Microstructure and densification behavior of liquid phase sintered Fe-Cu alloy powder using cold and hot compaction techniques

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Abstract

The physical and mechanical properties of atomized prealloyed Fe-Cu powders, blended with different amounts of liquid additions of lead (Pb), were studied in the as-sintered condition and hot compaction techniques. The influence of Pb content, compacting pressure and temperature on the densification, hardness, and the mechanical properties were investigated. During hot compaction, at a temperature of 500°C, the Pb liquid was found to spread uniformly among Fe-Cu solid particles. The effect of pores in Fe-Cu-Pb alloys, generated by sintering with transient liquid phase, had been studied. An attempt was made in order to study the properties of Fe-Cu-Pb particles and their behaviour, with respect to the consolidation of Fe-Cu-Pb powders. The density values of cold and hot compacts, at various pressures and temperatures values, were reported. The microstructure, hardness, and strength measurements were found to be dependent upon the compacting pressure. For the cold compacted alloys, the Pb powder particles were completely melted to form liquid pools. In addition, increasing the Pb content in the alloy matrix revealed a decrease of the pores percentage, hence the sample became denser. On the other hand, grain was found to be coarser and less porosity is obtained with increasing the Pb content in the hot compacting pressure of the cold and hot compacted samples revealed a homogenous, fine grain, and small pores appeared around the grain boundaries. The mechanical properties data showed improvement in the strength and hardness of the hot and cold compacted samples by increasing either the compaction pressure or temperature. Copyright © 2017 VBRI Press.

Keywords: Cold compaction, hot compaction, compaction pressure, compressibility factor, relative density.

Introduction

Fe-Cu is a commonly utilized material in powder metallurgy (PM), where copper acts as an alloying element and is not sensitive to oxidation [1-2]. Copper (Cu) is the most common alloying element added in powder form because of its low cost, availability and ability to improve the properties of alloys [3]. Cu as an alloying substance has unique properties in the field of sintered steels where it improves strength and rust resistance. In addition, Cu melts at a lower temperature, has a rapid surface diffusion over solid iron, and a liquid phase where iron easily diffuses [4]. In PM It is well established that residual pores are deleterious to mechanical properties and corrosion resistance. To improve densification process of Fe-Cu parts produced using PM technique a sintering process called liquid

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phase sintering (LPS) is common used. Accordingly, LPS of ferrous materials can be utilized in systems where it is difficult to achieve high density [5]. LPS is a sintering technique in which at least one of the constituents of the powder mix is in liquid state at the sintering temperature, or below the sintering temperature. In LPS process, the sintering treatment is accomplished above the melting point of Cu content. The main objective of producing a liquid phase during sintering is to improve densification due to the enhancement of the diffusion processes that take place in a more efficient way in liquid than in solid state [6]. During LPS, a coexistence of liquid phase and solid particulate takes place at sintering temperature [7]. When the liquid presents a good wetting ability and the solid has pores in its structure, then the liquid will infiltrate through the porosity channels in the substrate due to capillarity effect which resulted in significant

improvement in porosity morphology and, in turn, mechanical properties of parts is attained. Besides, the liquid will tend to penetrate through pores with smaller diameter, because of higher capillarity forces [7]. Sintering will improve all wetted surfaces which plays an important role in achieving full densification, providing that the material of the solid is soluble in the liquid-phase and the characteristics of liquid's wetting solid particulate are in good shape. This improved sintering would lead to growth along the wetted grain boundaries, where grains situated at free surfaces will consequently expand in all lateral directions. At the same time, the un-wetted free surface is depleted of material, which could result in a smoothing effect on the material surface [9-10]. In addition, it is common known that LPS releases enough internal force in conventional PM through liquid capillary action on the particulate solid, such that there would be no need for external forces for the compaction during sintering [8]. Accordingly, LPS allows the commercial development of PM parts further via decreasing sintering temperature and time.

Densification during LPS could be occurring through several mechanisms, including solid-state diffusion, particle rearrangement, and the dissolution and solutionreprecipitation stage [11]. When the powder compact is heated to sintering temperatures, the binder becomes liquid and spreads through the void space in between the solid hard-phase particles [12]. Accordingly, the filling of pores by the liquid is the process for densification and governed the overall sintering kinetics. For most systems, the rate of densification usually increases when the liquid forms, dissolving solid-solid contacts and exerting capillary forces to enable better packing through particle rearrangement [13]. Capillary force provided by the liquid pulls solid grains together by spreading into the pores of the solid-liquid-pore structure and wetting the particles; at the same time, delivers a rapid diffusion rate whereas its motion eliminates pore; meanwhile, a softening takes place in the solid due to elevated temperature which aids further in densification [14-15]. Explanation of liquid phase sintering way of action could be summarized in three overlapping actions; as shown in Fig. I (as shown in the supplementary information file) for the case of two mixed powders, where a rearrangement and rapid shrinkage occurs for the solid material; meanwhile, a dissolution and re-precipitation takes place along with densification; moreover, an occurrence of coalescence when the liquid phase disappears [16]. Efficient densification could be achieved with guaranteeing the essential requirement of particle rearrangement. On the other hand, the possibility of rapid sintering densification of pretty large pre-alloyed powders is attained by surpassing the solidus temperature. However, this process isn't common and is seldom used, due to the large probability of distortion [14]. It is worth mention here that, the densification during LPS is analogous to viscous flow sintering; the matrix viscosity decreases as the liquid volume fraction increases. Therefore, more liquid is required to enhance the sintering process [17-18].

sintering temperatures, Fe and Cu particles are wetted and bind strongly together by the molten Pb powders. The sintering behavior of Fe-Cu alloys, made from mixed elemental powders, is found extensively in literature [7, 9-10, 19]. Sintered Fe-Pb-Cu porous bearing act is considered to be as good as sintered bronze porous bearing [20]. The mixture benefits from the addition of Pb in terms of increased softness, which makes it very close to steel shafts, and improved lubrication capability, as a result of its low melting point. In other words, on one hand, the soft phase acts as lubricant and provides an easily sheared surface layer. On the other hand, the hard matrix phase is strong enough to support relatively high load [21]. The manufacturing sintered Fe-Pb-Cu porous bearings doesn't have standard conditions; however, there are many attempts in literature to study this issue, where some publications have dealt with the densification of granulated materials [22-26].

Mixtures of Fe and Cu powders are frequently adopted

to produce high strength steel parts. At supersolidus

Accordingly, the density, porosity, sintering temperature and time, alloying elements, compacting pressure etc. are the important factors which govern the mechanical properties of components manufactured by PM route [27]. In this research, a novel study was carried out to explore the effects of Pb content, compacting pressure and compacting temperature on the physical and mechanical properties of the Fe-Cu compact. The alloys were fabricated using two different PM techniques; cold compaction followed by sintering cycle and hot compaction technique. Consequently, all the produced samples of the two techniques were subjected to heat treatment.

 Table 1. The production parameters of Fe-5Cu-(x)Pb alloys.

	C	P	HP		
Condition	P(MPa)	t(min)	P(MPa)	T(°C)	t(min)
Production					
parameters	300	10	300	500	10
Sintering					
parameters	900°C/1h/FC			-	

 Table 2. Different condition used for the production of Fe-5Cu-4Pb.

Condition	СР			HP
	P(MPa)	P(MPa)	T(°C)	t(min)
		250	500	10
		300	300	10
Production	250-up to- 600		400	10
parameters	•			5
			500	10
				15
		350	500	10
Sintering parameters	900oC/1h/FC			

Experimental

Preparation of specimens

The elemental powders of Fe, Cu, and Pb were used as the as-received powders, with purity greater than 99%; average particle size less than 10 μ m; and manufactured

by ALDRICH, Germany. The various powder components were mechanically mixed forming the nominal composition as namely, Fe-5Cu, Fe-5Cu-4Pb, Fe-5Cu-8Pb, and Fe-5Cu-12Pb weight percentages. The weights of powders and compaction parameters were designed to produce specimens with specific listed in **Table 1**. compositions; Two different PM techniques were used in the fabrication of LPS specimens; cold compaction and hot compaction. Among the different Fe-Cu-Pb alloys, this study concentrates deeply on the Fe-5Cu-4Pb alloy. Table 2 shows the different cold and hot compaction conditions used in this study.

Powder processing

Cold compaction technique

Cold compaction was performed in a single-acting piston cylinder arrangement. The die bore was smeared with graphite powder to reduce the die-wall friction. Desired weights of mixed alloys were used for each compact. A hydraulic testing machine, of 200 tons capacity, was used to perform the compaction of the alloy powder, with a constant cross head speed of 2 mm/min. The height of the green compact was measured directly before and after die ejection; moreover, the final height was also calculated from the load-displacement curve. After unloading, the elastic recovery of the compacts was neglected. No lubricants were mixed with powders; however, graphite powder was applied to the die wall before powder filling. The annular disc specimen had an outer diameter of 45 mm. After the compact operation, the samples were covered with aluminum foil and embedded in a graphite powder, to protect their surfaces from reacting with atmospheric oxygen and nitrogen during the sintering process. The samples were heated at a constant heating rate of 20 °C/min to reach a temperature of 900°C, kept in furnace for one hour, and left to be cooled to accomplish the sintering process. Porosity and density of the compacts after sintering operation were calculated.

Hot compaction technique

All the hot compaction samples were subjected to single sided uniaxial hot compaction in a single acting piston cylinder arrangement as shown in **Fig. II** (as shown in the supplementary information file) **[28]**; to obtain cylindrical samples of 10 mm in diameter and 15 mm in height. The hot compaction die had a punch surrounded by an electric heater; it was manufactured from high strength steel mold. The die bore was smeared with the intension of powders reducing die-wall friction; the desired weights of mixed composites were used for each compact. A hydraulic testing machine of 200 tons capacity was used to perform the compaction of the alloy powder with constant cross head speed of 2 mm/min. The hot compaction temperature was measured using a NiCr-Ni thermocouple (type K), which was inserted into the die

near the sample cavity. The temperature was maintained at the required level (500 °C), with a tolerance of \pm 5 °C. The setup was heated up to a preselected temperature, which was fixed for 30 minutes; in order to homogenize the temperature throughout the powder alloy. Consequently, compact was pressed under 314 MPa, with duration of 10 min; therefore, the forming pressure was lowered for all tested hot compacts. Heat treatment operations were conducted for some cold and hot pressed alloys; heating was done at 900°C for 8 hours, which was followed by water quenching.

Mechanical and microstructure characterization

Compression tests were carried out for both cold and hot compacted samples, to determine the mechanical behavior of Fe-Cu-Pb alloys at room temperature, using an Instron 8562 universal testing machine, under quasi static loading and strain rate of $8 \times 10^{-5} \pm 5\%$ s⁻¹; where samples were deformed until failure. Cylindrical specimens were prepared from compacted rods, with a diameter of 5 mm and height of 7.5 mm. Three identical samples were prepared for each test case and exposed to the same loading conditions, to ensure consistency and homogeneity. The mean test value for all of the three samples was reported in results. The stress-strain responses of cold and hot compaction process, of the Fe-5Cu-8Pb specimen (at 300 MPa), and two cold pressed of Fe-5Cu-4Pb specimens (at 300 and 500 MPa), were measured from a uniaxial compression test performed according to ASTM E-9 standard of metals. The crosshead speed was maintained at 1 mm/min [29]. Rockwell hardness measurements were performed for different compacted samples at 60 kg load, using digital Rockwell hardness tester. A Leica Eclipse optical microscopy (OM) was used to produce microstructural images for both the cold and hot compacts. Microscopic examination was performed using a Jeol 5400 scanning electron microscopy (SEM), with a Link EDX detector attachment, to observe particle morphology, size, shape, and agglomeration.

Results and discussion

The primary objective of the present work is to develop a low melting element, to be used as a liquid phase in the sintering of Fe-Cu compacts. The effect of Pb content on the microstructure of Fe-5Cu-(x) Pb specimens; which are cold pressed, with 300 MPa, and sintered at 900 °C for one hour then furnace cooling, is shown in **Fig. 1**. The Pb powder particles are completely melted to form liquid pools. The liquid phase penetrates the particle boundaries, which possesses higher free energy. Increasing the Pb content in the alloy matrix resulted in decreasing the pores (**Fig. 1a-to-d**). Most of the liquid phase is localized at the site originally occupied by the Pb powder; as shown in **Fig. 1**. Therefore, it gradually penetrates the particle boundaries of the Fe-Cu matrix.

For the hot-pressed samples (Fig. 2), increasing the Pb content leads to the increase in wettability of solid

particles, by the liquid spreads all over grains within a thin film of the liquid phase.



Fig. 1. Microstructure of Fe-5Cu-xPb alloys cold pressed at 300 MPa for 10 min and sintered at $900^{\circ}C/1$ h/FC for a Pb percentage of (a) 0, (b) 4, (c) 8, (d) 12 wt%.

From **Fig. 2a-to-d** it can be revealed that, grain sizes are found to increase with the increase of Pb content and less porosity is obtained; for hot compaction samples at 500°C under compacting pressure of 300 MPa. In addition, Pb liquid encircles Fe-Cu matrix grains, on the sintering onset. **Fig. 3** presents the effect of applied pressure on the microstructure of Fe-5Cu-4Pb specimens; produced by cold and hot compaction techniques, at various compaction pressures, with holding time of 10 min. For the cold pressed samples (**Fig. 3a, c**), it is clear that the Cu particles present within the structure were significantly less deformed into Fe matrix of the cold compacted sample pressed at lower pressure (250 MPa).



Fig. 2. Microstructure of Fe-5Cu-xPb alloys hot pressed at a pressure of 300 MPa and a temperature of 500 °C for 10 min for a Pb percentage of (a) 0, (b) 4, (c) 8, (d) 12 wt%.

It was visible that the cold compacted samples showed irregular particle shape and inhomogeneous particle size distribution after compaction, with a number of pores still remaining, which also varied considerably with different pressures. Higher compacting pressure resulted in a homogenous, fine grain, and small pores appeared around the grain boundaries. Consequently, the material becomes denser.



Fig. 3. Microstructure of (a, c) cold pressed and (b, d) hot pressed Fe-5Cu-4Pb alloy pressed at a pressure of (a, b) 250 MPa and (c, d) 350 MPa.

The interconnected porosity shown in **Fig. 3a** could be due to the lower compacting pressure during green compacts forming. The sintered Fe-5Cu matrix compact can only reach a sintered density of about 82.7% of the theoretical density due to compacting without Pb which melt to form liquid phase. The minimum porosity of 12.5 %, for cold compaction technique, is obtained under a compacting pressure of 600 MPa.

For the hot compaction samples, upon increasing the compacting pressure, the atoms of the liquid phase diffused inside the solid powder matrix and the liquid film thickness decreased gradually. The characteristics of powder aggregates changed and the microstructure appeared more clearly, when increasing the hot compaction pressure; as presented in Fig. 2d (P = 350 MPa) compared to Fig. 2b (P = 350 MPa). The diffusing severity of liquid atoms into the solid matrix is the disappearing of liquid phase partially from grain boundaries. The molted Pb and Cu inter-spread and after reaching the equilibrium state, Pb receded from the copper. In addition, increasing the hot compacting pressure, (i.e. increasing the densification rate) resulted in producing of denser products with smaller grain sizes than achieved by sintering. It is worth mentioning, the formation of liquid phase during sintering or hot compaction plays an important role in enhancement the densification process.

Scanning electron microscopy (SEM) investigation was conducted on hot pressed specimens. **Fig. 4** shows the microstructure of the hot-pressed Fe-5Cu-8Pb alloy, at 300 MPa/500°C/10 min. General chemical analysis using EDX technique showed that the Pb content of a sample containing, initially, 8 wt.% Pb decreases after hot compaction to 2.17 wt%. This finding could be attributed to Pb liquid leakage after particles wetting and pores filling, which would lead to an enhancement in matrix densification. **Table 3** (as shown in the supplementary information file) illustrates the major constituents of Fe-5Cu-8Pb hot pressed alloy yielded from the EDX analysis at different spot locations.

The compressibility factor (C_j) is defined as the ratio between density change and the compacting pressure (P_p) , the compressibility factor can be measured according to the following relationship **[18]**:

$$Cf = (\rho m - \rho a) / \sqrt[3]{Pp}$$
(1)

where, ρ_m is the measured density, and ρ_a is the apparent density after applying initial load of 1 KN.

Fig. 5a shows the dependence of the relative density on the compacting pressure for the cold and hot compacted Fe-5Cu-4Pb. From Fig. 5a it is clear that the relative density of the cold and hot compacted Fe-5Cu-4Pb alloy increases with the increase of compact pressure. Fig. 5b shows plots of compressibility factors versus the cube root of the applied pressure, for cold and hot-pressed alloys. Such relationships are found to be linear, and the compressibility factor decreases upon the increase of the compacting pressure. Accordingly, increasing the compacting pressure can assist all three porosity reduction mechanisms (movement of particles into voids, deformation of particles, and flatting of the microscopic and submicroscopic features on the particle surface). As compaction pressure increases, the distance between powder particles gets closer and the destruction of the oxide layer on the surface of powders is accelerated, resulting in increased green density [17]. It is worth mention here that, LPS is a crucial densification process used to achieve multiple-phase components and materials with high-performance due to the significant role of the binder which melt and assist in filling the matrix voids. In addition, increase in density is explicable by increase in pressure, since it increases the number of contact points in powder bits.



Fig. 4. (a) SEM and (b) spot locations (of point 2 in Fig. 4a) of EDX analysis for Fe-5Cu-8Pb hot pressed alloy at 300 MPa/500 °C/10 min.

Accordingly, densification increased more significantly, in hot process compared to cold, by increasing the compacting pressure; and the porosity decreased slowly (**Fig. III**). The highest value of total porosity was observed for Fe-5Cu sample, and the lowest for sample with 12 wt% of Pb, at the same sintering cycle (900°C for 60 minutes, followed by furnace cooling) which can be attributed to the crucial role of the binder which melt forming the liquid phase which assist in filling the matrix pores resulting in improving the sample density.

Fig. IVa (supplementary information file) shows the variations of relative density, with the compaction time; for both cold and hot compacted samples at pressure of 300 MPa. It is evident that the hot compaction process yields more satisfactory results, as far as the relative density is concerned; this finding is probably attributed to the heating effect during that process. **Fig. IVb** (supplementary information file) shows the effect of compaction temperature on both of the relative density and porosity of the hot compacted Fe-5Cu-4Pb alloy.

From **Fig. IVb** it is clear that the relative density of the Fe-5Cu-4Pb alloy increases by increasing the compaction temperature due to the deterioration in the porosity of the hot compacted samples by increasing the compaction temperature. In addition, the increased compressibility can be related to the enhanced plastic deformability of the metallic powders by increasing the temperature.



Fig. 5. The influence of the compaction pressure on (a) the relative density and (b) the compressibility factor of cold and hot compacted Fe-5Cu-4Pb compacts.

Fig. 6a illustrates the true stress-strain curves for cold and hot compaction process of Fe-5Cu-8Pb alloy; previously pressed at 300 MPa. Densification of the cold compacted alloys is, generally, less than the hot compacted ones. In addition, it is noticeable that the strength of the sintered compacts has been improved with the increase in the compacting pressure (**Fig. 6b**) for the Fe-5Cu- 4Pb, which is a result of a closer packing density of the powder particles. The addition of Pb in the Fe-Cu leads to a noticeable increase in strength; which is revealed in **Fig. 6a** (Fe-5Cu-4Pb alloy) compared to the lower content alloy (Fe-5Cu-4Pb alloy) cold pressed at the same compacting pressure (300 MPa) showing a yield stress of ~ 140 MPa and ~100, respectively. This can be attributed to the significant effect of the binder content in filling the matrix pores during LPS. Additionally, from **Fig. 6b**, it can be revealed that the increase in densification with increasing the compacting pressure for the cold compacted Fe-5Cu-4Pb alloy which can be referred to the increase in sample densification with increasing the compaction pressure (**Fig. 5a**); therefore, the rise in the applied pressure from 300 to 500 MPa resulted in increasing the alloy strength (**Fig. 6b**).



Fig. 6. True stress-strain curves for (a) Fe-5Cu-8Pb and (b) Fe-5Cu-4Pb alloys processed using (a) hot and cold compaction and (b) cold compacted at 300, 400, and 500 MPa.

The effects of compaction pressure on the hardness values of cold and hot-pressed alloys are illustrated in Fig. 7a. It is revealed that the hot-pressed alloys are much harder compared with the cold pressed counterparts, at the same compacting pressure; this is due to higher densification of the hot-pressed alloys than that of cold pressed alloys. Accordingly, the higher densification rate of the hot compacted alloys resulted in decreasing the porosity which leads to denser alloys (Fig. IVa). The pores in the hot compacted Fe-5Cu-xPb alloys (Fig. 2) are smaller, rounder, and uniformly distributed throughout the samples compared to the cold pressed samples (Fig.1); hence, higher strength and hardness are achievable. On the other hand, at elevated temperature, the lubricants can be redistributed from inter-particles to the die-sample interface. Consequently, higher density is attained.

Increasing the hot compaction temperature also resulted in increasing the compactibility and strength of the compact, this trend can be attributed to the reduction of the yield stress of the powder with temperature rise. It is notable that in the compaction of metal powders, the hot compaction is employed to improve the compactibility and strength of metallic compactions. **Fig. 7b** shows the effect of the Pb contents on the both cold and hot-pressed Fe-5Cu-(x)Pb. From **Fig. 7b**, it revealed that the linear slight increase in hardness, with the rise in Pb content, for both hot and cold pressed alloys. In addition, heat treatment process gives the hardness increases for both cold and hot-pressure as shown in **Fig. V** due to the rise in metal hardness after water quenching.



Fig. 7. The effect of (a) compaction pressure on the hardness of Fe-5Cu-4Pb alloy and (b) Pb content on the hardness of Fe-5Cu-(x)Pb alloys.

Conclusion

The physical and mechanical properties of atomized prealloyed Fe-Cu powders, blended with different amounts of Pb were studied in the as-sintered condition and hot compaction techniques. The influence of Pb content, compacting pressure and temperature on the densification, and Rockwell hardness were analysed. For the cold compacted alloys increasing the Pb content in the alloy matrix revealed a decrease of the pores percentage, hence the sample became denser. On the other hand, grain was found to be coarser and less porosity is obtained with increasing the Pb content in the hot compacted. For the cold compacted samples pressed at low pressure, it is clear that the Cu particles present within the structure were significantly less deformed into Fe matrix. The low compacting pressure of the cold compacted samples during green compacting resulted in a noticeable interconnected porosity. Increasing the compacting pressure of the cold and hot compacted samples revealed a homogenous, fine grain, and small pores appeared around the grain boundaries. Accordingly, the minimum porosity of 12.5 % is obtained in the cold compacted samples under a compacting pressure of 600 MPa. The EDX analysis of the hot compacted Fe-5Cu-8Pb alloy revealed a decreasing in the Pb content after hot compaction to 2.17 wt% due to the spreading of the Pb through the void. It is worth mention that the compressibility factor decreases upon the increase of the compacting pressure. In addition, the densification increased more significantly, in hot processed sample compared to cold ones, by increasing the compacting pressure. The mechanical properties data showed improvement in the strength and hardness of the hot and cold compacted samples by increasing either the compaction pressure. The addition of Pb in the Fe-Cu leads to a noticeable increase in strength. Additionally, the hot pressed alloys showed higher hardness compared to the cold pressed counterparts for the same compacting pressure.

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Author's contributions

All authors have equal contributations. Authors have no competing financial interests.

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Supporting informations



Fig. I. A schematic of the microstructure changes during LPS [16].

Table 3. EDX analysis at different spot locations for Fe-5Cu-8Pb alloy.

Spot Location	Element Fe	Composition, Cu	wt. % Pb	
1	95.33	2.5	2.17	
2	94.7	5.3	-	
3	84.6	8.18	7.22	
4	75.7	-	24.3	
5	5.63	94.37	-	



Fig. II. Die setup of the hot compaction powder metallurgy compaction technique.



Fig. III. The influence of the compaction pressure and Pb contents on the porosity of cold and hot compacted Fe-5Cu-4Pb compacts.



Fig. IV. The effect of (a) compaction time and (b) compaction temperature on (a, b) the relative density and (b) porosity of Fe-3Cu-5Pb alloy.



Fig. V. Effect of heat treatment on hardness values of (a) cold and (b) hot compaction Fe-5Cu-4Pb alloy.