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Effects of Powder Hardness and Particle Size on the Densification of Cold Isostatically Pressed Powders

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Abstract

The densification behaviour of aluminium, iron and alumina powders under cold isostatic pressing (CIP) are examined. For this purpose, a unit which can withstand up to 650 MPa pressure was designed and constructed. With this pressure unit, aluminium, iron and ceramic powders, having different hardness values and different particle sizes, were pressed and their mechanical behaviours were investigated at pressures between 100 and 500 MPa. Attained densities were about 100% for aluminium, 90% for iron and 65% for alumina powders. Pressing resulted in significant increases in the microhardnesses of iron and aluminium powders. Light and scanning electron microscopy examinations revealed that severe plastic deformations took place in the powders of aluminium and iron, and no plastic deformation was observed in alumina powders.

Key words: Cold Isostatic Pressing, Metallic and Ceramic Powders, Mechanical Behaviour.

Introduction

Isostatic pressing of metallic and ceramic powders is one of the important achievements in high technology material processing. In isostatic compaction, a uniform pressure is applied simultaneously to all the external surfaces of a powder body. For this purpose the powder is sealed in a flexible container and the assembly is immersed in a fluid which is pressurized (German, 1984; Lenel, 1980). If the isostatic pressing is done at room temperature, then it is called cold isostatic pressing (CIP). There are two types of CIP toolings available: wet-bag tooling and dry-bag tooling. Wet-bag tooling is more common and versatile (Wheeler, 1986). Compared with die-compaction, CIP provides more uniform pressure distribution within the compact. The major reasons for this are the absence of die-wall friction and the greater area over which pressure is applied. Furthermore, distinct advantages may often be gained from the evacuation of air from the loose powder before compaction. Consequently, CIP provides increased and more uniform density at a given compaction pressure, and relatively defect-free compacts when applied to brittle or fine powders. Another characteristic of the process is the elimination of lubricant additives. Their absence eliminates problems associated with lubricant removal prior to or during final sintering (Koerner, 1978; Jackson, 1967; Meiners and McCall, 1987; James, 1983; Koizumu and Nishiara, 1992; Thompson, 1981).

Densification of a powder body is dependent upon a number of powder characteristics. The first group of these are the material features, e.g., hardness, work-hardening and cold-welding responses. The second group are the geometrical features, e.g.,

particle shape, particle size and distribution, and the effect of lubricant additives that govern their interparticle movement and interlocking during pressing (James, 1977). Densification of powders has generally been accepted to take place in three stages (Gethin *et al.*, 1994; Sheppard and McShane, 1980; Fischmeister and Arzt, 1983). In stage one, densification is produced by rearrangement, where both the packing density and coordination number increase with small levels of pressurisation (less than 0.05 MPa) (Jones, 1960). In stage two, at intermediate pressures, more defined elastic-plastic deformation occurs at interparticle contact areas. As illustrated in Figure 1, coordination number (number of contacts) and contact area increase with the increase in pressure while porosity decreases (Fischmeister *et al.*, 1978; German, 1989). With further pressurisation, the number of contacts inreases as particle rearrangement and sliding occur. Cold welding and/or mechanical interlocking of the particles contribute to the green strength of the compact. For brittle particles, the onset of plastic deformation can lead to fracture, giving way to fragmentation of the original particles and causing densification by repacking of the fragments. At very high compaction pressures, massive deformation occurs, leaving small pores in the junctions of three or more particles. This third stage typically starts at densities over 95% and compaction pressures in excess of 1 GPa (German, 1989).

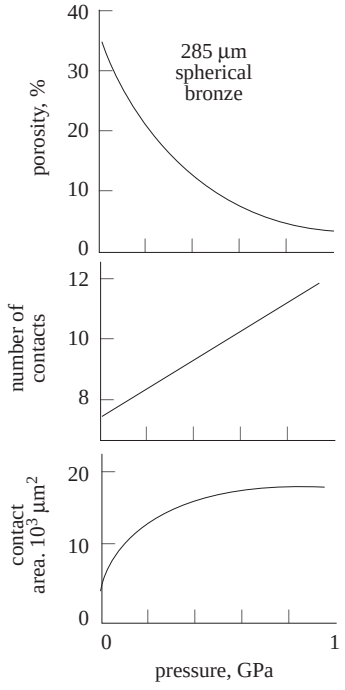


Figure 1. The compaction behaviour of spherical bronze particles.

Kawakita analysed CIP and proposed the following equation for particulate matter. It relates the relative reduction in volume of the powder mass, C, to pressure, P (James, 1983):

$$C = \frac{V_0 - V_p}{V_0} = 1 - \frac{D_a}{D_p} = \frac{abP}{1 + bP} \qquad (1)$$

where V_o is the initial volume of the powder mass, V_p is the volume of the powder mass under pressure P, $D_a$is the relative apparent density of the loose powder mass, D_p is the relative density of powder mass under pressure P and a and b are constant. Rearranging equation (1) gives

$$\frac{P}{C} = \frac{1}{ab} + \frac{P}{a} \qquad (2)$$

Figure 2 illustrates the application of Kawakita's equation of state for a series of monosize fractions of metal powders pressed over a pressure range of 77-770 MPa. The well defined linearity is typical of Kawakita's equation the slope is 1/a, while the intercept at zero pressure on the P/C axis is given by 1/ab. The generally accepted interpretation of the constant "a" is taken to be a measure of the initial (loose) porosity, 1-D_a, of the powder.

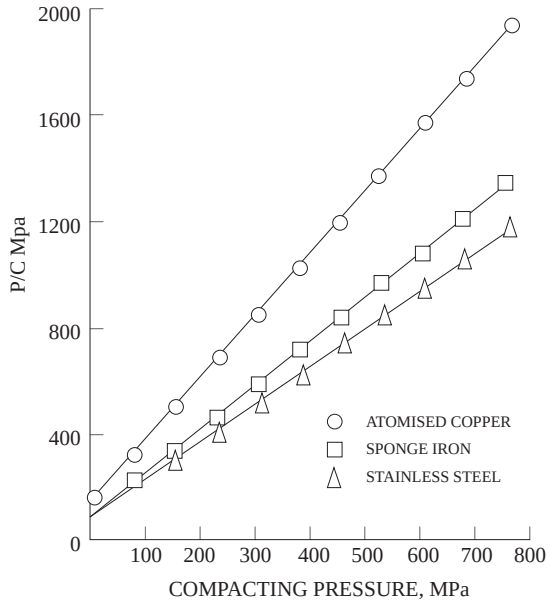


Figure 2. Kawakita relationships for monosize fractions of metal powders.

Materials and Methods

CIP Unit: A CIP unit (wet type) consisting of a compression piston and a pressure chamber was designed and constructed (Figure 3). Pressures of up to 650 MPa were obtained in the chamber by the application of force through a 2000 kN capacity testing machine (ELE). The piston and the chamber are made of SAE 1040 steel and heat treated to a hardness of 60 HRC. Their cylindrical surfaces were ground to have a roughness of $R_a = 0.3$ μm. Water and mineral oils were used as the pressurizing media. A copper tube (Figure 4) was used as the flexible container for the powder mass. The internal volume of the flexible container was $V_i = 4.0$ cm^3. The air in the flexible container was evacuated by vacuum pump up to 10^{-2} torrs.

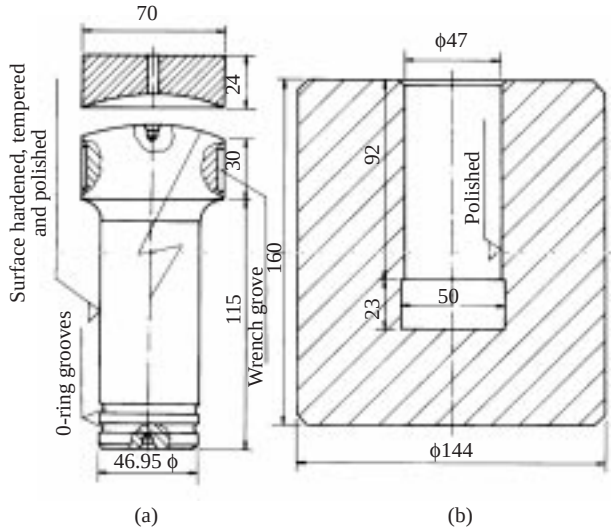


Figure 3. (a) Compression piston, (b) Pressure body.

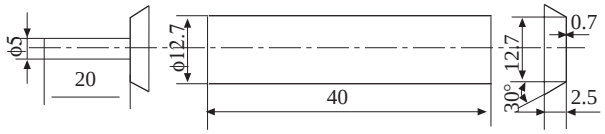


Figure 4. Flexible container.

CIP: The specifications of the powders used in this study are given in Table 1. Each powder was sieved to different size fractions. Aluminium (produced at Gazi University), iron (Höganas ASC100.29) and alumina (Aldrich) powders were used. One of the end caps of the flexible container was first brazed and then it was filled with the powder sample was filled in. Later, the other end cap was brazed. The total volume of the flexible container before (V_0) and after (V) pressing was measured using a graduated measuring cylinder according to the Archimedes principle. The flexible container was immersed into the pressure chamber, the piston was placed into the hole and then the pressures of 100 to 500 MPa was applied using a 2000 KN capacity ELE testing machine. Volume change ($\Delta V = V_0 - V$) in the flexible container is due to densification of the powders since the volume of the copper tube is constant. The volume of the pressed powder, V_p is given as

$$V_p = V_i - \Delta V \tag{3}$$

The mass density D_p of the pressed powder may be calculated as

$$D_p = \frac{W}{V_p} \tag{4}$$

where W is the weight of the powder inside the flexible container. The percent theoretical density D_t of the pressed powder is then determined by the relation

Table 1. Characteristics of powders used.

Powder,	(No)	Manufacturing Method	Apparent Densit, %	Hardness HV	Size Range μm
Aluminium,	(1)	Atomised	60	18	-45
	(2)	(gas)	50		-150 + 106
Iron,	(3)	Reduced	40	65	-63 + 45
	(4)		35		-150 + 106
Alumina,	(5)	Fused	47	2000	-45 + 10
	(6)		30		-106 + 75

$$D_t(\%) = \frac{D_p}{D} x100 \qquad (5)$$

where D is the picnometric density of the powder material.

Metallography: Deformed flexible containers were cut perpendicular to their axis and then mounted in plastic for metallographic preparation. The copper tube of some of the deformed flexible containers was carefully cut partly and then the sample was broken to cause fractured surfaces of compressed powders. The morphology and microstructure of the pressed samples were examined by scanning electron microscope (SEM) and light microscope. It was not possible to prepare samples of alumina for examination since they were loose powders. The same difficulty was encountered for iron samples pressed below 200 MPa and aluminium samples pressed below 100 MPa. In addition, the change in microhardness was measured for aluminium and iron samples embedded in plastic.

Experimental Results and Discussion

In this study, aluminum, iron and alumina powders of hardnesses and different sizes were pressed to 100-500 MPa using a cold isostatic pressing unit and their densification behaviour was investigated. An empty flexible container where the two end caps at both ends were brazed was immersed into the chamber and pressure was applied as a test of the deformation behaviour of an empty flexible container. It was observed that the container become totally flat (Figure 5(b)) under an applied pressure of 20 MPa, which is rather low compared to the pressures applied for the densification of powders. The deformation of the flexible container which was filled with powders is not uniform and buckling of the tube took place as seen in Figure 5. This is attributed to the constraints applied by the brazed end caps to the deformation of the copper cylinder.

CIP of aluminium powders was done for two different powder particle sizes (Table 1). The aluminium powder particles before pressing ligamental in shape as shown in Figure 6 (a). The packing density of powder No.1 is 60% of the theoretical density (TD), and of powder No.2 is 50% of the TD. As can be seen in Fiure 7, the densification in both powders reached 98% of the TD at a pressure of 400 MPa, and stayed constant up to 500 MPa. The remaining 2% may be attributed to voids between the particles. The SEM examination of the specimen pressed at 100 MPa shows the initiation of plastic deformation (Figure 6(b)). At this stage, the point contacts were partly replaced with plane contacts. In specimens pressed to 300 and 400 MPa, initial particle shapes totally disappeared and plane contacts were formed as seen in Figure 6(b) and 6(c). In cold isostatically pressed monosized metal powders a spherical particle turns into a dodecahedron shape when fully densified. The light microscope examination showed uniform densification as seen in Figure 8. The microhardness of aluminium increased from 18 HV to 63 HV with increasing pressure up to 500 MPa, even though the densification stopped at 400 MPa (Figure 9). This three-fold increase in microhardness is the result of work hardening taking place due to severe plastic deformation during CIP.

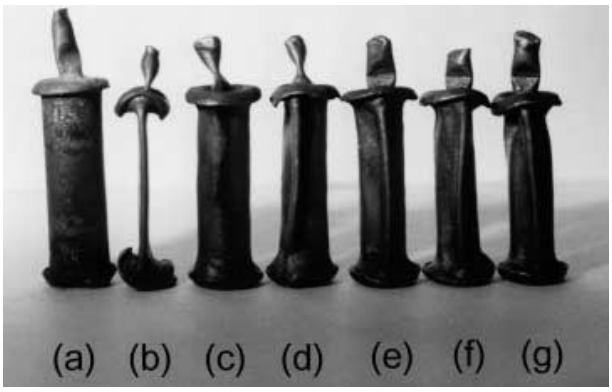


Figure 5. The deformation of the flexible container under applied pressures.

The densification of iron powders of two different particle sizes was investigated in two different CIP procedures: the first group without removing the air between the powder particles (as in the case of aluminium) and the second group with air removed up to a vacuum of 10^{-2} torrs. Original iron powder is seen to have an irregular and porous structure because of water atomisation as shown in Figure 10 (a). The densification behaviour of iron powders ders with two different particle size is similar, as seen in Figure 11. At 100 MPa pressure and below no compact was produced and the powders were loose. Pressing at 100 MPa pressure produced a density increase of 17% and powders were loose, which means that the main densification stage was repacking with very little plastic deformation. The tensile strength of iron powders may be considered to be about 21.6 kg/mm^2 (216 MPa) since the tensile

strength is about one third of the hardness. Additionally, some of the applied pressure goes to interparticle friction since no lubricant addition was done (German, 1984; Sarıtaş, 1995). Thus up to applied pressure in the order of 250 MPa most of the densification is due to the repacking of particles. In iron powders pressed at 400 and 500 MPa, plastic deformation was initiated at contact surfaces by yielding and this caused local deformation. Thus the local deformation, under excessive pressure, of the material into the neighbouring voids increased the density. The light microscope examination (Figure 12) shows that porosity decreased with increasing pressure, the initial shape of particles changed completely and homogenous densification took place.

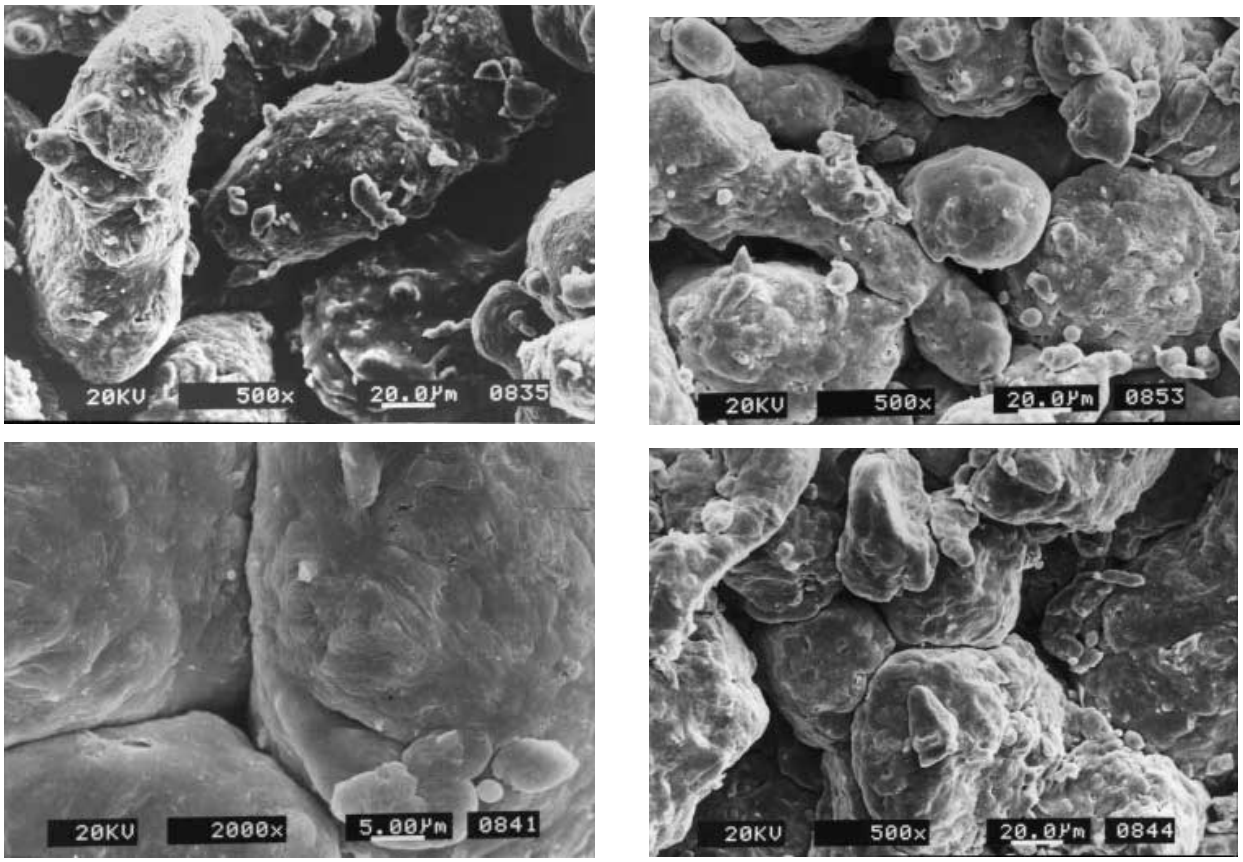


Figure 6. SEM micrographs of aluminium powders: (a) Before compaction, (b) 100 MPa, (c) 400 MPa, (d) 500 MPa.

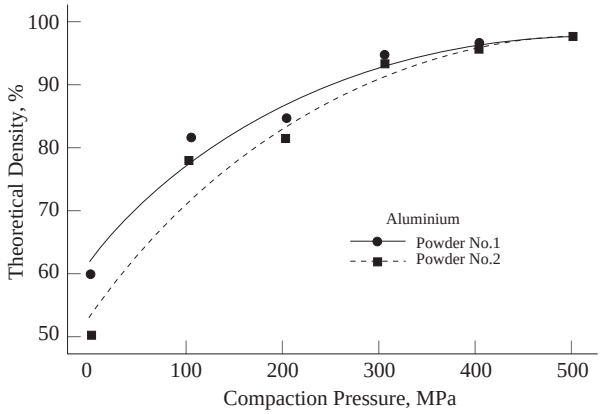


Figure 7. Densification in aluminium powders.

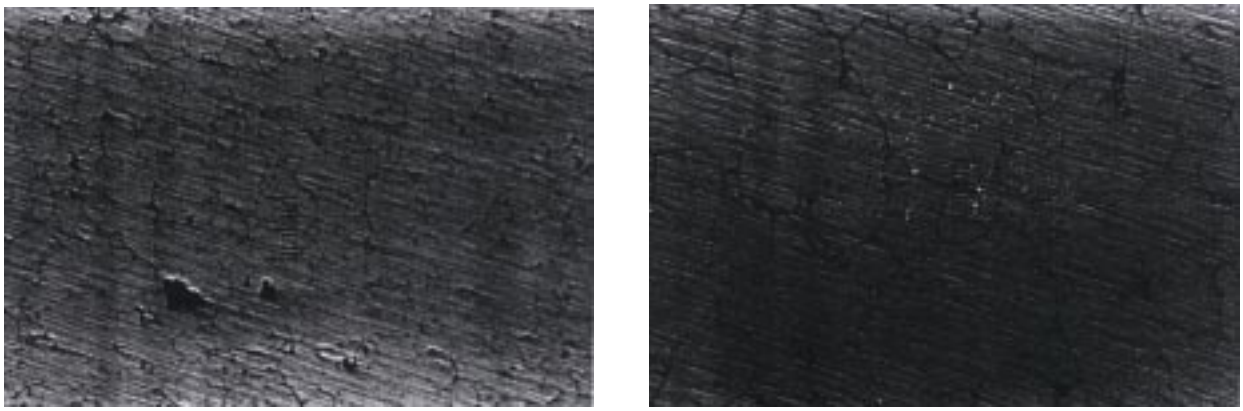


Figure 8. Light microscope photographs of aluminium compact: (a) 300 MPa (100X), (b) 400 MPa (500X).

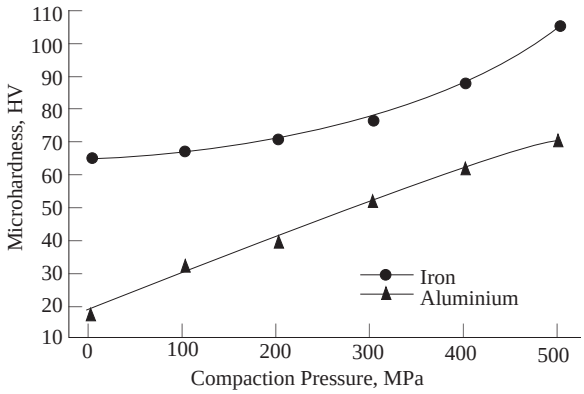
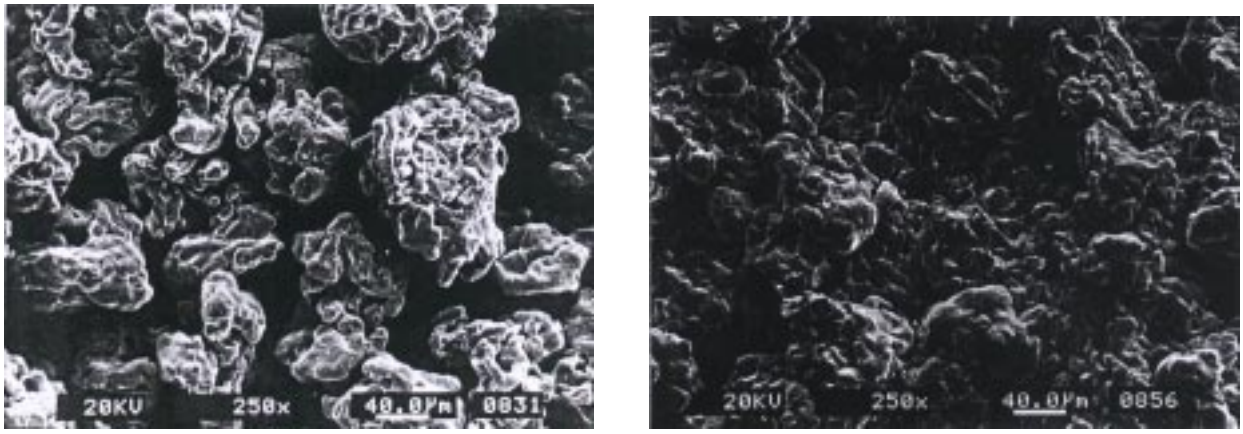


Figure 9. Change of microhardness of powders due to the CIP.



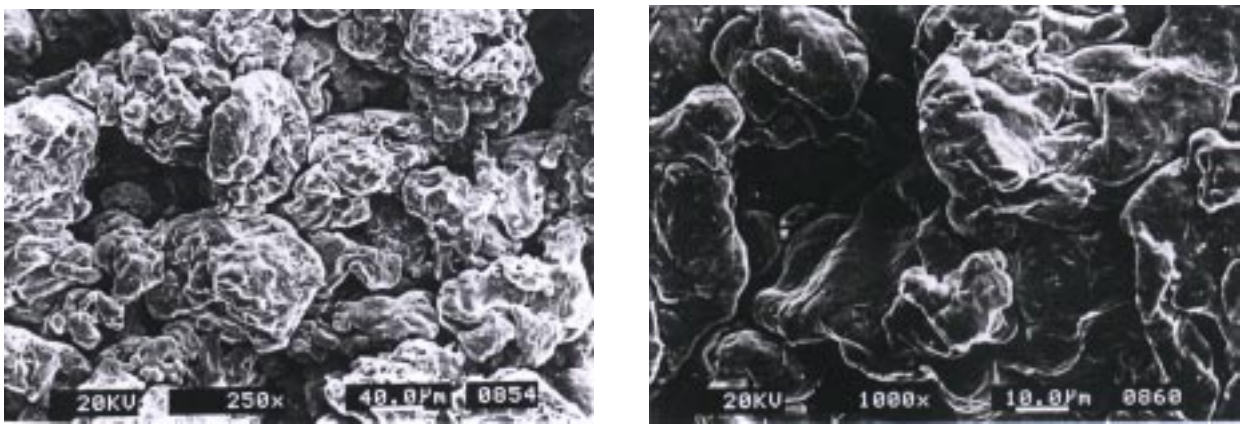


Figure 10. SEM micrographs of iron powders: (a) Before compaction, (b) 200 MPa, (c)300 MPa, (d) 500 MPa.

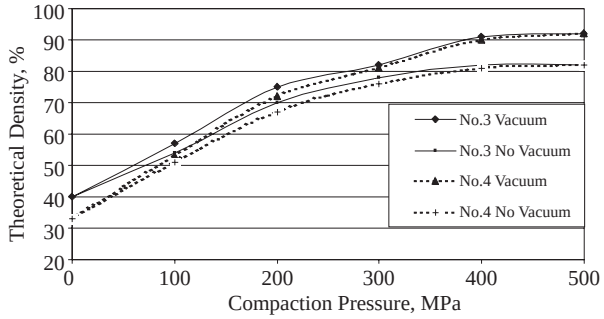


Figure 11. Densification in iron powders for No. 3 and No. 4.

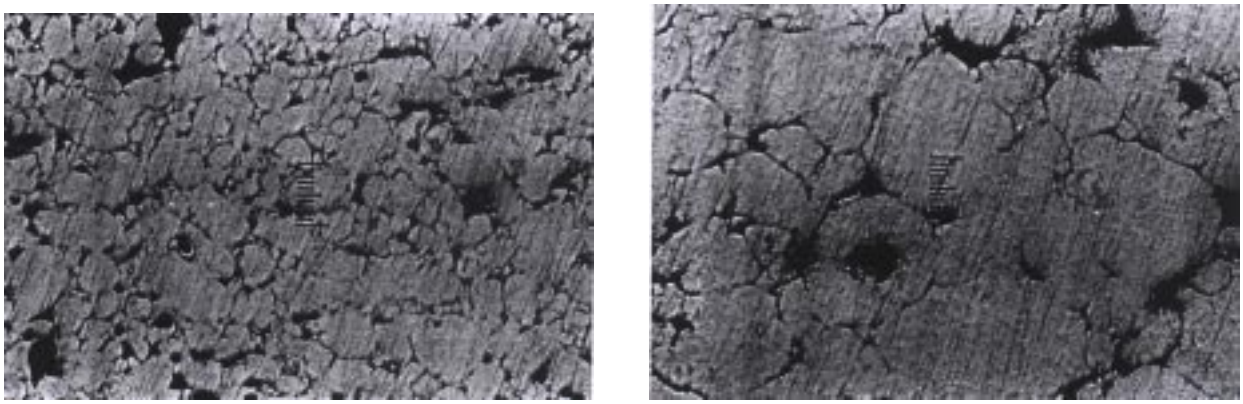


Figure 12. Light microscope photographs of iron compact: (a) 300 MPa (100X), (b) 400 MPa (500X).

The density increase of severely hard alumina powders reached only 20% for 500 MPa applied pressure (Figure 13). The increase in densification under the applied pressures is attributed to the repacking of powders since the applied pressure was below the yield strength of alumina particles.

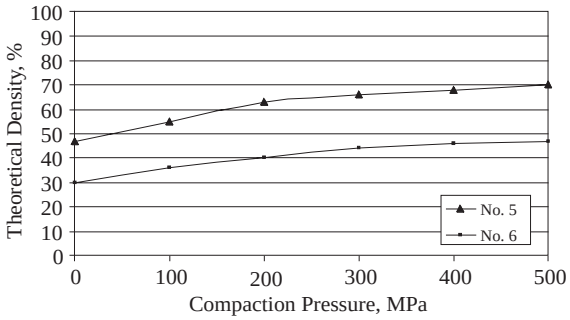


Figure 13. Densification in alumina powders.

Conclusion

1. Full densification was almost attained with CIP in soft and easy to deform aluminium powders.
2. For iron powders of medium hardness and plastic behaviour, density of 82% of the TD without removing air and 92% of the TD with removing air to a vacuum of 10^{-2} torrs were achieved at 500 MPa.
3. The microhardness of aluminium powders increased three-fold while the microhardness of iron powders increased about two-fold during CIP at pressures up to 500 MPa.
4. Severely hard alumina powders attained 70% of the TD under 500 MPa pressure. No plastic deformation was observed in alumina powders. Densification is due to the rearrangement of powders.

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