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EXPERIMENTA STUDIES ON SUPER NG SINTERED LOW ALLOY STEELS FABRICATED BY METAL INJECTION MOLDING

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Wan Sharuzi Wan Harun

Supervisor
Professor Dr. Hideshi Miura

Department of Mechanical Engineering
Graduate School of Engineering
Kyushu University

P K/11/14 PERPUSTAKAAN
UNIVERSITI MALAYSIA PAHANG
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ABSTRACT

Metal injection molding (MIM) process is an advanced powder processing technique because of net shaping with shape complexity at low processing energy and 100 % material utilization. This study has been performed to clarify and to optimize the relationship between the mechanical properties and the microstructures for obtaining the superhigh strengthening sintered low alloy steels (Fe-Ni system) by using MIM process. The influence of nickel particle sizes, nickel content, and sintering conditions on the microstructure and mechanical properties of superhigh strengthened Fe-Ni steel compacts have been systematically investigated. As starting materials, the mixed elemental of carbonyl iron and water-atomized nickel powders were utilized. Tempered compact added 6 mass% fine nickel powder followed by sintering at 1250 °C for 1 hour showed superhigh strength of 2040 MPa with elongation of 8.1 %, which was the best properties among reported data in P/M low alloy steels so far. These excellent mechanical properties is due to the fine heterogeneous microstructure consisted of nickel rich phase surrounded by a networks of tempered martensitic structures.

The mechanical properties of MIM compacts are highly dependent on two major factors; the porosity, and the microstructural morphology in the matrix. Both factors were cautiously considered in the present work. The porosity studies was carried out on 440C sintered steel, which was a high strength material with numerous pore contents. For the latter, the superhigh strengthened Fe-Ni steel compact, which is a primary alloy steel in this study was employed for microstructural studies on the matrix. Not only experimental work but also numerical simulation by finite element method was engaged to understand how these factors work.

440C steel compact has been purposely used as an example material to examine the pore factor. The utilization of 440C steel compact was due to homogeneous microstructure of matrix although contained many residual pores. The porosity study begins with experimental works, followed by numerical simulation for verification. The model demonstrated that tensile properties was enhanced at reduced pores and depreciated when the porosity was increased. Also, when mechanical properties of the

compacts with similar porosity level is compared, the pore factor can be disregarded due to their minimum influences. However, the pores became a major factor when comparing compacts of different porosity levels.

After the pore factor was successfully tested and evaluated, the effort had extended to the core focus of the present study. The effect of heterogeneous microstructure was treated in order to evaluate superhigh strengthened Fe-Ni steel compacts. Sintered density of all Fe-Ni steel compacts obtained in this study was 95-96 %, it means the porosity levels were about similar. Therefore, the pore factor has been simply omitted.

The microstructure of all superhigh strengthened Fe-Ni steel compacts have been consistently structured by heterogeneous condition. The microstructural heterogeneity aspects of the compact were changed by the characteristics of Ni powder, such as particle size, shape, and content, which play important roles in the deformation behavior. A complex network of higher Ni region which firmly bounded by the lower Ni region (matrix region) has been comprehensively observed.

The high ductility and high strength offered by the superhigh strengthened Fe-Ni steel compacts were probably also due to mechanically induced martensitic transformation that takes place during deformation. The material was initially metastable retained austenite, which was relatively ductile phase and the ductility was enhanced by the martensitic transformation-induced plasticity (TRIP) phenomenon. The high strength was due to the transformation of the soft austenite phase to the hard martensitic phase during the deformation as experimentally observed.

In order to understand how the microstructure results these high mechanical properties, finite element modeling based on the spatial distribution obtained experimentally was developed. Some parameters were prepared to control heterogeneity in the representative volume element. The simulated results were compared to experimentally obtained behavior, and showed good agreements. These capabilities of successful simulation of the actual microstructure by FEM resulted possibility to identify and design an optimum microstructure theoretically for Fe-Ni system.

TABLE OF CONTENTS

Page

ABSTRACT i
DEDICATION iii
ACKNOWLEDGMENT iv

CHAPTERS

1. INTRODUCTION

1.1 BACKGROUND 2

1.1.1 Fe-Ni Steel Compacts by MIM 2

1.1.2 Microstructural Simulation 3

1.1.3 Steel Compact 5

1.1.4 Metal Injection Molding Process 6

1.2 OBJECTIVE OF STUDY 10

1.3 OUTLINE 11

1.4 REFERENCES 12

2. EFFECT OF PORES ON THE MECHANICAL PROPERTIES OF HIGH STRENGHNING STEEL COMPACTS

2.1 INTRODUCTION 17

2.2 EXPERIMENTAL METHOD 18

2.2.1 Powder Characteristic 18

2.2.2 Binder Characteristic 19

2.2.3 The Compact 22

2.2.4 Experimental Procedure 22

2.3 RESULTS AND DISCUSSION 23

2.3.1 The Effect of Powder Loading on the Mechanical Properties of High Strength 440C Steel Compact 23

2.4 NUMERICAL SIMULATION 27

2.5 SUMMARY 36

2.6 REFERENCES 37

3. **EFFECT OF HETEROGENEOUS MICROSTRUCTURE ON THE MECHANICAL PROPERTIES OF SUPERHIGH STRENGTHENING MIM Fe-Ni STEEL COMPACTS**

3.1 INTRODUCTION 40

3.2 EXPERIMENTAL METHOD 41

3.3 RESULTS DISCUSSION 43

3.3.1 Experimental Data 43

3.3.2 Nickel Diffusivity 52

3.3.3 Nickel Rich Phase 53

3.3.4 Mechanical Properties 54

3.3.5 Transformation Induced Plasticity (TRIP) 55

3.3.6 Data Comparison 57

3.4 SUMMARY 59

3.5 REFERENCES 61

4. **FINITE ELEMENT SIMULATION OF HETEROGENEOUS MICROSTRUCTURE AND MECHANICAL PROPERTIES OF SUPERHIGH STRENGTHENING MIM Fe-Ni STEEL COMPACTS**

4.1 INTRODUCTION 64

4.2 EFFECT OF NI VARIATION ON THE HIGHER NI REGION 65

4.2.1 Procedure 65

4.2.2 Results and Discussion 67

4.2.3 Summary for Ni Variation Effect on the Higher Ni Region 69

4.3 CONNECTED AND DISCONNECTED HIGHER NI REGION 69

4.3.1 Procedure 69

4.3.2 Results and Discussion 72

4.3.3 Summary for Connected & Disconnected Higher Ni Region 74

4.4 GRADIENT OF NI CONTENTS 74

4.4.1 Procedure 74

4.4.2 Results and Discussion 79

4.4.3 Summary for Gradient of Ni Content 81

4.5 SUMMARY 82

4.6 REFERENCES 83

5. CONCLUSIONS AND FUTURE DIRECTIONS

5.1 CONCLUSIONS 85

5.2 FUTURE DIRECTIONS 87

5.2.1 Modeling of Microstructure 88

5.2.2 Simulation 88

5.2.3 3D Observation 89

5.3 REFERENCES 90

CHAPTER 1

Introduction

1.1 BACKGROUND

1.1.1 Fe-Ni Steel Compacts by MIM

The excellent mechanical properties offered by superhigh strengthened Fe-Ni steel compacts have been widely known. Since then, numerous efforts have been put into placed by various researchers in order to have better explanations on the mechanics of strengthening Fe-Ni alloys system.

Especially, Miura et al. reported that most important point was not only the solid solution of Ni but also fine heterogeneity which consists of Ni rich phase surrounded by a network of tempered martensitic structure as schematically shown in Fig. 1.1 [1)]. This heterogeneity resulted in good balance of strength and ductility. From their works, the best mechanical properties attained by tempered Fe-6Ni-0.5Mo-0.2Mn-0.4C using mixed elemental powders of carbonyl Fe and Ni, Mo, and Fe-25Mn were 1985 MPa tensile strength and 5 % elongation [2)]. The Ni concentration profile obtained by electron probe microanalyzer (EPMA) found out that peaks and valleys of Ni content were dispersed throughout the mezzo heterogeneous microstructure matrix [1-3)].

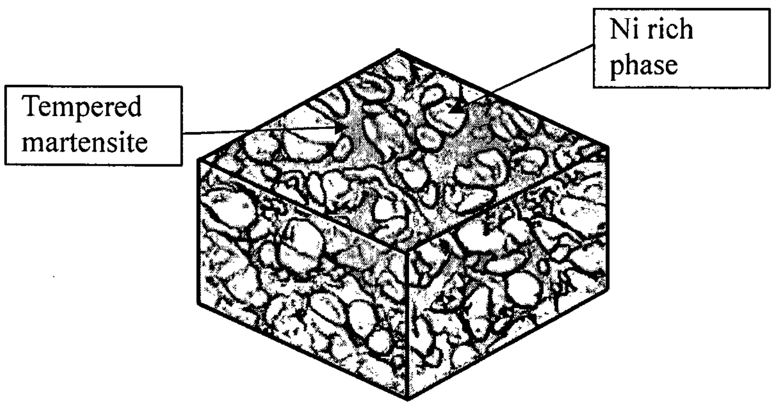

Fig. 1.1 Schematic diagram of 3D superhigh strengthened Fe-Ni steel microstructure where Ni phase surrounded by tempered martensite structure.

Furthermore, comparative studies for the effect of prealloyed and mixed elemental-based powders on the mechanical properties of AISI 4600 (Fe-2Ni-0.2Mn-0.5Mo-0.89C) and AISI 4100 (Fe-0.2Cr-2Mn-0.5Mo-0.89C) have been extensively investigated by Miura [4)]. All prealloyed-based compacts exhibited homogeneous microstructure, while the microstructures of mixed elemental-based compacts were heterogeneous. However, as sintering temperature increases, the microstructure of mixed elemental-based compact became similar to that of prealloyed-based homogeneous compact. It is worth noting that all mechanical properties of mixed elemental-based compacts were found superior over the prealloyed-based compacts [4-5)].

Until now, numerous interesting experimental data are available especially on heterogeneous nature of superhigh strengthened Fe-Ni steel compact microstructures by Miura [1-5)] and other researchers [6-9)]. However, there has been no attempts reported to numerically explain the strengthening mechanism of this distinctive heterogeneous structure. In this study, a FEM simulation work has been also implemented in order to gain better comprehensive understanding about the nature of heterogeneous microstructure of superhigh strengthened Fe-Ni steel compacts.

1.1.2 Microstructural Simulation

For design and development of high-performance materials using FEM modeling, it requires a thorough understanding and careful selection of factors that control a microstructure and its effect on mechanical properties. This is particularly challenging to make reliable numerical demonstration of the multiphase and heterogeneous nature of most high-performance alloy like Fe-Ni steel compacts. It is a complex problem to model and to predict the overall elastic-plastic response and local damage mechanisms in heterogeneous microstructure-based alloys, in particular in the metal injection molding (MIM) components [10-14)].

For the compacts with heterogeneous microstructure, numerical modeling techniques, such as the finite element method (FEM), are often more effective than mathematical modeling since the deformation and damage characteristics, particularly on a local scale, can be certainly revealed. In order to model the behavior of heterogeneous microstructure, assumption of simple geometry in a unit cell model is typically taken [15-17]. Unit cell models have been employed to model fracture, void nucleation, growth and coalescence of voids within a metallic matrix [15], and crack growth along the particle/matrix interface [18].

Another important aspect of the microstructure in a compact is the effect of spatial distribution of the alloy elements. The link between spatial distribution and mechanical behavior has not been modeled extensively [18-19]. Ghosh and co-workers [20-21] used a serial sectioning technique to obtain the spatial distribution of SiC particles, and quantified the spatial distribution by a tessellation scheme. Modeling of damage in the compact was conducted on two-dimensional (2D) sections by approximating the particle morphology as ellipsoids, so the deformation assumed a 2D stress state (plane stress, plane strain, or modified plane strain). A three-dimensional (3D) elastic Voronoi cell is also being developed [22], once again using ellipsoid particles. Boselli et al. [19] modeled the effect of crack growth using idealized 2D microstructures, consisting of circular disks embedded in a metal matrix. It was found that clustering had a significant effect on the local shielding and "anti-shielding" effects at the crack tip. Llorca and co-workers [18] modeled the effect of particle clustering on damage in metal matrix compacts. The particles, modeled as spheres, were incorporated with different degrees of clustering (as quantified by a radial distribution function). A similar modeling approach was taken by Bohm and co-workers [23-24].

In this study, the primary work was to discover interrelation between the heterogeneous distribution of Ni concentration throughout the matrix and the mechanical properties of superhigh strengthened Fe-Ni steel compacts. The work was accomplished experimentation followed by numerical simulation.

Before carrying out the simulation for heterogeneous microstructure, numerical simulation for pore structure was performed to check effectiveness of FEM simulation in the present work. It should be noted, for P/M parts that residual pores are usually found in the compact. These residual pores could be a major unfavorable factor to mechanical properties if not appropriately considered. For working components manufactured by MIM process, maximum acceptable porosity level is 5 % [28,29,31,33)]. Therefore, a preliminary studies which focused on the mechanical properties of the compacts containing pores was also performed. Since the porosity factor is a common issue for compacts, the work in this study begins dealing with the effect of pores on the mechanical properties of the compacts in the field of P/M or more specific in the MIM.

1.1.3 Steel Compact

As briefly mentioned in the previous section, the steel compacts fabricated by powder metallurgy (P/M) or metal injection molding (MIM) are typically characterized by residual pores after sintering, which quite detrimental to the mechanical properties of the compacts [25-33)]. The nature of the pore is controlled by several processing variables such as green density, sintering temperature and time, alloying additions, and particle size of the initial powders [34)]. In particular, the fraction, size, distribution, and morphology of the residual pores have a profound impact on the mechanical properties [35-37)].

Regardless characterized by these residual pores, the compacts are considerably utilized as structural parts, and the demand has been expanded favorably in wide variety of engineering fields. As a result, higher density and high mechanical properties have been required to the sintered structural parts. As one of the solution, MIM technique is found to be the best candidate as a manufacturing process for meeting the above requirements, because MIM process offers near full dense and net shaping of complicated components with a relatively low processing cost [35)].

When mechanical properties of the steel compact is considered, residual pores are not the only factor, the microstructural morphology is equally crucial, especially in high density compacts. Some alloy compact provides perfect homogenous nature, while the others are characterized by heterogeneous microstructure. Usually, when dealing with high-performance steel compacts, the nature of the microstructure is established on multiphase and heterogeneous. Especially for superhigh strengthened Fe-Ni steel compacts, the microstructure is characterized with complex variations of Ni concentration [2-5]. And this is a subject of interests to be explored on how this heterogeneity nature affects the mechanical properties of the superhigh strengthened Fe-Ni steel compact.

1.1.4 Metal Injection Molding Process

Metal injection molding (MIM) offers several advantages over other production technologies. The MIM technology has progressed substantially over the past 25 years and the maturity of the technology is demonstrated by the growing number of components, alloys, size, and shape complexity. Complex-shaped parts can be simply manufactured by MIM process without or with very little secondary finishing. Also, undercuts in the parts, which are impossible with conventional sintering processes, can be realized with the MIM process. The surface of MIM parts is far superior to that of precision cast parts. Thereby, finishing and polishing costs can be eliminated or reduced. MIM parts usually do not have to be mechanically refinished, the harder the machined material is, the more advantageous the MIM process is. Summary of advantages offered by the MIM process is shown in Fig. 1.2 [29,37].

Although relatively expensive equipment are needed for MIM, the process is competitive, above all, in the fields where greater quantities of complicated products are required. Smaller products are usually manufactured by MIM process because tolerance deviations increase with the size of the product. The greatest advantage of the MIM process is that all sintering-suitable (sinterable) materials can be processed for complicated shapes.

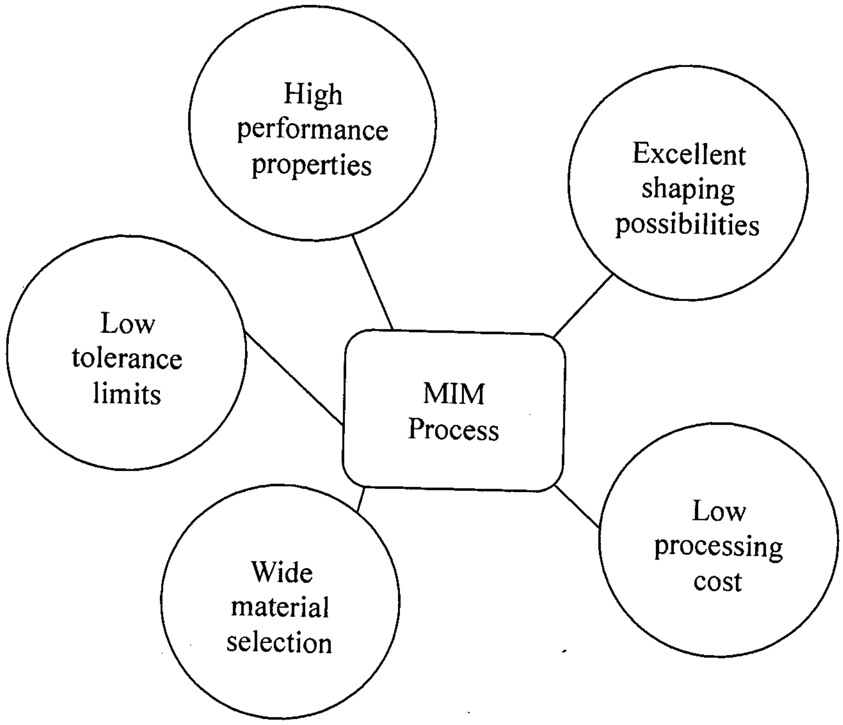

Fig. 1.2 The advantages of MIM process.

Another good point about MIM process, the feedstock material can be recycled nearly 100 %. This will give high benefit in cost reduction especially for the expensive materials. The total cost of mold, and equipment for debinding and sintering processes can be reduced by increasing the amount of production. In other words, the mass production is suitable for low cost of MIM process. The summary is that, to produce complex components at low cost, MIM process is expected to be one of suitable processes. Furthermore, it is possible to treat the components thermally or mechanically in order to achieve higher mechanical properties, narrower tolerances and lower surface roughness.

To know when in the process the heterogeneity occurred in the compact, flow of the MIM process should be explained here. In this study, promotion of heterogeneity structure into the compact begins at a very first instance during fabrication process. Two different elemental powders were prepared; Fine carbonyl Fe, and ultra-high pressure water-atomized Ni. These elemental powders were gently blended to form a balance dispersal of Fe and Ni particles in the powder mixture. It also has been reported that the heterogeneous microstructure of alloy containing Ni is easily attained. During sintering step, when Ni diffuses into Fe matrix, the carbon tends to repel the Ni, since Ni increases the chemical potential of carbon [38)].

The flow of the MIM process is illustrated in Fig. 1.3. The process of metal injection molding is very similar to that of polymers [25,28-29)]. The size of the powder particles ranges between 1 μm and a few of 100 μm. The most appropriate size of an individual particle for the process of MIM is smaller than 30 μm, and the average size of a particle is approximately 6 to 7 μm. The powder particles can be of various shapes, although the desired shape is a round one. As a result, it is possible to overcome certain anisotropic characteristics of a product while shrinking.

Molding is done by a conventional injection molding machine. The mold is designed similar to that for polymer but with consideration of shrinkage during subsequent debinding and sintering process. The parts are removed from the mold. At this point, the part known as "green" parts.

Before sintering, binder has to be removed. The binder is removed by heating, chemical extraction, or catalytic reaction. The debound part is called "brown". The binder removal process is called the debinding process.

The brown parts then subjected to sintering process. The purpose of sintering is to densify the powder and to remove most of the void space. The final density of the sintered part reaches from 94-99 % of the theoretical density. Ability to acquire high density compact which is comparable to the wrought alloys, is a crucial factor that contribute to the excellent mechanical properties offered by the compact.

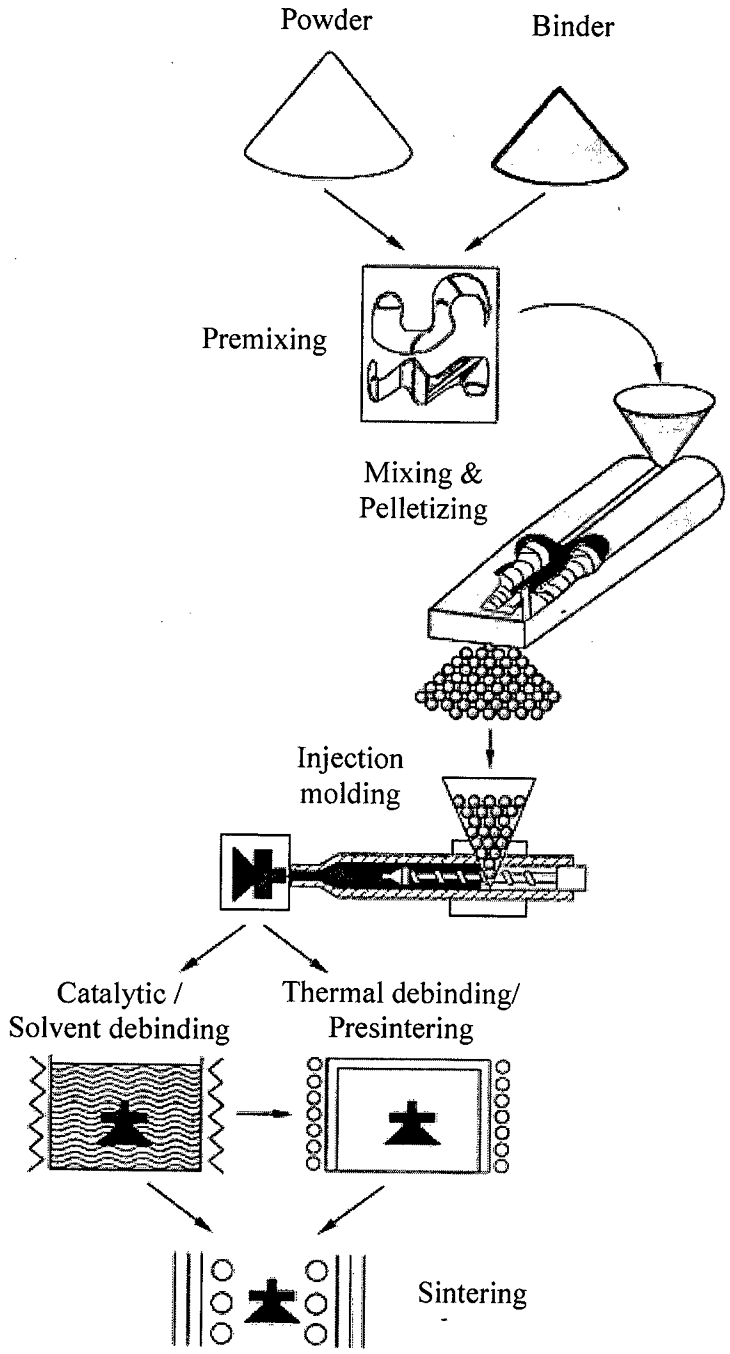

Fig. 1.3 Schematic diagram of MIM process [28,37].

1.2 OBJECTIVE OF STUDY

The work presented in this thesis is a first steps in understanding the complex microstructural morphology of the superhigh strengthened MIM Fe-Ni steel compacts at micro level. The experimental works have been extended numerically for more comprehensive studies of the matter. The data gathered here will serve as the foundation for the continuing design and development through understanding and careful control of microstructure and its effect on the properties of high-performance alloy compacts due to their heterogeneous and multiphase nature.

Firstly, the work began by systematically examination of the effect of residual pores on the tensile behavior of high strengthened steel compact experimentally, before continued by numerical simulation for verification. The 440C steel compact was selected as a model material. Although the compact was comprised by many residual pores, the compact offered high strength with homogenous microstructure. Three different compact densities were examined. Two-dimensional (2D) pore-based numerical models were then utilized for simulation of the compacts with various natures of pores cluster in order to efficiently gauge the effect of pores to their mechanical properties performance. The simulated mechanical properties results were then compared to experimentally obtained data.

The primary focus of this study is to clarify and to optimize the relationship between mechanical properties and the nature of heterogeneous microstructure for superhigh strengthened Fe-Ni steel compact through varying particle size of the Ni powders, Ni contents, and sintering conditions (temperature and time). In this study, the heterogeneity structure is mainly referred to the various concentration of Ni content throughout the compact matrix. There are two microstructural regions have been identified that play important roles in controlling mechanical properties; the higher Ni region, and the surrounded matrix (lower Ni region). These regions are the fundamental elements of complex Ni network of superhigh strengthened Fe-Ni steel compact. From crystalline structure point of view, the higher Ni is the region of Ni rich phase while the lower Ni region structured by the tempered martensite [1]. Thus, a thorough understanding about the nature of this complex heterogeneous Ni structure is needed before further adventures into reinforcement phase of the superhigh strengthened Fe-Ni steel compact.

Further efforts through FEM modeling to characterized unique connections between variations of Ni content to their mechanical properties have been carried out. This has been done by creating micromechanical models based on the microstructural data available experimentally. Modeling by the FEM was divided into three different parts; geometry, boundary conditions, and constitutive material properties.

1.3 OUTLINE

A general introduction is given in Chapter 1 together with its comprehensive background information about superhigh strengthened Fe-Ni steel compact, microstructural simulation, steel compact, and injection molding process. The chapter also clarified objectives of the study. Then, Chapter 2 describes comprehensive studies about the effect of residual pores on the mechanical properties of the high strength steel compacts through experimental followed by FEM analysis. After the effect of pores on the mechanical properties is concluded, an experimentation of superhigh strengthened Fe-Ni steel compacts continued in Chapter 3. All data obtained from experimental works are appropriately presented and discussed. In Chapter 4, FEM simulation of superhigh strengthened Fe-Ni steel compact is performed. The 2D microstructure-based model is developed based on actual heterogeneous microstructure that of experimentally obtained from Chapter 3. In Chapter 5, all conducted research works are thoroughly concluded. The chapter also gives clear directions of possible future works about current study.

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CHAPTER 2

Effect of Pores on the Mechanical Properties of High Strengthening Steel Compacts

2.1 INTRODUCTION

Though the general effects of pores on the tensile behavior of MIM compacts have been reported [1-5], a comprehensive and quantitative understanding of the effect of pores on the mechanical properties of MIM compacts is still not enough. Although the steel compact demonstrated high strength characteristics with homogeneous microstructure, many residual pores are also found throughout the matrix. Since the pores decrease mechanical properties, this is a common challenge when dealing with any alloy steel fabricated by P/M process. In this chapter, the effect of pores on the tensile behavior of a high strengthened steel is systematically examined using experimental and numerical techniques. Finite element method (FEM) was used to simulate the microstructural effect. Similar methodology will be also utilized to evaluate the heterogeneous microstructure of superhigh strengthened Fe-Ni steel compact in chapter 4.

The 440C stainless steel was employed as an example material to evaluate the effect of pores. It is a high carbon martensitic stainless steel with moderate corrosion resistance, high strength, excellent hardness and wear resistance. Due to these excellent properties, the steel has been used for many applications, such as ball bearings and races, gage blocks, molds and dies, knives and measuring instruments. However, when high strength 440C steel compact is considered over wrought alloy steel, many residual pores are typically characterized usually after sintering, which quite detrimental to their mechanical properties [6-7].

In this chapter, the main factor investigated by a series of experiments was on variability of the powder loading of the 440C alloy steel feedstock. As a high performance process, MIM has very high demands on compact with high sintered density. In this respect, the powder loading of a feedstock plays a key role. It has to be as high as possible in order to obtain higher densification during sintering. On the other hand, if it is higher than critical powder loading, this may result in lower sintered density due to increase in resistance between powder particle during injection molding and may cause process instability. The importance of the powder loading for obtaining good sintered density or less residual pores has been widely discussed in the literature [(8-9)].

Quantitative analysis on the microstructure of the high strength steel compact was then performed in order to determine the pore size distribution and the pore shape. Finally, pore microstructure-based numerical models were successfully developed to simulate tensile strength and elongation behaviors, before compared with experimentally obtained data.

2.2 EXPERIMENTAL METHOD

2.2.1 Powder Characteristic

An ultra-high pressure water-atomized 440C stainless steel powder was supplied by Mitsubishi Steel Mfg. Co. Ltd., Japan (MHT440C). The mean particle size was in the range of 9 to 12 μm (D_{50}=9.64 μm). As shown in Fig. 2.1, the powder shapes were dominated by irregular which will provide an excellent inter-locking effect between particles in the compact [10)]. However, it also has a major influence on the apparent density, flow properties, green strength, and compressibility of the powder; it also affects sintered properties, including dimensional change and mechanical properties. The irregularity of the shapes causes more contact points between particles during molding.

Therefore, higher compacting pressure was required to achieve proper green compact density, which is related to mechanical properties after sintering [11-12)]. More detailed powder characteristics are given in Table 2.1.