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ÇUKUROVA UNIVERSITY
INSTITUTE OF NATURAL AND APPLIED SCIENCES

MSc THESIS

Ali Hakan YÜZBAŞIOĞLU

SINTERING OF COLD ISOSTATICALLY PRESSED METAL POWDERS



DEPARTMENT OF MECHANICAL ENGINEERING

ADANA, 2004

151 436

ÇUKUROVA ÜNİVERSİTESİ
FEN BİLİMLERİ ENSTİTÜSÜ

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YÜKSEK LİSANS TEZİ

MAKİNA MÜHENDİSLİĞİ ANABİLİM DALI

Bu tez 16/12/2004 Tarihinde Aşağıdaki Jüri Üyeleri Tarafından Oybirliği/Oyçokluğu İle Kabul Edilmiştir.

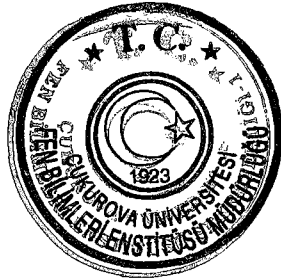
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Kod No: 2494



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Bu çalışma Çukurova Üniversitesi Araştırma Projeleri Birimi Tarafından Desteklenmiştir. FBE 2002YL375

Not: Bu tezde kullanılan özgün ve başka kaynaktan yapılan bildirişlerin, çizelgele, şekil ve fotoğrafların kaynak gösterilmeden kullanımı, 5846 sayılı Fikir ve Sanat Eserleri Kanunundaki hükümlere tabidir.

ABSTRACT

MSc THESIS

SINTERING OF COLD ISOSTATICALLY PRESSED METAL POWDERS

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DEPARTMENT OF MECHANICAL ENGINEERING
INSTITUTE OF NATURAL AND APPLIED SCIENCES
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Year: 2004, Pages: 84
Jury : Assoc. Prof.Dr. Abdulkadir EKŞİ
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Al and Fe metal powders have been chosen for study due to widely used in the industrial applications. Powders are pressed until 600 MPa pressure respectively, and sintered. In order to observe, effects of pressing and sintering operation to density, densities have been measured in per stages, for metallographic researches Optic Microscope and Scanning Electron Microscope (SEM) test machines are used. Keywords: Cold Isocratic Pressing, Density Measurement, Metallurgical Research, Light Microscope, Scanning Electron Microscope.

ÖZ

YÜKSEK LİSANS TEZİ

SOĞUK İZOZTATİK OLARAK PRESLENMİŞ METAL TOZLARININ
SİNTERLENMESİ

Ali Hakan YÜZBAŞIOĞLU

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MAKİNA MÜHENDİSLİĞİ ANABİLİM DALI

Danışman : Doç.Dr. Abdulkadir EKŞİ

Yıl: 2004, Sayfa: 84

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Bu çalışma için endüstriyel uygulamalarda yaygın olarak kullanılan Al ve Fe tozları seçilmiştir. Tozlar göreceli olarak 600 MPa basınca kadar sıkıştırılmıştır. Sıkıştırma ve sinterleme işlemlerinin yoğunluğa etkilerinin gözlemlenmesi amacıyla yoğunluklar her aşamada ölçülmüş, metalografik inceleme amacıyla Optik Mikroskop ve Taramalı Elektron Mikroskobu kullanılmıştır.

Anahtar Kelimeler: Soğuk İzostatik Presleme, Yoğunluk Ölçümü, Metalurjik İnceleme, Işık Mikroskobu, Taramalı Elektron Mikroskobu

ACKNOWLEDGEMENTS

I am so grateful to my supervisor Assoc. Prof.Dr. Abdulkadir EKŞİ for his guidance, inspiration and encouragement during all my thesis studies.

I also thank my special committee members; my supervisor Assoc. Prof.Dr. Abdulkadir EKŞİ, Prof.Dr. Melih BAYRAMOĞLU, and Assoc. Prof.Dr.Cengiz Duran ATIŞ and for their enthusiasms and many helpful suggestions.

I would like to thank to all my friends for his endless support: Research Asistant Durmuş Ali BİRCAN and Harun UÇAK in Çukurova University, and also thanks to Volkan ARIKAN in Gazi University, Beytullah ONAT, Murat ÇAKIRCA, İlteriş SARIGEÇİLİ, and Ela KÖSE

I have to thank to Assoc. Prof.Dr. Halil ARIK in Gazi University for his special attention during my laboratory works. He has let me use all possibilities of Gazi University Laboratories for microstructure analysis and sintering operations.

Finally, special thanks to my family for their endless supports, and my supervisors at Antalya Airport for their helps to study my work even in the job.

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NOMENCLATURE

Fe	: Ferrius Metal
Al	: Aluminum Metal
γ_{sv}	: Solid state Density
R	: Small Sphere of Radius
σ	: Stress
x	: Neck Dimension
x	: Weight of specimen
y	: Carrier material weight
z	: Weight of specimen in the water
b	: Difference of weight
a	: Weight without carrier bond
d	: Density
V_p	: Volume of the pressed powder
ΔV	: Volume change
W	: Weight of the powder
D_p	: Mass density
D_t	: Percent theoretical density
D	: Picnometric density

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1. INTRODUCTION

Powder Metallurgy may best be defined by comparing it with “Fusion Metallurgy”. In fusion metallurgy a metal or alloy is melted and cast into a mold. The mold may have the shape of the desired product, in which case a casting is produced, or the shape of an ingot may be cast. The ingot is then formed into a wrought product by rolling, forging, extrusion, drawing, machining, etc. In powder metallurgy, metal powders, i.e. metals in finely divided form rather than molten metal, are the starting material. The powders are consolidated into products with a given shape. The basic steps in powder metallurgy are therefore powder production and powder consolidation.

The most common sequence in powder consolidation includes pressing the powder in a die into a compact and sintering the compact which means heating it to a temperature below the melting point of the metal or alloy to give it the desired physical, chemical and mechanical properties. As in fusion metallurgy the consolidation may lead to a part with the desired shape corresponding to a casting or to a compact corresponding to an ingot which must be further shaped by forming.

Metal powders are employed for many other purposes besides consolidation into shapes. They are used in paints, varnishes and printing inks, and reagents in chemical industry, as explosives particularly in military applications and as food additives. In metal working they are employed in cutting and cleaning and producing metallic coatings, as e.g. for welding electrodes. None of these uses of metal powders are part of powder metallurgy.

Why would one want to produce a metal product with a given shape starting with metal powders rather than molten metal? One important reason has an economic basis. The cost of producing a part of a given shape to a required dimensional tolerance by powder metallurgy may be lower than by casting or as a wrought product. The powder metallurgy product, often called a “P/M structural part” must, of course, have adequate properties, usually adequate mechanical properties, for the application. But its cost rather than any unusual properties is the main reason for fabricating it from powder.

The market for structural parts by powder metallurgy has greatly expanded in the last 30 years. Structural parts are the largest field of application for powder metallurgy products, not only as to the amount of powder consumed, but also all volume. Parts from iron and low alloy steel constitute the bulk of structural parts production, but parts are also produced from copper, bronze, brass, nickel, silver, stainless steel, aluminum and titanium alloys.

Production of structural parts by powder metallurgy has important advantages compared with competing methods. It conserves material because parts do not need subsequent machining. Parts are produced on automatic compacting pressed and sintered in continuous furnaces, so that the powder metallurgy approach saves labor and insures high productivity. Fabrication by powder metallurgy requires less energy than other methods and is ecologically clean.

It has long been known empirically and discussed theoretically in powder mechanics that if we fill a cylindrical or other shaped mold with a powder and than compress it in order to mold a high density body, the body that results is not uniform in density. With the coming of the idea that a uniformly dense body might result if the powder were compressed with equal force in all directions, isostatic pressing was born.

Applying pressure with a fluid makes possible a technique for compressing the powder equally in all directions. Pascal` s principle states that if we increase the pressure at one point in a stationary fluid that is completely enclosed in a vessel, then the pressure at all points within the fluid will increase by exactly the same amount. Therefore, by filling a soft envelope with powder and compressing it with a surrounding fluid, we can make a uniformly compacted molded body. Density distribution is nearly perfect in this method. And also economy and high pressure capacity are the other important reasons for Cold Isostatic Pressing.

1.1. Powder Metallurgy Process

In the powder metallurgy generally, metal powders and additives mixing, compacting, sintering and according to the conditions optional machining applied. These basic steps showed in the Figure 1.1.

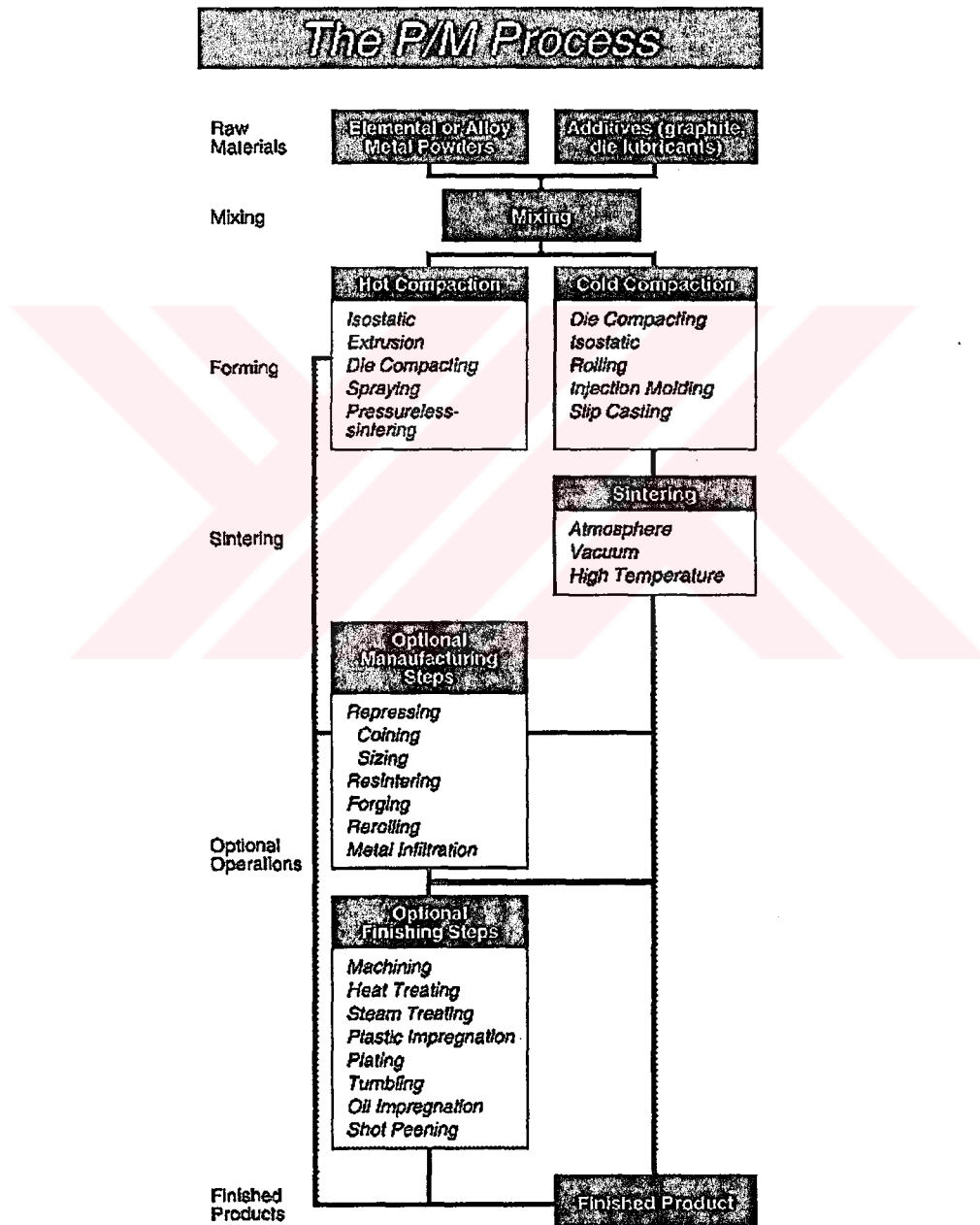


Figure 1.1. Powder Metallurgy Process Scheme

1.2. Powder Production

In order to produce metal powders, generally 5 different techniques widely used. General characteristics of these methods explained at the bottom.

1.2.1. Solid State Reduction Technique

This has been for long the most widely used method for the production of iron powder. Selected ore is crushed, mixed with carbon, and passed through a continuous furnace where reaction takes place leaving a cake of sponge iron which is then further treated by crushing, separation of non-metallic material, and sieving to produce powder.

Since no refining operation is involved, the purity of the powder is dependent on that of the raw materials. The irregular sponge-like particles are soft, and readily compressible, and give compacts of good green strength.

Refractory metals are normally made by hydrogen reduction of oxides, and the same process can be used for copper.

1.2.2. Electrolysis Technique

By choosing suitable conditions - composition and strength of the electrolyte, temperature, current density, etc., many metals can be deposited in a spongy or powdery state.

Extensive further processing - washing, drying, reducing, annealing and crushing may be required.

Copper is the main metal to be produced in this way but chromium and manganese powders are also produced, by electrolysis. In these cases, however, a dense and normally brittle deposit is formed and requires to be crushed to powder. Electrolytic iron was at one time produced on a substantial scale but it has been largely superseded by powders made by less costly processes. Very high purity and high density are two distinguishing features (Lawley A.,1978).

1.2.3. Atomization Technique

In this process molten metal is broken up into small droplets and rapidly frozen before the drops come into contact with each other or with a solid surface. The principal method is to disintegrate a thin stream of molten metal by subjecting it to the impact of high energy jets of gas or liquid. Air, nitrogen and argon are commonly used gases, and water is the liquid most widely used. The principal method is to disintegrate a thin stream of molten metal by subjecting it to the impact of high energy jets of gas or liquid.

A commercial scale plant was set up in Japan to produce iron powder using paraffin as the atomizing liquid the object being to keep the surface oxygen content as low as possible. The process was technically successful, but the advantages did not justify, in commercial terms, the extra cost involved. However, interest has not entirely evaporated and work is going on elsewhere. By varying the several parameters: design and configurations of the jets, pressure and volume of the atomizing fluid, thickness of the stream of metal etc. It is possible to control the particle size distribution over a wide range.

The particle shape is determined largely by the rate of solidification and varies from spherical, if a low heat capacity gas is employed, to highly irregular if water is used. In principle the technique is applicable to all metals that can be melted, and is commercially used for the production of iron, copper, including tool steels, alloy steels, brass, bronze and the low-melting-point metals, such as aluminium, tin, lead, zinc, and cadmium. The readily oxidisable metals, for example chromium-bearing alloys, are being atomized on an increasing scale by means of inert gas, especially argon. In addition, there are several other processes that are finding increasing application, an important one being centrifugal atomization in which droplets of molten metal are discharged from a rotating source (KUHNS H.A.,1978)

There are basically two types of centrifugal atomization processes:

- In one a cup of molten metal is rotated on a vertical axis at a speed sufficient to throw off droplets of molten metal, or a stream of metal is allowed to fall on a rotating disc or cone;

- In the other a bar of the metal is rotated at high speed and the free end is progressively melted e.g. by an electron beam or plasma arc.

This latter process is called the Rotating Electrode Process (REP), and the bar may be rotated either on a horizontal or on a vertical axis.

A special advantage of these processes is that they can be carried out in a sealed vessel in a controlled atmosphere - even vacuum - and thus produce 'clean' powders of highly reactive metals. Differences between gas atomization and water atomization can be seen in the Figure 1.2 and Figure 1.3 at the bottom.

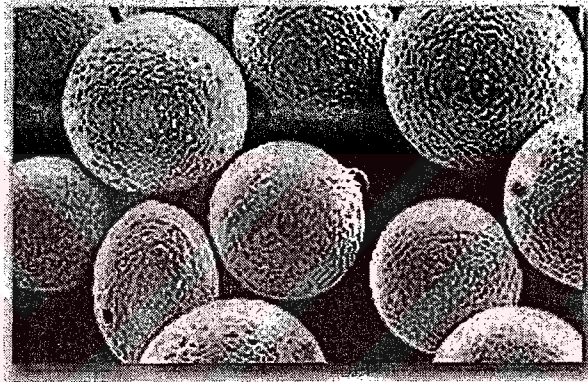


Figure 1.2. Shape of gas atomization powders

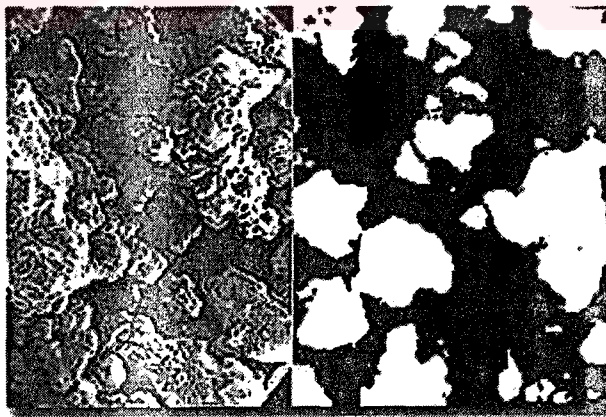


Figure 1.3. Shapes of water atomization powders

With the REP process the avoidance of contact with refractory is a potent means of reducing the number of non-metallic inclusions in the powder, and in components manufactured from the powder.

Atomization is particularly useful for the production of alloys in powder form, since the constituents metals are fully alloyed in the molten state. Thus each powder particle has the same chemical composition. Additionally the process is used to produce compositions such as copper-lead, in which the lead, though soluble in the liquid state, comes out of solution on solidification. If a casting of such an alloy is made, serious segregation of the lead results, but if the liquid is atomized, the end product is copper powder containing a very fine and uniform distribution of lead inclusions within each powder particle (KUH N H.A.,1978).

1.2.4 Mechanical Process for Powder Production

Brittle materials such as inter-metallic compounds, ferro-alloys, ferro-chromium, and ferro-silicon, etc. are pulverized mechanically in ball mills, and a process known as the Cold stream Process is finding increasing application for the production of very fine powders such as are required for injection molding (qv). In this process, granular material, this may be coarsely atomized powder, is fed in a stream of gas under pressure through a venturi and is cooled and thereby embrittled by the adiabatic expansion of the gas before impinging on a target on which the granules shatters (KUH N H.A.,1978).

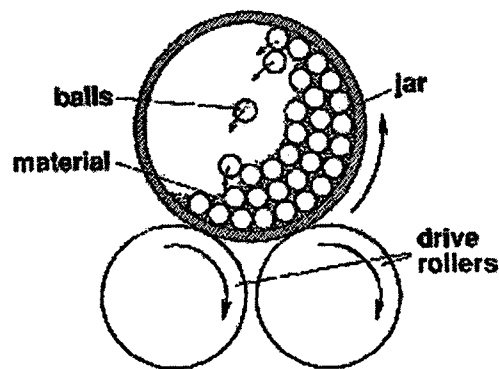


Figure 1.4. Basic Mechanical Process

1.2.5 Chemical Processes for Powder Production

Thermal decomposition of a chemical compound is used in some cases, a notable one being nickel carbonyl. This Carbonyl Process was originally developed as a means of refining nickel, crude metal being caused selectively to react with carbon monoxide under pressure to form the carbonyl which is gaseous at the reaction temperature and which decomposes on raising the temperature and lowering the pressure. The same process is used for iron, and carbonyl iron powder finds small scale application where its very high purity is useful. Recently, demand for very fine powders for the injection molding process has given a considerable impetus to the carbonyl process. Typically the particle size of carbonyl iron powder is 1-5 μ m, but, as in the case of nickel, it can be tailored to suit particular requirements. Another case of thermal decomposition is platinum powder of which is made from sponge produced by heating salt - platinum ammonium chloride. In the Sherritt-Gordon process, nickel powder is made by hydrogen reduction of a solution of a nickel salt under pressure (Gummesson P.U., 1972).

Chemical precipitation of metal from a solution of a soluble salt is used in other cases e.g. silver, powder of which is produced by adding a reducing agent to a solution of silver nitrate. This is, of course, the same basic process as is used to produce black and white photographs.

1.3. Powder Characteristics

The further processing and the final results achieved in the sintered part are influenced by the characteristics of the powder as explained at the bottom:

- Particle size,
- And size distribution,
- Particle shape,
- Structure
- And surface condition.

A very important parameter is the Apparent Density (AD) of the powder, i.e. the mass of a given volume, since this strongly influences the strength of the compact obtained on pressing. The AD is a function of particle shape and the degree of porosity of the particles.

i) The choice of powder characteristics is normally based on compromise, since many of the factors are in direct opposition to each other:

An increase in the irregularity and porous texture of the powder grain, i.e. decrease in apparent density, increases the reduction in volume that occurs on pressing and thus the degree of cold-welding, which, in turn, gives greater green strength to the compact. This increase in contacting surfaces also leads to more efficient sintering.

The same process is used for iron, and carbonyl iron powder finds small scale application where its very high purity is useful. Recently demand for very fine powders for the injection molding process has given a considerable impetus to the carbonyl process. Typically the particle size of carbonyl iron powder is 1-5 μ m, but, as in the case of nickel, it can be tailored to suit particular requirements. Additionally the greater reduction in volume necessary to give the required green density may require greater pressure and consequently larger presses and stronger dies. The ease and efficiency of packing the powder in the die depends to a large extent on a wide particle size distribution.

So that the voids created between large particles can be progressively filled with those of smaller size. Fine particle sizes tend to leave smaller pores which are easily closed during sintering. An excess of fines, however, reduces flow properties with the results already detailed above.

ii) The purity of the powder is critically important. Impurity levels which can be tolerated depend to a large extent on the nature and state of combination of the substances concerned.

For example, the presence of combined carbon in iron tends to harden the matrix so that increased pressures are required during compaction. Free carbon, however, is often an advantage, acting as a lubricant during the pressing operation. Most metal powder grains are coated by a thin oxide film, but in general these do not

interfere with the process, since they are ruptured during the pressing operation to provide clean and active metal surfaces which are easily cold-welded. Their final reduction under the controlled sintering atmosphere is essential for complete metal bonding and maximum strength. Stable oxide films or included oxide particles, such as SiO_2 and Al_2O_3 are more serious, since these are generally abrasive and lead to increased tool wear (Kouzumi M., Nishihara M, 1992).

Furthermore they cannot be reduced during subsequent sintering and their presence may adversely affect the mechanical properties especially impact strength of the finished part.

1.4. Mixing of Metal Powders

Unless a single metal powders is to be compacted without addition of lubricant, a mixing operation must precede compacting. The most common type of mixing equipment for base metal powder mixes are double cone mixtures. The important consideration in mixing is that the powder must not fall freely through the air during any stage of mixing because this will always cause segregation. For this reason the cylindrical part of double cone mixers is kept short. This point must also be considered when other types of mixers, e.g. barrile type mixers with baffles, are used.

When parts are pressed in rigid dies, lubrication must be provided to reduce friction between the compact being pressed and diewall and core rod. The lubricant which has a low shear strength keeps the metal surfaces and there will be friction due to contacts the metal asperities which puncture the lubricant film. Lubricants are chosen which attach themselves strongly to the metal surfaces and are not easily penetrated. The most common lubricants for base metal powders are stearic acid, metal steerages such as zinc and lithium steerage and synthetic waxes, such as accrawax. Without lubrication the pressure necessary to eject compacts from die would increase rapidly. After a few compacts had been pressed, they would size in die during automatic compacting.

1.5. Behavior of Metal Powders Under Pressure

When metal powders are pressed in a die, the resulting compacts are generally strong enough so that they can be handled without breaking. The green strength will, of course, depend upon the type of metal powders and upon the pressure being applied. For soft metal powders quite low pressures, less than 35 MPA are sufficient to produce compacts which can be handled. For harder powders higher pressures are necessary (Popper P., 1976).

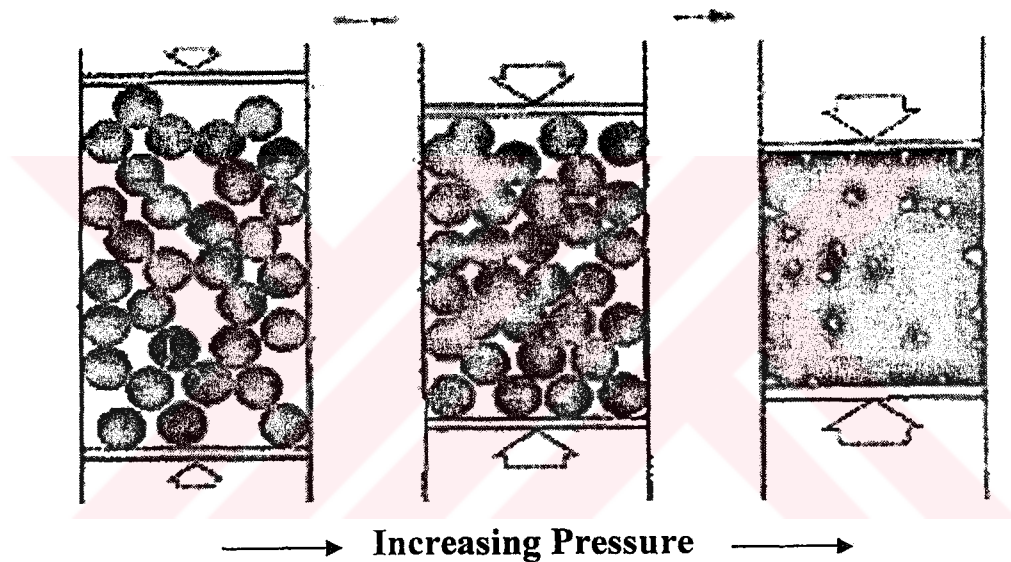


Figure 1.5. Behavior of Powders Under Pressure

The question of what physical forces produce adhesion between metals and, for that matter, between other solids is at the bottom of understanding the green strength of metal powder compacts. When clean metal surfaces are made to touch each other, the adhesion between them is small because the real area of contact between them is small. The area between the surfaces can be increased when pressure is applied. The pressure produces elastic deformation, which for geometries such a sphere on a flat surface can be calculated from Hertz classical equation. For most practical cases of adhesion of surfaces under pressure, the amount of elastic deformation is negligibly small since the weight of the specimen alone will already

cause plastic flow. Under the circumstances, the area of contact, regardless of the particle type or shape of surface asperities, will be roughly proportional to the force applied, but to produce complete contact extremely high loads are required (German R.M., 1994).

The analysis of fundamental basis is complicated by the fact that metal surfaces, and in particular, the surfaces of metal powder particles, are generally not clean, but are covered with an oxide film. In addition layers of gas molecules adsorbed on these surfaces. The oxides themselves can be cold welded, but the strength of the bond is generally low compared with that of metals. On the other hand, when metals are rubbed together, which is what happens during compacting of metal powders, the oxide films are penetrated or rubbed off and metal to metal contacts is established (German R.M., 1994).

The process occurring when a column of loose powder is compacted in a die have been described qualitatively by Seelig and Wulff who postulate a series of stages. The first stage is restacking of the powder particles in the column, also called packing, in which the bridging which always occurs in a randomly arranged stack of particles is at least partially eliminated. The second stage involves elastic and plastic deformation of the particles. As discussed above, elastic deformation plays only a minor role. How much plastic deformation occurs depends on the ductility of the metal and may be of minor significance in very hard powders such as tungsten or tungsten carbide. In most metals plastic deformation causes work hardening, which diminishes the amount of further deformation under stress. It may eventually lead to the third stage in which the particles fracture under the applied load and smaller fragments. This third stage is more important during compacting of nonmetallic powders. The three stages of Seelig and Wulff are not clearly, but overlap.

The progressions of motions for such a tool set during pressing are shown in Figure 1.6. The die provides the cavity into which the powder is pressed and gives lateral constraint to the powder. Most compaction is performed using both an upper and lower punch. The upper punch is retracted during powder filling. The lower punch position during powder entry is termed the fill position. That allows for entry of a predetermined amount of powder. The powder is vibrated into the die form an

external feed shoe and any variations in the pressed parts. The fill position differs from the lower punch position during pressurization to allow pressing in the center of the die. The position of the lower punch can change during powder fill to aid uniform powder placement throughout the cavity. After filling, the lower punch drops to the pressing position and the upper punch is brought into the die. Both punches are loaded to generate stress within maximum stress. Finally, the upper punch is removed and the lower punch is used to eject the compact. The cycle then repeats with a new fill of powder.

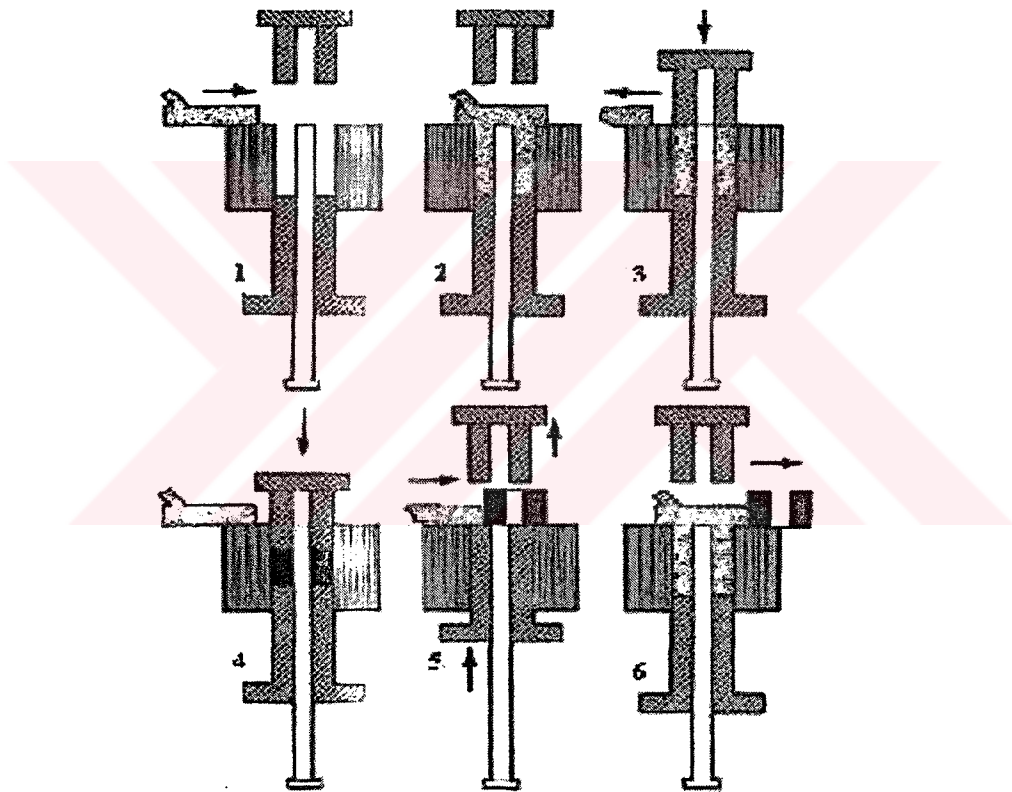


Figure 1.6. Powder Compacting Process Scheme

With increasing applied pressure the density of the powder column will increase, or its porosity will decrease. The relationship between applied pressure and the density or porosity of the powder compact has been studied by a large number of investigators, who have attempted establishing mathematical relationships between pressure and average relative density (Kouzumi M., Nishihara M, 1992).

1.6. Density and Stress Distribution in Compacts Pressed in Rigid Dies

In any discussion of density and stress distribution in powder compacts it is useful to recall the difference in the transmission of pressure in a liquid and in a confined powder column. When a liquid is subjected to hydrostatic pressure inside a confined die the pressure is transmitted evenly upon any area of the die regardless of whether the liquid must flow only in the direction of the applied force or whether it must flow around corners. Powders do not behave this way. When they are compressed in a confined die they flow mainly in the direction of the applied pressure.

When pressure is applied from top, the powder in the sidearm of the die will not be compressed at all, but will stay as loose powder. It is important to note that at the present time at all compacting operations in rigid dies are arranged in such a way that pressure is applied either only from the top or from the top and bottom but not from the sides. Presses which would apply pressure from the side could be from the sides. Presses which would apply pressure from the side could be built. The dies in such a press would have to be taken apart in order to remove pressed piece from the die. Also it would be difficult to avoid a plane of weakness in the compact where the powder is compressed from the top and from the side. Because pressure is applied only from the top and the bottom in compacting metal powders in rigid dies, the shapes can be compacted are limited. Pieces with reentrant angles, with holes with an angle to the direction of pressing or screw threads can, in general, not be produced by compacting (Kouzumi M., Nishihara M, 1992).

Another consequence of the fact that pressure is transmitted primarily in the direction of pressing is the observation that when compacts with different level of thickness are pressed compact have uniform density. The loss in force transmitted by a punch to a compact will depend upon the shape of the compact. The smaller pressing area upon the punch is exerts pressure and the larger the wall area of the compact, the greater will be the loss and the lower will be the density of the compact. In other words, compacts which are short and squat. For simple shapes with no variation in section thickness in the pressing direction.

When the density distribution in cylindrical compacts is determined, it is found that density gradients exist not only in the vertical, but also in the radial direction. The results of measurements of density distribution in metal powder compacts clearly show that the distribution of both magnitudes and direction of stresses during pressing of metal powder compacts in rigid dies must be quite complex. The early theoretical treatment of stress distribution in cylindrical powder compacts assumed that the vertical stresses are distributed uniformly over the cross section of the cylinders, that the radial stresses are a constant fraction of the vertical stresses and that the coefficient of friction is independent of the pressure.



2. PREVIOUS STUDIES

2.1. Cold Isostatic Pressing

2.1.1. Preface of Cold Isostatic Pressing

When powders are molded under pressure, the compaction process generally proceeds as follows;

- i) Rearrangement of the particles,
- ii) Elastic and plastic deformation of the particles, or fracture.

Of course, these phenomena do not occur separately; they overlap considerably. With brittle materials, the particles fracture rather than undergo plastic deformation, but with ductile materials they do not. In contrast, with substances such as ceramic powders having extremely hard particles, the particles undergo only elastic deformation. The above compaction process proceeds in the same way when molding under isostatic pressure as when molding with metal molds.

With the coming of the idea that a uniformly dense body might result if the powder were compressed with equal force in all directions, isostatic pressing was born. Applying pressure with a fluid makes possible a technique for compressing the powder equally in all directions. Pascal's principle states that if we increase the pressure at one point in a stationary fluid that is completely enclosed in a vessel, then the pressure at all points within the fluid will increase by exactly the same amount. Therefore, by filling a soft envelope with powder and compressing it with a surrounding fluid, we can make a uniformly compacted body. Isostatic pressing thus conceived was first attempted by the Westinghouse Lamp Company in America in order to manufacture billets from heat resisting metals. The process used the 1913 patent of H.D. Madden (Koizumi M., 1991).

Diversified industrial use of Isostatic Pressing did not come about until the Second World War and beyond. However, the isostatic pressing was first done at

order to manufacture billets from heat resisting metals. The process used the 1913 patent of H.D. Madden (Koizumi M., 1991).

Diversified industrial use of Isostatic Pressing did not come about until the Second World War and beyond. However, the isostatic pressing was first done at room temperature, what we call cold Isostatic Pressing (CIP). And also, Hot Isostatic Pressing (HIP), where compacting a powder and sintering it at high temperature are both done simultaneously, did not appear until the mid 1950s.

In order for isostatic pressing to succeed, it was necessary to design pressure vessels in which high pressures could be applied to powders. These pressure vessels consist of high pressure cylinders having closures to seal both ends. The two types- the threaded closure type and the press frame type- depend upon how the loads on the closures are supported. In the threaded type, stress concentrations readily arise in the threaded part and reduce its life span under repeated loadings. The press frame type has no threads in the top and bottom closures and thus eliminates the dangers of fatigue fracture due to stress concentrations. Not only is it thus superior in safety, it is also well-suited for automated production because of the ease in opening and sealing the closures. In applying pressure isotropically to a body under treatment within the high pressure vessel, a pressure medium of liquid or gas must be used. In CIP at ordinary temperatures the medium is oil or water. Pressures in isostatic pressing are 100-400 MPa for CIP in small scale equipment, and usually 100-200 MPa for CIP in large scale equipment.

2.1.2. Isostatic Pressing Equipment

There are two types of isostatic pressing: Free Mold or “Wet Bag” method and the Fixed Mold or “Dry Bag” method. They are identical in principal, achieve similar metallurgical properties, and require the same equipment. They differ, however, in the configuration and placement of the tooling.

Both methods utilize a pressure vessel with a closure device designed to contain a fluid under high pressures, along with a pressurization system and controls. Today’s more advanced production-scale isostatic presses are equipped with various

flexible molds are first filled with the powder, and then vibrated, jolted or subjected to vacuum to remove entrapped air and assure optimum distribution of the loose powder. After filling, the molds are sealed water-tight and placed into an immersion basket or rack assembly for introduction into the pressure chamber during a given cycle. The chamber is then filled with the hydrostatic medium (water or oil), the cover is closed and secured, and the vessel is pressurized to the required pressure.

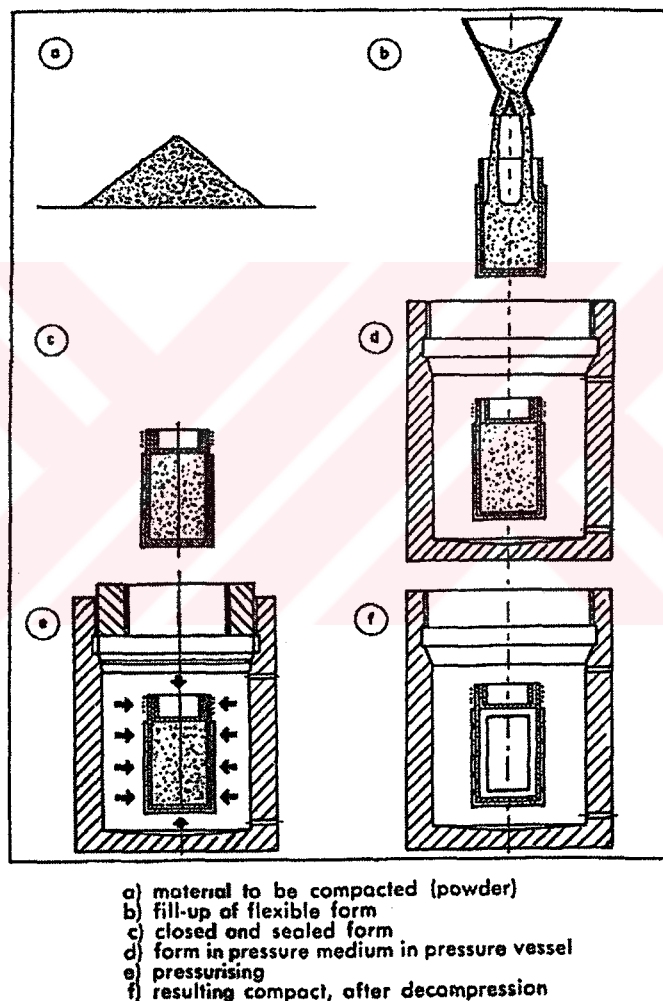


Figure 2.1. Schematic description of isostatic pressing principle

The Fixed Mold Process, depicted in Figure 2.2 is characterized by the flexible mold positioned directly inside the pressure chamber. The mold may be supported by perforated outer shell and with a lip on the mold sealing against the side wall of the

chamber. An annulus is provided between the vessel wall and the outside surface of the mold to allow the hydrostatic fluid to enter during the pressing cycle.

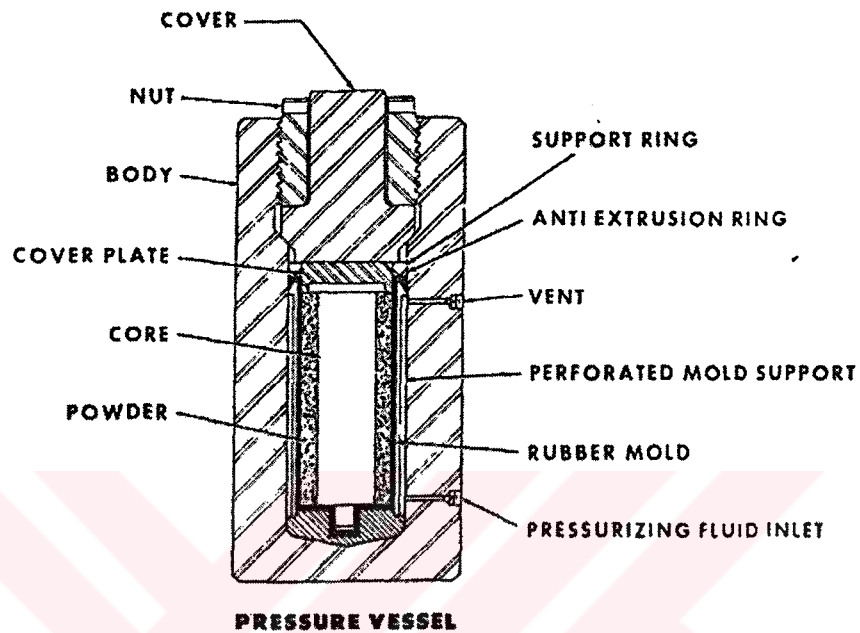


Figure 2.2. Fixed mold process

As with Free Mold Pressing, equal and simultaneous pressure can be applied to the mold. The powder fill is made into the cavity of the fixed mold, which act as an integral part of the pressure vessel. Hydrostatic fluid does not into contact with the operator's hands. Evacuation of air prior to pressing if necessary is generally achieved with a vibration device or vacuum connection in the pressure vessel cover. The sequence then proceeds as in Free Mold Pressing that shown in figure 2.3.

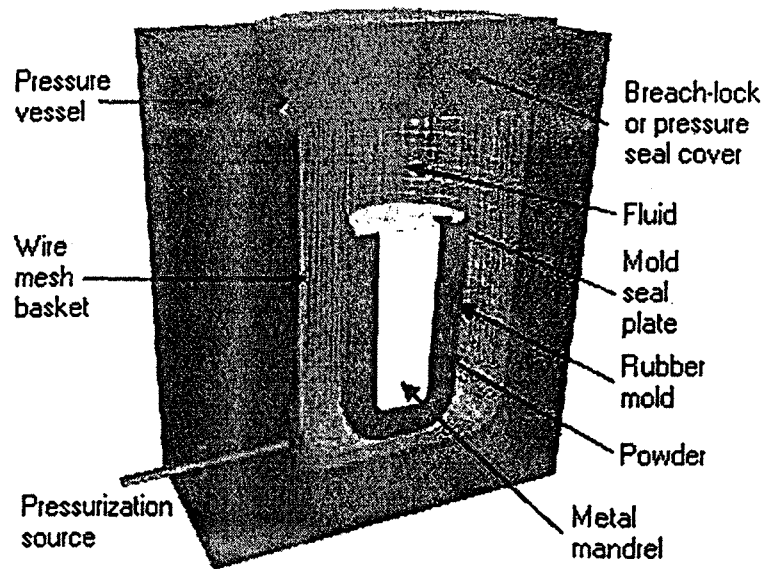


Figure 2.3. Wet Bag Cold Isostatic Pressing Unit

In Fixed Mold Pressing, one part is ordinarily pressed per cycle per pressure chamber. The process lends itself to such shapes as bars, rods or tubes. Undercuts can be also added if the largest dimension of the compacted part will clear the smallest dimension of the mold when withdrawn. Such advances as multi-chamber Fixed Mold Process and automated powder fill and parts ejection mechanisms have made this process feasible for many mass production applications.

Automated systems are available for both free Mold and Fixed Mold Isostatic Pressing. The selection of one process over the other is dependent upon the configuration of the part, production rates required, and cost considerations (Popper P., 1976).

2.1.3. The Relation Between Pressure and Density for CIP

Compared with the die-compaction, isostatic compaction provides more uniform pressure distribution within the compact. The major reason being the

absence of die wall friction, another factor is the greater area over which the pressure is applied. Furthermore, distinct advantages may often be gained from the ease with which air can be evacuated from the loose powder before compaction, if necessary, and because of starting with a uniformly packed powder. Consequently, isostatic pressing provides increased and more uniform density at a given compaction pressure, and relative freedom from the compact defects when applied to brittle or to fine powders. It is a result of the uniform compaction Pressure-Density distribution that the cross-section: height ratio is not limiting feature as it with die-compaction (Koizumi M., 1991).

Another characteristic of the process is elimination of die lubricants. Their absence permits higher pressed densities and eliminates problems associated with lubricant removal prior to or during final sintering.

The most important differences between the mechanisms of isostatic and die-pressing are:

- a) Compaction forces and movements which act simultaneously from all sides result in less linear displacement of an important portion of the powder mass for obtaining comparable density levels. This results in somewhat less frictional work of each of the powder particles.
- b) Existing pressures in and outside the powder environment are equal. Thus any trapped air within the powder mass cannot escape; so that it would be compacted between the powder particles, and would influence pressure-volume relation of the powder mass.

It is evident that in all cases a combination of these factors will occur simultaneously or in a certain order of progression depending on the pressure level (Hausner H., 1971). The pronounced change in slope of the pressure density relationship clearly shown in figure 2.4, for die pressed powder, appears to occur also for the isostatic pressing. Results for aluminum, beryllium, copper, and iron powders show such a change in gradient at high applied pressure which suggests that this is related to strain-hardening. The relative densification obtained by die and isostatic compacting is shown in figure 2.5, for several powders (Schmid V., 1986).

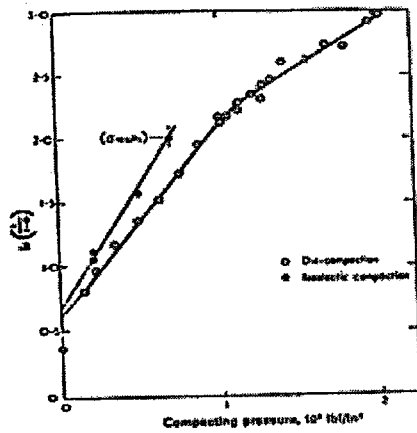


Figure 2.4. Comparison of The Green Density of sponge iron powder after die and isostatic pressing (Schmid V., 1986)

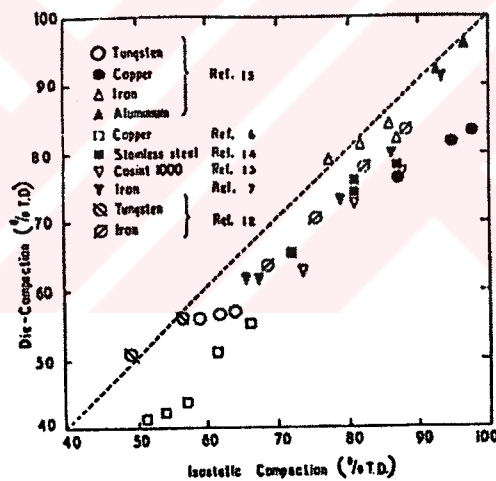


Figure 2.5. Green Densities with various metal powders for die and isostatic compaction at the same pressure (Schmid V., 1986)

The pressure distribution within a die-pressed compact may become isostatic. Density relationship should be identical with that isostatic compacting only if the density distribution is equally uniform. Because with isostatic pressing the effects of die-wall friction are absent, much more uniform densities are obtained. This is contrast to die pressing where it is well established that die-wall friction exerts a major influence on density distribution in the absence radial power flow.

2.1.4. Progress in Compaction Under Isostatic Pressing

If the molding conditions for the powders and the equation for the stress-strain relation are obtained, then it becomes possible in principle to analyze mechanically the compaction state, using the equation for the equilibrium of forces and the boundary conditions. When the stress distribution is not uniform, the finite element method is probably most effective technique for obtaining the density distribution or the shape of the molded body. Presently, there are finite element analysis techniques that treat materials as elasto-plastic solids and other techniques that treat materials as rigid-plastic solids.

The rigid-plastic finite element method used in the analysis of molding under isostatic pressure was developed only recently and thus can not yet be called a firmly established analytical technique. However, many applications have been reported. Figure 2.6. shows the density distribution of the density ratio for a body of powder after its molding under isostatic pressure in a cylindrical capsule (Diameter to height ratio of 1.1), both for a copper powder and a ceramic powder. The shape of each body after molding is also shown (Shima S.& Nakanishi R., 1988);

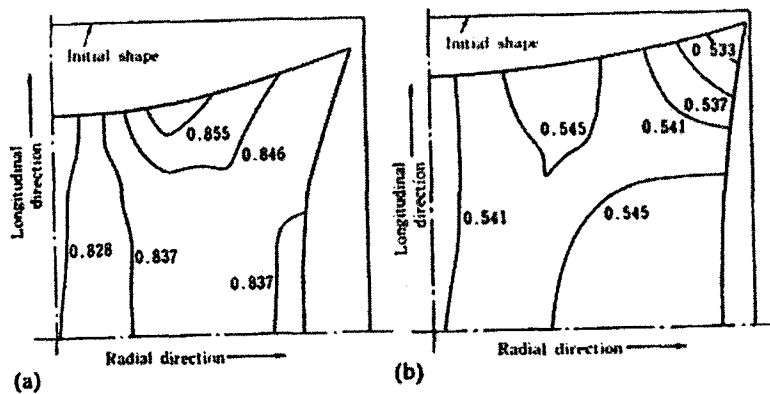


Figure 2.6. Distribution of Density Ratio in Molded by Isostatic Pressing.

(a) Electrolytic Copper Powder $P=410$ Mpa (b) Ceramic Powder $P=656$ Mpa (Shima S.& Nakanishi R., 1988)

The density distribution and the shape clearly differ depending on the type of powder. In addition, Figure 2.7. illustrates the shape of molded bodies obtained when using two different materials for the capsule. Even for the simple shape, it is easy to see how much the state of compaction differs with differing kinds of powders and different capsule materials. This shape was a comparatively simple one, but this kind of analysis will surely be applied to complicated shapes as well in the future (Shima S.& Nakanishi R., 1988).

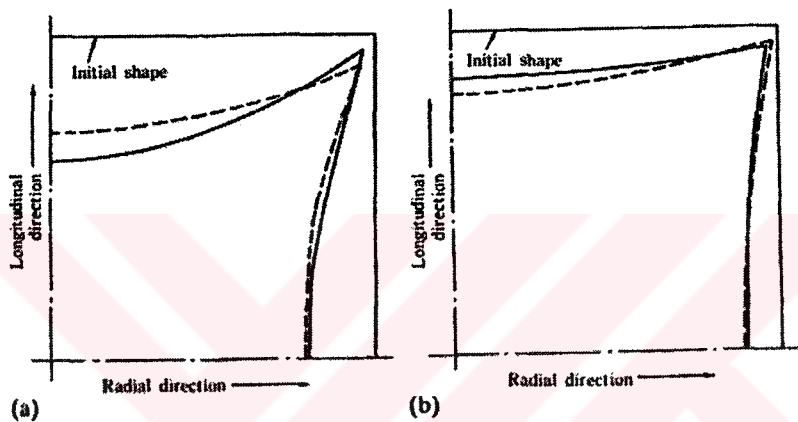


Figure 2.7. Difference in Molded Body Shape Between Two Types of Capsule (a) Electrolytic Copper Powder $P=410$ Mpa, ___ Sturdy Capsule, --- Frail Capsule (b) Ceramic Powder $P=656$ Mpa, ___ Sturdy Capsule, --- Frail Capsule(Shima S.& Nakanishi R., 1988)

The other thing for CIP is pressure reducing. Pressure reduction is the most important step in CIP molding, for during the pressure reduction process, the rubber mold that had adhered tightly to be molded body returns to its original state and separates from or slips off the molded body. Moreover, the air that had been sealed inside the molded body travels through internal pores and enters the space between the molded body and the rubber mold. The pressure favors these phenomena varies with the properties of the powder and the characteristics of the rubber mold, and can only be determined with experience. Failure to select appropriate pressure reduction rate in this pressure range leads to breakage of the molded body, and therefore a wide variety of pressure reduction mechanisms have been proposed (Koizumi M., 1991).

2.1.5. Molds in CIP Forming and Molding

The factor in CIP molding that exercises the greatest influence over the quality of the molded body is the forming mold. It also requires of technologists the greatest know-how and effort. The forming mold must meet the following requirements (Koizumi M., 1991 and Khol R, 1970):

- ✓ Not break the molded body when the pressure is reduced.
- ✓ Not give rise to an uneven density in the molded body.
- ✓ Produce to molded body close to the shape of the final product.
- ✓ Yield precise dimensions in the molded body.
- ✓ Be capable of easy,
- ✓ Be readily separable from the molded body.
- ✓ Be easily packed with powder.
- ✓ Not break during molding.
- ✓ Not deteriorate with repeated use under high pressure.
- ✓ Be easy to produce and low in cost.

2.1.6. Special Features of Cold Isostaic Pressing

1. High density of the molded body: Compared with other molding methods at the same molding pressure, such as molding with metal molds or injection molding, higher densities can be obtained; and because, the molded body is thus stronger, handling it is comparatively easy prior to sintering and pre-sintering treatments.
2. Homogeneity of the molded body: Because the body is pressed isostatically, there is no friction between the body and the mold, and the molded body that results has a uniform density with little residual stress. This reduces the warping or deformation during sintering. High density and homogeneity are extremely important advantages of CIP.
3. Significant reduce in molding aids: In the molding itself, there is basically no need for additives, but several Per cent of molding aids are added to enhance to fluidity of the powder or uniformity of the compaction. Since only a limited

quantity of a molding aid (wax) is used, the dewaxing process prior to sintering can be omitted or abbreviated.

4. No Restrictions on Size or Dimensional Ratios of The Molded Body: As long as the object can be accommodated in the high pressure vessel, than anything designable with a rubber mold –whether large, long, or irregularly shaped- can be molded into the homogeneous product.
5. Low Mold Costs: If there is a master mold for the rubber mold forming the body, then the mold can be produced at low cost. Mold costs in this method are very reasonable compared with those in other molding methods.
6. Applicability to Molding Complicated Shapes: By utilizing the contraction of the powder or by using assembled or disposable rubber molds, it is possible to mold complicated concave and convex that are impossible with metal molds.
7. Applicability to Molding Composite Products: Composite products can be molded using CIP, either by layering different types of materials or by separating them at the time of packing. These techniques are also possible when molding with metal molds, but complications in the flow of the powder make packing difficult. In CIP, the powder is pressed isostatically, so the molded products is similar in shape the packed powder. Moreover, CIP also makes it possible to use multiple packing in molding a body.

The rigid-plastic finite element method used in the analysis of molding under isostatic pressure was developed only recently and thus can not yet be called a firmly established analytical technique. However, many applications have been reported.

2.2. Overview of Hot Isostatic Pressing (HIP)

Hot Isostatic Pressing is a process which only superficially resembles cold isostatic pressing. Whereas in cold isostatic systems, the compaction phenomenon is basically mechanical, with HIP there are significant metallurgical effects taking place such as solid state diffusion. In principle, the powder is placed inside a sealed mold and then is subjected to isostatic pressure at any elevated temperature, in excess of 538 °C and usually at 982 °C, 1260 °C, 1427 °C or 1649 °C depending upon the

product being processed. Temperatures are usually selected to avoid any liquid phase although in some complex alloys or mixtures a partial liquid phase is unavoidable, particularly in binder materials. It is because of the elevated temperatures that procedures and techniques vary from that of cold processes. Because rubber or other conventional elastomers cannot withstand the imposed heat, metal or glass are used as tooling. This automatically restricts shape and configuration to relatively simple forms. Thus the process is normally carried out in the free mold manner. The reasons for this will become evident when the typical cycle is described. Tooling is of the isostatic type, although some experimental work has been done to produce complex configurations using a quasi-isostatic tooling system wherein a high temperature ceramic such as alumina is used as the rigid member.

2.2.1. Typical Cycle of Hot Isostatic Pressing

The powder to be compacted is placed inside a container or mold, usually made from mild steel. The filled can is then closed by welding a top to it fitted with a suitable evacuation connection. The evacuation and outgassing of the powder is usually carried out at a moderately elevated temperature. A suitable level is 427-649 °C for alloy steels and super alloys. This is accomplished by connecting a vacuum pump to the evacuation which is extended to reach outside the preheating furnace. The container is pumped down until a level of at least 1,000 micron is reached and the work is then placed inside a preheat furnace and brought up to temperature. Once temperature is reached, vacuum pumping is continued until the 1,000 micron level is reached inside the container. The work is then removed from the furnace, still under vacuum and the connection tube is crimped and welded shut close to the can and the excess length trimmed off to permit efficient loading into the hot isostatic press. These two steps can be done independently HIP unit so suitable loads can be prepared in the advance and stockpiled to keep the HIP unit efficiently utilized. One or more prepared cans of material are loaded into the work space of the HIP unit. The number and arrangement are function of the geometry involved, modified only by the necessity to insure that the desired temperature uniformly be maintained and that

adequate mechanical support is provided. Conventional alumina kiln furniture is used for some parts. In other cases crucibles and/or boat scan handle small parts. Various grades of alumina powders and balls can also provide the required mechanical support. It is common to bench pack a large mid steel cylinder, which is sized to fill the work space of the system with as many parts as feasible so that only a single element need by actually inserted and removed from the press. In small laboratory size equipment, loading is done directly into the HIP unit, but the method described previously is used on all larger systems. The HIP unit is closed and locked, evacuated to minimize to contamination of the pressurizing gas, and then the pressure and temperature are usually raised simultaneously. In some cases, e.g. use of glass as a canning material, minimum temperature levels are established first, followed by pressurization to insure that the canning material used is adequately ductile. In other cases, pressurization to an intermediate level is done cold and then the pressure is raised to the designed working level by increasing the temperature. After reaching the desired pressure and temperature levels, the system is kept at a "hold" or soak condition to insure that the center of the load ha reached the set conditions. No generalizations can be made covering the time required since it is totally function of the load characteristics, shape, mass, composition, configuration of loading, etc. Thermocouples placed in the load indicate completion of the minimum soak time. Additional time might be specified to achieve particular metallurgical properties. These are separately evaluated on the basis of diffusion rates and similar factors in a given application. Finally, pressure and temperature are allowed to drop. Normally the furnace is merely shut off completely and the temperature is allowed to decrease at its maximum rate. Pressure is usually allowed to drop in thermodynamic reaction to the temperature drop since bleeding off gas to reduce the pressure slow down the cooling rate. Some materials require a slower, controlled cooling which can, of course, be achieved in most HIP systems. Cycles may be timed so that the cool down phase can take place at night. The system is ready venting the zero pressure and reloading. This is normal production cycle condition. There are some operations which can achieve two cycles per day, while

others, by the nature of the cycle required by the product, might run up to two or three days. The vessel is then opened, reloaded and the above steps repeated.

2.3. Sintering

The main task of powder metallurgy and ceramic science is to supply development people with insight into essential parameters in the processing of materials. This makes it possible for them to bring some order into their excess of empirical experience. There are a number of clear-cut advantages. First of all, as one always uses a working hypothesis, a good theory can help to save a lot of work. Even a poor model is of practical use if it provides workable ideas for experiments. Secondly, considerable effect may result from experiments guided by analogy. Achievements in one part of the powder metallurgy or ceramic art resulting from a good theoretical background can cause an avalanche of achievements in other parts. And thirdly, if the person in the development laboratory is well up on the theoretical background it will give him great satisfaction to understand what he has done.

Industries which can make use of synthetic and purified raw materials for their products are in a much more favorable position to apply the principles brought forward by scientific studies of the sintering process. To illustrate the complexity of real systems, the factors in the technology of sintered products this can have a dominant effect.

Powder:

- Particle size
- Particle size distribution
- Particle shape
- Intra-particle porosity
- Agglomeration
- Homogeneity in chemical composition
- Absorbed and included gases
- Impurities
- Reactivity (Non-equilibrium defects, etc.)

Pressed Product:

- Packing density
- Pore distribution
- Pore size distribution
- Density distribution
- Effects from granulation (Binders)
- Crystallographic texture

Sintering:

- Temperature
- Temperature gradients
- Temperature cycle
- Gas atmosphere
- Second phase
- Liquid phase
- Activated sintering
- Pressure

Sintering is the most common technique for consolidating powders. Essentially, it is the removal of the pores between the starting particles, combined with their growth and strong mutual bonding. The process is carried out by heating up the "green" part at about 80% of the melting temperature, until full strength is obtained (10 min. to several hours). The biggest problem of this technique is shrinkage which causes cracking and distortion. There are many methods for sintering a component. The most important are: vapor-phase sintering; solid-state sintering; liquid-phase sintering; reactive liquid sintering. Overpressure sintering uses also pressure to accelerate densification.

As the term 'sintered part' implies, sintering is a key part of the operation. It is here that the compact acquires the strength needed to full fill the intended role as an engineering component. In general, sintering requires heat. The ISO definition of the term reads: The thermal treatment of a powder or compact at a temperature below the melting point of the main constituent, for the purpose of increasing its strength by

bonding together of the particles. Theories about exactly what happens during sintering have provided the subject matter of innumerable conferences and learned scientific papers, but these need not concern us here. Suffice to say that atomic diffusion takes place and the welded areas formed during compaction grow until eventually they may be lost completely. Recrystallisation and grain growth may follow, and the pores tend to become rounded and the total porosity, as a percentage of the whole volume tends to decrease; but see the section on 'dimensional changes'. The operation is almost invariably carried out under a protective atmosphere, because of the large surface areas involved, and at temperatures between 60 and 90% of the melting-point of the particular metal or alloys. For powder mixtures, however, the sintering temperature may be above the melting-point of the lower-melting constituent, e.g. copper-tin alloys, iron-copper structural parts, tungsten carbide-cobalt cemented carbides, so that sintering in all these cases takes place in the presence of a liquid phase, hence the term liquid phase sintering. It is, of course, essential to restrict the amount of liquid phase in order to avoid impairing the shape of the part. Control over heating rate, time, temperature and atmosphere is required for reproducible results. The type of furnace most generally favored is an electrically heated one through which the compacts are passed on a woven wire mesh belt. The belt and the heating elements are of a modified 80/20 nickel/chromium alloy and give a useful life at temperatures up to 1150 °C. For higher temperatures walking beam furnaces are preferred, and these are increasingly being used as the demand for higher strength in sintered parts increases. Silicon carbide heating elements are used and can be operated up to 1350 °C. For special purposes at still higher temperature molybdenum heating elements can be used, but special problems are involved, notably the readiness with which molybdenum forms a volatile oxide. Molybdenum furnaces must operate in a pure hydrogen atmosphere.

2.3.1. Sintering Reactions

The surface of a solid differs from the interior in that energy γ_{sv} (at the interface between the solid phase and the gas phase) the excess energy that accompanies the

scission of two chemically bound species. The type of stress that acts depends on the curvature of the surface and that of γ_{sv} . In the initial stage of sintering, if a neck with a radius of curvature ρ forms upon the fusion of two small spheres of radius R , then a stress $\sigma = \gamma_{sv}(1/x - 1/\rho)$ (where $\rho = x^2/4R$) acts upon the neck surface. This neck is shown in figure 2.8.

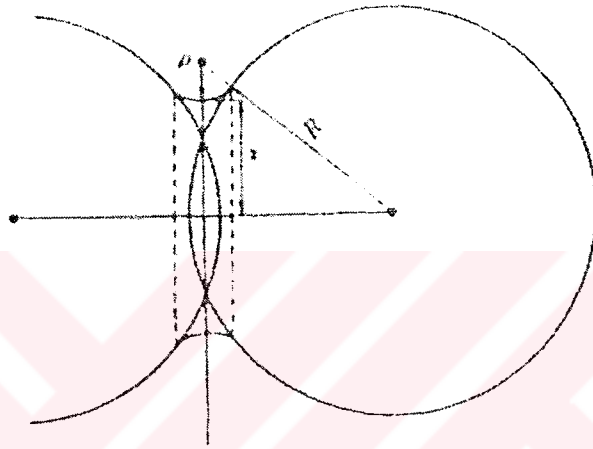


Figure 2.8. Model of sintering between two similar spheres

In the initial stage, $x \gg \rho$, so the stress acting upon the neck portion becomes a tensile force $\sigma = -\gamma_{sv} / \rho$, the driving force for fusion. In the initial sintering stage, the radius of curvature of the neck is small. Thus, as stated above, a large difference in the void concentration is created by the large stress acting upon the neck. As a result, even if isostatic pressure in order of 100-200 MPa were to be applied from the exterior at this stage of sintering, it would have hardly any effect on the sintering reaction. However, if the sintering reaction proceeds and the radius of curvature of the neck increases, that is $\rho \rightarrow x$, the stress on the neck practically disappears, the difference in the void concentration approaches 0, and the void concentration gradient that is necessary for eliminating the sealed pores during the middle and the final stages of sintering disappears. External pressure applied at this stage is very effective in the elimination of pores (German R. M., 1996).

3. MATERIAL AND METHOD

3.1. Material

3.1.1. Work piece Materials

In this study, Al and Fe powders are used for compacting process as powder. Powders are obtained from Hastek Toz Metal Sanayi from İzmit and size range defined by this company. Typical characteristic of powders are shown in table 3.1.

Table 3.1. Specifications of used powders

Powders	Shape	Manufacturer	Manufacturing Method	Hardness (HV)	Apparent Density,%	Size Range, μm
Aluminium	Ligament	Hastek (İzmit)	Gas Atomized	18	50	-150+106
Iron	Irregular	Hastek (İzmit)	Water Atomized	65	35	-150+106

A Wet Type CIP unit used for the study and this unit consisting of a compression piston and pressure chamber was designed and constructed by Assoc. Prof. Dr. Abdulkadir EKŞİ and used his previous researches also. The piston and the chamber are made of SAE 1040 steel and the heat treated to hardness of 60 HRC. Their cylindrical surfaces were ground to have a roughness of $R_a = 0,3 \mu\text{m}$. Technical drawing of piston and chamber mechanism is depicted in figure 3.1.

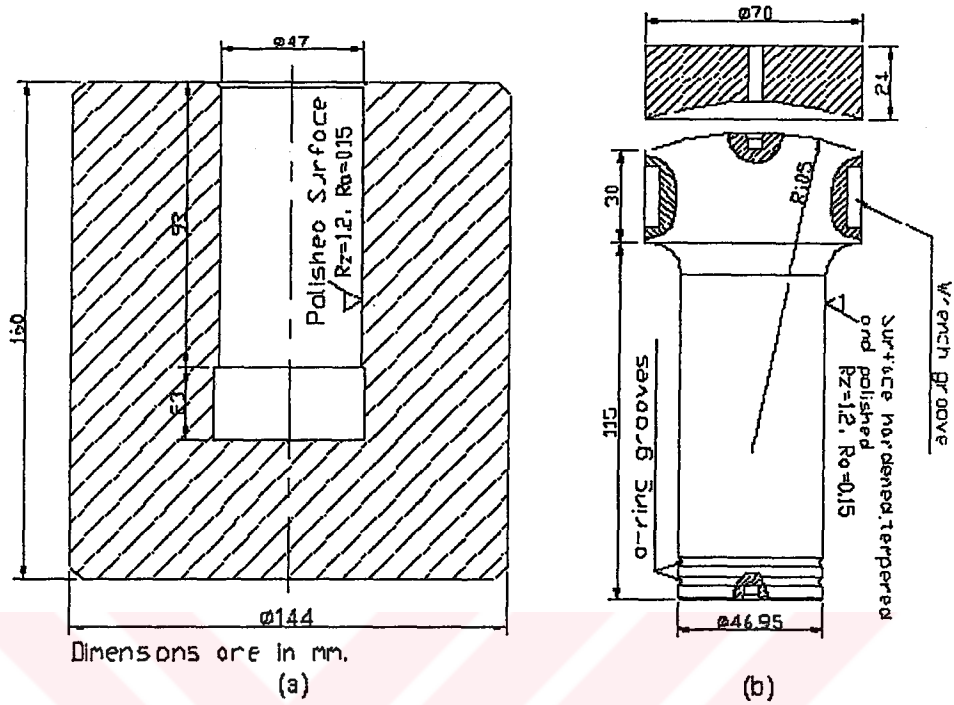


Figure 3.1. Wet Bag CIP unit used, (a) Pressure body, (b) Compression piston

Water and mineral oils can be used as the pressurizing media. For this work, Boron Oil is used for pressurizing media, because it is cheap and easy to obtain. The copper tube was used as the flexible container for the powder mass as shown in figure 3.2. The internal volume of the flexible container was $V_i = 4 \text{ cm}^3$.

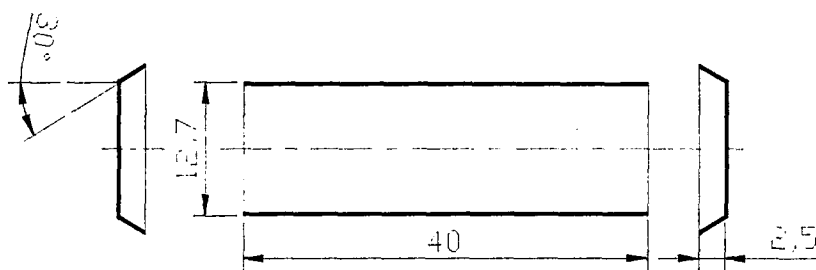


Figure 3.2. Flexible container

One of the end caps of the flexible container was first brazed and then filled with the powder sample filled in. Later, the other end cap was brazed.

3.1.2. Compaction Process

Compaction of the powder specimens has been established in hydraulic press. The force in the hydraulic test machine was increased 680 kgf in per second. This force is applied to compression piston, and piston compressed the pressure medium with flexible container which in established pressure body. According to the Pascal's Principle, the pressure at all points within the fluid will increase by exactly the same amount by effect of applied force. Wet bag CIP unit in the hydraulic test machine is shown in figure 3.3.

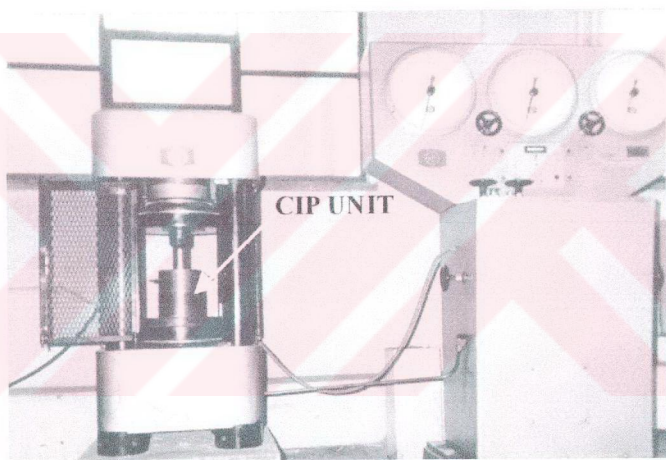


Figure 3.3. Hydraulic testing machine and CIP unit

The flexible container with the powder immersed into the pressure chamber, the piston was placed into the hole and then pressures of 100 MPa to 600 MPa was applied. And pressing degree is increased uniformly in the pressing tool. There is always air in filled flexible container, when we increase the pressure it is compressed between the particles and it causes porosity in the compacted body. Thus, pore

structures occurred between the powders. After compaction process, deformation on the flexible container under applied process shown in figure 3.4.

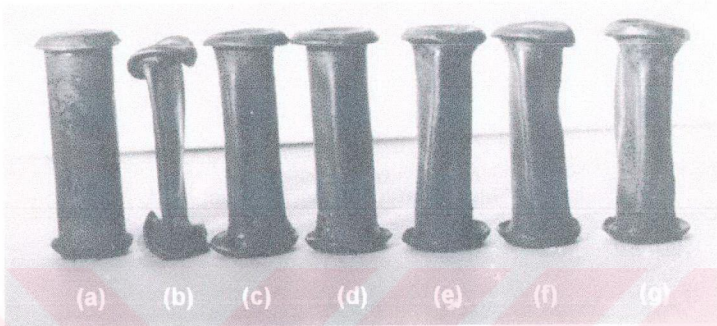


Figure 3.4. The deformation of the flexible container under applied pressures (a) before pressing, (b) empty pressing (20 MPa), (c) 100 MPa, (d) 200 MPa, (e) 300 MPa, (f) 400 MPa, (g) 500 MPa

3.1.3. Density Measurement

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 in equation (3.1)

$$V_p = V_i - \Delta V \quad (3.1)$$

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

$$D_p = \frac{W}{V_p} \quad (3.2)$$

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

$$D_t(\%) = \frac{D_p}{D} \times 100 \quad (3.3)$$

Where D is the picnometric density of the powder material.

Green density and density of sintered parts measured with Archimedes Principle method. Archimedes Principle is easy to apply and results can be calculated rapidly. Especially, when precision balance is used the results can be confidential. In order to measure densities, SARTARIUS 200EX Precision Balance test machine which has 1/10.000 precision of scale and SARTARIUS Density Measurement Kits are used in GAZİ University Mechanical Research Laboratories. This precision balance test machine can be seen in figure 3.5.



Figure 3.5. SARTARIUS 200EX Precision balance test machine

In order to measure density with Archimedes Principle, SARTARIUS Density Measurement Kit is also used. This kit specially produced with this balance consist of liquid tube carrier table, specimen holder, glass liquid tube and fitted temperature for liquid. Pure water is used for measuring liquid media at 22 °C for this study. SARTARIUS Density Measurement Kit can be seen in figure 3.6.



Figure 3.6. SARTARIUS density measurement kits

According to Archimedes principle;

x : weight of specimen

y : carrier material weight

z : weight of specimen in the water

b : difference of weight

a : weight without carrier bond

d : density

k : 0,9999 (Special constant for pure water at 18-26 °C temperature from handbook of the Density measurement kits)

$$a = z - y, \quad (3.4)$$

$$b = x - a \quad (3.5)$$

$$d = k \frac{x}{b} \text{ gr/cm}^3 \quad (3.6)$$

From these equations, the densities have been calculated easily and all data used for density comparisons.

3.1.4. Sintering

Sintering of all materials is achieved in GAZİ University Mechanical Research Laboratories and Reinmehr automatic controlled high temperature gas atmosphere is used as a furnace as shown in figure 3.7. The maximum temperature capacity of furnace is 1800 °C. The gas for sintering atmosphere was able to running in the furnace during sintering time.



Figure 3.7. Reinmehr automatic controlled high temperature gas atmosphere furnace

In sintering process, Al parts are sintered in 600 °C, 620 °C, and 640 °C temperatures during 20 minutes. Temperature increased 5 °C per minute until sintering temperature, then parts are waited in this temperature 20 minutes. After furnace is closed and temperature decreased 8 °C per minute until 300 °C, then decreasing temperature became 6 °C until room temperature. And also Argon gas was always running in the furnace until reaching room temperature. Tree different Al

sintering conditions are illustrated in figure 3.8-3.10. for 600, 620, 640 °C temperatures in 20 minutes respectively.

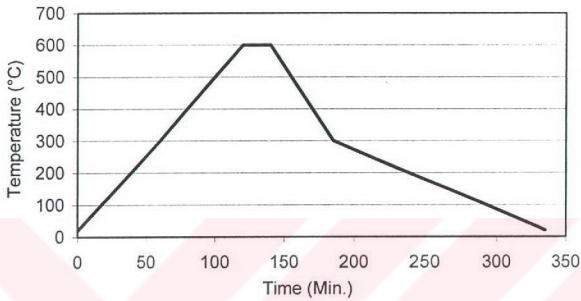


Figure 3.8. Time-Temperature gradient of Al specimens for 600 °C

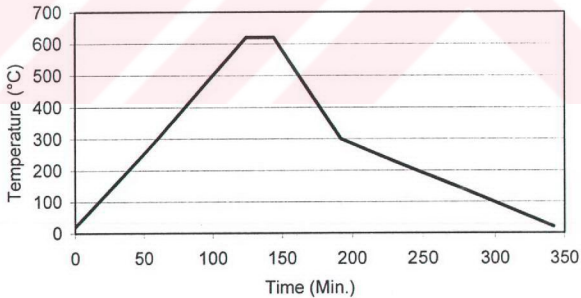


Figure 3.9. Time-Temperature gradient of Al specimens for 620 °C

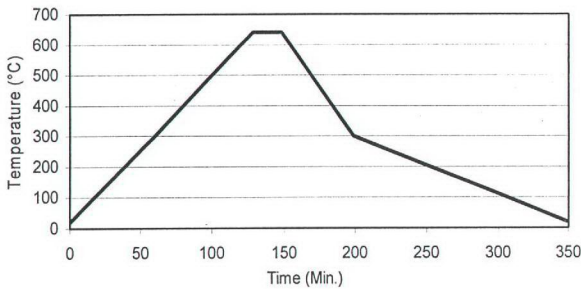


Figure 3.10. Time-Temperature gradient of Al specimens for 640 °C

Fe parts are also sintered in 1200 °C for 30, 60, and 90 minutes waiting. These conditions created very different metallurgical structures due to time and temperature. As shown in figure 3.12-3.15. for 30, 60, 90 minutes waiting times respectively. Temperature increased 5 °C per minute until sintering temperature then parts are waited that temperature for 30, 60, and 90 minutes. After this time, furnace is closed and temperature decreased 8 °C per minute until 600 °C, then temperature decrease became 6 °C per minute approximately until room temperature. Also Argon gas was always running in the furnace until reaching room temperature. 3 different Fe sintering conditions are illustrated in figure 3.11-3.13. for 600, 620, 640 °C temperatures in 20 minutes respectively.

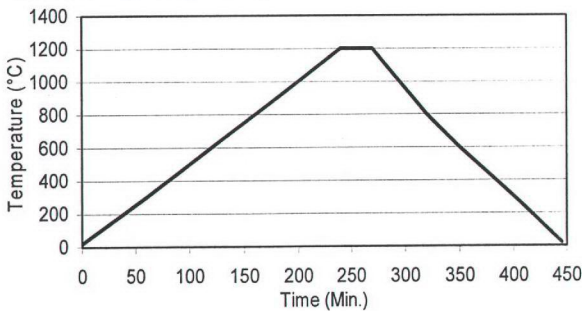


Figure 3.11. Time-Temperature gradient of Fe specimens for 30 minutes

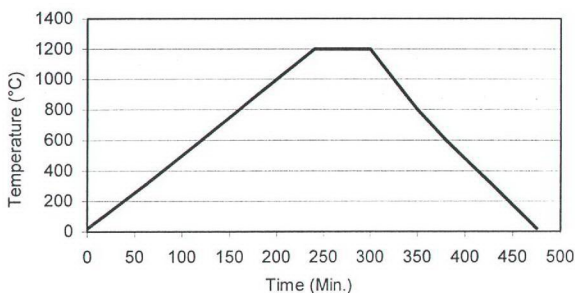


Figure 3.12. Time-Temperature gradient of Fe specimens for 60 minutes

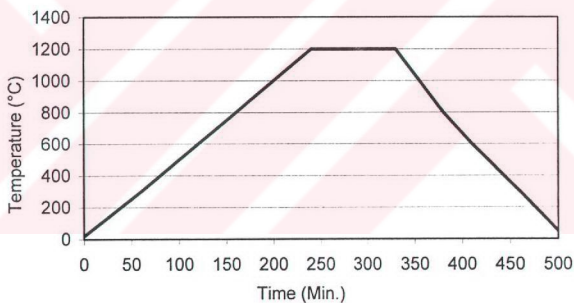


Figure 3.13. Time-Temperature gradient of Fe specimens for 90 minutes

3.1.5. Microstructure Analysis

In order to analyze compacted parts; deformed flexible containers were cut perpendicular to their axis and then mounted in plastic with 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 Optic Microscope and Scanning Electron Microscope (SEM). It was not possible to prepare samples of iron samples for examination due loose powder under 200 MPa pressure.

3.1.5.1. Optical Microscopy

Specimen preparation has become a long established art in the optical microscopy of materials, particularly in the fields of metallurgy and mineralogy. The specimens are prepared ideally representative of the structure of interest and free from damage and contamination. In this study, preparation of specimen has 5 steps and all these steps are illustrated schematically in figure 3.14. After preparation steps, all specimens investigated with optic microscope and photographed. Preparation and photographing specimens are managed again in GAZİ University Mechanical Research Laboratories.

Cutting → Mounting → Grinding → Polishing → Etching → Optical Microscopy

Figure 3.14. Specimen Preparation Steps

For cutting operation with as little deformation as possible, is required to facilitate and expedite further preparation. Consequently, the most appropriate sectioning method is Abrasive Wet Cutting, which will introduce the least amount of damage on the cut specimen and compacted parts were cut with this method. STUERS Abrasive Wet Cutting Test Machine shown in figure 3.15.

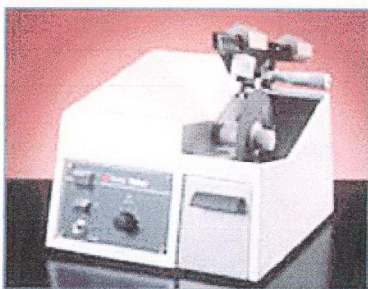


Figure 3.15. STUERS Abrasive wet cutting test machine

Mounting of samples are embedded in bakalite to facilitate their handling and to improve the preparation result. Specimens that need perfect edge retention or protection of layers require mounting. For the best possible result, samples were cleaned prior to mounting. And the surface is made free from grease and other contaminants for the best possible adhesion of bakalite to sample. Samples are put into the hole on the machine through the plain surface then bakalite powder added. Cover is closed and after machine started, heating bakalite formed and covered the parts. This STUERS Labopress mounting unit test machine shown in figure 3.16.



Figure 3.16. STUERS Labopress mounting test machine

For grinding and polishing operation STUERS Labopol is used. On the grinding sand paper with different abrasive dimensions which 800 μm , 600 μm , 400 μm , 200 μm , 100 μm is used respectively, and changed from bigger to small in successively finer steps to remove material from the surface until the required result is reached. After grinding, sandpaper is changed with polishing material. Specimens polished with this machine on the different layer. STUERS Labopol Machine is illustrated in figure 3.17.



Figure 3.17. STUERS Labopol Grinding and Polishing Machine

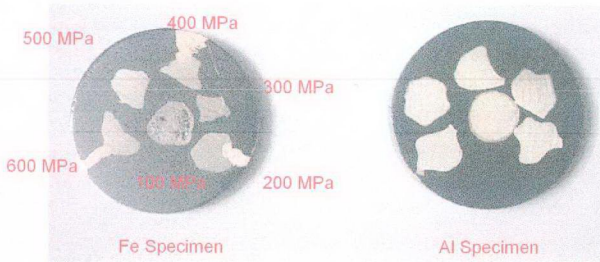


Figure 3.18. Prepared specimens parts

Finally, two different mediums were used in the etching step. For Al parts %0,05 HF + %99,95 H₂O solution is used and the Al parts immersed into the solution for 15 seconds approximately and etching time differs with pressure. This phenomenon obtained empirically. %5 HNO₃ + %95 H₂O solution medium is used for Fe parts and etching time differs according to the pressure also.

3.1.5.2. Scanning Electron Microscope (SEM)

The Scanning Electron Microscope is commonly used to examine the microstructure of bulk specimens. It is an electron-optical instrument which uses a source of electrons to illuminate the specimen. These electrons are accelerated down the column and pass through a combination of electromagnetic lenses and apertures to form a fine probe at the surface of the specimen in the chamber area. Both the column and the chamber are held under vacuum to avoid high voltage discharge and scattering of the electrons along their path by residual gas atoms. A variety of signals are produced as a result of the interaction of the beam with the specimen, which may be collected by appropriate detectors. There is a trade off between good count rate and low noise images and ability to see very fine specimen detail (McCall J.L. 1973)

Generally, two types of electron microscope are used; Scanning Electron Microscope (SEM) and Transmission Electron Microscope (TEM). Differences between optical and electron microscopes are shown in figure 3.19.

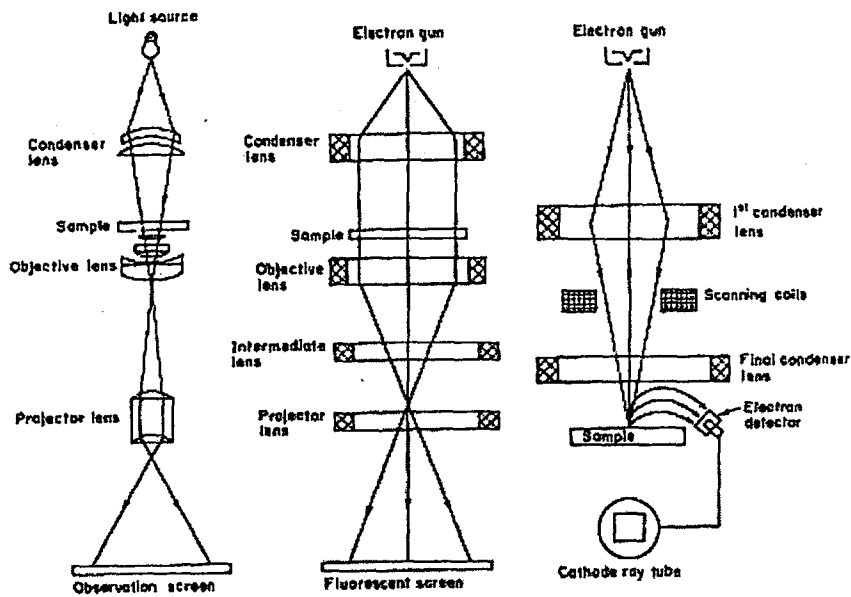


Figure 3.19. Comparison of Optical Microscope, Transmission Electron Microscope and Scanning Electron Microscope (McCall J.L. 1973)

In order to obtain well researchable images from the parts, compacted specimens were broken and cracked surfaces were investigated. Materials before and after sintering were researched. Plastic and elastic particle deformations, effects of pressure and sintering can be seen easily with SEM photos.

Scanning Electron Microscopy (SEM) investigations are managed in ERCİYES University Technology Research and Applying Center and LEO 440 Computer Controlled Digital Scanning Electron Microscope is used and shown in Figure 3.20.



Figure 3.20. LEO 440 Computer controlled Scanning Electron Microscope in ErciyesUniversity Laboratory

Special features of LEO 440 SEM counted at the bottom

- 5X-3.000.000 X enlargement capacity
- 40 kV maximum acceleration voltage
- Tungsten Streamer electron resource
- 5 Axis motorized control (X=100mm, Y=120mm, Z=60mm, Tilt=0-90°, Turning=360°)
- Specimen chamber sizes: 270mm X 270mm X 250 mm
- Video-copy unit
- Easy control with joystick in all directions

4. RESULTS AND DISCUSSION

4.1. Investigation of Density

4.1.1. Investigation of Density for Al Parts

Packing densities are measured with weight-volume relation and green densities and sintered part densities are measured with Archimedes Principle.

Al Green density distribution is shown in figure 4.1., comparison of % Packing Density and Green Density of Al parts are shown in figure 4.2

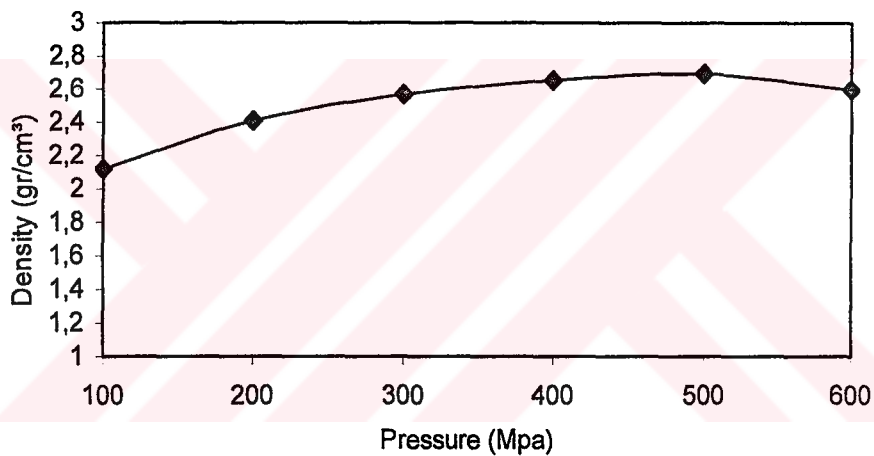


Figure 4.1. Al Green density distribution

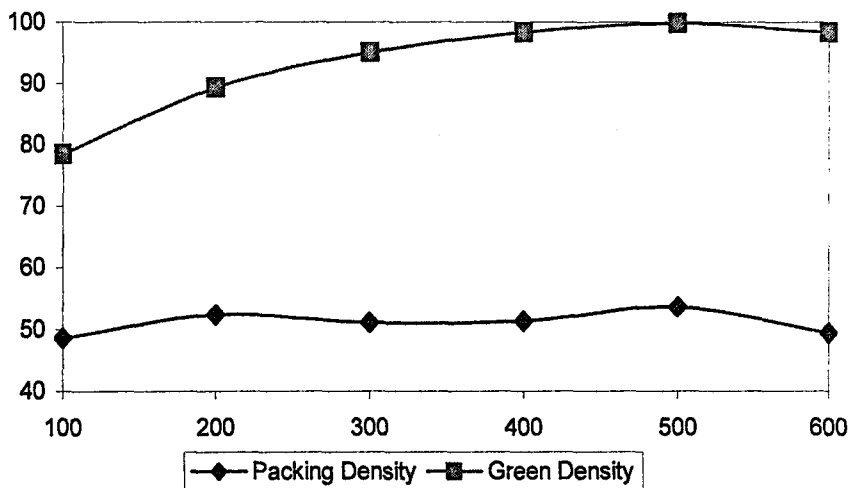


Figure 4.2. Comparison of % Packing Density and Green Density of Al parts

Green densities are measured after compaction with Archimedes Principle method. Increasing pressure causes higher density with non-sintered parts due to particle deformation and reducing pores. When pores reduce, particles are yielded and volume is decreased. Thus density is increased.

Calculated densities with Archimedes Principle method for 600, 620, 640 °C and 20 minutes sintered for Al parts are shown in Figure 4.3-4.5.

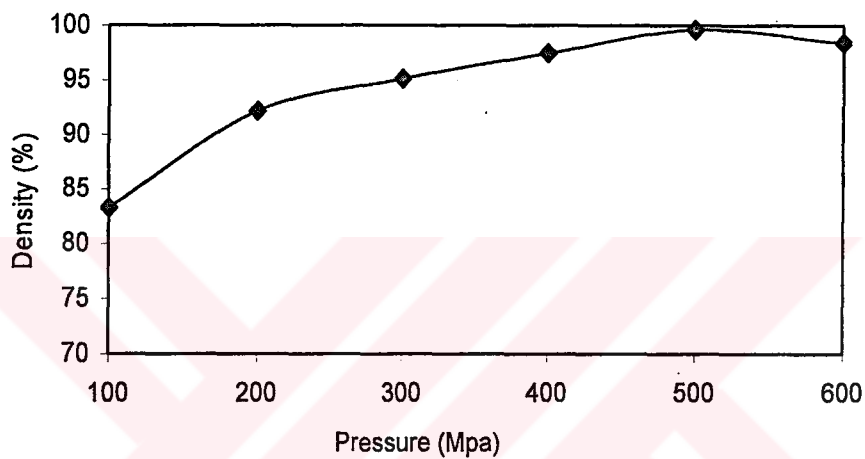


Figure 4.3. Al 600 °C/20 min. sintered density distribution

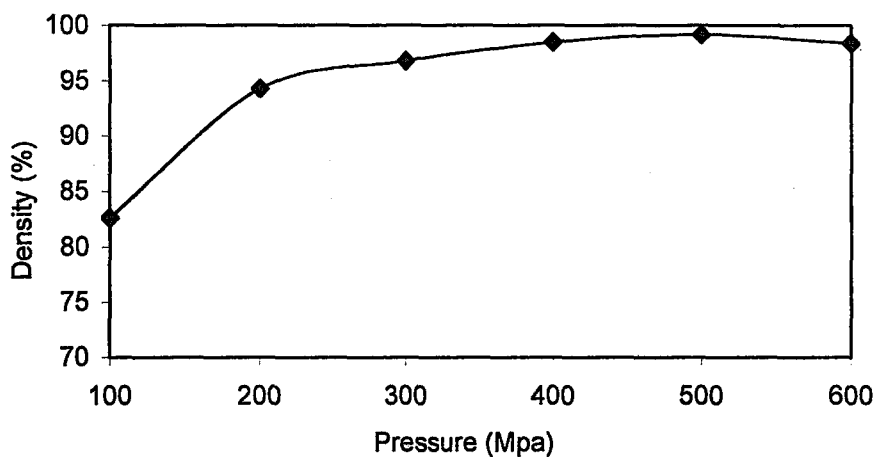


Figure 4.4. Al 620°C/20 min. sintered density distribution

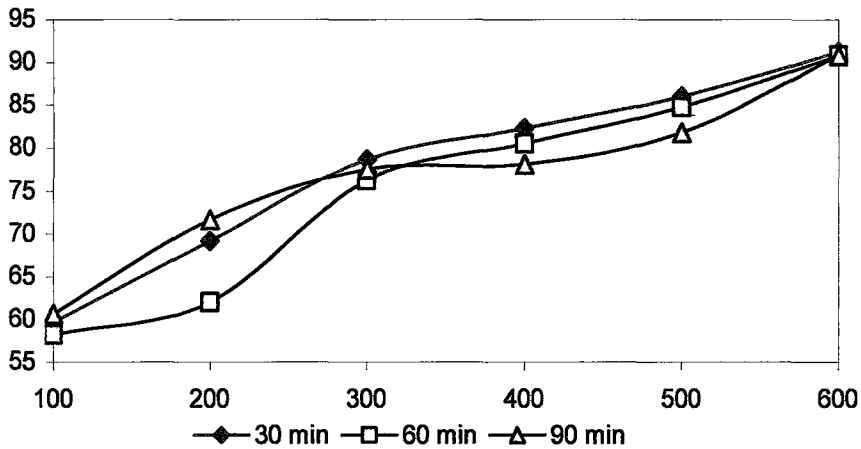


Figure 4.13. Comparison of % densities 1200 °C temperatures in 30, 60, 90 minutes sintered Fe parts

Densities increased with sintering operation due to decreasing pores, and grain necks which occurred between the particles. The maximum density, $d_{\max} = 90,2163$ is obtained with 1200 °C and 30 minutes sintering condition. The comparison of green density and density obtained at 1200 °C/30 min. sintering conditions are given in figure 4.9., in order to see the effect of sintering on the green density. Density increased only %1,74 after sintering operation with ferrous. Sintering effected with small amount to density. But, increasing pressure effected to density notably. In order to good mechanical features, density must be increased.

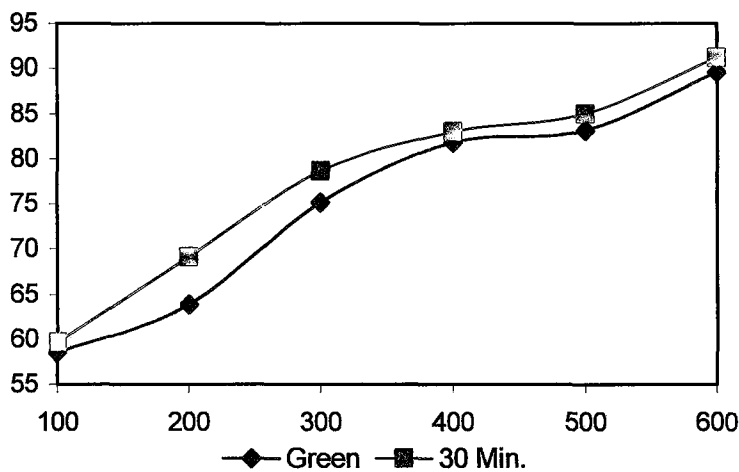


Figure 4.14. Comparison of % green density and density of 1200 °C/30 minutes sintered Fe parts

sintering operation. Density increased after sintering operation only %1,9 with aluminium.

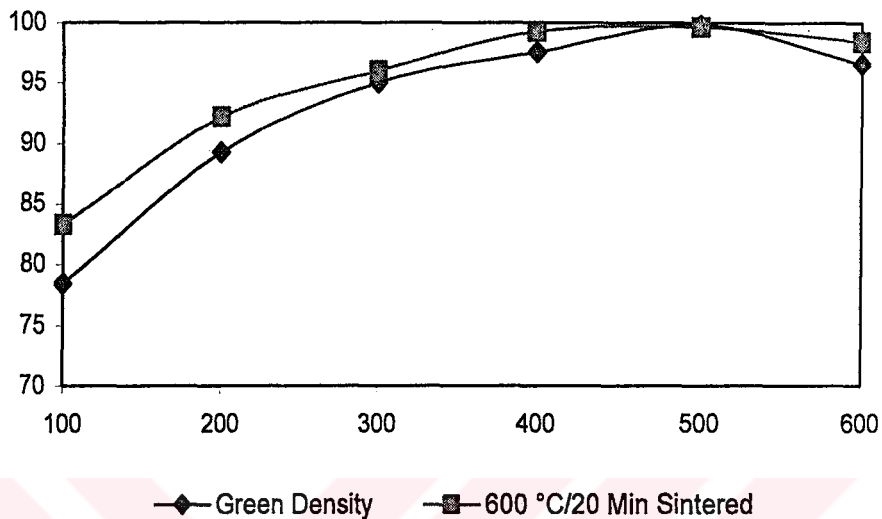


Figure 4.7. Comparison of % green density and 600 °C/20 minutes sintered Al parts

4.1.2. Investigation of Density for Fe Parts

Fe theoretical green density distribution is shown in figure 4.8. Green densities and sintered part densities are shown in figure 4.9.-4.10. for Fe specimens.

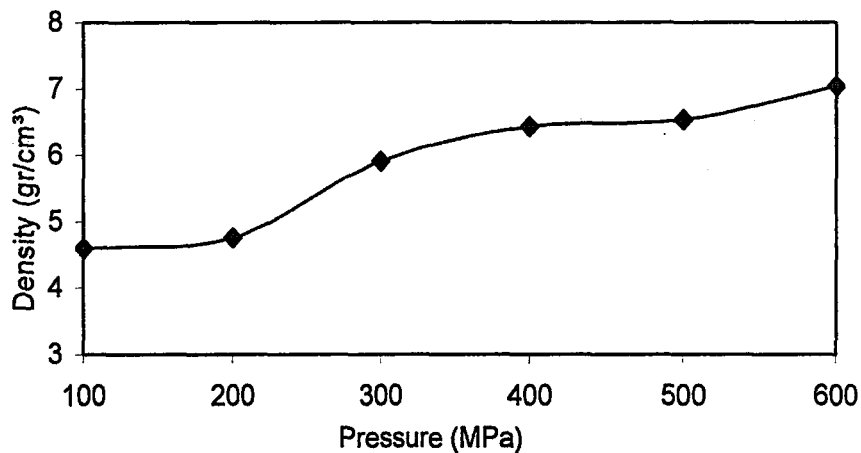


Figure 4.8. Fe theoretical green density distribution

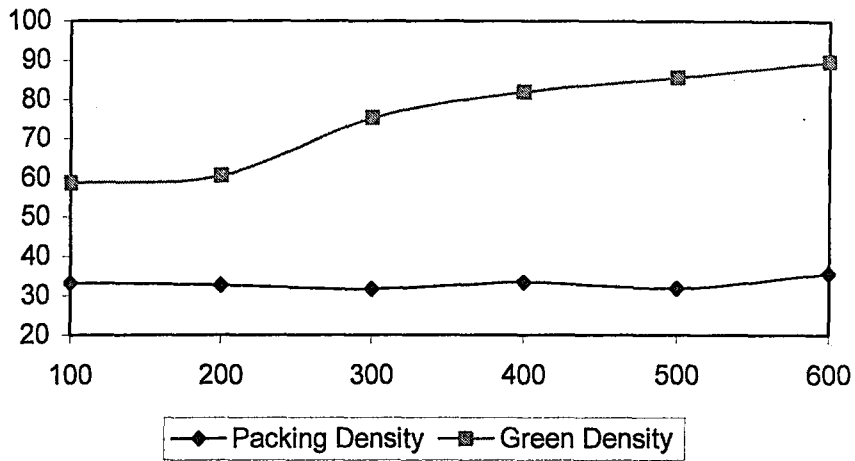


Figure 4.9. Comparison of % Packing Density and Green Density of Fe parts

The maximum density %d = 89,5848 is obtained with 600 MPa pressure. Pressure is increased notably with 300 MPa, cause particle deformation. Replacing powders and plastic particle deformation is initiated with 300 MPa pressure.

Density distributions of 1200 °C temperature and 30, 60, 90 minutes sintered Fe parts are shown in figure 4.10-4.13.

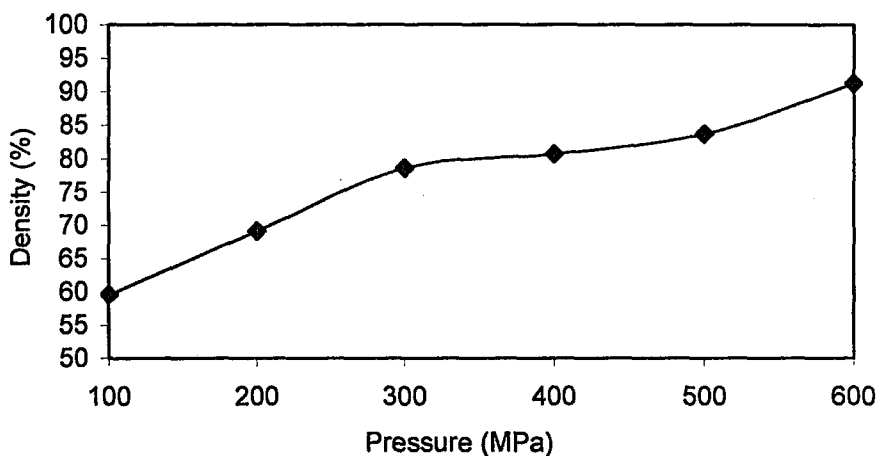


Figure 4.10. Density distribution of 1200°C/30 min sintered Fe powder parts

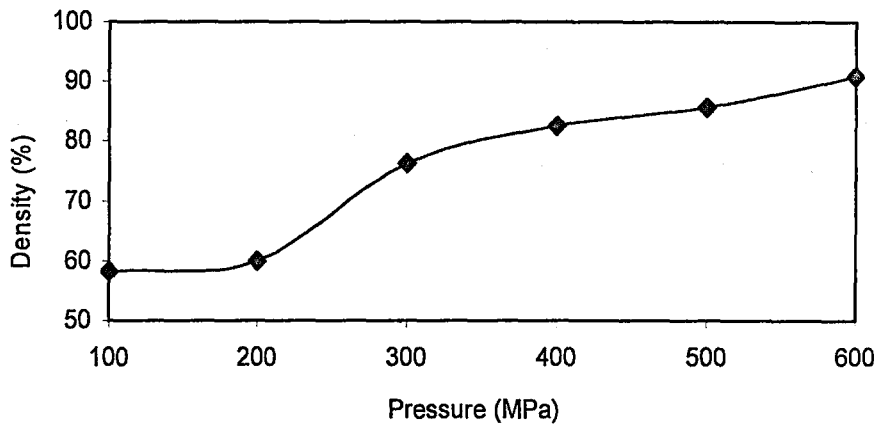


Figure 4.11. Density distribution of 1200°C/60 min sintered Fe powder parts

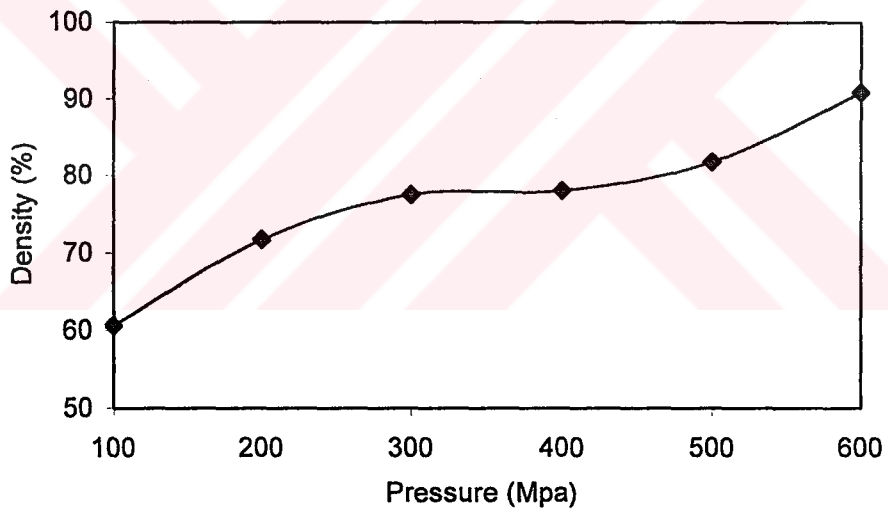


Figure 4.12. Density distribution of 1200°C/90 min sintered Fe powder parts

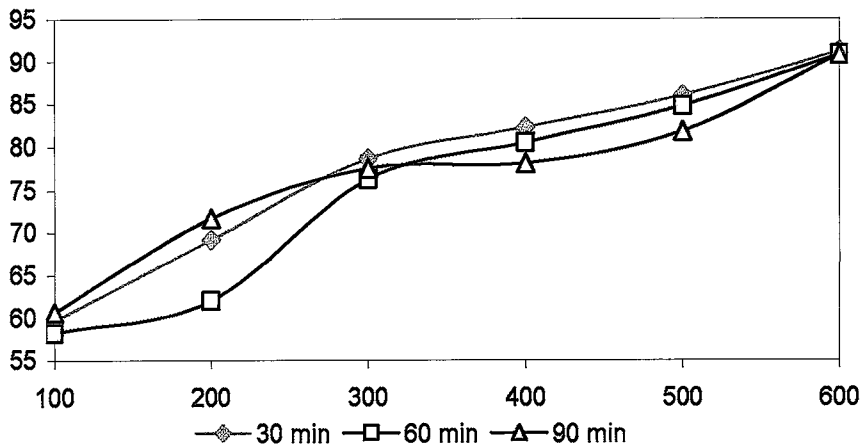


Figure 4.13. Comparison of % densities 1200 °C temperatures in 30, 60, 90 minutes sintered Fe parts

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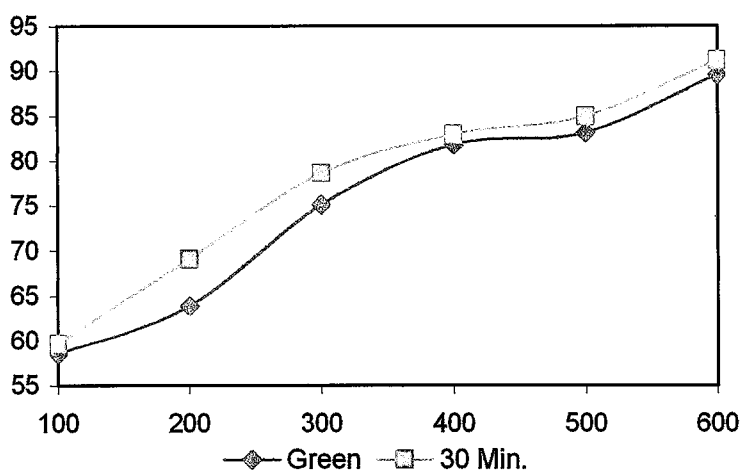


Figure 4.14. Comparison of % green density and density of 1200 °C/30 minutes sintered Fe parts

4.2. Optical Microscopy

4.2.1. Investigation of Non-Sintered Al Parts

Polished and etched Al parts photographed to investigate for this study. Before and after sintering operation, photographs are taken in 100X and 400X scales from optic microscope with video capture. General pore structures on the surface can be seen clearly with 100X scaled photographs. Structure of non-sintered Al compacted parts are shown in Figure 4.15.-4.18.

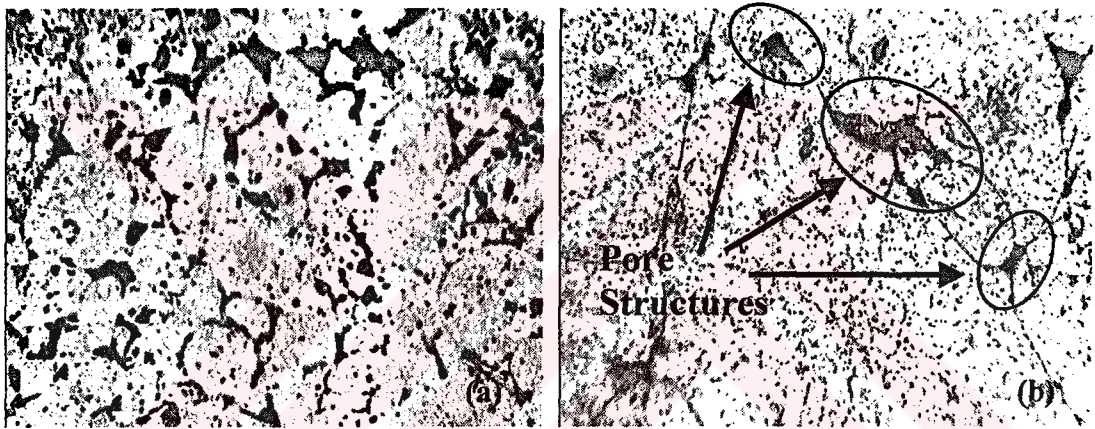


Figure 4.15. Al Non-Sintered Structure for 100 MPa Pressure (a-100X, b-400X)

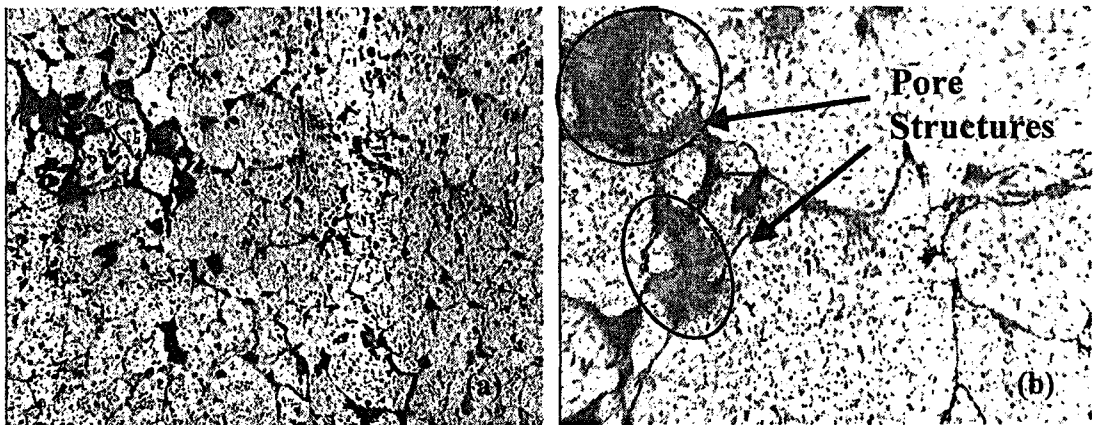


Figure 4.16. Al Non-Sintered Structure for 200 MPa (a-100X, b-400X)

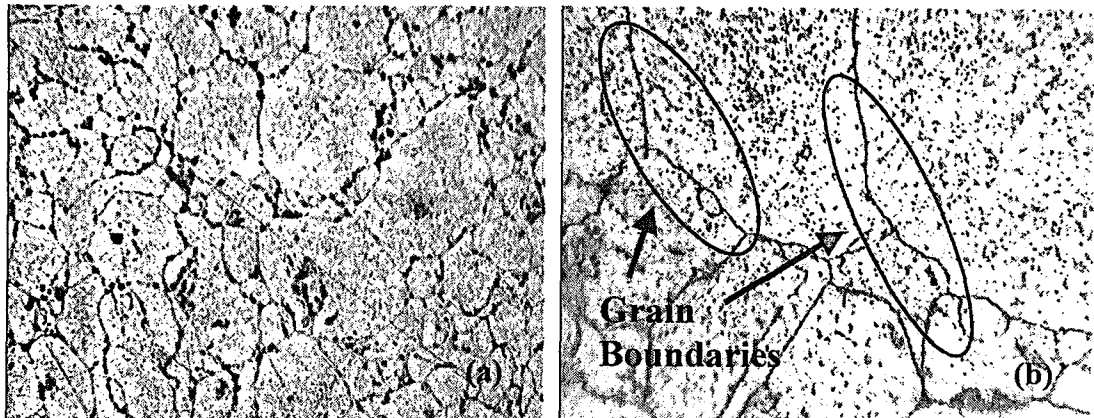


Figure 4.17. Al Non-Sintered Structure for 500 MPa (a-100X, b-400X)

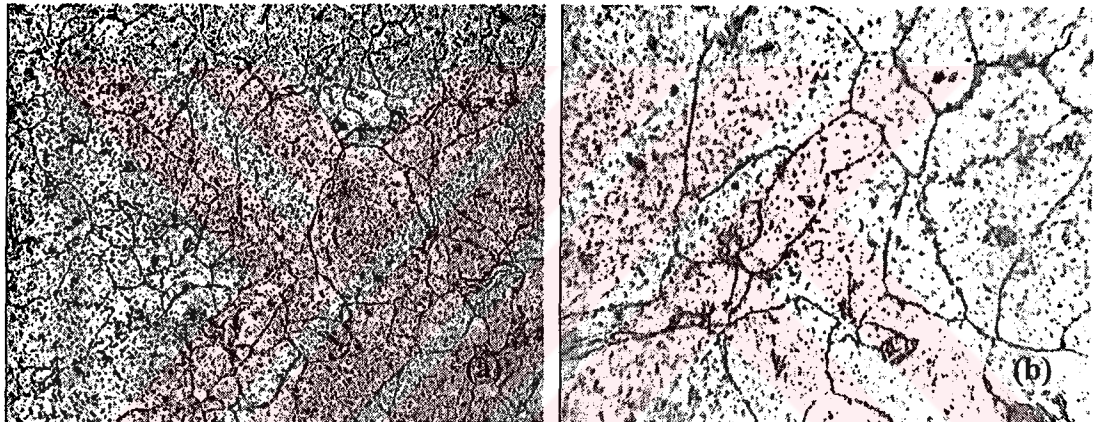


Figure 4.18. Al Non-Sintered Structure for 600 MPa (a-100X, b-400X)

As seen in the figures, structures are become homogenous and pores between the powder particles are reduced with increasing pressure. Also grain boundaries appeared notably in figure 4.17. Pore structures and grain boundaries are signed in figure 4.16-17.

4.2.2. Investigation of Sintered Al Parts

Structure of 20 minutes 600 °C temperature sintered Al specimens is shown in Figure 4.19-4.22. respectively in 100X and 400X scales. In order to show changes with structure 200, 400, 500 and 600 MPa pressures are shown.

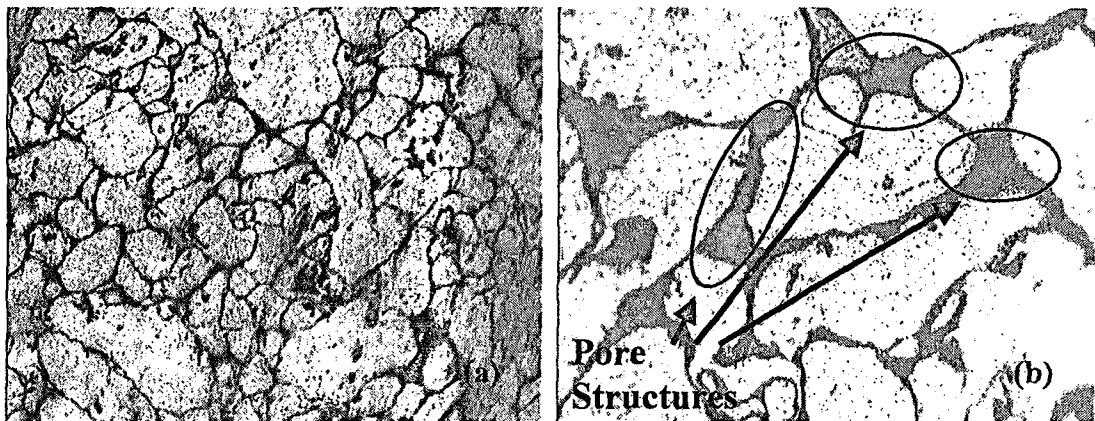


Figure 4.19. Al 20 min., 600 °C Sintered Parts for 100 MPa (a-100X, b-400X)

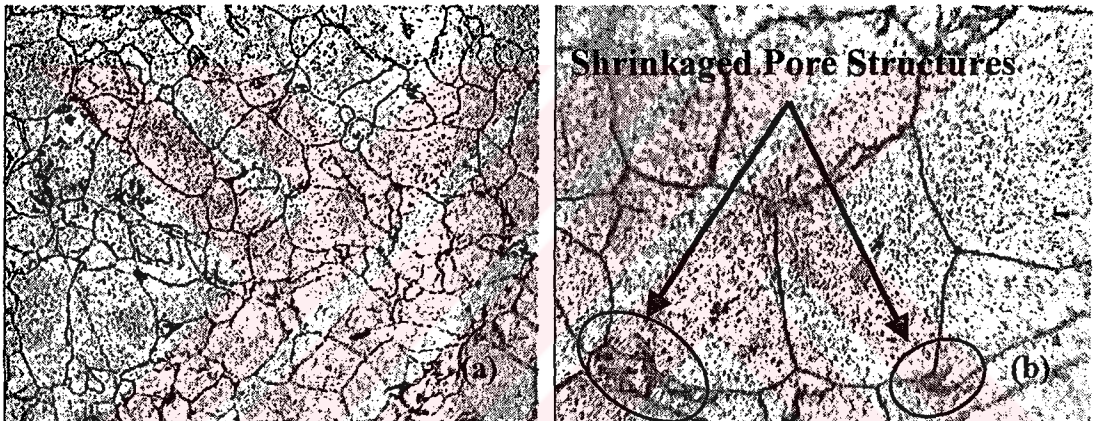


Figure 4.20. Al 20 min., 600 °C Sintered Parts for 300 MPa (a-100X, b-400X)

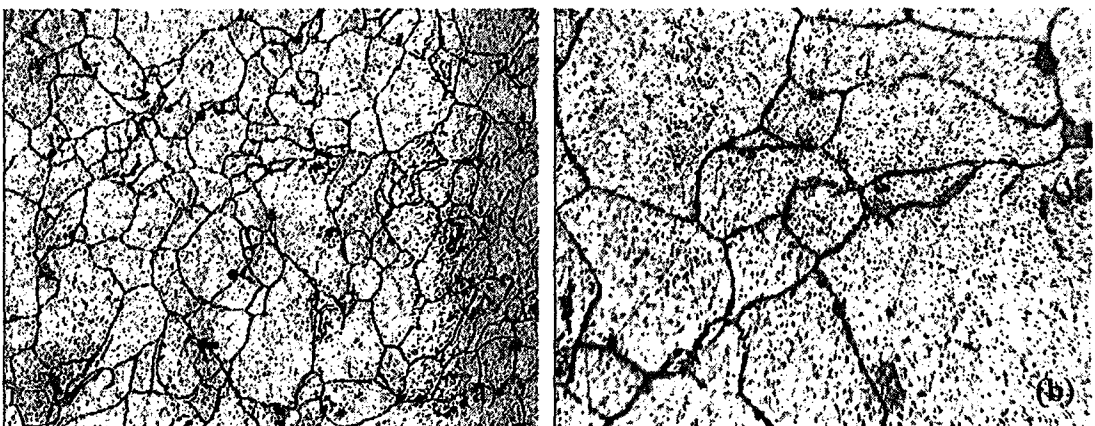


Figure 4.21. Al 20 min., 600 °C Sintered Parts for 500 MPa (a-100X, b-400X)



Figure 4.22. Al 20 min., 600 °C Sintered Parts for 600 MPa (a-100X, b-400X)

600 °C /20 minutes sintering condition, reducing and shrinkage of the pores initiated with 200 MPa in figure 4.19. Grain necks are constituted with 500 MPa notably and signed in figure 4.22.

Structure of 20 minutes 620 °C temperature sintered Al specimens are shown in Figure 4.23-4.25. respectively in 100X and 400X scales. Shown parts are pressed with 200, 500 and 600 MPa pressures.

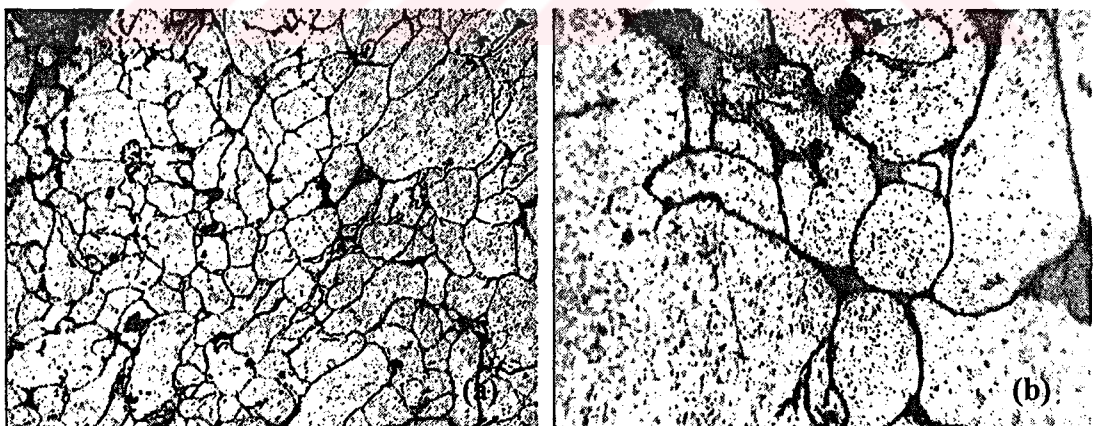


Figure 4.23. Al 20 min., 620 °C Sintered Parts for 200 MPa (a-100X, b-400X)

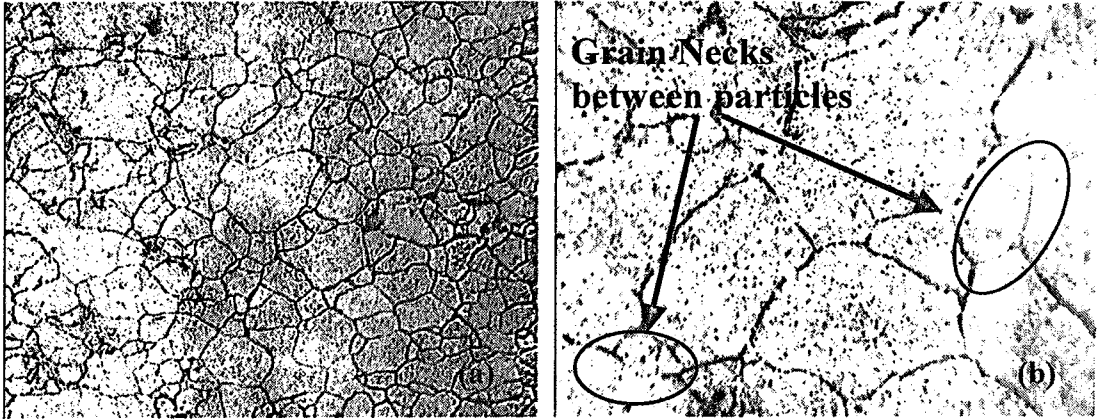


Figure 4.24. Al 20 min., 620 °C Sintered Parts for 500 MPa (a-100X, b-400X)

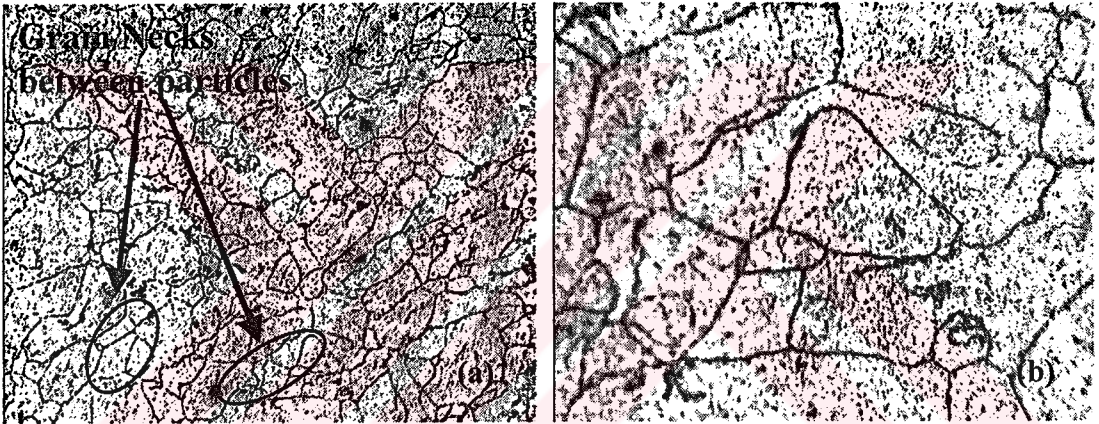


Figure 4.25. Al 20 min., 620 °C Sintered Parts for 600 MPa (a-100X, b-400X)

620 °C /20 minutes sintering condition, reducing and shrinkage of the pores initiated with 300 MPa. Grain necks are constituted notably and homogeneous structure obtained, smooth and less porosity surface can be seen in 500 MPa pressed part in figure 4.24. as signed.

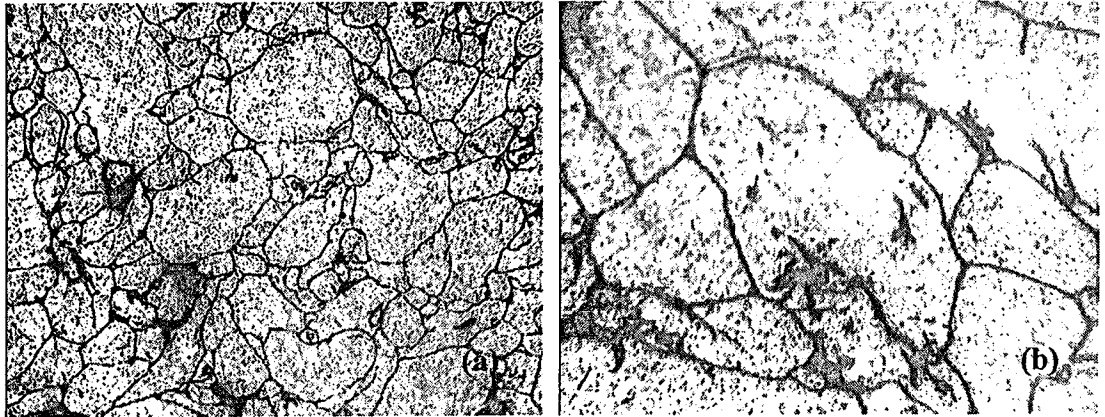


Figure 4.26. Al 20 min., 640 °C Sintered Parts for 200 MPa (a-100X, b-400X)

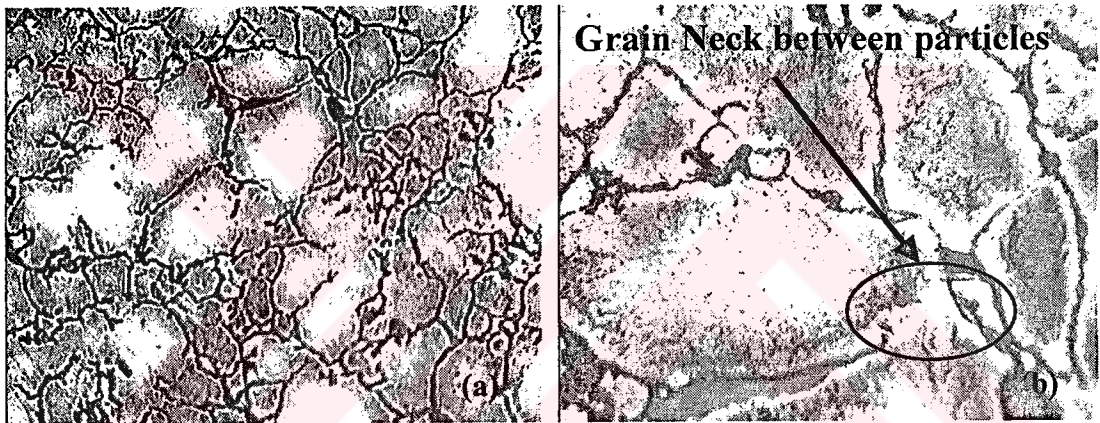


Figure 4.27. Al 20 min., 640 °C Sintered Parts for 500 MPa (a-100X, b-400X)

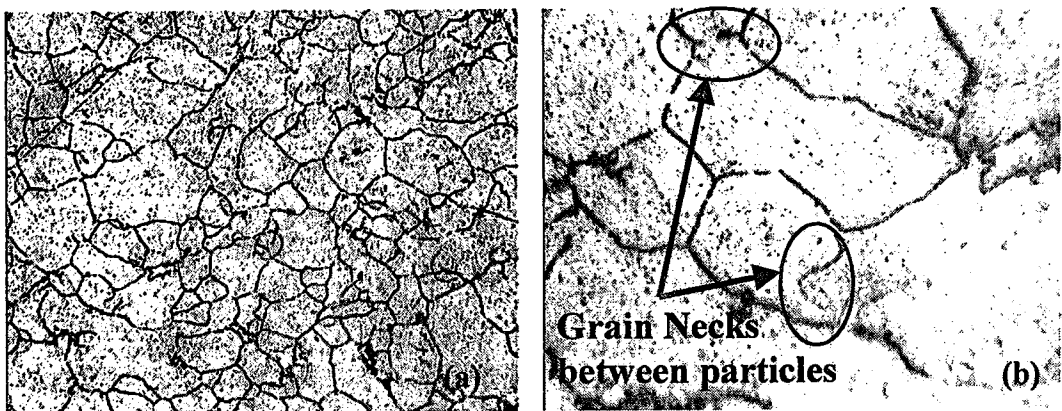


Figure 4.28. Al 20 min., 640 °C Sintered Parts for 600 MPa (a-100X, b-400X)

The Optical Microscopy examinations for aluminium powder compacts show that porosity decreased with increasing pressure, the initial shape of particles changed completely. Reduction in the volume of porosity resulted increase in the density of specimens. This is desired structure in the powder metallurgy due to the mechanical properties such as hardness, impact strength, notch strength, yield strength, toughness and durability etc. Also, after sintering operation grain sizes become larger, grain boundaries can be seen clearly, pore structures become smaller and are shrinkaged. Thus, density is increased with pressure and sintering.

4.2.3. Investigation of Non-Sintered Fe Parts

Prepared non-sintered and sintered Fe specimens for optic microscope exam investigated in this section. With 100 MPa pressure Fe compact is not managed successfully to loose powder. But in order to show porosity and effect of increasing pressure the structure, is added. Structures of non-sintered Fe parts are shown in the Figure 4.29-4.31.

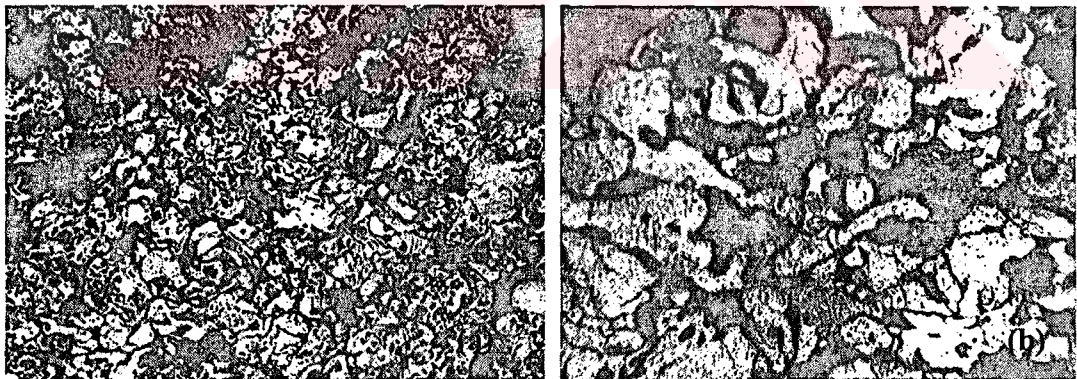


Figure 4.29. Fe Non-Sintered Parts for 200 MPa (a-100X, b-400X)

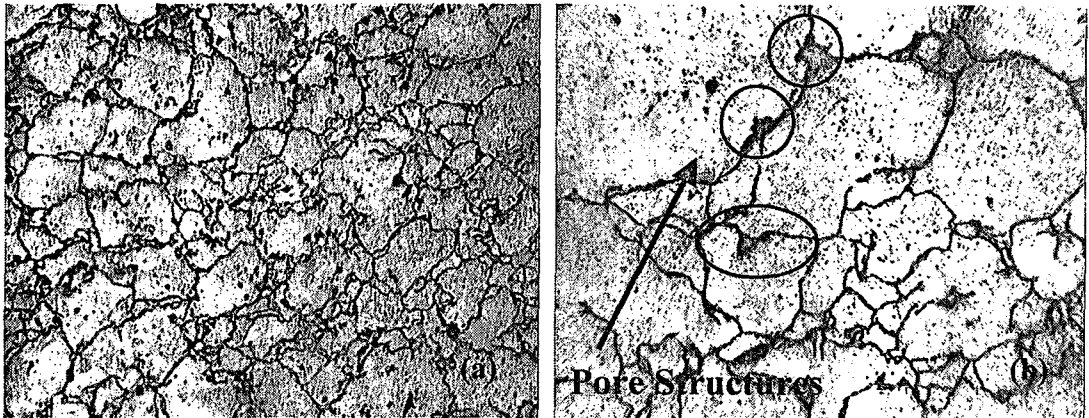


Figure 4.30. Fe Non-Sintered Parts for 600 MPa (a-100X, b-400X)

As seen in the graphics, porosity decreased increasing pressure generally. Grain boundaries are scattered due to shape of powder particle. But general structure is become homogeneous. Shrinkage of pores and grain boundaries are seen in figure 4.31. as signed.

4.2.4. Investigation of Sintered Fe Parts

Structure of 1200 °C temperature and 30 minutes sintered Fe specimens are shown in Figure 4.31-4.33. respectively in 100X and 400X scales. Shown parts are pressed with 200, 500 and 600 MPa pressures.

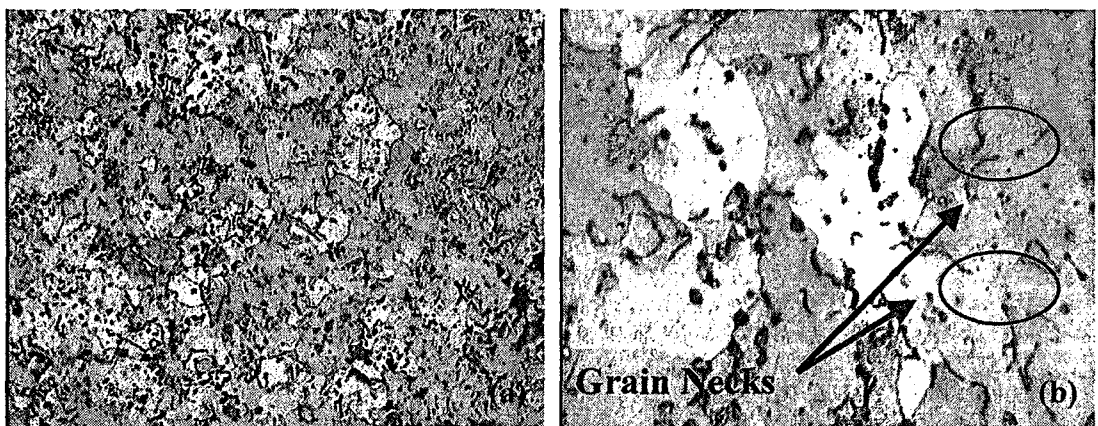


Figure 4.31. Fe 1200 °C, 30 min. Sintered Parts for 200 MPa (a-100X, b-400X)



Figure 4.32. Fe 1200 °C, 30 min. Sintered Parts for 500 MPa (a-100X, b-400X)

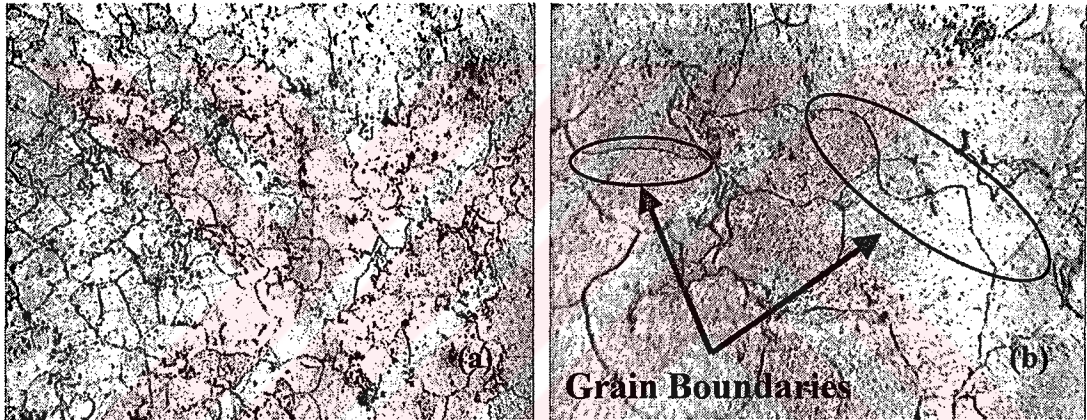


Figure 4.33. Fe 1200 °C, 30 min. Sintered Parts for 600 MPa (a-100X, b-400X)

Pores and grain necks are signed on the figures for 1200 °C/60 minutes sintered parts. Boundaries between the particles become clear and structure is homogeneous as shown in figure 4.33. Shrinkage of pores increased with high pressures.

Structure of 1200 °C temperature and 60 minutes sintered Fe specimens are shown in Figure 4.34-4.36. respectively in 100X and 400X scales. Shown parts are pressed with 200, 500 and 600 MPa pressures.

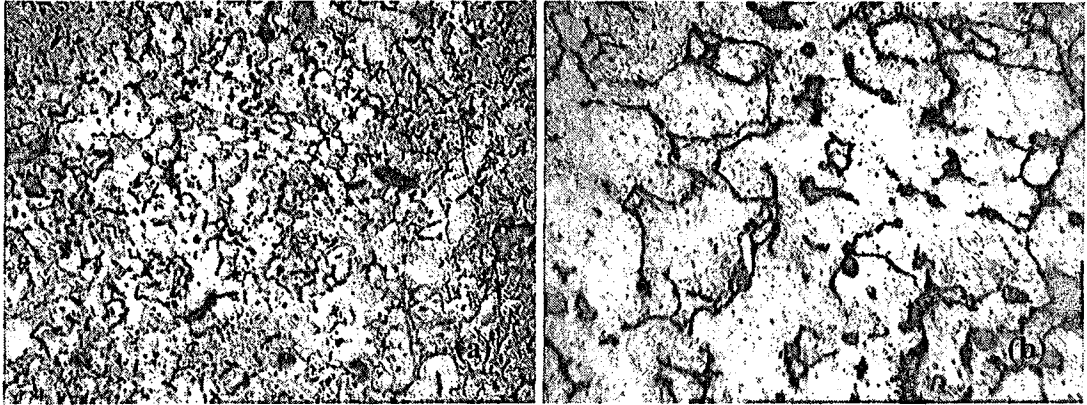


Figure 4.34. Fe 1200 °C, 60 min. Sintered Parts for 200 MPa (a-100X, b-400X)

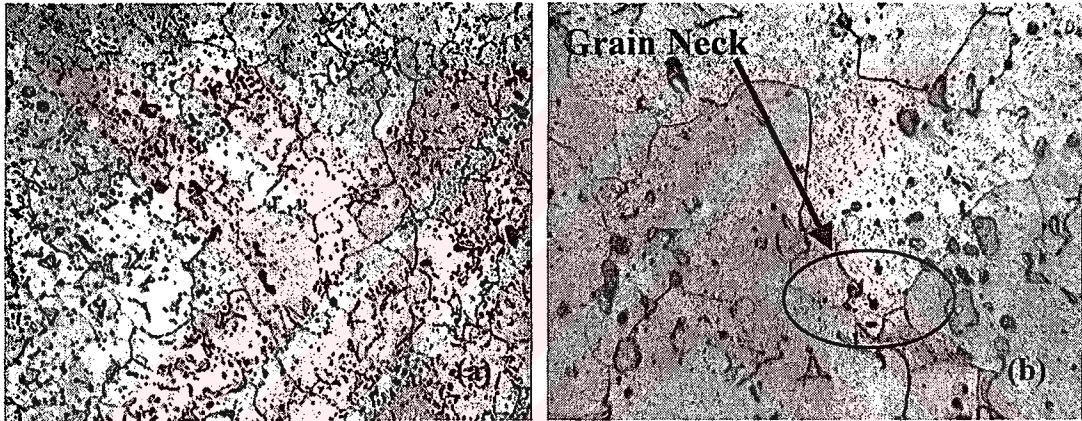


Figure 4.35. Fe 1200 °C, 60 min. Sintered Parts for 500 MPa (a-100X, b-400X)

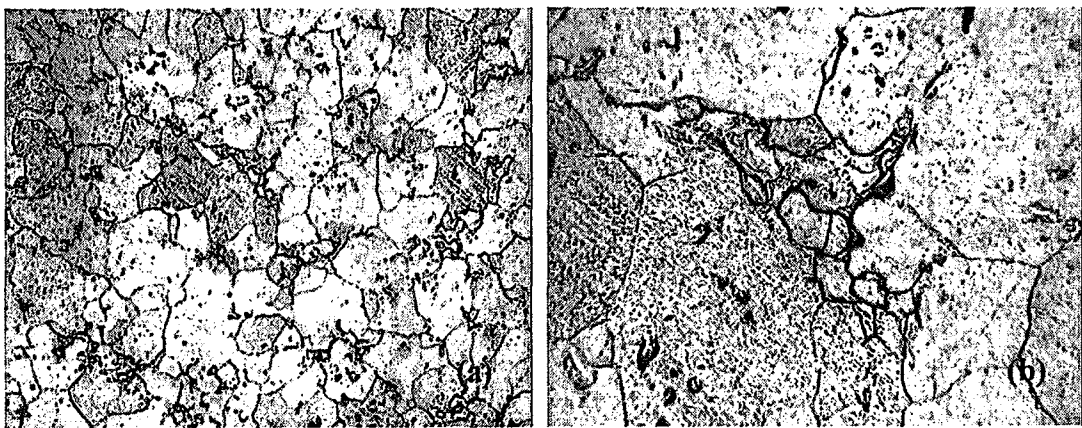


Figure 4.36. Fe 1200 °C, 60 min. Sintered Parts for 600 MPa (a-100X, b-400X)

1200 °C/60 minutes sintered Fe parts, pore structures did not change with 200 MPa in figure 4.34. But, boundaries become clear with 500 and 600 MPa pressures due to be constituted grain necks between the particles in figure 4.36-37.

Structure of 1200 °C temperature and 90 minutes sintered Fe specimens are shown in Figure 4.34.-4.36. respectively in 100X and 400X scales. Shown parts are pressed with 200, 500 and 600 MPa pressures.



Figure 4.37. Fe 1200 °C, 90 min. Sintered Parts for 200 MPa (a-100X, b-400X)

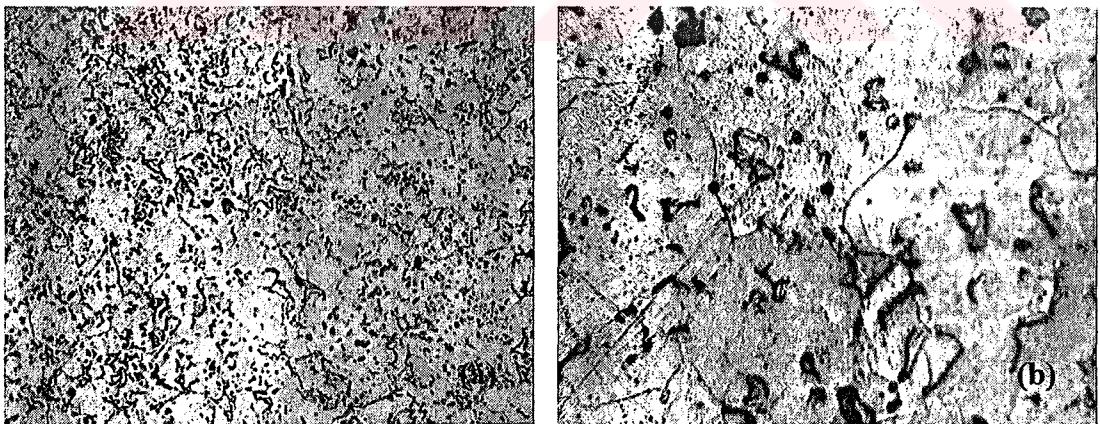


Figure 4.38. Fe 1200 °C, 90 min. Sintered Parts for 500 MPa (a-100X, b-400X)

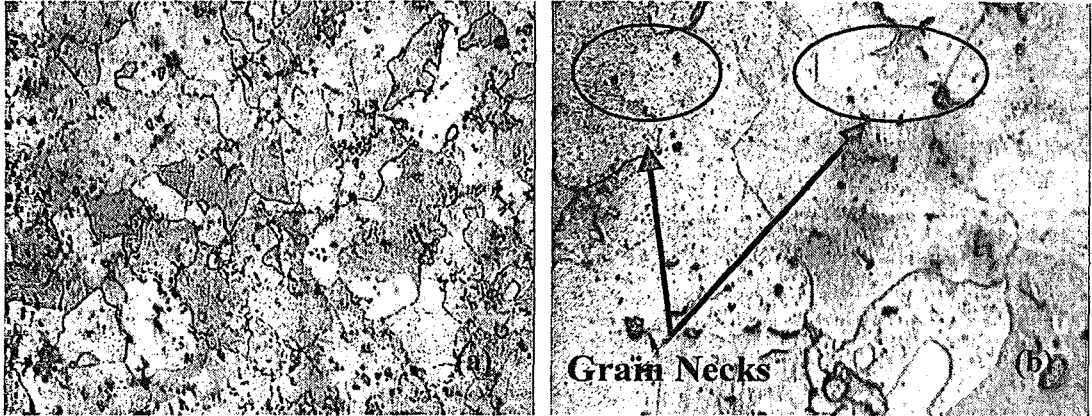


Figure 4.39. Fe 1200 °C, 90 min. Sintered Parts for 600 MPa (a-100X, b-400X)

In the Fe compaction examinations under 100 MPa no compact produced and powder loose. Density of Fe compacts increased with pressure. Porosity also decreased but not like Al compacts. The smoothest pore structure obtained with 1200 °C, 30 minutes sintering condition, thus it caused the highest density. Shrinkage of pores can be seen in this condition. In additionally, plastic deformation was initiated after 300 MPa, contact surfaces by yielding and caused local deformation due to hardness of ferrous material. In the sintered parts, grain necks are formed and can be seen clearly with high pressures.

4.3. Scanning Electron Microscope (SEM)

Scanning Electron Microscope photographs are taken at Erciyes University Technology Research and Applying Center Laboratories with LEO 440 SEM test machine. 250X, 500X, 750X, 1000X and 1500X scales are used.

4.3.1. Investigation of Non-Sintered Al Parts with Scanning Electron Microscope

Non-sintered Al specimens are researched and shown in Figure 4.41-4.43. 500X scale is used and it is enough to show the structure clearly.

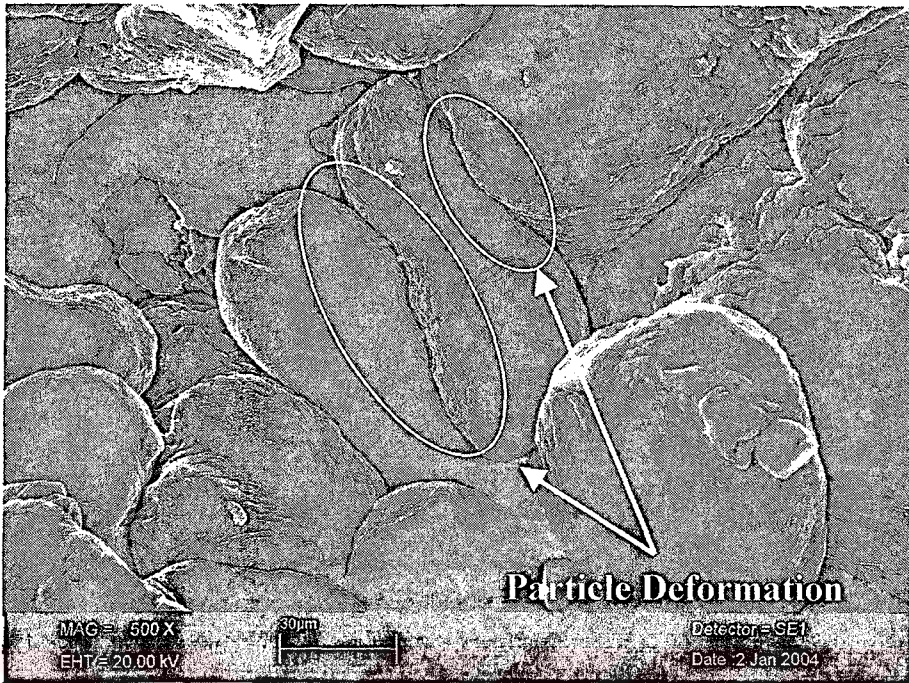


Figure 4.40. Non-sintered Al part for 200 MPa

Particle deformation is initiated even in 200 MPa due to hardness of Al powder particles. Shape of soft Al powders are changed easily and plastic deformation occurred. These changes are signed on figure 4.41.

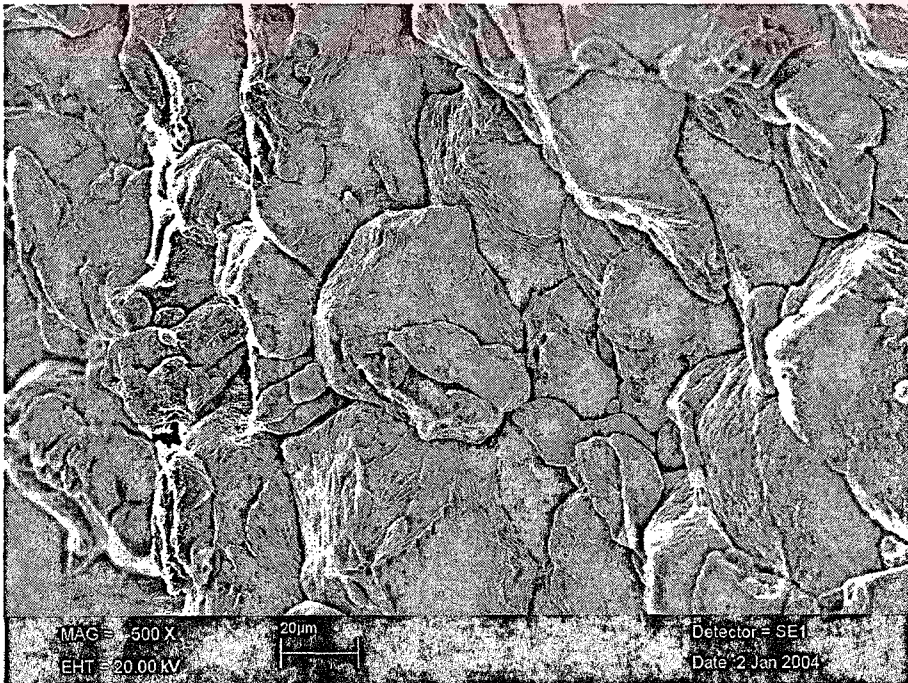


Figure 4.41. Non-sintered Al part for 400 MPa Pressure

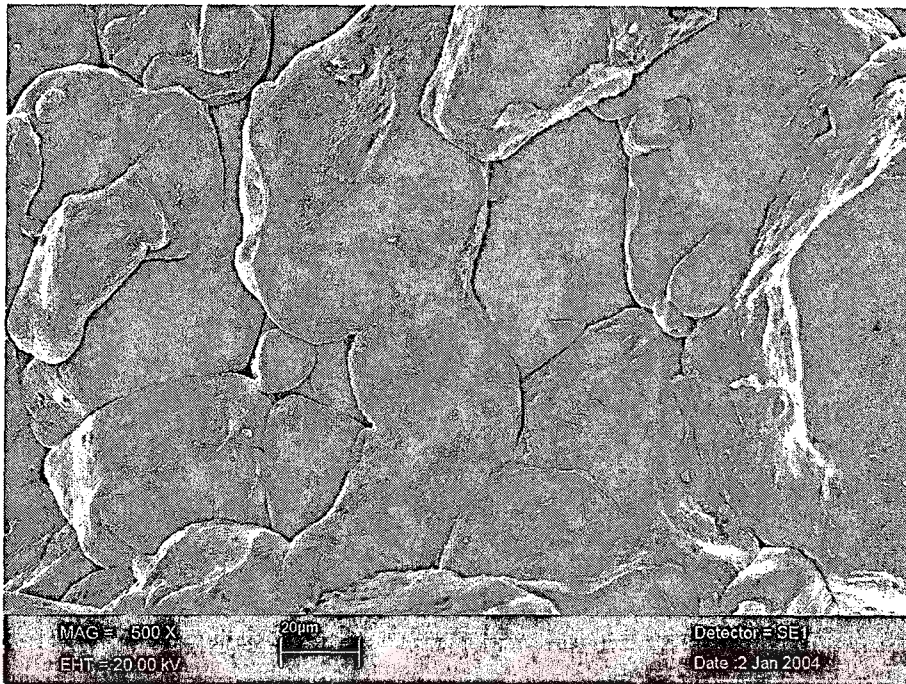


Figure 4.42. Non-sintered Al part for 500 MPa

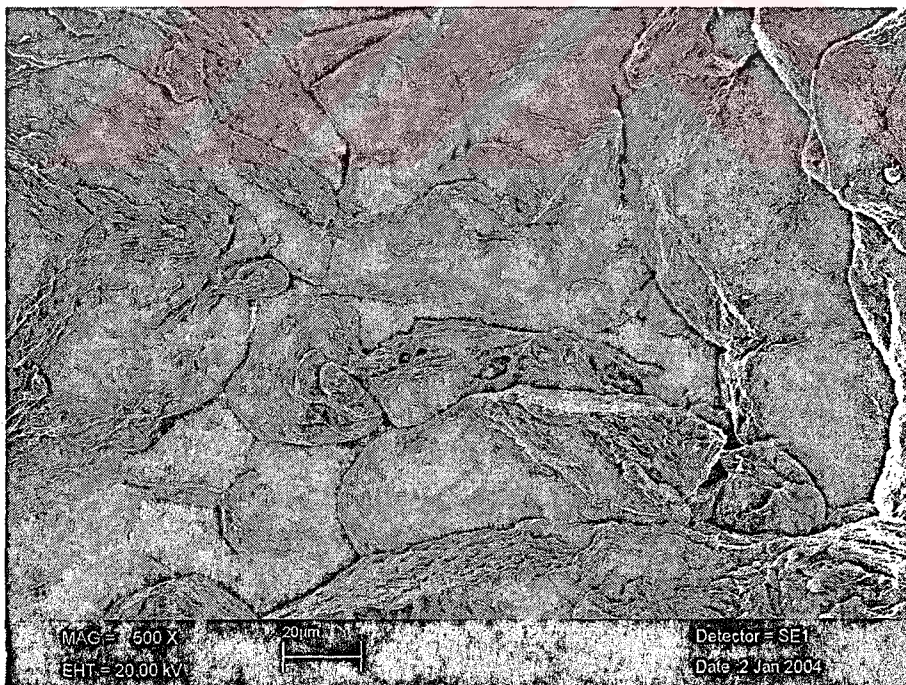


Figure 4.43. Non-sintered Al part for 600 MPa

These SEM photos show all particle deformations and effect of increasing pressure clearly. Particle deformation was initiated with 100 MPa and is appeared with 400 MPa notably. Particles are yielded and initial particle shapes disappeared and plane contacts formed. Pores decreased and plastic deformation occurred. Spherical powder particles changed their shapes and yielded. Homogeneous structure with plastic deformation can be seen in figure 4.42-43 clearly. This compaction condition is desired form in P/M process and causes full density structure. Also, hardness, strength and toughness are increase. For further properties, sintering operation must be applied to the P/M parts.

4.3.2. Investigation of Sintered Al Parts with Scanning Electron Microscope

640 °C temperature and 20 minutes sintering condition Al specimens investigated with SEM in Figure 4.44.-4.46. Differences between non-sintered and sintered parts can be seen clearly with these SEM photos.

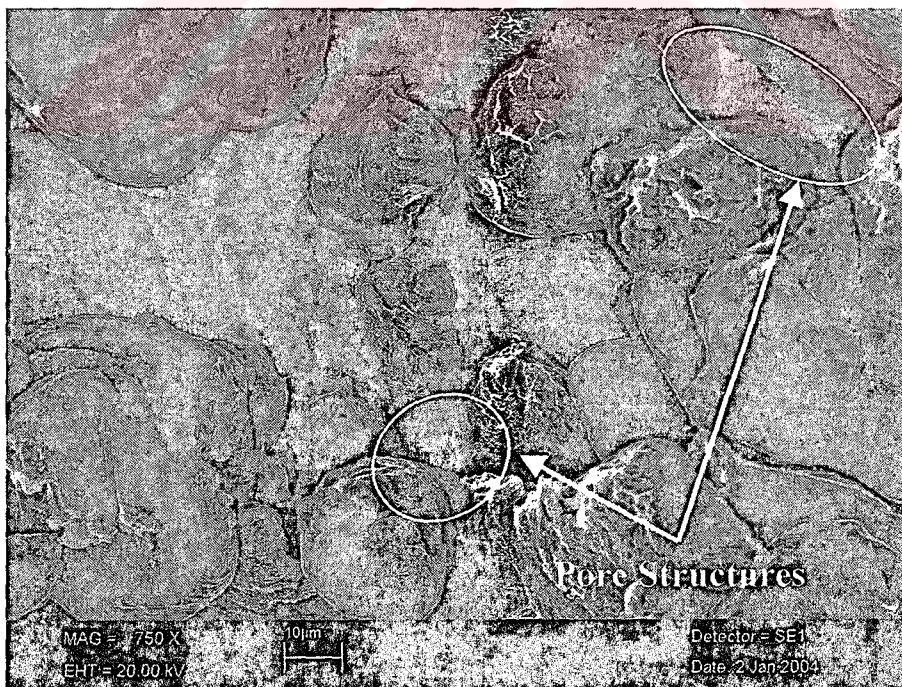


Figure 4.44. 20 minutes, 640 °C sintered Al part for 200 MPa

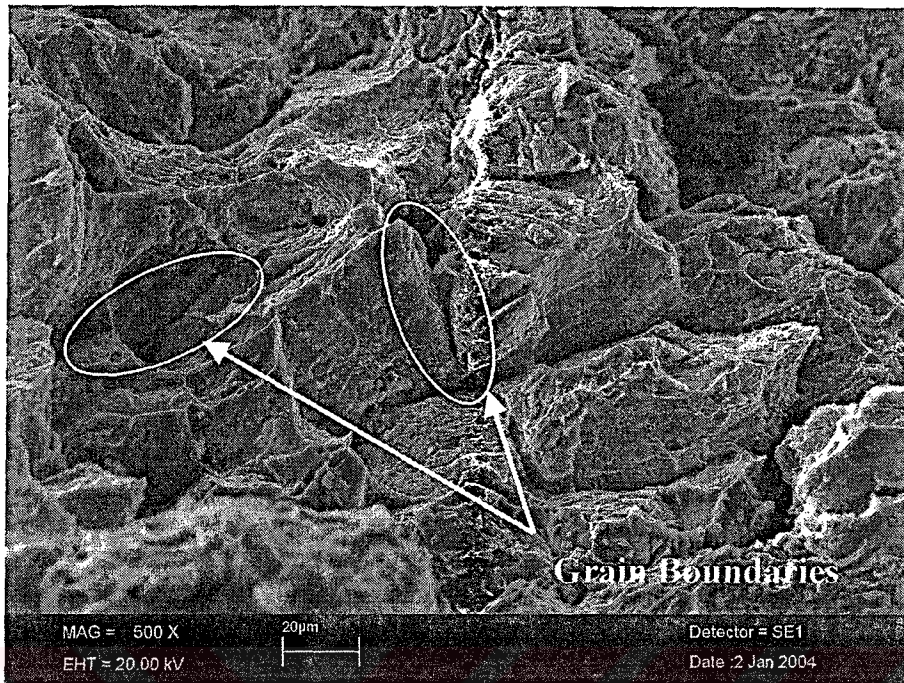


Figure 4.45. 20 minutes, 640 °C sintered Al part for 400 MPa

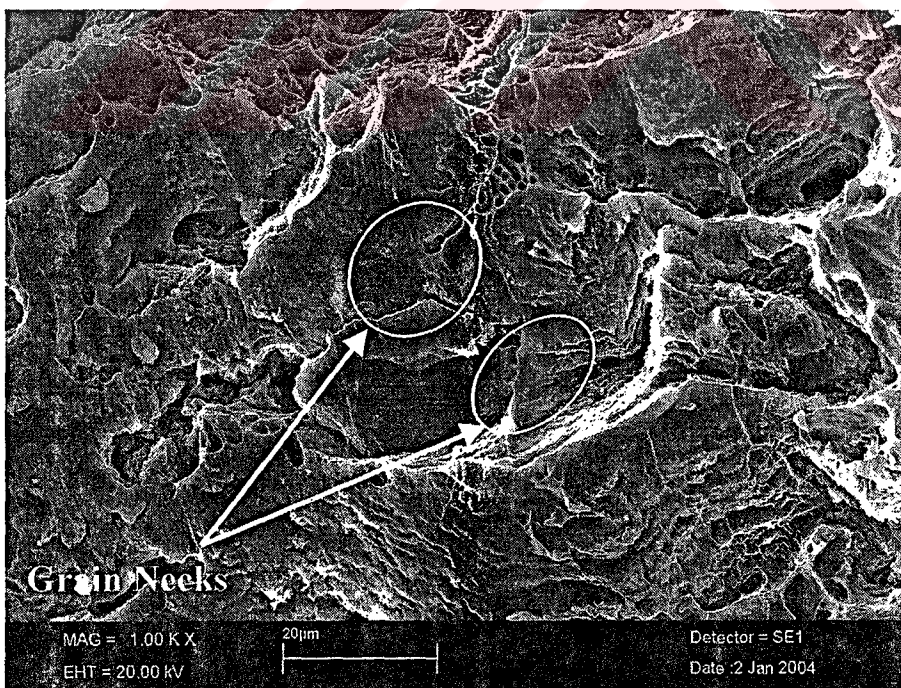


Figure 4.46. 20 minutes, 640 °C sintered Al part for 600 MPa

After sintering operation compacted parts are cracked and crack surface is researched. Particle deformation and yielding can be seen but grain necks not occurred in figure 4.44. With increasing pressure, structure of powder particles changed from spherical shape to the dodecahedron. This is an ideal structure for compacts. Grain boundaries appeared and pores decreased after 200 MPa pressure. In order to obtain best structure sintering is not enough. High pressure degrees are more effective to the structure and mechanical properties than sintering. The best structure is formed with 600 MPa pressure in figure 4.46. In this case, grain boundaries clearly disappeared and pores shrinkaged. Grain necks between the particles effected to strength due to homogeneous structure. Thus, full density is obtained and strength of compact increased. Grain necks could not occur with 200 MPa,

4.3.3. Investigation of Non-Sintered Fe Parts with Scanning Electron Microscope

Non-sintered Fe specimens investigated in Figure 4.47-4.50. 750X and 1500X scales are used and these scales are enough to see structures clearly.

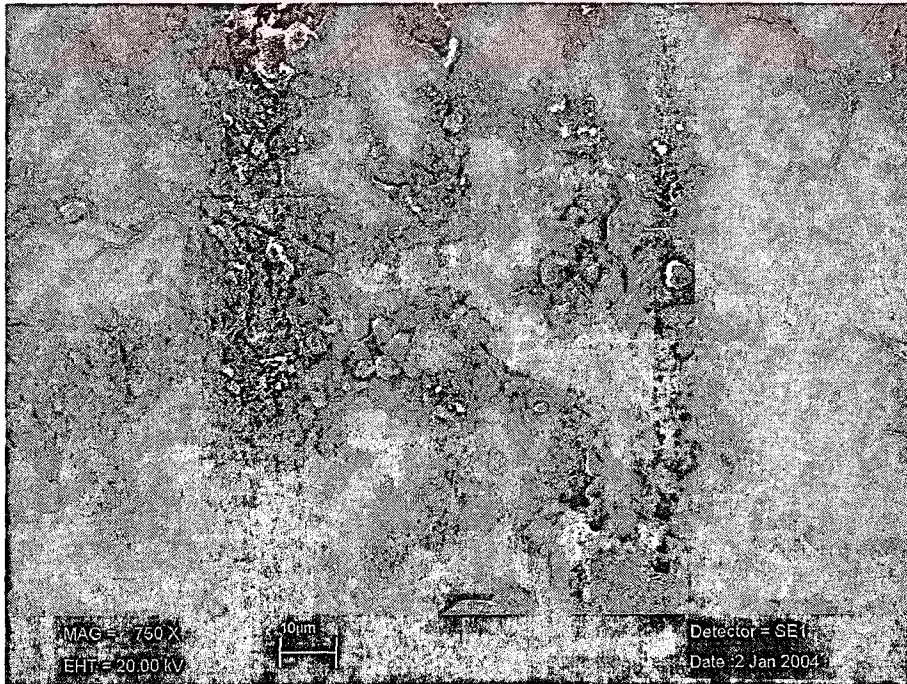


Figure 4.47. Non-sintered Fe part for 200 MPa



Figure 4.48. Non-sintered Fe part for 400 MPa

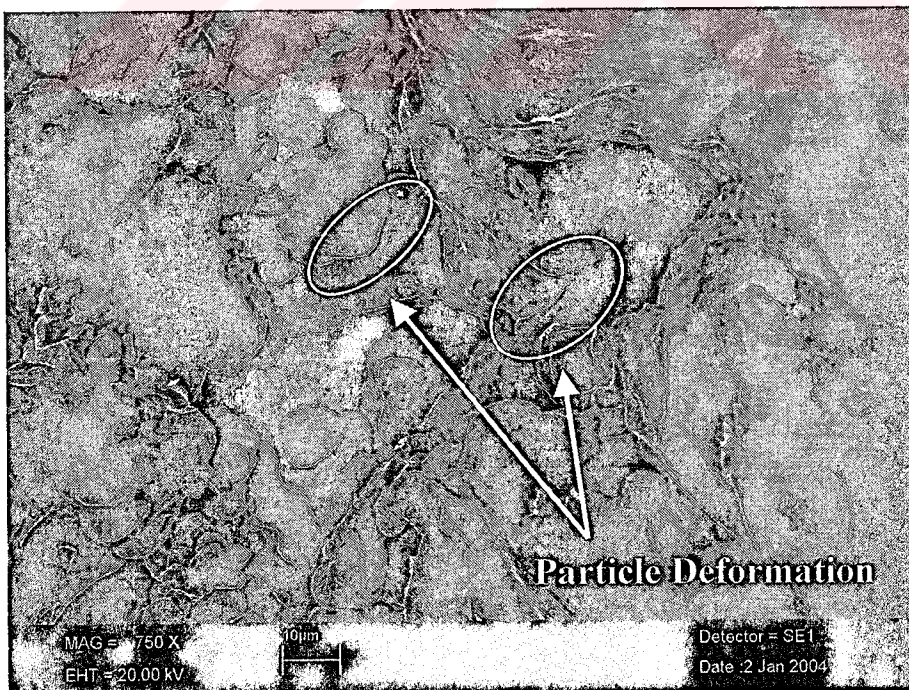


Figure 4.49. Non-sintered Fe part for 600 MPa

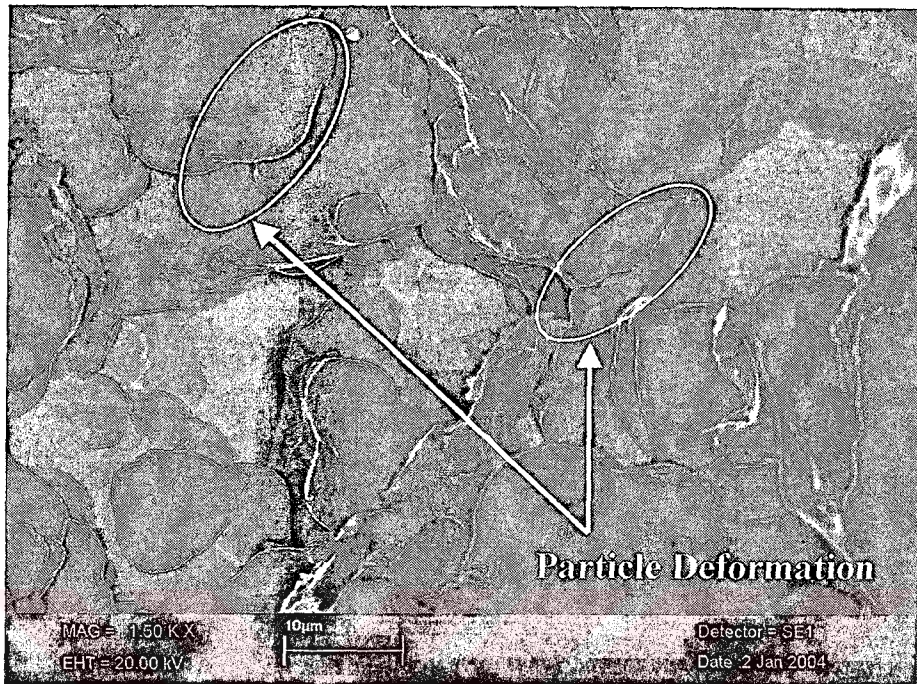


Figure 4.50. Non-sintered Fe part for 600 MPa

Powder structures differed with ferrous and aluminium. Fe powders are produced in Water Atomization Method and particle shapes are not uniform. This non-uniform structure can be seen in figure 4.47. and 4.48. clearly. With increasing pressure structure become homogeneous and pores are decreased. This structure effect the notch impact strength. Particle deformation occurred even in 200 MPa with Al compacts, but it initiated with 400 MPa with Fe compacts in figure 4.48. Applying 500 and 600 MPa pressure, plastic deformation occurred at contact surfaces by yielding and this caused local deformation. Thus, the local deformation, under excessive pressure, of the material into the neighboring voids increasing the density.

4.3.4. Investigation of Sintered Fe Parts with Scanning Electron Microscope

Photographs of 1200 °C temperature and 90 minutes sintered parts for 200, 400, 600 MPa pressures are shown in Figure 4.53-4.56.

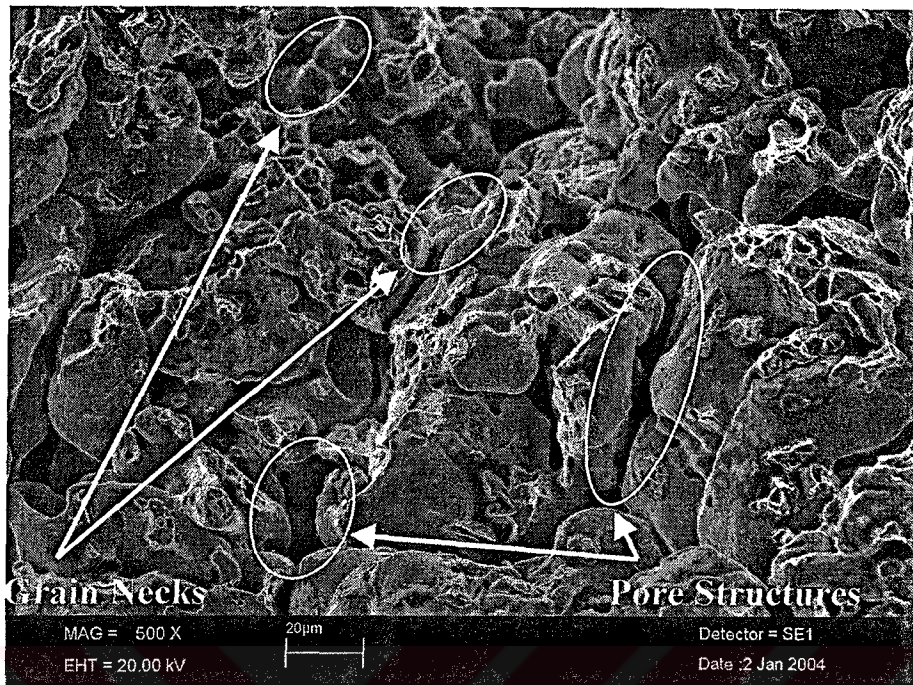


Figure 4.51. 1200 °C, 90 minutes Sintered Fe Part for 200 MPa

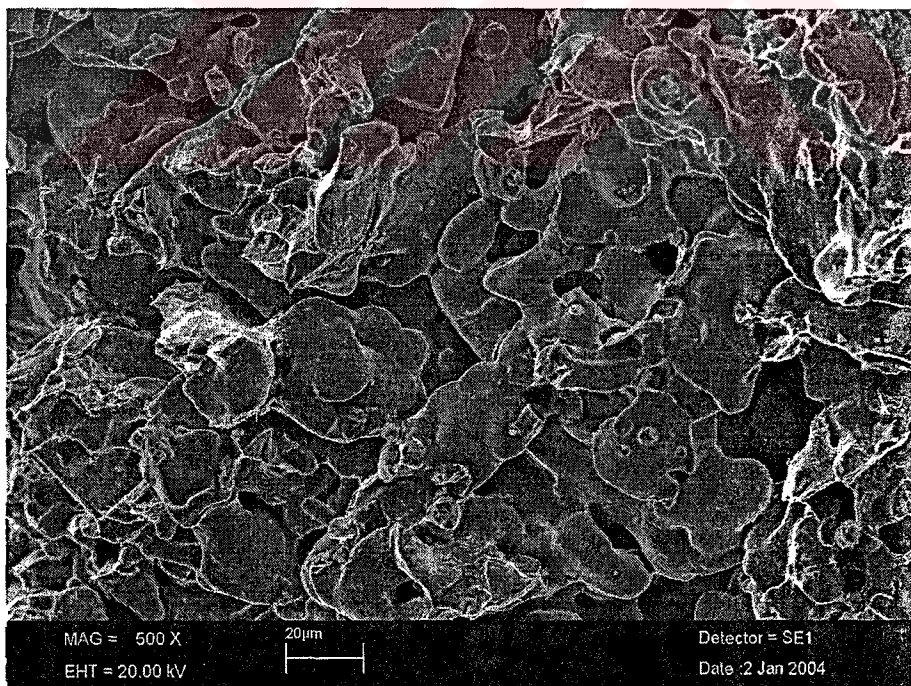


Figure 4.52. 1200 °C, 90 minutes Sintered Fe Part for 400 MPa

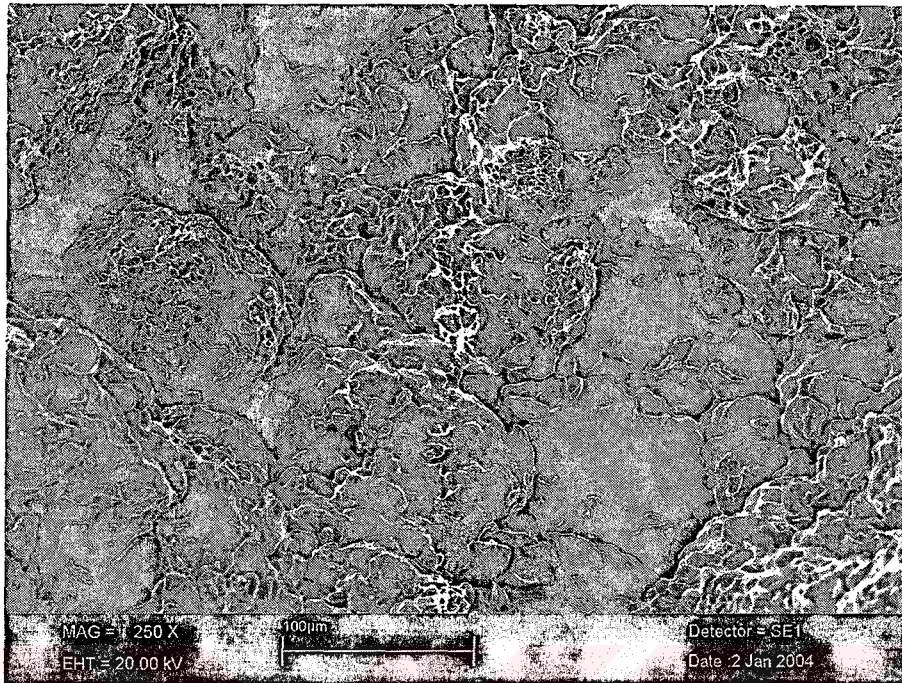


Figure 4.53. 1200 °C, 90 minutes Sintered Fe Part for 600 MPa

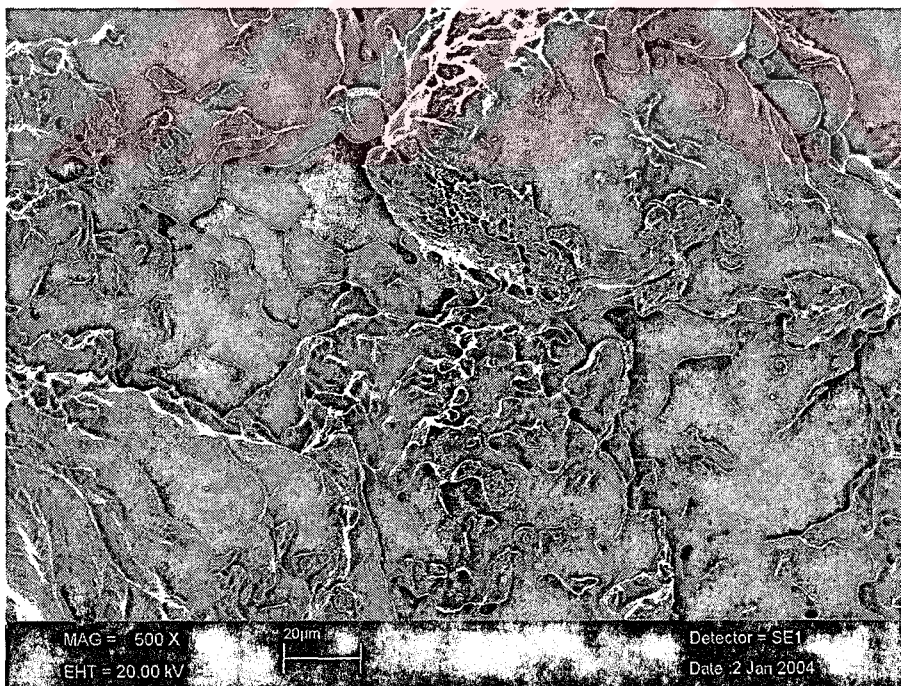


Figure 4.54. 1200 °C, 90 minutes Sintered Fe Part for 600 MPa

SEM photos illustrate grain boundaries, grain necks and pores clearly. Particles deformed plastically and homogeneity occurred, this homogeneous structure causes increasing density. And also other mechanical properties like notch impact strength, yield strength, and toughness improved. Pores can be seen with 200 MPa pressure in figure 4.53, but it decreased after 400 MPa in figure 4.54. Grain necks could occur in 400 MPa pressure and increased with sintering operation. This situation can be seen clearly in figure 4.53. After sintering operation with 400 MPa and higher pressures homogeneity increased notably. Shrinkage of pores increased also.



5. CONCLUSION AND RECOMMENDATION of FUTURE STUDIES

5.1. Conclusion

In this study, features of CIP process researched and sintering effects to the materials were investigated. Different pressures and sintering conditions are used, in order to obtain different metallurgical structures. These structures analyzed with density and microstructure. Other mechanical properties as hardness, strength, notch impact strength, yield strength, and toughness etc. could not measured due to shape of the compacted part bodies. Specimen bodies have not appropriate to measure these properties. According to these analyses, results are pointed;

- ✓ Full densification was almost obtained by using cold isostatic pressing method for in soft and easily deformed aluminium powders with 500 MPa pressure
- ✓ For ferrous powders of medium hardness and plastic behavior density of %89 is achieved,
- ✓ Maximum green density of Al parts %d_{max} : 99,81 with 500 MPa,
- ✓ Maximum green density of Fe parts %d_{max} : 89,58 with 600 MPa,
- ✓ Maximum density of Al parts after sintering operation is %d_{max}: 99,62 with 500 MPa pressure and 600 °C/20 minutes sintering condition
- ✓ Maximum density of Fe parts after sintering operation is %d_{max}:91,216 with 600 MPa pressure and 1200 °C/30 minutes sintering condition
- ✓ Porosity reduced and structures became homogeneously with arising pressure. Reducing porosity effects of the density directly. Cause, shrinkage of pores is decreased volume of compacted parts, and weight did not change. Thus, density increased. High density means that high strength of compacted parts, and it is desirable feature with powder metallurgy.
- ✓ High density also effect to mechanical properties like strength, toughness and hardness etc.
- ✓ Al compaction parts deformed notably according to the Fe parts due to differences on the hardness of powder particles. Alimunium is soft, and ferrous

is medium-soft material. Differences between the deformation on the pressed powders are shown in SEM pictures clearly.

- ✓ Etching times varied with pressures. Increasing pressure, etching time decreased. 100 MPa>20 seconds, 200 MPa>18 seconds, 300 MPa>16 seconds, 400 MPa>15 seconds, 500 MPa>12 seconds, 600 MPa>11 seconds etching time are experienced and applied for mounted Al parts,
- ✓ In order to appear necks between the powder particles sintering is not enough. Grain necks occurred with increasing pressure. Grain necks occurred in 200 MPa for Al, and 300 MPa for Fe parts,
- ✓ Structure become homogeneous with pressure due to shrinkage of pores and yielding of particles,
- ✓ Plastic deformation initiated with aluminium specimens over 200 MPa, although for ferrous initiated at 400 MPa pressure due to hardness of powders.
- ✓ Shape of the powder particles changed to dodecahedron shape,

5.2. Recommendation of Future Studies

This study has been concentrated on the effect of different pressures and different sintering conditions to the density and microstructure with wet bag Cold Isocratic Pressing technique.

In future, the following considerations could be improved:

- High pressure equipments are almost using with CIP, but expensive in operation. Costs could be reduced.
- Molding in CIP is more expensive, it may be improved.
- Initial shape differs final shape after compaction, and it causes extra operations. It may be researched.
- The air between the powder particles causes more pores, this problem solved with vacuum CIP molds, but still small amount stays.

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CIRRICULUM VITAE

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