

Investigation of mechanical properties before and after sintering of cold isostatically pressed metallic powders

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Abstract

In this work, Al and Fe powders are pressed and sintered up to 600 MPa with 100 MPa pressure intervals in a CIP unit made of SAE 1040 steel that can withstand up to 650 MPa pressure. Packing densities were initially 48 % and 33 % and these values have reached to 98 % and 89.2 % under pressures of 600 MPa for Al and Fe powders, respectively. Pressed specimens were sintered at 600, 620 and 640°C for 20 minutes for aluminium and 1200°C for 30, 60 and 90 minutes durations for iron under argon atmosphere in a tube furnace. In order to observe the effects of pressing pressure and sintering temperature and duration on sintering behaviour of samples after and before sintering densification values of samples have been measured. When these pressed specimens were sintered at 600°C/20 minutes and 1200°C/30 minutes, the increase in amount of densification for Al and Fe powders was to 1.3 % and 2.5 %, respectively. Pressing process resulted with significant increase in microhardness of iron and aluminium powders. All products were examined by Scanning Electron Microscope (SEM) in order to determine the morphological change of powders in block samples. Light and scanning electron microscopy examinations revealed that severe plastic deformations took place in the powders of aluminium and iron.

Key words: sintered aluminium and iron, mechanical properties, cold isostatic pressing

1. Introduction

Powder metallurgy (PM) is an attractive, widely used, cost-effective manufacturing technology for the industrial production of small gears to large cam shaft sprocket which require both medium strength and high hardness for wear resistance. One attraction of powder metallurgy is the ability to fabricate high quality, complex parts to close tolerances in an economical manner. Key steps include the shaping or compaction of the powder and the subsequent thermal bonding of the particles by sintering [1–4].

Cold isostatic pressing (CIP) and hot isostatic pressing (HIP) have been used in the industry to make metallic and ceramic products by molding powders. The isostatic pressing of metallic and ceramic powders is one of the important achievements in high technology material processing [5–8]. The principles of CIP and HIP are based on the Pascal's theory and were applied as an industrial technique by Madden [9].

Sintering is usually evident at temperatures in excess of approximately one-half of the absolute melting temperature. Materials melt over a wide range of temperatures; accordingly, sintering is performed over an equally wide range of conditions [10]. Essentially, it is the removal of the pores between the starting particles, combined with their growth and strong mutual bonding.

The densification mechanisms undergone in a powder body are dependent upon a number of powder characteristics. These are the material features, e.g., hardness, work hardening, cold-welding responses, geometrical features, particle shape, particle size and distribution [1, 6].

The purpose of the present study is to investigate the influence of applied pressure and different sintering conditions on mechanical behaviour and properties of aluminum and iron powders, which are processed by the CIP method and commonly used in industry.

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Table 1. Specifications of used powders

Powders	Shape	Manufacturer	Manufacturing method	Hardness (HV)	Apparent density (%)	Size (μm) $d_{m(0.5)}$
aluminium	ligament	Hastek	gas atomized	18	50	107
iron	irregular	Hastek	water atomized	65	35	112

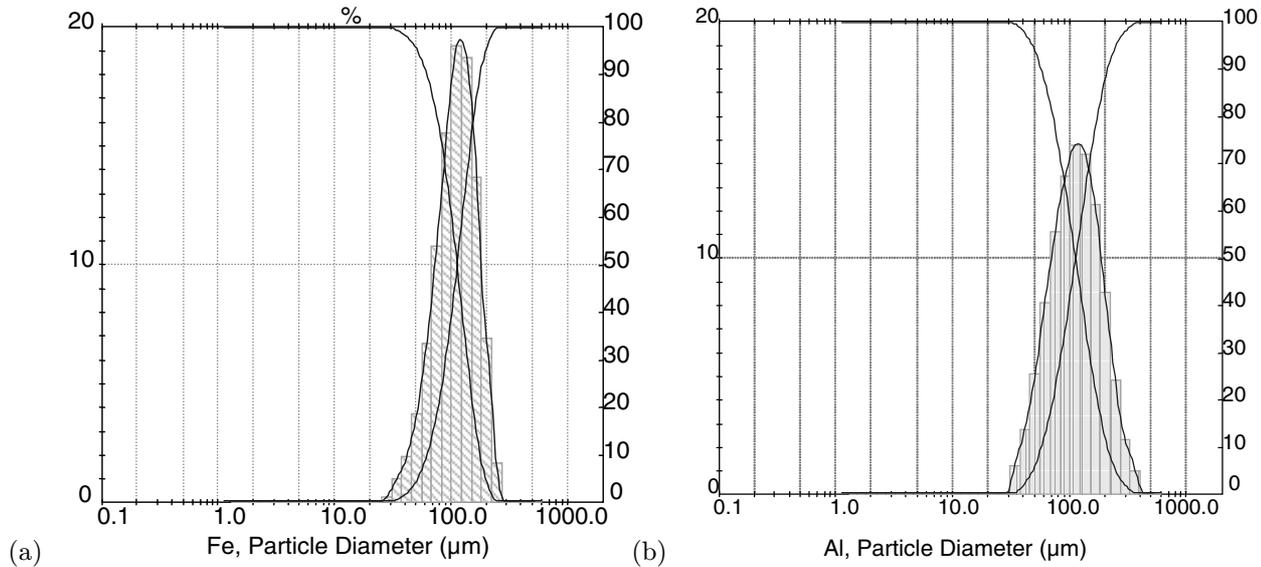


Fig. 1. The size analysis of powders used: (a) Fe, (b) Al.

2. Experimental procedure

2.1. Material

Al and Fe metal powders have been chosen, in this study, due to their wide use in industrial applications. Powders were obtained from Hastek Toz Metal Sanayi from İzmit, Turkey. Typical characteristic properties of powders are given in Table 1. As illustrated in Fig. 1, the particle size of aluminium and iron powder used in this study was measured by Malvern Master-sizer E apparatus at the Gazi University. The medium size of powders is $d_{m(0.5)} = 107 \mu\text{m}$ for aluminium and $112 \mu\text{m}$ for iron.

2.2. CIP unit and method

A wet type CIP unit is used in this study, and this unit consisting of a compression piston and pressure chamber was designed and constructed, because the wet-bag tooling is the most common and versatile one [11]. The piston and the chamber are made of SAE 1040 heat-treated steel with a hardness of 60 HRC. Their cylindrical surfaces were ground to have a roughness of $R_a = 0.3 \mu\text{m}$ (Fig. 2). For this work, Boron oil is used for pressurizing media, since it reduces friction and eliminates corrosion, and is cheap

and easy to obtain. The copper tube was used as the flexible container for the powder mass as shown in Fig. 3. The internal volume of the flexible container was $V_i = 4 \text{ cm}^3$. The air in the flexible container was not evacuated until the desired densification of powders under the pressure was obtained.

An empty flexible container, whose two end caps at both ends were brazed, is immersed into the chamber and pressure was applied to test the deformation behaviour of the empty flexible container. Then, it was observed that the container became totally flat (Fig. 4b) under the applied pressure of 20 MPa that is rather low value compared to the pressures applied for densification of powders. The deformation of the flexible container, which is filled with powders, is not uniform and buckling of the tube took place as seen in Fig. 4. This is attributed to the constraints applied by the brazed end caps to the deformation of the copper cylinder.

2.3. Density and microhardness measurement

Mechanical properties were studied by densification and microhardness measurements under pressure before and after sintering. Powders were filled into a copper tube, which worked as flexible container. The

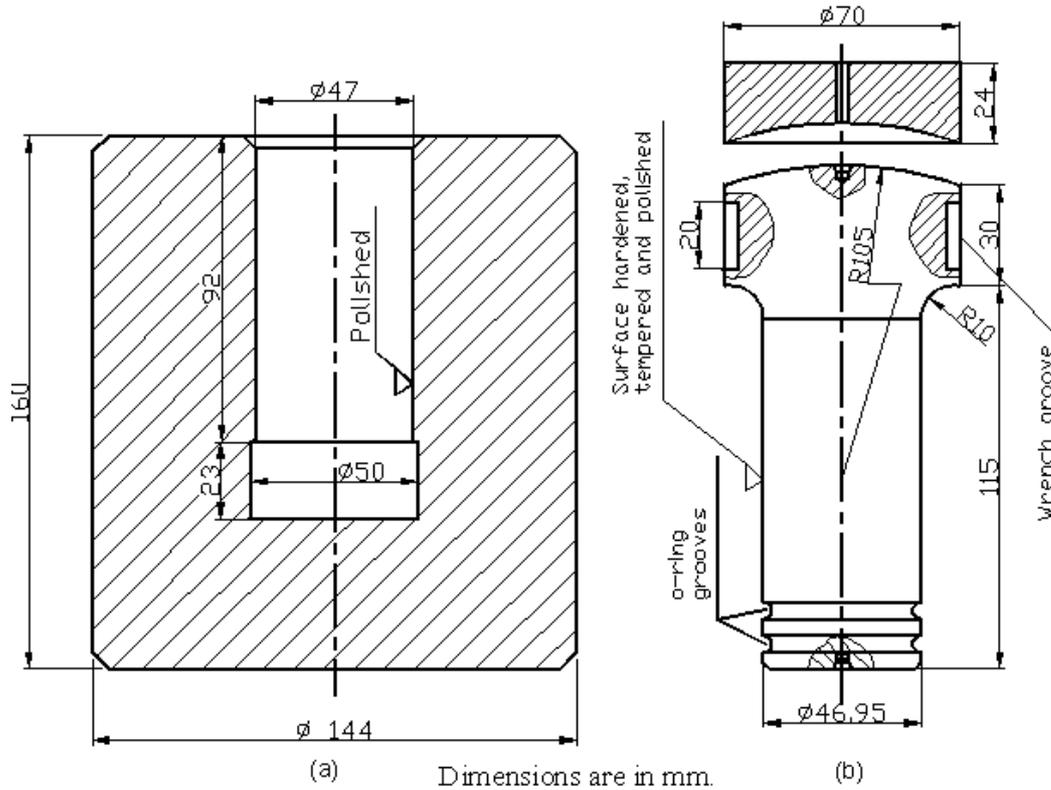


Fig. 2. Wet Bag CIP unit used: (a) pressure body, (b) compression piston.

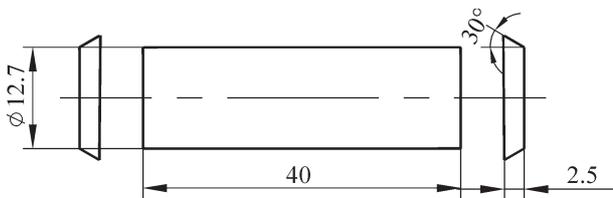


Fig. 3. Flexible container.

ends of the copper tube were sealed by brazing caps to them. Copper tube was 12.7 mm in diameter and 40 mm in length with a wall thickness of 0.7 mm. Before and after filling the powders into the flexible container, it was weighted to an accuracy of ± 0.001 g. The change of volume of the flexible container due to the CIPing was measured by Archimedes's principle. Thus, % theoretical densities before and after CIPing were calculated. Sintered specimens were measured by the same method as well. The procedure of sealing



Fig. 4. The deformation of the flexible container under applied pressures: (a) before pressing, (b) empty pressing (20 MPa), (c) 100 MPa, (d) 200 MPa, (e) 300 MPa, (f) 400 MPa, (g) 500 MPa, (h) 600 MPa.

and measurement of volumes of flexible mould was explained in previous works in details [12].

The microhardness of the powder particles was determined before and after sintering using a Reichert Hardness Tester. To determine the microhardness of the powder particles and specimens before and after sintering, the powders and specimens were mounted in bakelite. The indentation was made using 10 g for aluminium and 20 g for iron, the average length of the diagonals of the indentation was measured, and the microhardness was determined. The recorded values represent an average of 10 readings for each powder sample.

2.4. Sintering

Sintering of all materials was made in an automatically controlled high temperature gas atmosphere tube furnace in Gazi University P/M laboratory. In sintering process, Al compacts were sintered at 600°C, 620°C, and 640°C temperatures for 20 minutes. Temperature was increased by 5°C per minute up to sintering temperature, and then parts were kept at this temperature for 20 minutes. Following that, the temperature controller of furnace was set to 300°C, and temperature was allowed to decrease by 8°C per minute down to 300°C, and then the temperature was decreased by 6°C per minute up to room temperature.

Fe parts were sintered at 1200°C for 30, 60 and 90 minutes time intervals. These conditions created very different densities and metallurgical structures due to time and temperature variations. Temperature was increased by 5°C per minute up to sintering temperature, and then the compacts are kept at that temperature for 30, 60 and 90 minutes. Following this time, the temperature controller of furnace was set to 600°C, and temperature was allowed to decrease by 8°C per minute down to 600°C. Then the furnace was turned off, and temperature was decreased by 6°C per minute approximately down to room temperature. Argon gas was always running in the furnace until reaching room temperature for both Al and Fe testing procedure.

3. Experimental results and discussion

3.1. Densification behaviour of Al specimens

The cold isostatic pressing of powders is carried out for almost the same powder particle sizes (Table 1 and Fig. 1). Different pressure and sintering conditions were used in order to obtain different metallurgical structures in materials. Initially, after compaction process, green density of samples was measured using Archimedes principle method. It was observed that the flexible container became totally flat (Fig. 4b)

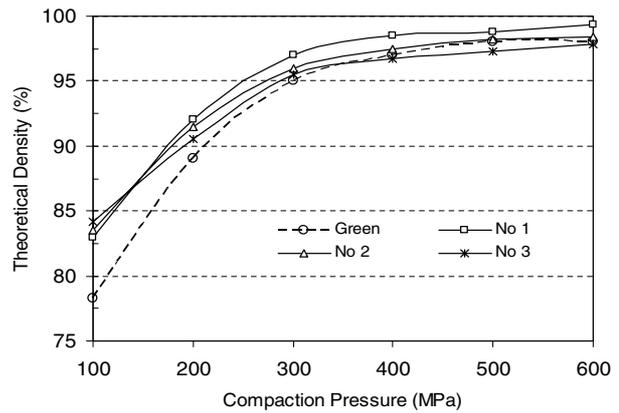


Fig. 5. Comparison of % densities of green and sintered Al compacts.

under the applied pressure of 20 MPa, that is rather low value compared to the pressure applied for densification of powders.

Increasing pressure caused higher density in non-sintered parts due to particle deformation and reducing pores. When pores reduce, particles are yielded and volume is decreased, thus density is increased.

A significant cold-welding was observed between the particles in non-sintered specimens pressed with 100 MPa or above for Al powders due to plastic deformation.

Calculated densities with Archimedes principle method for 600, 620, 640°C and 20 minutes sintering for Al parts are shown in Fig. 5. As can be seen in Table 2 and Fig. 5, after sintering, the highest densities were obtained in cycle No. 1. Pore structures and shrinkages among the boundaries and density are more affected at the temperature of No.1 case when compared to No. 2 and No. 3 case. Comparison of green density and sintered density of 600°C/20 min is shown in Fig. 5, which shows increasing density after sintering operation. Density increased by only 1.5 % after sintering operation (No. 1). During the sintering, lower temperature makes more contribution to densification. Higher temperature caused melting of aluminium and there can be very small evaporation of aluminium.

3.2. Densification behaviour of Fe specimens

The maximum density $\rho_{\max} = 89\%$ is obtained with 600 MPa pressure. Beyond 400 MPa pressure, the density is increased but with decreasing rate. Increasing pressure also caused particle deformation. Interlocking among the powders and plastic particle deformation is initiated at about 300 MPa pressure.

Density distributions of green and 1200°C temperature and 30, 60, 90 minutes sintered Fe compacts are

Table 2. Density values of Al under different pressures and sintering conditions

Pressure (MPa)	Green densities (%)	Sintering conditions and sintered densities (%)		
		Ar, 5 °C/min-600 °C, 20 min (No. 1)	Ar, 5 °C/min-620 °C, 20 min (No. 2)	Ar, 5 °C/min-640 °C, 20 min (No. 3)
100	78.3	83	83.5	84.2
200	89	92	91.5	90.5
300	95	97	96	95.5
400	97.5	98.5	97.5	96.7
500	98	98.8	98.2	97.3
600	98	99.3	98.4	97.8

Table 3. Density values of Fe under different pressures and sintering conditions

Pressure (MPa)	Green densities (%)	Sintering conditions and sintered densities (%)		
		Ar, 5 °C/min-1200 °C, 30 min (No. 1)	Ar, 5 °C/min-1200 °C, 60 min (No. 2)	Ar, 5 °C/min-1200 °C, 90 min (No. 3)
100	58	59	58	60
200	64	70.7	67	71
300	70.5	78	76	76.7
400	80	84	80.7	81
500	83.5	89	86.5	85
600	89	91.5	88.5	89

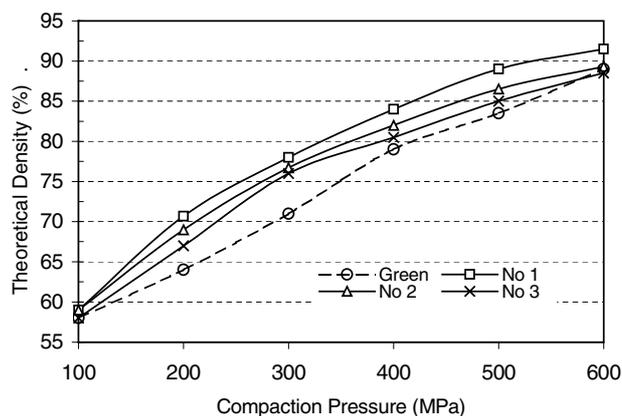


Fig. 6. Comparison of % densities of green and sintered Fe compacts.

shown in Fig. 6. Densities increased with sintering operation due to decreasing pores between the particles. The maximum density $\rho_{\max} = 91.5\%$ is obtained with 1200 °C and 30 minutes sintering condition according to green density. Density increased by only 2.5 % after sintering operation with ferrite. It is observed that sintering affects the density in a very small amount. As can be seen in Table 3 and Fig. 6, after sintering, the highest densities have been obtained in cycle No. 1. As illustrated in Fig. 6, the green and sintered densities showed similar pattern.

3.3. Morphology and microstructure of Al and Fe

Figure 7 shows the photographs of SEM of the Al (a) and Fe (b) before compaction. The aluminium powder particles before pressing have the ligamental shape as shown in Fig. 7a.

Figure 8 shows the photographs of SEM of the aluminium before and after sintering with 600 MPa pressure, respectively. A complete plastic deformation is observed under the pressure, which can be seen clearly in the Fig. 8. Pores amount decreases to its minimum value with the increase of applied pressure. As can be seen in Fig. 5, the densification in green specimen has reached 97.5 % of theoretical density at a pressure of 400 MPa, and remains nearly constant up to 600 MPa. The remaining 2% may be attributed to the voids between the particles before sintering. Almost complete densification was obtained after sintering of aluminium powders at 600 °C for 20 min (Fig. 8b).

Figure 9 shows the photographs of SEM of the iron powder before and after sintering. Iron has an irregular shape as seen in Fig. 7b. For ferrous powders of medium hardness and plastic behaviour, density of 89 % is achieved. Porosity amount is very high for Fe specimens. A complete plastic deformation can be seen from Fig. 9. A high density means high strength of compacted parts, and this is a welcome feature in the powder metallurgy. For iron powders pressed at

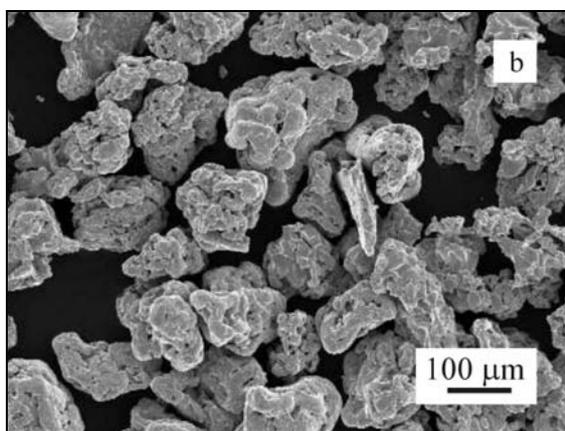
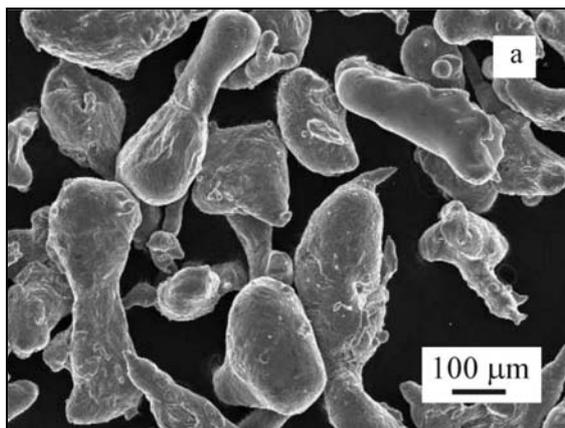


Fig. 7. The photographs of SEM before compaction: (a) Fe, (b) Al.

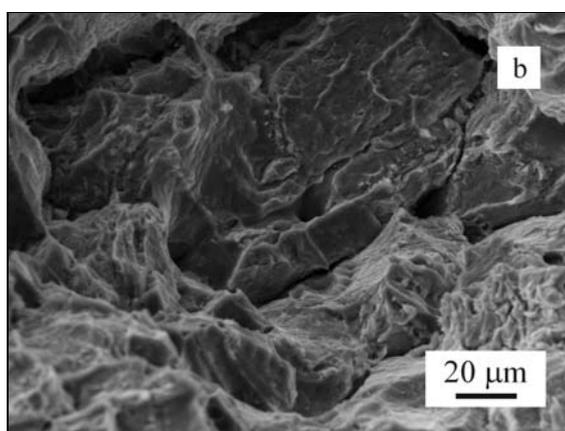
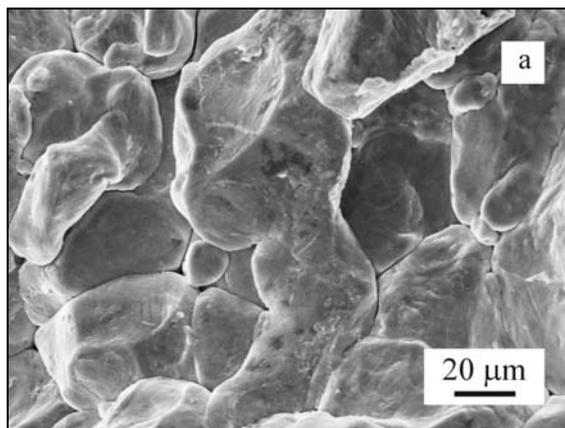


Fig. 8. SEM micrographs of the Al specimens: (a) before sintering, (b) after sintering.

400 MPa and above, plastic deformation was initiated at contact surfaces by yielding and this caused local deformation (see Fig. 9a). Thus, the local deformation, under excessive pressure, of the material into the neighbouring voids increased the density. The examination with light microscope shows that the porosity has decreased with increasing pressure, the initial shape of particles has changed completely and homogenous densification has taken place as seen in Fig. 10.

3.4. Microhardness behaviour of Al and Fe

In the case of low hardness and plastic behaviour of aluminium powders, which are softer than iron powders, maximum density is 98 %. For iron powders, which are harder than aluminium, density is 89 % when the pressure reached up to 600 MPa before sintering. As illustrated in Fig. 11, microhardness values of aluminium and iron powders are 67 and 107 HV under applied pressure of 600 MPa, respectively. The microhardness of Al increased from 18 HV to 67 with increasing pressure up to 600 MPa, even though the densifica-

tion almost stopped at 400 MPa (see Fig. 5). The increase in microhardness, which is the result of work-hardening taking place due to severe plastic deformation during CIP, for Al is almost three-fold.

Microhardness values of green iron powders under pressure also increased. They continue to increase although the densification ratios for both powders remain almost unchanged. Saritas [12] stated that this is due to the fact that plastic deformation occurs with increasing pressure. With the occurrence of plastic deformation strain hardening is developed. The strain hardening corresponds to the motion of large number of dislocations. This large number of dislocations results in the hardening of powder.

But, microhardness values of the both powders decreased after sintering, because the stress occurred on the powders by increasing pressure has decreased with temperature during sintering, which caused a relaxation in the powder body.

4. Conclusions

1. For iron powders with a medium hardness and

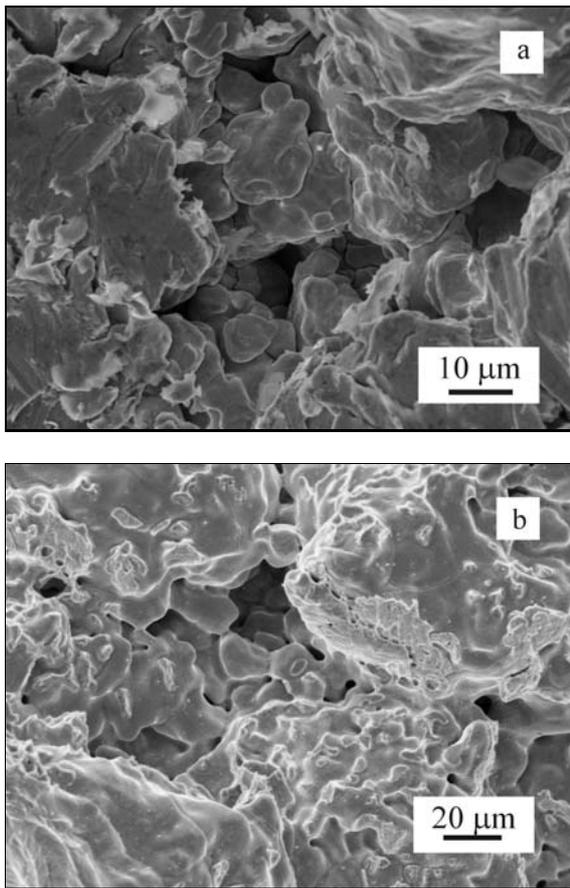


Fig. 9. SEM micrographs of the Fe specimens: (a) before sintering, (b) after sintering.

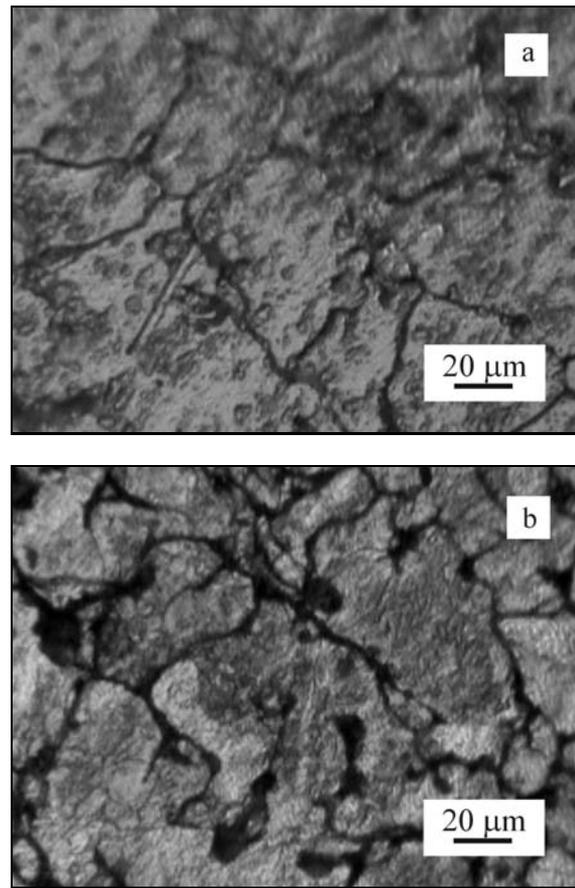


Fig. 10. Light microscope photographs of (a) aluminium and (b) iron compact.

plastic behaviour, green measured density was 89 % of theoretical density of iron at 600 MPa pressure.

2. Almost complete densification (97.55 % theoretical density) was obtained with CIP method for soft and easily deformed aluminium powders at 400 MPa pressure.

3. Plastic deformation was initiated above 100 MPa and 300 MPa pressure due to hardness of powders for Al and Fe, respectively.

4. Maximum density of Al compacts after sintering operation is found as 99.3 % of theoretical density at 600 MPa pressure and 600 °C/20 minutes sintering condition.

5. Maximum density of Fe compacts after sintering operation is found as 91.5 of theoretical density of Fe at 600 MPa pressure and 1200 °C/30 minutes sintering condition.

6. Before sintering process, although, the ratios of theoretical densities of both powders Al and Fe have not changed much even after 300 and 400 MPa pressure, respectively, increasing pressure resulted in a rise in the microhardness of the aluminium and iron powders.

7. It is found that sintering process decreased mi-

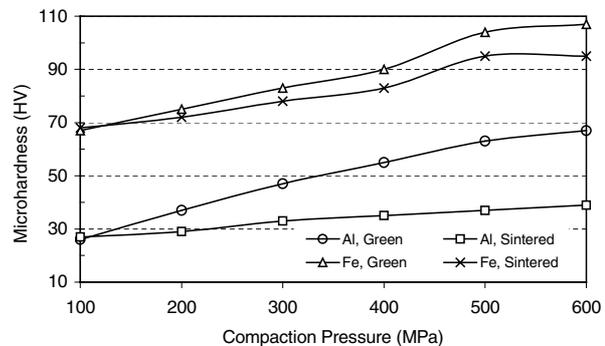


Fig. 11. Change of microhardness of powders before and after sintering under pressures.

crohardness for both powders when compared to unsintered case.

8. It is observed that sintering conditions have important role in the densification mechanism.

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