the properties of milled powder (toughness, hardness, ductility, strain hardening ability, etc.), milling environments (wet/dry), and existence of other materials (alloying elements, ceramic particles, etc.) [2, 3]. We should differentiate between particle size and grain (crystal) size; in milling, they experience different baths by different mechanisms and may finally receive different results. Brittle phases undergo a direct refining effect by a virtue of fast crack growth along preferred planes and directions throughout all the milling process till reaching critical sizes where the particles strength more than applied stresses because of little or no defects in bodies of fine particles. While ductile and to some extent semi-ductile phases face a continuous fracture/re-weld cycles under impacts of milling balls and examine highly strained high rate plastic deformations. Generally the collision time of balls is about 2×10^{-6}s, with typical values of strain rates about 10^3-10^4 s^{-1} [4]. Depending on extent of milling, ductile particulates will encounter the following consequences; coarsening by agglomeration (fracture < re-weld), then refining (fracture > re-weld), and finally a near stable sizes despite extent of time (fracture ≈ re-weld) [5]. Of course for a particular material the time to reach stable state is not constant, but depends on milling conditions (frequency, time, B/P ratio, and balls density and hardness). On the other hand the internal crystals will continuously decrease in size as plastic deformation increases till nano range sizes. After, with continuous deformation the nanocrystals gradually loss their crystalline states and convert finally into amorphous phase [6,7]. Microstructures are affected not only by milling, but also by sintering variables (time, temperature, and atmosphere) as well as compaction variables (stress and temperature). During sintering, amount of residual stresses from milling or compaction have influences on recrystallization temperature and time. Sintering temperature controls amount of grain growth; also does sintering time. Impurities (including oxides and reinforcing particulates) can restrict the dislocations and grain boundaries mobility, so minimizing growth rates [8, 9].

2. MATERIALS AND PRACTICAL WORK

Powder of copper (Merck KGaA-Germany) with purity more than ≥ 99.7 %, mean size of particles less than 63μm, and dendritic shape, is used as starting material. Final consolidated copper samples are cylindrical 8mm diameter with height/diameter ratio equal to 3/2. At first, the pure copper powder is divided to four quantities and introduced into jars of high energy milling (planetary rotation, balls of stainless steel, tray rotational speed 250rpm, and ball/powder weight ratio equal to 4.34). The second quantity is milled for one hour, third quantity is milled for three hours, fourth quantity is milled for five hours, but the first quantity is left as received (for comparing) and compacted without milling. A steel cylindrical mold was used for powder compaction by a stresses about 175 MPa from both upper and lower sides (double plungers), some selected samples are pressed at 233 MPa for purpose of XRD comparison. The compacted parts are ready to sintering. Using electrical resistance tubular furnace protected by inert gas (argon) flow the samples were sintered for 50 minutes at temperature 0.810 of the T_m (absolute temperature of melting of copper). Also, some selected samples are sintered at 0.754 T_m for purpose of XRD comparison

3. RESULTS AND DISCUSSIONS

3.1. XRD Characterization

In figure 1, patterns of x-ray diffraction of three consolidated parts are presented. Sample (b) is sintered at 0.754 T_m as same as sample (a), but it attains about double amount of plastic deformations than samples (a) and (c). Sample (c) has the higher sintering temperature (0.810 T_m), but gains same amount of deformation as same as sample (a). So, we have as deformation (b) > (a), (a) = (c), and as temperature of sintering (c) > (a), (a) = (b). By inspecting figure 1 which includes XRD patterns of samples (a), (b), and (c) we recognize a clear differences between patterns in their intensities and broadening. Strengthening of peak intensity approves a bigger crystallites sizes and enhancement in crystallinity (which in part comes from decreasing grain boundaries fraction) by means of releasing stored energy to diminish lattice strain, minimizing overall lattice defects, and encourage recrystallization in sever deformation-induced amorphous regions. On the other hand, peak broadening means refining in crystals sizes and losses in in the overall crystallinity (partly arises from increasing of grain boundaries fraction, which are amorphous regions) by creation of residual stresses and strain in lattices, increasing lattice defects, and induce amorphous regions by continuous high rate highly strained plastic deformations. Looking back to figure 1; according to peaks intensities sample (b) has crystallites with sizes bigger than what sample (a) has, though samples (a) and (b) have same sintering temperature. This is because the bigger amount of plastic deformations sample (b) subjected to; it encourages earlier recrystallization (at lower temperature) and grain growth. Also samples (a) and (c) possess about same plastic deformations and stored energy, but sample (c) has more crystallinity and bigger crystals sizes. This is because it has a hotter sintering temperature, which provides a higher rate of diffusion and therefore a faster grain growth. Sample (b) gains about double what (c) does of plastic deformation, while (c) has a temperature just 0.056Tm hotter than sintering of (b), but sample (c) is more crystalline and has a bigger grains than (b)! Comparing (b) and (c) leads to conclude that temperature is more efficient in recrystallization and grain growth than residual stresses and deformation stored energy. After all, these patterns clearly insure the crystal size refining and/or the loss of crystallinity of starting powders after exposure for prolonged milling time.

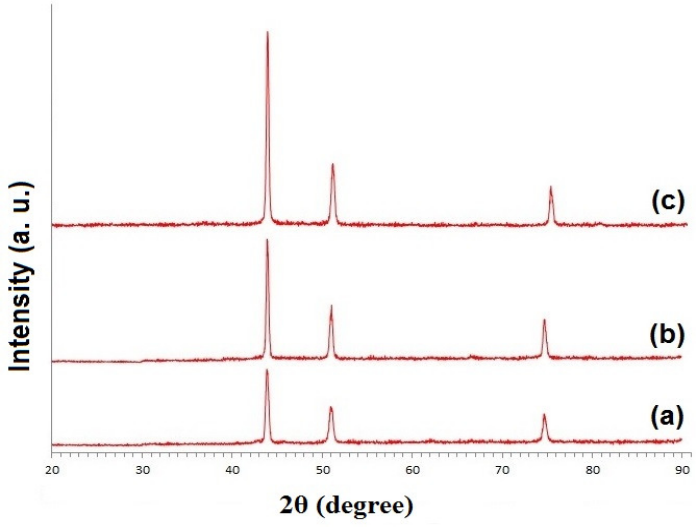

Figure 1 Figure 1 XRD patterns of three different samples.

3.2. SEM and EDS Characterization

Figure 2 shows SEM photos and EDS of copper powder, for non-milled (2-a) we notice the fine three-dimensional treelike dendrites (electrolytically deposited) and EDS analysis indicating highly pure powder. After 1hour of milling (2-b) the powder particles become more homogenized and many shapes appear like rounded, flakey, granular, and others. Some agglomeration can be seen; it produced by re-welding of two or more ductile particles under action of impact. Particles become denser by enclosing interdendritic micro cavities. EDS refer to same chemical composition. In light of fracture/re-weld phenomena the photo of three hours milled powder (2-c) reveals a high rate of re-welding mechanism much more than fracture by milling. Besides the increasing in agglomerated particles size there are more refining for the rest of other smaller particles, and this means increasing in size ranges and nonhomogeneous size distribution. Also no changes in composition as EDS reports. Finally, for five hours milled powders (2-d) it seems that fracture rates become faster than re-weld rates. The sizes of powder now more homogeneous and have tendency toward smaller sizes ranges. There also appear smaller sizes of particles. The shapes are now closer to rounded, granular, and alike. The particles at this stage become harder and less ductile by accumulative developments of strain hardening. Chemical compositions still without noticeable change as EDS supports.

Figure 3 contains SEM photos of consolidated copper. Starting powder milled for 1hour (b), 3hours (c), and 5hours (d), in addition to non-milled powder (a), all are sintered at 825º C for same period of time. From a general looking to figure we find that grains sizes for all specimens range from sub- or one µm towards 20-30 µm. Morphology of specimen (a) is characterized by narrow size distribution leaning towards small mean size. With just one hour milling (b) the trend is forward smaller sizes with few coarser grains. Samples (c and d) reveal more refining in major side plus increasing in size of some grains in other side, but (d) exhibit a little fraction of coarse ones. All four specimens have same sintering temperature, so this can be ignored in explaining the differences between them. The factor affecting here is amount of plastic deformation and residual stresses i.e. time of milling. As it increases, it can lower temperature of starting recrystallization and reduce time to complete it, and initiate grain growth early. Sample (a) has the least value between all, in real it has no plastic deformation (except these coming from compaction), so gets the minimum recrystallization and has narrow size distribution. For other samples (b, c, and d), the deformations and residual stresses are in ascending increasing, which can clarify the emergence of newly smaller sizes of grains (recrystallization), at same time with coarser ones.

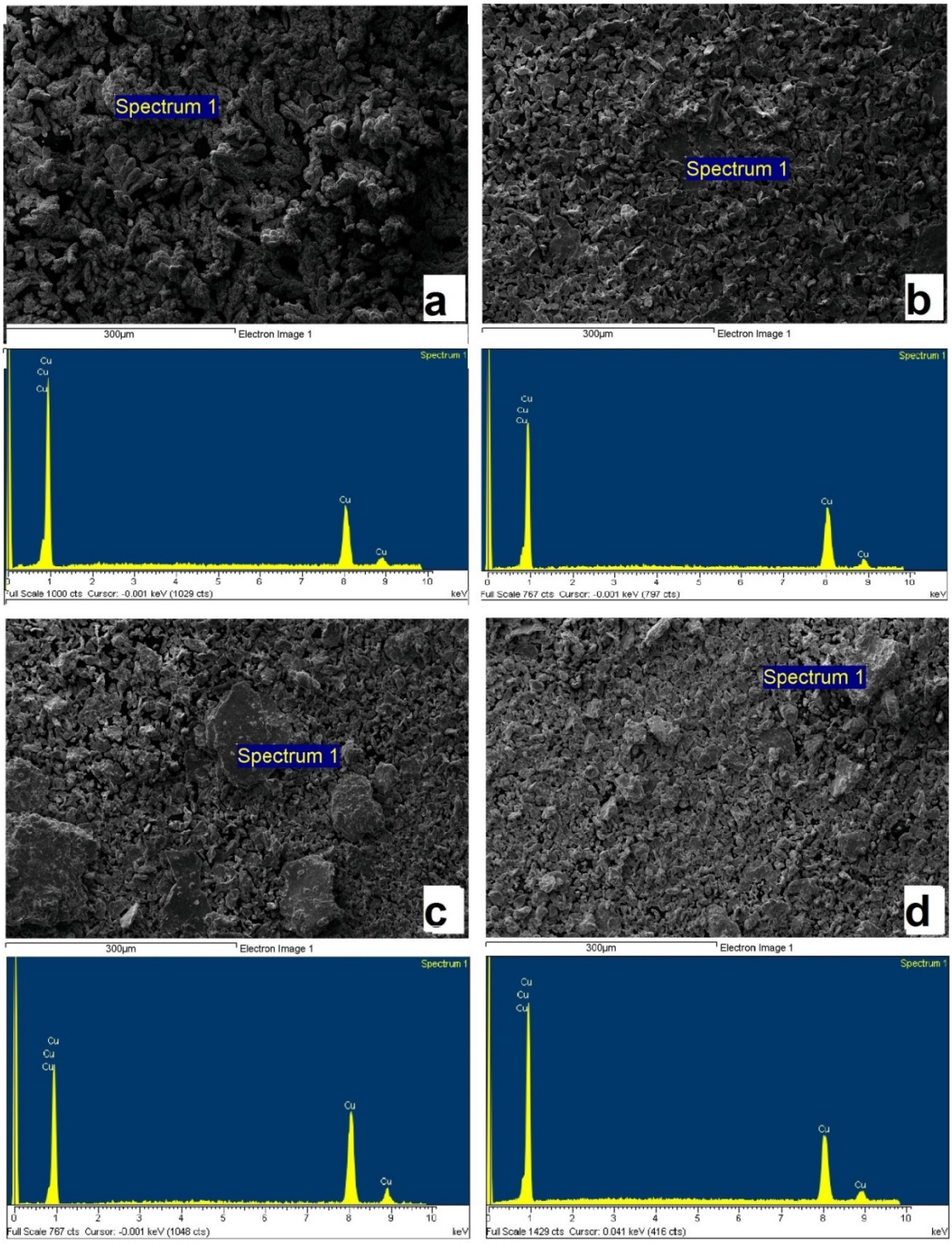

Figure 2 SEM photos and EDS of copper powder, (a) non-milled, (b) 1hour milled, (c) 3hours milled, and (d) 5hours milled.

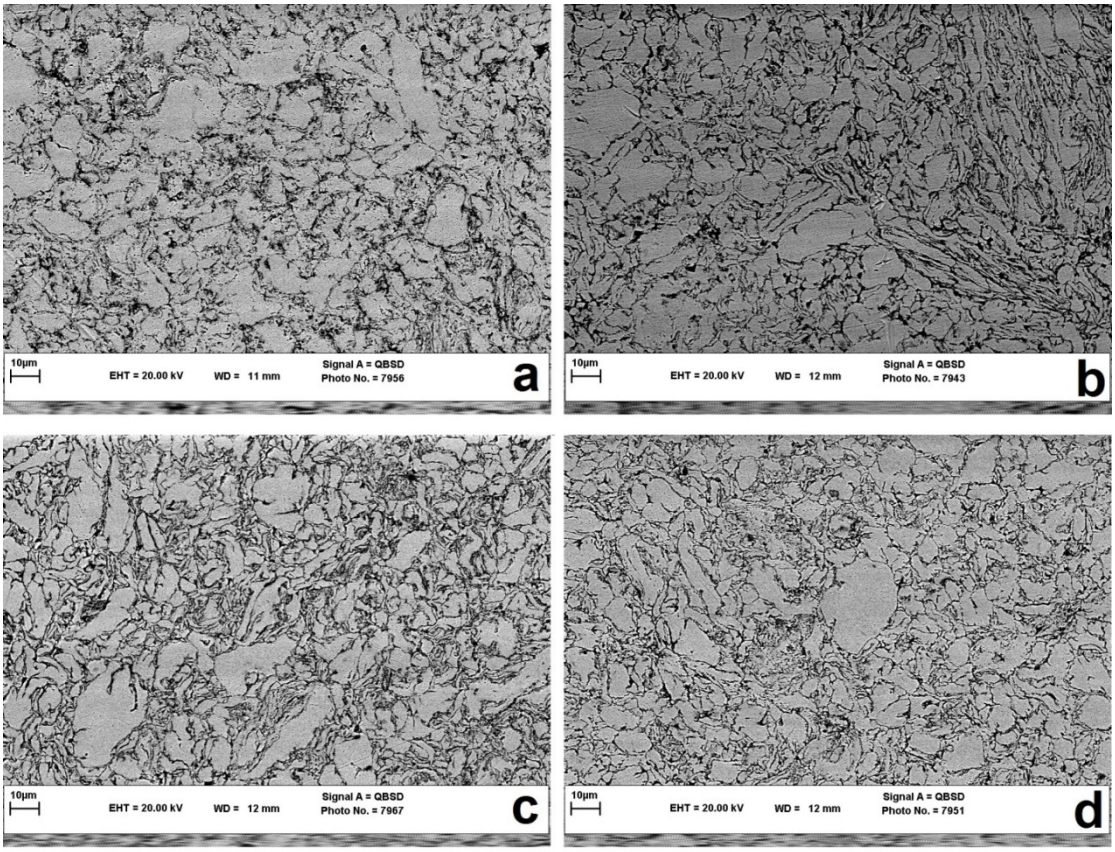

Figure 3 SEM photos of consolidated copper, all sintered at 825º C. non-milled (a), 1hour milled (b), 3hours milled (c), and 5hours milled (d).

4. CONCLUSIONS

Milling leads to some agglomeration and increase in particulates sizes especially in first and mid stages, but during all milling stages it always leads to refinement in crystals sizes and this action if continues for long time will cause some degree of crystallinity loss. Also in final stages of milling, an extended loss of toughness is involved with high rates of strain hardening. Stored energy of sever deformations and temperature of sintering (the first is dependent factor) both motivate recrystallization and grain growth, but sintering temperature is the more efficient one. Finally, the process has a recommended purity of products by EDS.

REFERENCES

[1] Isaac Chang, Yuyuan Zhao, " Advances in Powder Metallurgy: Properties, Processing and Applications", Elsevier, 2013.

[2] Alexandre Nogueira Ottoboni Dias et al., "Effect of High Energy Milling Time of the Aluminum Bronze Alloy Obtained by Powder Metallurgy with Niobium Carbide Addition", Materials Research, 20(3), (2017) 747-754.

[3] Wei Zeng et al., "Microstructural Evolution of Cu-10at%C Nanocomposite Powder During High Energy Mechanical Milling", Materials Research, 18(1), (2015) 152-157.

[4] Carl C. Koch, "Nanostructured Materials Processing, Properties, and Applications", William Andrew Inc., 2007.

[5] Thakur Prasad Yadav et al., "Mechanical Milling: a Top Down Approach for the Synthesis of Nanomaterials and Nanocomposites", Nanoscience and Nanotechnology, 2(3), (2012) 22-48.

[6] J. Ruzic et al., "Influence of mechanical alloying time on particle size of copper matrix composite", Journal of Achievements in Materials and Manufacturing Engineering, 68(2), (2015) 53-58.

[7] Viseslava M Rajkovic et al., "The properties of high-energy milled pre-alloyed copper powders containing 1wt.% Al", J. Serb. Chem. Soc., 72(1), (2007) 45–53.

[8] Paula Rojas et al., "Effect of the Powder Metallurgy Manufacture Process on the Electrochemical Behaviour of Copper, Nickel and Copper-Nickel Alloys in Hydrochloric Acid", Int. J. Electrochem. Sci., 11 (2016) 4701-4711.

[9] Matteo Zanon et al., "The sintering behaviour of electrolytic and water-atomized copper powders", Euro PM2015 Proceedings: Sintering, 2015