IOP Conf. Series: Materials Science and Engineering **377** (2018) 012209 doi:10.1088/1757-899X/377/1/012209

Effect of compaction pressure on microstructure, density and hardness of Copper prepared by Powder Metallurgy route

Manish Dixit[1] and Prof. R. K. Srivastava[1]

[1]Department of Mechanical Engineering, Motilal Nehru National Institute of Technology, Allahabad, 211004, India

manish.dixit39@gmail.com

Abstract. The Copper samples were prepared by powder metallurgy route and conventional sintering. The objective of the present work is to study the effect of compaction pressure on the bulk hardness, densification behaviour and microstructure of the Copper. The Copper powder is compacted with compaction pressure 500, 600, 700 and 730 MPa. The sintering of all green compact is performed at 750^0C for 1.5 hour in conventional muffle furnace. Compaction pressure highly influences the pore size as well as number of pores. With increase in compaction pressure the surface contact area of the powder particles increase which reduce the porosity. The maximum valve of the density and hardness is obtained for 700 MPa and 3 min dwell time. Different characterization techniques such as Scanning electron microscope (SEM), X-ray diffraction (XRD) were used to characterize the sintered copper.

1. Introduction

Copper is a candidate material for industrial application due to its good electrical and thermal conductivities, low thermal expansion co-efficient, high melting point and good corrosion resistance. But it does not have wide range of applications due to its poor mechanical and wear resistance property. To overcome these limitations, hard second phase particles such as carbides, nitride, sulphides and borides are added into the copper matrix. Copper based metal matrix composites (MMCs) are widely use in electrical sliding contact, heat sink, aerospace and automotive application. The several techniques are used to synthesize the Cu based MMCs such as powder metallurgy, Stir casting and ingot metallurgy. Among these techniques powder metallurgy (PM) is widely use to synthesize the Cu base MMCs as it can produce near to net shape product with complex geometries, lowers the machining cost and high volume production. The PM process mainly consists of three basic steps in sequence: mixing (blending) of powders, compaction and sintering. In blending, adequate composition of different powders are mixed in a ball mill in order to obtain a uniform mixture of composite powder. This composite powder is placed in a dedicated die and punch assembly and then gradual load is applied to achieve desire compaction pressure. Due to high compaction pressure free powder particle are combined together by the mechanical bonds and form a solid part of desire shape. This solid part is known as the green compact. It is very fragile in nature and has poor strength to work. In sintering, Green compact is heated to 0.75 to 0.85 of melting point of the main constituent to allow for solid-state diffusion. Due to solid state diffusion at elevated temperature, the weak mechanical bonds are replaced by the strong metallic bond. As in PM, initial constituents are in powder form so it facilitates mixing the wide range of compositions.

Mahani Y et al. study the effect compaction pressure on microstructure and mechanical properties of Cu-TiC composite for different pressure ranging from 100 to 600 MPa. With increase in the pressure the tendency towards the plastic deformation of Cu matrix and fracturing of reinforcing TiC particles increases. Fracturing of reinforcement may give adverse effect on mechanical properties.

SEM images reveal the plastic deformation of Cu matrix without fracturing of reinforcing TiC particles up to 600MPa. The high plastic deformation of Cu matrix attributes the increase of green density. With increase in compaction pressure the contact area of powder particles increase SEM images reveal the plastic deformation of Cu matrix without fracturing of reinforcing TiC particles up to 600 MPa. The high plastic deformation of Cu matrix attributes the increase of green density. With increase in compaction pressure the contact area of powder particles increase which causes reduction in porosity. Presence of porosity lowers the mechanical properties like density and hardness. It also decrease the electrical conductive by restricting the movement of free electron.

With the increase in compaction pressure, the mechanical property (density and hardness) and electrical conductivity increases [1]. De Mello et al. discussed the effect of compaction pressure on microstructure, oxide content, hardness, and surface topography for sintered iron. The effect of compaction pressure on different properties is analysis for 300, 400, 500, and 600MPa. As the compaction pressure increases the powder particle come closer and porosity decreases. Results indicate that low porosity is always associated with the high compaction pressure. Similarly, Compaction pressure greatly influences the pore size of the composite. At high compaction pressure, ductile powder undergoes high plastic deformation this lead to dense packing of powder particle. Moreover, due to increase in effective particle contact area it may lead to decrease the pores volume and porosity [2]. Pranav Garg et al. study the effect of compaction pressure on green density and sintering of fine molybdenum powders. Green density corresponding to the compaction pressures of 280, 560 and 840 MPa are 63%, 73% and 79% respectively. The masters sintering curves (MSCs) are experimentally obtain at different compaction pressures using dilatometer analysis. Dilatometer analyses optimize the process and reduce the number of sintering runs. It is observed that the densification of finer molybdenum powder is high at all compaction pressures. The densification increases with increase in compaction factor [3]. Wen-Fung Wang pointed that the compaction pressure greatly influences the green density, sintered density and hardness of the composite [4]. Maximum density and hardness of iron powder is obtained for 500 MPa compaction pressure. Yongping Jin et al. suggested that the copper graphite composite exhibit excellent mechanical properties for 700 MPa compacting pressure and 30 s dwell time. As compaction pressure exceeds 700 MPa, the fracture of powder particle may starts which reduce the compaction effect. In this paper, the milling effect of powder on the different processing and properties is discussed [5].

From the above literature it is clear that compaction pressure have great influence on the density, hardness and electrical conductivity. The objective of the present work is to analyze the effect of compaction pressure on microstructure, density and hardness of the fine electrolytic Copper. The green compacts of Copper are prepared for different compaction pressure. Effect of compaction pressure on the green density is studied for Copper powder. After sintering, samples are prepared to study effect compaction pressure on the microstructure, density and hardness.

2. Experimental Procedures

In present study, electrolytic Cu with 40 micron average particle size and 99.9% purity is selected as working material. The high purity Copper is supplied by Sarda Industrial Enterprises Jaipur. The Cu powder is preheated in a hot air oven at 150^0C for one hour. It will remove the moisture content present in the powder. After cooling to room temperature, the powder is placed in dedicated double acting compaction die. The die have 20 mm inner diameter. Zinc stearate is use as die lubricant which facilitates the removal of punch and green compact. The compaction is carried in a Universal Tensile Machine with different compaction pressure of 500, 600, 700 and 730 MPa. The compaction is carryout under gradually applied load with 3 minute relaxation time. After confined compaction the Cu powder is bonded with mechanical bonds and forms a Green Compact. The compact after pressing is called a green compact. Five samples are compacted at each compaction pressure. The Green compact forms after pressing possess poor mechanical strength. Sintering is performed to improves the strength of Green Compact. Sintering is carried out at elevated temperatures of 750^0C for 1.5 h using a

IOP Conf. Series: Materials Science and Engineering **377** (2018) 012209 doi:10.1088/1757-899X/377/1/012209

conventional Muffle furnace. Due to sintering the weak mechanical bonds of green compact are converted into strong metallic bonds.

3. Work Methodology

3.1 Process and material parameters affecting the densification behaviour

Generally, compaction of powder is carried out in die and punch assembly in room temperature. The die and punch assembly is placed in hydraulic press. As compaction pressure increases, the metal powder experiences the uni-axial compression. During compaction, powder particles mainly experience the compressive stress but particles near the die wall surface possess some tensile response. While mathematical modeling the densification behavior, the difference in compressive and tensile behavior of powder particle should be consider. So mathematical modeling of generalized yield criteria for uni-axial powder compaction in a die is given by the equation below.

$$F = \left(\frac{q}{\sigma_m}\right)^2 + \alpha(1-\rho)^{\beta}\left(\frac{p}{\sigma_m}\right)^2 - \rho^{\delta} \quad (1)$$

Where,
$q \rightarrow$ effective stress, $p \rightarrow$ hydrostatic pressure,
$\rho \rightarrow$ relative density, $\sigma_m \rightarrow$ flow stress of the matrix material
α, β, and $\delta \rightarrow$ material parameters

The above equation does not consider the heating effect because the valve of flow stress of the matrix material σ_m varies with the temperature. So it became very difficult to assign a numerical of flow stress of the matrix material σ_m for different temperature. In order to avoid the heat generation, compaction process should be carried out in slow manner in room temperature. The value of material parameters α, β, and δ can be obtained by tri-axial test. The conventional procedure to find the valve of material parameters α and β is very expensive, time taking and difficult to use in industry as it needed some special purpose equipment. For simplicity, the material parameters α and β are considered to be independent of metal powder. So based on the uniaxial compression and tension tests, Shima and Oyane proposed the valve of material parameters α and β are 6.20 and 1.028 respectively [6]. These values of material parameters are successfully apply for stainless steel and copper powder. The value of flow stress of the matrix material σ_m (at room temperature) may be calculated from the equation given below.

$$\sigma_m = a_m + b_m e_m^{n_m} \quad (2)$$

Where,
a_m, b_m, and $n_m \rightarrow$ material parameters
$e_m \rightarrow$ effective strain of matrix material

The valve of material parameters δ, am, bm, and nm, can be obtain from the cylindrical die compaction test. The experiments result shows the material parameters δ is not only the material property but also the powder property. So, based on uniaxial compression tests, Shima and Oyane proposed a constitutive model that predicts the densification behavior of powder during die compaction.

$$F = \left(\frac{q}{\sigma_m}\right)^2 + 6.20(1-\rho)^{1.028}\left(\frac{p}{\sigma_m}\right)^2 - \rho^5 \quad (3)$$

The assumption for above equation is to keep co-efficient friction as low as possible. It can be achieve by minimum punch movement and proper use of die lubrication on the die wall surface.

3.2 Calculation of Green Density.

To study the effect of compaction pressure on the green density, we have to establish the density-pressure relationship over the entire compaction pressures range [7]. The green density may be obtained with the help of single acting die compaction test. Compaction test take place in a die and punch assembly of 20 mm in diameter with proper tolerance. The density may be calculated with the help of powder weight and volume. With increase in pressure, punch makes linear movement in confined die space like forward motion of piston in a piston cylinder arrangement. The displacement of punch may be calculated for corresponding load. As shown in fig. 1 the cross-sectional area of die is constant and displacement of the punch is known so the volume of metal powder can be calculated for corresponding pressure.

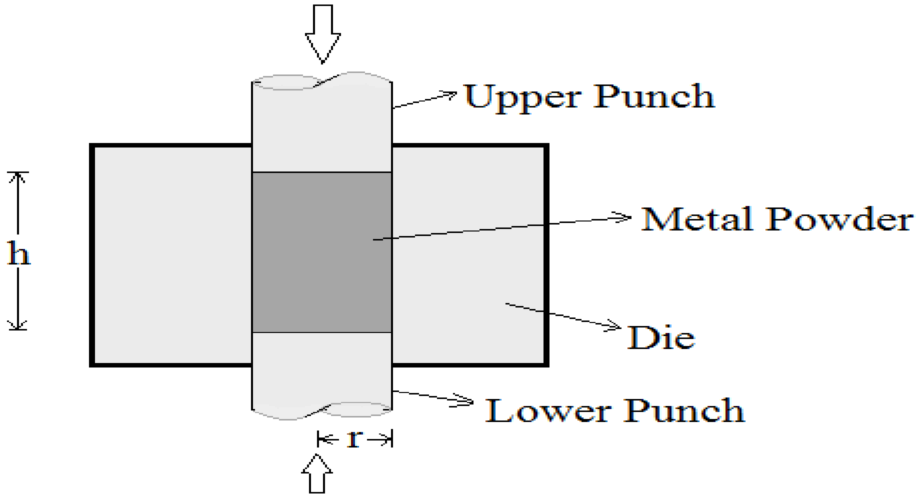

Figure 1: illustrate the process of powder compaction

The weight of the metal powder remains almost constant throughout the compaction. So with the help of mass and volume, the green density of compact can be easily calculated for corresponding pressure.

$$\rho_g = \frac{m}{v} \qquad (4)$$

$$v = \pi r^2 h \qquad (5)$$

Where,

$\rho_g \rightarrow$ Green Density, m → Powder weight
h → Powder height, r → Die radius v → volume

The fig. 2 shows the variation of green density with compaction pressure. The green density increases with the increase in compaction pressure. The graph shows that green density is very poor at low compaction pressure.

4. Result and Discussion

4.1 Green and Sintered Density

The sintered density of the specimen is calculated by using Archimedes principle. With increase in compaction pressure the consolidation of loose powder particle start. Upto 200 MPa applied pressure, the powder particles are rearranged so it possesses poor packing of powder.

Table: 1 Effect of compaction pressure on densification response of Copper

Compaction Pressure (MPa)	Green density (% theoretical)/g cm–3	Sintered density (% theoretical)/g cm–3	Densification parameter
500	7.89 (88.45%)	8.08 (90.58%)	0.18
600	8.27 (92.71%)	8.41 (94.28%)	0.22
700	8.58, (96.2%)	8.70 (97.53%)	0.35
730	8.65 (96.97%)	8.72 (97.75%)	0.26

As compaction pressure increases from 200 MPa to 500 MPa, the deformation of powder particles take place and particles are associated with the weak mechanical bonds. The fig.3 shows that due to plastic deformation, green and sintered density significantly increases at low 500 MPa. Above 500 MPa compaction pressure, Cu particles undergo high plastic deformation which attributed to increase in green and sintered density.

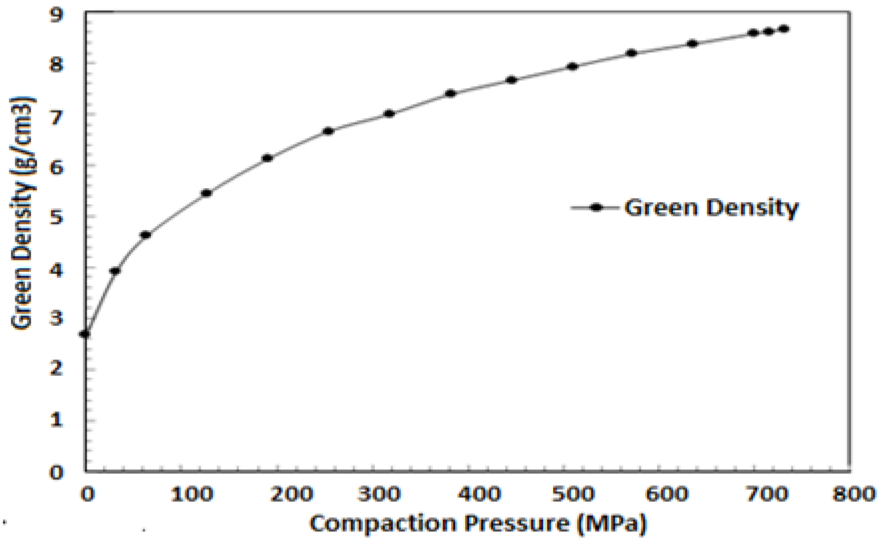

Figure 2: Shows the effect compaction pressures on the Green density of Copper.

Increase in compaction pressure lead to increase in particle contact area due to which high densification may be achieved. Increasing the compaction pressure influences the work hardening of the green compact. It improves the green density of copper. Fig. 3 confirms that with the increase in compaction pressure both green and sintered density increases. The maximum value of density is obtained for 730 MPa. The value of green density, sintered density and densification parameter for different compaction pressure are shown in table-1.

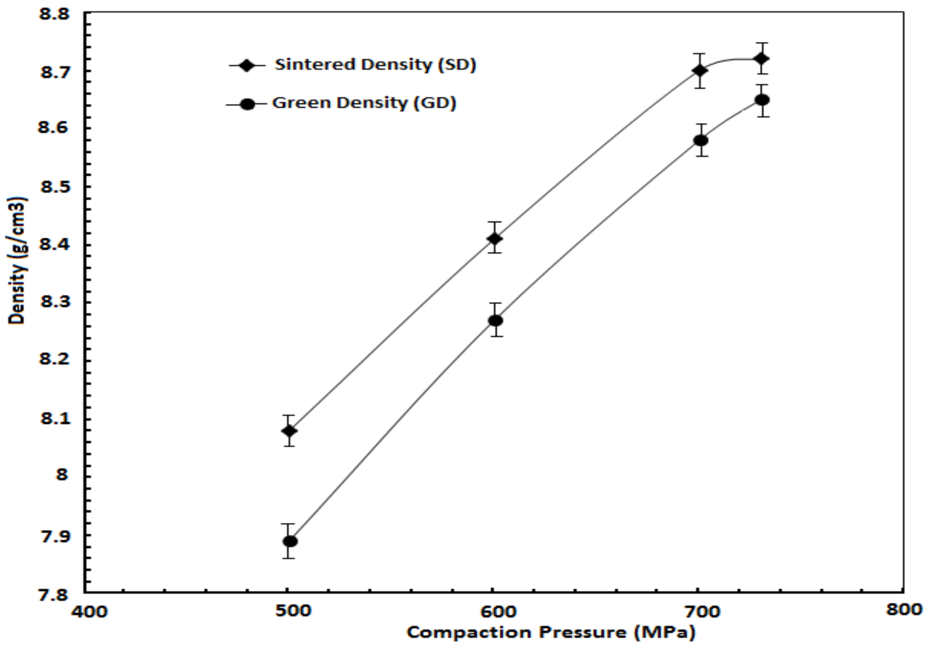

Figure 3: Shows the effect compaction pressures on the Green and Sintered density of the Copper

IOP Conf. Series: Materials Science and Engineering **377** (2018) 012209 doi:10.1088/1757-899X/377/1/012209

Positive value of densification parameter confirms the shrinkage of Cu during sintering moreover it confirms the reduction in porosity [8]. Result shows that maximum value of densification parameter obtains for 700 MPa which confirm the maximum densification for Copper at 700 MPa. The plot in fig. 3 shows that the sample pressed at higher pressure exhibit higher density as compared to pressed at lower pressure.

4.2 Scanning electron microscopy

Morphology and microstructure of the sintered specimens are examined under a scanning electron microscope, FEI NOVA Nano SEM. Figure 4 represents the features of the microstructures observe for different compaction pressure. At low compaction pressure, the effective contact area of powder particle is less due to poor plastic deformation of powder particles.

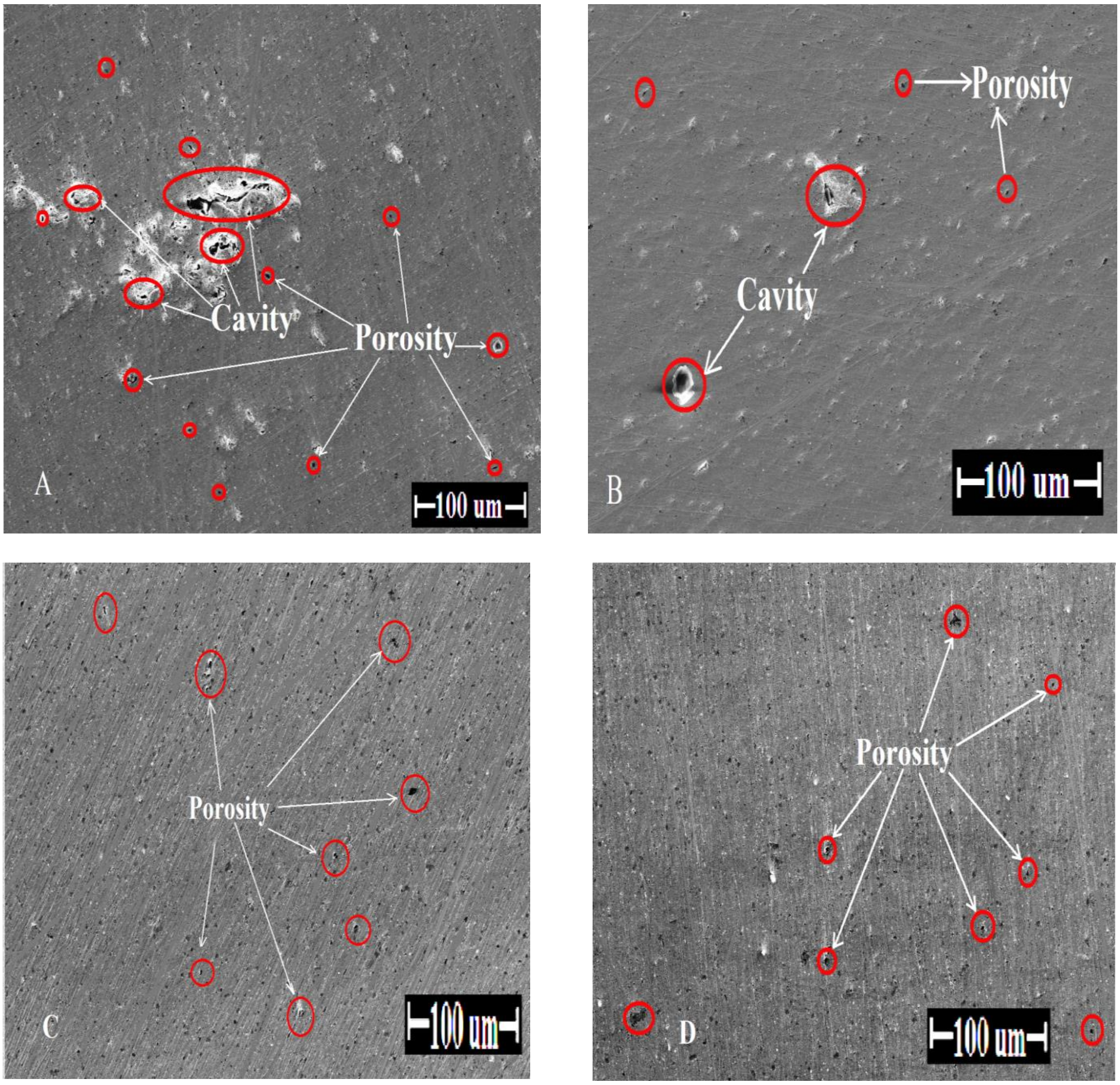

Figure 4: Shows the unetched SEM images of Copper at A. 500; B. 600; C. 700; and D. 730 MPa compaction pressure.

Low compaction pressure lead to segregation of pore along the interstitial gap between the particles. Due to this pores are pile up along the interstices which may lead to formation of cluster. This may attributed to quite large and irregular size of the pore [8]. Fig. 4a shows that the pores are in the form of voids and also irregular in size at low compaction pressure. Micrograph shows the presence of irregular cavity at 500 MPa. The formation of cavity confirms the segregation of pores at low compaction pressure. As pressure increases above the 500 MPa, the effective stresses along the grains boundary exceeded the elastic limit which causes higher plastic deformation along the contact points. It attributes the reduction in pore segregation which further lowers the cluster formation. Microstructure in fig 4b shows the formation of small voids and tiny size pores at 600 MPa. Due to ductile nature, copper particle experiences more plastic deformation. As compaction pressure reaches to 700 MPa, due high plastic deformation pores cluster are disappear and remains the small tiny size pores. The SEM micrograph in fig 4c shows the formation of small tiny size pores. It may be attributing to cold working (work hardening) due to increase in pressure. Due to work hardening induced stress increase and enhances the plastic deformation due to which the pores diameter decrease and it converted into small tiny size. At high compaction pressure 730 MPa, fig. 4d shows the significant reduction in porosity.

4.3 Hardness

To measure the bulk hardness of sintered Copper, Brinell Hardness Tests are performed. The value of hardness is measured for the average of at least three tests result for each compaction pressure. Fig 5 shows the variation of hardness with the compaction pressure. At low compaction pressure (500 MPa); the value bulk hardness is considerably low. It may explain from the fig. 4a, the microstructure analysis confirms that at low pressure pore are in the form of large voids and irregular in size. At the region of void formation, stress concentration center is developed due to which hardness decreases.

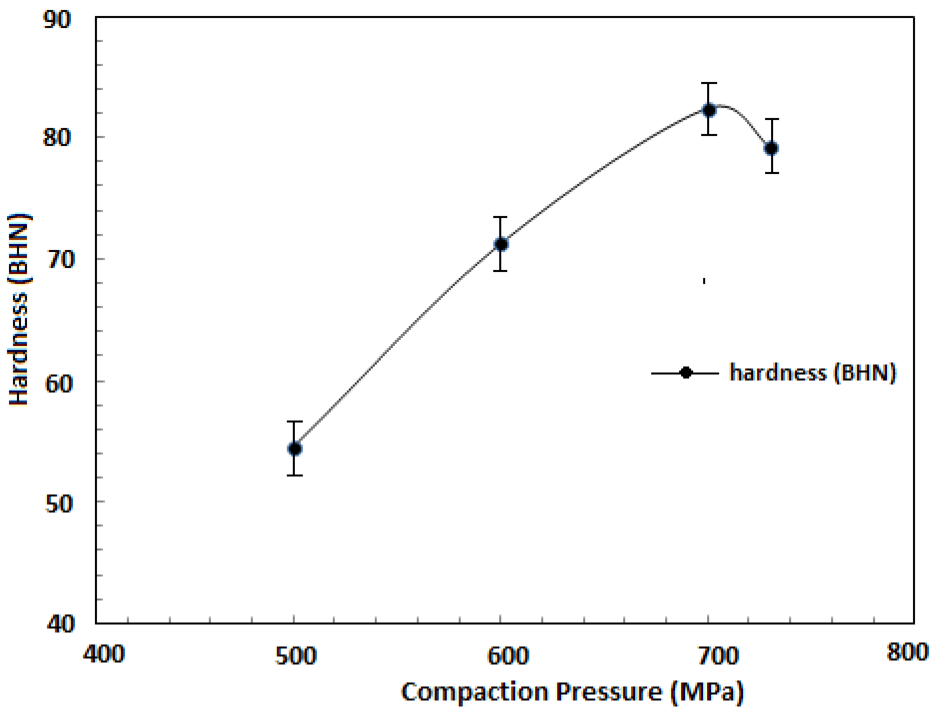

Figure 5: Shows the effect compaction pressures on the hardness of the copper

As compaction pressure increase, it will decrease the pore size. From the fig 4c it is clear that at 700 MPa the pores are of tiny size. So no stress concentration center appears at the surface hence it will increase the hardness. The maximum value of hardness is associated with 700 MPa compaction pressure. The value hardness is decrease at 730 MPa as compared to 700 MPa compaction pressure. It may due to high plastic deformation; the fracturing of copper particle may be initiated at high compaction pressure [1]. So it will reduce the hardness. In general high hardness is associated with the high compaction pressure.

4.4 X-ray diffraction analysis

Fig. 6 shows the XRD spectra of the Copper prepared with 700 MPa compaction pressure and 750^{0}C sintering temperature. The XRD spectra detected the strong peaks of Cu and weak peaks of Cu_2O. The weak peak of Cu_2O confirms the slight involvement of Cu with the atmospheric oxygen during conventional sintering.

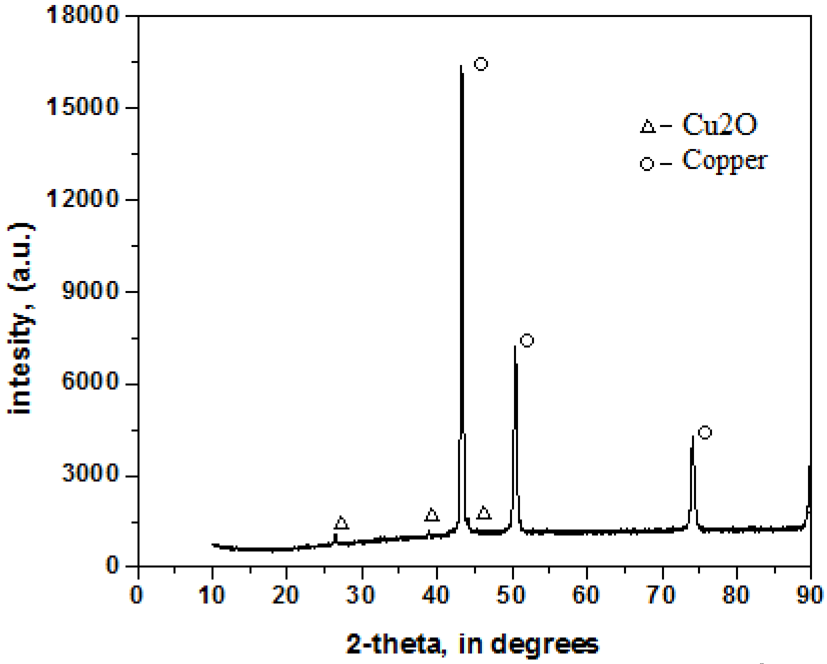

Figure 6: Shows the XRD spectrum of Copper conventionally sintered at 750^{0}C for 1.5 h

5. Conclusion

(1) The present study highlighted the relationship among the factors which are associated with the densification behavior of copper such as effective stress, relative density, flow stress of the matrix material, friction and die wall lubrication.

(2) Microstructure analysis reveals that at low compaction pressure pore clusters and voids are present due to segregation of pore but as pressure increases large voids are replace by the small tiny pores.

(3) High green and sintered density is associated with the higher compaction pressure for a fixed grade of Copper powder. The maximum densification parameter achieve at 700 MPa.

(4) Bulk hardness increases with the applied pressure but as heavy plastic deformation initiate the fracture of particle its start decreasing.

(5) The optimum value of densification and bulk hardness is associated with 700 MPa compaction pressure.

IOP Conf. Series: Materials Science and Engineering **377** (2018) 012209 doi:10.1088/1757-899X/377/1/012209

References

[1] Mahani Y, Zuhailawati H 2013 Effect of Compaction Pressure on Microstructure and Properties of Copper-based Composite Prepared by Mechanical Alloying and Powder Metallurgy International Conference on Sustainable Materials Engineering (ICoSM) Pg 26-27

[2] J. D. B De Mello, R. Binder, A. N. Klein, and I. M. Hutchings 2001 Effect of compaction pressure and powder grade on microstructure and hardness of steam oxidised sintered iron Powder Metallurgy Vol. 44

[3] Pranav Garg, Seong-Jin Park and Randall M German 2005 Effect of die compaction pressure on densification behavior of molybdenum powders International Journal of Refractory Metals & Hard Materials

[4] Wen-Fung Wang 2006 Effect of Powder Type and Compaction Pressure on the Density, Hardness and Oxidation Resistance of Sintered and Steam-treated Steels Journal of Materials Engineering and Performance Volume 16

[5] Yongping Jin and Ming Hu 2011 Densification of Graphite/Copper Compound Powders IEEE

[6] Shima S, Oyane M. 1976 Plasticity theory for porous metals International Journal of Mechanical Science Vol 18

[7] R. W. Heckel 1960 Density-Pressure Relationships in Powder Compaction Transactions of the metallurgical society of AIME

[8] S. Hadas, A. Raja Annamalai and A. Upadhyaya 2011 Effect of compaction pressure and tempering on mechanical and tribological properities of sinter hardened Ancorsteel 4300–0.6Gr. Materials Technology Volume 26