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HARDNESS AND DENSIFICATION BEHAVIOUR OF COPPER AND BRONZE POWDERS COMPACTED WITH UNIAXIAL DIE AND COLD ISOSTATIC PRESSING PROCESSES

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In this study hardness and densification behaviour of copper and bronze powders under wet bag cold isostatic and uniaxial die pressing processes are examined. In uniaxial pressing the specimens were compacted up to a pressure of 800 MPa. Cold isostatic pressing (CIP) resulted in better densification for both of the studied powder materials. Attained densities were 94 % for copper and 82 % for bronze powders. In uniaxial die pressing greater pressurisation was needed to attain the same densification that obtained with CIP. The microhardness of both of the studied materials were measured before and after pressing processes. Higher pressure resulted in dislocation and strain hardening and increased hardness of powders.

Key words: *cold isostatic pressing, uniaxial pressing, densification, microhardness*

Ponašanja tvrdoće i povećanja gustoće prahova bakra i bronce kompaktiranih procesima hladnog prešanja u matrici smjerom jedne osi. U ovom radu se istraživala promjena tvrdoće i povećanje gustoće prahova bakra i bronce prešanih na hladno u matrici smjerom jedne osi, te izostatski pomoću fluidnog medija. Pri hladnom prešanju u matrici smjerom jedne osi uzorci su kompaktirani s tlakovima do 800 MPa. Hladno izostatsko prešanje (CIP) rezultira znatnijim povećanjem gustoće kod oba praškasta materijala. Dobivene gustoće bile su 94 % za bakarni i 82 % za brončani prah. Kod prešanja u matrici smjerom jedne osi bilo je potrebno upotrijebiti veće tlakove da bi se postigla ista gustoća kao ona dobivena CIP postupkom. Mikrotvrdoća oba proučavana materijala mjerena je prije i nakon procesa prešanja. Veći tlakovi uzrokuju očvrščavanje dislokacijama i naprezanjem, te povećavaju tvrdoću prahova.

Ključne riječi: *hladno izostatsko prešanje, prešanje smjerom jedne osi, povećanje gustoće, mikroočvrščavanje*

INTRODUCTION

In powder metallurgy process different methods are used for consolidation of metal powders into structural shapes. Uniaxial die pressing and cold isostatic pressing (CIP) methods are two of these methods which are widely used to manufacture near net shape (NNS) components. In this study hardness and densification behaviour of bronze and copper powders are investigated using uniaxial die and wet bag cold isostatic pressing methods. In uniaxial die pressing process automatic hydraulic or mechanical presses of 10 to 1000 tones capacity are used to produce "green" or "as-pressed" compacts at rates typically between 250 and 1250 per hour [1].

Actual rates depend on the press size, compacted component size and complexity. A schematic drawing of uniaxial die pressing cycle is shown in Figure 1. At the first stage the powder supplier moves over the die with the lower punch and required weight of powder is introduced. After the powder supplier has withdrawn the top and the bottom punch moves relatively to the die to compress the powder at the pressures between 400 to 800 MPa. After compaction the top punch is withdrawn and the bottom punch moves upwards to eject the compacted powder. Finally the powder supplier moves again across the top surface of die, refills it and pushes the green component onto a moving belt as seen in Figure 1. Compaction tools are mostly manufactured from conventionally hardened and tempered die steels as to obtain longer service life, carbide inserts can be incorporated into the die [2]. During die compaction the tools have to endure both high stresses and wear. Thus, tool fracture occurring during production

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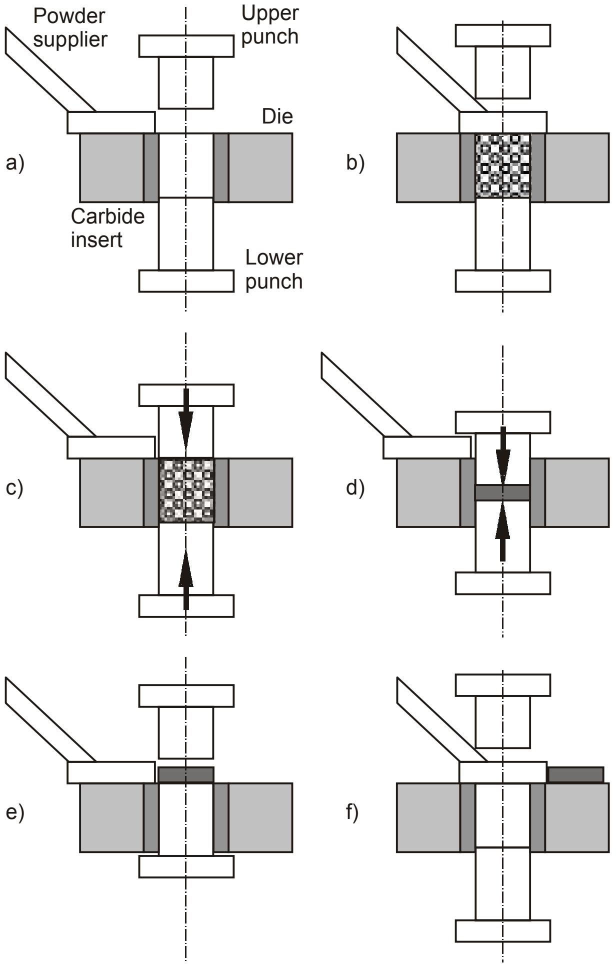

Figure 1. **A schematic illustration of uniaxial die compaction cycle: a - initial position of die set, b - filling die with powder, c - compaction start, d - compaction completed, e - part ejection, f - part removal - die refilling)**

Slika 1. **Shematski prikaz ciklusa prešanja u matrici smjerom jedne osi: a - početni položaj matrice, b - punjenje matrice prahom, c - početak prešanja, d - završetak prešanja, e - izbacivanje uzorka, f - uklanjanje i ponovno punjenje**

is a costly factor whose probability is related to the complexity of the tools. Another drawback of die compaction is that it is not possible to achieve a homogenous green density distribution in the structure of compacted components [3].

The density is more or less inhomogeneous depending on the part of geometry, the tool design and the friction between powder and die wall. As a result of these problems, the compacted parts undergo shape distortions during sintering, or cracks may develop. The main reasons for preferring uniaxial die pressing in manufacturing PM components are; material and energy severity, possibility of pressing components that have large geometrical shape, precision and repeatability tolerances in pressed component dimensions and high rate productivity (250 - 1250 component per hour) [4].

In isostatic pressing process an uniform pressure is applied equally and simultaneously to all of the external surface of powder. Thus the powder is compacted with the same pressure in all directions and since no lubricant is needed, high and uniform density can be achieved. To obtain uniform pressure on powder particles, the powder is contained in a flexible mold and then immersed in a fluid kept at very high pressure. Isostatic pressing eliminates some of the constraints that limit the geometry of parts compacted unidirectionally in rigid dies [5 - 6]. Isostatic pressing that is done at ambient temperature is called cold isostatic pressing (CIP). The CIP is used primarily for producing green bodies (powder compacts prior to sintering). With the CIP process it is possible to reach pressures as high as 700 MPa.

Long thin-walled cylinders and parts with undercuts can be manufactured effectively with CIP. The dimensions of compacted parts are approximately 100 mm in diameter and 460 mm in length [7 - 8]. Due to the absence of die-wall friction and the greater area over which pressure is applied for each of powder particle, CIP provides more uniform pressure distribution than uniaxial die pressing. Transferring air out from loose powder before compaction, improves the properties of compaction. The absence of lubricant in CIP process eliminates the problems related with lubricant removal prior to sintering [1, 5]. Densification of a compacted powder body depends upon powder characteristics such as: shape, hardness, cold welding behaviour, size distribution, effect of lubricant and interlocking during pressing process [8, 9]. Densificaton phenomenon of powders can be assessed in three stages. In the first stage densification is obtained by rearrangement of powders at small levels of pressurisation. The density improvement by rearrangement is dependent on the powder characteristics. The first 5 to 10 percent decrease in porosity can be attributed to rearrangement. In the second stage at intermediate pressure, elastic-plastic deformation occurs at the interface of particle contact area. Increasing pressure level results in increase of the number of contacts and contact area of every particle and decrease the porosity as shown in Figure 2.

At the final stage, with the effect of high pressurisation massive deformation occurs leaving small pores between particles. There are two types of CIP process: wet bag CIP and dry bag CIP. Filling of the mold with powder takes place outside of the pressure unit in wet bag CIP. The mold is then placed into the pressure unit and directly immersed into the fluid as seen in Figure 3a. Wet bag CIP process is used for pressing larger parts and complex forms. Compaction cycle takes a few minutes. Components with different shapes can be pressed in the same pressure unit during one cycle in wet bag CIP [10]. In dry bag CIP compaction chamber of powder is separated from the pressure fluid by an elastomeric sleeve as seen in Figure 3b. The

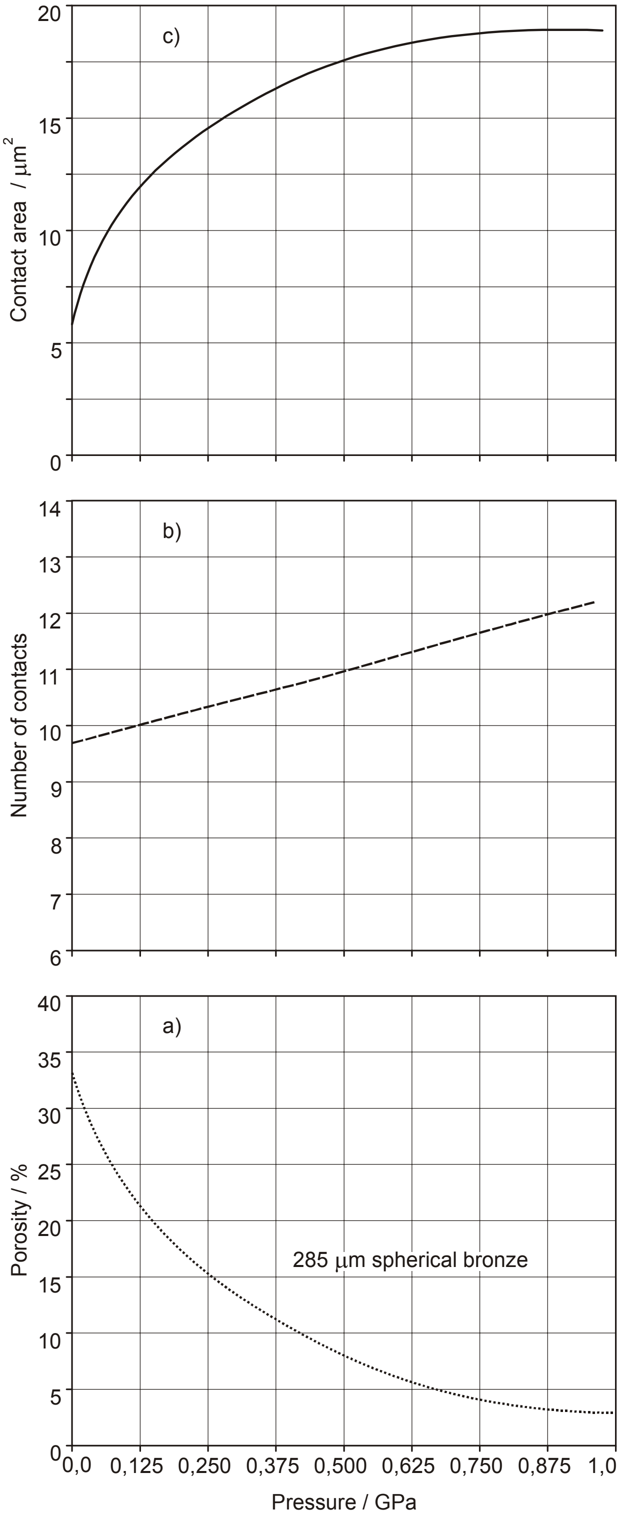

Figure 2. **Behaviour of spherical bronze particles during compaction process. a) decrease in porosity with pressurisation, b) increase in the average number of contacts per particle (repacking), c) contact area / deformation mechanisms**

Slika 2. **Ponašanje sfernih čestica bronce za vrijeme procesa prešanja: a) smanjenje poroznosti s povećanjem tlaka, b) povećanje prosječnog broja kontakata po jednoj čestici, c) kontaktna površina / mehanizmi deformacije**

mold is fixed inside the pressure unit. This process is mostly used for smaller parts such as tubes, rods and nozzles. Typical cycle time for dry bag CIP compaction of a component is about 20 to 60 seconds [4, 10].

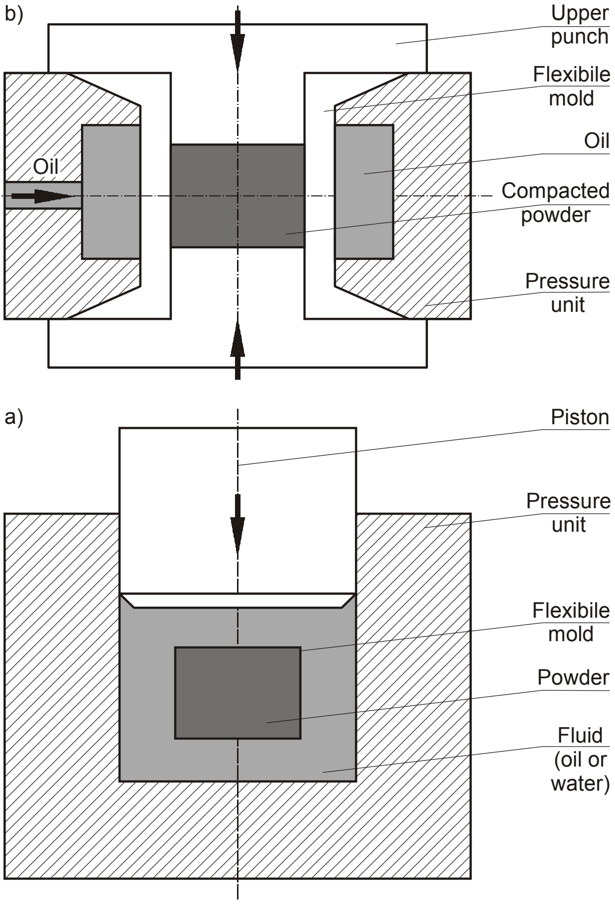

Figure 3. **Cold isostatic pressing processes: a) wet bag CIP, b) dry bag CIP**

Slika 3. **Proces hladnog izostatskog prešanja: a) mokri postupak CIP, b) suhi postupak CIP**

MATERIALS AND METHODS

In this study hardness and densification behaviour of commercial bronze and copper powders are investigated using uniaxial die and wet bag type CIP process. Both of the powders used in experiments were manufactured with atomising method and have spherical shape. The particle sizes of copper and bronze powders were measured with “Malvern Mastersize E” apparatus. Measured powder sizes were d_m = 54,12 μm for copper and d_m = 201,8 μm for bronze. Detailed properties of powder materials are given in Table 1.

Uniaxial Die Pressing

The powders used in experimental studies were compacted with uniaxial compaction as seen in Figure 1. and

Table 1. **Properties of powders used in experiments**
Tablica 1. **Svojstva prahova korištenih u eksperimentima**

Powders	Properties				
	Shape	Particle size μm	Manu-facturing method	Apparent density $g\cdot cm^{-3}$	Tap density $g\cdot cm^{-3}$
Bronze	Spherical	201,80	Atomised	4,5-5,7	4,9-5,7
Copper	Spherical	54,12	Atomised	4,7-5,8	5,1-6,0

Powders	Chemical Analyse		
	Copper	Tin	Phosphorus
Bronze	Balanced	9,73	0,03
Copper	99,58	-	0,05

wet bag CIP as seen in Figure 3a. In compaction processes to obtain approximately the same sample height for both copper and bronze, the same amount of powder material (8g) was used. For uniaxial die compaction of copper and bronze powders "Instron 1081" type 200 kN (max) load capacity press machine was used. It was possible to reach 800 MPa during uniaxial die pressing process. The die set used in uniaxial die compaction was manufactured from high carbon speed steel material. The dimension of compaction chamber in die were 10 mm in diameter and 70 mm in length. The dimensions of the compacted specimens with uniaxial die pressing process were 10 mm in diameter and 5 mm in thickness. Die wall and the surfaces of both punches were lubricated with zinc stearate solution to prevent friction. After each compaction the die wall and punch surfaces were cleaned and re-lubricated for the next pressing operation. In uniaxial die compaction processes, double acting compaction was used to obtain better densification distribution. After compaction process the dimensions (diameter and thickness) of the compacted specimens were measured using a micrometer with an accuracy of ± 0.001 mm. The mass of the specimens were measured with an accuracy of ± 0.001 g. Increase in density at applied pressure was measured using dimensional measurement method. Punch displacements were recorded continuously when applied load increasing. From the experimental displacement data the green density was calculated using the equations given below:

$$d_g = \frac{M}{V_g} \tag{1}$$

$$V_g = \pi r^2 \left(h_i - h_{dp}\right) \tag{2}$$

where:

d_g is the green density,
M is the mass of the green compact,
r is the radius of the die,
h_i is the initial powder height that calculated from the tap density,
h_{dp} is the displacement of the punch.

Wet Bag CIP

A 200 kN (ELE) concrete testing machine was used to generate required pressurisation for wet bag CIP process. It was not possible to reach the pressure level of uniaxial die pressing (800 MPa) with wet bag CIP process.

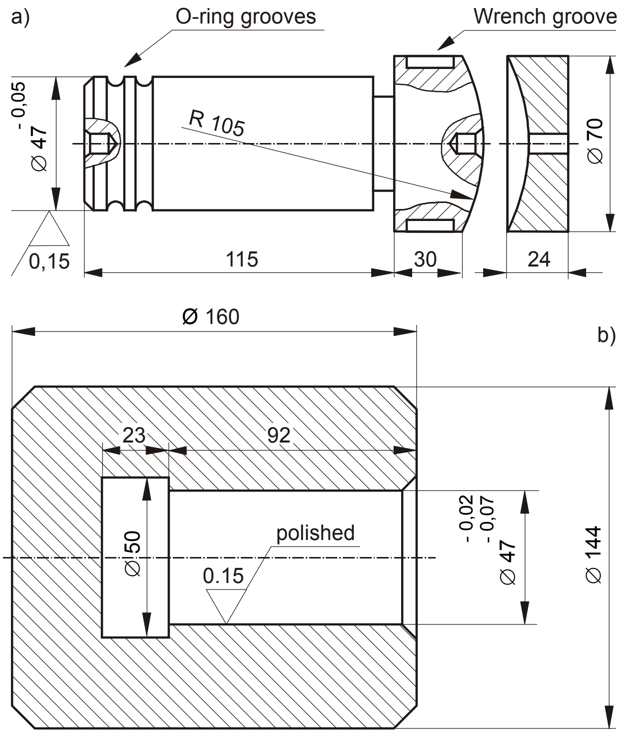

Figure 4. **CIP unit used in the study: a) compression piston, b) pressure unit**
Slika 4. **CIP uređaj upotrijebljen u ovom radu: a) tlačni klip, b) tlačna jedinica**

The O-rings which were used to prevent leakage deformed over 600 MPa pressures. Because of this restriction 500 MPa (max) pressurisation was used for wet bag CIP process. Piston and pressure unit used for CIP process were made of SAE 1040 steel. Details and dimensions of piston and pressure unit are given in Figure 4. Water and mineral oil were used as pressurising media. Flexible mold for powder mass was made of copper in the dimensions given in Figure 5. The internal volume of the flexible mold was mea-

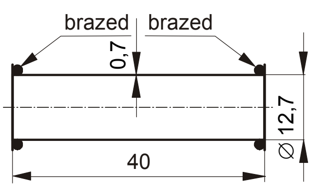

Figure 5. **Dimension of flexible mold (copper tube) used in CIP**
Slika 5. **Dimenzije fleksibilnog kalupa (bakrena cijev) koji je upotrijebljen u postupku CIP**

sured as V_i = 4 cm^3. First, one of end caps of copper tube was brazed, then it was filled with powder. Finally the other end cap was brazed. According to Archimedes principles, the volume of the flexible mold before (V_0) and after pressing (V) was measured using a graduated measuring cylinder. The flexible mold was immersed into the pressure chamber and then pressure piston was fixed to pressure unit. Gradually 100 to 500 MPa pressures were applied to flexible mold using 2000 kN capacity (ELE) testing machine. Densification of powders under applied pressure, resulted in volume change (ΔV) in flexible mold:

$$\Delta V = V_0 - V \tag{3}$$

The volume of pressed powder (V_p) and the mass density of pressed powder (d_m) can be calculated by following equations:

$$V_p = V_i - \Delta V \tag{4}$$

$$d_m = \frac{W}{V_p} \tag{5}$$

where:

W is the weight of powder inside the flexible mold.

The micro-hardness of powder particles before and after compaction was determined using Reichert Hardness Tester. To measure the microhardness, the powders and compacted specimens were embedded in bakelite. Applied force for indentations were 10 and 20 g for copper and bronze respectively. The lengths of diagonals were measured and converted to the Vickers micro-hardness (HV) value. Recorded HV values represent an average of 20 readings of powder and compacted sample. The Vickers micro-hardness value was determined using the following equation:

$$HV = \frac{1854,4\ L}{d^2} \tag{6}$$

where:

L is the applied load in g,
d is the length of diagonals in mm.

RESULTS AND DISCUSSION

Hardness and densification behaviour of copper and bronze powder under applied pressures with different pressurisation methods were investigated. The results of micro-hardness of bronze and copper powders are given in Figure 6. As seen in the Figure pressurisation method does not change the ratio of hardness increase at a defined pressures e. g. almost the same hardness increase values were measured for both of uniaxial die and CIP pressing processes. Initial micro-hardness of bronze and copper powders were measured as 120 HV and 72 HV respectively. Hardness increase at 500 MPa pressure for both of the pressurisation methods were measured as 41 % (101,5 HV) for copper and 44 % (173 HV) for bronze. Measured maximum hardness values of copper and bronze under pressure at 800 MPa with uniaxial die pressing were 118 HV for copper (64 % increase in initial hardness) and 192 HV for bronze (60 % increase in initial hardness). Increasing pressure value results in large numbers of dislocations [9, 10]. Hardness increase of powder can be manifested with the strain hardening as a result of dislocations. Densification ratio of copper and bronze powders under pressure applied

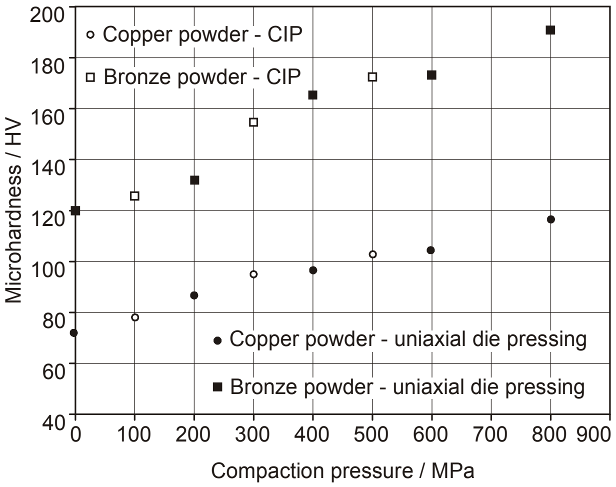

Figure 6. **Micro hardness of bronze and copper powders and compacted specimens under different pressure values obtained with CIP and uniaxial die pressing**

Slika 6. **Mikrotvrdoća prahova bronce i bakra te kompaktiranih uzoraka dobivenih različitim tlakovima postupcima CIP i prešanjem u matrici smjerom jedne osi**

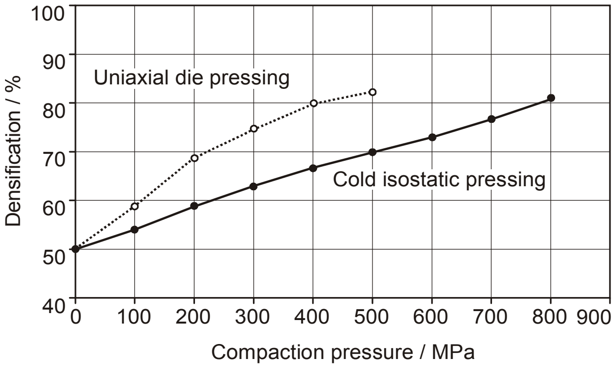

Figure 7. **Obtained densifications of compacted bronze powders with CIP and Uniaxial die pressing processes**

Slika 7. **Dobivena povećanja gustoće kompaktiranog praha bronce sa procesima CIP i prešanjem u matrici smjerom jedne osi** with both of the process are given in Figure 7. and Figure 8. respectively. Packing density for copper is 54 % of theoretical density (TD) and for bronze is 49 % of TD. For both of the powders at 100 MPa pressure the density improved about 10 %. This improvement in density can be attributed to rearrangement of powders with very little plastic deformation [1, 2]. If densification values obtained at 500 MPa pressure level compared for both of the process and the same powder material, it is seen that wet bag CIP process gives better densification than uniaxial die pressing. At 500 MPa pressure, CIP resulted in 15 % better density than uniaxial die pressing for copper and 18 % for bronze powder (at 500 MPa pressure obtained densities are: for copper 82 % uniaxial, 94 % CIP - for bronze 70 % uniaxial, 82,5 % CIP) These better densification results for CIP process can be explained with the situation that in CIP process pressure is applied over greater area than uniaxial pressing [11]. Obtained densities at 500 MPa with CIP process are 94 % of TD for copper and 83 % of TD for bronze. To reach the same density levels with uniaxial pressing 60 % more pressurisation is needed as seen in Figures 7. and 8.

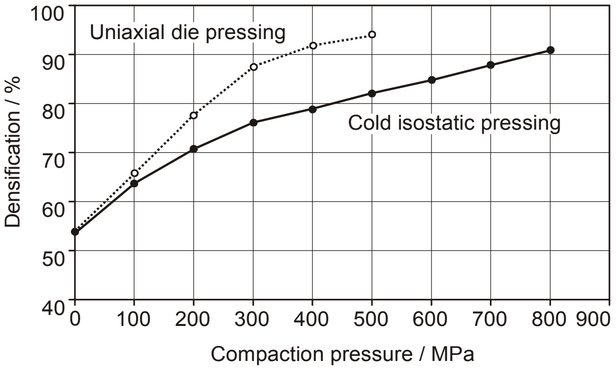

Figure 8. **Obtained densifications of compacted copper powders with CIP and Uniaxial die pressing processes**

Slika 8. **Dobivena povećanja gustoće kompaktiranog praha bakra sa procesima CIP i prešanjem u matrici smjerom jedne osi**

CONCLUSION

From the experimental results given above, following conclusion can be drawn.

1. CIP process transmits the pressure omnidirectional and greater area is effected so it is possible to have higher densification ratio than at uniaxial pressing. At 500 MPa pressure; for copper powder 15 %, for bronze powder 18 % better densification obtained with CIP than uniaxial pressing.
2. Almost full densification (94 %) was obtained for copper powders at 500 MPa pressure with CIP process. For bronze powders densification ratio was measured as 82 % at the conditions.
3. For uniaxial die pressing, 60 % greater pressurisation was needed to attain the same densification as obtained with CIP at 500 MPa.
4. Pressurasing method does not change the ratio of increase of hardness of powders for a defined pressure level. Measured micro-hardness values for both of the pressurising methods at a specific pressure value were same for both of the studied powders (measured micro-hardness values at 400 MPa for copper was about 97 HV for both of uniaxial and CIP process and for bronze was about 170 HV for both of the pressurisation methods).
5. At 800 MPa pressure the micro-hardness of samples increased in the ratio of 64 % of initial hardness for copper and 60 % for bronze. Increase in the hardness can be explained with the dislocations due to pressurisation.

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