STUDY ON THE HARDNESS AND MICROSTRUCTURE PROPERTIES OF NITRIDED FERRITIC STAINLESS STEEL POWDER AFTER SINTERING

by

Mohd Faizal Bin Muhamad Tajudin

Dissertation submitted in partial fulfillment of
the requirements for the
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(Mechanical Engineering)

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CERTIFICATION OF APPROVAL

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Approved by,

AP Dr. Patthi Bin Hussain

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
December 2010

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

MOHD FAIZAL BIN MUHAMAD TAJUDIN

ABSTRACT

A powder nitriding experiments has been conducted on AISI 430 ferritic stainless steel samples at temperatures of 700°C, 900°C and 1200°C using Carbolite Horizontal Tube Furnace in nitrogen gas flow. The main objective of the research is to investigate the hardness and microstructure properties of AISI 430 ferritic stainless steel powder and to improve the hardness and microstructure properties of ferritic stainless steel powder by nitriding process. The treatment time was 1 hour and 3 hours. The structure and composition of the nitrided surface layer were analyzed by optical microscope. Also, the hardness technique was used for testing the surface hardness of the nitrided samples. Effect of nitrogen on the surface hardness and microstructure of the treated samples was studied. The hardness increases with increasing of nitriding temperature. Also, by increasing nitriding time can improve the hardness of the samples. The nitrided phase, which are improved in terms of compactness of material particles and less pores area were observed.

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CHAPTER 1: INTRODUCTION

1.1. BACKGROUND OF STUDY

Nitriding is one of diffusion treatments applied to the surface of machine parts, tools, and other metallic objects to enhance their surface hardness and to improve a number of other useful properties. Founded in 19th century, a surge of interest in the possible hardening of ferrous surfaces, as well as in resulting industrial applications, occurred only in 1900.

In the beginning of industrial implementation, the nitride layers were obtained in ammonia atmosphere, in the course of a long process, on parts manufactured from special nitriding steel. The introduction of the concept of nitriding potential K_N, representing the nitriding capability of an atmosphere, began the period of development of controlled gas nitriding in NH_3-N_2 and NH_3-dissociated NH_3 mixes. Between 1970 and 2000, a number of further modifications of gas, salt bath, and plasma nitriding processes have appeared, aiming at improving both the properties of treated materials, as also the technological advantages of the process itself.

Recently, high-temperature gas nitriding (HTGN) or solution nitriding was introduced as a method for adding nitrogen to stainless steels. This new nitrogen addition method involves a diffusion process for nitrogen to permeate the surface of stainless steel through heat treatment in N_2 atmosphere at high temperatures [1–3]. In general, HTGN has usually been applied to Cr based austenitic, martensitic and duplex stainless steels,which have high nitrogen solubility in the austenite phase. However, the low nitrogen solubility of a ferrite single phase prevents HTGN of ferritic stainless steel. Ferritic stainless steels were adopted to use in the manufacture of engine mufflers, nuts, bolts and heat resistant tools. In some cases, such steels require high surface hardness. In this study, the effect of a nitriding treatment and the tempering of 430 stainless steel powder were examined as a function of the surface phase changes.

1.2. PROBLEM STATEMENT

As explained above, the purpose of nitriding is to investigate the hardness and microstructure properties of ferritic stainless steel powder. The main factor that enhances those properties is the diffusion of nitrogen into the powder. However, the nitrogen will not be uniformly distributed until the core of the steel if the nitriding process is done on the bulk surfaces. Thus, the diffusion of nitrogen is limited to the surface of the material only. The diffusion of nitrogen is therefore become important properties in this heat treatment. By nitriding the powder of ferritic stainless steel, the diffusion will be entered to the core of the steel and uniformed throughout the material.

1.3. OBJECTIVES AND SCOPE OF STUDY

The objectives of the research are:

- To investigate the hardness and microstructure properties of ferritic stainless steel.
- To improve the hardness and microstructure properties of ferritic stainless steel by nitriding process.

As this project is classified under material engineering field the main idea for creating this project will not escape from material study. The scope of study for the research is to study on the hardness and microstructure properties of ferritic stainless steel when involving the nitriding process. It is a need to know the improvement of both properties by doing hardness and microstructure test after nitriding process at certain variable is done.

CHAPTER 2: LITERATURE REVIEW

2.1. INTRODUCTION

In chemistry, a **nitride** is a compound of nitrogen with a less electronegative element where nitrogen has an oxidation state of −3. Nitrides are a large class of compounds with a wide range of properties and applications.

There are several other attributes, which makes the used of nitriding stainless steel favorable compared to the more conventional alloys. Some of these are; (i) high yield strength, tensile strength, and ductility, (ii) high strength-fracture toughness combination, (iii) high strain hardening potential, (iv) low magnetic permeability and (v) favorable corrosion properties (M. Adithan., A.B. Gupta, 2007).

Recent years have seen a rapid development of nitriding stainless steel with improved properties. The combination of number of properties such as strength, fracture toughness, wear resistance, magnetic properties and corrosion resistance have given a unique advantages and offers a number of prospective applications today in many industries and engineering applications (P. N. Rao, 2009).

2.2. FERRITIC STAINLESS STEEL

The structural form of pure iron at room temperature is called ferrite or α-iron. Ferrite is soft tile. Since ferrite has a body-centred cubic structure, the inter-atomic spaces are small and pronouncedly oblate, and cannot readily accommodate even a small carbon atom (P N Rao, 2009). Therefore, solubility of carbon in ferrite is very low, of the order of 0.006% at room temperature. The maximum carbon content in ferrite is 0.05% at 723°C. In addition to carbon, a certain amount of silicon, manganese and phosphorous may be found in ferrite (Serope Kalpakjian., Steven R. Schmid.,2007).

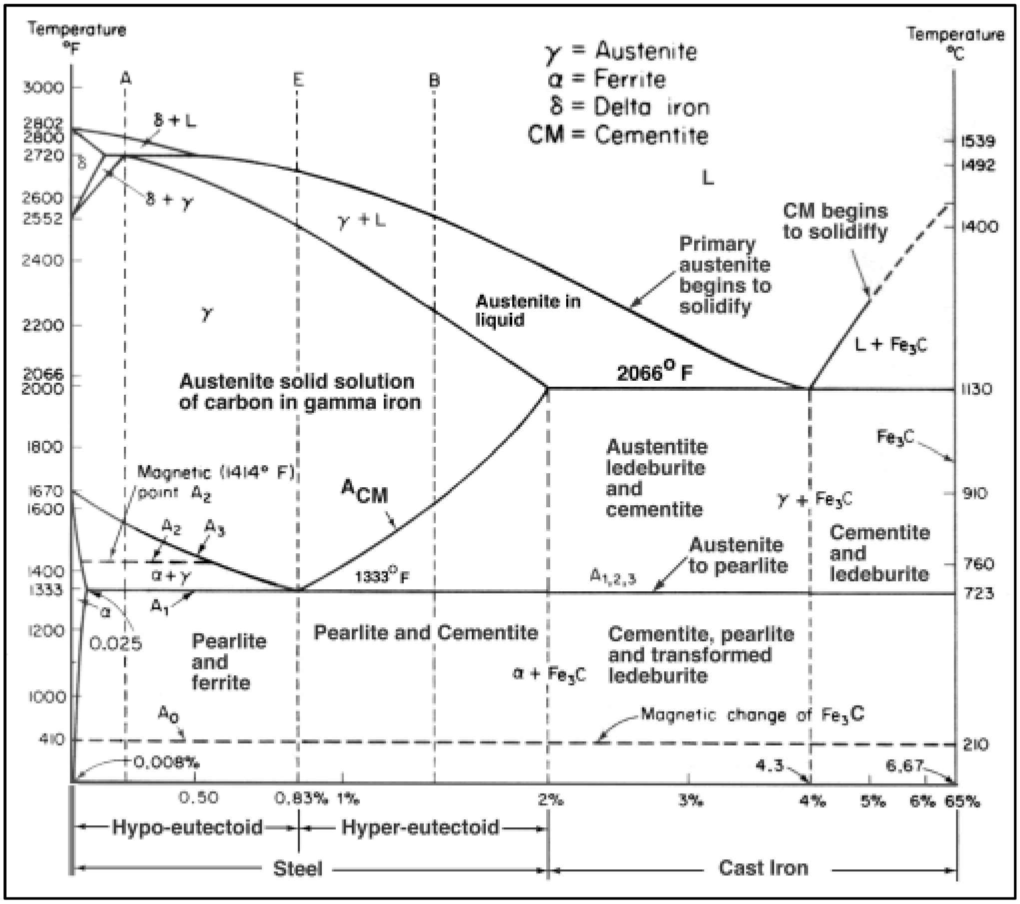


Figure 1.1. Iron-carbon Equilibrium Diagram. Reference: P N Rao, 2009.

Ferritic stainless steels are straight-chromium 400 series grades that cannot be hardened by cold working and are not heat treatable. They usually have a chromium content of between 10 to 27% and lower carbon contents than the martensitic stainless steels. Ferritic stainless steels are magnetic, have good ductility and resistance to corrosion and oxidation but have lower ductility than austenitic stainless steel. It is stronger than carbon steel and is utilized in applications where thinner materials and reduced weight are advantageous. They are often specified due to their superior corrosion resistance and resistance to scaling at elevated temperatures. Generally, it is used for nonstructural applications, such as kitchen equipment and automotive trim (M. Adithan., A.B. Gupta, 2007).

2.3. NITRIDING

Nitriding, enhancing corrosion resistance on most steels, impairs it on stainless steel. This is because chromium, being a strong nitride-forming element, readily combines with diffusing nitrogen, thus depleting the steel matrix of its chromium content in solution, necessary for the formation of a protective film. Alleviation of this affect can be obtained by nitriding at low temperatures, involving, however, long processing times. Nitrided layers on precipitation-hardening steels exhibit similar features to those a martensitic stainless grades.

Stainless steel is nitrided mainly to enhance wear resistance in cases where non-ferromagnetic properties are required but where corrosion resistance is not a major requirement. Gas nitriding at 350°C (662°F) produces in 304 steel a total nitriding depth of 5 µm after 24 hours, at the same temperature plasma nitriding produces 7.5 µm after 16 hours (E. Menthe., A. Bulak., J. Olfe., A. Zimmermann., K.-T. Rie., 2000).

Faster nitriding rates, and deeper cases, can be obtained through a high temp process (1050-1150°C, 1922-2102°F) in atmosphere containing a high proportion of molecular nitrogen. The proposed name for the process is **solution nitriding**. At this temperature level, thermal dissociation is sufficient to supply atomic nitrogen than can diffuse into surface. A quench is necessary to avoid chromium nitride precipitation. For best results some optimization of material composition and process conditions is advisable. Stable ferritic steels cannot be treated in this manner-too fast grain growth in the core (K.H. Lo., C.H. Shek., J.K.L. Lai., 2009).

2.4. CHARACTERISTIC OF IRON

2.4.1. Crystallography

According to Randall M. German (1998), iron is an unalloyed element. As a solid, iron form a crystal where the atoms align in periodic structures as diagrammed in Periodic structures occur when we neatly pack cannon balls in front of the armory or stack brick to build a wall. Atoms can align in any of 14 periodic structures, but iron atoms exist in only two common arrangements. Nornally, the crystal structure of iron changes between these two arrangements at specific transformation temperatures, but these temperatures can be manipulated by **alloying and rapid cooling**.

The two important crystal structures of iron are body-centered cubic and face-centered cubic. These are sketched in **Figure 1.2.**

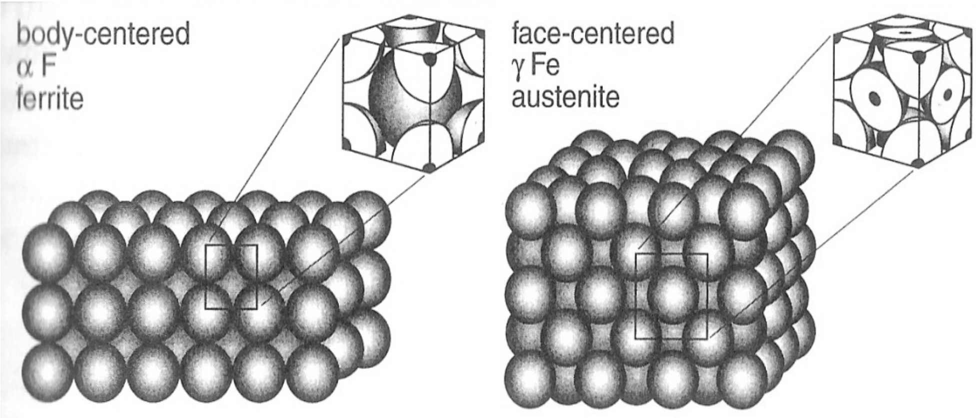


Figure 1.2. Crystal struture of body-centered cubic and face-centered cubic. Reference: Randall M. German (1998).

The body-centered cubic structure has an atom on each corner and one in the center of the cube. For iron this structure is also known as *ferrite* or *alpha iron*, α-Fe. The alternative structure is *austenite* or *gamma iron*, γ-Fe. It consists of the same corner atoms, but now one centered on each face.

For pure iron, the body-centered cubic structure is stable up to 910°C (1670°F) and again from 1392°C (2538°F) to the melting temperature of 1537°C (2800°F). Although the same crystal structure as α-iron, the high-temperature body-centered cubic form is designated as *delta iron*, δ-Fe. Between 910° and 1392°C, the stable crystal structure is face-centered cubic. The change in crystal structure with temperature is called ***polymorphism.*** Iron is polymorphic element. Changes in the crystal structure influence the properties.

On other change occurs for iron that is not due to a crystal structure change. This is the magnetic response. Pure iron is magnetic up to about 770°C (1420°F), but the crystal structure change to face-centered cubic does not occur until 910°C (1670°F). The nonmagnetic form of body-centered cubic iron was formerly known as *beta iron*, β-Fe, but this is nomenclature is rarely used anymore.

2.5. PROPERTIES OF IRON AND STEELS

The simplest steel consists of iron and carbon. Carbon is expensive and very potent in terms of strengthening. These simple alloys are called *plain carbon steels*, and usually range from 0.2 to 0.8% carbon. **Table 1** provides the classes of ferrous alloys.

TABLE 1. The Main Alloying Classes of Steels. Reference: Randall M. German (1998).

Alloy Class	Alloying Level	Key Uses
Iron	None	Magnets
Plain Carbon Steel	≤0.8% carbon	Moderate strength, general purpose
Low-alloy steel	≤5%, some carbon	High strength
Stainless steel	≥12% chromium	Corrosion resistance
Tool steel	30-50%, ≥0.4% carbon, includes carbide forming metals	Corrosion resistance
Cast iron	High carbon levels	Large structures, castings, automobile engines
High-heat alloy	≤25%, high chromium and aluminium	High heat, jet engines, furnaces, heating elements
Special alloy	≤50%	Special applications, electronics, glass seals, filters
Silicon alloy	≤8% silicon	Electrical transformers
Magnetic alloy	≤50% nickel, many special compositions	Magnetic components

2.5.1. Alloy Types

Randall M. German (1998) says that when more than 0.8% carbon is present, the steels are brittle. Alloys with about 0.8% carbon are very special and are termed *eutectioid steels*. Less carbon gives a hypoeutectoid steel (below the eutectoid), and more carbon gives a hypereutectoid steel (above the eutectoid).

The stainless steels have the presence of chromium that provides corrosion and oxidation resistance associated with the stainless characteristics. As illustrated by the saltwater corrosion data in **Figure 1.3, at least 12% chromium is required for protection against corrosion.** Other alloying ingredients might include nickel, manganese, carbon, molybdenum, or copper. Usually if nickel is present in large quantities, then carbon is present.

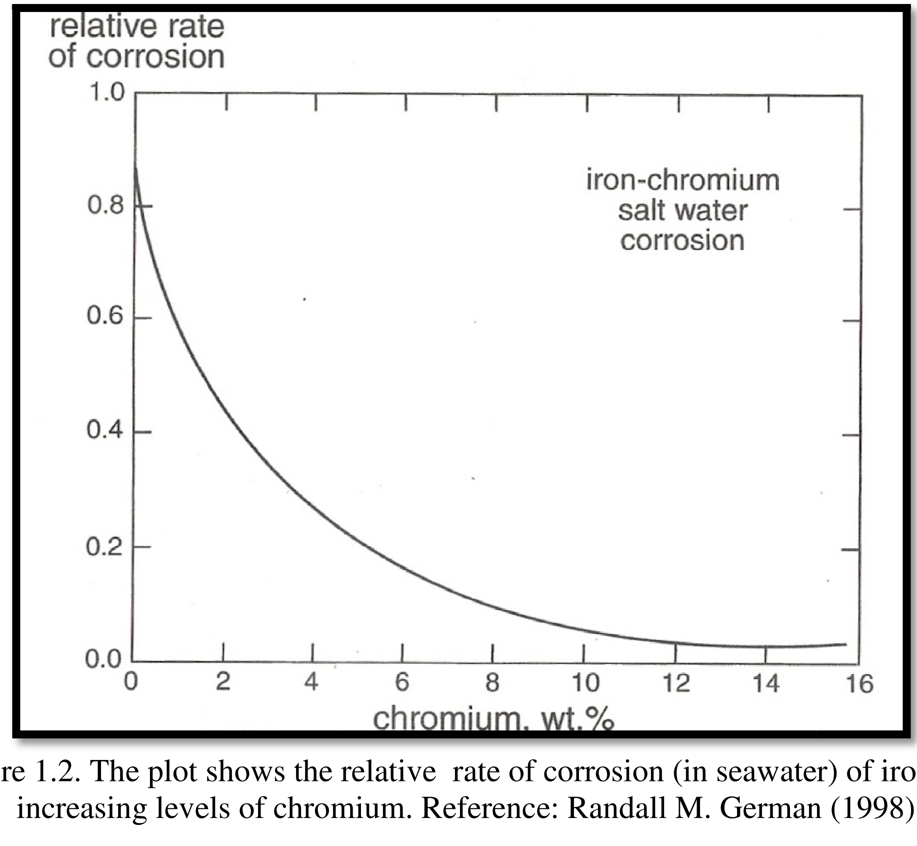


Figure 1.2. The plot shows the relative rate of corrosion (in seawater) of iron with increasing levels of chromium. Reference: Randall M. German (1998).

Table 2 summarizes some steels by giving examples form different alloy types, including the common name, composition, theoretical density, and mechanical properties (Randall M. German ,1998, p.13)..

Table 2. Composition, Name, and Mechanical Properties of Common Steels. Reference: Randall M. German (1998).

Designation	Type	Composition wt% (Balance Iron)	Density (g/cm^3)	Yield Strength (MPa)	Tensile Strength (MPa)	Hardness
Iron	Pure	0	7.9	133	266	21 HRB
Malleable	Cast-iron	2.5C-1Si-0.5Mn	7.3	231	364	75 HRB
4340	Low-alloy	1.8Ni-0.7Mn-0.7Cr-0.2Mo-0.4C	7.9	1675	1875	50 HRC
M2	Tool	6W-5Mo-4Cr-2V-0.3Mn-0.3So-0.9C	8.0	1650	1860	65 HRC
250 marging	Special	18Ni-8Cp-5Mo-0.4Ti	7.8	1750	1820	49 HRC
316L	Stainless	17Cr-13Ni-2Mo	8.0	280	550	85 HRB
420	Stainless	13Cr-0.2C	7.7	1365	1610	51HRC
A286	High-heat	26Ni-15Cr-2Ti-1.3Mo-0.2Al	7.9	735	1022	28 HRC
Invar	Special	36 Ni	8.1	280	490	75 HRB

2.5.2. Iron-Carbon Steels

According to Randall M. German (1998), carbon is not very soluble in ferrite (α-Fe) at room temperature, yet it is soluble in austenite (γ-Fe or austenite), the high-temperature form of steel. **Figure 1.4** shows the phases, carbon solubility, and melting behavior of iron versus carbon solubility and temperature. The maximum solubility of carbon in ferrite is 0.025% at 727°C (1340°F), while the maximum solubility in austenite is 2.1% at 1148°C (2098°F). This nearly 100-fold difference in carbon solubility makes it possible to strengthen ferrite by quick cooling iron-carbon alloys from austenite. Carbon dissolve in austenite, and on quick cooling becomes frozen in the steel as austenite tries to form ferrite. The net result is a distortion of the crystal structure and formation of a hard phase known as *martensite*, as distorted ferrite with dissolved carbon. If a high-carbon austenite is slow-cooled, then the carbon precipitates out of solution, just as snow forms in cooled clouds, to form a carbide Fe_3C called *cementite* (p. 14). It is a new crystal structure, consisting of 6.67 wt% carbon (25 atomic percent).

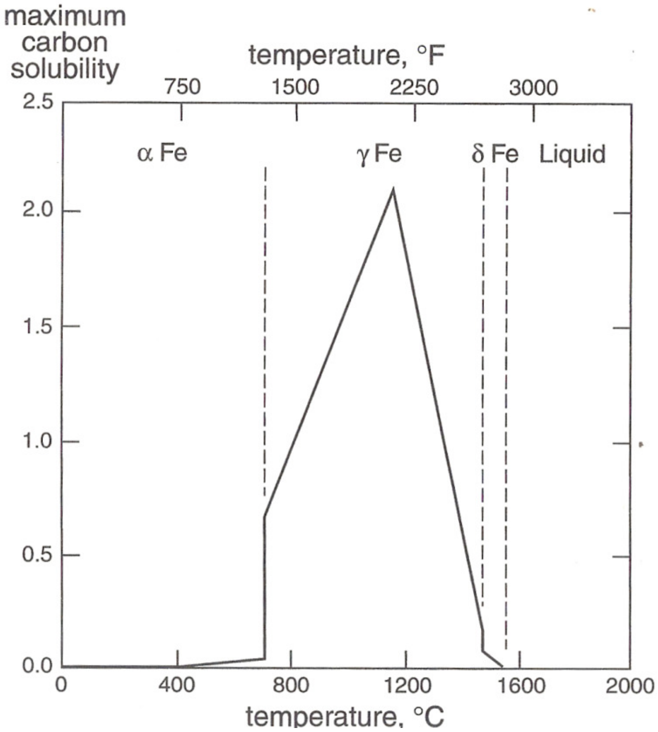


Figure 1.3. The solubility of carbon in iron changes with temperature Reference: Randall M. German (1998).

2.6. IRON AND STEEL POWDERS

The powder production is organized into four divisions. First, the language for discussing powders and their characteristics - size, shape, packing, and flow. The second sections describes powder production techniques with information on the powder characteristics associated with each. Third, the powder characteristics will be contrasted and compared. Finally, the attention is turned to the selection of powders for various applications.

2.6.1. Particle Size

Particles can be sized by maximum dimension, mass, volume, surface area, minimum diameter, or a randomly selected dimension. **Table 3** shows the comparison of ferrous powder size based on different production techniques (Randall M. German ,1998, p.50),

TABLE 3. Comparison of Ferrous Powder Production Techniques. Reference: Randall M. German (1998).

Process	Particle Size Range (µm)	Particle Shape	Packing Density	Alloying	Cost
Carbonyl decomposition	2-20	Spherical	Moderate	No	Moderate
Centrifugal atomization	50-1000	Spherical	High	Yes	Variable
Electrolytic	5-300	Irregular, dendritic	Low	No	Moderate
Gas atomization	10-300	Spherical	High	Yes	High
Mechanical comminution	2-500	Angular	Moderate	Some	High
Melt spinning	200-1000	Flake	Moderate	Yes	High
Millscale reduction	20-800	Sponge	Low	No	Low
Oxide reduction	30-500	Sponge	Low	No	Moderate
Plasma atomization	5-80	Spherical	High	Yes	Very high
Spark erosion	0.1-20	Spherical	Moderate	Some	Very high
Water atomization	5-400	Irregular, ligamental	Low to moderate	Yes	Moderate

2.6.2. Particle Shape

Particle shape varies significantly between iron powders. Further, shape influences packing, flow, and powder compaction. Because measuring particle shape is difficult, qualitative descriptors are used to convey the particle shape. **Figure 1.5** is a collection of electron micrographs that illustrate the variations possible in particle shape. The best particle shape descriptors are simple words, especially words that invoke wide-spread images. A full set of shape descriptors includes the following: spherical, angular, irregular, dendritic, sponge, cubic, polygonal, flake, ligamental, acicular, and rounded (Randall M. German ,1998, p.56).

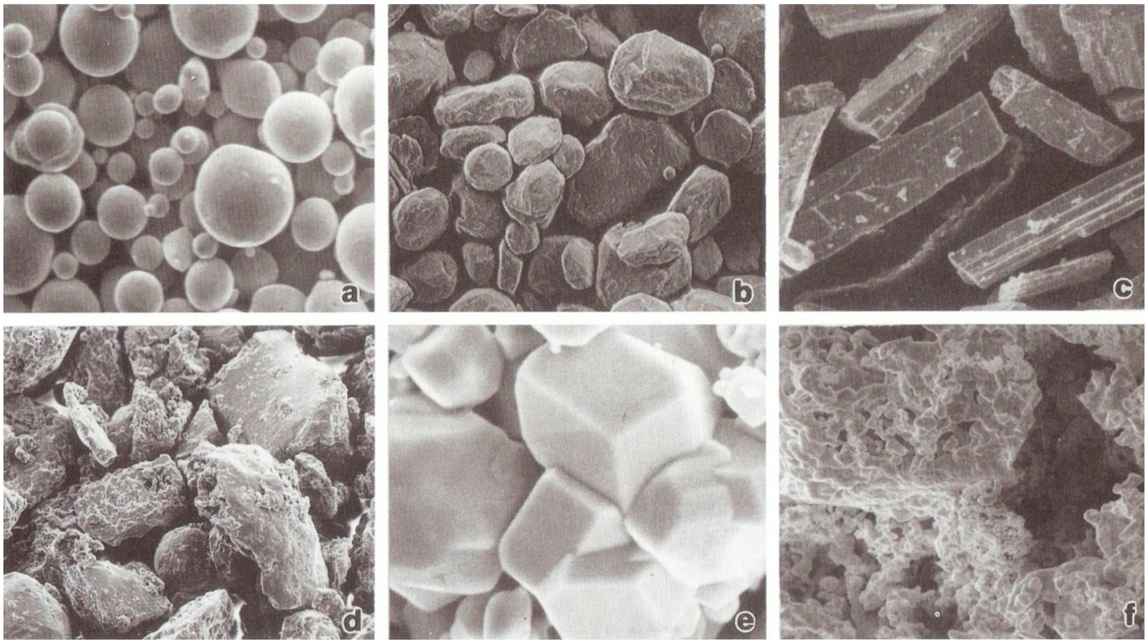


Figure 1.4. SEM showing the shape diversity in powders, The simple terms can be used to describe particle shape: (a) spherical; (b) rounded; (c) angular; (d) irregular; (e) polygonal or cubic; (f) sponge. Reference: Randall M. German (1998).

2.7. PRODUCTION OF FERROUS POWDERS

2.7.1. Mechanical Techniques

A conceptually simple means for making a powder is to grind, file, or hammer a piece of steel. Eventually the steel will disintegrate into powder. Milling or grinding are part of another powder production process.

The mills for grinding powders consists of balls, hammers, impellers, and meshing jaws to mechanically impact on the material, causing it to fracture into powder. Milling via mechanical impaction of falling hard balls in a rotating chamber is a classic approach to grinding brittle materials. These are known as *ball mills* or *jar mills*. A most common techniques uses a cylindrical jar filled with balls and the material to be milled. The jar is rotates at a speed where the balls are lifted to fall on the material. As the particle size decreases, further grinding becomes less productive.

Generally, the louder the mill, the more efficient the grinding operation. For optimal milling, the diameter of the grinding balls should be approximately 30 times the diameter of the charge material. The volume of balls should be about half the jar volume, and the fill of the input material should be about 25% of the jar volume. Fluids or protective atmospheres are used to reduce contamination.

Variants on ball milling include jet milling, attritor milling, vibrator milling, and hammer milling. Each of these techniques uses a mechanical means to reduce the particle size (Randall M. German ,1998, p.67).

A micrograph of an iron powder produced this way is shown in **Figure 1.6**.

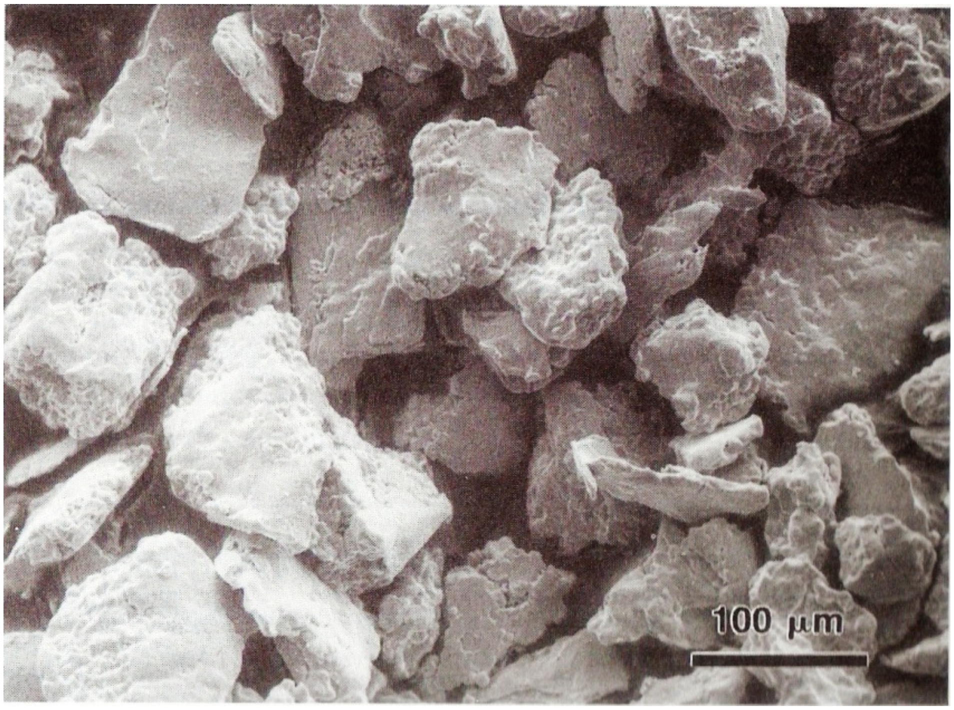


Figure 1.5. The angular powder was fabricated by milling machining chips to form small particles that were subsequently hydrogen-reduced to soften and purify the powder. Reference: Randall M. German (1998).

2.7.2. Atomization

2.7.2.1. Gas Atomization

The desired particle shape is spherical. In this instance, melting is performed under a vacuum in an induction melter. The vacuum melting is required to meet the purity demands of many biomedical, aerospace, and electronic alloys.

The expanding gas around the molten metal stream causes a dramatic disruption of the melt stream. The resulting droplets are accelerated to high velocities and solidify before reaching the walls of the chamber – requiring a large chamber. To ensure that the melts is not frozen in the nozzle, it is heated much over its melting temperature. This also allows the particles to spherodize before solidification. Final particle size is determined by several factors, with higher gas pressures and gas flow rates being necessary for producing small particles. Most powders are in the 15-300 μm range, but smaller powders are possible (Randall M. German ,1998, p.74).

Figure 1.7 is a scanning electron micrograph of a gas-atomized stainless-steel powder.

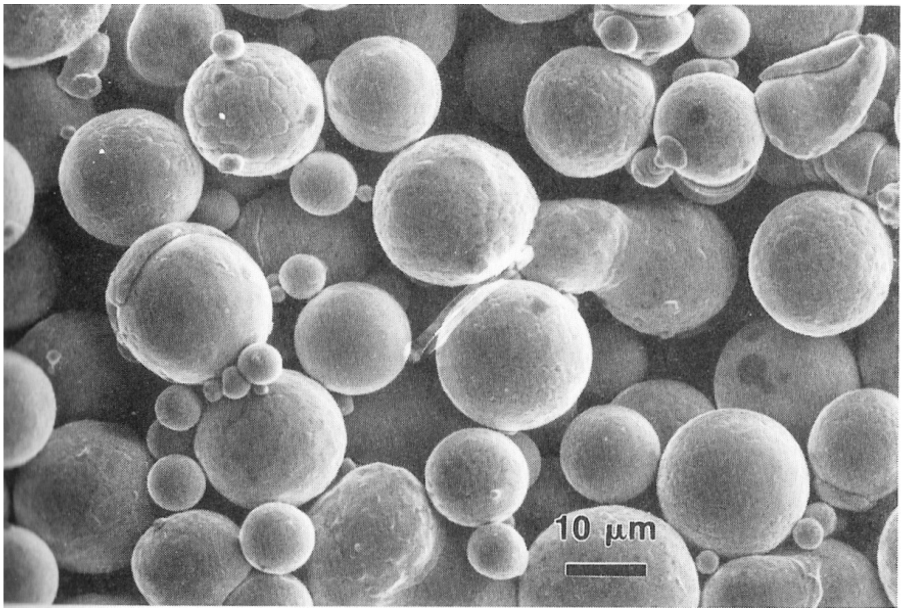


Figure 1.6. SEM of a gas-atomized stainless-steel powder. Reference: Randall M. German (1998).

2.7.2.2. Water Atomization

Water atomization is the most common techniques for producing elemental and alloy ferrous powders. Two examples of water atomizer powder are shown in **Figure 1.8** (Randall M. German ,1998, p.76).

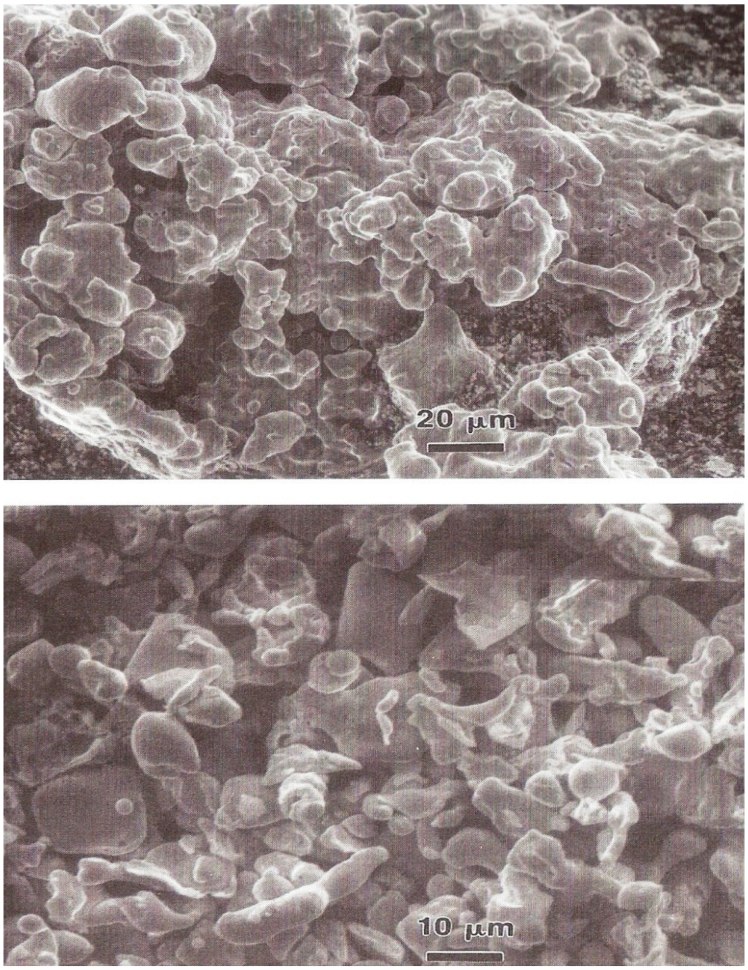


Figure 1.7. The upper photograph is the typical compaction grade powder with many bumps and nodules on the surface. Reference: Randall M. German (1998).

The upper photograph is the typical compaction grade powder with many bumps and nodules on the surface. In the lower picture, a very small, ligamental shaped stainless-steel powder is evident, one useful for injection molding. As contrasted with gas atomization, powder produced by water atomization is not spherical.

2.7.2.3. Centrifugal Atomization

Randall M. German (1998) says that several specialized atomization processes use a spinning disk or other means to impact centrifugal force to a melt. Droplets are thrown off as a fine spray that solidifies into spheres. An example of the centrifugal atomization is shown in **Figure 1.9.**

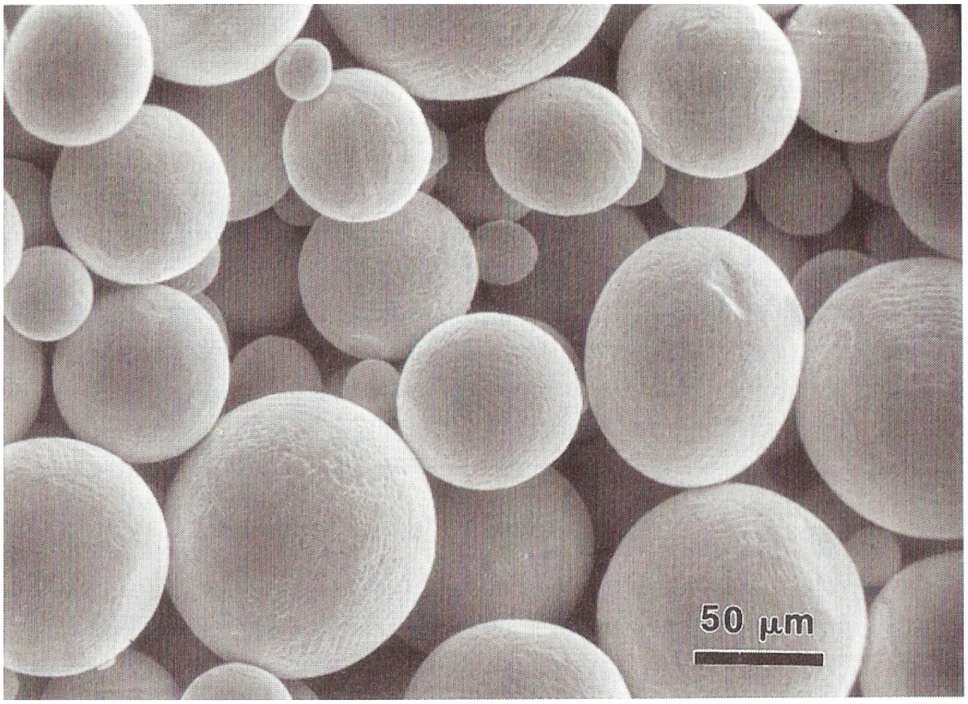


Figure 1.8. A SEM picture of a steel alloy powder formed by centriugal atomization, showing the nearly perfect spheres of powder. Reference: Randall M. German (1998).

Benefits of centrifugal atomized powders are the purity, spherical shape, and uniform apparent density. The disadvantages are a low production rate, high equipment and processing costs, and coarse particle size.

2.8. . CHARACTERISTICS OF COMMON FERROUS POWDERS

Table 4 summarizes the characteristics of several commercial iron and steel powders. Chemistry is an important variable. Low-allow steel, stainless-steel, and tool steel powders are all available. In each alloy class, the oxygen content is a good measure of the powder production skills. Also, particle size and particle shape are widely different. For some applications, a low-packing density, sponge powder is most desirable, such as for brakes. Further, high porosity powders are useful for situations in which swelling occurs during sintering, especially in iron-copper alloys. The water-atomized iron powders can vary considerably, but industry standardization keeps most of the offerings close in terms of powder characteristics.

TABLE 4. Example of Metal Powders and Characteristics by Various Processes. Reference: Randall M. German (1998).

Powder	Process	Shape	Median size (µm)	Oxygen Level (%)	Apparent Denstiy (g/cm^3)	Tap Density (g/cm^3)	Flow Time (s)
Iron	Water atomization	Irregular	75	0.11	2.9	3.4	26
Iron	Water atomization	Irregular	104	0.15	2.9	3.5	26
Iron	Water atomization	Irregular	106	0.08	2.9	3.1	26
Iron	Gas Atomization	Spherical	66	0.15	4.5	5.0	9
Iron	Carbonyl	Spherical	5	0.38	2.7	4.3	NF[a]
Iron	Carbonyl	Spherical	4	1.00	3.6	4.5	NF[a]
Iron	Oxide Reduction	Porous	77	0.39	2.6	-	29
Iron	Oxide Reduction	Porous	50	1.0	1.9	3.0	35
Iron	Centrifugal Atomization	Spherical	75	0.10	4.7	5.0	14
4600 Steel	Water Atomization	Irregular	70	0.10	2.9	3.0	26
Stainless Steel	Gas Atomization	Spherical	12	0.10	3.8	4.7	38
Stainless Steel	Water Atomization	Irregular	60	0.20	2.6	3.7	30
Stainless Steel	Water Atomization	Rounded	10	0.46	3.8	4.5	NF[a]
Tool Steel	Gas Atomization	Spherical	10	0.01	5.0	55	NF[a]
Tool Steel	Water Atomization	Nodular	70	0.10	1.8	2.4	50

Usually, the higher-purity powders exhibit the highest compressibility. The classic is water-atomized powders do not pack to a high density, but do undergo extensive deformation during compaction that results in a high green strength. The gas-atomized and centrifugally atomized powders exhibit high packing densities due to the spherical shapes. However, they prove difficult to compact and have low compressibility and negligible green strength. Generally, spherical powders are used where packing density is the primary concern, since compaction is difficult and green strength prove low. Then, carbonyl and small atomized powders are most useful in injection molding, tape casting, and slip casting. Often powders are mixed to customize the packing, flow, compaction, shrinkage, or other engineering attributes.

Some grades are ferrous powders are prealloyed, including, including low-alloy steels, tool steels, and stainless steels. Alloying additions are required for powder that will be used in applications where certain properties are a concern-magnetic, corrosion, strength, machinability, or electrical. Usually, the alloying additions increase the price and reduce the compressibility of the powder (Randall M. German ,1998, p.85).

2.9. SINTERING BEHAVIOR

2.9.1. Changes Due to Sintering

According to Randall M. German (1998), sintering is the key step in transforming the green compact into a high-strength structre. It involves heating the compact to a temperature where the particles weld to each other, much like ice cubes in a freezer bind to one another. Because of strengthening during sintering, it is easy to follow the progress by measuring mechanical properties. Improved strength occurs by events at the atomic level. Weld bonds between particles form and grow through the motion of individual atoms.

Besides particle bonding, sintering contributes to changes in the structure as evident through a microscope, termed the *microstructure*. During sintering, the microstructure also exhibits changes in porosity, pore size, and pore shape. Sometimes the particles move relative to each other, giving a dimensional change to the compact. At the sintering temperature the high rate of atomic motion progressively leads to growth of bonds between the particles, termed *interparticle necks*.

2.9.2. Fundamental Steps in Heating

In the sintering furnace, the compact is heated to a peak temperature for a few minutes to allow heat to uniformly soak throughout the load. The first even during heating is the extraction of binders and lubricants. This is accomplished at temperature below 550°C (1022°F). Subsequently, the compact is heated to a high temperature that induces atomic motion and sinter bonding, typically in the temperature range of 1100-1350°C (about 2000-2460°F).

It is near the peak temperature in the sintering cycle that most of the particle bonding occurs. Although some time at the peak temperature is beneficial to ensure complete heating of the compact, extended times prove detrimental. *Oversintering* refers to the situation in which a steel exhibits a loss of properties with prolonged time at the peak temperature. Such events microstructures coarsening are evident by enlarged pores and large grains.

After the soak at the peak temperature, the sintered compact is cooled to room temperature and removed from the surface. The phase transformations in a steel can be manipulated during cooling to induce high strength. Thus, cooling rates may be controlled for some steels. This practice of heat treating during cooling is termed *sinter-hardening*. The alternative is to reheat the compact in a separate furnace after sintering (Randall M. German, 1998, p.191).

2.10. SINTERING ION AND STEEL POWDERS

The atomic structure of iron affects the inherent sintering rate. Because of the phase transformation from alpha iron (α-Fe) or ferrite [the body-centered cubic crystal structure of iron stable below 910°C (1670°F)], to gamma iron (γ-Fe) or austenite (the face-centered cubic crystal structure of iron stable at most sintering temperatures), there is a dramatic shift in sintering rate on heating. Atoms diffuse faster in the body0centered cubic crystal structure, making ferrite the favored phase for sintering. Hence, iron exhibits faster sintering at temperature near 900°C than at 1000°C (1652°F and 1832°F0. Consider that most ferrous sintering occurs between 1100 and 1300°C (2000 and 2375°F). Thus, on heating the sintering rate progressively increases, but the phase transformation from alpha to gamma slows sintering. As a demonstration of the behavior (Randall M. German ,1998, p.193).

CHAPTER 3: METHODOLOGY

3.1. PROJECT FLOW CHART

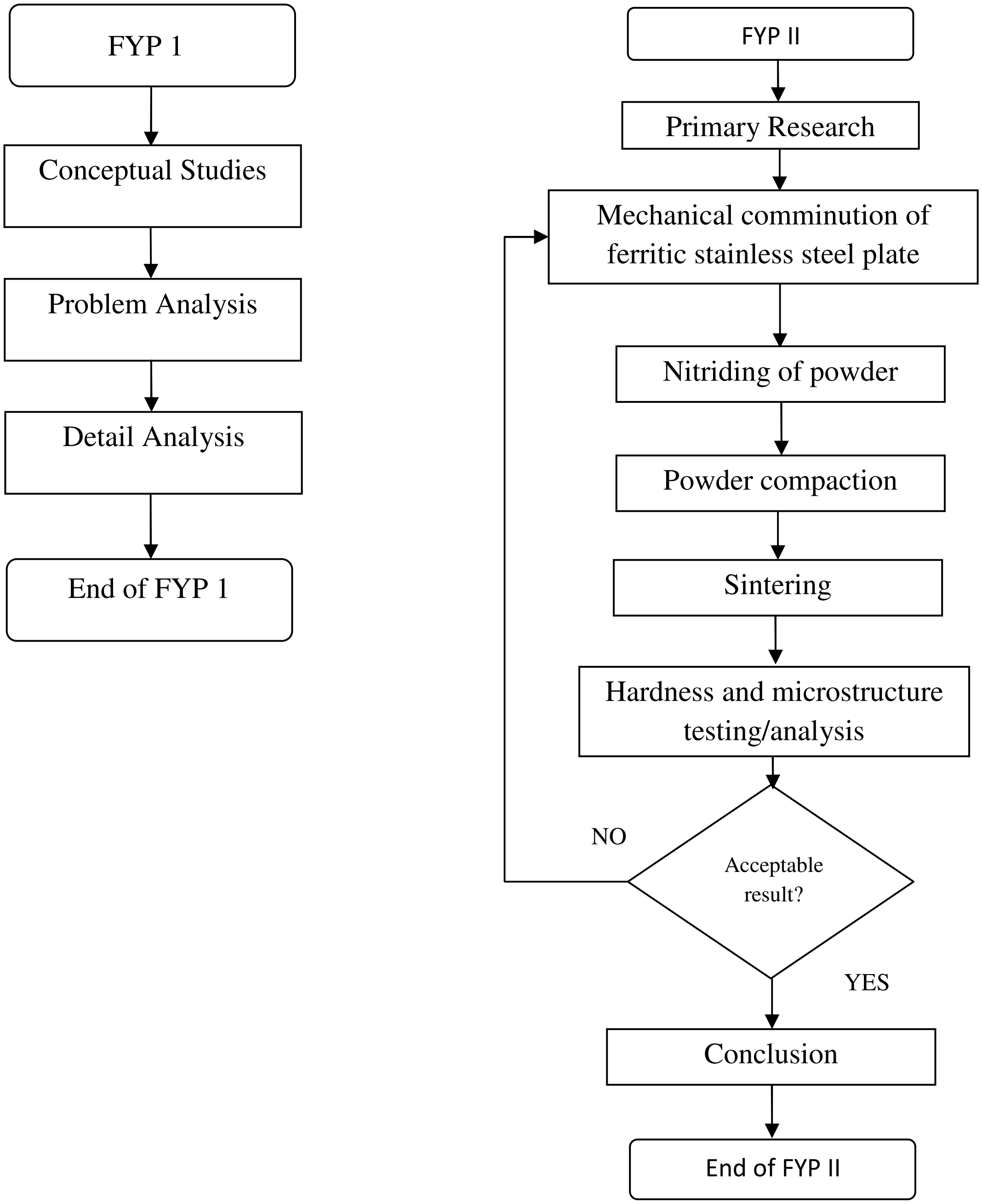


Figure 2.1. Shows the flow chart of the project.

3.2. EXPERIMENTAL PROCEDURE

3.2.1. NITRIDING OF POWDER

Nitriding was carried out in Carbolite horizontal tube furnace as shown in **Figure 2.2**. For each treatment sample was placed in the boat and inserted into the heating zone. Before the samples were heated, the air in the furnace was purged with nitrogen for 15 minutes at a flow rate of 1000 cm^3/min to prevent oxidation of the sample. Heating at 5ºC/minute was started immediately after the purging was completed. Nitrogen was introduced into the furnace with the flow rate of 1000 cm^3/min. The temperature variables were set at 700°C, 900°C and 1200°C while 1 hour and 3 hours of time period was set for time variables. At the end of the process, the samples were directly had slow cooling at 5ºC/minute. The examples of powder after having nitriding process are shown in **Figure 2.3** below.

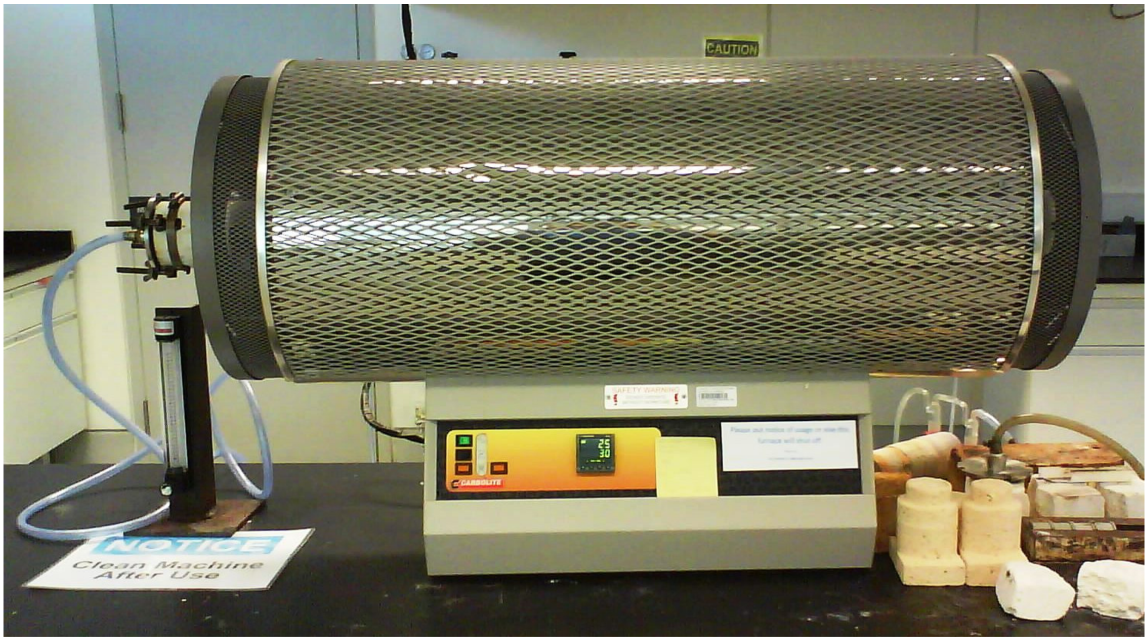


Figure 2.2. Carbolite horizontal tube furnace.

Figure 2.3. Samples of AISI 430 ferritic stainless steel powders after having nitriding process.

3.2.2. POWDER COMPACTION

Compaction was carried out on AISI 430 ferritic stainless steel powder after sintering process was completed. Samples were pressed in the diameter of 13 mm with 17000 Pa pressure for 15 minutes of dwell time. The result was carried out on 3 mm thickness of pallet. The machine used is Auto Pallet Machine. After powder compaction has been done, the pallet will undergo sintering process for 3 hours at temperature of 1200°C.

3.2.3. HARNESS AND MICROSTRUCTURE ANALYSIS

After grinding pallets with emery paper (#4000), the samples were being etched by the solution of 40ml hydrochloric acid, 5g copper chloride, 30ml water and 25ml ethanol. The solution is also known as Fry’s reagent. Vickers micro hardness measurements were carried out under the load of 300 gf on cross-section section of the nitrided material for 15 seconds. Optical Microscope was employed to observe the microstructure of the treated samples.

CHAPTER 4: RESULT AND DISCUSSION

4.1 MICROSTRUCTURE CHANGES OF AISI 430 FERRITIC STAINLESS STEEL

The original microstructure of the sample is typical ferritic stainless steel microstructure as shown in **Figure 3.1.** It shows not evenly distribution of ferrite over the whole cross section and mostly segregation bands at most places with some black spot which is assumed as pores.

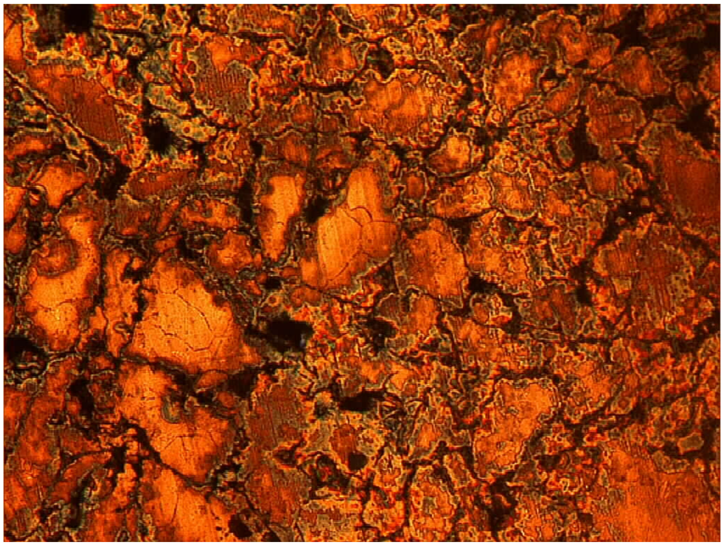


Figure 3.1. Optical micrograph image of cross section of the typical AISI 430 steel.

Before a series of nitriding treatments were performed, a heat treatment without nitrogen atmosphere was conducted. This would be used later as a comparison with the solution nitriding result. It was also to study the influence of nitrogen in the treatment. To attain the objective, the sample was heated to three different temperatures (700°C, 900°C, and 1200°C), holding for one hour and three hours in air atmosphere and slowly cooled. The microstructure of the material after the treatment is given in **Figure 3.2**. The microstructure consists mainly of segregated ferrite grains.

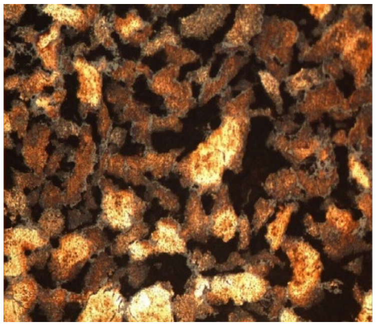
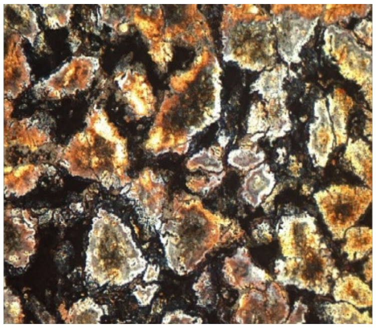
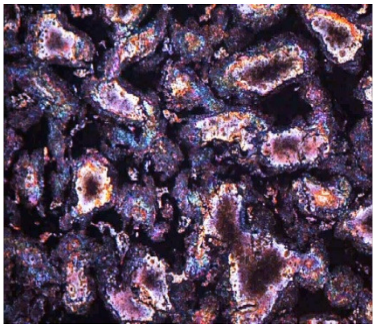
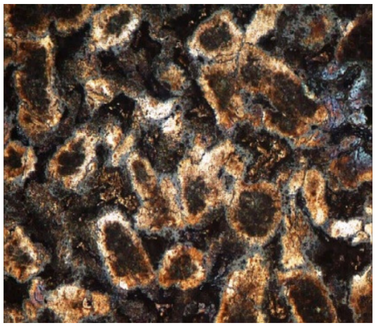
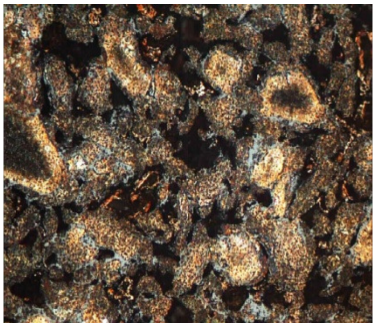
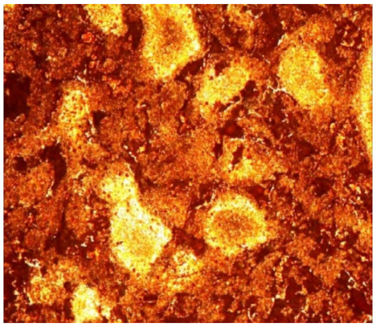
Time (hour)	Nitriding Temperature		
	700°C	900°C	1200°C
1			
3			

Figure 3.2. Optical micrograph images of the cross section of nitrided AISI 430 stainless steel samples.

Microstructures of the nirided samples are given in Figure 14. Only one phase (ferrite) can be clearly indicated in the images. Martensite phase cannot be seen through the optical microscope because the samples were having slow cooling. If the samples were imposed to quench (quick cooling), there is a probability for it to have two phases (martensite and ferrite).

According to **J.H. Sung, J.H. Kong, D.K. Yoo, H.Y. On, D.J. Lee, H.W. Lee., (2008)**, due to an incomplete transformation of austenite to martensite, retained austenite may be formed. This was found when solution nitriding of AISI 430 ferritic stainless steel was carried out at lower temperature (*i.e*. at 1100ºC). However, austenite was not found in this work.

With increasing of the nitriding temperature and time, the particles of the AISI 430 ferritic stainless steel is likely to be more bonded and thus decreasing the pore spaces between particles. It proves that the nitrogen in nitriding process has diffused into the core of the particles hence gives more strength to the particles bond. Also, from the optical micrographic image, it can be seen that the particles are not homogeneous. The sizes of particles are not uniform due to mechanical communition.

4.2 HARDNESS OF NITRIDED AISI 430 SAMPLES

The hardness of the surface of the samples is given in **Table 5**. The average of Vickers hardness is calculated from ten different fixed locations on the surface. The hardness of the nitriding samples is apparently has risen as short as one. A very high hardness is shown by all samples that apply nitriding. This hardness is in accordance with the microstructure of the steel.

TABLE 5. Vickers Hardness (HV) Value of AISI 430 Ferritic Stainless Steel.

Nitriding		**Average Vickers Hardness (HV)**
Time (hour)	**Temperature (°C)**	
Unnitrided		176.3
1	700	222.4
	900	223.2
	1200	245.8
3	700	234.0
	900	239.3
	1200	250.3

Table 5 shows the hardness variation after the nitriding treatment at given time and temperature variables. The results shown in the table also indicates that higher hardness is achieved by nitriding samples at longer time. Longer nitriding time is expected to diffuse more nitrogen into the steel hence thickens the ferritic particles. By assuming the thickness of the particles increasing with increasing of nitriding time, therefore the diffusion of the nitrogen to the core of the steel also increasing. Thus, it can be concluded that higher nitrogen content lead to higher hardness. This find is accordance with **Hudiyo Firmanto, Patthi Hussain, Othman Mamat (2008)**, where their findings showed that higher hardness was achieved by samples nitrided at longer time.

Figure 3.3 below shows the comparison chart of Vickers hardness with respective to the times and temperatures.

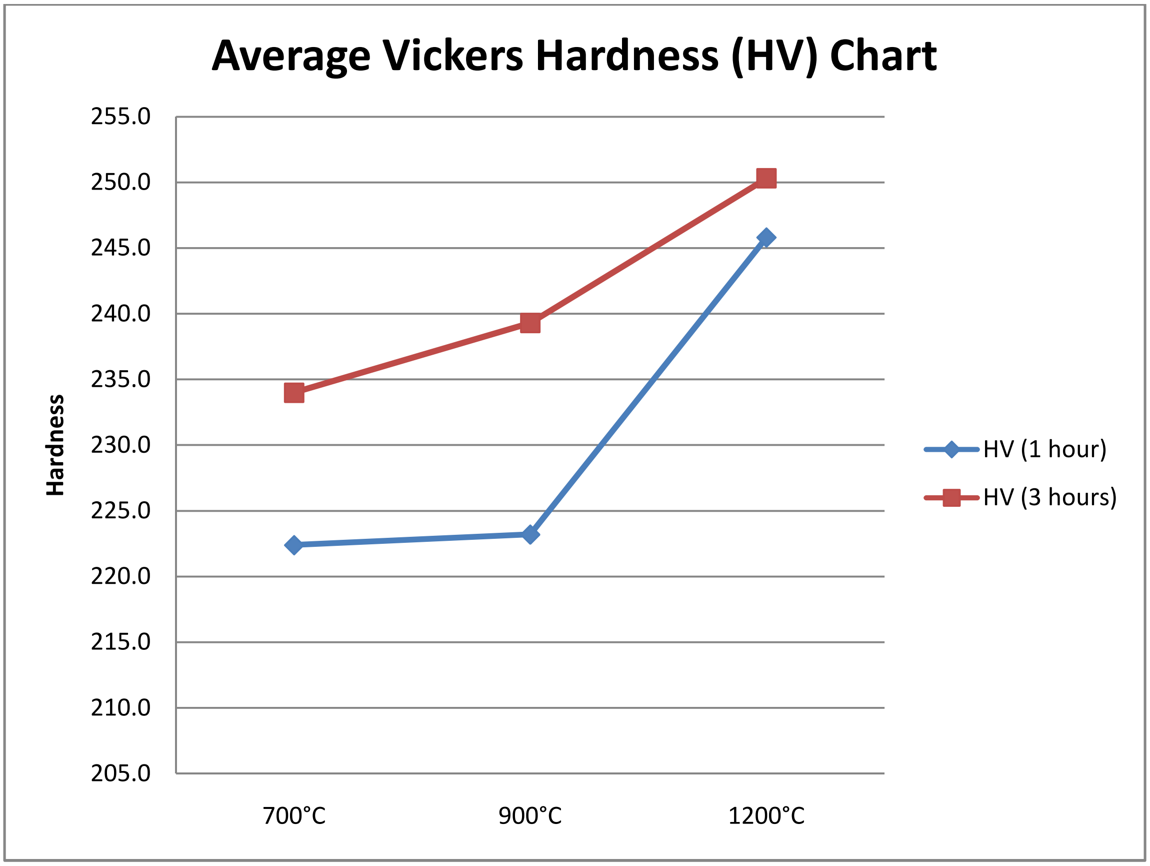


Figure 3.3. Average Vickers Hardness (HV) of nitrided samples with respective to the times and temperatures.

As shown in the Table 5 and Figure 15, the hardness was relatively increased with the increasing of nitriding temperature after the treatment. Among this three different nitriding temperatures (700°C, 900°C and 1200°C), temperatures at the highest value will give the highest hardness value compared to the others for the same period of time. The highest hardness (~250Hv) was observed at 1200°C for 3 hours. According to the **J.H. Sung, J.H. Kong, D.K. Yoo, H.Y. On, D.J. Lee, H.W. Lee (2007)**, this hardness variation is related to the nitrogen content and phases appeared on the surface layer.

A lot of improvements have been done through this nitriding process because it is comparable to the sample that has not exposed to the nitrogen. It is clearly shown that the hardness has increased approximately 26 to 46 percent from the unnitrided sample. The hardness differences between nitrided samples are only occurred at the value of maximum 13 percent. One of the additional reasons that can be assumed for answering these phenomena is regarding the bonding between the particles itself.

Figure 3.4 shows the microstructure changes according to the increasing hardness. From the observation, the compactness of the particles and pores area influenced the hardness value. When the particles were exposed to the nitrogen in the nitriding process, the size of particles increased hence will reduce the pores area between them and finally will give more strength to the particles bond. The diffusion of nitrogen into the core of the steel also assist in the occupying the pores area.

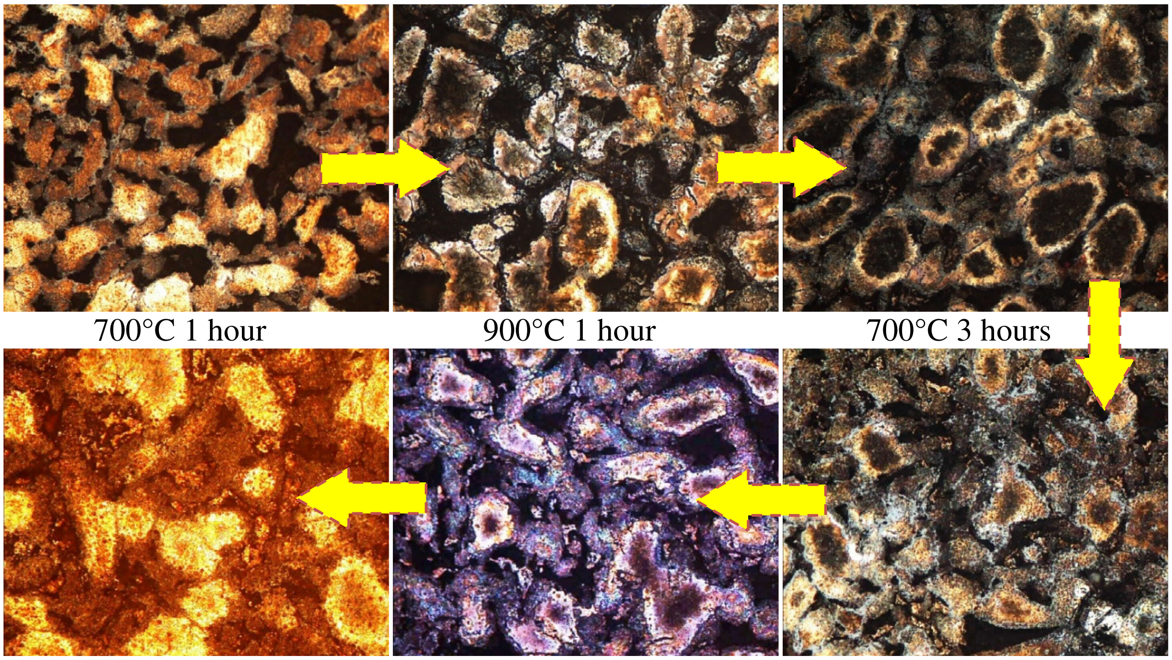

700°C 1 hour
900°C 1 hour
700°C 3 hours
1200°C 3 hours
1200°C 1 hour
900°C 3 hours

Figure 3.4. The microstructure changes of samples according to the increasing hardness.

CHAPTER 5: CONCLUSION AND RECOMMENDATIONS

5.1. CONCLUSION

Even thought there are some difficulties in interpreting the result, the author managed fulfill the entire main objectives of the project. The nitridation of AISI 430 ferrictic stainless steel samples has been completed at different temperatures and times. Both hardness and microstructure investigation indicated that the effect of the nitrding increased with nitriding temperatures and times.

The nitriding has successfully enhanced the hardness of AISI 430 ferritic stainless steel. The hardness was enhanced by the nitrogen diffusion into particles. Prolonging the nitriding yields to more nitriding diffusion of the particles. It can be concluded that the improvement of the hardness is due to the compactness of the materials and also the reduction in pores area.

The study on the microstructure indicated that the particles of the material are not uniformly distributed after the nitriding treatment. Analysis of the surface reveals that the nitrogen diffusion yields good enhancement which can be clearly seen with pores area. The pores area at the lowest temperature (700°C) shows many black spots which is not good for the properties of the materials. The corrosion might be occurred at the pores area hence reduce the strength.

4.2.1 RECOMMENDATIONS

As stated in the discussion there are some recommendations for this project. First and for most the powder of AISI 430 ferritic stainless steel preferably to be changed from mechanical comminution to centrifugal or gas atomization method because the particles size are not well uniform and this can lead to weaken the strength of samples. This is contribution from the pore spaces that left when the powder are compacted after nitriding process.If greater bond from smaller and uniform particles size, it can have greater hardness and microstructure.

Other than that, the mechanical and physical properties can be further investigated in the future to improve the result and also give the detail description of the nitriding process. This is to ensure by nitriding the powders of the material, the properties improvement is greater rather than nitriding a bulk of material. The author's experimental is a new invention in nitriding area.

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