

# **WEAR PERFORMANCE INVESTIGATION OF METAL MATRIX COMPOSITES**

**BY:  
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## ABSTRACT

To improve the abrasive wear resistant which is governed by a combination of both high hardness and toughness, WC particles were used to reinforce the Hadfield steel. This latter is known with its good work hardening property. WC is mainly considered for its combination of suitable hardness and toughness. Various characteristic techniques were applied to study the microstructure, interface layer, and reinforced region of the MMC. The microstructure characteristics of WC was analyzed using an Optical microscope (Alicona), Scanning Electron Microscope (SEM), Vickers Hardness tester, and lab wear tester. The findings indicate that WC particles were evenly distributed inside the MMC and a well-fabricated metallurgical bond at interface has been formed between the matrix (Hadfield steel) and the WC particles. A significant increase in wear resistance and hardness was observed in all MMC samples in comparison with matrix.

**Keywords:** Metal Matrix Composites, Hadfield Steel, Tungsten carbide

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## TABLE OF CONTENTS

<b>1. INTRODUCTION</b>	<b>8</b>
1.1. Background	8
1.2. Objective	9
1.3. Thesis Layout	9
<b>2. LITERATURE REVIEW</b>	<b>10</b>
2.1 Hadfield Steel	10
2.1.1 Properties of Hadfield Steel	11
2.1.2 Alloying elements	11
2.2 Manufacturing and Heat Treatment	12
2.2.1 Heat Treatment	12
2.2.2 Microstructure, Grain Size and Mechanical Properties	13
2.3 Failure Reasons for Hadfield Steel	13
2.4 Factors Affecting Mechanical Properties	14
2.4.1 Temperature and Time	14
2.4.2 Carbon and Carbides Effect	15
2.5 Tungsten Carbide	15
2.5.1 Production of Tungsten Carbide	18
2.6 Composites	20
2.6.1 Matrix	26
2.6.2 Reinforcement	28
2.7 Metal Matrix Composites	30
2.7.1 Production and Processing of Metal Matrix Composites	31
2.7.2 Characteristics of Light Metal Composite Materials	31
2.7.3 Metal Matrix Composites Applications	34
2.7.4 Determination of MMCs damage:	34
2.7. Hadfield steel – Matrix	35
Reinforcement – Tungsten carbide	35
2.8 Manufacturing methods	35
2.8.1 Liquid state processing	36
2.8.2 Solid-state processing	37
<b>3. EQUIPMENTS USED</b>	<b>39</b>
3.1 Energy Dispersive X-Ray Spectroscopy (EDS)	39
3.2 Sample Preparation	40
3.3 Alicona	40
3.4 Scanning Electron Microscopy	41
3.5 Hardness Tester	42
3.6 Lab Wear test	42

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<b>4. EXPERIMENTAL PROCEDURE</b>	<b>43</b>
4.1 Material data	43
4.2 Materials mixing	44
4.3 EDS Analysis	44
4.4 Pressing	44
4.5 Casting	45
4.6 Sample Preparation	45
4.6.1 Sample Cutting	45
4.6.2 Hot Mounting	45
4.6.3 Grinding & Polishing	46
4.7 Characterization Techniques	47
4.7.1 Optical Microscope - Alicona	47
4.7.2 Scanning Electron Microscope (SEM)	48
4.7.3 Hardness Tester	50
<b>5. RESULTS AND DISCUSSION</b>	<b>50</b>
5.1 Powder processing: Mixing, pressing and casting	50
5.2 Characterization techniques	51
5.2.1 B*05 & C*05	51
5.2.2 C*XA & C*XA1	55
5.2.3 C*XB and C*XB1	59
5.3 Hardness	63
<b>6. Wear Test</b>	<b>64</b>
<b>7. Conclusion</b>	<b>65</b>
<b>8. Future work</b>	<b>66</b>
<b>9. Reference</b>	<b>67</b>

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## TABLE OF FIGURES

Figure 1 Ashby diagrams for materials. Left: Young's modulus vs. strength, suitable: wear-rate constant vs. hardness [12].....	17
Figure 2 Designation of each grade based on the WC grain size [12] .....	18
Figure 3 Comparison of steel, Al with composites based upon physical & mechanical properties [23] .....	20
Figure 4 Composite material formation [22].....	20
Figure 5 Classification of Composite Materials [23] .....	22
Figure 6 Properties of some thermoset resins [27] .....	23
Figure 7 Properties of some Thermoplastic resins [27].....	24
Figure 8 Strength vs. density and Young's modulus vs. density for different class of materials [23].....	24
Figure 9 Types of Composite materials [27].....	27
Figure 10 Reinforcement types: a) continuous long fibers, b) discontinuous short fibers or whiskers and c) particles in the matrix [18] .....	28
Figure 11 Structure of a unidirectional endless fiber-reinforced aluminum composite material (transverse grinding) [27] .....	32
Figure 12 SiC monofilament/Ti composite material [27] .....	32
Figure 13 Short-fiber reinforced light-alloy formation [27].....	32
Figure 14 Hybrid reinforced light metal composite material structure [27].....	33
Figure 15 Various production process for MMCs [21] .....	36
Figure 16 Melt infiltration (left) and Pressure infiltration (right) [21] .....	37
Figure 17 Squeeze casting infiltration [21] .....	37
Figure 18 Vacuum/gas sintering [21].....	38
Figure 19 Demonstration of working of EDS [29].....	39
Figure 20 (a) Disk Cutting Machine, (b) Hot Mounting Machine at LTH.....	40
Figure 21 Infinite Focus Alicona at LTH.....	41
Figure 22 Demonstration of working of SEM at geocentrum I.....	41
Figure 23 Vickers Hardness testing machine [30] .....	42
Figure 24 Schematic representation of Vickers Hardness Test [20] .....	42

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Figure 25 Selected samples-WC .....	43
Figure 26 Selected samples – TiC.....	43
Figure 27 EDS analysis of powders showing a) good mixing b) poor mixing.....	44
Figure 28 sample embedded in epoxy.....	46
Figure 29 Alicona images of one of the samples .....	47
Figure 30 Alicona images of all samples reinforced region.....	48
Figure 31 SEM images at reinforced region .....	48
Figure 32 SEM images at interface between matrix and reinforced region .....	49
Figure 33 Hardness values graph .....	50
Figure 34 Alicona Images at lower magnifications (a) B*05 (b) C*05 .....	51
Figure 35 Interface layer between Matrix and Composite of B*05 (Left) & C*05 (Right) .....	51
Figure 36 SEM image of B*05 reinforced region at different mag.....	52
Figure 37 SEM image of C*05 reinforced region at different mag.....	52
Figure 38 SEM images of (a)&(b) B*05 interface and (c)&(d) C*05 interface.....	53
Figure 39 Comparison graph between B*05 and C*05.....	54
Figure 40 Graph between Avg. Hardness Vs Avg. Particle Size of B*05 & C*05	55
Figure 41 Alicona images at a magnification (a) C*XA (b) C*XA1 .....	55
Figure 42 Interface layer between matrix and composite (a) C*XA & (b) C*XA156	
Figure 43 SEM image of C*XA1 reinforced region. ....	56
Figure 44 SEM images of C*XA1 interface .....	57
Figure 45 Comparison graph between C*XA and C*XA1 .....	58
Figure 46 Graph between Avg. Hardness Vs Avg. Particle Size of C*XA & C*XA1 .....	58
Figure 47 Alicona Images of (a) C*XB and (b) C*XB1 .....	59
Figure 48 Interface layer thickness of C*XB (Left) and C*XB1 (Right) .....	59
Figure 49 SEM image at reinforce region of C*XB (left) & C*XB1 (right) .....	60
Figure 50 SEM images at interface layer of (a)&(b) C*XB and (c)&(d) C*XB1 interface.....	61
Figure 51 Comparison of Average hardness values for C*XB and C*XB1 .....	61

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Figure 52 Graph between Avg. Hardness Vs Avg. Particle Size of C*XB & C*XB1 .....	62
Figure 53 Graphs of average hardness values at different locations (Hadfield steel, Interface, MMC at interface and MMC-inside).....	63
Figure 54 Graphical representation of wear ratio of all the samples.....	64

## **LIST OF TABLES**

Table 1 Sample naming data .....	44
Table 2 Sequence of Grinding and Polishing Steps .....	46

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# 1. INTRODUCTION

## 1.1. Background

Material characterization and mechanical property testing have become more important in the current era. As technology advances, it is necessary to improve material properties. Currently, many industries are concentrating on achieving high production parameters and mechanical properties of a material. Mechanical properties vary depending on the application. It is essential to create a material that is suitable for a particular area. For instance, in the mining sector, machines are used for crushing massive rocks into more manageable pieces that continuously enhances in their mechanical properties. As a result of such applications, many new advanced materials are created to withstand extreme wear conditions, extending lifespan of the machine.

Metal Matrix Composite (MMC) is quite possibly the best material to generate good mechanical properties. In particular, MMCs have gained a great deal of attention from industries, as they combine the great strength of the matrix and the high hardness, wear resistance properties of the fiber. The main important factors to be concerned are wear resistance, significant impact loads.

In other words, choosing the right materials for this activity can fundamentally increase the machine's lifespan, consequently decreasing its cost. In these applications, Hadfield steel is widely used due to its high hardness, high wear resistance, and high hardenability. Even though Hadfield steel has excellent mechanical properties, it couldn't maintain a long life due to low impact resistance, replacing the tool involves significant expenses which is a costly process to fix.

A few studies have been conducted to identify a material for a heavy-duty application that could meet the essentials while reducing expenses and overcoming this systematic process of replacing tools.

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As a result, a composite material was developed using Hadfield steel as the matrix, and cemented carbides as the reinforcement. This has prompted many industries to invest in creation of MMC. As part of the research, this thesis focuses on developing a MMC that uses Hadfield steel as the matrix material and Tungsten Carbide as the reinforce material. The MMC was analyzed with Alicona and SEM-EDS, Vickers hardness tester as well as performed wear analysis.

## **1.2.Objective**

The main aim of the thesis is to develop a MMC with WC reinforcement and perform wear analysis. By finding a proper material and suitable production method to create MMC. In order to determine whether MMC is feasible, a detailed examination of the microstructure is studied followed by wear test analysis.

## **1.3.Thesis Layout**

This thesis began with a proper introduction explaining the background and purpose of the work. An extensive literature review was conducted to gain an understanding of MMC, Hadfield steel, WC, and manufacturing methods. Using the literature review, the key findings are presented with references and justified appropriately. This project describes and explains the experimental process along with the equipment used in the lab. Several samples were produced, which include a material description. A detailed analysis of the MMC was performed using Optical microscope (Alicona), Scanning Electron Microscope (SEM), Vickers Hardness tester and the results are presented along with wear analysis.

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## 2. LITERATURE REVIEW

The aim of the literature review is to summarize the findings that we were able to gather after reading journals, articles, and books. This study concentrates on Hadfield steel, its manufacturing process, properties, Composites, MMC, tungsten carbide.

The research methodology used in this thesis is a combination of qualitative research in terms of gathering data and a lots of action research where we have performed actions, evaluation and changes have been made based on the results.

### 2.1 Hadfield Steel

Hadfield steel is the first-ever Manganese Austenite Steel, with carbon and manganese being the main constituents. It started with experiments where manganese was present in a high percentage (Over 50%) along with carbon and iron being the remaining share [4][6]. The material did not possess desired mechanical properties. The trials still were continuing in search of material that can have good mechanical properties. After specific trials, a material with properties like carbon steels was attained [5]. Then Sir Robert Hadfield started to perform experiments with percentages close to that ratio and discovered an alloy with superior mechanical properties to much other high-strength steel. The first chemical composition involved 1.35% carbon and 13.76% manganese; after performing various tests, he concluded that this alloy has better properties. Hadfield has taken patent for this material in 1883, but has not let it out in public; he experimented with the compositions for the next 5 years. After the tests, he has finalized that Hadfield steel can have the best properties with 12-14% of manganese and 1.2% of carbon. After a few more years of study and research by other researchers, it is finalized that the composition could be 10-14% manganese and 1 to 1.4% carbon [5].

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### **2.1.1 Properties of Hadfield Steel**

The properties possessed by Hadfield steel were high toughness, high ductility, excellent wear resistance. Apart from these properties, Hadfield steel was known for one major characteristic, i.e., work hardening ability; Hadfield steel has a very high work hardening ability than other materials. The drawbacks are low thermal conductivity, low hardness, low machinability and it is difficult to achieve good mechanical properties in high thickness specimens [1][2][3][4]. Hadfield steel also has good impact strength, which can absorb sudden shocks given during the operation. A combination of such good mechanical properties means a wide variety of applications where wear resistance is critical. Some of the major application areas of Hadfield steel are in industries like cement, construction, mining, and railways where high strength, high wear resistance qualities are essential [1][3].

### **2.1.2 Alloying elements**

There are many choices of elements to add to this alloy depending upon the additive characteristics being boosted in the Hadfield steel. This decision is entirely based on the type of application and the customer requirements. Numerous additions can be made to Hadfield steel, where each material has some impact on properties. Elements used till now are: Manganese, Carbon, Silicon, Chromium, Molybdenum, Nickel, Aluminum, Phosphorous, Sulphur, Titanium, Boron, Cerium. Many researchers have performed tests by adding different alloying materials and studied the properties of the compositions. Some of the study results are that the addition of chromium helps in increasing wear resistance. 1.7 % of the total weight is the best contribution. Adding more amount of chromium, leads to an increase in carbide formation, decreasing the toughness. Titanium can be added to attain fine-grained structure and increase hardness. Vanadium helps to increase the hardness of the material (Tested in XRD) [5][6]. Aluminum boosts deoxidation and reduction of carbides at the boundary. It also increases the solubility of carbon in the austenite crystal network. Further more additions of aluminum decreases the mechanical properties. Nickel is a strong austenite stabilizer that prevents the complete transformation and

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precipitates of carbides even at decreased cooling rates during the quenching process.

Similarly, in the same pattern, analysis for different material addition has been performed. A proper bracket of the percentage of weight added to the alloy has been determined, which could help attain the required mechanical properties. The addition of a disproportionate percentage of any material has adverse effects on the mechanical properties of the alloy [4][5][6].

## **2.2 Manufacturing and Heat Treatment**

A low superheat casting process is used to manufacture Hadfield steel.

### **2.2.1 Heat Treatment**

Annealing is the most critical process in heat treatment. It is necessary to study the composition and decide the operational temperature, temperature, and process time. The temperature range depends on the thickness and length of the materials. Dimensions play a crucial role in determining thermal properties like the melting point of the material.

The temperature range of heat treatment for Hadfield steel is 900-1150 degrees. The increase in temperature rate is dependent on one of the thermal characteristics, i.e., thermal expansion coefficient. The temperature rising rate is inversely proportional to the thermal expansion coefficient, which means the increase in expansion coefficient decreases a temperature increase rate. For Hadfield steel, the temperature in the heat treatment must be increased gradually as the steel has an expansion coefficient of 3%. The temperature rising rate must not be more than 100 degrees per hour [4][6][7]. The holding time of metal in heat treatment depends on the thickness of the material, expansion coefficient, and the mechanical properties desired for the outcome. For Hadfield steel, holding time is at least 1 hour/25 mm of the maximum wall thickness of the part passing through the furnace [6].

After the annealing, immediate quenching is required. It is important to follow proper cooling medium, temperature, and cooling rate, just as with the heat treatment. Salt can be added while quenching in water as it helps in

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the cooling process and increases the dissolution of carbides. After many experiments, they concluded that the quenching process for Hadfield steel needs to be carried out with water to have proper microstructure [4][6].

### **2.2.2 Microstructure, Grain Size and Mechanical Properties**

Hadfield is a fully austenitic single-phase structure. This phase is acquired only after undergoing austenitic heat treatment after casting. Immediately after casting, the phase formed is austenitic with carbide. Having heat treatment below 1000 degrees can help reduce carbides on boundaries. Very important to consider the melting temperature. If the temperature is too high, it leads to solidification, resulting in high grain size and porosity. If the heat treatment temperature is limited to a point close to the melting point, then less grain size can be obtained considering the thickness of the material. Quenching is performed in water [8] [7] [5].

Olivine (Light Weight) And Chromite (Heavy Weight) Sandstone Are Most Suitable for Moulds.

- To keep the grain size reasonably small, use a low pouring temperature.
- To facilitate the dissolution in the austenitic range, it is essential to have the correct temperature and holding time in the heat treatment process. The quenching must be optimized to minimize the precipitation of carbides inside the grains and grain boundaries [3].

## **2.3 Failure Reasons for Hadfield Steel**

Based on a literature review, researchers has performed experimental tests on various samples from a breakdown crusher and analyzed to find the reasons behind failure. After detailed analysis, following reasons are observed: Having inappropriate material compositions could lead to improper mechanical properties for the application, leading to failure. For Hadfield steel, it is important to have proper temperature while pouring the molten material into the mould. To sustain high temperature, a mould should be made of suitable material. Wrong pouring practice of molten metal could

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lead to cracks or air bubbles, leading to failure. In heat treatment, having improper parameters like temperature, increasing rate, there is a possibility of forming carbides on the grain boundaries, leading to the failure of the material.

Material must be applied in the proper location. For instance, when the material is designed for crushing small rocks, but is applied to crushing much larger rocks, the material tends to fail because of high impact energy and hard rocks [3] [9].

## **2.4 Factors Affecting Mechanical Properties**

After some researches, it was observed that many factors affect the mechanical properties of the materials. The most common and frequent factors, as per our knowledge, are Grain Size, temperature, time, carbide formations, and section thickness. All these factors are interlinked. Majority of the factors are depended on temperature and time parameters [3] [9].

### **2.4.1 Temperature and Time**

In an article, the author has performed heat treatment with rising in temperature for four specimens of same type. There was a difference of 100° C maintained between each specimen. The results obtained from this article describes following statements. In heat treatment, rise in temperature leads to decrease in the amount of carbide in the structure and increase in mechanical properties, like wear resistance, yield strength, tensile strength, and hardness. The major drawback is that the impact energy decreases along with the elongation ratio of the material [7][3] [9].

The main reasons for their change in characteristics is due to increase in grain size because of the temperature rise, and the dissolution of carbides also increases as the temperature increases. The author has concluded that to attain a perfect austenite structure; it is important to have the heat treatment parameters accurate and make sure that the grain size does not exceed than required [3].



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## 2.4.2 Carbon and Carbides Effect

The nominal carbon percentage bracket had been finalized as 1-1.4%, while most industries prefer to use 1.2% as it has shown more favorable properties [5]. The main advantage of having more carbon content is that it increases the abrasion resistance. The amount of carbon reacts with the other alloying metals to form carbides. The more amount of carbides present in the material, the increases hardness, Yield strength, stiffness. The primary property for which Hadfield steel is known is work hardening ability which is also depends on the carbon percent.

- An increase in carbides at grain boundaries leads to an increase in possibilities of brittle nature.
- Decreases toughness, ductility, and impact strength.
- An increase in the thickness of the specimen leads to an increase in carbide particles.
- A slow rate of cooling while quenching leads to an increase in carbide formation at grain boundaries.

## 2.5 Tungsten Carbide

Tungsten carbide is one of the significant refractory carbides which has a promising future in various industrial applications. 'Refractory' a term that refers to materials with a high melting point (Over 1700 C) and a high inertness to chemical reactions [24]. Tungsten was first found in Erz Mountains in Saxony, Germany. It was called with different names in German: Wolfart, wolfram, wolframite. In 1758, a Swedish chemist Cronstedt found a heavy mineral named Tungsten (Swedish name for heavy stone). These minerals are found in the form of tungstates which have a considerable content of two materials, i.e., wolframite ((Fe, Mn)WO<sub>4</sub>) and scheelite (CaWO<sub>4</sub>) [25] [26]. China has the largest bank of tungsten ores, followed by Canada, the US, etc. A sintering process produced the first-ever Tungsten Carbide to draw dies and rock drills [26]. In the current scenario, over 90% of the cemented carbide tools are made of tungsten carbide. With improved design options and manufacturing technologies, the development of cutting tools has been dramatically increased. This development has led

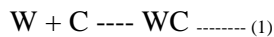
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to the advancement of the performance of these cemented carbides and has increased the application scope of these materials [32] [33]. Due to the additions of alloy elements, material gained properties like abrasion resistance, shock resistance which has opened a possibility for many other applications. The significant areas that can be focused on Tungsten Carbide are:

1. Developing a composition that can produce better mechanical properties.
2. Finding alternative methods for efficient way of producing WC.
3. Modifying the present compositions by adding other elements.

Most of the applications of WC have involvement of a binder phase which is a ductile. This is because tungsten has good properties like hardness and wear resistance but lacks in adequate toughness. So, tungsten carbide needs a ductile binder phase to add toughness and other properties [26].

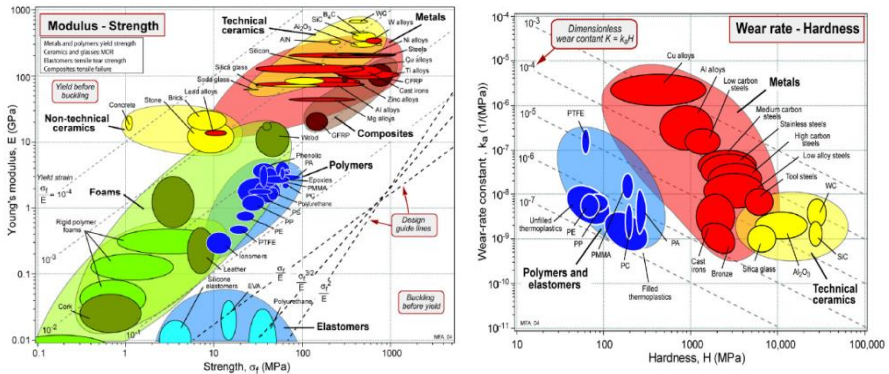
Cemented carbides are widely used in various industrial applications like metal machining, oil drilling, mine excavation, etc., which provides an excellent combination of hardness, strength, toughness and chemical stability. It consists of two major phases: complex refractory phase WC and soft metal binder phase [9] [12]. Tungsten carbide is formed by mixing pure tungsten powder with pure carbon at a high temperature of around 1500°C in a vacuum or the presence of hydrogen [9].



According to the Tulhoff, 2000: Tungsten carbide can be manufactured through several methods which are: tungsten oxides (WO<sub>3</sub>), tungstic acid (H<sub>2</sub>WO<sub>4</sub>), ammonium Paratungstate and scheelite (CaWO<sub>4</sub>) [9]:



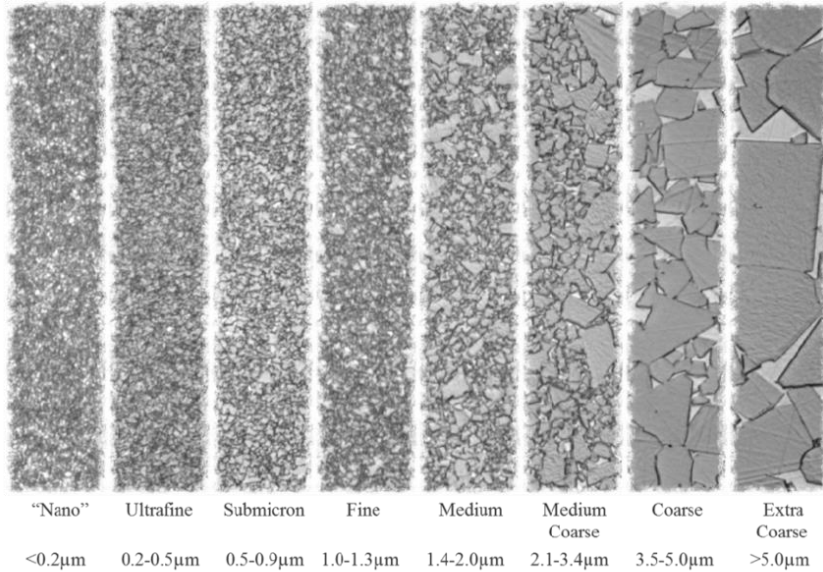
Tungsten carbide is a hexagonal close packed (HCP) structure which is a refractory carbide. It has high toughness and plastically can deform by forming slip planes instead of experiencing brittle failure [10].



**Figure 1** Ashby diagrams for materials. *Left: Young's modulus vs. strength, suitable: wear-rate constant vs. hardness [12]*

These materials are commonly used in nuclear refractory parts, micro-drills, medical equipment, wear-resistant parts, cutting tools because of their mechanical properties like high hardness, wear resistance, melting point, and corrosion resistance [11].

Tungsten carbide can be found in immense applications in powdered form, mainly to add the mechanical properties as desired for an application. Grain size of the elements will show the effect of the mechanical properties, i.e., a decrease in WC grain size increases compressive strength, hardness, and bending strength whereas the toughness, impact strength, and rupture strength decrease.



*Figure 2 Designation of each grade based on the WC grain size [12]*

### 2.5.1 Production of Tungsten Carbide

Tungsten carbide powders are acquired from tungsten-bearing ores. Scheelite ( $\text{CaWO}_4$ ) and Wolframite ( $(\text{Fe},\text{Mn})\text{WO}_4$ ) are the primary mineral wellsprings of tungsten powder. W's high melting point limits the extraction options to hydro process instead of a pyrometallurgical process. The essential initiation step of extracting any material is carried out by understanding the ore and removing the impurities to the highest possible extent. Either roasting or leaching does this with the help of some dilute acids for lower grade mineral ores, while concentrated acids are used for high-grade mineral ores. The following step is to reduce the whole ore to more minor compounds where extraction would become more accessible. And finally, the compound is reduced, and the remaining impurities are cleared to obtain the final material.

Production of Tungsten Carbide (WC) was performed by carburization process. It is essential to have a homogenous mixture of tungsten and carbon, so mixing is performed before carburization. Ball milling or a conical blender

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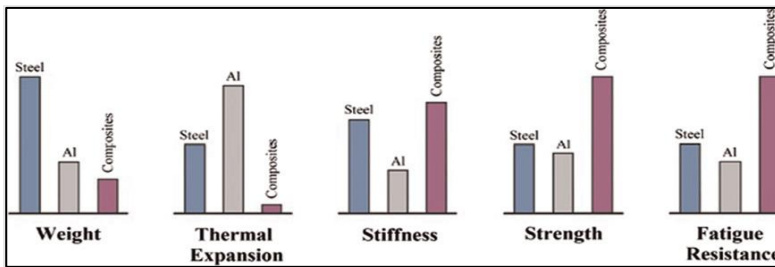
at high RPM are used to mix the powders because of the big difference between the densities of tungsten and carbon. One area of concern, while selecting carbon material is purity. Sulfur is the most common impurity found in carbon, has a significant effect on the grain size of WC. The mixing method also depends on the form of W (Individual particles, firmly bonded polycrystalline pseudomorphs of the original Ammonium Paratungstate (APT) crystals). The mixing of individual particles is relatively easy, but it is important to choose a proper mixing technique for APT crystals. Pseudomorphs are supposed to be broken down effectively to achieve uniform carbide formation. If not, the pseudomorphs weld upon each other, resulting in carbides with high grain size. The carburization process is carried out by the mixture of tungsten and carbon powder and the presence of some external gas with some pressure of carbon. The carburization process is performed in either graphite tube furnaces or a high-frequency induction furnace.

In a graphite tube furnace, the W, C powders mix is placed on graphite boats and excited by non-stop electric power in the furnace. Hydrogen gas is sent into the furnace from the discharge end. When it comes to the induction furnace, the main parts are an induction coil and a graphite crucible susceptor surrounded by insulation. Similar to the process in graphite tube furnaces, the mixture of W, C is placed in a graphite tray and kept inside the graphite susceptor. Hydrogen gas is let inside through a small opening in the susceptor; the charge is heated to the carburization temperature and held stable for a couple of hours. After the cooling of carbides under hydrogen surroundings; the outcome is broken down in a hammer mill and grinding in a ball mill. The final product from the ball mill is separated into different particle sizes using a vibrating screen. Depending upon the temperature and time, the particle sizes found to be altering during carburization. It is observed that the increase in carburization temperature leads to an increase in particle size of WC. The ideal temperature for carburization is considered to be between 1300-1500 degrees.

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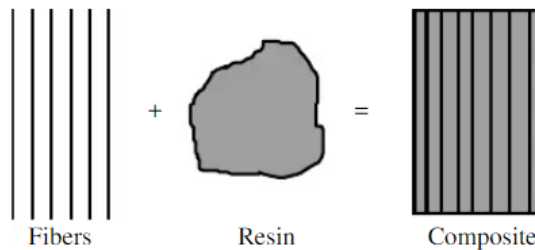
## 2.6 Composites

In 1960, composite materials were found in the form of polymeric-based composites, which started to gain focus from industries. These materials are widely used in common engineering applications for a long time to resolve technical problems for different industries like automotive, aerospace, automotive, and marine. These types of materials became a competition to other materials in the global market due their product performance as lightweight components. Composites are commonly used in the replacement of steel and Aluminum for better performance. Further, component weight can be saved 60-80% in replacement. Nowadays, composite materials are a possibility for various engineering applications [22].



*Figure 3 Comparison of steel, Al with composites based upon physical & mechanical properties [23]*

Generally, composite materials are formed by reinforcing fibers in a matrix resin as shown in the **Figure 4** [22].



*Figure 4 Composite material formation [22]*

Composites are found in nature, not invented by humans. For example, wood is a composite of cellulose fibers in lignin, a natural glue matrix. Researchers

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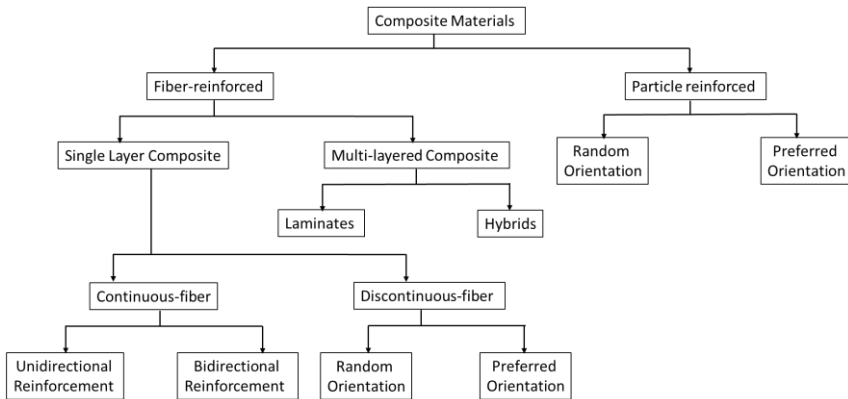
have found that spider web fibers are stronger than synthetic fibers. For several hundred years, in India, Greece, and other countries, husks or straws were mixed with clay to build houses. An example of particulate composite is combining husk or sawdust in clay, and mixing with straws in clay is an example for short fiber composites. These types of reinforcements are used to improve the performance of particular applications [22].

Main mechanical properties of composite material are [23]:

- High stiffness and strength
- High young's modulus
- High wear and corrosion resistance
- Low density
- High fatigue strength at elevated temperatures

Composite materials combine two or more materials that produce properties that do not exist in one material, and these are widely used for producing a product because of their higher mechanical properties like strength and stiffness. It is obtained by two materials which are matrix and reinforcement. The two materials are bonded together to produce a composite material that consists of good machinability, toughness, high hardness, and wear resistance. The matrix material is used for distributing stress and protecting the reinforcement material. The reinforcement material's role is to provide high mechanical properties for composite material and strengthen the matrix in favorable directions. The composite material properties depend upon the reinforcement and matrix behavior, reinforcement formation (fibers, particles), and the amount of matrix and reinforcement used [13] [14].

The composite materials have advantageous properties like light weight, flexibility, good properties like high corrosion resistance, impact strength, etc. These properties are the main reason for being considered an option for traditional materials with applications in various fields like automotive, aerospace etc. The material properties can be customized by selecting materials with the required properties for matrix and reinforcements.



*Figure 5 Classification of Composite Materials [23]*

The matrix is the main constituent with more than 50% of the composition, and reinforcement is the material that is supposed to have the properties that the matrix material lacks. The reinforced materials can be in two primary forms, either particulate or fiber, which is why the composites are named on type of reinforcement i.e. particulate composites and fibrous composites. In composites, the matrix has the highest weight percentage; so, called to be continuous, while reinforcement materials are known to be in a discontinuous phase.

Fiber Reinforced Plastic (FRP) is one of the critical parts of the engineering material market. FRP possesses excellent properties, but the only negative point is the manufacturing methods. The materials used in their composites are readily available as a replacement for traditional materials at acceptable costs, but the manufacturing process has been the bottleneck for this case. There are many methods to manufacture FRP, namely:

1. Vacuum Bagging
2. Autoclave
3. Filament winding
4. Pultrusion
5. Matching die set compression molding
6. Resin Transfer Molding (RTM)
7. Resin Infusion



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Most of these methods come under the liquid composite molding technique. The primary concern regarding liquid composite molding is that the machining process is quite expensive and lengthy cure times.

Polymer Matrix Composites (PMC) have a wide variety of applications, covering the central part of composite types. PMC has fascinating properties which can be matched to a wide range of requirements by making the proper choice of constituent materials and their parameters. PMC is known for having less weight and properties like high strength, stiffness, good chemical stability. There are two types of resins used in manufacturing a PMC; they are thermosets and thermoplastics. In the current scenario, thermoset resins are used for most applications. There are many types of thermosets, but the most of the used thermoset resins, along with some of their properties, are mentioned in below *Figure 6*.

Resin	Density (g/cm <sup>3</sup> )	Tensile strength (MPa)	Tensile modulus (GPa)	HDT (°C)	Cure shrinkage (%)	Glass transition temp. (°C)
Epoxies	1.2–1.3	55–130	2.75–4.10	48	1–5	100–270
Polyester	1.1–1.43	34.5–103.5	2.1–3.45	60–205	5–12	70–120
Vinyl ester	1.12–1.32	73–81	3.0–3.5	93–135	5.4–10.3	102–150
Phenolic	1.00–1.25	30–50	3.6	165–175	0–0.01	260
Polyimide (PMR-15)	1.32	38.6	3.9	–	0–0.006	320–330
Polyurethane	1.1–1.5	1–69	0.069–0.69	50–205	0.02	135

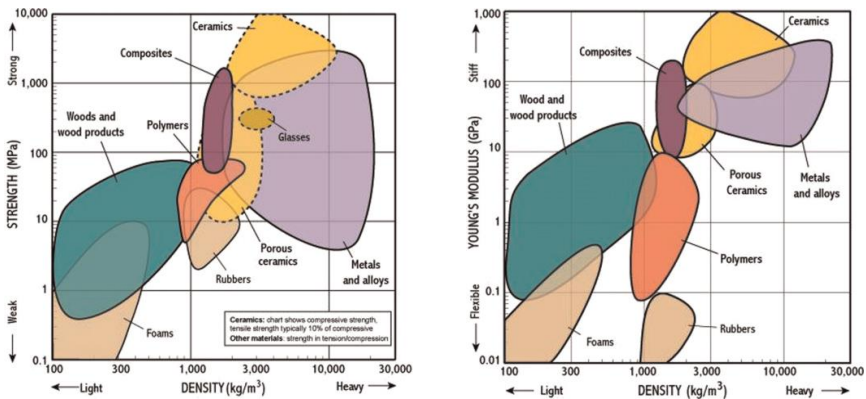
*Figure 6 Properties of some thermoset resins [27]*

Thermoplastic resins are non-reactive solids; the use of pressure and heat can operate these. They can be quickly restructured whenever required. Some of the types of thermoplastic resins and their properties are listed **Figure 7**.

Resin	Density (g/cm <sup>3</sup> )	Tensile strength (MPa)	Tensile modulus (GPa)	HDT (°C)	Cure shrinkage (%)	Glass transition temp. (°C)
Acrylonitrile-Butadiene-Styrene, molded	0.88–3.5	24.1–73.1	0.78–6.1	65–220	–	105–109
Polystyrene	1.02–1.18	17.9–60.7	0.3–3.35	62–98	0.002–0.008	83–100
Nylon (PA6)	1.12–1.14	41–166	2.6–3.2	68–85	0.003–0.015	47
Polycarbonate (molded)	0.95–1.51	46.1–93.1	1.8–3.0	78–187	–	143–152
Polysulfone	1.37–1.48	60–131	–	172–213	–	–
Polyethylene, HDPE (glass filled)	0.94–1.53	11–113	0.7–13.6	51.7–127	0.001–0.003	–

**Figure 7 Properties of some Thermoplastic resins [27]**

Reinforcing Materials used are fibers. Fibers tend to be more assertive when produced in smaller dimensions, and the selection of fibers can be altered to attain customized properties. Fibers are available in various forms, i.e., yarn, roving, chopped strands, woven fabric, and mats. There are four types of fabrics: glass fibers, carbon fibers, aramid fiber, and boron fiber.



**Figure 8 Strength vs. density and Young's modulus vs. density for different class of materials [23]**

The interface boundary between matrix and reinforcement material plays a crucial role in obtaining good mechanical properties. A better interface can increase bonding strength, preventing decohesion between matrix and reinforcement. For example, the difference in the thermal expansion of the

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two phases led to the increased crack formation, which is not preferable. To improve the wettability of composites for forming a good interface, there are three ways to change the composition of reinforcement material, additional metal elements included in the metal matrix, and modifying the surface of the ceramics. In addition, a strong interface can be achieved by reducing the reinforcement dimensions as it provides an increase in the net contacting area between matrix and reinforcement [13].

## **Functions of Matrix and Fibers**

A composite material is created by reinforcing fibers with a matrix. One should understand the roles of fiber and matrix in a composite material. Some of the fiber and matrix functions are discussed below [22].

In composites, the main functions of the fibers are:

- In a structural composite, 70-90% of the load is carried out by the fibers.
- To provide strength, stiffness, thermal stability, and other structural-mechanical properties.

The main functions of the matrix material are:

- To provide rigidity and shape to the structure
- Good surface finish quality
- Protects fibers against damage due to wear and chemicals.
- Stops or slows the crack propagation by isolating the fibers so that fibers can act separately.
- Performance characteristics such as ductility, strength, toughness can be increased.

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## **Disadvantages of Composites:**

Apart of immense benefits of composites, they also have some drawbacks, which are [22]:

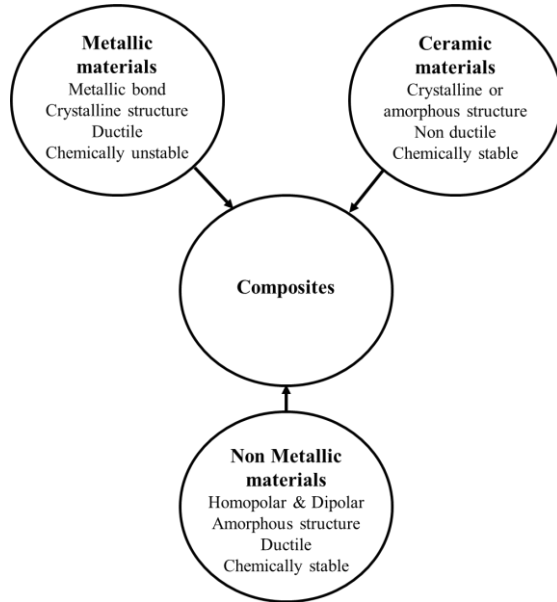
- High material cost in comparison with steel and Aluminum
- Lack of high-volume production methods
- Lack of database in designing parts with composites
- Properties can be influenced by the parameters like temperature, dimensional stability, chemical resistance.

### **2.6.1 Matrix**

The primary purpose of the matrix is to hold up the reinforcement together by its cohesive and adhesive nature. Matrix provides a solid structure to the composite, which is necessary for discontinuously reinforced composites. Often, the matrix is a weak link in the composite as the reinforcement fibers are stronger and stiffer from a structural point of view. Further, the matrix controls transverse properties, interlaminar strength, and high elevated temperatures of the composite. The matrix still allows the reinforcement strength to be utilized by providing load transfer effectively from external load to the reinforcement [23].

Machining of composite materials depends upon the content and properties of their respective materials used. Composites are classified into three types based upon the matrix material used [13] [14] [23]:

1. Polymer matrix composite (PMC)
2. Metal matrix composite (MMC)
3. Ceramic matrix composite (CMC)



*Figure 9 Types of Composite materials [27]*

In PMCs, common types of reinforcement are strong and brittle fibers containing soft and ductile polymeric matrices. The applications of PMCs are in the manufacturing of structural panels, aircraft, beams, parts for automobiles, etc., The life span for these types of composites are low as the matrix leads to softening, chemical decomposition at moderate temperatures. MMCs are commonly used in aerospace industries, automobile engine parts. These are used for machines that require high operating temperatures. In this process, the matrix used for binding with reinforcement is metal. However, in some applications, MMC has some disadvantages compared with metals and PMCs, high cost, complex production methods, and lack of knowledge over technology [23]. When it comes to the CMCs, they are used to improve the fracture toughness of unreinforced ceramics. Continuous fibers, Discontinuous fibers(whiskers) or particles are used as reinforcement materials in CMC [13] [14].

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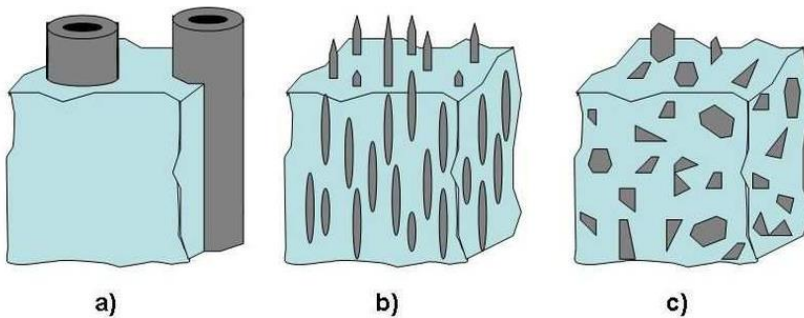
## 2.6.2 Reinforcement

Reinforcements are more substantial than the matrix, which is also called the reinforcing phase. It is generally possess following properties [23]:

- Low density
- Good mechanical and chemical properties
- Good thermal stability
- High compression and tensile strength
- High young's modulus
- Economic efficiency

For optimal mechanical properties, the interface in a composite should be robust, and a interface layer with a suitable thickness occurs. Generally, reinforcement is classified into continuous reinforcement (Mono-filaments) and intermittent reinforcements (particles). A complex demand profile is required for reinforcement in metal matrix composites formed by the matrix arrangement of the composite material. This specific profile is crucial for the formation of composite material [17]. Different types of reinforcements are

- a) Continuous fiber reinforcement.
- b) Discontinuous short fiber or whiskers reinforcement
- c) Particle reinforcement [18]



*Figure 10 Reinforcement types: a) continuous long fibers, b) discontinuous short fibers or whiskers and c) particles in the matrix [18]*

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## **Particle Reinforcement**

Hard ceramic particles are generally chosen for the reinforcement of magnesium alloys. Particle materials are carbides (ZrC, TiC, W<sub>2</sub>C, WC), nitrides (BN, TiN, ZrN), oxides (ZrO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>), and borides (TiB<sub>2</sub>, ZrB<sub>2</sub>, WB). The chemical reactivity between the matrix and the particles is considered while choosing the reinforcement type. An interface layer is essential for transferring the external and internal stresses to achieve good mechanical properties. Type and size of reinforcement influence the composite properties along with the shape of the particles. Three types of particle shapes are available, which are round, blocky, and platelet. Generally, platelet shape is used to form a good bonding, contact with a crystal plane that is not much possible in round particles. Usually, sharp edge particles are not considered because the edges can act as a start point for cracks when the material is loaded. For attaining a low-density composite, the reinforcement particle density should be low [18].

## **Continuous Fiber Reinforcement**

In continuous fiber reinforcement, primarily carbon (graphite) or ceramic are utilized. Ceramic types are alumina, silica, boron, zirconia, boron nitride, boron carbide. These types of filaments are weak, defect materials [19]. Only fewer materials are available in continuous fiber shape when compared with the particles. There are two types of fibers which are monofilament and multifilament. Either one of the fiber types is used in most of the composites [18].

## **Short Fiber / Whiskers Reinforcement**

Short fibers stand in the middle between long fibers and particles concerned with the reinforcing and strengthening effect, whereas the continuous fiber results in high anisotropic mechanical properties, and particle reinforcement results in nearly isotropic properties. It is possible to vary the mechanical properties by using short fibers with the right amount and reinforcing fibers.

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When it comes to whisker reinforcement, whiskers are tiny, needle-like single crystals with a diameter of 1 $\mu$ m. They are processed from oversaturated gases from solids or electrolysis solutions. Due to the production conditions, they contain low defect density. Because of their small size has led to a discussion about the health risks. They can be inhaled, leading to carcinogenic effects [18].

## 2.7 Metal Matrix Composites

Metal matrix composites are a group of materials used for the applications such as space and aviation industries, automobile industries, etc., that require high hardness, wear-resistance, and strength. Ceramic particles increase the wear resistance, melting point, hardness, and conductivity while depending upon the matrix material. If the composite material is bonded correctly, then a good level of corrosion resistance is maintained [15].

The main aim of implementing MMCs is to integrate properties of matrix and reinforcement. Metal matrix composites combine a reinforcement phase (ceramics or metallic) and a metallic matrix that consists of 50 % of the total material volume. The advantages of the MMCs are an increase in specific strength and stiffness, high-temperature limits, low density, and high wear resistances. A significant consideration for the selection of matrix is the nature of the reinforcement. The selection of MMC material is based upon an evaluation of individual characteristics and bonding between each other. [23].

The main objectives for the development of MMCs are [27]:

- Increase in creep resistance at elevated temperatures
- Increase in tensile strength and yield strength
- Increase in fatigue strength at high temperatures
- Increase in Young's modulus property
- Improvement in corrosion and thermal shock resistance
- Decrease in thermal elongation



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An increase in the usage of MMCs has led to solutions for many problems, but when it comes to the light metal area, there is no progress. Because of these, insufficient reliability and process results in production problems and incapability in economic efficiency.

In the present paper, Hadfield steel is considered as a matrix and Tungsten carbide as reinforcement materials.

### **2.7.1 Production and Processing of Metal Matrix Composites**

Different techniques can be used to produce metal matrix composite. The selection of the desired process entirely depends upon the reinforcement type, matrix, quantity, and application type. Although the composition and number of elements are the same, different characteristic profiles can be obtained by altering the manufacturing techniques, reinforcement formation, and processing.

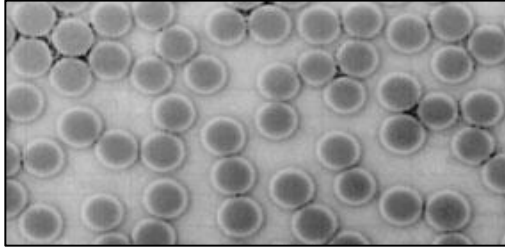
Following are the product engineering types for MMCs [27]:

- Melting metallurgical processes
- Hot isostatic pressing of powder mixtures and fiber clutches
- Joining and welding of semi-manufactured components
- Finishing by machining techniques

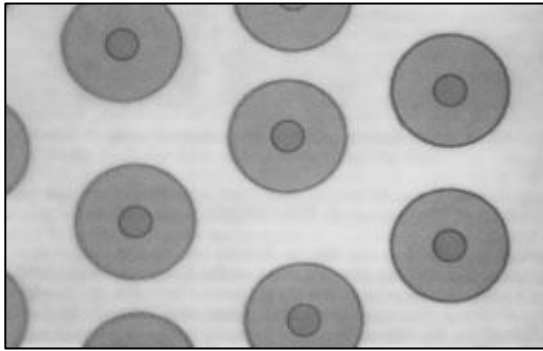
### **2.7.2 Characteristics of Light Metal Composite Materials**

The formation of the composite materials is determined by the reinforcement components type and form where the manufacturing processes vary the orientation and distribution of these components. In the composite materials, different reinforced types show different results.

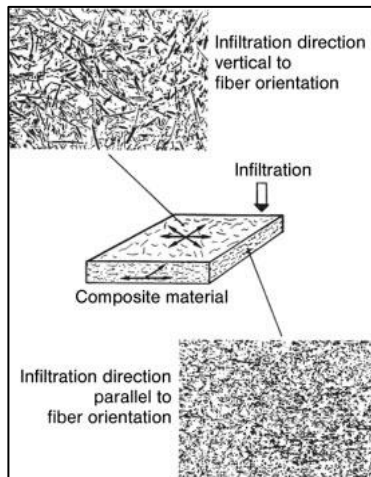
As a result of infiltration of fiber, the fiber/fiber contacts and non-reinforced areas are visible for multi-filament-strengthened composites. Even defects in the structure like pores, non-reinforced areas are recognizable, which have a more significant influence on the composite materials [27].



*Figure 11 Structure of a unidirectional endless fiber-reinforced aluminum composite material (transverse grinding) [27]*



*Figure 12 SiC monofilament/Ti composite material [27]*



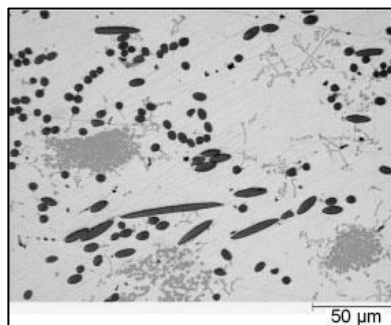
*Figure 13 Short-fiber reinforced light-alloy formation [27]*

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Above mentioned **Figure 13** Shows the formation of short-fiber reinforced composite materials. Due to the fiber molded padding production process, a planar-isotropic distribution of the short fibers originates. Generally, the direction of the infiltration is perpendicular to the material. Short fiber reinforcements tend to increase the strength by increasing the fiber content.

When it comes to particle reinforcement, the distribution of particles is based upon the processing methods used. In pressure die-cast materials, the particle distribution is more efficient, and even a good outcome can be obtained after the extrusion of the material. In gravity die casting, due to solidification conditions, non-reinforced areas can be visible. In all powder manufactured composite materials, the extreme particle homogeneous distribution is visible after the extrusion of powder materials.

Sometimes, the combination of fibers and particles for the formation of hybrid-reinforced composite material is possible. Adding the particle into the light metal composites increases the hardness, young's modulus, tensile strength, wear resistance, and these kinds of improvements depend upon the processing method and particle content. The content of the particle increased over 40% which are obtained from the extrusion along with fine-grain matrix structure with increase in strength and young's modulus. But, the fracture toughness and elongation are not good; however, these results are better than when compared with the cast materials [27].



*Figure 14 Hybrid reinforced light metal composite material structure [27]*

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### **2.7.3 Metal Matrix Composites Applications**

MMC replaced other materials like polymer matrix composites, unreinforced metals in most applications for different purposes. The substitution mainly increases strength, wear resistance, modulus, and decrease in density, thermal expansion, and cost. MMCs are grouped based upon the field of the market area. The primary industries that use MMCs widely are automotive, aerospace, electronic packaging, and commercial products. The aerospace industry is more focused on MMCs due to the weight reduction factor [28].

Properties like high young's modulus, specific strength, temperature resistance, and low thermal expansion are concentrated in the aviation industry, which is better with MMCs when compared with polymers [27].

### **2.7.4 Determination of MMCs damage:**

Over the recent years, one of the commonly used methods for analyzing the MMCs damage is scanning electron microscope (SEM), which helps to visualize the damage and quantitative data. However, this method only provides the information damage on the surface, whereas the inner surface can be different.

The development of the damage can be determined in more detail by the microtomography that helps to view the sample's microstructure in a non-destructive way. The main advantage of this technique is that due to the non-destructive way, it can find the features of the inner surface of the material. Another technique to analyze the MMCs damage is elastic stiffness measurement of a sample obtained by partial unloading in mechanical tests. Thus, the stiffness reduction is combined with the reinforcement of deformation-induced damage. Even Kouzeli et al., a researcher, has investigated the relationship between the reduction in weight and the damage caused by deformation [27].

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## **2.7. Hadfield steel – Matrix**

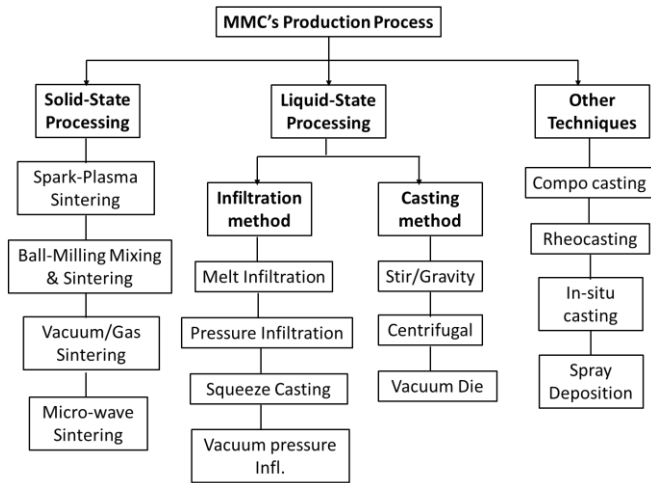
In MMCs, a matrix material can act as a binder to provide sufficiently strong bonding and decrease abrasive wear with the ceramic particles. Suppose when a load is applied, then the matrix shifts the load to reinforce it by improving the load capacity of the composite material. High manganese steels contain a high impact, wear resistance, good ductility, and toughness, which resist the propagation of cracks [13]

### **Reinforcement – Tungsten carbide**

For the past several years, ceramic particles served as reinforced material for steel matrices and are commonly used for wear resistance components. Carbides consist of good mechanical properties such as high wettability by molten metals, high hardness. Tungsten carbide (WC) is one type of carbide containing hot hardness and is mainly used as reinforced material for iron matrix composites. The disadvantage of the WC is low heat formation (easy to dissolve by molten metals) [16]. Tungsten carbide is an ideal combination of hardness, strength, toughness, and good chemical stability. These properties all together make it ideal for application in fields like metal machining, mining excavations, and many other industrial applications. Tungsten carbide exhibits a hexagonal structure, a high melting temperature of 2600 C, and high electric and thermal conductivity values.

## **2.8 Manufacturing methods**

There are numerous processes used to produce MMCs. Generally, all the methods are classified based upon the temperature of the metal matrix during the operation into three types which are Liquid state processing, Solid-state processing, and other processing like deposition techniques, and two-phase (solid-liquid) processes [13] [17] [21].



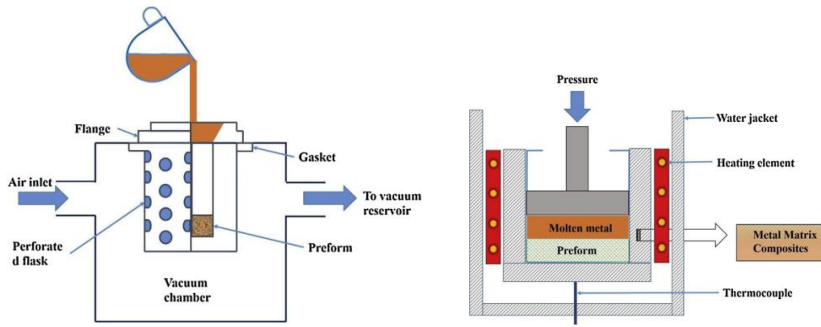
*Figure 15 Various production process for MMCs [21]*

### 2.8.1 Liquid state processing

In MMC's, liquid state processing techniques are eye-catching to various industries because of their economic and straightforward features, widely used in various industries. It includes either infiltration methods of molten metal into preforms or casting methods. Infiltration methods consist of gas pressure, vacuum pressure, melt, high pressure centrifugal forming a distributed phase into a molten matrix metal followed by the solidification phase[21]. A good interface bonding between dispersed and liquid matrix phases must be formed to provide high mechanical properties. Liquid state fabrication of MMC: Stir casting, Squeeze casting infiltration or pressure die infiltration, and gas pressure infiltration [17].

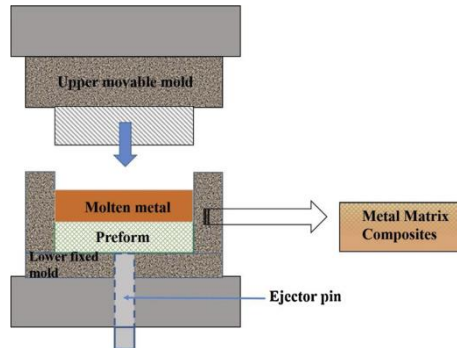
#### **Infiltration methods**

The permeation of molten metal onto a preform by the infiltration process. This can be achieved either by pressure less infiltration or by pressure infiltration. In pressure less infiltration, reinforcements are placed in the die, and then the molten alloy is poured on it and later solidified without any external pressure. External pressure is applied through or directly as an inert gas, vacuum pressure, centrifugal force, and squeeze infiltration [21].



*Figure 16 Melt infiltration (left) and Pressure infiltration (right) [21]*

In manufacturing processes, casting is a primary technique capable of producing complex shapes for different materials. In this process, molten metal is poured into a cavity and allowed to solidify to form a predefined shape. Generally, obtained products have high compressive strength. This technique is cheapest when compared to the other manufacturing processes [21].



*Figure 17 Squeeze casting infiltration [21]*

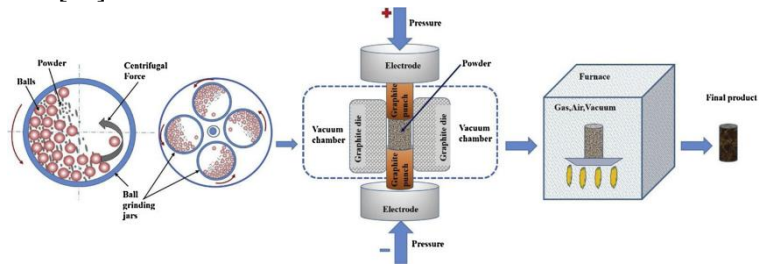
## 2.8.2 Solid-state processing

In this process, MMC is produced because of the interface bonding between matrix metal and distributed phase due to mutual diffusion occurring between them in solid state at high temperatures and pressures [21].

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## Vacuum/Gas sintering

It is one of the commonly used sintering methods because of its good controllability and complex features. It is similar to the HEBMS; the only difference between these methods is vacuum or gas sintering after compacting. Gao et al. observed that when there is an increase in sintering temperature, the alloys have less porosity, homogeneous microstructure, and high hardness [21].



*Figure 18 Vacuum/gas sintering [21]*

**Other processes:** Apart of liquid and solid-state processing techniques for producing MMCs, there are other processes such as semi-solid processes, compo-casting, spray deposition etc.,



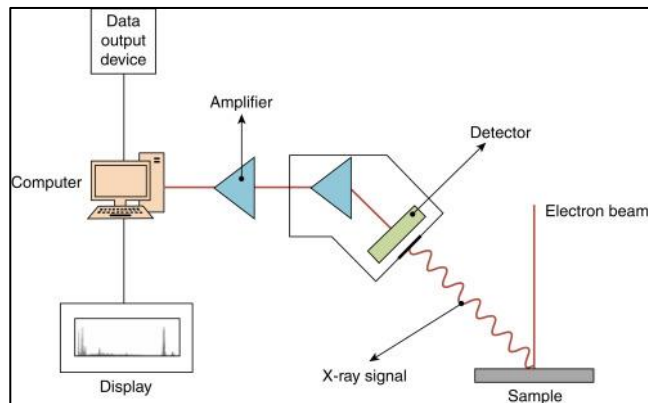
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### 3. EQUIPMENTS USED

The equipment's used in this thesis are:

1. Sample Preparation
2. Optical microscope (Alicona)
3. Scanning Electron Microscope (SEM)
4. Energy Dispersive X-Ray Spectroscopy (EDS)
5. Hardness Tester
6. Lab wear tester

#### 3.1 Energy Dispersive X-Ray Spectroscopy (EDS)



*Figure 19 Demonstration of working of EDS [29]*

Energy Dispersive X-Ray Spectroscopy (EDS or EDX) is used to perform chemical microanalysis in conjunction with scanning electron microscopy (SEM). This technique detects x-rays emitted by the specimen when bombardment by an electron beam. The primary purpose is to characterize the elemental composition of the analyzed volume. The unique feature of this is to measure particle size up to 1 micron.

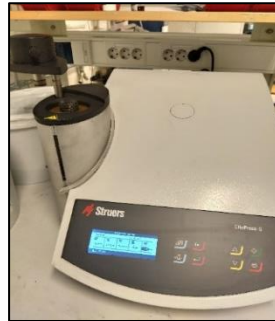
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## 3.2 Sample Preparation

Sample preparation involves different steps to prepare the sample in a better way to perform analysis. The heat-treated samples were cut to the desired size to prepare them for SEM analysis. The cutting process was carried out using the Struers Cutting machine using SiC blades. The cut samples are hot mounted (epoxy) using Struers Cito Press 5. The hot mounted samples were ground to remove the epoxy remains and flatten the surface. The ground surface is then polished to eliminate the scratches formed on the surface to provide the best surface for analysis. The grinding and polishing were performed using Struers Tegramin-30.



(a)

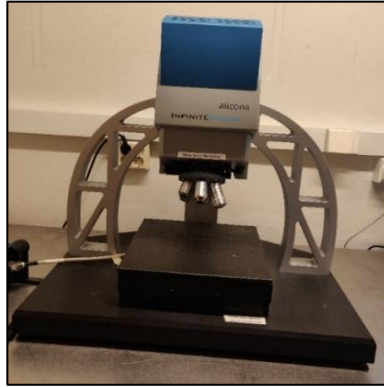


(b)

*Figure 20 (a) Disk Cutting Machine, (b) Hot Mounting Machine at LTH*

## 3.3 Alicona

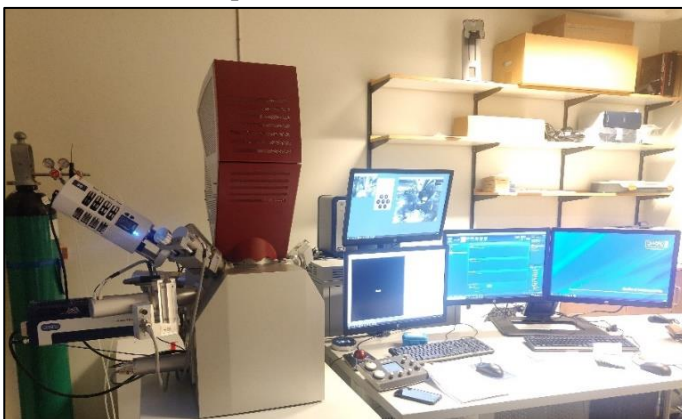
Alicona is an optical 3D measurement system that is fast, flexible, and provides accurate results. It is mainly used for measuring the surface roughness and dimensional accuracy of the objects. The current thesis uses alicona for optical images and particle size measurement. It is a user-friendly system that can capture from 2.5X to 100X magnification with high resolution.



*Figure 21 Infinite Focus Alicona at LTH*

### **3.4 Scanning Electron Microscopy**

Scanning Electron Microscopy [SEM] is used to identify imperfections, cracks, or some foreign material inclusions on a surface. It is also used to study the microstructure along with finding the shape and sizes of smaller particles. In SEM, the electron beam is reflected on the surface or even ionized atoms within the sample by liberating electrons instead of passing through the specimen. These electrons that are backscattered can serve as signals to build up the final image. SEM is used to attain a high-definition image of surface microstructure and provides a clear picture of the distribution of different samples' chemical elements.

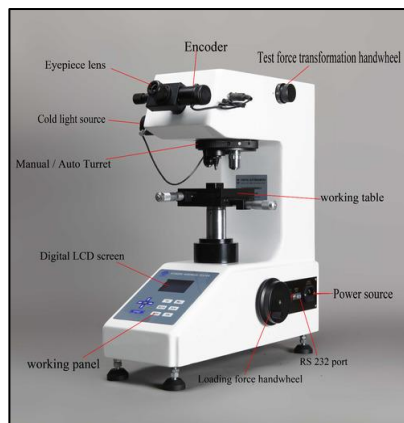


*Figure 22 Demonstration of working of SEM at geocentrum I*

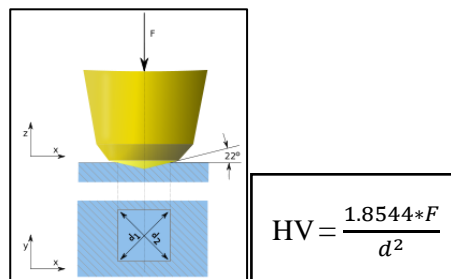
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### 3.5 Hardness Tester

This equipment is used for testing the hardness of the samples named Vickers Hardness testing Machine. The principle behind this is that hardness is measured to identify a material's ability to resist plastic deformation. This machine can be used on all types of metals, and it is the most comprehensive scale compared to the remaining hardness tests. The hardness unit can be represented in Vickers Pyramid Number (HV) or Diamond Pyramid Hardness (DPH). Hardness is the ratio of loading force to an actual contact area shown in the image below.



*Figure 23 Vickers Hardness testing machine [30]*



*Figure 24 Schematic representation of Vickers Hardness Test [20]*

### 3.6 Lab Wear test

The heat-treated plates are machined and prepared for the wear test.

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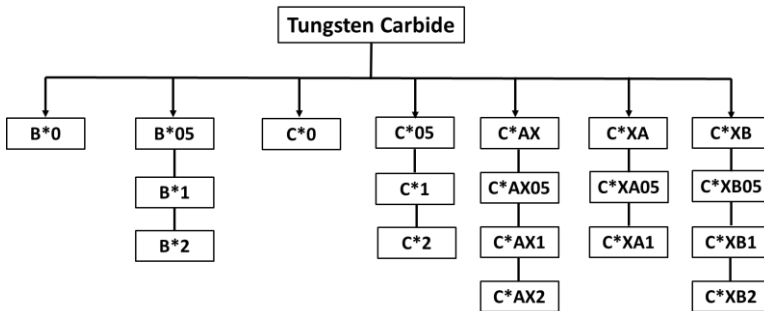
## 4. EXPERIMENTAL PROCEDURE

The thesis aims to develop a material consisting of Tungsten carbide that act as a reinforcement agent with Hadfield steel. Based on the literature review, identification of elements is crucial, followed by casting manganese steel and later analyzing the results.

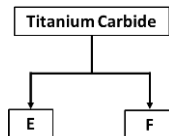
Using the literature review as a guide, we chose initial raw materials to be used in the MMCs. The experimental procedure steps involves powder processing, casting, sample preparation and characterization techniques.

### 4.1 Material data

The below table represents the data of the selected elements. Along with WC, we have also worked on Titanium carbide as a supplement to our work. As part of the project, WC was analyzed and compared.



*Figure 25 Selected samples-WC*



*Figure 26 Selected samples – TiC*

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Percentage	X	X	AX	XA	XB
xx	B*0	C*0	-	-	-
xx	-	-	C*AX	C*XA	C*XB
xx	B*05	C*05	C*AX05	C*XA05	C*XB05
xx	B*1	C*1	C*AX1	C*XA1	C*XB1
xx	B*2	C*2	C*AX2	-	C*XB2
xx	E	-	-	-	-
xx	F	-	-	-	-

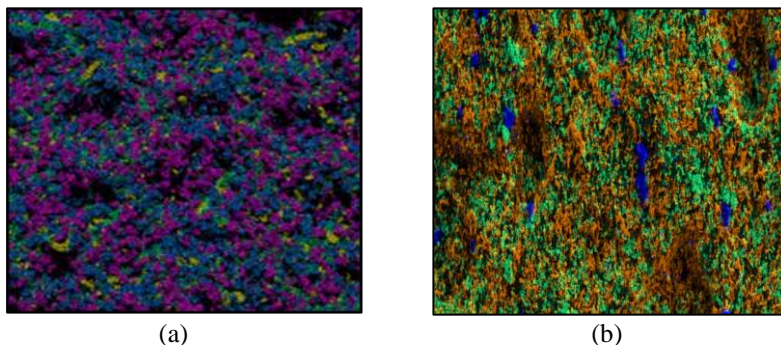
*Table 1 Sample naming data*

## 4.2 Materials mixing

Mixing was done for all the selected powders.

## 4.3 EDS Analysis

To differentiate the materials in analyzing, color coding was used. Each sample is analyzed at two different sites for accurate results. After examination, all samples have shown good mixing properties, but some agglomerations are found. An adjustment of mixing procedure has resulted in good mixing and no agglomerations were found.



*Figure 27 EDS analysis of powders showing a) good mixing b) poor mixing*

## 4.4 Pressing

All samples are pressed at required pressure. And Maximum green density obtained was 70%.

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$$\text{Green density} = \frac{\text{weight of the part}}{\text{geometric volume of the part}}$$

## 4.5 Casting

Casting is carried out using molten Hadfield steel. All the selected 21 powders are divided into two sets. Samples are placed in the mold before casting.

During visual inspection, one plate was found to be defective after casting and heat treatment. Due to the availability of another set of plates (B\*0 & C\*1) from a previous master thesis student two plates B\*0 and C\*1 were considered directly for wear test. It was evident from previous thesis that 2 of the plates selected showed significant results, but the wear test was not conducted due to time constraints. So, after visual inspection we have ruled out plate due to bad casting and also eliminated the two plates (B\*0, C\*1) for wear test. The remaining plates are subjected to further analysis.

## 4.6 Sample Preparation

### 4.6.1 Sample Cutting

As part of sample preparation, the first step is to prepare plates according to the required dimensions. There are altogether eight samples that were cut into small samples for characterization analysis.

In this process, two types of cutting machines were used for efficient usage of time. Initially, Wire Electrical Discharge Machine (WEDM) was used to cut larger plates, and then Struers cutting machines were used to cut small samples suitable for material characterization

### 4.6.2 Hot Mounting

The cut samples are embedded with epoxy. Struers Cito Press 5 was used for hot mounting. For the analysis, the best surface should face the bottom of the mold. The amount of resin and operational parameters are determined by the mold's size, in accordance with the standards. In this case, a 30mm diameter mold was used, since the sample's average size was 19mm\*17mm\*10mm

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(L\*B\*H). Polyfast resin was used in this process, which is recommended to be used for SEM analysis.

For this application, as per struers standards, 20ml poly fast was advised. But, considering the thickness of the specimen, 18ml used. This setting was heated up to 180 degrees with a pressure of 325 bars for 3.5 minutes as per the struers standards, and an intense cooling process took place for about 1.5 minutes.



*Figure 28 sample embedded in epoxy*

For the identification of samples, a diamond pencil is used to mark all the samples and proceeds further for grinding and polishing.

#### **4.6.3 Grinding & Polishing**

Struers Tegramin-30 was used for grinding and polishing. It is crucial to have a fine finish to get better results in the SEM analysis. A grinding process includes the initial four steps, starting with MD Piano 200, which has diamond crystals smaller than 9 microns, and ending with MD Piano 2000, which has finer diamond crystals. During grinding, the force on the specimen is set to 25N and the speed is constant at 300 rpm. After each step, a microscope was used to inspect the surface for improvements. It might be necessary to repeat some steps in order to achieve accurate results.

Step	Surface	Suspension	Lubricant	Force (N)	Rotation Speed (rpm)	Time (min)
1	MDPiano 220	-	Water	25	300	5
2	MDPiano 500	-	Water	25	300	5
3	MDPiano 1200	-	Water	30	300	5
4	MDPiano 2000	-	Water	30	300	5
5	MD Dac	DiaDuo 2-3 $\mu\text{m}$	-	5	180	6
6	MD nap	DiaDuo 1-2 $\mu\text{m}$	-	10	180	6
7	MD-Chem	OP-U Non-dry	-	10	200	6

*Table 2 Sequence of Grinding and Polishing Steps*



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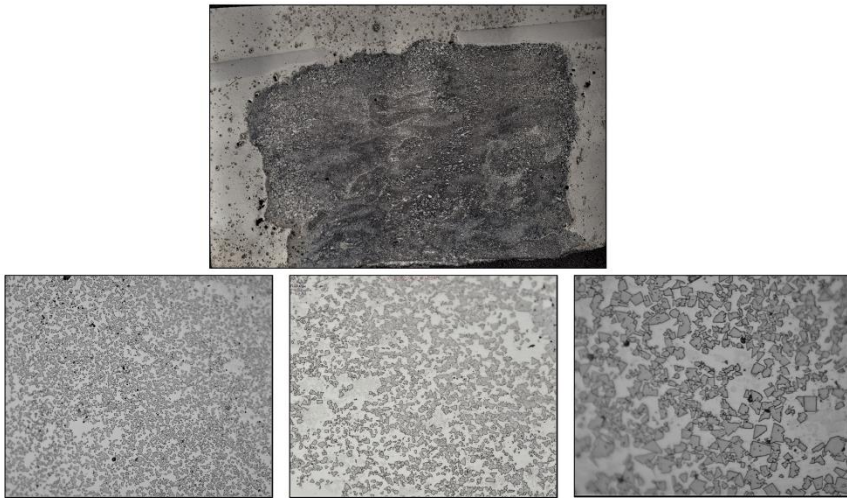
A mirror finish is achieved after polishing the samples. As a final step, the surface has been covered with ethanol to prevent corrosion and is packed.

## 4.7 Characterization Techniques

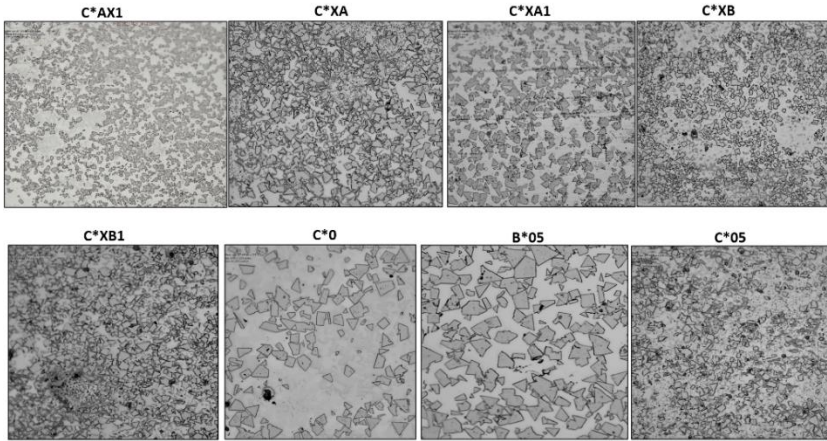
Material characterization is the most crucial step. In this thesis, the optical microscope (Alicona) and Scanning Electron Microscope (SEM) were used to analyze the microstructure of the all MMC samples.

### 4.7.1 Optical Microscope - Alicona

A high-performance optical microscope named Alicona was used to capture the images of the surface microstructure in the best possible way. The images are taken at the same magnification of all the samples for comparison. For an overview of the whole sample, the data has been captured at various magnifications. The particle size was also measured for further analysis.



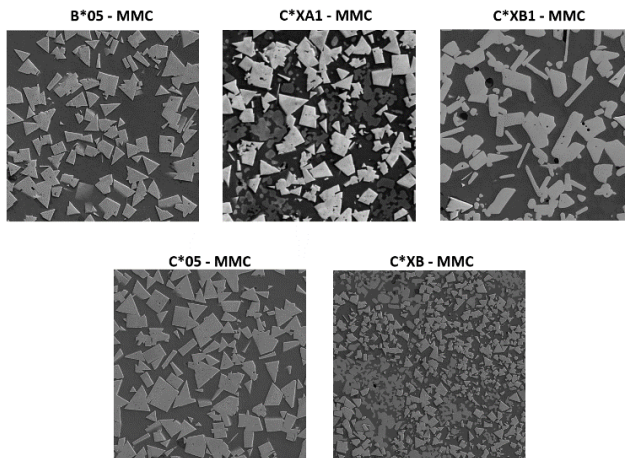
*Figure 29 Alicona images of one of the samples*



*Figure 30 Alicona images of all samples reinforced region*

#### 4.7.2 Scanning Electron Microscope (SEM)

Scanning electron microscopes can produce images at higher resolution. Only five of the eight samples were subjected to SEM analysis due to a lack of SEM equipment access and time constrain. Based on the literature review, heat treatment, and Alicona images, these five samples are considered. The below images are taken at the same magnification for identification of the particle size and distribution.



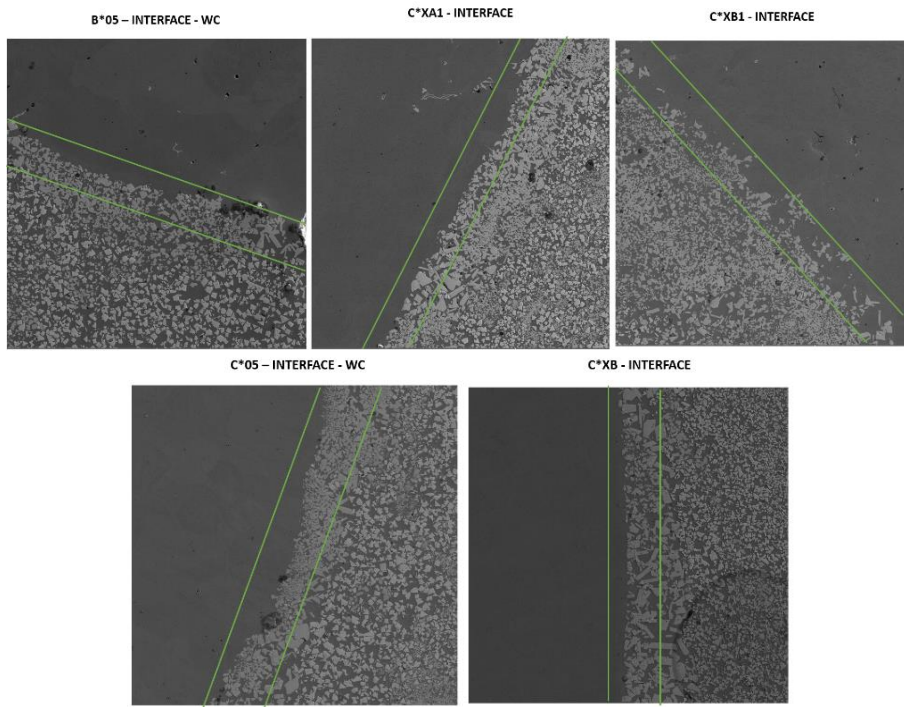
*Figure 31 SEM images at reinforced region*

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The WC particles are uniformly distributed throughout the composite region and firmly embedded with the matrix in all five samples. No cracks, pores, or defects are visible in the samples, which means all the samples succeeded in forming a good metallurgical bond. In all samples, the interface region shows a strong connection between the matrix and reinforcement.

WC particles mostly have prismatic shapes with sharp edges and no pores. The platelets shape in the particles can be found in some areas which enhance the toughness of metal matrix composites.

Interfacial bonding was affected depending on the initial raw material percent. In previous experiments, cracks were seen on the interface layer as the initial raw material varies.

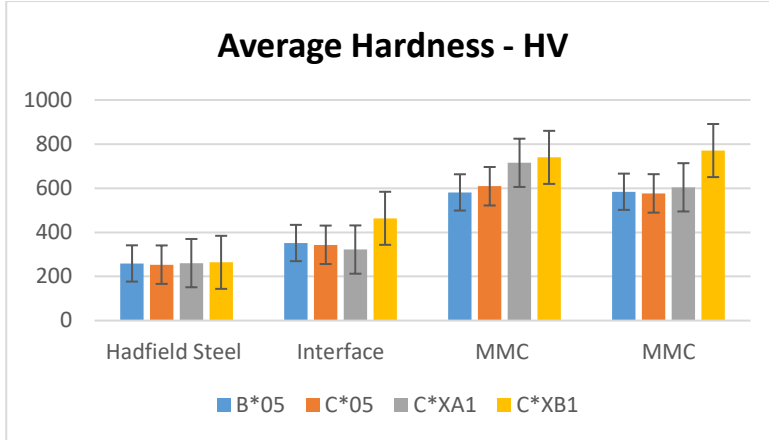


*Figure 32 SEM images at interface between matrix and reinforced region*

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### 4.7.3 Hardness Tester

In this thesis, Vickers hardness tester is used. A load of 1Kgf applied on the surface of all the samples. The measurement was taken on all the different phases (i.e., Hadfield steel, interface, and composite), with at least four indents on each phase. Hardness measurements were conducted horizontally for ease of identification.



*Figure 33 Hardness values graph*

## 5. RESULTS AND DISCUSSION

### 5.1 Powder processing: Mixing, pressing and casting

After powder mixing, EDS was used to evaluate the mixing. The powder mixture consists of agglomerations because of the improper mixing procedure, it is clear that a homogeneous mixture requires right mixing parameters and a proper mixing equipment for adequate results. All the mixed powders were pressed.

All samples have resulted in a good shape and crack-free samples were obtained. Out of all, one sample was broken because it was mishandled during pressing. The broken sample was not considered for further steps whereas the remaining samples will proceed for casting. The average green density obtained was 70%.

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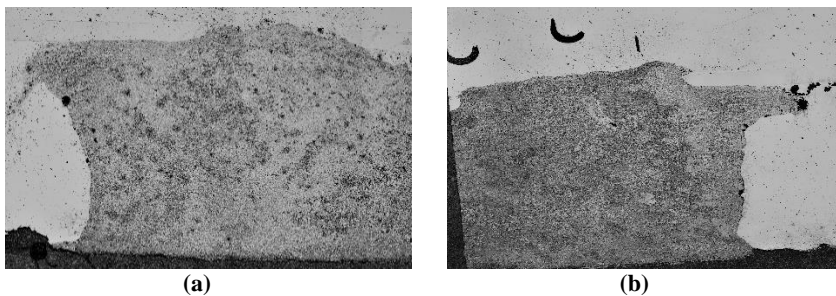
The casting process was successfully performed. On visual inspection, one plate has defects, while another has fewer defects and remaining all samples are in good state.

## 5.2 Characterization techniques

### 5.2.1 B\*05 & C\*05

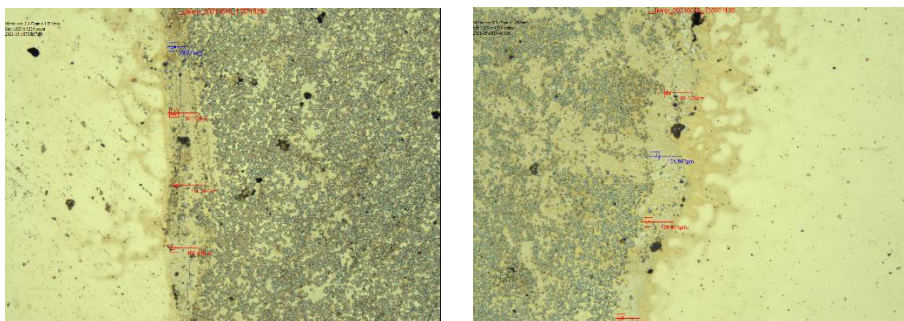
#### Optical microscope: Alicona

The microstructure of B\*05, C\*05 at different magnifications shown in the **Figure 34**. In Alicona, the particle distribution of WC particles observed similar to SEM and the particle size was measured.



*Figure 34 Alicona Images at lower magnifications (a) B\*05 (b) C\*05*

In the above magnification, it is clear that the matrix and reinforce regions are connected by a smooth, inline interface.



*Figure 35 Interface layer between Matrix and Composite of B\*05 (Left) & C\*05 (Right)*

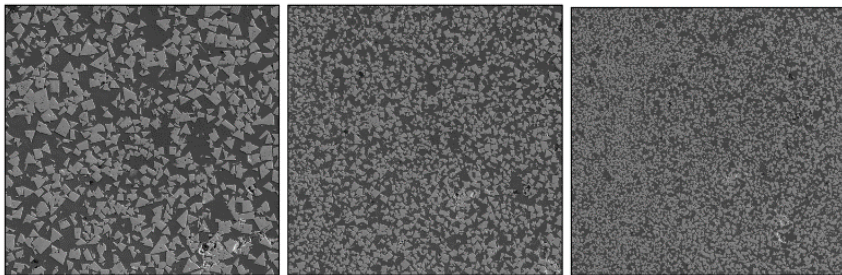
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In this case, the metallurgical bond of B\*05 and C\*05 with Hadfield steel was caused by the solvability nature of ceramic materials in high-temperature steel material.

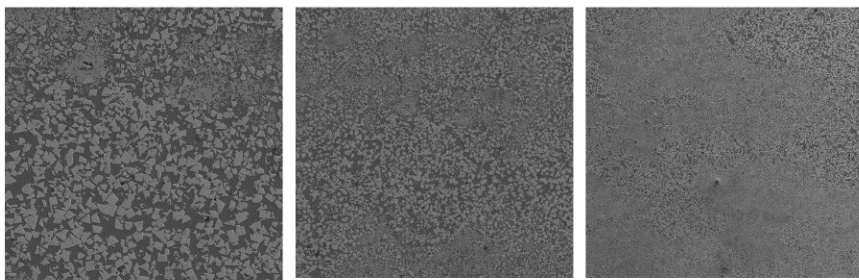
Even the thickness of the interface layer was investigated systematically by using Alicona. It was observed that B\*05 interface layer had an average thickness of 93.54 microns. C\*05 interface layer had an average thickness of 138.5 microns. The metallurgical bond of C\*05 with Hadfield steel (matrix) was more than the B\*05.

### Scanning Electron Microscope (SEM)

The overall view of B\*05, C\*05 was taken at the same magnification shown in the below figures.



*Figure 36 SEM image of B\*05 reinforced region at different mag.*



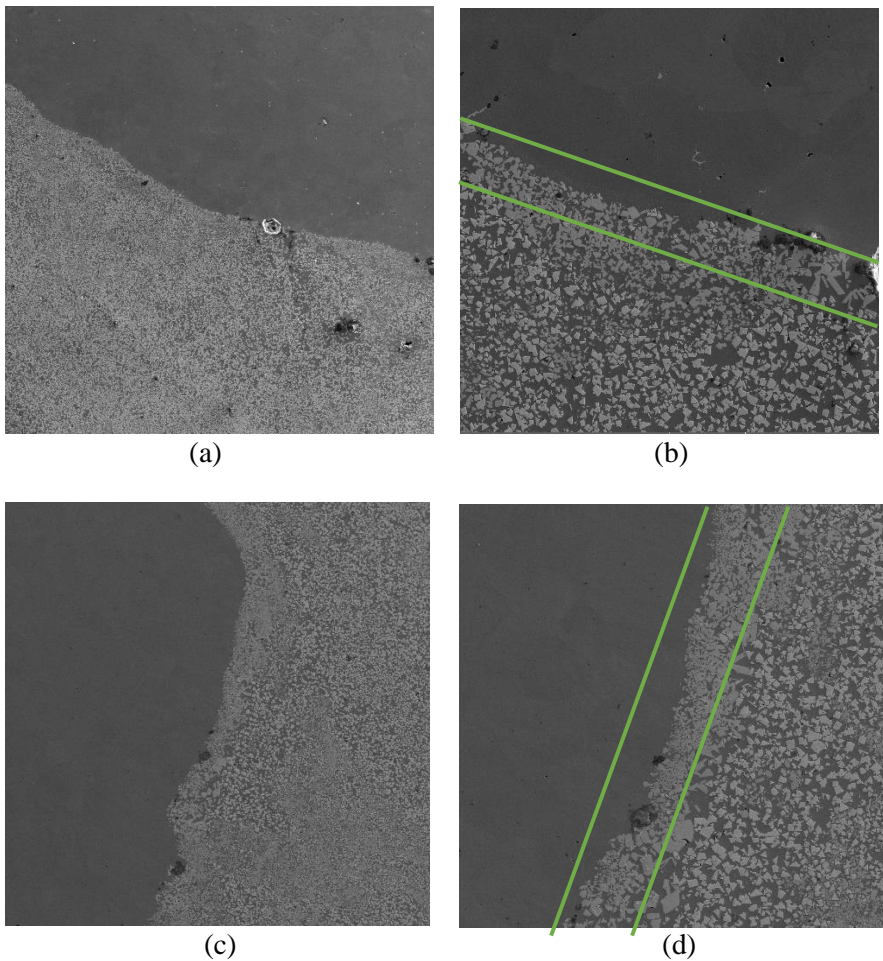
*Figure 37 SEM image of C\*05 reinforced region at different mag.*

There are no visible pores or cracks in the reinforcement region, which means both samples have a good metallurgical bond within the composite phase.

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In the reinforced area, WC particles have triangular shape, which means that a strong bond between ceramics and matrix was achieved. The average WC particle size of B\*05 varies with 3 – 13 microns approx. But in some regions, the particle size may differ. So, it is difficult to predict the exact average WC size through measuring at specific area.

The average particle size of C\*05 varies from 5-16 microns and the particle size was little bigger than B\*05. In the comparison with B\*05 and C\*05, WC was reinforced with Hadfield steel in both the samples. The particle size of the C\*05 is slightly higher than the B\*05.



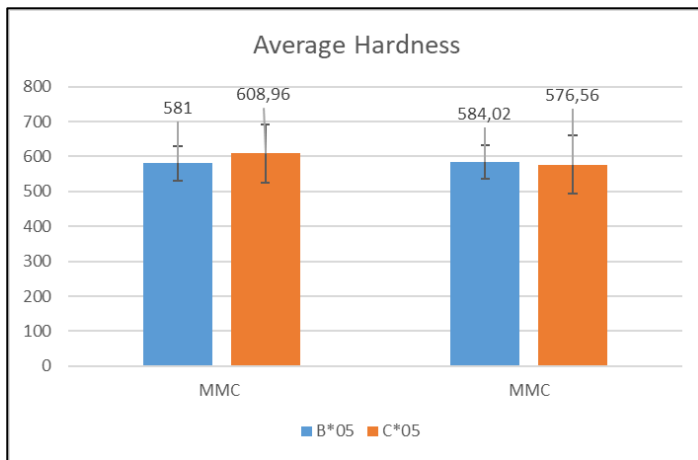
*Figure 38 SEM images of (a)&(b) B\*05 interface and (c)&(d) C\*05 interface*

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From **Figure 39** (a) & (c) It can be observed that the B\*05 and C\*05 have good interface with Hadfield steel without any cracks or pores in between. From **Figure 39** (b) & (d) the area between two lines indicates the interface layer discussed in the Alicona section about its thickness.

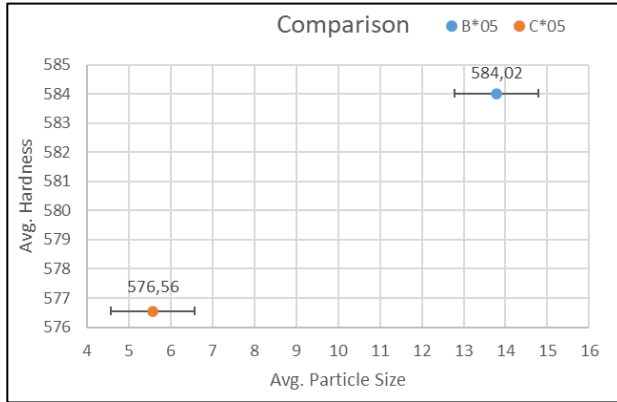
### Hardness

The hardness values of B\*05 and C\*05 are taken at four different locations (Hadfield steel, Interface line, Interface layer, MMC). The obtained values show an increase from matrix to MMC. The result shows that the B\*05 have avg. hardness of 584 HV and C\*05 has a hardness of 576.6 HV. The data have shown that the MMC at the interface layer has high hardness values. Both samples have a similar variation in hardness values.



*Figure 39 Comparison graph between B\*05 and C\*05*





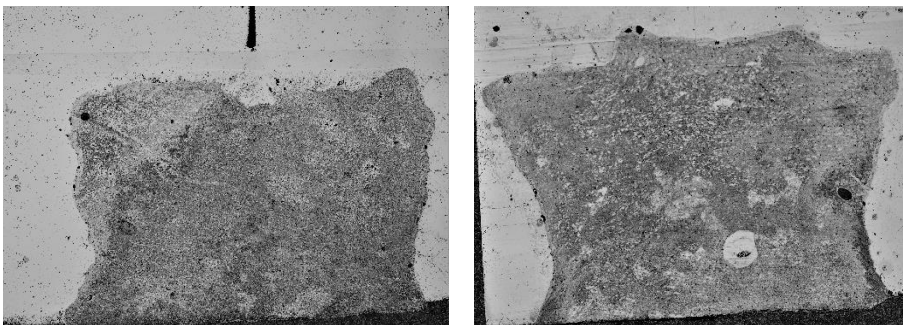
**Figure 40** Graph between Avg. Hardness Vs Avg. Particle Size of B\*05 & C\*05

The maximum hardness value of Hadfield steel is 261 HV, and the hardness value at the interface line between matrix and interface layer is 467.3 in C\*05 which higher than B\*05. In the MMC, the highest hardness value obtained for C\*05 is 747.1, and for B\*05 is 639.9 HV. Hardness values of B\*05 and C\*05 did not differ much, indicating that a durable wear resistance property was built and has a higher value than Hadfield steel.

### 5.2.2 C\*XA & C\*XA1

#### Optical Microscope: Alicona

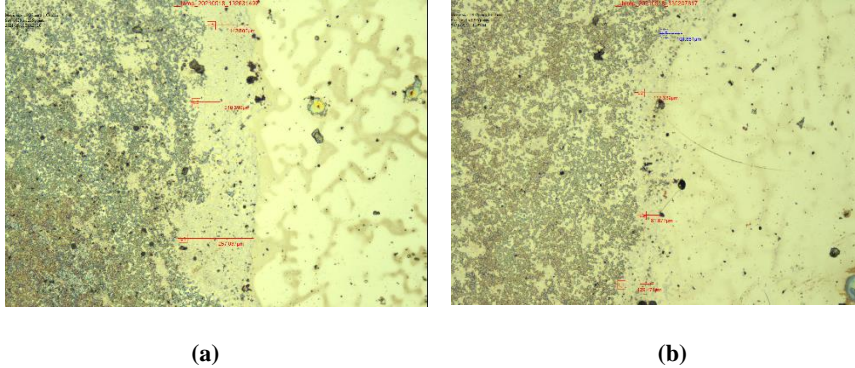
Overall view of the sample reinforced with the matrix was captured at a lower magnification in Alicona.



**Figure 41** Alicona images at a magnification (a) C\*XA (b) C\*XA1

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With Alicona images, the sample can be seen. C\*XA and C\*XA1 have a flawless interface with the matrix, without any defects or cracks.

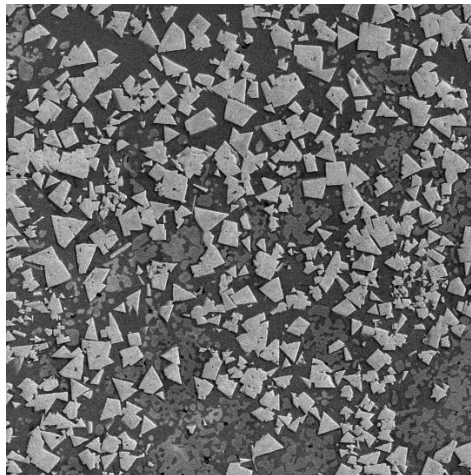


*Figure 42 Interface layer between matrix and composite (a) C\*XA & (b) C\*XA1*

The average thickness of the C\*XA interface layer is 232 microns, where C\*XA1 is 292 microns. As a result of a change in raw material percent, a thicker interface layer has formed between the matrix and reinforcement in the sample. This meant that the matrix and reinforcement were more securely bonded to one another in C\*XA1.

### **Scanning Electron Microscope (SEM)**

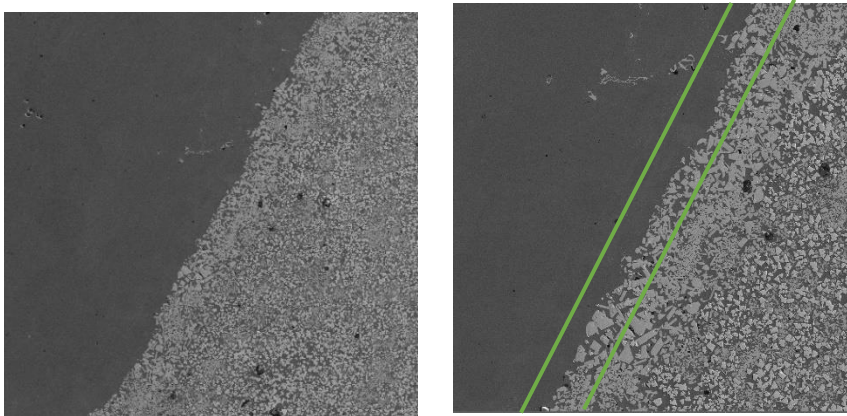
C\*XA1 reinforced region was captured at higher magnification through SEM.



*Figure 43 SEM image of C\*XA1 reinforced region.*

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C\*XA1 distributed uniformly throughout the region without any cracks or pores. A SEM analysis has not been performed on C\*XA; instead, a comparison has been made based on its optical microscope images. The particle size of C\*XA1 varies between 8-18 microns at a particular scanned location. Approximately 5-13 microns of particle size can be found in C\*XA, which means higher initial raw material percent have larger particle size than with lower percent. The particle size of C\*XA1 was significantly larger than B\*05 and C\*05.

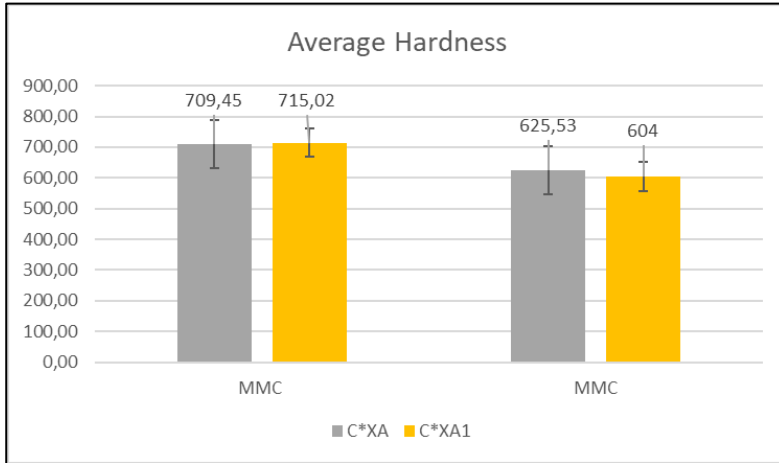


*Figure 44 SEM images of C\*XA1 interface*

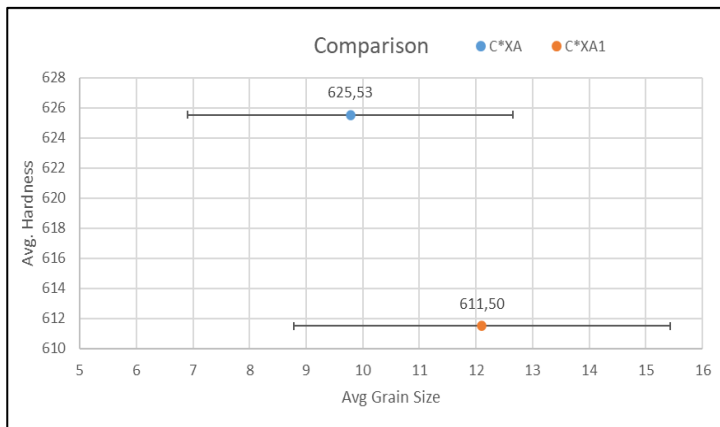
In the Alicona section, C\*XA1 was shown to have an excellent interface bond to the metal matrix without cracks or pores. There are very few intergranular pores in the region. It was perfectly aligned with the matrix by forming an interface layer in between. In the context of particle size analysis, for a higher raw material content, particle size increases, thus XA has less initial raw material resulted in a smaller particle size, whereas XA1 contains a higher raw material content, thus the particle size increases.

### **Hardness**

A very slight variation can be seen in the average hardness values of C\*XA and C\*XA1. Both the samples have high hardness at the interface region. However, the matrix and composite formed a strong bond. Particles with smaller sizes have higher hardness values, while particles with larger sizes have a lower hardness value.



**Figure 45 Comparison graph between C\*XA and C\*XA1**



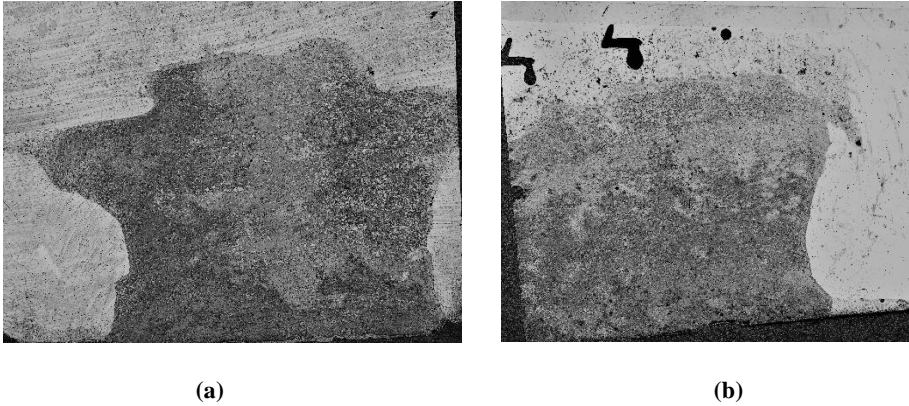
**Figure 46 Graph between Avg. Hardness Vs Avg. Particle Size of C\*XA & C\*XA1**

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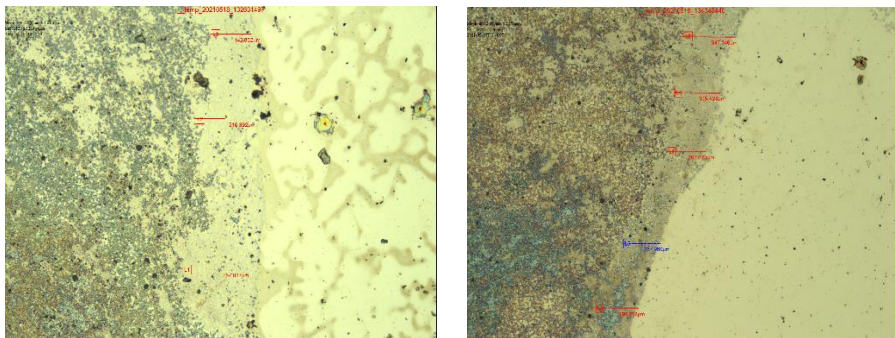
### 5.2.3 C\*XB and C\*XB1

#### Optical Microscope: Alicona

Overall view of the C\*XB and C\*XB1 was shown at a magnification in Alicona.



*Figure 47 Alicona Images of (a) C\*XB and (b) C\*XB1*

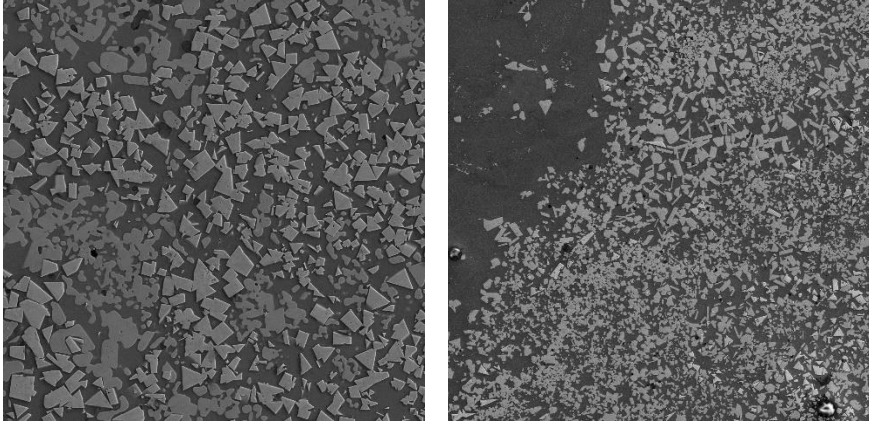


*Figure 48 Interface layer thickness of C\*XB (Left) and C\*XB1 (Right)*

The thickness of the interface layer formed for C\*XB was 158 microns whereas, for C\*XB1, it was 293 microns which is more superior than with the initial raw materials with low concentration. So, if the raw materials percentage increases, then the thickness of the interface layer increased.

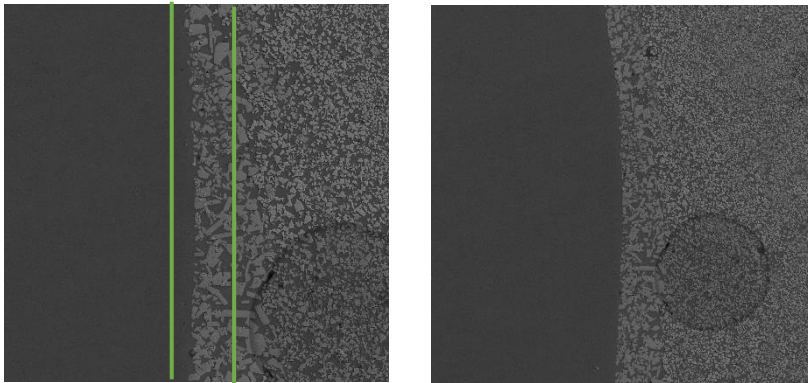
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## Scanning Electron Microscope (SEM)



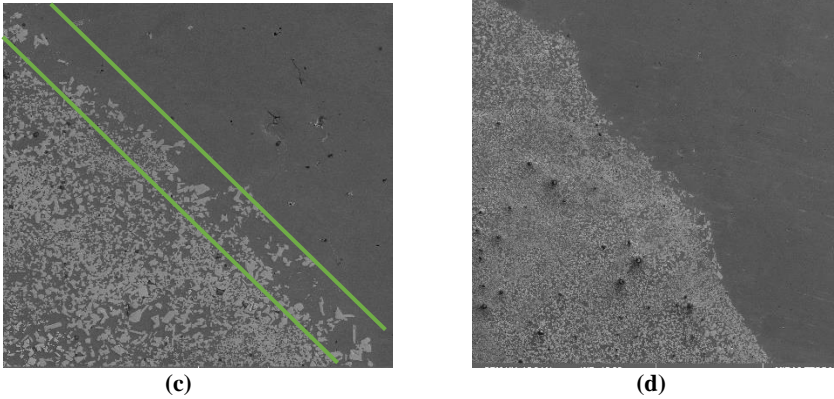
*Figure 49 SEM image at reinforce region of C\*XB (left) & C\*XB1 (right)*

The particle sizes are measured from the Alicona, showing that the size of C\*XB was 2-6 microns approx. And the C\*XB1 particle size varies from 5-12 microns approx., which means the initial raw materials percentage increase has led to an increase in the particle size at this particle location. Data provided only pertains to the scanned area and expected to be similar overall the sample.



(a)

(b)

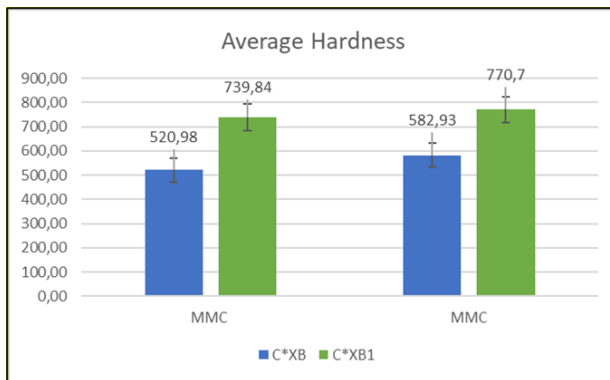


*Figure 50 SEM images at interface layer of (a)&(b) C\*XB and (c)&(d) C\*XB1 interface*

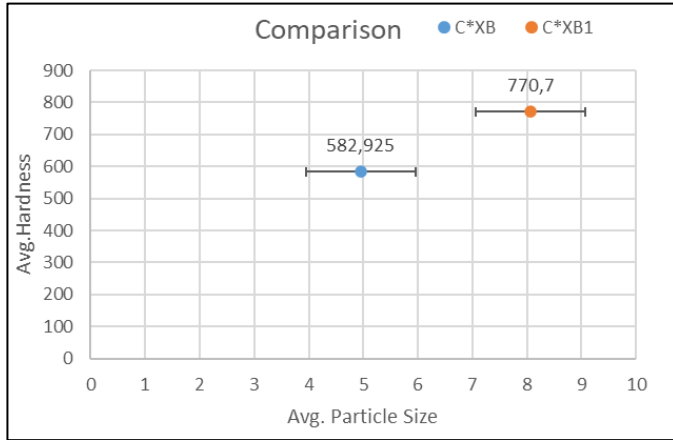
The interface layer of both samples has a solid metallurgical bond between matrix and reinforcement without any defects.

### Hardness

From the obtained data, both samples showed increases in hardness as they progressed from matrix to composite. Increases in initial raw materials results in increased particle size, which in turn increases the hardness values, so the C\*XB1 has high hardness values.



*Figure 51 Comparison of Average hardness values for C\*XB and C\*XB1*



*Figure 52 Graph between Avg. Hardness Vs Avg. Particle Size of C\*XB & C\*XB1*



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## 5.3 Hardness

The hardness value was shown only for four samples in four different locations in this section. Material and location affect the hardness values. These graphs demonstrate an increase in average hardness values between 250HV and 770HV, indicating a change in strength due to the cement-reinforced steel matrix. Hadfield steel has the lowest hardness, which is 260HV, while MMC – Inside, which has indents in the reinforced region, has the highest hardness of 770HV (C\*XB1).



*Figure 53 Graphs of average hardness values at different locations (Hadfield steel, Interface, MMC at interface and MMC-inside)*

The hardness of each sample varies with the particle size. The larger the particle size the lower hardness of the material and vice versa. It is necessary to adjust the particle size according to the initial raw materials, so that the hardness can be changed for different applications.

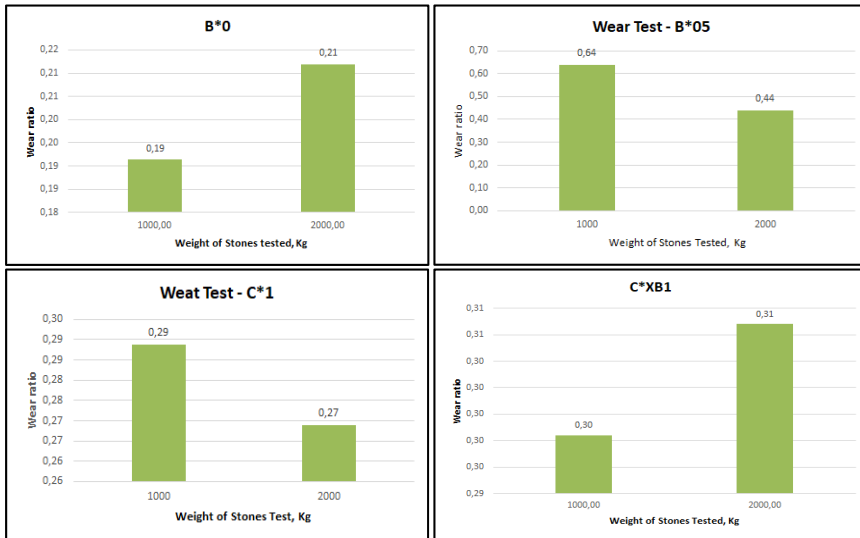
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## 6. Wear Test

Wear test was performed on four sets of plates: B\*0, C\*1, B\*05, and C\*XB1. There are two main reasons for selecting four sets of plates; obtained results from the SEM, Hardness evaluation, and due to time constraint. In spite of this, results from this wear test showed that MMC has a significant increase in wear resistance.

Specially for the wear test, the selected plates are mixed and cast again. Dalby mix stones are used as a crushing material in the Lab wear test. Stationary plates have a higher wear ratio than moving plates due to continuous contact at a fixed location. So, the wear ratio depends on the movements of the plates and on work hardening property.

A significant decrease in wear ratio has been observed in two sets of plates (C\*1 and B\*05), and two more sets of plates after two tons exhibit slightly higher wear ratios than after one ton; this is because of two parameters:



*Figure 54 Graphical representation of wear ratio of all the samples*

Since the impact is less (2 tons), the work hardenability has not been achieved effectively. As a result, the matrix regions are more susceptible to wear than the reinforced areas, which means ceramic composites act as a wear resistance against impacts.

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## 7. Conclusion

Tungsten Carbide (WC) particles were reinforced successfully with Hadfield steel. Optical microscope (Alicona), Scanning Electron Microscope (SEM), Vickers hardness tester, and a wear test were used to examine the mechanical properties and morphology of the WC reinforcement.

The conclusions are:

- A microstructural analysis has shown excellent results for WC particles in Hadfield steel. Neither pores nor cracks have been found.
- All MMC samples exhibit a strong metallurgical bond between the matrix and reinforced regions.
- The composite region has a gradual increase in hardness compared to the matrix region (base alloy).
- A notable improvement in the wear performance with MMC compared to the matrix.

A detailed interpretation of the microstructure of WC particles was achieved using SEM and SEM-EDS. The results indicate that tungsten carbide was homogeneously distributed and has good bonding properties in the interface region for all the samples. It can be observed that the particle size varies with changes in the initial raw materials. Particle sizes have a strong effect on the hardness results. Hardness increases with increasing the particle sizes, which enhances wear resistance.

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## **8. Future work**

- 1) To conduct more SEM analysis in order to investigate more the microstructure features.
- 2) To perform X-Ray Diffraction (XRD) analysis on the samples in order to evaluate the different phases present in the samples.
- 3) To do more wear experiments in order to gather more statistical data and to be able to draw more conclusions.
- 4) To investigate more materials.

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