

Research Note

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The effect of sintering pressure on the wear behavior of bronze (85/15) alloys produced using hip method

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KEYWORDS

Hot isostatic pressing; CuSn; Microstructure; Wear; Microhardness. **Abstract.** In this study, new material was produced using Hot Isostatic Pressing (HIP) method, upon which Co and Ni were added in different proportions into a CuSnbased (85/15) alloy. Considered as its very important advantage, HIP method allows simultaneous application of pressure and temperature during the process. A constant sintering temperature of 800°C, a constant sintering time of 15 min, and two different sintering pressures of 20 and 30 MPa were used. The sintering process was performed in a vacuum after the initial burning process. The produced samples were subjected to various metallographic processes as well as Scanning Electron Microscope (SEM) and Energy Distribution Spectrometer (EDS) analyses. Afterward, a wear test was carried out on the samples. The study investigated the effects of Ni and Co addition and sintering pressure on the wear behavior. It was found that as the amount of Co and the sintering pressure increased, the wear resistance increased.

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1. Introduction

The Hot Isostatic Pressing (HIP) method is a process in which heat and pressure are applied at the same time to consolidate the produced material. In the consolidation process employing inert gas, the use of isostatic pressure eliminates internal porosity and provides condensation as a result [1]. The HIP is a very flexible process and has many applications in many fields. It allows processing different immobilization matrices and different waste streams without the challenges associated with nitrification. HIP cycle yields no off-gas. One does not need any system to pour out or empty the product [1,2]. Powder metallurgy hot isostatic pressing is a modern method used in high-rate components and complex productions (i.e., for various metals and/or ceramics) and in the chemical, oil, mold, and energy industries. It has become widespread in the market [3,4] and it features many more advantages than various traditional production methods such as casting and forging, as well [4,5]. In practice, it is often too difficult to expect that isostatic pressed samples would exhibit isotropic shrinkage under pressure in theory. Previous studies have revealed the presence of irregular shrinkage and even deterioration [4,6,7]. This problem causes long delivery times as well as high costs for postprocessing. To increase cost efficiency, manufacturers need to produce HIP parts near the net-shape within the geometric limits of the first shot. This can only be

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done with the support of an HIP simulation tool [4]. HIP is a standard finishing process applied to turbine blades made of Ni-based superalloys. This closes the micropores and causes prolongation of stress-fracture life. A subsequent heat treatment is essential to having the desired fine γ - γ' microstructure. HIP is also used to rejuvenate turbine blades in service. Here, creep porosity is removed, thus enabling turbine blades to be reused upon additional heat treatment. The highly specialized and already optimized microstructure can be enhanced using rapid quenching in HIP. For example, Lopez-Galilea et al. pointed out that a high cooling rate inside the HIP unit distinctly tuned the γ - γ' micro-structure of single crystal nickel based on super alloys [8].

Almangour et al. examined the $TiB_2/316L$ stainless steel composite produced using the Selective Laser Melting (SLM) method. They applied the HIP process to increase the final density of the components produced using SLM. Having applied the method due to its annealing effect at high temperatures, they found that there was a significant decrease in hardness and wear resistance [9]. Islam and Farhat stated that the HIP reduced pore size and total porosity and decreased the hardness. The correlation between the benefit of low porosity and detrimental effect (caused by low hardness) ultimately determined the final wear rate [10].

According to Yalçın, the irregular pore formation accelerates upon an increase in the total porosity [11]. Ye et al. found that a decrease in the density of voids led to an increase in the wear resistance and voids, which was considered a stress raiser, thus increasing the probability of cracking and fracture [12]. Chen et al. reported that trapping of wear debris in large surface pores would reduce the plastic deformation and contact pressure near the pores as well as decrease the wear rate. They stated that the pore size was the most important parameter in terms of entrapping wear debris. Low porosity and small pore size make it difficult to capture the debris. Wang et al. reported that pore coalescence on the subsurface layers and microcrack nucleation in the subsurface both contributed significantly to the wear [13]. Qiu et al. examined the impact of HIP temperature on the microstructure and tensile properties of nickel-based superalloy powder [14]. Fu et al. used a HIP technique to produce different Cr_3C_2 containing Ni₃Al based composites. Cr_3C_2/Ni_3Al composites were found to increase wear resistance when Cr_3C_2 content was within a certain range. Upon the addition of excess Cr_3C_2 , the wear resistance of composites decreased [15]. Cios et al. stated that when chromium carbides were present in iron, cobalt, and nickel matrices, they exhibited excellent high-temperature oxidation resistance and high-temperature hardness and, also, had a good

wetting ability [16]. Yıldız et al. investigated the effect of sintering temperature on the microstructure and mechanical properties of alloys produced by the HIP method and found that their microstructure and mechanical properties improved upon increasing sintering temperatures [17]. Han et al. produced samples with a high hot pressing method by adding Ni, Fe, and Co to 75% Cu-25% Sn alloy. They examined the microstructure and mechanical properties of the produced samples. They stated that when the sintering temperature was above 700°C, the weight loss of Fe and Ni doped samples was low, while that of Co doped samples was high [18]. Cai et al. examined the effects of annealing on the wear behavior of pure copper. They stated that the annealing process increased the grain size of Cu and caused a decrease in hardness. They determined that the coefficients of friction decreased following a rise in normal load, while the rate of wear increased [19]. Ram Kumar et al. produced samples with liquid metallurgy by adding the SiC compound to the Cu-Sn alloy. Mechanical properties and dry sliding wear behaviors of the produced samples They stated that as the applied were examined. load and sliding distance increased, the rate of wear increased [20]. Sathishkumar et al. investigated the microstructure and wear behavior of heat-treated Cu-11Ni-6Sn alloys. The wear rate represented a factor that would reduce the hardness of the alloy and follow the wear adhesion theory [21]. Radhika and Sam added SiC and Al_2O_3 compounds to the Cu-10Sn-5Ni alloy, strengthened them using a horizontal centrifuge cast technique, and investigated the wear behavior of these allovs [22]. Martínez et al. evaluated the microstructure, mechanical properties, and tribological properties by producing nanocrystalline Ni with hot pressing and HIP method. Accordingly, by performing hot pressing at low temperatures, Ni prevented grain growth, which led to its excellent mechanical and tribological properties [23].

In this study, with the addition of Ni and Co elements to CuSn (85/15) alloy, the effects of these elements and the sintering pressure at the production stage on the wear resistance of CuSn were investigated. This study aims to increase the wear resistance of CuSn alloys, which are widely used in the automotive industry, sliding bearings, and bushings, and to reduce wear losses.

2. Experimental work

This study was conducted to produce a new matrix material by adding Cobalt and Nickel into an 85/15 Bronze alloy at certain ratios. The grain sizes of the powders are as follows:

• Bronze, 70 μ m;

- Cobalt, 35 μ m;
- Nickel, 5 μ m.

Before initiating the sintering process, the powders were prepared in the percentages listed in Table 1. The polyethylene glycol of 1% was added into

the mixture to process the powders in a specific shape and size. Co and Co alloys were evaluated as alternative materials for the diffusion barrier layer due to their excellent wettability and low solubility. The composition was then mixed in a mixing unit for 20 minutes. Iron chains and beads of different diameters were added to the mixing unit to obtain a more homogeneous mixture. Finally, the samples were applied to the pre-pressing with the sizes of $10 \times 10 \times 40$ mm. To perform a better sintering process, graphite mold was oiled and pre-pressed samples were taken into oiled graphite molds.

The new matrix materials were produced by Celmak Industry and Commerce Ltd. company. The constant sintering temperature of 800°C, a constant sintering time of 15 min, and two sintering pressures of 20 and 30 MPa were employed in the production of the samples. Sintering temperature was taken as 2/3 or 4/5 of melting temperature for single-component systems. On the other hand, in multicomponent systems, sintering temperature was selected below the melting temperature of the component with the highest melting temperature and above the melting temperature of the component with a low melting temperature (melting temperature of Cu was 1085°C; melting temperature of Sn was 231.9°C). The samples were produced with a HIP method, which is a powder metallurgy production method. The sintering process was performed in a vacuum after initial burning. The matrix materials were produced in $10 \times 10 \times 40$ mm dimensions. Table 1 shows the production parameters of metal powders used during production.

Hot pressing is conducted by applying heat and pressure to the sample simultaneously, which in turn



Figure 1. Wearing apparatus.

ensures the removal of the majority of the gaps and a bond strength with a uniform flow between all of the particles. The proposed hot pressing method is a single-stage process and is widely used in the industry.

The samples were prepared through the production process for metallographic examinations. The Energy Distribution Spectrometer (EDS) analysis was performed to determine the ratios of the elements in the samples. Before that, the samples were etched with ether. Microhardness measurement was performed at a load of 50 gr in the waiting period of 10 seconds and at a distance of 1 mm to determine the hardness of the samples. Afterwards, the samples were subjected to an abrasive wear test to determine their wear resistance. The abrasive wear samples were worn after being shaped into $10 \times 10 \times 10$ mm sizes. Figure 1 shows the wear apparatus. The abrasive wear test was specifically performed on the surface of the sample by using an abrasion apparatus with a pin-on-disc system and a

Group no	Sintering pressure (MPa)	$\begin{array}{c} {\bf Sintering} \\ {\bf temperature} \\ (^{\circ}{\bf C}) \end{array}$	$egin{array}{c} {f Sintering} \ time \ (min) \end{array}$	$egin{array}{c} {f Bronze} \ ({f wt}.\%) \end{array}$	Co (wt.%)	Ni (wt.%)
N1	20	800	15	97	0	3
N2	30	800	15	97	0	3
N3	20	800	15	92	5	3
N4	30	800	15	92	5	3
N5	20	800	15	87	10	3
N6	30	800	15	87	10	3
N7	20	800	15	82	15	3
N8	30	800	15	82	15	3

Table 1. The parameters for the sample production.



Figure 2. Scanning Electron Microscope (SEM) images of samples N1 and N2 produced without Co.

120-mesh SiC sanding cylinder and by applying a 10 N load individually at distances of 10, 20, 30 m. Likewise, the turning lathe rotation speed and feed rate were set to 16 rpm and 2.8 mm, respectively. Total distance was found by calculating the circumference of the cylinder while taking its diameter into account. The rotation speed of the turning lathe was 16 rpm. In the system, the sample works by applying a load to abrasive wear from the top with a straight angle of 90°.

3. Results and discussion

3.1. Microstructure results

Figure 2 shows the Scanning Electron Microscope (SEM) images of samples N1 and N2 produced without Co, but rather by adding 3 wt.% Ni at 800°C under 20 and 30 MPa of pressure. These images point to the increase in the density of the grains upon a rise in pressure over sample N2. After sintering, the crystallite size increases, while the number of crystalline defects decreases [23].

Figure 3 shows the EDS analysis of sample N1. This sample had no Co element, which was supported by the EDS analysis. Ni element of 2.10 wt.% was found in this sample group with 3 wt.% Ni.

Figure 4 shows SEM images of samples N3 and N4 produced with 5 wt.% Co and 3 wt.% Ni at 800° C un-

der 20 and 30 MPa pressure. The comparison of these images illustrated that the grains with concentrated Ni and Co elements became more apparent.

Figure 5 shows the EDS analysis of sample N3. This sample contained 5 wt.% Co. A fibrous structure was detected using EDS analysis with 0.21 wt.% Co and 0.29 wt.% Ni.

Figure 6 shows SEM images of samples N5 and N6 produced with 10 wt.% Co and 3 wt.% Ni added at 800°C under 20 and 30 MPa pressure. Here, it was observed that the structure of these samples could be closer to one another than the other samples.

Figure 7 shows SEM images of samples N7 and N8 produced with the addition of 15 wt.% Co and 3 wt.% Ni at 800°C under 20 and 30 MPa pressure. It was observed that Co was accumulated to a greater degree on grain boundaries and was more homogenous under higher pressure.

3.2. Microhardness results

Figure 8 shows the microhardness graph of the samples N1, N2, N3, N4, N5, N6, and N7 produced under pressure of 20 MPa and 30 MPa. According to this graph, the microhardness increased as the rate of Co elevated. The microhardness was 197 HV in the sample N1 without Co addition produced at 20 MPa of pressure and was 201 HV in the sample



Element	Weight %	Atomic %
Ni Cu Sn	$2.10 \\ 85.85 \\ 12.05$	$2.40 \\ 90.78 \\ 6.82$
Total	100.00	

 $10 \ \mu m$ Electron image 1

Figure 3. Energy Distribution Spectrometer (EDS) analysis of sample N1.

T. Yıldız et al./Scientia Iranica, Transactions B: Mechanical Engineering 28 (2021) 2259–2266



Figure 4. Scanning Electron Microscope (SEM) images of samples N3 and N4 produced with 5 wt.% Co.



Element	Weight $\%$	Atomic %
Co Ni Cu Sn	$\begin{array}{c} 0.21 \\ 0.29 \\ 86.33 \\ 13.17 \end{array}$	$0.24 \\ 0.34 \\ 91.92 \\ 7.51$
Total	100.00	

 $3 \ \mu m$ Electron image 1

Figure 5. Energy Distribution Spectrometer (EDS) analysis of sample N3.



Figure 6. Scanning Electron Microscope (SEM) images of samples N5 and N6 produced with 10 wt.% Co.



Figure 7. Scanning Electron Microscope (SEM) images of samples N7 and N8 produced with 15 wt.% Co.



Figure 8. The microhardness results for the samples produced at different ratios under 20 MPa and 30 MPa.

N7 with addition of 15 wt.% Co produced under 20 MPa of pressure. Correspondingly, it was found that microhardness values increased with increasing ratio of Co. The microhardness of the sample N2 without Co produced under 30 MPa pressure was 210 HV, whereas it was 216 HV in the sample N8 with addition of 15 wt.% Co under 30 MPa pressure.

3.3. Wear rest results

Figure 9 shows the wear resistance graph of the samples produced under 20 MPa pressure and a load of 10 N at 800° C at distances of 10, 20, and 30 m. The wear results were transferred by establishing a mass loss-distance relationship. It was found that the wear resistance increased due to the increased rate of cobalt. Moreover, the sample N7, which contained 15 wt.% Co, exhibited the highest level of wear resistance.

Figure 10 shows the wear resistance of the samples produced under 30 MPa pressure and a load of 10 N at 800°C at distances of 10, 20, and 30 m. Similar to the samples produced with a sintering pressure of 20 MPa, it was found that among the samples produced with a sintering pressure of 30 MPa, the highest wear resistance was observed in the sample with the highest 15 wt.% Co. It was observed that the rise of pressure would ultimately increase the wear resistance because of mass loss reduction. Following an increase in the Sn content in the grinding wheel, the Cu–Sn bond becomes more brittle and self-sharpening, which means that the bond is highly prone to wear and the grinding efficiency improves remarkably [18].

4. Conclusions

The proposed study presents a new material produced using the Hot Isostatic Pressing (HIP) method. Co and Ni were added in different proportions into a CuSn based (85/15) alloy. The production process includes the parameters of a constant sintering temperature of 800°C, a constant sintering time of 15 min, and two different sintering pressures of 20 and 30 MPa. Obtained results of the experiments are briefly listed below:



Figure 9. Wear graph of the samples manufactured under 20 MPa of pressure with 10 N load.



Figure 10. Wear graph of the samples manufactured under 30 MPa of pressure with 10 N load.

- 1. The sintering pressure, sintering temperature of metal powders, and proportion and quantity of alloying elements were found important parameters that changed the properties of the parts;
- 2. The production of the samples went very smoothly at no cost. This is one of the most important advantages of Hot Isostatic Pressing (HIP) compared to the powder metallurgy production method;
- 3. Based on the increasing sintering pressure in the microstructure images, the metal powders in the samples were homogeneously distributed;
- 4. In terms of hardness measurements, the amount of hardness increased due to increase in cobalt ratio and sintering pressure;
- In terms of the applied load and wear distance, samples N7 and N8 exhibited the highest level of wear resistance. This occurred when the Co ratio was 15 wt.%;
- 6. Increase in sintering pressure and cobalt ratio was effective in increasing the wear resistance.

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Abbreviations

HIP	Hot Isostatic Pressing
PM HIP	Powder Metallurgy Hot Isostatic
	Pressing
SEM	Scanning Electron Microscope
EDS	Energy Distribution Spectrometer
SLM	Selective Laser Melting

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