DEVELOPMENT OF AN ADVANCED COMPOSITE MATERIAL CONSISTING OF IRON MATRIX REINFORCED WITH ULTRA HIGH TEMPERATURE CERAMIC PARTICULATE (TiB_2) WITH OPTIMUM PROPERTIES

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Title

Development of an Advanced Composite Material Consisting of Iron Matrix Reinforced with Ultra High Temperature Ceramic Particulate (TiB_2) with Optimum Properties

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The Supervisory Committee certifies that this ***disquisition*** complies with North Dakota State University's regulations and meets the accepted standards for the degree of

MASTER OF SCIENCE

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ABSTRACT

This study was intended to investigate the mechanical properties and microstructure of iron based composite reinforced by ultra high temperature ceramics fabricated by powder metallurgy technique. The fabrication parameters were optimized and composite samples with different volume fraction of TiB_2 were fabricated and were subjected to different mechanical tests. The results indicated improving in mechanical properties of Fe-TiB_2 composites by increasing the volume fraction of TiB_2 up to 20 vol%. More TiB_2 particles didn't improve the mechanical properties of composite, instead adversely affected it due to increasing the chance of agglomeration and porosity in entire microstructure. Another finding showed the twofold characteristic of TiB_2 on mechanical properties of composite via increasing the hardness and decreasing the bulk density of composite. Finite Element Analysis (FEA) have also been performed on microstructural based developed models to simulate failure of composites. Numerical simulation results could verify the experimental results.

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My profoundest thanks go to my advisor, Dr. Fardad Azarmi. His respectful professional character along with his sincere commitment and patience to help the students to be better versions of themselves in their research and future careers, make Dr. Azarmi an exceptional advisor to work with and I'm grateful for the chance of being in his research team.

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DEDICATION

To My Wife & Parents

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1. INTRODUCTION

Innovation and development of advanced materials to meet the demands of emerging new applications is a very important area of research in academia and industry. Particularly, materials which can exhibit better performance at extreme working conditions while can be produced by possible cost effective techniques would be very attractive. In this regard Metal Matrix Composites (MMCs) have attracted much attention due to their capabilities to offer a various range of mechanical and physical properties. Ductility and formability of metals along with strengthening properties of reinforcements have made MMCs competitive candidates for wear-resistant and high temperature applications in aerospace and automotive industries. In the present work, Iron-based matrix (Fe) has been reinforced with Titanium Diboride (TiB_2) ceramic particles. TiB_2 ceramic particles are expected to be a suitable reinforcement for iron and steel matrix composites due to their high thermal stability at very high temperatures, high modulus of elasticity, good wettability, low density, and relatively strong bonding between matrix and reinforcement. MMCs reinforced with Ultra High Temperature Ceramics (UHTCs) can be a potential candidate in applications with high wear resistance properties, applications with high ratio of strength to weight and applications which require mechanical and chemical stability at high temperatures.

The effect of manufacturing parameters of Powder Metallurgy (P/M) technique on mechanical properties of Iron-based composited reinforced by UHTCs are not well documented yet. There are several PM process parameters affecting mechanical and physical properties of MMCs. However to the author's knowledge, there is no specific standard on PM process parameters to fabricate Fe-TiB_2 composites. The main objective of this study is optimizing PM process to fabricate Fe-TiB_2 composite using PM technique. Effects of pressure, time, and temperature during the cold pressing, hot pressing and sintering on mechanical properties of Fe-

TiB_2 have been investigated experimentally and an optimized cost-effective PM process was suggested for fabrication process.

In the present study, 80 vol% Fe and 20vol% TiB_2 samples were first fabricated using PM technique to optimize the fabrication parameters. This vol % for the reinforcement phase was only chosen as a starting point for this study. Next, four sets of MMC samples were fabricated with different vol% of TiB_2: 0, 10, 20 and 30 vol%. The microstructural and mechanical characterizations were performed on MMC samples. The results were compared in order to explore the influence of the volume fraction of the TiB_2 on the microstructural and mechanical properties of Fe based composite.

2. LITERATURE SURVEY

2.1. Introduction

A composite material can be defined as a combination of two or more different materials, which the combination has superior properties than individual components. Generally the constitutional materials in a composite are reinforcements such as fibers, particles, and flakes which embedded into a matrix such as polymers, metals, or ceramics. The matrix is responsible for holding the reinforcement to form the required shape while the reinforcement increases the mechanical properties of the composite. If designed and manufactured properly, the fabricated composite shows better strength than each individual ingredients [1]. As studied by Jartiz, [2] composites can show significant characteristics which cannot be gained from its individual components. Kelly [3] emphasized that composites should not be simply considered just as a combination of two or more materials but in a broader context, composite is new material that has own specific characteristics. Physical and mechanical properties of composite materials have been shown that are improving rather than its components alone. Berghezan [4] mentioned: "the composites are compound materials which differ from alloys by the fact that the individual components retain their characteristics but are so incorporated into the composite as to take advantage only of their attributes and not of their shortcomings". Van Suchetclan [5] considered the composites as heterogeneous materials consisting of two or more solid phases, which are in intimate contact with each other on a microscopic scale. He also believes composites can be considered as homogenous materials on a macroscopic scale, considering this point that any small portion of composite might have same physical characteristics [5].

Composite materials are being used in a broad range of applications and still due to continuously growing demand for new high-performance materials, more new composite materials

are needed to be designed. Currently, composite materials can be found in very diverse applications, from sport and recreational applications to automotive, biomedical, and aerospace applications [6-9]. For instance in aerospace and aviation industry in 1980 less than 10% of airplane components were made from composites but right now approximately 50% of components are made from composites. For this specific field of application, the major advantage of composites is reduced weight and assembly simplification. In fact steady improvement in transportation/automotive market, aerospace and construction sectors as well as recovery in the wind energy market are stimulating more demands of composite market. Currently China, the United States, and Germany are the largest markets for composite materials [10-11]. In the U.S. composite market is expected to reach $12 billion by 2020 [10]. There are some driving forces behind this growth. Figure 2.1 shows the forecast of composite materials for different sections of applications by 2020 in the US market [11].

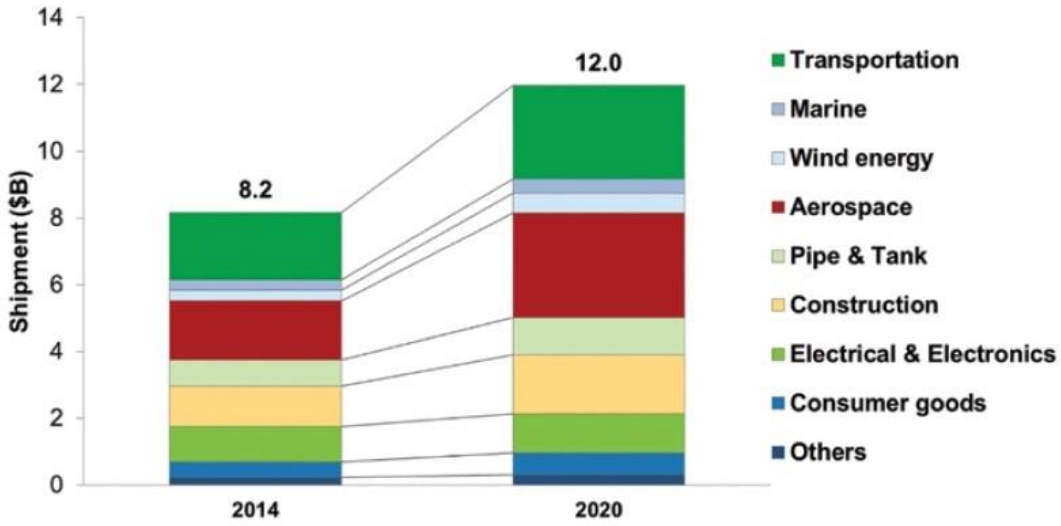


Figure 2.1. Forecast of composite materials for different sections of applications by 2020 in the US market [11].

Composite materials can be divided in many different ways, but classification based on the materials of matrix and the geometry of reinforcement are the most common ways. Based on

the material of matrix phase, composites can be categorized into: Metal Matrix Composites (MMC), Ceramic Matrix Composites (CMC), and Polymer Matrix Composites (PMC). Based on the geometry of reinforcement, composites can be divided into three main groups of particle, fiber, and structural reinforcement. [12, 13]. Figures 2.2 and 2.3 show the two classifications of composites, based on the material of matrix and the shape and geometry of reinforcement.

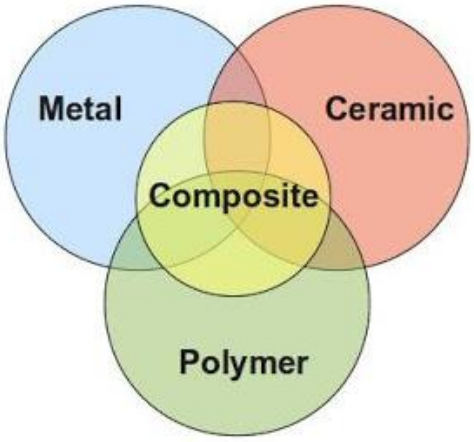


Figure 2.2. Composite Classification based on the materials of matrix.

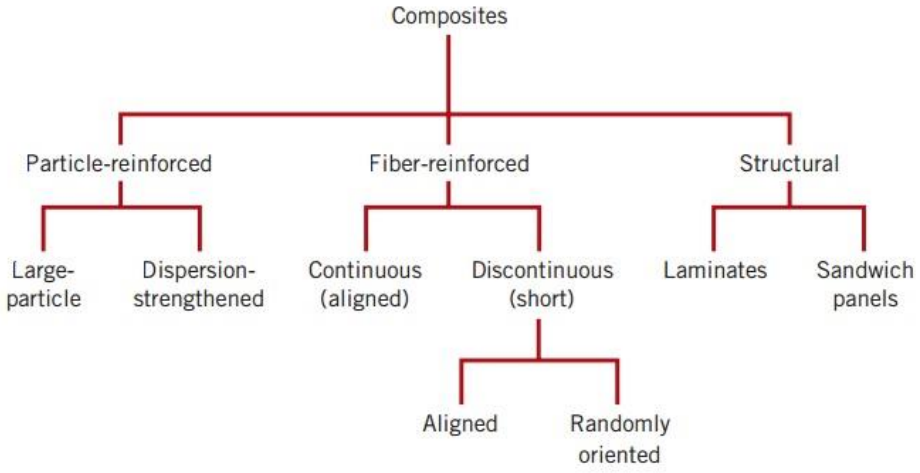


Figure 2.3. Classification of composite materials based the shape of reinforcement. Adapted from Fig.16.2, Callister 8e [13].

2.2. Metal Matrix Composites (MMCs)

Innovation and development of advanced materials to meet the demands of emerging new applications is a very important area of research in academia and industry. Particularly, materials which can exhibit better performance at extreme working conditions while can be produced by possible cost effective techniques would be very attractive. In this regard MMCs are potential candidate for many applications in automotive industry, biomedicine, aviation and etc. In fact MMCs are multi-phase materials in which a strong and stiff reinforcing phase, typically a ceramic, is incorporated with a soft and ductile metal phase [14]. The possibility of combining various material systems (metal-ceramic-nonmetal) gives the opportunity for unlimited variation. The properties of these new materials are generally determined by the properties of individual components [15]. Adding ceramic particles such as SiC, Al_2O_3, TiC, and TiB_2 to the metal matrix, improves mechanical properties and wear property of composite [16, 17]. MMCs have many applications in the ground transportation, thermal management, aerospace, industrial, recreational and infrastructure industries. This vast domain of applications is enabled due to functional properties such as excellent wear resistance, good thermal and electrical characteristics, high strength and stiffness and improved strength to weight ratio compared to non-reinforced alloys [18]. Figure 2.4 shows some applications of particulate reinforcement metal matrix composites.

Figure 2.4. Some applications of MMCs with ceramic reinforcements in biomedical and industrial parts [19].

As it can been seen in Figure 2.5, high Young's modulus of ceramics can make them a very good candidate for strengthening the metal matrix phases. In recent years, many studies have been reported on the use of conventional ceramics as reinforcing phase in composites, with metal matrix of aluminum, copper, iron, cobalt and their alloys. However, most of the studies have focused on Al based systems, dealing with the effect of reinforcement size, shape, and/or volume fraction [20].

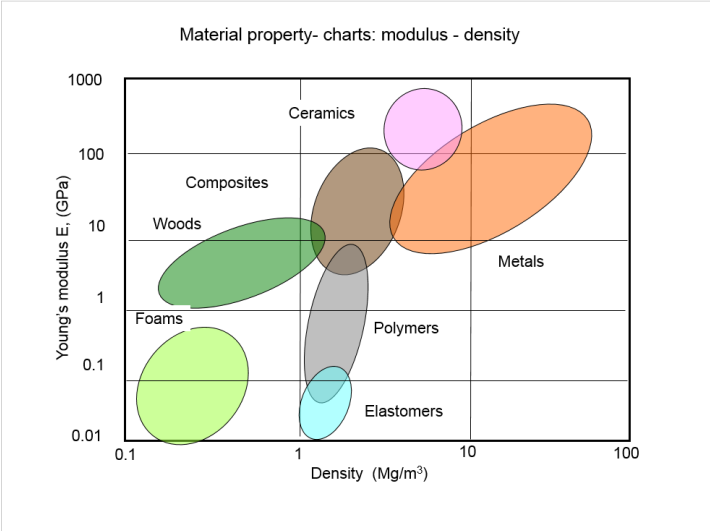


Figure 2.5. Classification of engineering materials based on Young's modulus [21].

2.3. Classification of Metal Matrix Composites (MMCs)

MMCs can be classified in different ways. One classification is considering the geometry and contribution of reinforcement in matrix phase. In broader view, MMCs can be divided in two main groups. As it can be seen in Figure 2.6, the first group are reinforced by discrete particles and the second ones are reinforced either by fibers or filaments. The second group can be further categorized into continues reinforcement (mono or multifilament) or discourteous reinforcements (whiskers or short fibers).

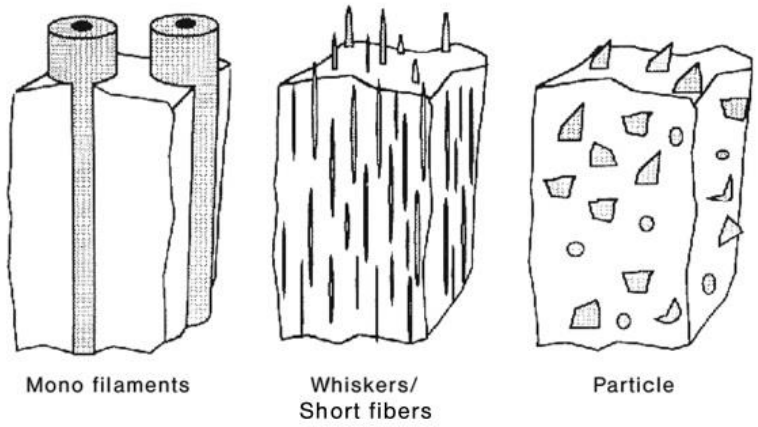


Figure 2.6. Schematic presentation of three shapes of metal matrix Composite materials [22].

2.3.1. Dis-continuously Reinforced MMCs

2.3.1.1.Particulate MMCs

Since uniformly distributed particles can create isotropic property, these type of reinforcement are being widely used in MMCs fabrications. Oxides, Nitrides, Borides, and Carbides are among the most common ceramic particles that are currently being used. The particle can be spheres, cubes, or irregular particles [23].

2.3.1.2.Short Fiber MMCs

Fiber reinforced metal matrix composites have reinforced by discontinuous fibers that are typically circular and vary in diameter from 0.1mm to 0.1 cm [24], with aspect ratios ranging, typically, from 3 to 100 [14]. Fibers increase the strength of composite in the direction of their orientation. Lower strength in the direction perpendicular to the fiber orientation is characteristic of continuous short fiber reinforced MMCs [25]. Formability is one of the main characteristic of this composites [26].

2.3.1.3.Whisker Reinforced MMCs

Although whisker reinforcements are very similar to the short fiber ones, there is a big difference between two types of reinforcements. Whiskers are mono crystalline [27] and usually have a diameter less than 1μm, with aspect ratios up to several hundred [28]. Whiskers are characterized by their fibrous, single crystal structures, which have no crystalline defect. Numerous materials, including metals, oxides, carbides, halides, and organic compounds have been prepared under controlled conditions in the form of whiskers [29].

2.3.2. Continuously Reinforced MMCs

Carbon fibers and ceramic such as alumina, silica, boron, zirconia, magnesia, boron nitride, titanium diboride, and etc. are being used as continuous reinforcements as continuous reinforcements. The common similarity among many of these ceramic fibers is that they are brittle and flaw-sensitive reinforcements. It should be taken into the account that these types of reinforcements are very sensitive to the size effect. It has been proven that from a certain size the strength of these fibers decreases as the length increases. It also should be added that these types

of reinforcements mostly are coated with materials like boron carbide to make better joints at interfaces [30].

2.3.2.1. Monofilament MMCs

These reinforcements have large diameter typically 100μm to 150μm. The materials are usually consisting of SiC, boron, or aluminum oxide or refractory metals which have been deposited by chemical vapor deposition on to a carbon or tungsten wire core [28, 31]. The monofilament fibers are usually aligned in a unidirectional way within the matrix. It should be noted that these fibers do not show so much flexibility [14].

2.3.2.2. Multifilament MMCs

In comparison with monofilament reinforcements, multifilaments are small in diameter in the range of 5μm to 30μm which enables them to be woven, knitted, stitched, braided, or wound. These fibers have a small bend radius that improves their flexibility. Because of the flexibility of these fibers they can be incorporated into a matrix in a unidirectional manner or combined [14].

2.3.2.3. Layered MMCs

This type of MMCs have reinforcements in layer shapes. These layers can be in many different thicknesses ranging from few nanometers to centimeters. These reinforcing layers might be coated with some extra materials in order to create better joints. Layers can be used as prepared tapes or can be deposited on the matrix phase [32].

2.4. Materials Selection for MMCs fabrication

The best properties can be obtained in a composite system when the reinforcement and matrix are as physically and chemically compatible as possible. Special matrix alloy compositions,

in conjunction with unique whisker coatings, have been devised to optimize the performance of certain metallic composites [37-41].

2.4.1. Matrix

In a composite material, the matrix can be defined as a continuous monolithic material which its principal role is giving shape to the composite sample, moreover it holds the reinforcements in their positions. Isolating the reinforcement from one another can prevent abrasive reinforcement to touch each other. In fact the matrix is like a bridge which transforms applied stress to the reinforcement [23]. Besides load transferring, in some composites the matrix is responsible to conduct heat and electricity in some specific applications of MMCs. In material design of MMCs, several factors should be taken into account when materials are being selected for matrix phase. Chemical compatibility with reinforcing materials and ability to wet the reinforcement are the main considerations in choosing a material as matrix [33]. Obviously, the favorable matrix allows sufficient reactivity to be done for enough wetting without creating excessive unwanted reaction which can significantly degrade the reinforcement properties in composite. Beside this, the matrix should be able to transfer the applied load to reinforcement which requires a coherent interface between matrix and reinforcement [34, 35]. Other potentials that a matrix could have as advantages are factors such as, low concentration of impurities which helps for creation of a better bonding [36]. The selection of a matrix of a composite is also directly depends on the requirements of final composite, for instance, in contact points which are being used in high-voltage posts, the parts should be highly conductive and on the other hand should have high strength and resistant to wear. In such a case a metallic matrix reinforced by ceramics or intermetallic can be a good candidate. Aluminum and its alloys, Nickel, Iron, Copper,

Magnesium, and Titanium alloys are among matrix materials that are being widely used in industry [25].

2.4.2. Reinforcements

The responsibility of the reinforcement phase is to strengthen and stiffen the composite by preventing deformation of matrix. In fact reinforcing phase acts like a mechanical restraint, which this restraint is basically a function of many factors such as size of reinforcement. In many application reinforcement phase is the major load-bearing component in the composite [25]. Basically by adding of reinforcement into the composite system, the strength, stiffness, and temperature capability increase meanwhile the coefficient of thermal expansion of resulting MMC probably decreases. When combined with a metallic matrix of higher density, the reinforcement also serves to reduce the density of the composite, thus enhancing properties such as specific strength [23, 25, 42, and 43]. Specifically speaking in MMCs, common reinforcement materials are ceramic materials such as nitrides, carbides, borides and oxides which are well known for their high strength and stiffness at room and elevated temperatures [44]. Multi factors should be considered for selecting a material as reinforcement. Some factors are listed as follow [15, 22, 45, and 46]:

1) The dimension, size, and aspect ratio of reinforcement.
2) Shape of reinforcement: fiber, whisker, spherical or irregular particulate, and flake.
3) Smoothness and roughness of reinforcement's roughness.
4) Crystal structure of reinforcement; Poly or single crystal.
5) Inherent defects in the structure of reinforcement.
6) Composition of the layer/film formed on the surface of reinforcement.
7) Impurities of reinforcements.

8) Mechanical/physical properties of reinforcement: strength, modulus and density.

2.5. Fabrication of Metal Matrix Composites

There are several fabrication techniques available to manufacture the MMC materials depending on many factors such as materials of matrix and reinforcement, shape and size and reinforcement, required properties from final samples and etc. All fabrication techniques can be classified into four main categories:

(a) Solid state methods,

(b) Liquid state methods,

(c) Deposition techniques, and

(d) In-Situ technique.

2.5.1. Solid State Methods

In solid phase processes, MMCs are formed as a result of creation of bonding between blended elemental powders in solid state at elevated temperature and under pressure. Powder Metallurgy (P/M), diffusion bonding, hot rolling, extrusion, drawing, explosive welding and pneumatic impaction are examples of solid state methods. In solid state methods, the low temperature of process compared to liquid state techniques, is an advantage. In fact at low temperatures, many undesirable reactions at interface of matrix and reinforcement cannot take place [47-49]. Solid state processes are generally used to obtain the highest mechanical properties in MMCs, particularly in discontinuous MMCs this is because segregation effects and brittle reaction product formation are at a minimum for these processes, especially when compared with liquid state processes [50].

2.5.2. Liquid State Methods

In this technique, the reinforcements are introduced into a molten metal, then liquid composite is cast into various shapes by conventional casting techniques or can be cast into ingots for secondary processing and finally the system ends up by solidification of matrix phase. To reach the highest level of mechanical properties of composite, a good wettability between the dispersed phase and the molten metal is required. In some cases, in order to improve the wettability between the dispersed reinforcement and matrix phases, the reinforcement can be coated or wetting agents can be added into the liquid. The coating can not only increase wettability but it can prevent some undesirable chemical reactions between two phases [49]. A big number of MMCs are fabricated by liquid state methods. The main advantages of liquid state method is that the cost of relative low cost of the process. However, this technique is also associated with some disadvantages such as non-wettability between the reinforcement and matrix phases which can cause the rejection of reinforcement particles from the melt and consequently non-uniform distribution of reinforcement and preferential segregation and significant decrease in mechanical properties. Liquid sate methods can be achieved in various ways such as Liquid metal infiltration, Squeeze casting, Spray co-deposition method, Stir casting, and Compocasting [23, 44, and 51].

2.5.3. Deposition Techniques

Deposition techniques for MMCs fabrication involves a process, in which matrix metal is deposited together with the dispersed reinforcement phase, followed by diffusion bonding to form a consolidated composite plate or structural shape. Since the composite is composed of identical units, the microstructure is more homogeneous than that of cast composites. There are several advantages in using deposition techniques such as (a) Good control on impurity during process, chemistry and homogeneity in composites (b) Clean environment and process, (c) Repeatability

of process is high .Several deposition techniques are currently available such as Immersion plating, Electroplating, Spray deposition, CVD, PVD, Spray forming techniques. Electrolytic, Spray and Vapor depositions are different ways of this technique [52-53].

2.5.4. In-Situ Technique

In these techniques the reinforced phase is formed in situ. The composite material is produced in one step from an appropriate starting alloy. There are several different processes that would fall under this category. Directional solidification of eutectics in which one of the phases solidifies in the form of fibers is one example of such processes. Inherent limitations in the nature and volume fraction of the reinforcement and the morphological instabilities associated with thermal gradients have resulted in a decrease in the interest in these types of composites. Exothermic reactions, such as directed metal oxidation, are one family of processes for the production of in-situ composites. The major advantage of this class of composites is that the in-situ reaction products are thermodynamically stable. [54, 55].

2.5.5. Powder Metallurgy

Powder Metallurgy (P/M) process is the most commonly used technique for fabrication of discontinuous reinforced MMCs [56]. This process is able to be used as a fabrication technique for both particle and whiskers reinforcements. Basically in this technique the powders of matrix and reinforcement are completely mixed. After mixing, the powders are fed into a mold which is designed in desired shape. The next step is cold pressing in which, pressure is applied to the powders in the mold. In order to increase the chance and possibility of bonding between the powder particles, at the next stage which is called hot pressing, the compacted powders are heated while powders are under pressure. It should be noted that compacted powders at hot pressing stage are

heated to a temperature below the melting points of matrix and reinforcement, but this temperature is sufficiently high to make bonding at the interface of matrix and reinforcement. At the sintering stage which can be either under pressure or without pressure, the compacted powders are heated in a controlled atmosphere to reduce the amount of porosity and consequently increase the density of composite. Some MMCs might need some secondary operations such as extrusion or forging [23]. Figure 2.7 shows different shapes of particulate reinforcements commonly used in P/M technique.

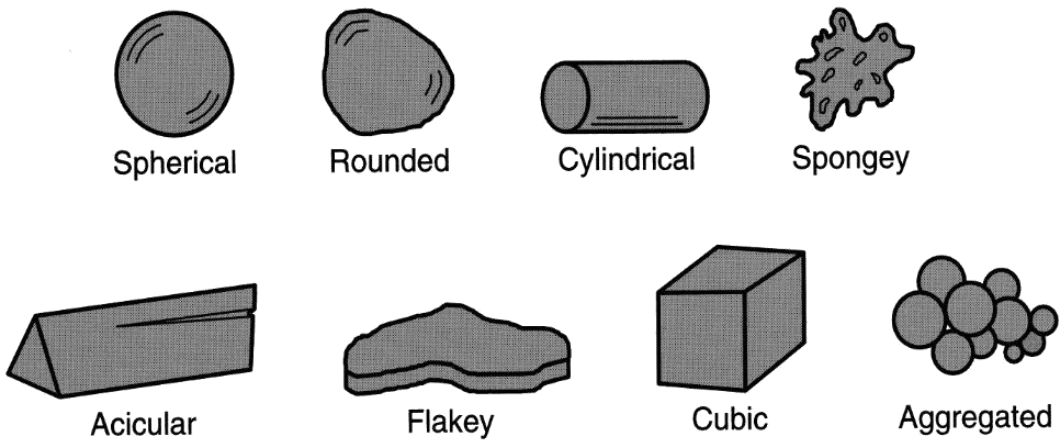


Figure 2.7. Different shapes of powders used in P/M technique [12].

2.5.5.1. Mixing and Blending the Powders

Mixing of powders is an important stage in fabrication of MMCs because it can affect the final mechanical properties of MMC. Mixing of reinforcement particles in matrix can be time consuming and expensive due to high level of cleanliness and reaching to a homogenous distribution of reinforcement. Any kind of inclusions incorporated into the mixing process can adversely affect the mechanical properties such fracture toughness and fatigue life [14, 57, and 58].

As it can be seen in Figure 2.8, there are different ways of mixing the powders in P/M technique but it can be said that double corner mixer, ball milling and V-mixer are the most common ones. Basically, there are some factors that make the mixing process difficult. For instance, as the size of powders get smaller, or when there is big difference between the mass of particles the mixing process would be more difficult. In this regard, it should be added that irregular shapes are more difficult to mix rather than regular shapes [57and 59].

Many factors play an effective role in the mixing of powders which some of them are listed below:

1. Type of mixer

2. Volume of mixer

3. Geometry of mixer

5. Constructional material of mixer

9. Volume ratio of component powders

4. Inner surface area of mixer

6. Surface finish of mixer

7. Characteristics of powders

8. Rotational speed of mixer

9. Mixing temperature

10. Humidity when mixing in air

11. Mixing time

12. Mixing agent

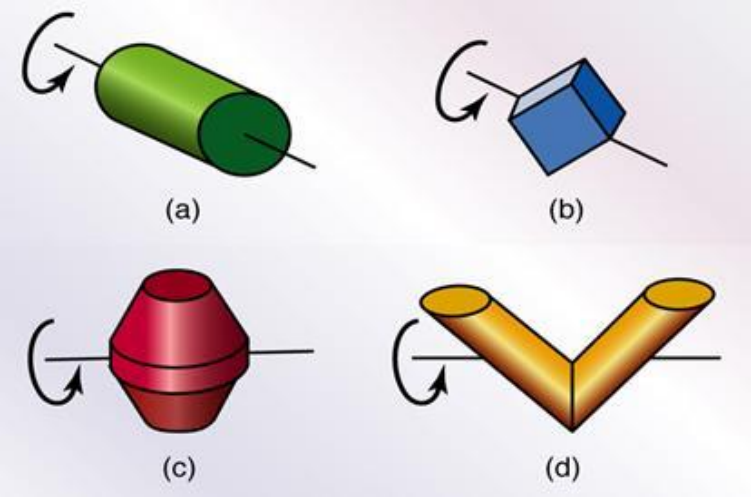


Figure 2.8. Schematic of Different methods of mixing the powders [60].

2.5.5.2. Powder Compaction

2.5.5.2.1. Cold Pressing

After mixing the powders, the next step would be cold pressing. Basically, cold pressing has three main functions [59].

(a) Creating the desired shape and dimensions

(b) Control the porosity level

(c) Creating adequate strength for handling the sample for the rest of the process

The pressure at this stage, creates a kind of cold welding bonds which holds the part together. The outcome of cold pressing is compacted billet which is called green billet. As it can be seen in Figure 2.9, cold compaction can be performed in two ways: single and double unidirectional pressing (two moving punches) [61]. It has been seen that if the ratio of height/diameter of cold compacted billet is height, then more density variation of final sample is expected [62]. The pressure at which the powders are pressed has a large effect on the green density of the material [63].

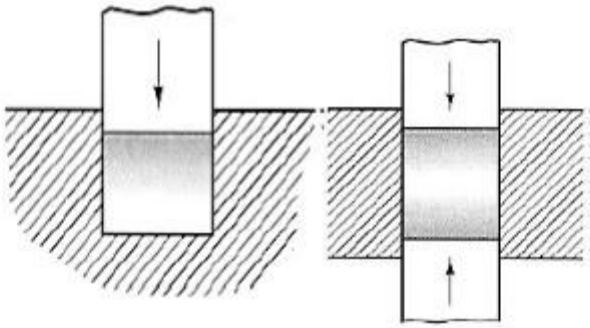


Figure 2.9. Single and double unidirectional pressing [59].

2.5.5.2.2. Hot Pressing

Generally, metals become softer at elevated temperatures (rather than room temperature) and for this reason it is usually possible to reach to higher densities by applying pressure at high temperatures. Although in some cases hot pressing process can be skipped from P/M process, in general it can significantly improves the mechanical properties of final parts. Some factors that can limit its application such as high cost of dies and tools which should tolerate high temperature and high pressure, the slow rate of process and the high costs of energy [64].

2.5.5.3. Sintering

Sintering is a solid state process carried out at temperatures below the melting point. In fact sintering is a diffusion based process which from thermodynamic point of view, its driving force comes from surface area reduction of joining particles and hence reduction in surface energy and consequently the energy of system decreases [12, 14]. Temperature and time of sintering are the main process factors in sintering stage which can affect the strength, elastic modulus and ductility of the composite. Increasing the sintering time and temperature can increase the density of composite and decrease the porosity of microstructure and hence decreasing the porosity means

improvement of mechanical properties [59, 65]. Time and temperature of sintering are mainly determined by the type of consisting materials in the composite and also by expected strength from final fabricated parts [66]. Sintering can be divided into four separate stages as it shown in Figure 2.10: Primary or green compact, Initial stage of bonding called necks, Intermediate stage and final stage [67]. Figure 2.11 shows a schematic illustration of developing the necking zone which is associated by the reduction of surface areas of two joining particles.

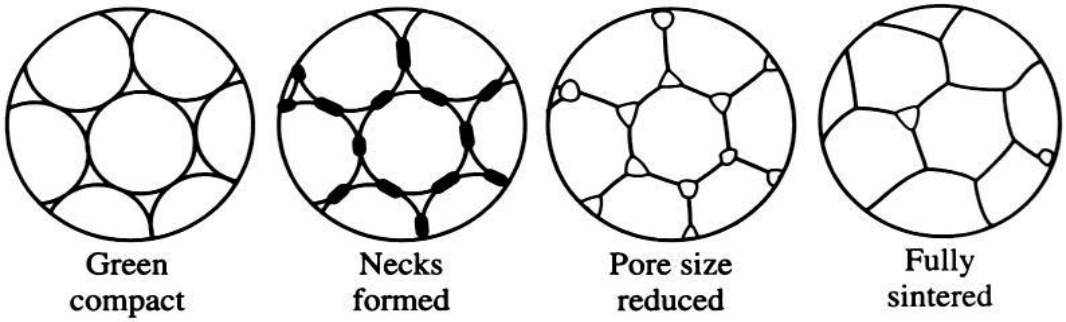


Figure 2.10. A schematic view of microstructural evolution during sintering process [12, 60].

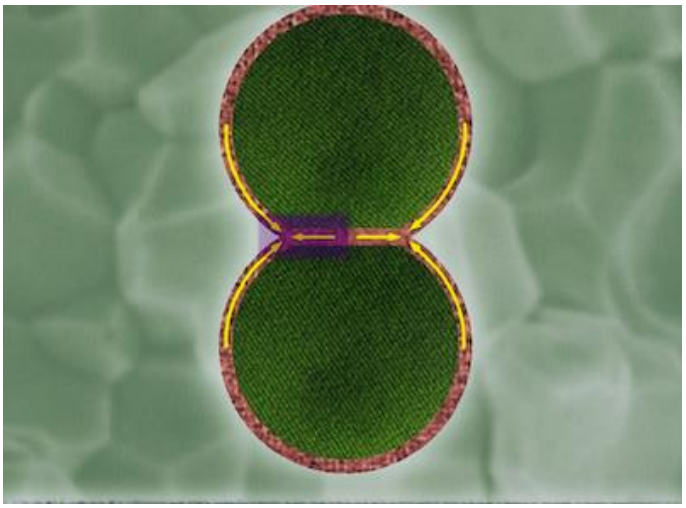


Figure 2.11. A schematic illustration and a high-resolution transmission electron microscopy image showing sintering process, Photo credit by: The cover of Journal of American Ceramic Society, August 2012 [68].

2.5.5.4. Secondary Operations

For some parts, the sintering can be the final stage of fabrication process while in some other cases depending on expecting properties or design considerations, some additional processes might be needed which are called secondary operations. Some secondary operation are listed below [66, 69]:

1) Heat treatment process such as annealing
2) Repressing
3) Machining and polishing
4) Forging, Rolling or Drawing
5) Coating
6) Infiltration

2.5.5.5. Advantages of P/M Process

P/M technique as a manufacturing process has many advantages that makes this technique as one the most attractive procedure for mass production in industry. Some of these points are listed below [66, 70, 71, 72].

1) The dimensions of finished parts are in very good accuracy. The Surface finishing quality is acceptable for many applications.
2) Unlike many other fabrications methods (Casting, Press forming…), the wasted material during the production is very low.
3) Some materials can only be used by P/M technique such as refractory metals and ceramics.
4) The production rate can be high.
5) Some metallurgical limitations such as phase diagram constraints which do not allow the formation of some alloys because of mutual insolubility, are not the case in P/M technique.

6) Effective control on the property of parts is high. Purity, density, particle size, precise chemical composition, porosity and etc. can be very well controlled.
7) P/M technique is the main fabrication technique of porous parts and for super-hard cutting tools bits.
8) In terms of health issues, the P/M workplace is quite and clean rather than many other mass production techniques.

2.5.5.6. Limitations of P/M Process

Beside many attractive points of P/M techniques, like any other fabrication technique it has limitations which are listed below [66, 70, 71, 72].

1) This fabrication technique is financially feasible for small-scale productions.
2) The tools and dies are expensive.
3) Metal powders and some ceramic powders are expensive and costly for long term storing.
4) The size of finished parts cannot be very big in comparison to casting methods.
5) Very complicated geometries cannot be fabricated by these technique because of poor flow ability of mixed powders.
6) Some deviations might be seen in density of finished parts.

2.6. Fe-TiB_2 Composites

Growing demand of advanced materials and composites for extreme condition applications motivated many scientists and engineers to search and study on new materials. In this regard, MMCs have been studied and improved over the years. In recent years, many studies were reported on using of conventional ceramics as reinforcing phase in composites with metal matrix of aluminum, copper, iron, cobalt and their alloys [69, 73-76]. The most commonly used ceramic

particles include oxides (e.g., Al_2O_3 and ZrO_2), nitrides (e.g., TiN and Si_3N_4), and carbides (e.g., TiC, Cr_3C_2, VC, and B_4C) [77, 78]. Among the reinforcing materials for MMCs, in recent years Ultra High Temperature Ceramics (UHTCs) have received special attention by high tech industries. The extraordinary mechanical and thermal properties of UHTCs make them a great candidate for being used as reinforcing phase for advanced MMCs. MMCs reinforced by particulate UHTCs are very desirable materials due to a synergetic effect caused by the combination of hard reinforcement and ductile metal matrix [79, 80]. Among these common ceramic reinforcements, TiB_2 is being vastly used for reinforcing steel matrix, due to its high thermal stability at higher temperature, high modulus of elasticity, good wet ability, low density and their relative stability with steel matrix [81-83]. These types of composites are promising candidates for many applications in the ground transportation, thermal management, aerospace, recreational and infrastructure industries. TiB_2 can be considered as a main candidate for reinforcing the MMC composites with applications where hardness, high melting point, electrical conductivity, reflectance, chemical inertness, radiation stability or light weight are expected. Such applications include microelectronics, electrodes, optical mirrors, cutting tools, wear- and erosion-resistant components [84–95], photovoltaic cells[87,96,97], chemical barriers that prevents diffusion [98] as well as many other applications. TiB_2 exhibits an extremely high specific modulus of 120 GPa/Mgm^{-3} and hence would be desirable in applications where component weight as well as stiffness are important. In this regard, F. Bonnet et al, suggested TiB_2 as the best reinforcement if the weight reduction as well as strength increasing are design criterions. [99]. High hardness (3400HV), high thermal conductivity (~110Wm^{-1} K^{-1} at 25℃) and low coefficient of thermal expansion (~7.2×10^{-6} K^{-1}) make TiB_2 unique among the family of ceramic reinforcements in terms of mechanical and physical properties. In terms of tribological properties, unlike most

ceramic reinforcements, TiB_2 exhibits a great chemical stability even in liquid iron [6, 100]. Moreover excellent wettability [4, 101-103] and good compatibility with iron-based matrices [102,103] are other advantages of using TiB_2.The microstructural characterization and damage mechanisms of Fe-TiB_2 have been investigated previously. Lime Cha et al. investigated the chemistry and atomic structure of Fe-TiB2 composites [104]. Their findings supported the fact that a good interfacial cohesion between Iron particles and Titanium Diboride particles enhanced the mechanical properties of resulting material. It has also been reported that due to the high strength of metal/ceramic material damage propagation in MMCs reinforced with TiB_2 mostly has an inter-granular character [105].

Depending on type of materials used as matrix and reinforcement, shape and size of reinforcement and the fabrication technique, MMCs can be designed and produced for different applications. For instance, environmental changes and global warming pushed the car manufactures toward designing new MMCs which exhibit higher strength while they have lighter weights in order to reduce fuel consumption and CO_2 emission. Iron-based composites have been considered a potential substitution for many conventional steel parts [106]. Manufacturing method, fabrication parameters and volume fraction of TiB_2, as the reinforcement, affect the mechanical properties of resultant MMC. Fe–TiB_2 composites can be produced by different techniques such as synthesis of TiB_2 phase in the system via in-situ techniques such as laser cladding [107], conventional melting and casting [108], plasma transferred Arc (PTA) [6], Aluminothermy Reduction [109], Self-propagating High-temperature Synthesis (SHS) [110], and Powder Metallurgy (P/M) [111-113]. P/M, is one of the most common methods for fabrication of MMCs reinforced by TiB_2 particles [114-117]. This technique involves mixing uniformly the powders of matrix and ceramic reinforcement followed by cold pressing, hot pressing and sintering. P/M

technique is associated with some inherent advantages such as being a versatile technique capable of modifying parameters based on desired properties, possibility of using ceramic-metal combinations not viable by liquid-state routes and producing near-net shaped components. Some of the drawbacks include cost-ineffectiveness, oxidization of powders which demands inert/protective gas conditions, and high porosity content (inevitable in powder metallurgy techniques) that leads to degradation in properties, especially the drastic reduction in ductility. [118,119].

2.7. Optimization of P/M Process

Basically optimization techniques are designed to determine the 'best' operating variables of a system that can create the highest achievable levels of performance for the system under the existing constraints. There different methods of optimization which can be used for different applications based on process design considerations. These methods can be divided into two main groups a) Deterministic methods and b) Probabilistic methods. In deterministic methods the outcome could be exactly predictable, but in Probabilistic methods only few estimations could be possible on the outcome. For example if a dice is rolled, it is expected that the result would be a number between 1 to 6 but it will be known only after physical visualization of the results. These two main groups of optimization techniques can be divide into many subcategories such as Blocking, Sizing designs, Plackett-Burman (screening) designs, Model validation, factorial designs, and etc. Reviewing all of the current optimization methods in detail, is not in the scope of this study, interested readers are encouraged to refer the main text books of industrial engineering or the sources provided in the reference sections [120-122].

2.7.1. Plackett-Burman (Screening) Design

According to the concept of probabilistic methods, it is possible to approximately predict the effect of process factors on the output of product but the degrees of influence and the relation between the technical process values and mechanical properties of product are still unknown. In this regard, Plackett-Burman design or screening methods were chosen to optimize the process. This technique is used when the process has different variables and the aim is distinguish the effect of most important factors. In fact in this technique the parameters and their effects are tested and screened to reach to the factors that create the desirable factors in final product [120], a schematic algorithm of this process is shown in Figure 2.12. The experimental outcome of this technique can be supported with mathematical models and equations which this part was not considered in this study because the experimental finding could create a meaningful relations between factors of process and properties of fabricated MMCs.

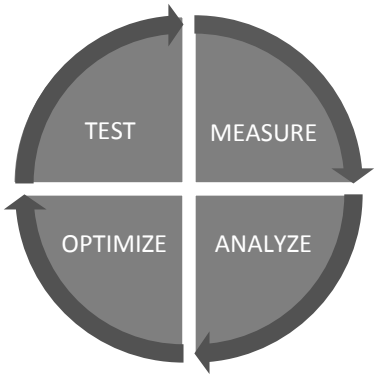


Figure 2.12. A schematic procedure of optimization process used; Plackett-Burman (screening) design.

2.8. Finite Element Analysis (FEA)

Finite element analysis (FEA) is a computerized technique which can help to predict the reaction of a product to the applied conditions in real world. The applied conditions can vary from force, heat, fluid flow, vibration as well as some physical effects. FEA is an effective technique in

academia and industry, especially when the conditions for doing real experiment are not technically or financially possible or in conditions when the experimental results need to be verified. In most of the FEA studies on particulate reinforced MMCs, for the reason of simplification, the reinforcing particles have been considered simple spheres or regular shaped phases uniformly or randomly distributed within metal matrix [123]. Prem et al. [124] showed that finite element analysis is able to accurately predict the properties of particulate reinforced metal matrix composites. Vaidya and Sun [125] used 2D And 3D axisymmetric models to predict the elastic properties this type of composites. Eckschlager et al. [126] used a simplified 3D model of uniform size spherical particles with random distribution to damage mechanism in particulate metal matrix composites. Soppa et al. [127] used 2D to study tensile behavior of MMC stripes reinforced by particle ceramics. Mammoli and Bush [128] showed that orientation of reinforcing particles can affect the elastic and plastic regions. Lin et al. [129] found that distribution of particles can significantly affect elastic and plastic properties. Huang and Bush [130] studied the effect of grain size on properties of particulate reinforced MMCs. Leon and Mishnaevsky [131] studied the effect of geometry of reinforcement on mechanical behavior of MMCs. Shuyi et al. [132], modeled the reinforcement with different geometries by finite element method, their findings showed a direct effect of particles shape on the thermal residual stress in MMCs reinforced with ceramic particulate. Since simplified standard models in FEA were not be able accurately predict the mechanical properties and damage mechanisms [133].

In this study a real microstructure-based computational scheme and FEA model was developed which was numerically simulated under compression load condition to obtain more reliable and results. In this regard, segmented and vector images were created from microstructure images using a computer assisted image processing technique. The two dimensional CAD model

from the actual microstructure was created using Solidworks 2012 x64 edition (licensee No.25734) software. The generated 2D model was used to develop FE model and simulate the compression test using commercially available ANSYS Workbench 14.5 (ANSYS Academic Research, Fully Licensed for NDSU) software.

3. EXPERIMENTAL PROCEDURE

In order to optimize P/M process for fabrication of Fe-TiB_2 composites, several cylindrical samples (Ø: 10 cm, H: 20 cm) were fabricated at different conditions as explained in this section. The main goal of optimization is to determine a P/M route which ensures the best possible mechanical properties through a simple, cost effective, and efficient process.

3.1. Material

Commercially available Iron powder with 99.9 wt. % purity (Alfa Aesar, Ward Hill, MA, USA) with nominal size less than 10 μm and TiB_2 powder with 99.5 wt. % purity (Alfa Aesar, Ward Hill, MA, USA) with nominal particle size less than 45 μm were used to fabricate P/M samples.

3.2. Tool Making

Cylindrical dies made from D-grade steel for fabrication P/M samples. Resistant to heat, high strength to tolerate applied pressures and very polished internal surfaces of die, were the main considerations in tool making. Figure 3.1 shows some parts of tools were used in this study. In order to prevent any friction between powders and internal area of die, some high temperatures dry lubricant was sprayed onto the internal wall of die.

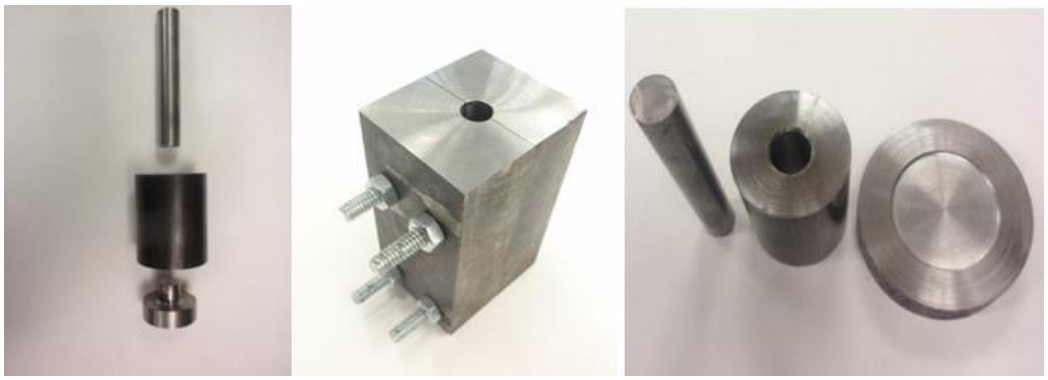


Figure 3.1: The tools and dies were designed and fabricated for this study.

3.3. P/M Technique to Fabricate Samples

3. 3.1. Blending and Mixing of Powders

TiB_2 powder at different volume levels were mixed with Iron powder. Powders were mixed and blended uniformly using a V-Blender, available at ME Department, NDSU, according to ASM V.7. It is the most recommended technique for effective uniform mixing and blending of the powders with large difference in hardness values [70]. Figure 3.2 shows a typical V-blender, this kind of blenders can be found in different sizes and rotation speeds. The optimum mixing time and speed was obtained after production of several samples at different speed and time and microstructural observation and image analysis using optical microscopy. For an easy measurement on mixing the powders, the volume percentage has been transformed into weight percentage as follow:

$$\rho = \frac{m}{V} \quad (1)$$

$$20\,Vol\%\ TiB_2 \rightarrow 4.5 = \frac{m_{TiB_2}}{\frac{20}{100}\,V_{total}} \rightarrow = 12\%\,Wt\%\ TiB_2$$

$$80\,Vol\%\ TiB_2 \rightarrow 7.8 = \frac{m_{Fe}}{\frac{80}{100}\,V_{total}} \rightarrow = 88\%\,Wt\%\ Fe$$

Figure 3.2. V-Blender was used to mix the powders.

3.3.2. Cold Pressing

Cold pressing of the powders followed the mixing stage and the powder mixture was fed into the die. A hydraulic press as shown in Figure 3.3 has been used at room temperature to apply the required steady loads on the powder mixture. Several cold pressed samples have been produced at six different pressures of 30, 35, 40, 45, 55 and 60 MPa. The effect of pressure on the hardness of cold-pressed samples was investigated to find the optimum applied pressure for cold pressing.

Figure 3.3. Cold Pressing instrument.

3.3.3. Hot Pressing

At relatively elevated temperatures metals were softened and therefore their formability increase, therefore it is possible to achieve higher density at high temperature pressing. The hot pressing process was associated with high pressures at relatively high temperatures. There were three main process variables in hot pressing stage; pressure, temperature, and time. In order to study these variables, several samples at different pressures (30, 45, 55 and 60 MPa) and temperatures from 400 to 700 ℃ at different times from 30 to 120 minutes have been fabricated to optimize the hot pressing parameters using the hot pressing machine shown in Figure 3.4. The parameters which haven examined in this study are presented in the Table 3.1.

Figure 3.4. Hot pressing machine.

Table 3.1. Process parameters of cold and hot pressing used in this study.

Sample No.	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
Cold pressure (MPa)	45								45		55			60		
Hot pressure (MPa)	30				45				45		55			60		
Temp (℃)	400		500		400		500		700	700	500					
Time (min)	30	60	30	60	30	60	30	60	60	120	30	60	120	30	60	120

Four thermostats were installed in different positions of die in touch with powders to monitor temperature distribution in the cold pressed samples as shown in Figure 3.5. As it can be seen in Figure 3.6 Figure 18 Thermocouples recorded the temperature of powders during hot pressing the die over the time.

Figure 3.5. Different thermometers were installed to control the temperature of compacted powders.

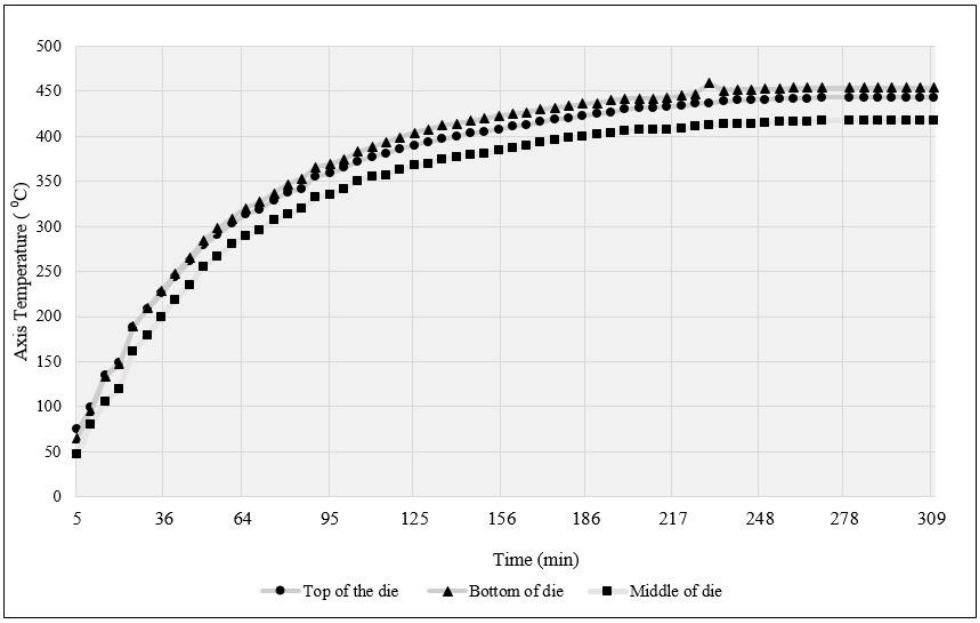


Figure 3.6. Temperature in different points of mold was monitored and controlled.

3.3.4. Sintering

Hot-pressed samples were sintered in Argon-controlled furnace at three temperatures of 900, 1000 and 1100℃ at 10 different dwell times ranged from 10 to 120 minutes to optimize the parameters for sintering samples in this study. Figure 3.7 shows the furnace was in this study.

Sintering parameters; time and temperature of sintering which were studied in this study are listed in Table 3.2.

Figure 3.7. Atmosphere-controlled sintering furnace.

Table 3.2. Process parameters of sintering process.

Sample No	1	2	3	4	5	6	7	8	9	10	11	12
Temp (℃)	900			1000			1100					
Time (min)	10	20	30	40	50	60	70	80	90	100	110	120

3.4. Optimization

The process parameters of each P/M route were designed based on desired properties of the final products and some fabrication considerations such as cost effectiveness. Table 3.3 shows

that the number of possible P/M processes if only 5 different values for each factor were considered. This big number of P/M routes on one hand and lack of specific technical information for fabrication of each composite on the other hand, makes the optimization as an absolutely necessary step in using P/M technique. In order to optimize the parameters of P/M process for fabrication of Fe-TiB_2 composite, 28 possible P/M routes were experimentally studied. In fact, at each section of P/M process (cold pressing, hot pressing and sintering) the effect of process parameters on the mechanical properties of the product were studied. As P/M processes optimized, pure Fe samples and MMC samples with 10, 20 and 30 vol% of TiB_2 were fabricated to study the effect of reinforcement volume fraction on mechanical properties of fabricated samples.

Table 3.3. Numerous P/M routes are possible.

<table>
<tr><th>P/M Route</th><th colspan="2">Parameters</th><th>Minimum factors for P/M route</th><th colspan="2">example</th></tr>
<tr><td>Cold Pressing</td><td>Pressure</td><td>1</td><td rowspan="7">6</td><td>5</td><td rowspan="7">$\frac{30!}{6! \times (30-6)!} = 593775$</td></tr>
<tr><td rowspan="3">Hot Pressing</td><td>Pressure</td><td rowspan="3">3</td><td>5</td></tr>
<tr><td>Time</td><td>5</td></tr>
<tr><td>Temperature</td><td>5</td></tr>
<tr><td rowspan="2">Sintering</td><td>Time</td><td rowspan="2">2</td><td>5</td></tr>
<tr><td>Temperature</td><td>5</td></tr>
</table>

3.5. Microstructural Study

All MMC samples fabricated by optimized P/M process were cross-sectioned, mounted using epoxy (Thermoset acrylic) as shown in Figure 3.8; all samples were grinded and polished prior to microstructural observation. The microstructures of samples were observed by OLYMPUS FV laser confocal microscope and ZEISS MAT optical microscope along with iVIEW software.

Porosity is one of the most important microstructural features of the materials which can affect physical and mechanical properties of the materials. Image analysis was performed to determine (a) presence, location, size and distribution of porosities, (b) morphology and distribution of reinforcing particles, (c) bonding and interface of matrix and reinforcement.

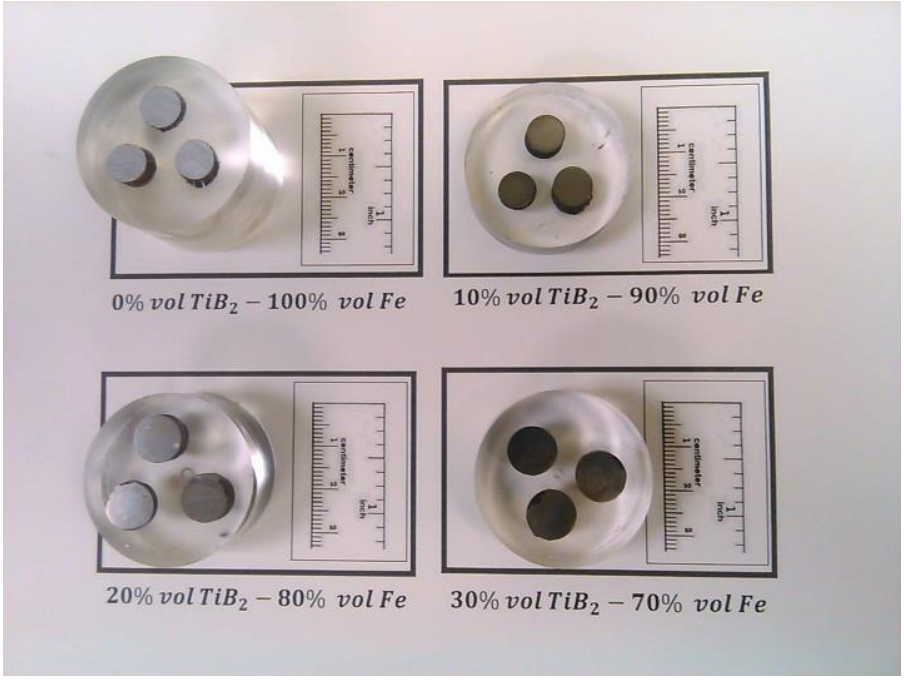


Figure 3.8. Mounted and polished samples for microstructural study.

3.6. Density Measurement

The density measurements have been performed after each processing step to understand the effect of the certain parameters of P/M process on the densities of the samples during the process optimization. As shown in Figure 3.9, the densities of the specimens were measured by water immersion method at room temperature according to ASTM B962-15 using the Eq (2).

$$\boldsymbol{\rho}_{solid} = \frac{\boldsymbol{m}_{solid\ in\ air}}{\boldsymbol{m}_{solid\ in\ air} - \boldsymbol{m}_{solid\ in\ water}} \times \boldsymbol{\rho}_{water} \quad (2)$$

Figure 3.9. Density measurement by water immersion method.

3.7. Micro-Hardness Test

Hardness is one of the main indications of mechanical properties of materials. The value of hardness represents the resistance of microstructure to deformation, densification, and cracking. The Vickers micro hardness test was used for measurement of hardness in different P/M processed samples. The tests have been done by using CLARCK CM-800AT machine. The applied load was 200 gf and the dwell time was 15s. Hardness test in porous materials associated with a big variations in test results, to minimize uncertainties arising from this variation, at least 21 tests at random locations were done on each sample to minimize the effect of the inhomogeneity of the microstructure and to make sure that all phases and features were covered by indenter. The results were reported as the average value along with Standard Deviations (STDV).Figure 3.10 and 3.11

respectively show the schematic view of an indentation during micro harness test and an actual Vickers indentation effect which was observed in this study.

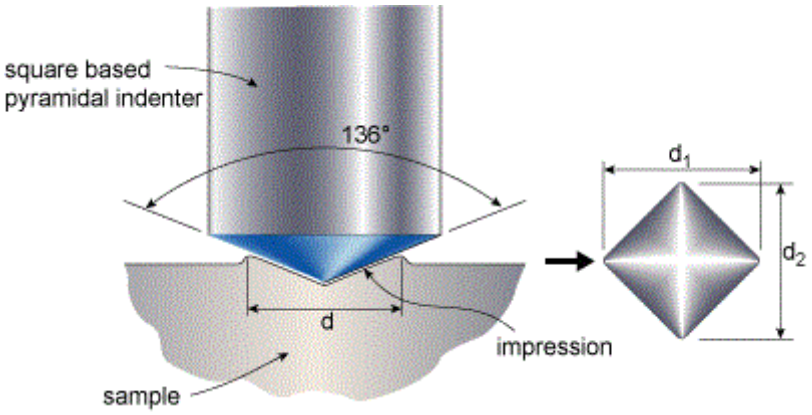


Figure 3.10. Schematic view of indenter and indentation effect during micro harness test [134].

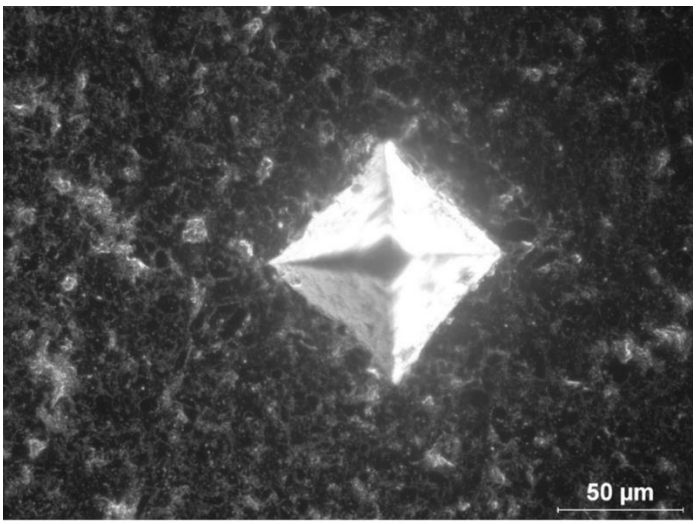


Figure 3.11. Actual indentation effect observed in this study.

3.8. Compression Test

Compression tests were also conducted on each sample to determine the mechanical properties such as Young's modulus, yield strength, and ultimate tensile strength. According to

ASTM E9-09 the cylindrical test samples with aspect ratio of 2:1 (Ø: 10 cm, H: 20 cm) were fabricated and tested as schematically shown in Figure 3.12. For MMCs with 0, 10, 20 and 30 vol% of reinforcement, at least five specimens were fabricated and tested for each class of samples. Since the compressing plates of MTS machine were too big for fabricated samples, specific grips as shown in Figure 3.13, were designed and fabricated to make sure that compressive test runs according the standard. Specimens were subjected to uniaxial compression load of 0.1 mm/min by using MTS Criterion C45 universal testing machine as presented in Figure 3.14.

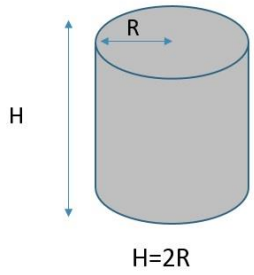


Figure 3.12. Schematic view of fabricated samples for compressive test.

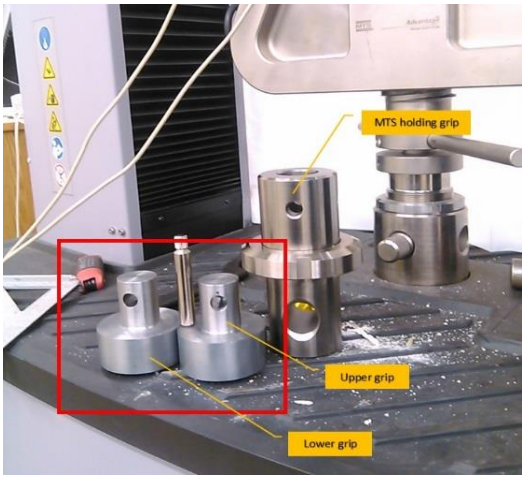


Figure 3.13. Compression test's grips were designed and fabricated.

Figure 3.14. Compression Test was done by MTS machine.

4. RESULTS AND DISCUSSION

4.1. Optimization of P/M Process parameters

Due to the porous structure of fabricated samples, the hardness test results had some variances which can be seen in Figure 4.1. In this regard to get more reliable results, at least 21 microhardness test were done on each sample. Table 4.1 shows the effect of cold pressing on the hardness and density of the fabricated samples.

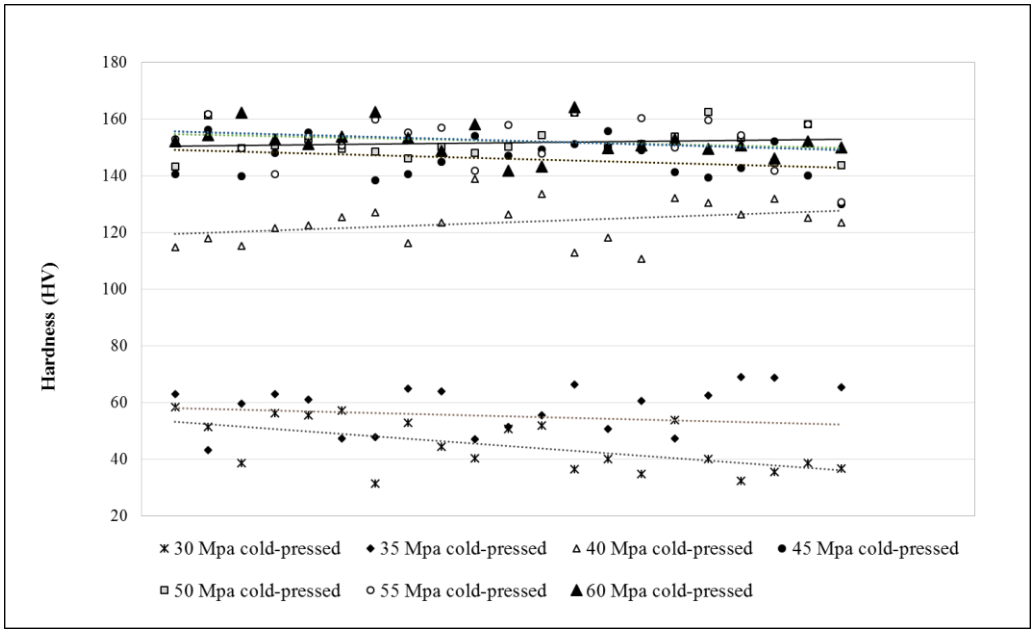


Figure 4.1. The effect of pressure on the hardness of cold-pressed samples.

Table 4.1.The effects of cold pressing on hardness and density of cold-pressed Fe-TiB_2 samples.

The applied pressure on cold-sample (MPa)	30	35	40	45	50	55	60
Micro-Hardness (HV)	44.74 ± 9.11	58.05 ± 8.32	123.63 ± 7.46	146.19 ± 7.08	151.87 ± 5.78	152.34 ± 10.5	152.5 ± 14.33
Density ($\frac{gr}{cm^3}$)	3.233	3.242	3.798	3.954	3.972	3.974	3.97

As it can be seen in figure 4.2 (a) and (b), the trend of average hardness of samples were aligned with the trend of the average density values, which can be attributed to the fact that

applying more pressure caused better intimate contacting areas between powders particles. On the other hand, more pressure caused the particles to be deformed, crushed and fill the existing pores in the microstructure. As the amount of applied load on mixed powders increase, particles can touch each other more effectively and consequently the possibility of more bond formation increase in the entire structure. It was also observed that by applying more pressure the hardness and density of samples did not increase more than a certain value. In fact, applying loads higher than 45 MPa, could not increase the hardness and density because powders were compacted enough to be capable of carrying the loads without showing any deformation or crushing. On the other hand, friction between the powders along with friction between the walls and powders are acting against the applied load. The amount of these friction forces increase by increasing the applied load which means that these existing friction forces can counterbalance the applied load.

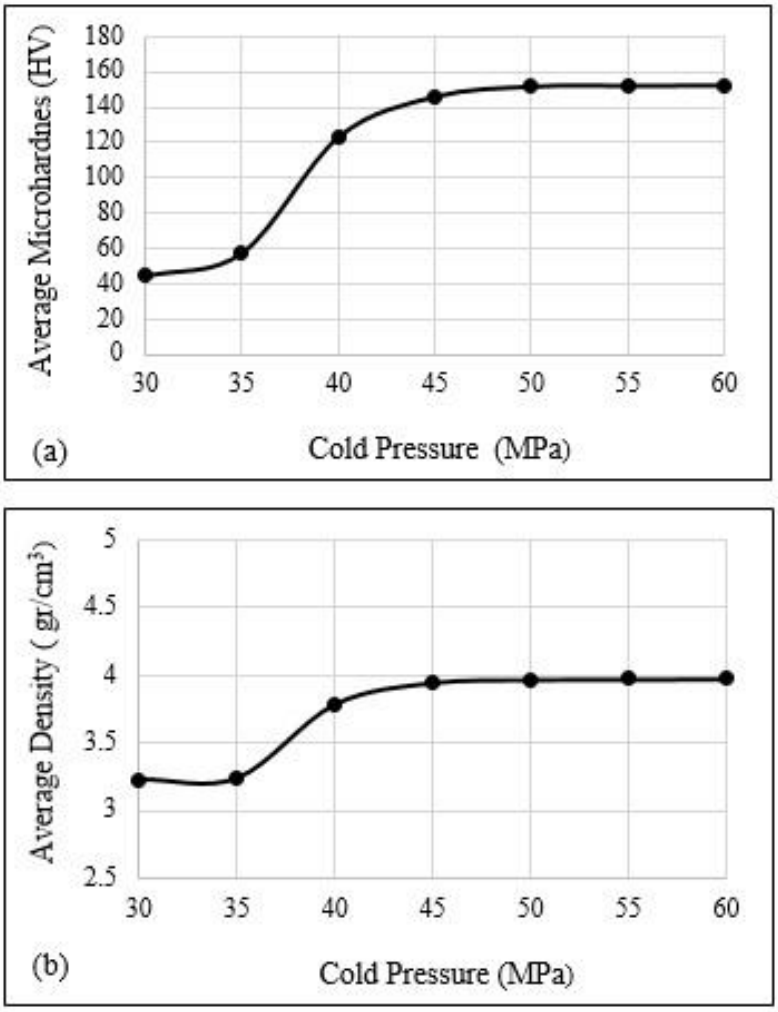


Figure 4.2. The effects of cold pressure on (a) hardness and (b) density of cold pressed samples.

Applying heat facilitated the mobility of powers by decreasing the internal friction between the powders and friction between powders and walls. Figure 4.3 shows the effect of hot pressing on hardness of the samples.

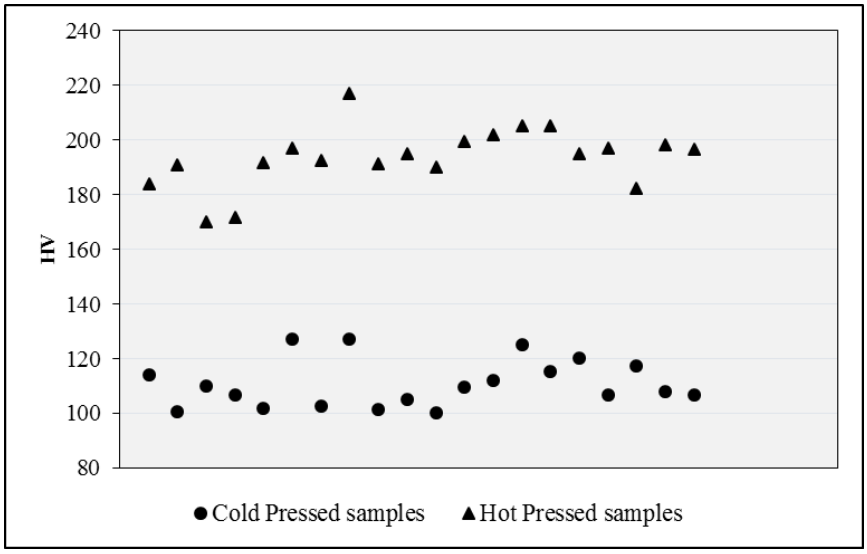


Figure 4.3. The effects of heat at same load application.

Figure 4.4 showed the individual and overall effects of hot pressing's process factors on density and hardness of hot-pressed samples.

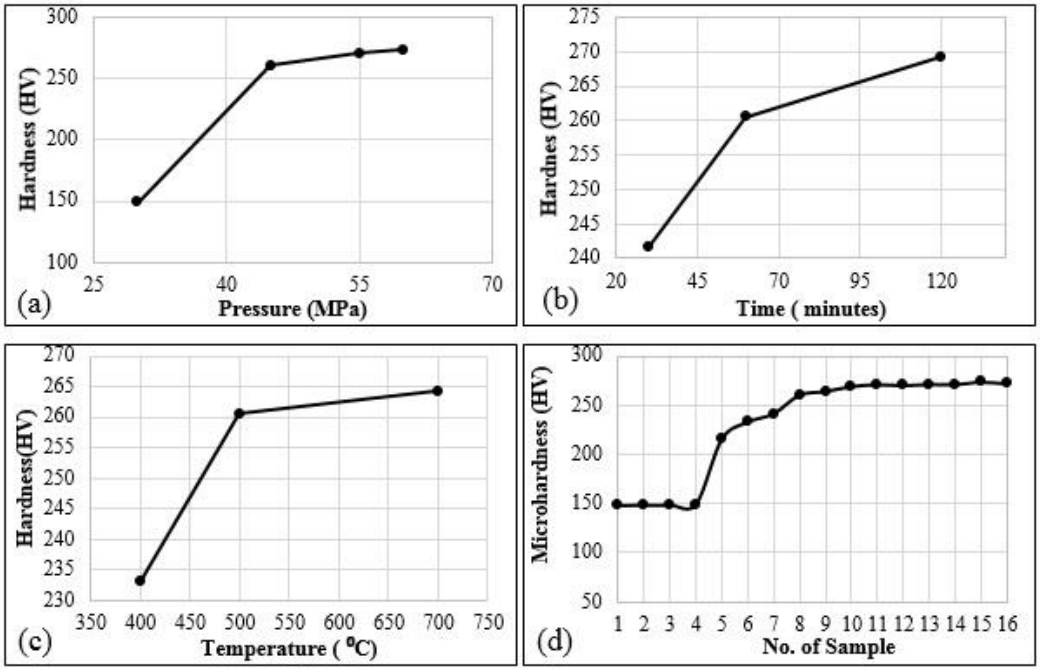


Figure 4.4. The effect of hot pressing parameters on hardness. (a): the individual effect of pressure, (b): time, (c): temperature and (d) :the overall effect of all parameters on hardness of hot pressed samples.

As it is shown in Figure 4.4 (a), increasing pressure from 30 to 45 MPa resulted in a big increase in the hardness value from 150 HV to 260 HV, however, application of pressures higher than 45 MPa showed no significant increase in hardness. Similar trends were observed for the effects of time and temperature on hardness of samples as shown in Figures 4.4 (b) and (c). Based on these curves, it was concluded that optimum values for each of parameters were pressure of 45 MPa, temperature of 500 ˚C and time of 120 minutes. Sample No. 8 which was fabricated using optimum process parameters exhibited the best mechanical properties. It is worth mention that the cost-effectiveness of the process was also taken to the account during optimization of the fabrication process parameters. Therefore, although increasing pressure above 45 MPa and temperatures above 500 ˚C should slight improvement in mechanical properties as seen in sample No.9, it was not considered as optimum parameters due to considerably higher energy consumption, mold deterioration and more frequent need to instrument maintenance. The optimization experimental processes were summarized in Table 4.2, in which the optimized cold and hot pressing process parameters and their results can be found.

Table 4.2. Process factors and results of cold and hot pressing.

No.	Cold pressure (MPa)	Hot pressure (MPa)	Temp (℃)	Time (min)	Density ($\frac{gr}{cm^3}$)	Average hardness (HV)
1	45	30	400	30	4.302	148.01 ± 14.95
2				60	4.309	148.27 ± 13.52
3			500	30	4.334	148.38 ± 16.57
4				60	4.349	149.27 ± 17.96
5		45	400	30	6.279	215.82 ± 27.10
6				60	6.352	233.13 ± 24.46
7			500	30	6.357	241.59 ± 26.77
8				60	6.55	260.56 ± 27.02
9	45	45	700	60	6.62	264.19± 34.41
10			700	120	6.635	269.26 ± 33.38
11	55	55	500	30	6.641	271.23± 35.18
12				60	6.640	270.36± 38.52
13				120	6.642	271.44± 22.64
14	60	60		30	6.641	271.19± 32.82
15				60	6.641	273.71± 41.11
16				120	6.640	272.12± 26.53

Since, porosity is an intrinsic part of all P/M samples, large scatter of the results and consequently large standard deviation were observed (4.2). The measured hardness values could vary depending on the positions of the indenter. For instance, as it can be seen in figure 4.5, if indenter touched a TiB_2 particle (white areas), which was the case in this figure, the reported value of hardness were higher than when indenter touched Fe particles (grey areas) or porosities (dark areas).

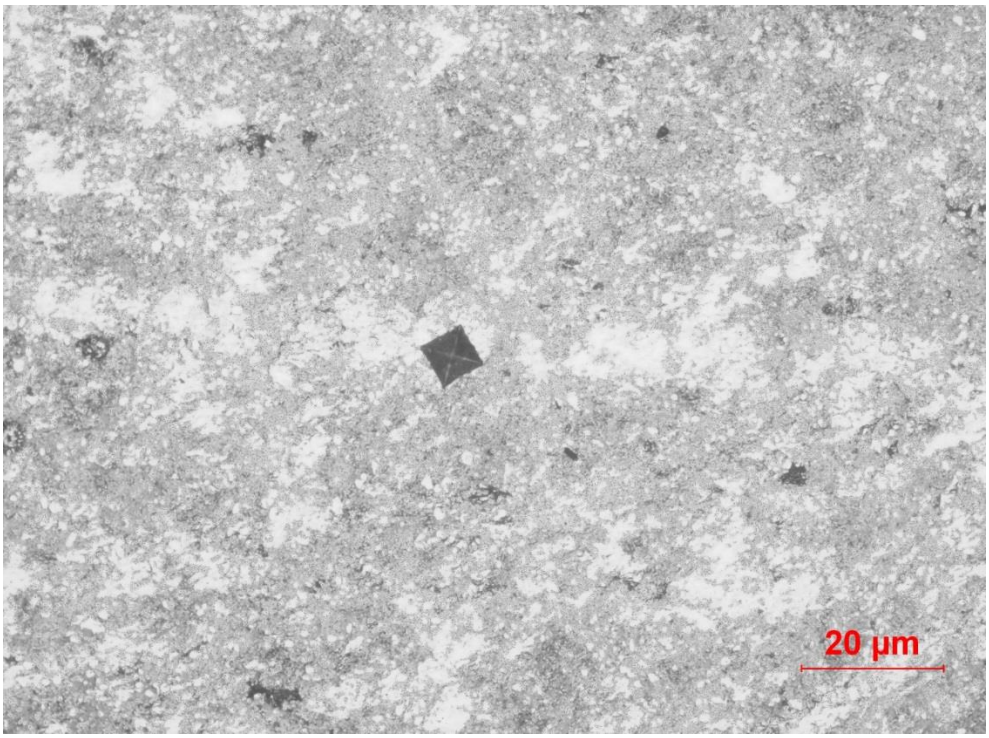


Figure 4.5. Indentation effect on the surface of a sintered sample.

Table 4.3 listed the effects of sintering parameters on hardness and density of samples. Two main variables in sintering process were temperature and duration of sintering process. As it can be seen in Figure 4.6 (a), sintering temperature should be higher than 1000 ℃ in order to get improved mechanical properties. In fact, it was observed that sintering at 900 and 1000 ℃ had almost negligible or minor effects on hardness values, while sintering at 1100 ℃ resulted in noticeable increase in hardness. It was also seen in Figure 4.6 (b), increasing the time of sintering

caused the increasing of hardness to some specific extent and after that, increasing the time showed adverse effect on hardness. It is clear that increasing time and temperature could help for better diffusion and atomic bonding and higher hardness but passing a certain level could have reverse effect and decrease the hardness values. One possible explanation could be grain growth due to higher diffusion rate at higher temperature and longer time. Based on the curves presented in Figure 4.6 (a) and (b), temperature of 1100 ℃ and time of 30 minutes were suggested as optimum values for sintering process. Figure 4.7 showed the combination effects of sintering parameters in one curve. Considering two factors of improved mechanical properties and cost effectiveness of the process, it was concluded that sample No.9 which was sintered at 1100 ℃ for 60 minutes met the optimization requirements. The findings in this research were in good alignment with previously reported values for sintering parameters during fabrication Iron-based composites by P/M technique [23, 24].

Table 4.3. Effects sintering parameters on hardness and density of samples.

No.	Temp (℃)	Time (min)	Average hardness (HV)		Density ($\frac{gr}{cm^3}$)	
			Before sintering	After sintering	Before sintering	After sintering
1	900	10	260.5±26	261±22	6.54	6.564
2		20		261±27		6.581
3		30		262±21		6.607
4	1000	10		263±33		6.611
5		20		264±27		6.615
6		30		266±25		6.638
7	1100	10		278±24		6.712
8		20		297±28		6.866
9		30		309±23		7.013
10		60		282±28		6.62
11		90		264±27		6.31
12		120		260±25		6.27

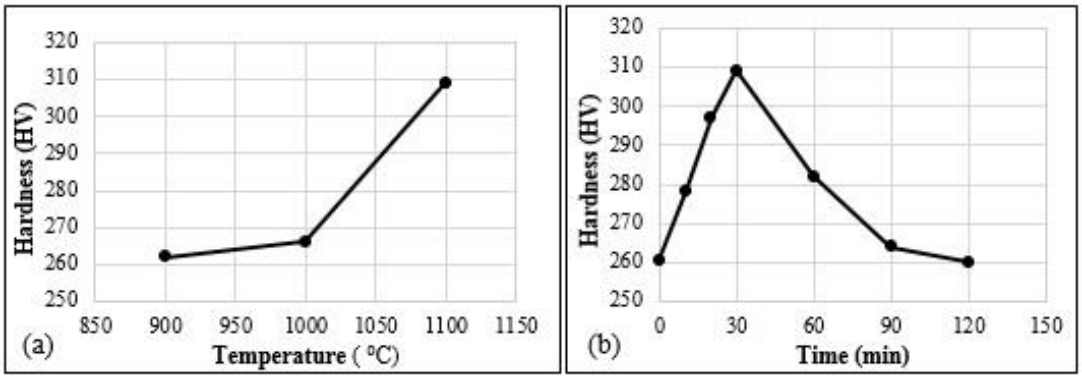


Figure 4.6. The effect of each individual parameters of sintering process on hardness of samples. The effect of (a): temperature and (b) time on hardness of sintered samples.

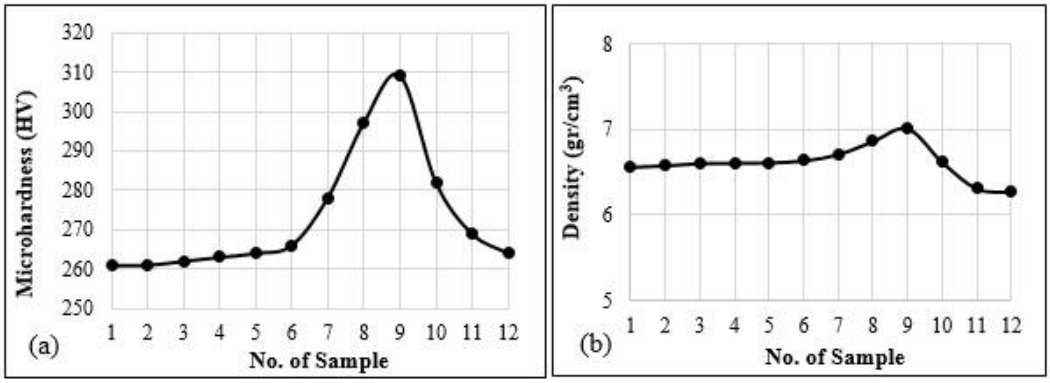


Figure 4.7. The effect of sintering process parameters on: (a) microhardness (b) density.

Figure 4.8 showed the optical microscopy images of a sample with 20 vol% TiB_2 after each step of P/M process. The porosity percentage of %55 in cold pressed sample, was reduced to %23 and 9% in hot pressed and sintered samples, respectively. Cold pressing increases touching points between particles, compacts the particles and eliminates the inter-particular spaces. As a result the density of mixed powder increases but still a significant amount of porosity remains in the microstructure. The outcome of cold pressing is creation of some interlocks between the particles which give the sample a structural integrity so it may be processed further. In hot pressing the touching areas between particles and joint points develop, this makes more voids and porosities eliminated from the microstructure (figure 4.8.b). Softening of iron powders at hot pressing helped

the porosity reduction. In sintering process due to high temperatures, diffusion activated bonding can be formed at interfaces which helps to increasing the density and mechanical properties. As it can be seen in figure 4.8.c, although porosity was not completely eliminated from the entire microstructure, sintering could significantly change the morphology from open and interconnected porosities into the closed and isolated ones, which had a huge role in improvement of the mechanical properties of materials. During P/M process iron particles diffused and fused to each other and could cover around TiB_2 particles. In fact, sintering at 1100 ℃ could create a uniform FE based structure.

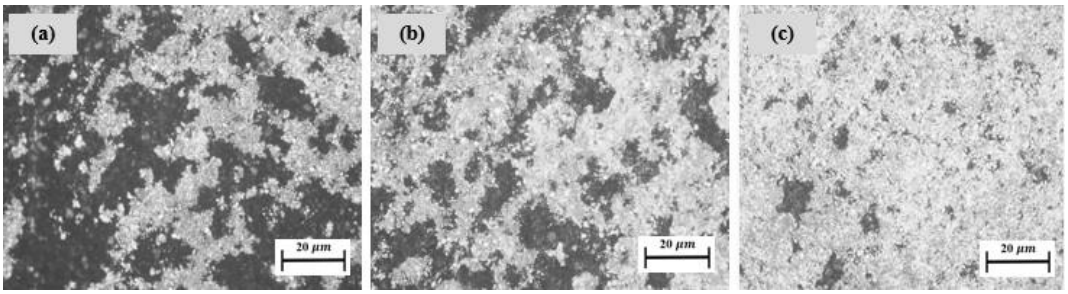


Figure 4.8. The process parameters on porosity content of sample No.8 at 50X. (a) After cold pressing (b) after hot pressing and (c) after sintering.

4.2. Effect of Reinforcement Volume

4.2.1. Density

Theoretical and bulk density of fabricated P/M samples with different volume fractions of TiB_2 produced using optimized P/M process are presented in figure 4.9.

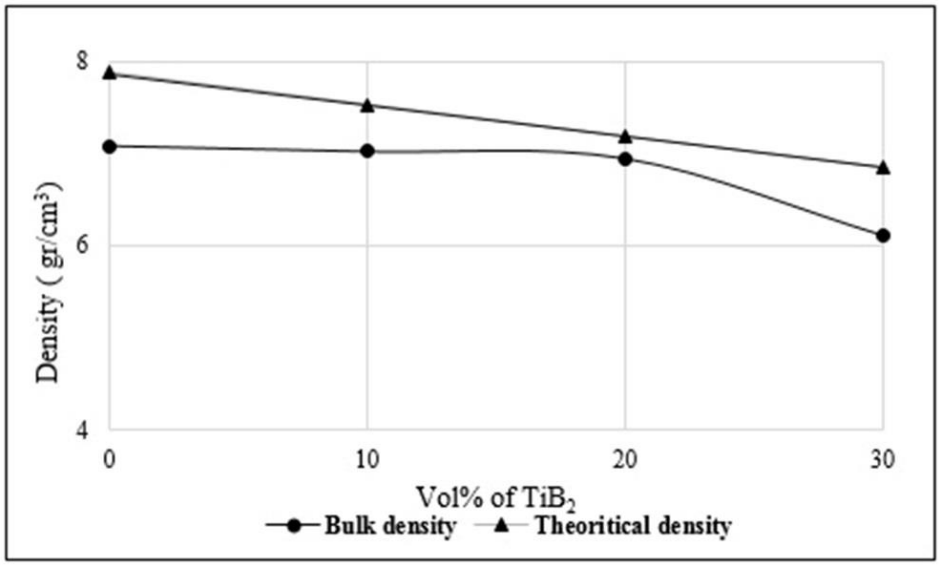


Figure 4.9. The effect of reinforcement vol% on theoretical and bulk densities.

According to the rule of mixture by increasing the vol% of TiB_2 which has a lower density than Fe, the density of composites would decrease. Relatively, similar trend was seen in bulk density which was measured by water immersion method. However, it was noted that reduction rate of bulk density did not exactly follow the same trend as theoretical density. The difference was attributed to the fact that formation of porosity in the microstructure of samples is not taken into the account in rule of mixture equation. As it can be seen in Figure 4.9, the bulk density of samples decreased with very slow rate up to 20 vol% of TiB_2, addition of more TiB_2 to the matrix, increased the reduction rate of density. Another finding showed that difference between bulk and theoretical density was minimum at 20 vol% of TiB_2. In other words, 20 % is the maximum volume percentage of TiB_2 which Fe matrix is capable to wet effectively. In some literatures [135,136] authors suggested that lattice mismatch between matrix and reinforcement and therefore subsequent agglomeration of ceramic particles can be one of the reasons of porosity formation. By considering this point, more volume percentage of TiB_2, could introduce more mismatch and more porosity into the composite which translates to the loss of density. In fact, lack of wetting and

consequently insufficient bindings between matrix and reinforcement particles at higher contents of TiB_2 leaves more particles unbounded and agglomerated which can increase the amount of the porosity and consequently, decrease the density of samples at 30 vol% of TiB_2.

4.2.2. Hardness

The findings as shown in figure 4.10, indicated that increasing the content of TiB_2 up to 20% increased the hardness of composite. The reason of hardness increasing, backs to this simple fact that adding UHTC particles with very high values of hardness to Fe matrix helped to improve the hardness of composite. Also it was observed that addition of more TiB_2, could not significantly change the hardness of composite and even resulted in slight reduction of hardness around 30% reinforcement. It was concluded TiB_2 can increase the hardness of composite when it was uniformly distributed in the microstructure. It seems that when the amount of reinforcement excesses a certain level, the possibility of unwanted brittle ceramic-ceramic interfaces and agglomeration of ceramic particles increases, that is why we loss of hardness at 30%.

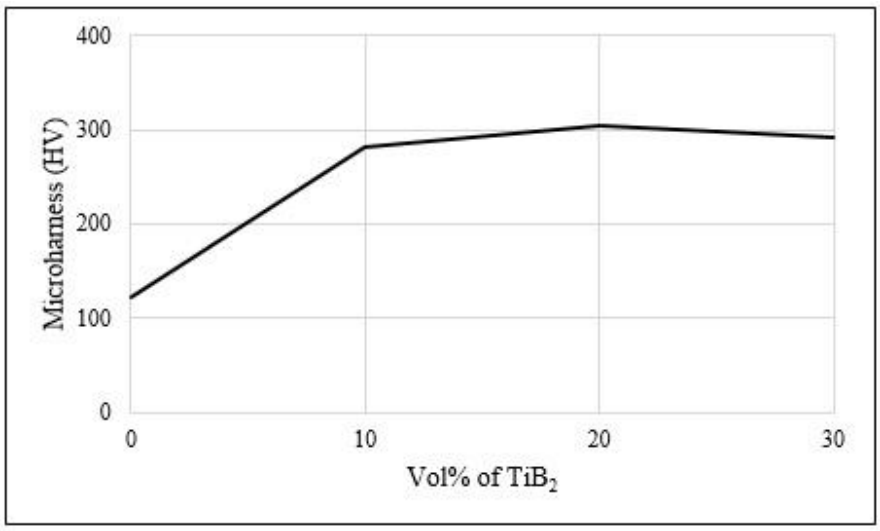


Figure 4.10. The effect of reinforcement vol% on average hardness of Fe-TiB_2 composites.

4.2.3. Compression test

As it can be seen in the Figures 4.11 and 4.12, the sample without reinforcement showed completely ductile deformation during compression test and failed at low compressive stresses while adding TiB_2 increased compressive strength and Young's modulus. Adding 10 and 20 vol% of TiB_2 increased the Young's modulus by 56% and 100%, respectively. Addition of 30 vol% TiB_2 have not significantly improve mechanical properties compared to un-reinforced pure Fe samples.

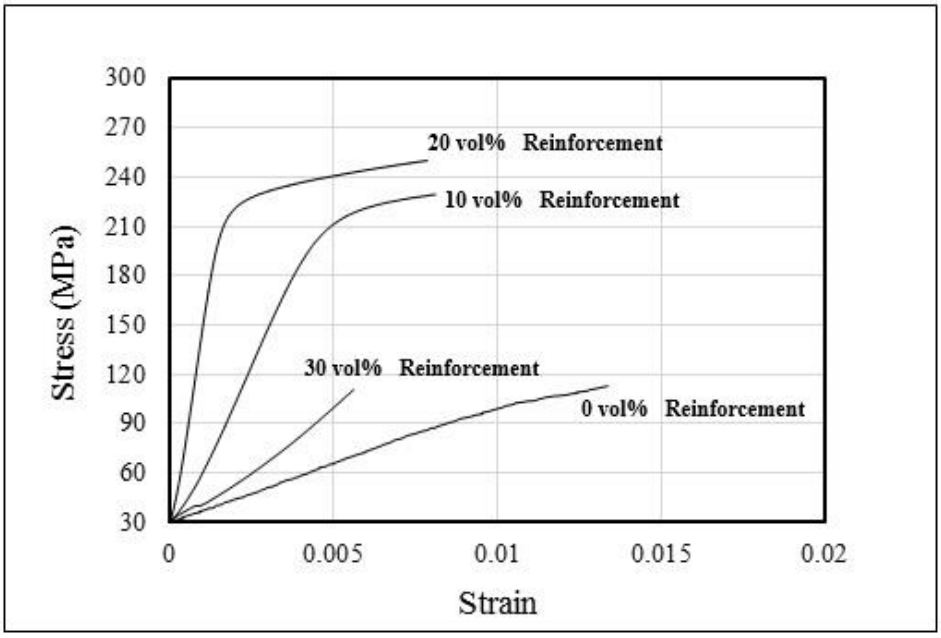


Figure 4.11. The stress-Strain curves of Fe-TiB_2 composites at different reinforcement vol% based on compression test.

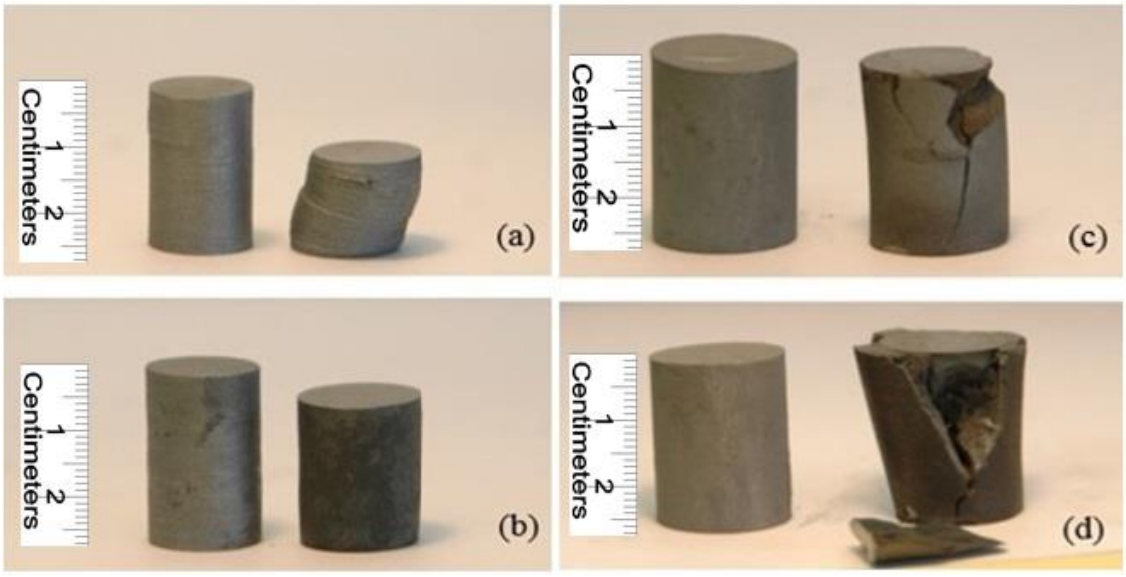


Figure 4.12. Fracture mode of samples under compression load changed from fully ductile.

It can be inferred to this point that TiB_2 particles inherently have very high hardness and Young's modulus, therefore, adding these particles up to a certain level, in this study 20 vol.%, could favorably increase the compressive strength of composites if they were uniformly and sufficiently been added to the matrix [137]. Addition of more than optimum volume percentage of TiB_2 to the matrix, could not be resulted in improvement in the mechanical properties of composite. As mentioned before in case of excessive amount of reinforcement as it can be seen for Fe-30 vol.% TiB_2 as it can be seen in Figure 4.13, The loss of mechanical properties were attributed to the increase in agglomeration of the particles as shown in more details in Figure 4.14. In this condition the possibility of formation of TiB_2-rich spots increases. Since 1100˚ C is significantly lower than melting point of UHTCs, the chance of formation of ceramic-ceramic bonding was negligible. Therefore, these excessive ceramic-ceramic points with no bonding, under the applied stress broke easily and developed sharp cracks inside the microstructure [138] and eventually failed in a brittle mode (Figures 4.11 and 4.12). It should be noted that TiB_2 has showed two opposite characteristics in strengthening of MMC in this study. On one hand TiB_2 increased

the strength of fabricated composites through its very high hardness and stiffness but at the same time it decreased the density of composites because of its low density. Moreover, as it discussed earlier more amount of reinforcement in the matrix resulted in more possibility of agglomeration.

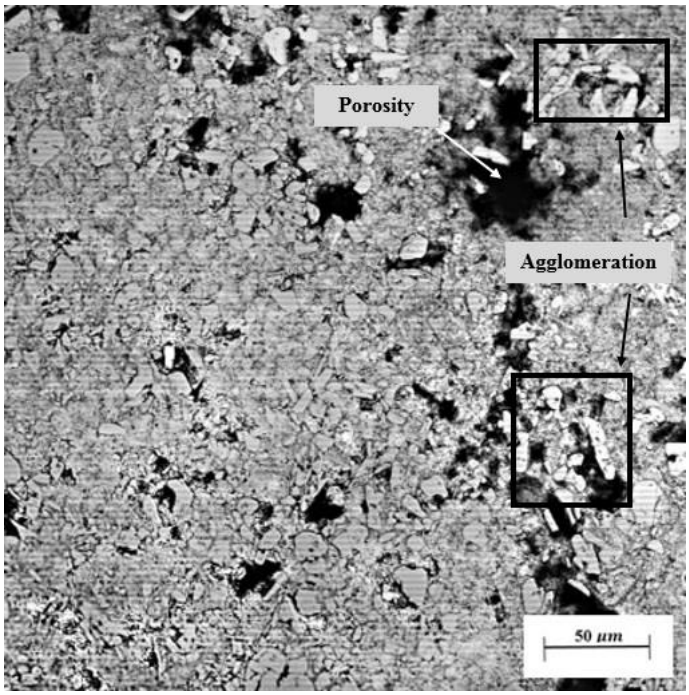


Figure 4.13. Confocal micrograph showing porosity and agglomeration of reinforcement particles observed in Fe-30 vol% TiB_2.

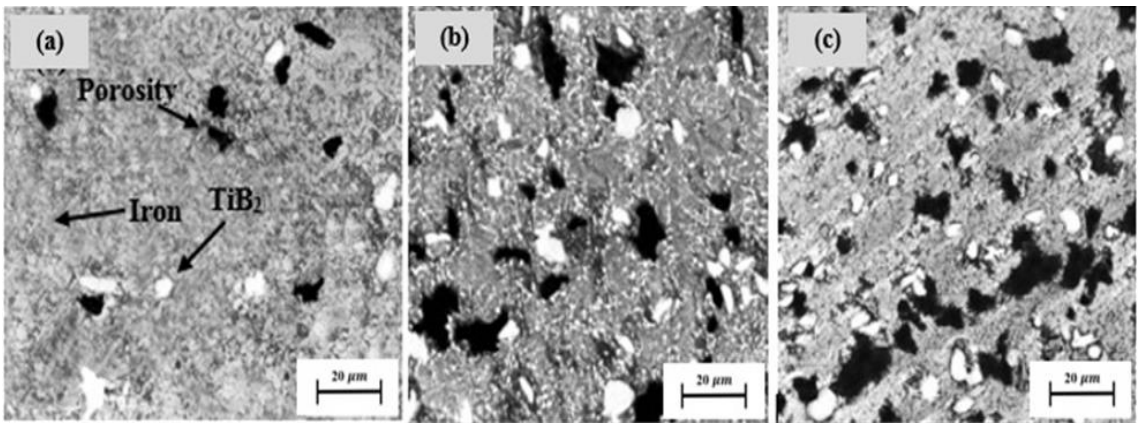
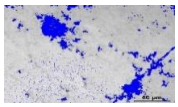
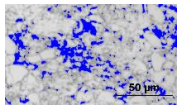
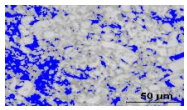
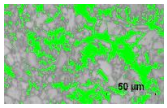


Figure 4.14. Microstructure of Fe-TiB_2 composites at different vol% of reinforcement.
(a) 10 vol%, (b) 20 Vol% and (c) 30 vol%.

Depending the amount of TiB_2, one of these mechanisms could govern the mechanical properties of Fe-TiB_2 composite. Moreover, by considering the difference between thermal expansion coefficients of TiB_2 and Fe, it was concluded that during cooling after sintering process, there would be possibility of creation of residual stress due to different rates of shrinkage. It could form stress fields around each TiB_2 particles. These stress fields of adjacent particles overlapped which made dislocation movement difficult and consequently resulted in work hardening [139]. Up to a certain level, work hardening could result in strengthening of samples but excessive work hardening due increasing the amount of TiB_2 introduced more internal energy into the structure and made it unstable [140]. The overall effects of TiB_2 content on mechanical and physical properties of Fe-TiB_2 composites were summarized in table 4.4.

Table 4.4. The effect of TiB_2 vol% on mechanical and physical properties of Fe-TiB_2 composite.

Metal-Matrix Composite	Hardness (HV)	Density ($\frac{gr}{cm^3}$)		Young's Modulus (GPa)	Porosity (%) Measured by *isolution* software Magnification :100X Indicator bar : 50μ	
		Actual (experiment)	Theoretical (rule of mixture)			
0% vol TiB_2 100% vol Fe	286 ±15	7.079	7.87	64	7.5	
10% vol TiB_2 90% vol Fe	286 ±15	7.018	7.53	100	8.4	
20% vol TiB_2 80% vol Fe	304 ±21	6.941	7.19	130	9.2	
30% vol TiB_2 70% vol Fe	291 ±34	6.101	6.859	71	14	

4.2.4. Finite Element Analysis (FEA)

In order to perform FEA, the matrix and reinforcement phases were meshed separately using three nodes plane stress triangular element. TiB_2 reinforcement was modeled as linear elastic materials and Fe matrix as a bilinear elastic hardening material to describe its elasto-plastic behavior. A uniaxial compressive displacement similar to our compression test as previously explained in sections 3.7 and 4.2.3 was simulated on developed models at the rate of 0.1 mm/min.

The finite element model was developed based on actual micrographs which were taken randomly from microstructure of the samples with different reinforcement phases, as shown in Figure 4.15.

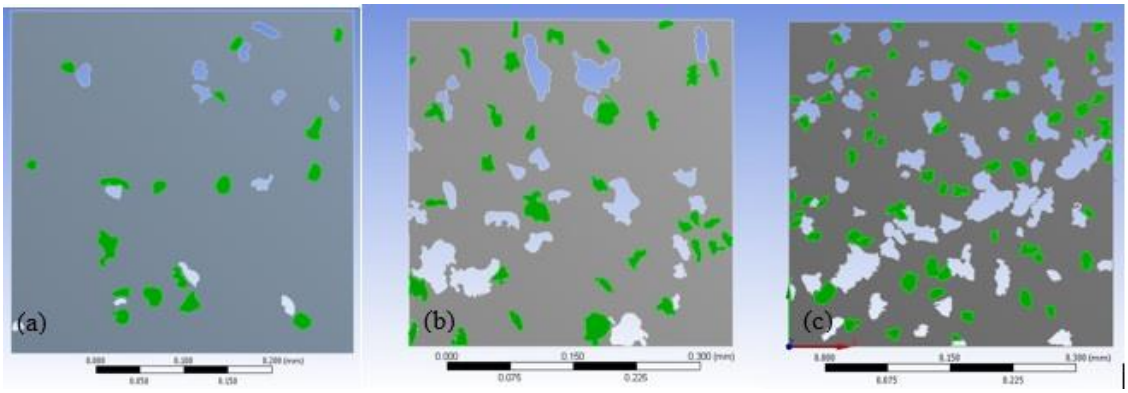


Figure 4.15. Segmented and vector images developed based microstructural images: (a): 10 vol% TiB_2 (b): 20 vol% TiB_2 (c): 30 vol% TiB_2.

TiB_2 particles were assumed to behave linearly elastic with Young's modulus of 500 GPa and Poisson's ratio of 0.17. Bilinear isotropic hardening stress-strain data was used as an approximation to model the plasticity in Fe matrix. Young's modulus of Fe matrix assumed to be 200 GPa with Poisson's ratio of 0.3, Yield strength of 250 MPa, and Tangent Modulus of 1.45GPa. Several steps in FEA study from processing images and conversion to CAD model and consequently creation of FE scheme ready for solution were shown in Figure 4.16.

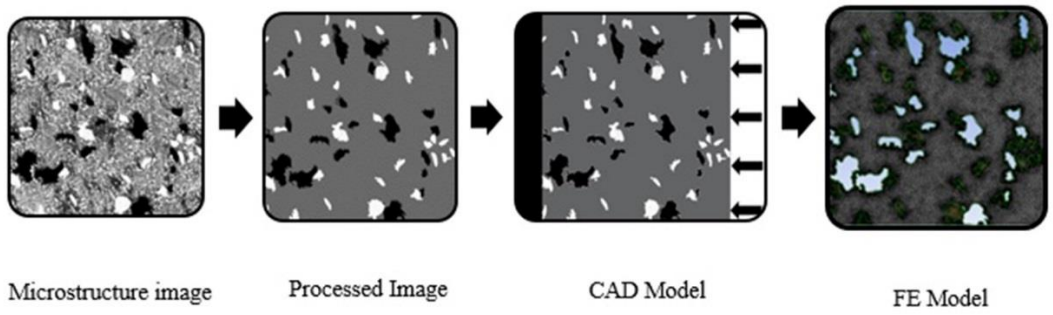


Figure 4.16. The steps of FEA in this study.

The stress distributions after the compression displacement were presented in Figure 4.17. During loading, Fe matrix which was a continuous phase, transferred the compressive stress flow to TiB_2 particles which were inherently designed to carry a big portion of applied load due to their higher Young's modulus.

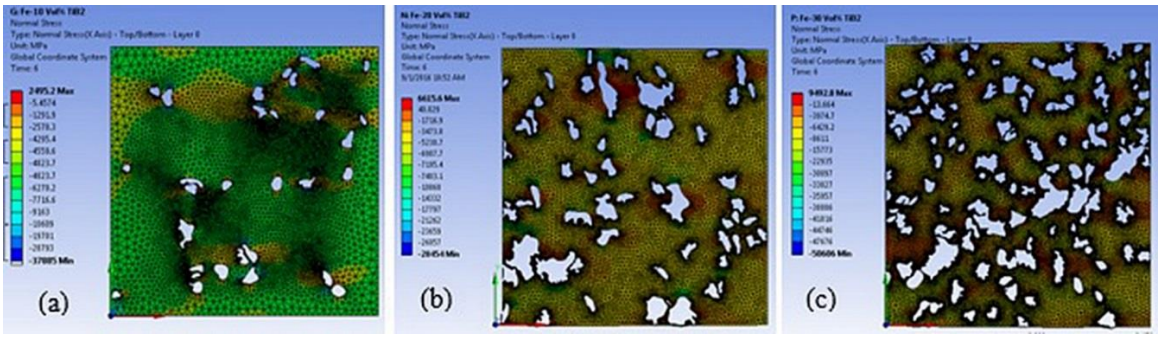


Figure 4.17. Developed FEA model on Fe-TiB_2 composites: (a): 10 vol% TiB_2 (b): 20 vol% TiB_2 (c): 30 vol% TiB_2.

As stress distribution maps showed in Figure 4.18, excessive amount of TiB_2, could significantly increase the local tensile stress concentration cells within microstructure. These stress concentration sites were considered as the potential sources for local crack initiation and propagation which could result in failure and brittle fracture in composites. In numerical results, this phenomena observed as changing the sign of developed stress from negative (compressive) to positive (tensile). These points with tensile stress (red color zones) were mostly located at interface of Fe and TiB_2 and around the porosities. Existing tensile stresses at the interface were the major reason for bonding separation between Fe matrix and UHTC reinforcement. One explanation for creation of such localized tensile stress within the microstructure could be large difference between the Young's modulus of ductile iron and stiff TiB_2 materials.

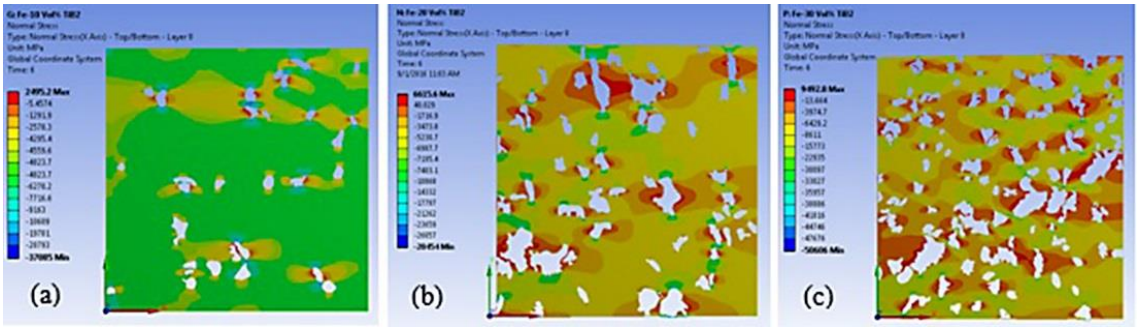


Figure 4.18. Normal stress distribution in Fe-TiB_2 composites with (a): 10 vol% TiB_2 (b): 20 vol% TiB2 (c): 30 vol% TiB_2.

The stress-strain curves of each composite were recorded and presented in Figure 45. The elastic modulus of 10, 20, and 30 vol% of TiB_2 composite were calculated to be 260.3, 280.1, and 91.6 GPa, respectively. Young's modulus were also calculated through rule of mixtures as 240, 270 and 300 GPa respectively for MMCs reinforced by 10, 20 and 30 vol% of TiB_2. Comparison between these two ways of calculations showed that for MMCs with 10 and 20 vol% of TiB_2 both techniques predicted the same trend for Young's modules, but in case of MMCs with 30 vol% of TiB_2 , rule of mixture showed increasing in Young's modulus while numerical analysis showed decreasing of Young modulus. The reason for such a difference could be referred to this fact that in rule of mixture calculation, the amount of porosities in the microstructure and their effects on mechanical properties were not taken into account. Yield strength of 10, 20, and 30 vol% of TiB_2 composite were also recorded to be 262, 269, and 181 MPa, respectively. The results have also shown that 30 vol% TiB_2 composite behaved as a brittle material which was inn very good agreement with experimental results from compression test as shown in Figure 4.19.

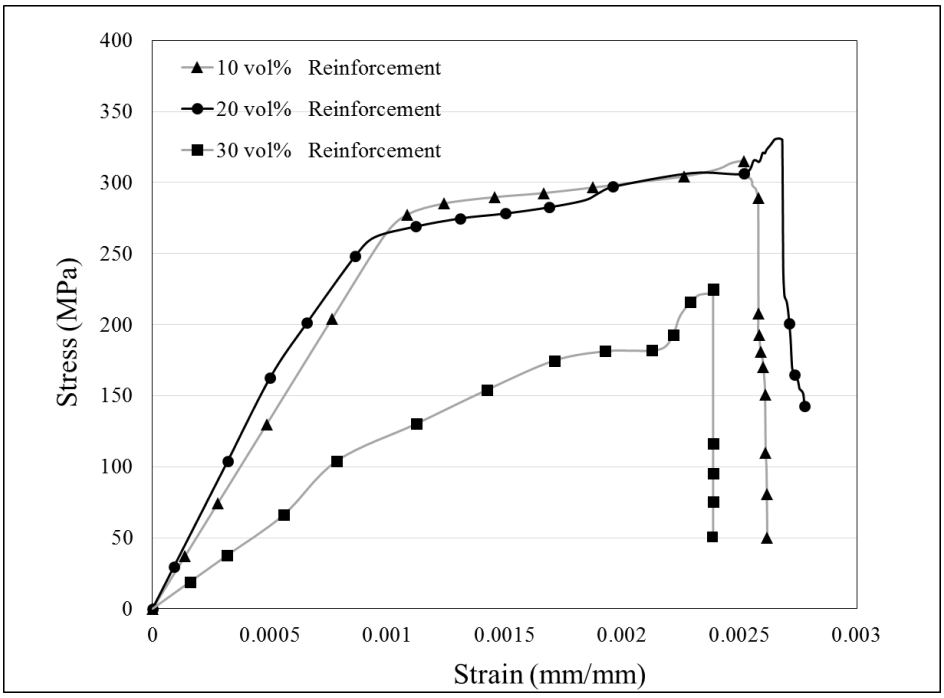


Figure 4.19. The stress-Strain curves of Fe-TiB_2 composites at different reinforcement vol% based on FEA.

The results of numerical simulation of compression test on composites with different amount of UHTC reinforcement confirmed the effectiveness of addition of reinforcement phase up to certain level as resulted before from experimental study. Stress maps generated from numerical solutions along with associated stress-strain curves were also in good agreement with experimental results. Both experimental and numerical analysis showed that there must be an optimum balance between the amount of reinforcement phase and matrix phase in the microstructure to improve mechanical properties of composites. At this optimum content, there is enough matrix phase to transfer the applied load and also enough UHTC reinforcement to carry the load. The future work on this study could be testing mechanical properties of such a composite at elevated temperature conditions to see effectivity of UHTC reinforcement on stability of iron to service at higher temperatures. It was observed that at any P/M route with specific process parameters, matrix has a certain level of capability to receive reinforcement particles. It is very

similar to solubility limitation in liquids. In fact, as it seen in this study, adding any extra TiB_2 particles more than 20 vol% resulted in unfavorable microstructures such as porosities and reinforcement agglomeration. The effect of these microstructural deteriorations were seen in mechanical behavior of sample with 30 vol% of TiB_2 in compression test which the sample failed in low stresses and exhibited a very brittle fracture.

5. CONCLUSIONS

In this study metal matrix composites reinforced by particulate ultra high temperature ceramics were fabricated by a P/M route which was experimentally optimized. In order to study the effect of volume percent of UHTC reinforcement on microstructure and mechanical properties of MMCs, different samples with different volume percentage of reinforcement were fabricated and studied. The results of this study is listed as follow:

- From manufacturing point of view, it was observed that uneven distribution of reinforcement particles can largely affects the densification process, in this regard the mixing process is critically important in any P/M route. It was also observed that frictional forces at the wall of the compacting die restrain the densification of mixed powders by acting against the applying pressure. Using high temperature dry-lubricants was very helpful to solve this problem.
- The P/M process for fabrication of Fe-TiB_2 composite was optimized and the process factors were suggested. The best results were obtained by the following route: cold pressing at 45 MPa, hot pressing at 45 MPa, 500 ℃ and 60 minutes and finally sintering at 1100 ℃ for 30 minutes.
- Density measurement, hardness and compression tests indicated that, addition of TiB_2 to Fe matrix and producing composite could improve the mechanical properties of Fe material.
- Increasing the volume percent of reinforcement improve the mechanical properties of Fe-TiB_2 composites up to 20 %vol and above this level adding more reinforcement adversely affected the mechanical properties.

- TiB_2 as a particulate Ultra High Temperature Ceramic reinforcement showed a two opposite behavior in Fe-TiB_2 composites. In fact on one hand due to its very high hardness, it increased the hardness of composite but its low density and formation of more porosity in composite compared to pure Fe material, it decreased the density of resultant composite. The overall mechanical properties of Fe-TiB_2 composite was the result of these two acting opposite factors.
- It was observed that adding more TiB_2 particles increased the chance of agglomeration of reinforcement particles in the microstructure. The TiB_2 rich areas, can be crack initiation zones inside the microstructures. In fact since the melting point and hardness of UHTCs are extremely high, it was very difficult to create ceramic-ceramic bonding between UHTCs particles during P/M process. The unbounded UHTCs particles agglomerated in a region could form and propagate the crack.
- Increasing the volume percent of TiB_2, increased the volume percent of porosity in composite due to the low density of TiB_2 which consequently increased the chance of formation of interconnected porosities inside the microstructure. These interconnected porosities negatively affected the mechanical properties of Fe-TiB_2 composites.

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