

A STUDY OF COMPACTION OF METAL POWDERS

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By

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DECLARATION

No portion of the work referred to in this dissertation has been submitted in support of an application for another degree or qualification of this or any other university or other institution of learning.

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ABSTRACT

The aims of this investigation on metal powders are to determine powder behaviours under an external applied pressure and to assess some mathematical expressions related to the compaction of powders. Two different types of irregular shaped atomized iron powders, spherical bronze powders and copper powders that were commercially available were pressed uniaxially in a floating action die at pressures up to 950 MPa. Powder compaction stages that occurred during consolidation were identified and evaluated. Work hardening effects on the green compact were found to be effective from the beginning of stage I.

CHAPTER 1 : INTRODUCTION

Compaction is one of the main stages in the fabrication processes of manufacturing engineering components from particulate materials. Compaction can be defined as a consolidation process of loose powder into desired shapes under external pressures. In terms of industrial powder metallurgy (P/M) production systems in industry , uniaxial compaction is one of the commonly used consolidation process. It has received a lot of attention by many researchers. That is because it is suitable for continuous production techniques, involving computer numerical controlled systems and it is relatively easy to produce high volumes of metal and ceramic components, explosive materials and pharmaceuticals. Therefore, most P/M parts are produced by using this process. It is the object of this study to determine the behaviour of powders of different shapes and sizes when consolidated by uniaxial compaction.

1.1 : Consolidation of Powders

During the last decades there have been great efforts to understand the mechanisms of the compaction process in both metals (Kamm, 1947; Bockstiegel, 1965; Vonazountas,

1992) and non-metals (Cooper, 1962; Oudemans, 1965; VaBen, 1992; Al-Tounsi, 1993; Ohji, 1994). Powder behaviour during compaction is of great importance in order to understand the mechanism of the densification phenomenon. Initial attempts to achieve this were made by Seelig - Wulff (1946) and Seelig (1947), who identified that three regions of compaction may exist.

I- The initial mechanism involves rearrangement and packing of the particles on application of the initial compaction pressure. In this stage, particle size, size distribution, particle shape and surface properties play an important role, as well as interparticle friction.

II- In the second stage, elastic and plastic deformation are dominant. This stage also includes inter-particle bonding, mechanical interlocking of the particles and particle-particle interactions.

III- In the final stage of compaction, increasing pressure causes fragmentation of powder particles; cold welding may occur at this stage.

However, subsequent studies have substituted this final stage with more stages. The different stages cannot be clearly separated one from another. Also, in the compacted body different stages of compaction occur in different parts of the body during the consolidation process, due to differences in density and stress

distribution throughout the compact body in uniaxial compaction.

Wang (1976) divided the compaction process into four stages. Besides the three stages of Seelig and Wulff, Wang added a final stage i.e., ejection of the compact body from the die. In Wang's model only stage two i.e., elastic-plastic deformation of particles, gives a strictly straight line relationship between compaction pressure and green density that can be expressed as a mathematical relationship.

Later, Fischmeister, Arzt and Olsson (1978) re-examined the first and second stages of the compaction process. They used spherical powders of ductile metals and found that deformation and sliding occur from the beginning of consolidation.

1.2 : Consolidation Techniques

Since one of the aim of powder compaction is to increase the green density and strength of the compact, various consolidation process are in use. In general, consolidation of powders can be divided into two main categories, mechanical and non-mechanical consolidation. Some examples of the latter are slip casting, injection moulding, tape casting and pressureless sintering. Mechanical consolidation can be subdivided into groups

according to;

- The rate of applied load,
 - static compaction
 - dynamic compaction
- Introducing heat or not,
 - rolling
 - extrusion
 - forging
- Uniaxial compaction,
 - single action
 - double action
 - Isostatical compaction,
 - hot isostatic pressing
 - cold isostatic pressing

Since component production rate is an important factor in P/M routes, uniaxial compaction has achieved a state of major importance. Thus, it is with this basic process of compaction that this study is involved.

Uniaxial compaction is one of the most commonly used P/M consolidation processes. Uniaxial compaction is a process by which powders are consolidated inside a die cavity into a designed shape by using applied pressure

acting uniaxially. This process achieves an adequate green strength of the compacted samples before this part is transferred to the sintering stage. Die compaction requires a die which is used to transmit external pressure into the powders and the use of a compaction machine with a capacity that depends on the powder material and its specifications such as, particle size, shape, size distribution etc,. The compacting machine should be at least capable of having compaction forces adequate for the minimum compaction stress for a given die specification in order to achieve sufficient green strength.

Uniaxial compaction can be classified into two main groups, single action and double action pressing. Single action compaction is used for relatively thin parts. The basic phenomenon is movement of the single punch against the other punch which remains stationary. Thicker or complex parts are compacted with a double action compaction mechanism in which both the upper punch and bottom punches move against the powder mass to compress it.

An account of the shaping of powders by using different types of consolidation processes, their advantages and disadvantages one over another and final compact properties is given by Meeks (1984), and by Randall

(1994).

In terms of the compaction rate, powder compacts can be assessed to be compacted under either static or dynamic conditions. A compaction rate below 10 s^{-1} is considered to be static, above it, up to 10^2 s^{-1} , it is called dynamic compaction. Compaction rates above 10^3 s^{-1} are designated high rates of compaction. There have been several studies in this area recently (Roman, 1980; Tunaboylu, 1994; Tong, 1994; Joslin, 1995). Sano (1977) studied dynamic (high speed) compaction and he concluded that dynamic powder compaction has advantages over static compaction (low speed) ; they are: higher green densities, more uniform green density distributions, lower ejection loads and improved dimensional accuracy. Evaluations of pressing characteristics for the static and dynamic compaction stages were also reported in this study.

CHAPTER 2 : LITERATURE REVIEW

2.1 : Factors Affecting Compaction Pressure and Compressibility

In many of the early studies, most of the consolidation processes were carried out using cold uniaxial compaction. Since the aim is to obtain fully dense powder compacts, the influence of the physical characteristics of the powder i.e., particle size and shape, materials being used, work hardening of the particles, etc., on the compaction process and compressibility has been studied extensively for many years. Compressibility or compactibility is described in ASTM B 243-94 as "the capacity of a metal powder to be compacted uniaxially in a closed die. It is expressed numerically as the pressure to reach a required density or the density at a given pressure".

In uniaxial pressing there is a fundamental compact initial length-to-diameter (L/D) ratio which affects the density distribution of the final compact body. This limit is suggested to be $L/D = \leq 4$ for uniaxial pressing by McEntire (1991). Powder compaction process involves several stages (Seelig 1947): these are as follows;

- Filling the die with loose powder,
- Packing and rearrangement (with or without tapping),
- With increasing pressure, elastic and plastic deformation occur,
- Expelling air from the die,
- Bulk deformation and fragmentation.

2.1.1 : Frictional forces

In uniaxial pressing, either single action or double action, the applied load is not transmitted fully to the powders (Ridgway 1992). That is because of frictional forces between particles and more importantly between the die wall and particles (Kamm, 1947). Balshin (1936) first recognized that density variations in compacts pressed uniaxially resulted from friction at the die wall. He found that the greatest density in a compact existed at the top near the outside edge while lower densities were found at the compact centre. Therefore, low friction is preferred (Briscoe & Evans 1991). In order to eliminate or minimize this adverse effect of friction, admixed and/or die wall lubricants are used

during compaction (Gethin 1994). This permits high effective pressures to be transmitted to the compacted mass and gives high green density for a given applied pressure. This also helps to prolong tool life and lower ejection pressures.

Frictional forces were divided into four groups by Leopold and Nelson (1968): these are as follows;

- I - Between moving punches,
- II - Interparticle friction,
- III - Particle and die wall friction,
- IV - The final stage, during ejection, between the compact and die wall.

Frictional forces between particles and the die wall are the major ones that exist during the compaction process. This is affected by the number of powder particles in the mould, which is itself a function of the shape and amount of powder, as well as surface conditions. Leopold and Nelson (1968) carried out work on admixed and die wall lubrication and concluded that die wall lubrication is best for higher densities at high pressure ranges, while admixed lubricant gives good results at relatively low pressure ranges. Thomson (1981) suggested that lubricating the die wall was preferable to addition of lubricant into the powder. Briscoe and Evans (1991)

measured die wall friction using ceramic powders.

The effect of lubricants on the compaction pressure and physical properties of the final compact has been studied by Leopold & Nelson (1968) and Siddiqui (1992). It has been reported (Siddiqui 1992) that zinc stearate improves the compressibility of atomized iron powders and enhances particle cohesion.

2.1.2 : Particle size and apparent density

Many of the compacted powder properties are related to the particle behaviour, size and shape (Smith, 1983; Seelig & Wulff, 1946). Particle size, size distribution and surface area have a high influence on the apparent and final compact density (Koerner, 1971; Smith, 1962; Huffine, 1963; Leiser, 1970; Lawrence, 1970). Initial powder height (which is related to apparent density) in the die cavity determines the compressibility of the powder materials.

Koerner (1971) carried out research into the effects of particle size distribution on the compaction of iron, nickel and iron-nickel powders. He concluded that for decreasing particle size, the flow rate and green

density decreases while the apparent density and green strength were very dependent on the type of the powder. Al-Tounsi (1993) investigated the effect of particle size on the compaction behaviour and green strength of the ceramic powder. He came to the conclusion that small (average) particle size powders gave better compressibility.

Unisized ceramic powders were used by Copper and Eaton (1962) to study compaction behaviour. They concluded that filling of the small holes in a powder mass required considerably higher pressures than the filling of large holes.

2.1.3 : Particle shape

In general, the surfaces of powder particles are uneven; it is assumed that even at high compaction pressures only limited particle contacts exist (Rhines, 1946). Therefore, in order to compact hard and brittle powders, micron particle sizes are usually employed to ensure a maximum number of contacting points and sufficient adhesion after pressing (Wulff, 1942). The effect of the particle shape on the compaction behaviour of the powder pressing has been studied by Seelig, 1947; Leiser, 1970; Kostelnik & et al., 1968; Meyer, 1969; and Fell, 1971. It was found that particle shape had a large influence

on the compaction behaviours of powders. Beddow (1968) studied the effect of particle shape on the pressure transfer characteristics of copper powders with three different shapes and surface characteristics. A result of this study was that spherical powders transmitted pressure most effectively and flake powders transmit pressure the least effectively. A high degree of roundness in shape may be advantageous, because of the good flow properties and packing density of such powders.

2.2 : Pressure - Density Equations

During the last 70 years much research has been carried out on pressure - density relationships. In order to describe the powder behaviour within the die or mould under an external pressure, a variety of mathematical equations, both theoretical and as a result of practical work, were suggested within a limited range of application. Most of these mathematical formulae were proposed in attempt to account for the affect of pressure on the green densities of compacts. Because of differences in density throughout the compact body, in all early derived equations the average density was the main measurement criterion. For isostatic compaction process, however, density was assumed to be the same

throughout the compact.

Empirical relationships are based on porosity, compact volume, green density and applied pressure. Using two variables, either applied pressure-compact volume or green density-pressure, straight lines were evolved to result in log-log or log-linear relationships. One of the early basic formula was derived by Balshin (1938). The equation given below was used for a limited range of compaction pressures,

$$\ln P = -aV + c \quad (1)$$

where V is the relative volume at a pressure P; a and c are constant. This expression is applicable for low pressures and specific powders. Later Seelig (1947) and Smith (1948) independently developed more basic equations. Their equations (using their terminology) are:xxx

$$\frac{\theta}{\theta_0} = \exp\left[-\left(\frac{P}{a}\right)\right] \quad (2)$$

$$D - D_0 = b \cdot P^2 \quad (3)$$

$$D - D_0 = c \cdot P^{1/3} \quad (4)$$

where, a, b and c are empirical constant, dependent on material properties, D_0 is the initial density of the powder and θ_0 is the initial powder porosity. D and θ are the compact density and porosity at pressure P, respectively.

Heckel (1961) investigated density - pressure relationships using Ni, Fe, Cu and W powders. He concluded that the rate at which density increases with applied pressure for metal powders was proportional to the volume fraction of pores (for pressures exceeding a lower limit), which is dependent on the powder. Heckel suggested the following equation,

$$\ln\left(\frac{1}{1-D}\right) = k \cdot P + \ln\left(\frac{1}{1-D_0}\right) \quad (5)$$

where D_0 is the relative density of the loose powder at zero pressure. $\ln(1/1-D_0)$ is a constant, called A, so putting the A into equation (5) above, gives

$$\ln\left(\frac{1}{1-D}\right) = k \cdot P + A \quad (6)$$

D is the relative density of the compact at pressure, P.

k is the slope of the linear region on the relationship; this was related to the yield strength (σ) of the material, i.e., $k \propto 1/3 \sigma_y$.

A quantitative compaction model was proposed by Cooper and Eaton (1962), who claimed that it was applicable to a wide range of ceramic powders. The model is based on the reduction in compact volume due to applied pressure. At intermediate pressures large voids are filled, and at relatively high pressures small voids are filled; the equation is,

$$V_1 = a_1 \cdot \exp\left(-\frac{b_1}{P}\right) + a_2 \cdot \exp\left(-\frac{b_2}{P}\right) \quad (7)$$

where,

$$V_1 = \left(\frac{V_0 - V}{V_0 - V_2} \right) \quad (8)$$

V_1 = fractional volume after compaction

V_0 = initial volume of compact

V = volume of the compact at pressure, P

V_2 = volume of the compact of theoretical density

a_1, a_2, b_1 and b_2 are constant related to the mechanics of compaction.

The Cooper and Eaton model did not take into account the loss of transmitted load in the powders.

Kawakita and Lüdde (1970) attempted to identify various stages in consolidation process and obtained a relationship,

$$C = \left(\frac{V_0 - V}{V_0} \right) = \frac{a \cdot b \cdot P}{(1 + b \cdot P)} \quad (9)$$

where,

$C = V_0 - V / V_0$ is a measure of volume reduction,

V_0 is the initial apparent volume,

V is the powder volume under the applied pressure, P , and a, b are constants, characteristic of the powder.

This equation is widely used in the field of powder compaction. Because of its consistency, it is used for many pharmaceutical products. After rearranging equation

(9),

$$\frac{P}{C} = \frac{1}{a \cdot b} + \frac{P}{a} \quad (10)$$

the constants can be evaluated graphically from the linear relationship between P/C and P . This equation, however, is limited on the range of soft and medical powders.

Green strength has been found to be related to the contact area between particles (Hyungmoon, 1985). Hyungmeon derived some simple equations from geometrical considerations to describe the relationship between green density and contact area.

Using a uniaxial strain model, Es-Saheb (1985) focussed on the influence of compression rate on the residual strength using density-pressure relationships: The Heckel and Kawakita diagrams as well as axial and radial pressures were examined. McEntire (1991) gave consideration to empirical and quantitative models to predict green densities during compaction.

Bockstiegel (1965) investigated uniaxial and isostatic compaction using iron powders; he critically evaluated

the Konopicky (1948) and Shapiro & Kolthoff (1947) equations, concluding that these mathematical expression required that prediction of the final compaction pressure was dependent on a known fraction of porosity.

Rong-de (1991) has recently proposed a powder compaction equation based on several previously published data for metallic and non-metallic powders. The equation is;

$$\text{Log} \left[\text{Ln} \frac{(1-D_0)}{(1-D)} \right] = A \cdot \text{Log } P + B \quad (11)$$

where,

D_0 is the relative density of the loose powder,

D is the relative density of the powder compact

under pressure, P ,

A and B are the constant.

2.3 : Physical and Mechanical Characteristics of Powders

The physical and mechanical properties of a particular powder in the die play an important role in their

densification, namely, friction, flow, fracture, lubrication and elastic & plastic deformation and interparticle bonding. Correlation of consolidation with the physical, chemical and mechanical properties of the compact, interparticle friction and interaction was examined under various compaction pressures, (Stanley 1987). The effect of additives, lubricants and binders on material properties has been researched earlier by Yarnton and Davies (1963). Hyungmoon (1985) found that bond strength increased by increasing the surface contact due to high plastic deformation.

Sarumi (1991) explored the effect of different compaction rates on powder compact densities. Sakuma (1992) reported that high purity iron powder had high compressibility. It was reported by Vanazountas & Davies (1992) that the addition of alloying elements lowered the compressibility of pure iron powders. Ohji (1994) studied the effect of pre-heat treatment of loose powders on the final densities, microstructure and strength of SiC compacts; he concluded that a pre-sintering heat treatment was effective in improving the microstructure, density and mechanical properties of pressed and sintered silicon carbide. Hirschhorn and Garey (1969) reported that the major effect of annealing was to decrease the degree of work hardening by reducing the dislocation density and increasing the grain size.

Song and Chandler (1990) found that adhesion between the powders and die-wall can sometimes be noticeable in the compaction of ceramics powders. Mechanical characteristics of individual particle under an external force were examined by Takahashi and Suzuki (1990); compaction behaviour of powders under consolidation were explained in terms of the individual particle interactions and the packing coordination of particles.

Stanley-Wood (1987) attempted to correlate uniaxial compaction processes with the physico-chemical and physico-mechanical properties of a coherent compact. Stepanov and Zubov (1990) carried out a work on the physical changes of porous metal powders during compaction and calculated stress-strain states.

2.4 : Micro-Hardness and Work Hardening Rate

One of the early study in this area was made by Jones (1937), proposed several phenomena to explain increased hardnesses of compacts with increasing compaction pressure; these consisted of grain size, surface oxide films and strain in the powder particles.

Hirschhorn and Garey (1969) examined the stage of plastic deformation during compaction of metal powders

and attempted to correlate this with compaction pressure which resulted in an increase in particle hardness due to work hardening.

Lawrence (1970) found that at equivalent applied pressures the relative densities of various mineral powders were lower for powders containing particles with high Young modulus and hardness.

A series of "mono-size" $-126+106\text{ }\mu\text{m}$ metal powders of different hardness and work hardening capacities were isostatically compacted over a pressure range 0-770 MPa by James (1977). He concluded that particle plastic deformation commenced rapidly in all powders where densification is at first governed by the yield stress of the material and later by its work hardening capacity and hardness. Powders of high work hardening capacity and low hardness densified more efficiently and vice versa.

Cooper and Eaton (1962) studied ceramic powders with four different hardnesses, and found that the harder and more brittle the powder, the higher was the final compact's ultimate porosity. They also included that the compaction behaviour of the ceramic powders depended markedly on their hardness.

Caroll and Kim (1984) proposed an empirical equation to

explain the effect of work hardening on the compaction of metal powders.

2.5 : Porosity

Initial porosity in a mass of powder has a major effect on the final density and the retained porosity of the product. Cooper and Eaton (1962) divided pores into two size groups. The first group pores are large that were filled during the rearrangement stage by particles of approximately the same size. The second group of pores are small that can only shrink by bulk movement or plastic flow or disintegration of the particles around the pores. Bockstiegel (1965) carried out work on porosity-pressure curves and the effect of pore-size distributions on the densification process. He expressed pore-size distribution and its change with increasing pressure by the relationship;

$$U_{(p)} = U_{(o)} \operatorname{erfc} \left[\operatorname{Log} \frac{D_o}{D_{\max(p)}} \right] \quad (12)$$

where,

$U_{(p)}$ is the porosity at pressure, P ,

$U_{(0)}$ is the porosity at zero pressure,

$D_{\max (p)}$ and $D_{(0)}$ are diameters of the largest pores at pressure P and 0 , respectively,

$\text{erf } c$ is the complementary error function.

Pore sizes are readily described when they are isolated in a static body; otherwise, description of the porosity at pressure is much more difficult. Linear relationships between porosity and pressure when plotted in either a semi-log or log-log scale were not obtained by Bockstiegel.

Ramakrishnan and Sivakumar (1990) evolved an expression for the contact area in terms of porosity, using results from the data of James (1977) and Fischmeister (1978) for a hollow sphere model. Their equation is as follows;

$$\text{Ln}[1 - (\frac{S_c}{S_0})] = m \cdot \text{Ln}[(\frac{Q}{1-Q})] + C \quad (13)$$

m has a value of $2/3$ for spherical voids

$$C = -(\frac{2}{3}) \text{Ln}[(\frac{Q_0}{1-Q_0})] \quad 28$$

(14)

where,

m is the slope of the linear plot between

$\ln [(Q/1-Q)]$ and $\ln [(1-(S_c/S_o))]$,

c is the y-intercept of the linear plot between $\ln [(Q/1-Q)]$ and $\ln [1-(S_c/S_o)]$,

S_c is the total contact area between all the particles,

S_o is the total surface area of all the particles or total surface of all the voids when $Q=Q_o$,

Q is the porosity,

Q_o is the initial porosity or "1-tap density".

CHAPTER 3 : EXPERIMENTAL PROCEDURE

3.1 : Materials

In the present work two types of atomized iron powders having different particle size, a spherical bronze powder and a copper powder were used. The size fraction for the iron powders ranged from 45 μ m - 150 μ m. The powder specifications are given in table 3.1. Zinc stearate and acetone were used in the volume fraction of 1/10 zinc stearate dissolved in acetone to act as a die wall lubricant.

The die barrel was supported by a spring so that the die performed in a double-action manner; this is termed "floating-action". The die used for compacting powders was made of high carbon high speed steel. The diameter of the die was 16 mm with a length of 70 mm. A 0.001 gm sensitive balance was utilised for weighing the powders and the final compacts. The compacting machine used for pressing was a type 1081 Instron, 200 kN max. load capacity, tension machine.

3.2 : Specimen Preparation

All the iron powder samples were weighed to approximately 7 gm weight with an accuracy of 0.001 gm. The copper and bronze powders were weighed to 8 gm to give an approximately similar sample height after compacting, which was 5 to 7 mm. This allowed good density distribution in the compact and reduced die wall friction. Before compacting the powders, the die wall was lubricated with the zinc stearate solution.

The surfaces of both punches and die wall were coated with the solution to leave a uniform coating of lubricant. After each compaction the die wall and punches were cleaned and relubricated for the next pressing.

3.3 : Consolidation of Powders

In the preliminary work, samples of weighed powder of a selected size fraction were pressed uniaxially using a single action die at room temperature using a range of pressures for the each powder size. Iron powders were compacted at pressures up to 850 MPa, up to 800 MPa was used for bronze powders and copper powders were pressed at pressures up to 950 MPa. This resulted in compacts of

4 to 7 mm thickness with a diameter of $16,04 \pm 0,05$ mm depending on the maximum compacting load. At the maximum load a hold time of 30 sec. was used for each compaction. The rate of compaction was determined from the movement of the upper punch. Cross-head speeds of 1 mm/min to 15 mm/min were used throughout the consolidation processes. The compacting force and displacement of the moving punch were recorded automatically.

Before application of the compacting pressure the powders in the die were tapped followed the method recommended is by American Society for Testing and Materials Standard ASTM B 527. This was carried out to achieve the maximum tap density in order to secure constant initial powder height. By maintaining a constant aspect ratio i.e., ratio of the compact thickness to compact length, density variations in the compact body could be assumed to be minimized.

3.4 : Density Measurements

Compacts of 16 mm diameter and 5 to 7 mm thickness were produced. The density of the compacted powders were determined by either a weighing and dimensional measurement method or by a punch displacement method

i.e., punch displacement was recorded continuously against increasing load. The maximum load was recorded an ejection of the compact from the die; this did not include a correction for elastic springback of compacts that occurs when load was released. Final compact thickness, diameter and weight were also measured after removing the compact from the die using a micrometer ($\pm 0.001\text{mm}$) and the mass was determined to $\pm 0.001\text{ gm}$. It was assumed that the final pressure was equal to the transmitted pressure on the compact because of the die wall lubrication and the small thickness / diameter ratio. From the displacement data the green density was calculated using simple equation below;

$$d = M/V$$

$$V = \pi \cdot r^2 \cdot (h_i - h)$$

Where,

d is the green density at pressure,

M is the mass of the powder,

V is the volume of the green compact,

r is the radius of the die cavity,

h_i is the initial powder height, calculated from the tap density,

h is the displacement of the punch.

Because of the difficulties in accurately determining the initial powder height, these density measurements (at pressure) did not yield such good reproducibility as those obtained by measurement after ejection of the compact from the die.

3.5. : Optical and Scanning Electron Microscopy

Optical microscopic examinations were performed to determine the fractional porosity and the pore shape; micro hardness measurements using a Reichart indenter were also performed. To examine the fracture surface and to determine contact area between particles a Phillips SEM-505 operating at 20 kV was used.

Some of the compacted samples were sectioned, and if necessary polished and etched for microstructural changes and porosity distribution. The etching agent used for iron compacts was 2 % nital solution.

For SEM examination, compacts were fractured by performing transverse rupture strength test on compacts following the recommendations of ASTM B 312 and Metal

Powder Industries Federation Standard MPIF 15.

3.6 : Micro Hardness

Micro hardness measurements were carried out on compacts of the 45 μ m-106 μ m sieve fraction iron powders (category I and II in Table 1). In order to avoid displacement of particles under the indenter both compacted and loose powders were moulded in a resin for 100 min under vacuum. These samples were then etched in 2% nital solution. Polished samples were tested using a Vickers diamond pyramid indenter with a load of 20 gm in a Reichert Hardness Tester; a holding time for each sample was 5 sec at a load of 20 gm.

The Vickers microhardness V_{MH} value was determined using the equation below,

$$V_{MH} = 1854,4 L/d^2 \text{ kg/mm}^2$$

Where,

L is the applied load, 20 gm.

d is the base diagonal in mm.

Samples of iron powders compacted at a constant cross

head speed of 1 mm/min in the load range 40 to 110 kN were selected for this particular experiment. From each set of samples, 20 impressions were taken per sample to obtain mean micro hardness values.

CHARTER 4 : RESULTS AND DISCUSSION

4.1 : Compressibility of the Iron, Copper and Bronze Powders

Using the experimental data, pressure-density, pressure-contact area, pressure-hardness correlations have been developed graphically. In this particular work iron powder II (Table 1,2 and 3) is found to have better compressibility than iron powder I up to a compaction pressure of 700 MPa (Fig: 4.1.1., 4.1.2). [The line drawn on the pressure - density plots is the mean of three separate measurements]. In the range 700 to 850 MPa the reverse was found. This can be justified in terms of the initial compact density. Apparent and tap densities, particularly tap density play an important role in the compressibility of the powders. Since iron powder II has lower tap densities than iron powder I (Table 2), it's potential compactibility is higher. At higher compaction pressures, however, the consolidated powders behaviour is indistinguishable from that of bulk material. Accordingly, porosity within individual

particles cannot be reduced even at high pressures, (Fig. 4.1.3; 4.1.4): this internal porosity affects the apparent density, so that nonporous particles tend to have higher compressibility.

Examination of the fracture surface after stage I shows that some fragmentation occur locally on the periphery of the contact areas between particles. Even when compaction stresses are low on the top surface of the powder compact they can be high at the small actual contact areas of individual particles. In this stage fragmentation is likely to arise particularly in the irregular shapes particles, as was observed for both types of iron powders (Fig.4.1.5).

4.2 : Density Measurement

4.2.1 : Tap density

Constant states of tap densities (Table 2, 3) were achieved before pressing started. The aim of doing this was to have high initial packed densities and consequently a small displacement in the rearrangement stage, stage I. Initial close packing in a particle

assembly decreases the amount of porosity and results in high compact density for a certain pressure. Typically, the change in poured density packing coordination going from the as poured state to tap density state caused an increase in apparent density of 14-38 % in iron powders; this increase was related to the particle size. For copper and bronze powders this was only 3-8 % i.e., the extent of the increase depends on the particle size distribution. The increase in density from apparent to tap density of spherical powders was small due to high relative density of the loose powders.

4.2.2 : True compact density

Because lubricant was added directly to the die wall and not admixed with the powder, the measured density is considered to be a green density of the metal powders alone and was determined in the standard manner by dividing the mass by the volume of the compact after consolidation. Dimensional expansion of the compact was noted after ejection of this green compact from the die. Because of relaxation of residual stresses, some "springback" occurred. The magnitude of this is related to the properties of the powder being compacted and the applied pressure. For iron powders, at different

compaction pressures (200 MPa - 850 MPa), springback was related to the diameter of the green compact after ejection (Table 4). The diameters of compacts of iron powder I were found to be slightly bigger than for iron powder II for compacting pressures greater than 350 MPa. This expansion in the volume of the compact after ejection gives rise to difference in the two measurement of green density i.e., the density at pressure in the die cavity and density after ejection were not equivalent.

The true compact density is expressed as a percentage of the theoretical density (specific density) of the materials being used. At a maximum pressure of 850 MPa, the highest true compact densities were achieved by iron powder I, i.e., 94 % (Fig 4.2.1; 4.2.2) . At 800 MPa, copper has a true compact density of 91 % and the bronze powder had 83 % at the same pressure. The differences can be explained in terms of the ability of these materials to deform plastically, this is especially low for bronze powders. The pressure - density correlation for copper and bronze powders are given in Figure 4.2.3 and Figure 4.2.4, respectively.

4.3 : Pressure - Density Relationships

4.3.1 : Powder behaviour during compaction

The process of compaction of powders in a die by uniaxial compaction consists of four main parts;

- 1) Filling the mould or die with powder,
- 2) Tapping the die to achieve a pre-set initial density,
- 3) Consolidation occurs under an external pressure,
- 4) Ejection of the compacted powder from the die.

In general, it is assured that with increasing applied pressure the density of the powder increases; in other words, when pressure increases, porosity decreases, (see Fig. 4.4.1; 4.3.2; 4.3.3). The rate of volume change is high at the first stage of application of pressure; this happens when the pressures are less than 200 MPa (as seen in Fig. 4.3.1; 4.3.2; 4.3.3). At this stage, smaller particles are forced to move into suitably sized voids, at the same time the air that occupies these voids is displaced and is expelled along the die wall surface.

For the iron and copper powders, the particle size range

was such that the fine particles can locate between the medium size particles, and medium size particles can fill voids in between coarse particles. This is illustrated in the photographs of the fracture surfaces (Fig. 4.3.4; 4.3.5).

4.3.2 : Compaction stages

The results of the present study confirm that the consolidation stage of powder compaction (stage 3) consists of four parts, as follows;

- I-** An initial stage, which is particle rearrangement and restacking,
- II-** A second stage, where particle deformation at contact points takes place,
- III-** A third stage is the elastic - plastic deformation and fragmentation of particles,
- IV-** A final stage that consists of cold working of the "bulk" material.

There is, however, no certainty that any one stage can be separated from another. In the initial stage of the compaction the amount of densification is controlled mainly by the external characteristics of the particles. Individual particles slide in order to fill the gaps between the particles with a minimum of deformation and fracture. This happens by the interparticle motion of particles with each other, this may cause elastic deformation, local fragmentation or limited plastic flow of particles. However, irregular shaped particles may produce some fine particles in this stage. Particle shape and size distribution play an important part during this stage. Fragmentation may occur even at very low compacting pressures and in the early (first) stage. This is because the pressure created at interparticle contact areas cause sufficiently high stresses to promote deformation by fracture. The rearrangement of particles are limited by the particle shape , size, size distribution, particle interactions and friction between particles. This stage takes place at a relatively low pressure range. After being tapped the amount of particle rearrangement may be low; following this, substantial particle deformation occurs in stage II and leads to further consolidation.

In the second stage, elastic - plastic deformation plays a major role. The voids between the primary particles

are smaller than the original particle sizes; these are filled by fragmentation and plastic flow. The plastic flow in the case of regular shape powders, such as copper, is the primary reason for densification. For irregularly shaped iron powders, on the other hand, fragmentation is a dominant process in the filling of voids. This is the major densification mechanism and leads to high green density and green strength by making the interparticle contact area significant. However, the extent of stage II is limited by the ductility, yield strength and work hardening characteristics of the particles being used. In the case of hard and brittle particles where there is little or no plastic deformation, stage III is encountered before any insignificant changes occur in stage II. It is particle fracture or fragmentation that becomes the main densification mechanism, even for regularly shaped particles.

In either stage I or II, stress distributions on individual particles may alter. In some cases this could be high enough to force particles into the pores plastically even though the applied load in general is in the intermediate range.

In the third stage where the development of substantial interparticle contact area occurs (Fig. 4.3.6), a state is reached that results in isolated pores and only a

little further densification takes place. Small pores are filled by the fragmented particles causing further deformation and densification. In this stage work-hardening of particles plays an important role. At very high pressures the intergranular porosities are eliminated (Fig. 4.3.7). From the figure it can be seen that at an applied of pressure 850 MPa, intragranular porosities remain but they are much smaller than at intermediate pressures (300-500 MPa), Fig. 4.3.6.

The final stage was found to be essentially cold working of the consolidated mass. Agglomerates are no longer able to reorient or fragment because of insufficient remnant cavities.

4.3.3 : Powder compaction equations

Various theories and mathematical analyses that were discussed in chapter 2 consisted of attempts to interpret the compaction process in terms of the relative density and applied pressure. However, none of the equations are completely satisfactory because they are unable to cover even the limited ranges of pressures, particle shape and sizes used in this investigation.

It will be recalled that the Kawakita and Heckel

equations attempt to correlate pressure and pressed density relationships. One of the aims of this investigation was to validate the green density - pressure curves obtained under uniaxially conditions for irregular shapes atomized iron powders, spherical copper and bronze powders that would obey the formulae of Heckel and Kawakita. The data obtained in this present investigation, shown in Figures 4.1.1; 4.1.2, were applied to equations (6) and (10) (chapter 2) to test their validity. Kawakita's equation, (10) was found to give a satisfactory relationship at pressures above 100 MPa (Fig. 4.3.9). The present data were also analyzed by plotting $\ln (1/1-D)$ vs pressure as shown in Figure 4.3.8, following Heckel. The diagram shows three regions, suggesting that three distinct mechanism take place at different compaction stages. It is interesting to note that the initial stage has a very narrow range. This could have been because of the procedure of standarding an initial tap density before compaction. A plot of P/C vs pressure (see equations 9 and 10, in chapter 2), following the Kawakita equation (Fig.4.3.9), however, produces a good linear relationship, similar to the mid-section of the Heckel diagram (Fig. 4.3.8).

4.4 : Compaction Pressure / Contact Area / Green Density

Spherical particles are easy to pack and can give high apparent densities without bridging or arching that create hollows. They move easily over one another due to low inter particle friction and lack of roughness in shape. This allows the particles to relocate effectively into the gaps in between the previously settled particles.

The effect of the applied pressure on the green density of compacts, for all the powders used, is presented in Figures 4.1.1; 4.1.2; 4.2.3; and 4.2.4. A general feature of all these curves (except Fig. 4.2.3) is that the slope of the curve decreases as pressure increases. This is due to the contribution of the mechanical properties of the powders, such as work hardening. For the copper powder (Fig. 4.2.3) however, the slope of the density / pressure curve remains constant after 500 MPa. This could imply that consolidation processes and particle deformation were complementary

Relationships between the compact surface area in contact with the die wall and compaction pressure are given in Figure 4.4.1; 4.4.2, for iron powders I and II. After an initial negative rate, the rate of the change of slope is very rapid for iron powder II and then

flattens out at about 400 MPa. Iron powder I, on the other hand, the rate of change of this material slope is more gradual to a pressure 550 MPa when it becomes constant. This behaviour is paralleled by the pressure - thickness / diameter relationships for iron powder I and II, given in Figure 4.4.3 and 4.4.4, respectively.

4.5 : Effect of Compaction Rate on Consolidation

Different rate of application of compacting load were used to investigate the effect of the compaction rate on the densification mechanism of iron powders; this was achieved by adjusting the cross head speed on the press. The pressing rate ranged from 1 mm/min to 15 mm/min, the maximum speed used. The results are given in Figure 4.5.1 and 4.5.2. For iron powder I (Figure 4.5.1), compacted density increased by 2.5 per cent at a final compaction pressure of 550 MPa. For iron powder II, on the other hand, an increase of 3.4 per cent, compared to the tap density, was obtained. This demonstrates that iron powder II is more "compaction rate sensitive" than iron powder I. The reason for this could have been initial particle hardness. For iron powder I the microhardness value was $\sim 79 \text{ kg/mm}^2$, with a powder size

fraction of 45-106 μm , whereas, for iron powder II, with the same size fraction, the micro hardness value was $\sim 65 \text{ kg/mm}^2$.

4.6 : Pressure - Density - Micro Hardness Relationships for Iron Powders

Micro hardness measurements were carried out on both iron powder I and II in order to examine the effect of pressure on the work hardening of powders. It is seen in Figure 4.6.1 that there is a significant increase in hardness with compacting pressure. The zero pressure hardness values on the diagram are the hardness values of loose powders (as-received materials). It is seen that the micro hardness increases from the start of stage I. A substantial amount of work hardening occurs in the early stage of compaction as shown in Figure 4.6.1. The slope of the curves are high up to 200 MPa compacting pressure and in the range of 400 MPa to 600 MPa. This demonstrates clearly that the work hardening effect is significant not only (as expected) in the latter stages of compaction (stage III) but occurs at the beginning of stage I. This could be due to extensive high interparticle motion and interparticle friction that may occur at this stage.

The compaction pressure - microhardness results can be correlated with the density / micro hardness behaviour. As can be seen from Figure 4.6.2, in general, when green density increases microhardness increases, at the beginning it rises rapidly and then levels off and finally rises sharply again. It is noted that for iron powder II micro- hardness does not increase continuously up to a final density achieved, but seems to saturate. The cause of this is that after a certain compacting pressure and compacted density state, particle motion stops and further densification is only achieved by reduction of the remaining porosity. Further increase of compaction pressure results in increased plastic deformation and work hardening of individual particles but no increase in green density.

The increases in microhardness in iron powder II was a maximum at ~91 per cent after compaction at 850 MPa, higher than that of iron powder I, that had a maximum value of ~67 per cent (Fig. 4.6.3). It was noted that iron powder II had a lower tap density. This may have lead to more interparticle motion and interparticle friction when pressure was applied to achieve a given green density than was the case for iron powder I. It transpires that the greater the amount of interparticle interactions that occur, the higher the microhardness values of the final product of compaction.

4.7 : SEM and Optical Microscopic Analyses

4.7.1 : Interactions between particles

A consequence of the particle size distribution within the powder body is that there will be variations in the degree of particle deformation in different part of the compact. Maintaining constant particle sizes and small length / diameter ratio for the compact, ensures that the degree of particle deformation within the compact might be homogenous. Figure 4.6.7 demonstrates qualitatively that for unisized (~220 μm) spherical bronze powder compacted 800 MPa there is relatively homogenous plastic deformation on the particles.

The compacts of spherical bronze powders compacted at 800 MPa disintegrated on ejection from the die cavity. This could be due to a lot of energy being stored as elastic deformation in the compact, and being recoverable, constitutes a fracturing process when the die constraint is removed rather than a strength producing mechanism involving plastic deformation. Elastic springback forces in the compact could have been high enough to break any interparticle attractive forces of compacting particles. A postulate by Rietema

(1991) that could be operative in this case is that contacting particles could give rise to electrically similar (repulsive) charges, since Coulomb's rule applies: interparticle forces on similar charged surfaces are repelling and not attracting, (Rietema 1991).

With increasing compaction pressure, work hardening rate increases in the readily deformable powders and this leads to an exhaustion of workhardening capacity which ultimately causes powders to act in a brittle manner and may lead to further densification by particle fragmentation. This is especially observed in the case of the irregular shaped iron powders (Fig. 4.6.5). It was previously shown that particle hardness increases from the beginning of the compaction process; this may cause fragmentation when the particles get harder, progressing through stage I ,II and stage III. This limits further densification due to a lack of plasticity.

4.7.2 : Relationships between compaction pressure and compact porosity

Figure 4.6.8, which is a cross-sectional view, confirm that iron powder I had a fairly wide particle size

distribution and that some of the particles had internal porosity. Microphotographs, Figures 4.6.9; 4.6.10, present the change in pore structure (size and shape) with increasing pressure for iron powder II; as the pressure increases from 300 MPa to 500 MPa big pores are largely eliminated. Interconnected pores shown in Figure 6.4.9 modify into the isolated pores shown in Figure 6.4.10. Intergranular porosities almost disappear but some intragranular porosities remain in individual particles.

4.7.3 : Examination of contact area by SEM

With increase in compaction pressure, the contact areas between powder particles increases. This leads to high relative motion at the contacts of individual particle i.e., centre-centre shortening. Relative interparticle motion becomes progressively more difficult so that high compaction pressures are required in stage III and IV. In the early stage (stage I) the initial areas of particle contacts are low i.e., almost point contacts, so that, at only very low loads, the pressures at these contacts are high. The surface topography is complex because of the range of types of interparticle contacts

that exist. Forces acting on the surface contacts determine the strength of the bond formed at particle interfaces. There was little or no evidence of cold welding on the contact surfaces of the spherical bronze or of the copper powders, (Figure 4.3.4; 4.6.7) even at high pressures. This indicates that the residual attractive forces between particles are weak. This is an expected for the plastically hard bronze powders but it is rather a surprising finding for the plastically deformable coppers powders, unless perhaps, the copper powders were coated with a thin film of oxide. [The oxide contents of the powders were not determined].

The metallographic observations give clear indications that substantial plastic deformation, pore closure, particle surface flattening and particle fracturing occurred at high pressures. Because of the surface roughness for the atomized irregular shape iron particles, only relatively small parts of their total surface area come into contact. This facilitates plastic flow at local areas even at low applied load, since enormous pressures could be generated over the infinitesimal areas of particle contacts. Bonding could be in various forms; for the irregular shape iron powders mechanical inter-locking was observed.

After compaction at ~800 MPa, the original spherical bronze powders had acquired the shape defined as

tetrakaidekahedron (James, 1977), shown in Figure 4.6.6.

CHAPTER 5: CONCLUSION

- The compaction equations quoted in chapter 2.2 did not give linear curves over the range of compaction pressures used in this investigation. Deviation from linearity occur at low compaction pressures.
- The Kawakita formula was found to be valid at pressures above 40 MPa in this particular work.
- It is difficult to sperate the mechanisms that lead to densification during the stages identified as I, II and III.
- Initial tap density has an influence on the compressibility of the powders as it determines the initial powder height in the die. As a result of this, iron powder II has a higher compressibility than iron powder I up to 700 MPa.
- Local plastic deformations occurs at even low compaction pressures for irregular shape powders, due primarily to high stresses at small contact areas on individual particles.
- The initial stage (stage I) was of short duration, because of the high tap densities obtained before

pressing commenced. Conversely, it may be concluded that a high tap density results in high effective compressibility.

- The effect of compaction rate on the irregular shaped iron powders was examined. The maximum compaction rate used, 15 mm/min, gave an increase in green density of 3.4 % at a final compacting pressure of 550 MPa.

- Microhardness assessments performed for iron powder I and II demonstrate that work hardening is operative even in the early stages of the compaction due to relative particle motion and interparticle friction.

CHAPTER 6: FOR FURTHER WORK

- Further investigation on the influence of the initial particle hardness of different size and shapes of metal powders on the work hardening of the compact should be studied.
- The effect of the grain size on individual particle microhardness during the compaction process needs to be researched.

The effect of evacuation of air from compacts following the application of a small compaction pressure would be worthwhile. The surface energies that exist in vacuum are higher than in air where surface contamination reduces the adhesion between particles. Accordingly, clean particles compacted in a vacuum should demonstrate better interparticle bonding than those with an oxide film on their surface.

BIBLIOGRAPHY

- Al-Tounsi, A. et al.**, (1993), J. of Materials Processing Tech., (37), 543-550.
- Balshin, M.Yu.**, (1936), Vestnik Metallopram, (16), No.16, 87-120.
- Balshin, M.Yu.**, (1938), "Theory of Compacting", Vestnik Metalloprom (Russian Journal), (18), No.2, 124-137.
- Beddow, J.K.**, (1968), Inter. J. of P/M, 94), No.1, 27-29.
- Bockstiegel, G.**, (1965), Modern Developments in Powder Metallurgy, Vol.1, 155-187, New York.
- Briscoe, B.J. & Evans, P.D.**, (1991), Powder Technology, (65), 7-20.
- Carroll, M.M.**, (1984), Powder Metall., (27), No.3, 153-159.
- Cooper, A.R. & Goodnow, W.H.**, (1962), Bull. American Ceramic Soc., 41, (11), 760-761.
- Cooper, A.R. & Eaton, L.E.**, (1962), J. American Ceramic Soc., (45), No.3, 97-101.
- Es-Saheb, M.H.H.**, (1985), Ph.D. Thesis, UMIST.
- Fell, J.T. et al.**, (1971), J.Pharm. Sci., (60), No.12, 1866-1869.
- Fischmeister, H.F. et al.**, (1978), Powder Metallurgy, (21), 178-187.
- Gethin, D.T. et al.**, (1994), Engineering & Technology, (30), No.4, 385-398.
- Heckel, R.W.**, (1961), Trans.AIME, (221), 671-675, 1001-1008.
- Hirschhorn, J.S.**, (1969), Int.J.of Powder.Metall., (5), No.1, 35-44.
- Huffine, C.L. and Bonilla, C.F.**, (1963), J. Am. Inst.

Chem. Engris, 8,490.

Hyungmoon, I. et al., (1985), Pow. Metall., (28), No.1, 21.

James, P.J., (1977), Powder Metall., (20), 199-204.

Jones, W.D., (1937), Principles of Powder Metallurgy, London.

Joslin, S.M. & Ashbee, K.H.G., (1995), Metallurgical and Materials Transactions A, (26A), 1087-1091.

Kamm, R. et al., (1947), Trans. AIME, (171), 439-453.

Kawakita, K. & Lüdde, K.H., (1970/71), Powder Technology, (4), 61-68.

Koerner, R.M., (1971), " Effects of Particle size and .." Modern Developments in P/M, by Hauser, H.H., (4), 115-125. Plenum press, New York.

Konopicky, K., (1948), Radex-Rundschau, 141-148.

Kostelnik, M.C., and et al., (1968), Int.J.Pow.Met., 4, (4), 19.

Lawrence, P., (1970), J.Materials Sci., (5), 663-668.

Leiser, D.P. & Whittemore, O., (1970), Bull.Am.Ceram.Soc., (49), No.8, 714.

Leopold, P.M. & Nelson, R.C., (1968), Perspectives in P/M., (3), 184-199, New York.

McEntire, B.J., (1991), "Dry Pressing", Engineered Materials Handbook (Ceramics and Glasses), Vol.4, 141-146, ASM International, ISBN 0-87170-282-7, Ohio, U.S.

Meeks, Henry S., (1984), "Cold Consolidation and Vacuum Sinter..", Modern Development in P/M., Vol.15, 237-250, U.S.

Meyer, R. et al., (1969), Pow.Metall., (12), No.24, 298-304.

Ohji, T & De Jonghe, L.J., (1994), J.Amer.Ceram.Soc., (77), No.6, 1685-1687.

Oudemans, G.J., (1965), Proc.Br.Ceram.Soc., Science of Ceramics 2, 133-146.

- Ramakrishnan, N & Sivakumar, K.,** (1990), Bull.Mater.Sci., (13), No.3, June, 217-225.
- Randall, M.German.,** (1994), Powder Metallurgy Science, 2nd Edition, 191-240, ISBN-1-878954-42-3, U.S.
- Rhines, F.N.,** (1946), Seminar on Sintering, Trans.AIME., Metals Tech., (166), 2043.
- Ridgway, K.,** (1992), "Compaction Science", Practical Aspects of Industrial Compaction, (Sym.Papers), No.4, 1.1-1.13, July 1992.
- Rietema, K.,** (1991), "The Dynamics of Fine Powders", 65-89, ISBN-1-85166-594-3, England.
- Roman, O.V. et al.,** (1980), Modern Developments in Powder Metallurgy, Vol.12, 325-337, ISBN-0-918404-51-7, New Jersey, U.S.
- Rong-de, D.,** (1991), The International Journal of Powder Metall., (27), No.3, July, 211-216.
- Sakuma, H. et al.,** (1992), Advances in P/M & Particulate Materials, (5), 327-336.
- Sano, Y.,** (1977), Int.J.P/M & Pow.Tech., (13), No.2, 81-April.
- Sarumi, M.A. & Al-Hassani, S.T.S.,** (1991), Powder Tech., (65), 51-59.
- Seelig, R.P.,** (1947), Institute of Metals Division, Trans. AIME, (171), 506-518.
- Seelig, R.P. & Wulff, J.,** (1946), "The Pressing Operation in the Fabrication ..", Trans.,AIME, (166), Metals Tech.
- Shapiro, I. & Kolthoff, I.M.,** (1947), J.Phys.Colloid.Chem., (51), 483-486.
- Siddiqui, M. & Hamiuddin, M.,** (1992), Powder Metallurgy International, (24), No.2, April, 79-83.
- Smith, D.W.,** (1983), "Elements of Powder Metallurgy", Powder Metallurgy, 7-38, ISBN-0-87170-154-5, Ohio.
- Smith, G.B.,** (1948), "Compressibility Factor", Met.Int., (72), 427.
- Smith, T.A.,** (1962), Trans.Br.Ceram.Soc., 61, 523.

Song, J.H. & Chandler, H.W., (1990), Br.Ceram.Trans.J., (89), 49-52.

Stanley-Wood, N.G., (1987), "Uniaxial Powder Compaction", Tribology In Particulate Technology (Book), Bristol.

Stepanov, G.V. & Zubov, V.I., (1990), Strength of Materials, (2296), June, 823-829.

Takahashi, M. & Suzuki, S., (1990), Ceramic Powder Science III, 473-484. California, U.S.

Thompson, R.A., (1981), "Mechanics of Powder Pressing I-III, Ame.Ceram.Soc.Bull., Vol.60, No.12, 237-245.

Tong, W. & Ravichandran, G., (1994), Appl.Phys.Lett., (65), No.22, 2783-2790.

Tunaboylu, B., et al., (1994), J.Ame.Ceram.Soc., (77), No.6, 1605-1612.

VaBen, R. & Stöver, D., (1992), Powder Technology, (72), 223-226.

Vonazountas, J. & Davies, T.J., (1992), Advances in P/M & Particulate Materials, Vol.2, 89-113, Publ:Metal Powder Industries Federation, New Jersey, USA.

Yarnton, D. and Davies, T.J., (1963), "Effect of Lubrication on the Compact...", Intl.J.of P/M and Powder Tech., (8), No.2, 51.

Wang, F.Y., (1976), Treatise on Materials Science and Technology, (9), Academic Press, New York.

Wulff, J., (1942), P/M.

TABLES

Table 1 : Powders used in this investigation.

No	Powder	Producer	Designation	Process
I	Iron	Höganäs	ASC 100.29	Atomized
II	Iron	Höganäs	NC 100.24	Atomized
III	Bronze	Makin Metal Pow.	Spherical bronze	Atomized
IV	Copper	Makin Metal Pow.	Spherical copper	Atomized

Table 2 : Iron powder specifications

Powder No	Apparent Density (gr/cm ³)	Tap Density (gr/cm ³)	Chemical Analysis (%)		
			H ₂ -loss	C	Fe
Iron I	2,88-3,03	3,30-4,05	0.15	0.01	BAL
Iron II	2.35-2.50	2.70-3.45	0.30	0.02	BAL

Table 3 : Atomized bronze (220 μm) and copper powders (15-55 μm) specifications.

Powder No	Apparent Den. (gr/cm^3)	Tap Den. (gr/cm^3)	Chemical Analysis (%)		
			Copper	Tin	Phosphorus
BronzeIII	4.5-5.4	4.9-5.7	BAL	9.73	0.03
Copper IV	4.7-5.8	5.1-6.0	99.58	-	0.05

Table 4 : Values of springback for the iron powders after compaction at different pressures

Powder No	Diameter of the compact after ejection. (mm)				
	200 MPa	350 MPa	550 MPa	700 MPa	850 MPa
Iron I	16.020	16.040	16.075	16.084	16.094
Iron II	16.020	16.036	16.070	16.080	16.090

FIGURES

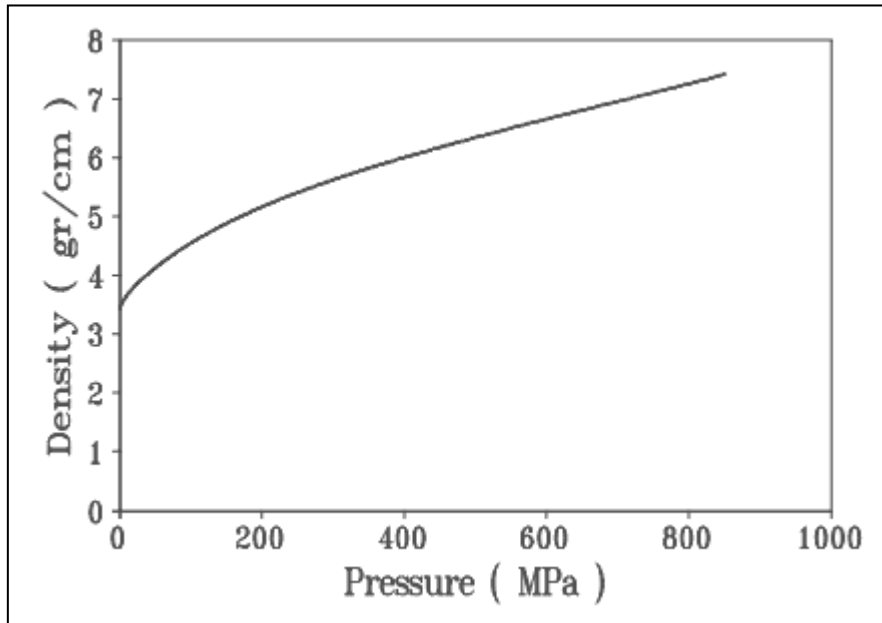


Figure 4.1.1 : Pressure - green density relationship of iron powder I, particle size 45-106 μm .

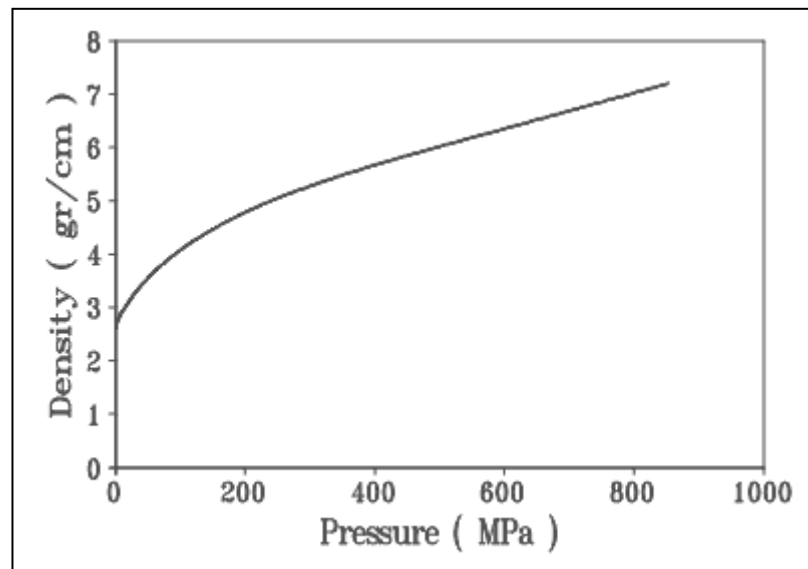


Figure 4.1.2 : Pressure - green density relationship of iron powder II, particle size 45-106 μm .

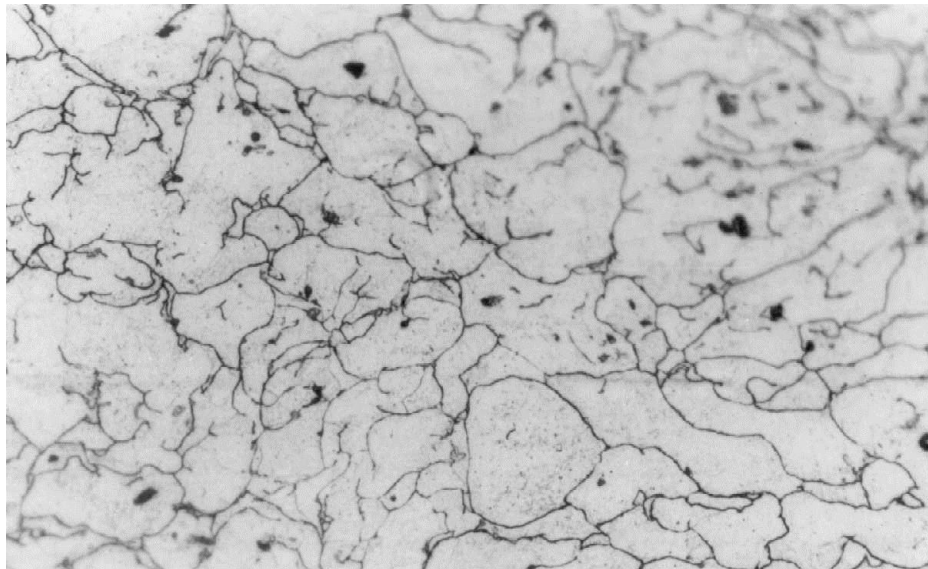


Figure 4.1.3 : Microstructure of iron powder II (45-106 μm) compacted at 500 MPa and etched with 2 % nital solution. 550x.

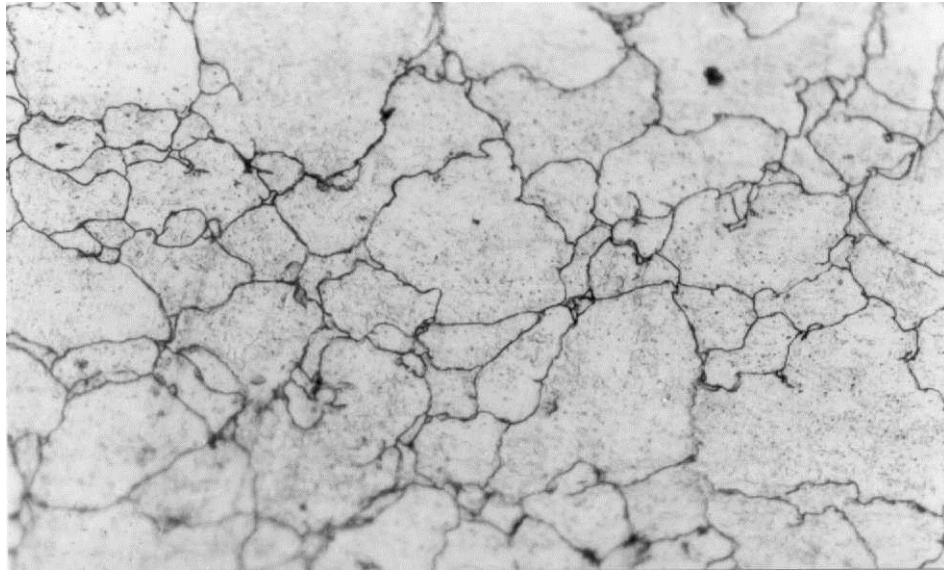


Figure 4.1.4 : Microstructure of iron powder I (45-106 μm) compacted at 500 MPa and etched with 2 % nital solution. 550x.

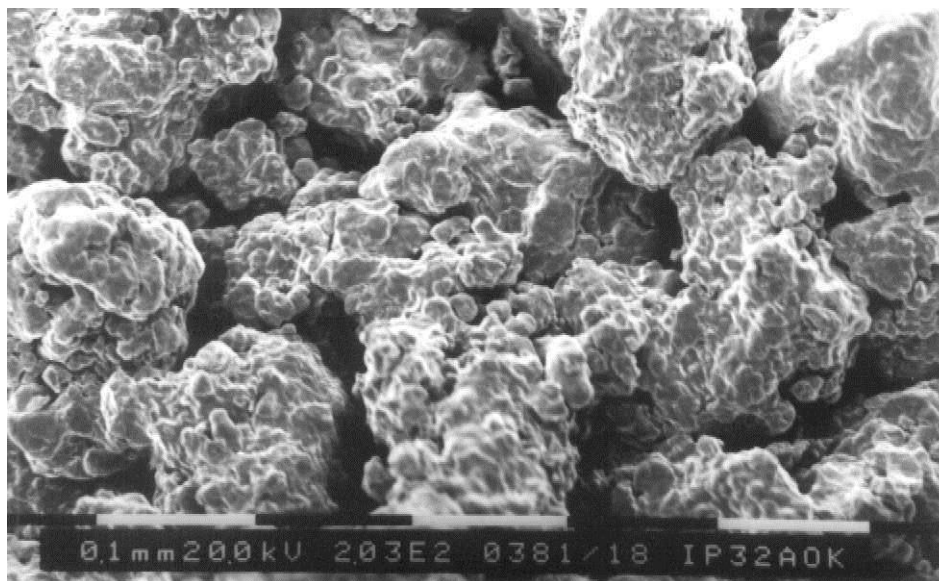


Figure 4.1.5 : Surface topograph of iron powder I (106-150 μm) compacted at 200 MPa, 200x.

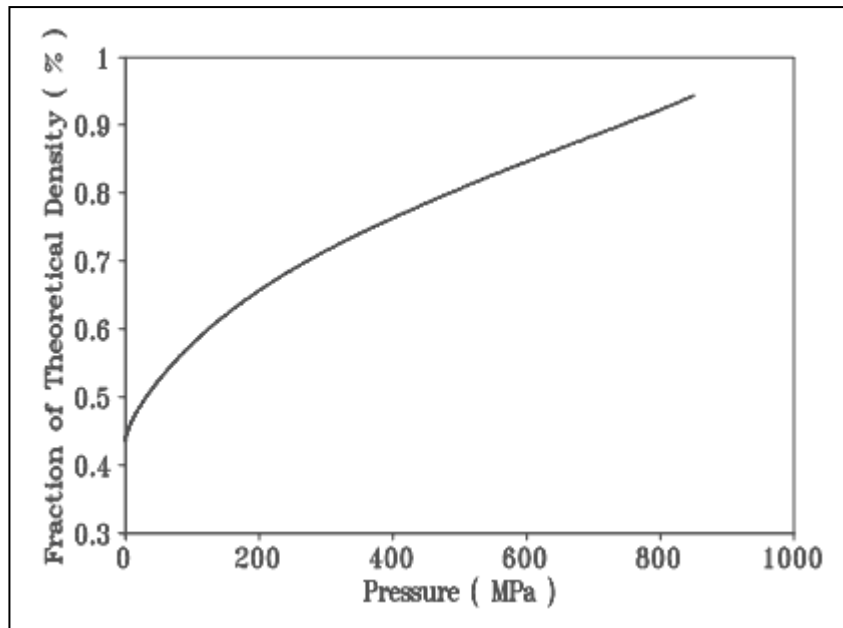


Figure 4.2.1 : The effect of pressure on the density of iron powder I, particle size 45-106 μm .

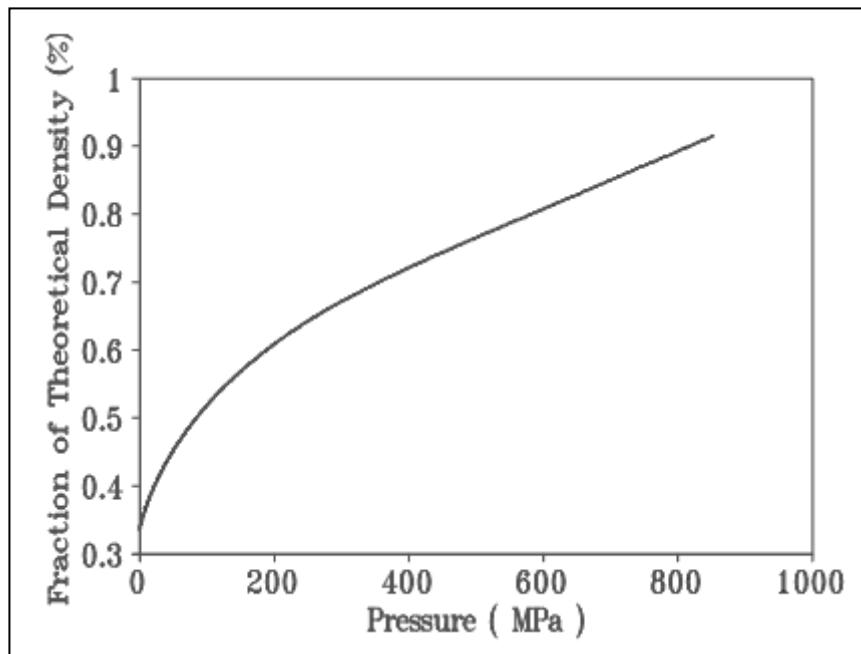


Figure 4.2.2 : The effect of the pressure on the density of iron powder II, particle size 45-106 μm .

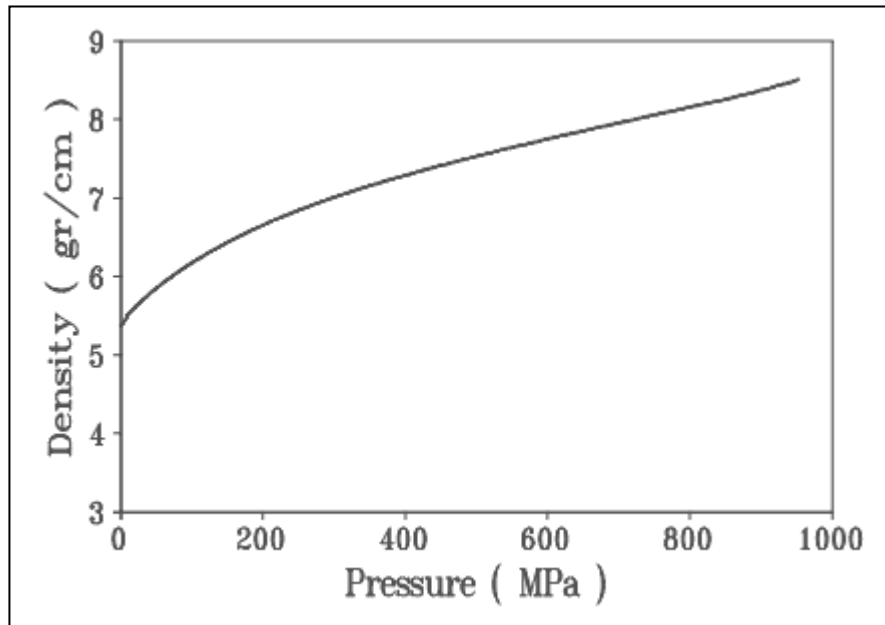


Figure 4.2.3 : The applied pressure - green density relationship of spherical copper powder, (15-55 μm).

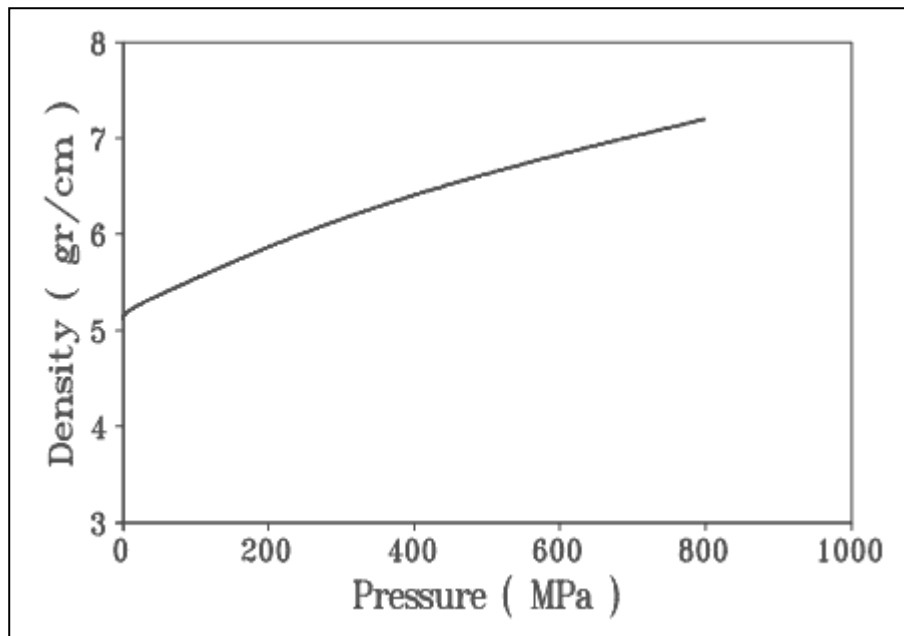


Figure 4.2.4 : The relationship between applied pressure and green density of spherical bronze powder, (220 μm).

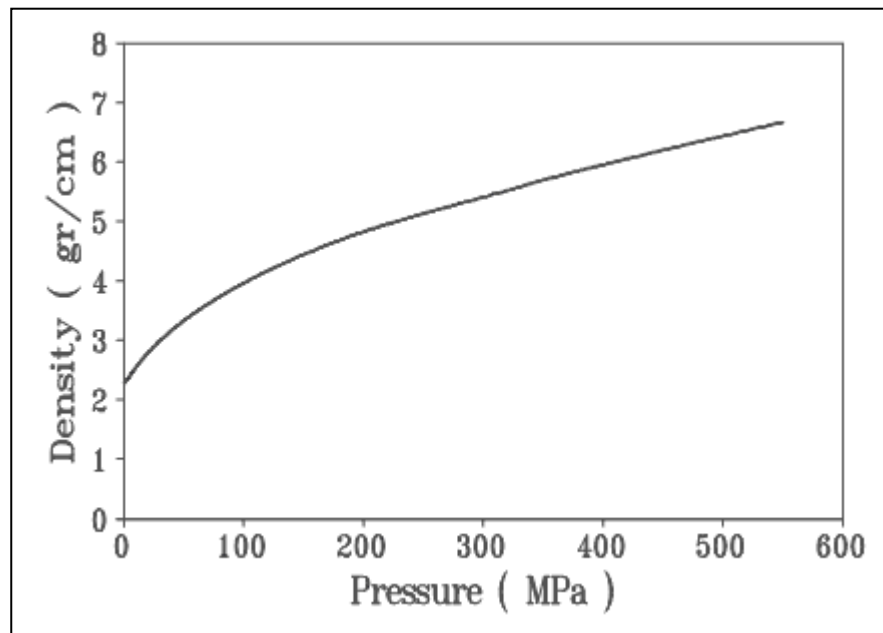


Figure 4.3.1 : The correlation between pressure and green density of 106-150 μm size fraction of iron powder II.

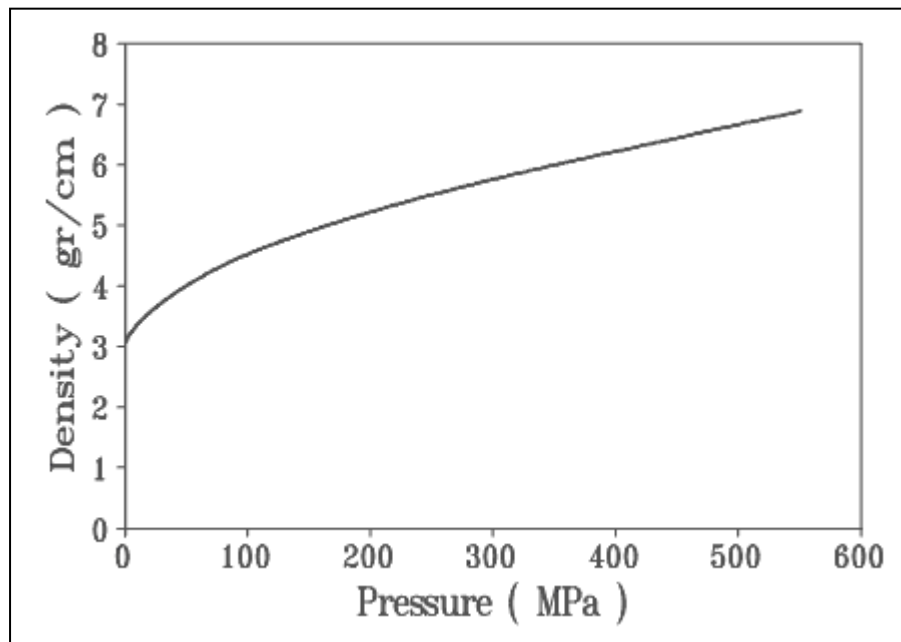


Figure 4.3.2 : The applied pressure - green density relationship of 106-150 μm size fraction of iron powder I.

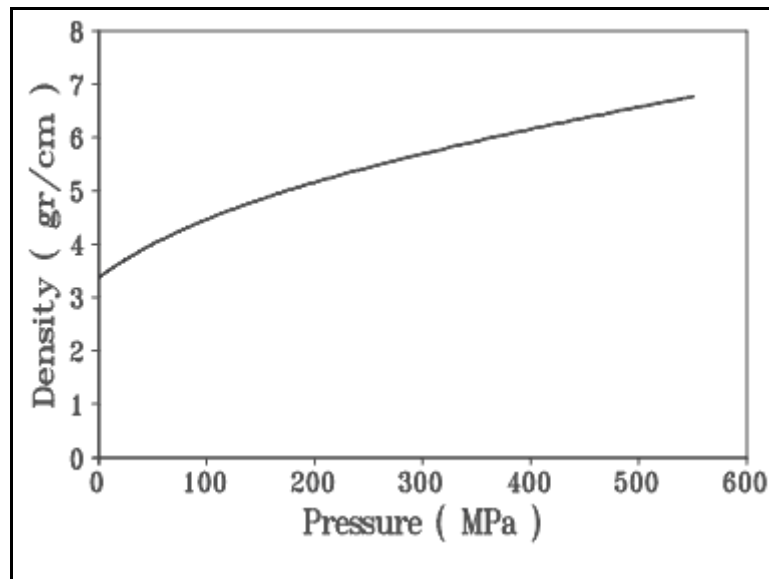


Figure 4.3.3 : The relationship between pressure and green density of -45 μm size fraction of iron powder

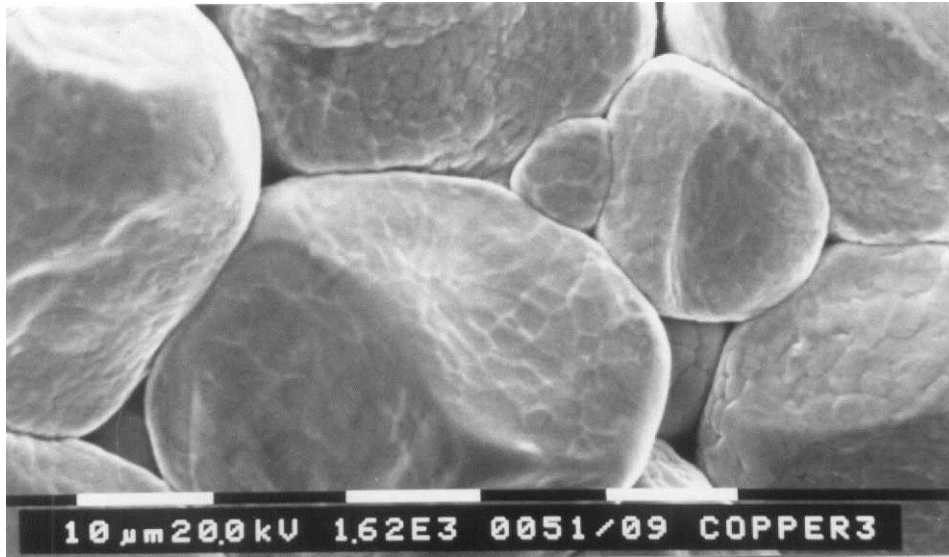


Figure 4.3.4 : SEM showing area contacts in copper powder (15-55 μm) compacted at 800 MPa, 1620x.

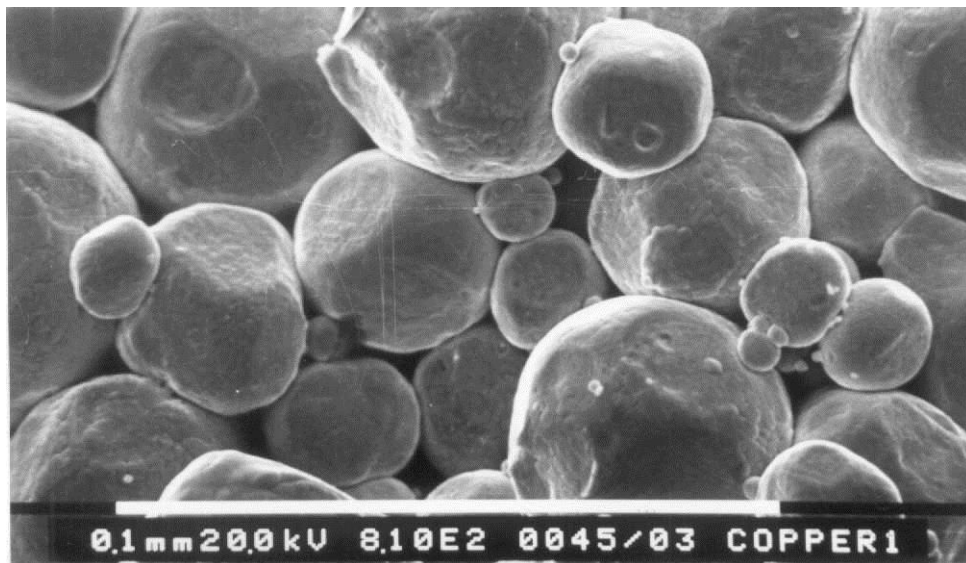


Figure 4.3.5 : SEM micrograph showing the packing characteristics of copper powder (15-55 μm) compacted at 400 MPa, 810x.

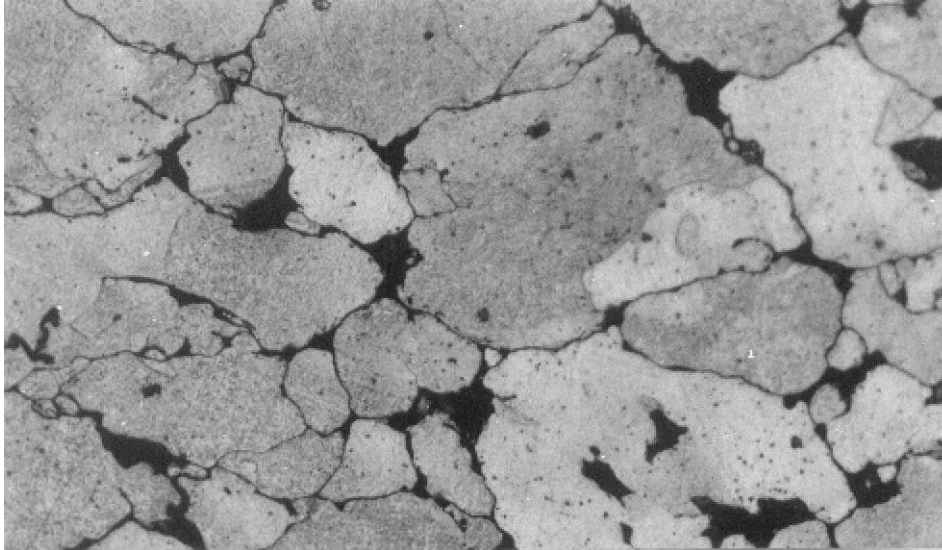


Figure 4.3.6 : Microstructure of iron powder I (45-106 μm) compacted at 450 MPa and etched with 2 % nital solution. 500x.

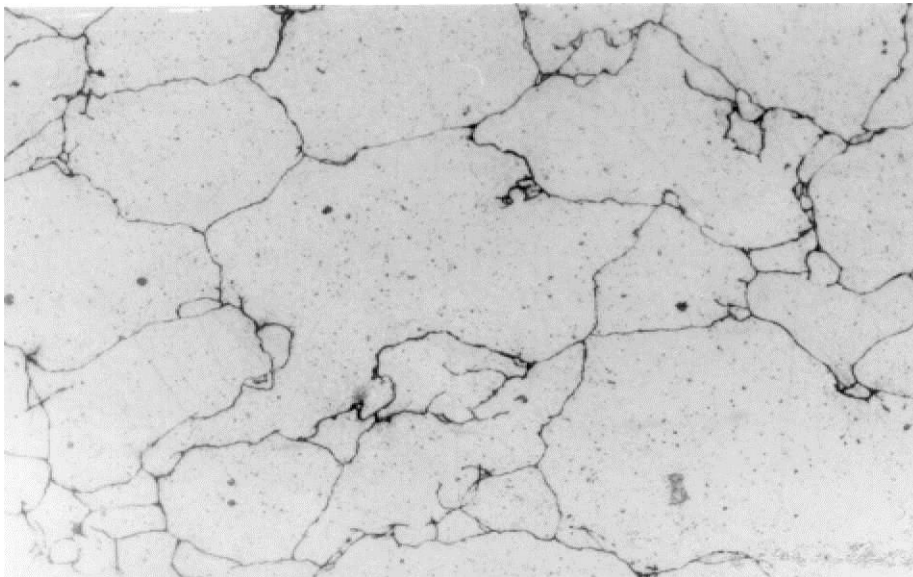
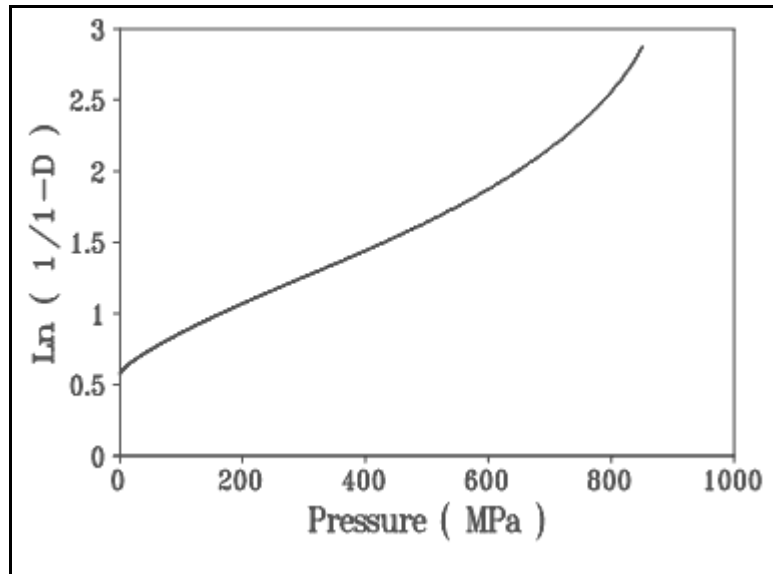
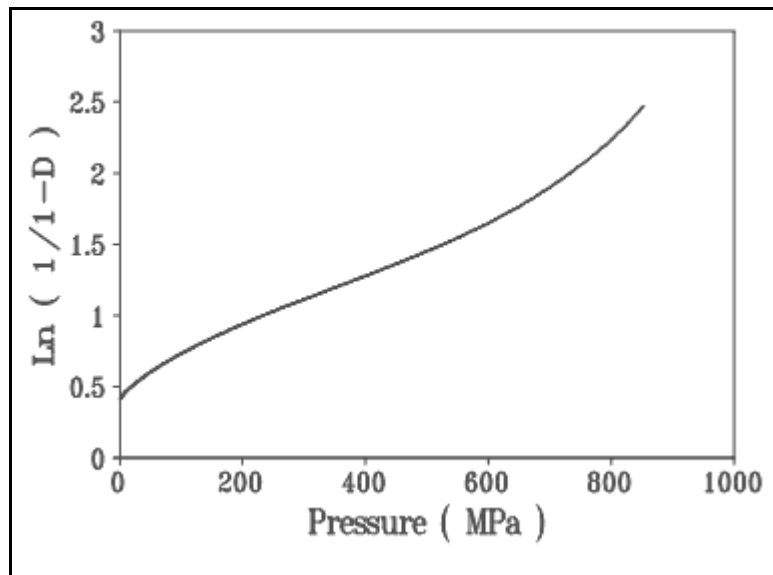


Figure 4.3.7: Microstructure of iron powder I (45-106 μm) compacted at 850 MPa and etched with 2 % nital solution. 550x.

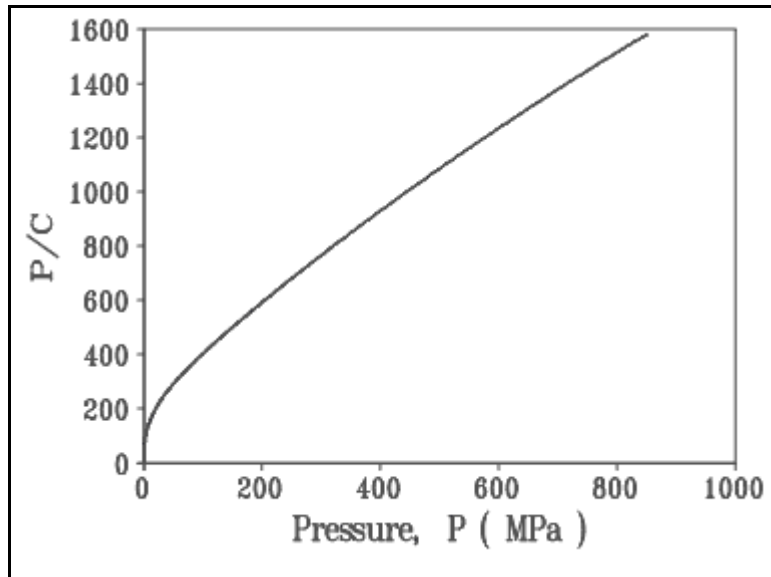


(a)

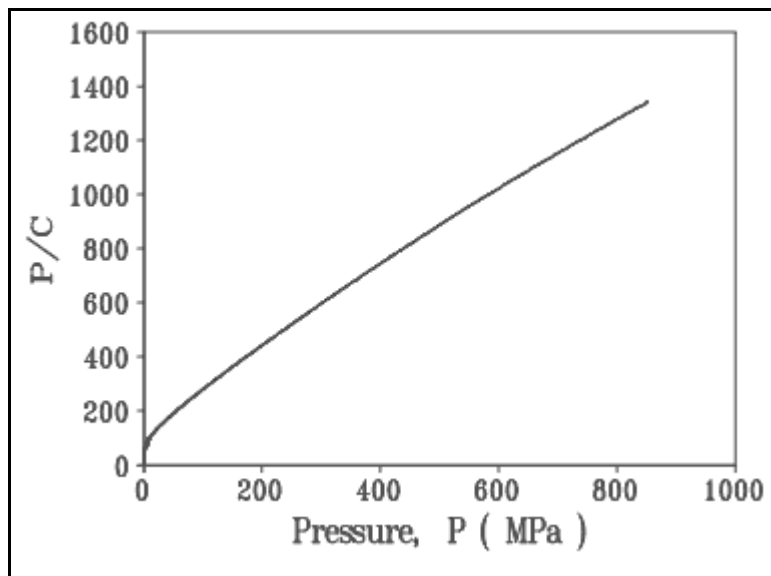


(b)

Figure 4.3.8 :The representation of the Heckel Diagram with the data for (a) iron powder I and (b) iron powder II (45-106 μm).



(a)



(b)

Figure 4.3.9 : Illustration of the Kawakita diagram with data of 45-106 μm size fraction of (a) iron powder I and (b) iron powder II.

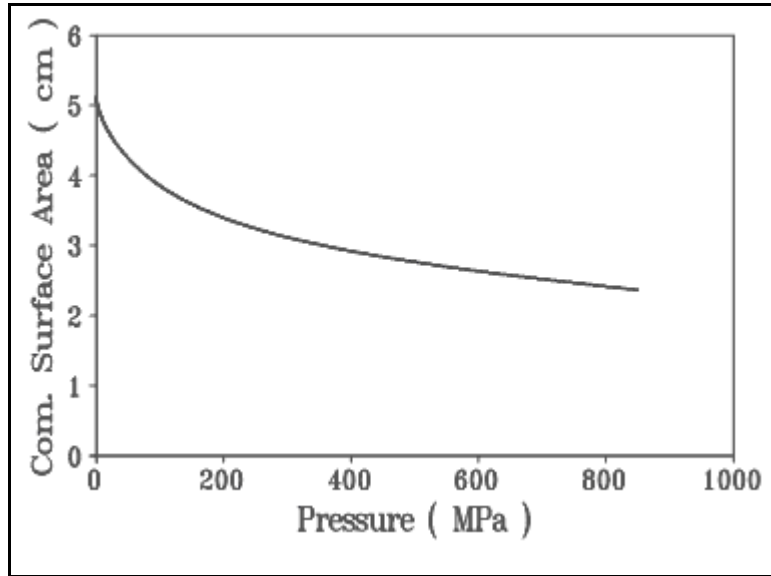


Figure 4.4.1 : The relationship between pressure and compact surface area contact with the die wall for 45-106 μm size fraction of iron powder I.

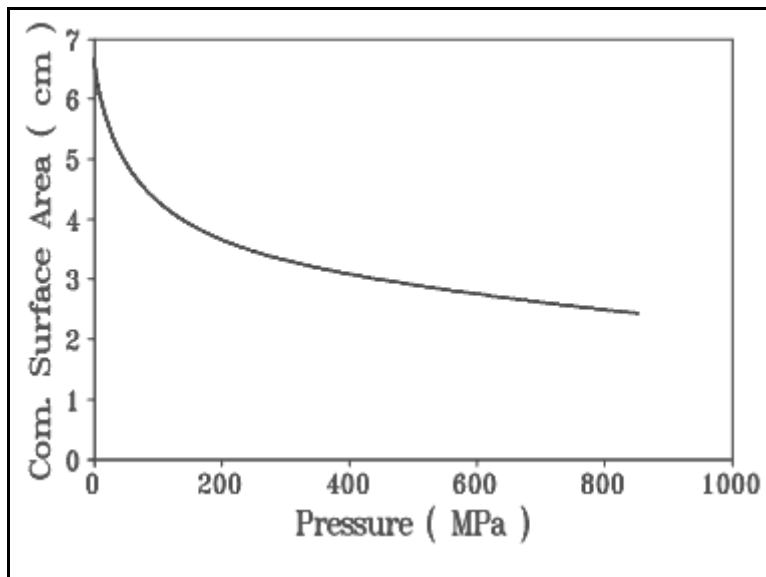


Figure 4.4.2 : The variation of the compact surface area contact with the die wall under applied pressure for 45-106 μm size fraction of iron powder II.

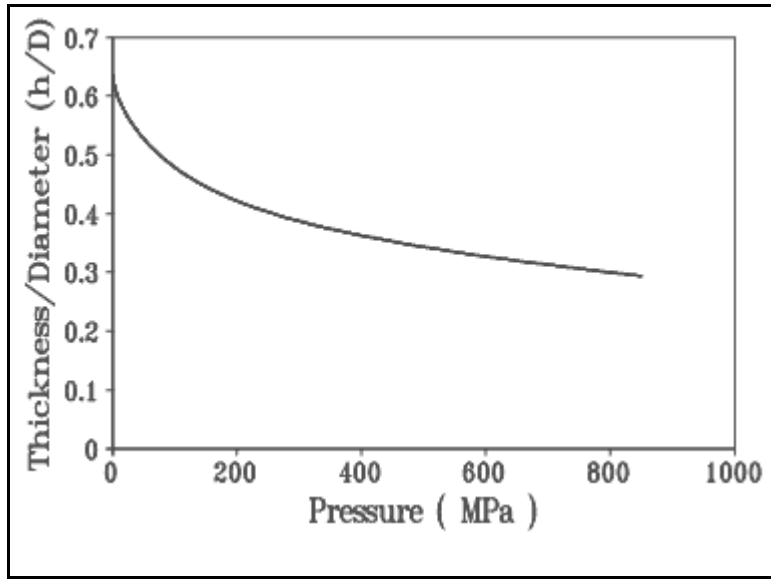


Figure 4.4.3 : The change of compact thickness/diameter (h/D) ratio under applied pressure for 45-106 μm size fraction of iron powder I.

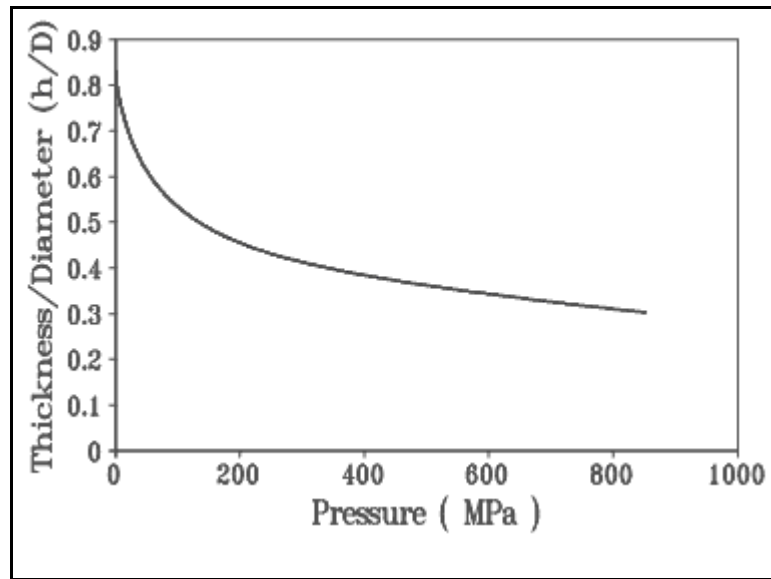


Figure 4.4.4 : The change of compact thickness/diameter (h/D) ratio under applied pressure for 45-106 μm size fraction of iron powder II.

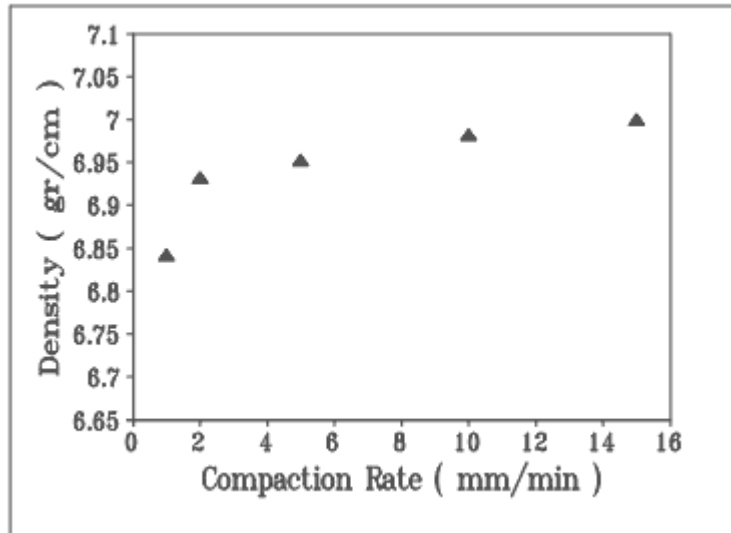


Figure 4.5.1 : The influence of compaction rate on the green density of iron powder I having size fraction of 45-106 μ m at constant pressure of 550 MPa.

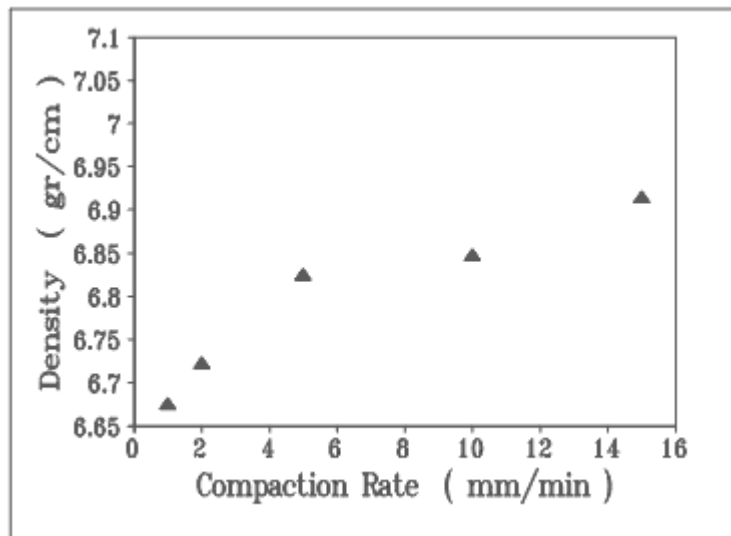


Figure 4.5.2 : The compaction rate effect on the green density of iron powder II having size fraction of 45-106 μ m at constant pressure of 550 MPa.

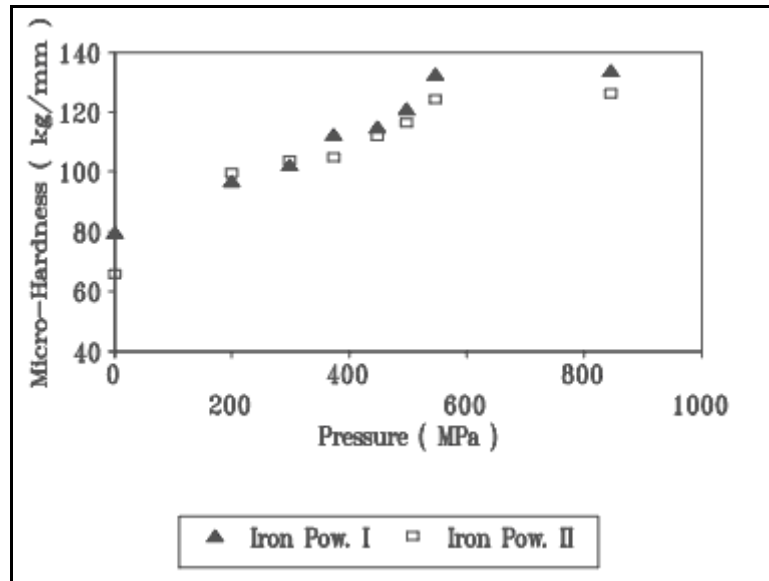


Figure 4.6.1 : Pressure - microhardness relationship of iron powder I and iron powder II having the same size fraction of 45-106 μm , compacted under identical conditions.

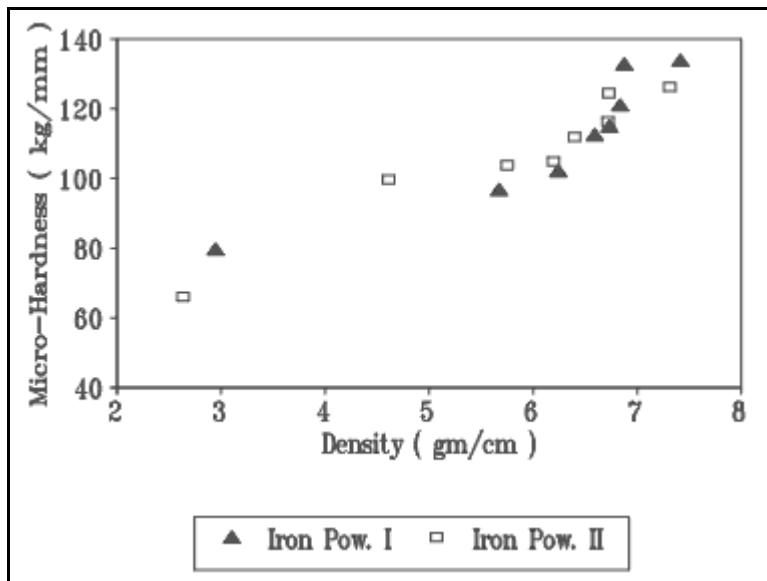


Figure 4.6.2 : Green density - microhardness relationship of iron powder I and iron powder II having the same size fraction of 45-106 μm , compacted under identical conditions (compacting pressure range 0-850 MPa).

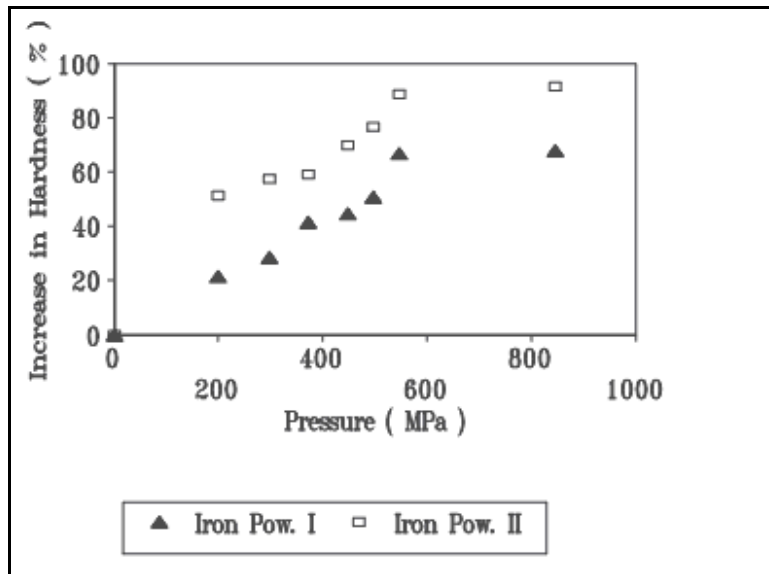


Figure 4.6.3 : Showing the increase in micro hardness of iron powder I and iron powder II, size fraction 45-106 μm , as a function of compacting pressure.

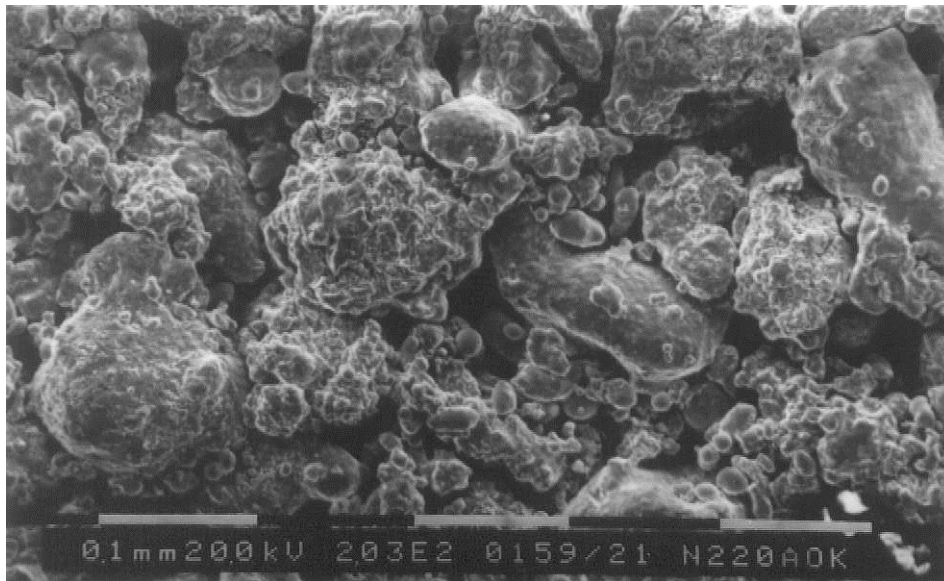


Figure 4.6.5 : SEM micrograph of surface and packing characteristics of iron powder I compacted at 250 MPa, 200x.

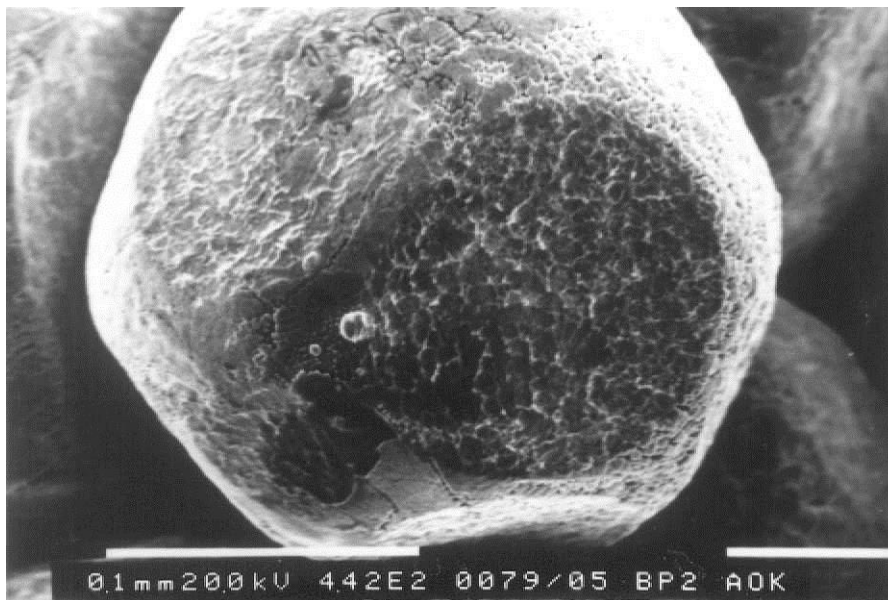


Figure 4.6.6 : SEM micrographs of particle shape of bronze powder (220 μm) after compacted at 800 MPa, 440x.



Figure 4.6.7 : SEM micrograph of surface characteristics of bronze powder (220 μm) compacted at 800 MPa, 250x.

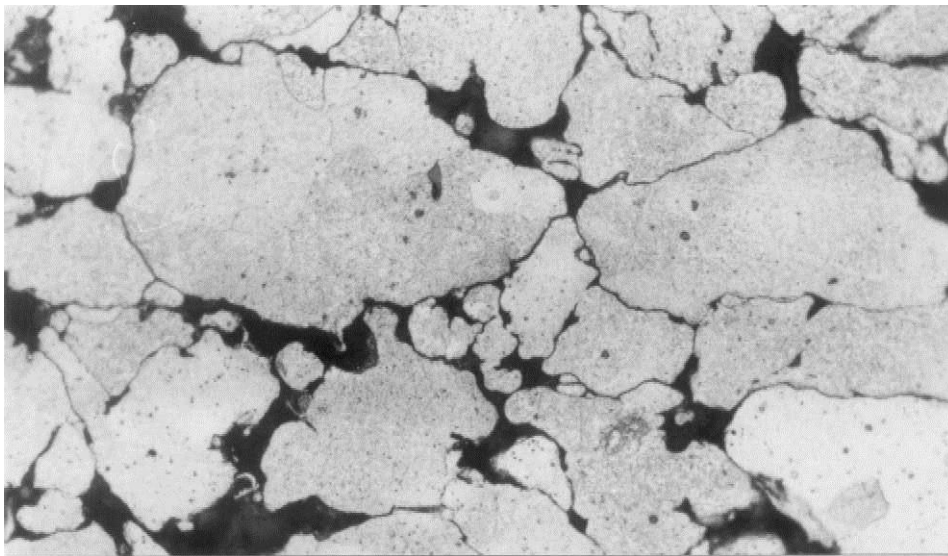


Figure 4.6.8 : Microstructure of iron powder I (45-106 μm) compacted at 450 MPa and etched with 2 % nital solution. 500x.

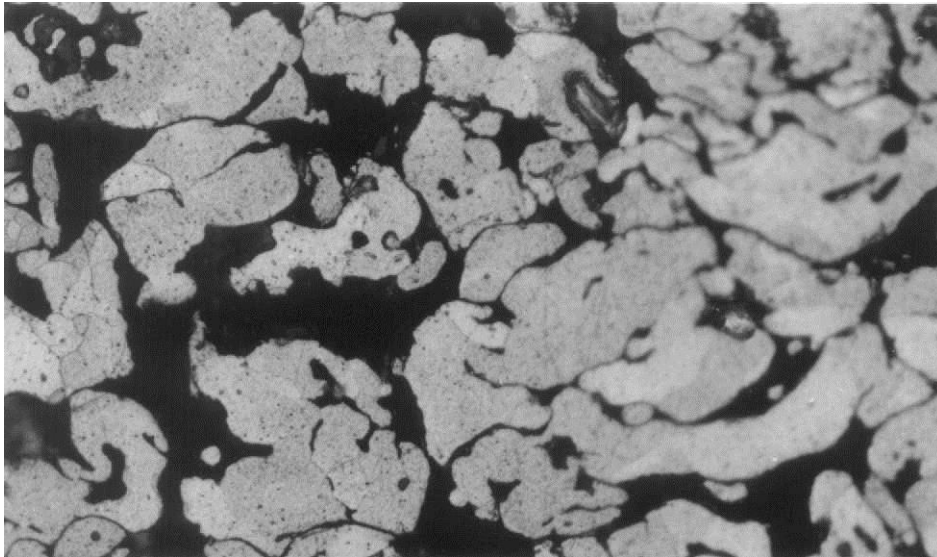


Figure 4.6.9 : Microstructure of iron powder II (45-106 μm) compacted at 300 MPa and etched with 2 % nital solution. 500x.

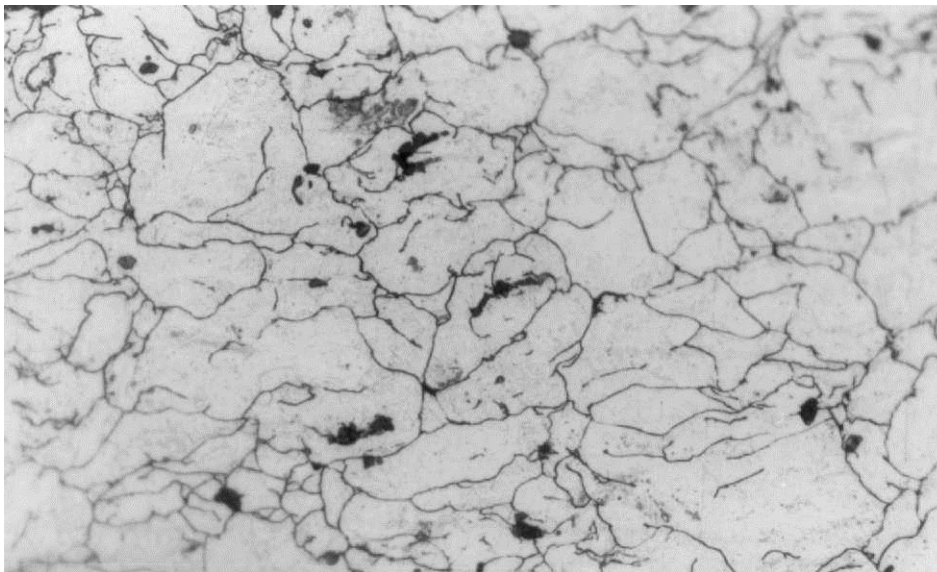


Figure 4.6.10 : Microstructure of iron powder II (45-106 μm) compacted at 500 MPa and etched with 2 % nital solution. 550x.