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**Option : Material Engineering**

***Thème***

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***Elaboration and Characterisation of In-Situ  
Metal Matrix Composites***

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# Acknowledgment

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## **ABSTRACT :**

In this study, we report on the microstructure and tribological characterization of in-situ *modified M50 steel* synthesized by in-situ reaction of Maxthal211 ( $\text{Ti}_2\text{AlC-Ti}_3\text{AlC}_2$ , MAX phase). The composite was elaborated from 5, 10, 15 and 20wt% of the MAX phase which fully reacted with M50 matrix at 1050 °C sintering temperature for 4 h; the MAX phase decomposed into TiC, and the released Al and Ti atoms diffused in M50 matrix.

Optical Microscopy was used to study the different microstructures and worn surface characteristics. Dry sliding properties of the composite under different normal loads were studied using a ball-on-disc Tribometer. Addition of 20 wt% MAX phase procured the highest hardness (63.083 HV5) which is about two times higher than that of pure modified M50 steel. Whereas all the reinforced composites exhibited better wear resistance.

## **Keywords:**

Composite, max phase, dry sliding, optical microscopy, sintering, tribometer, reinforcement.

## **Résumé :**

Dans cette étude, nous rapportons la microstructure et la caractérisation tribologique de l'acier modifié m50 in-situ synthétisé par réaction in-situ de Maxthal211 ( $Ti_2AlC$ - $Ti_3AlC_2$ , phase MAX). Le composite a été élaboré à partir de 5, 10, 15 et 20 % en poids de la phase MAX qui a complètement réagi avec la matrice M50 à une température de frittage de 1050 °C pendant 4 h ; la phase MAX s'est décomposée en TiC, et les atomes d'Al et de Ti libérés ont diffusé dans la matrice M50.

## **Mots clés :**

Composite, phase max, microscope optique, traitement thermique, tribomètre, renfort.

# Summary:

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## General Introduction:

Composites represent a kind of materials combining the main advantages of each material on property and functionality, which can be traced back to the ancient times that making grass stems reinforced clay as the primary construction materials. They are usually the products of various elemental materials through structural design and optimization at different scales and levels. The emergence of such high performance and new functions mainly results from complex synergistic effect,

Metal matrix composite materials (MMCs) originated from the demand of advanced technology areas such as aviation, aerospace, and national defense in the late 1950s. MMCs is a combination of ductile metal or alloy matrix strengthened by other metal, ceramic or intermetallic compounds, which are usually fabricated by continuously implanting the reinforcing materials into a monolithic metallic material matrix to reduce weight while enhancing its properties in terms of elastic modulus, specific strength and stiffness, wear and corrosion resistance.

The  $M_{n+1}AX_n$  phases (where M is an early transition metal, A is an A-group element and X is C or N, are layered hexagonal solids with two formula units per unit cell, in which near close-packed layers of M, are interleaved with layers of pure A-group elements, with the X-atoms filling the octahedral sites between M layers. They combine the merits of both metals and ceramics. Like metals, they are an excellent conductor of electricity and heat and most readily machinable, with low susceptibility to thermal shock. Like ceramics, they are quite tidy, lightweight and exceptionally wear resistance.

Since the early '90s in last century, an interesting combination of metallic and ceramic-like properties of MAX phases have motivated the development of MAX/metal matrix composites (MMCs). The layered MAX phases enhanced MMCs exhibited not only high strength but also sufficient deformation capacity by virtue of unique deformation behaviors of MAX in terms of delamination and the formation of kinking band, which facilitate the coordination deformation between reinforcement and metal matrix. As the precursor of *in situ* ultrafine binary carbide/nitride particulates, MAX phases triggered its close derivatives (MX) have also provided a general strategy for the synthesis of a wide range of in-situ MMCs. For the in-situ MX particulates enhanced MMCs, the binary MX particulates are transformed topologically from hexagonal MAX accompanied by de-intercalation of A-site element and then diffusing into metal matrices. As the

Formation of ultrafine and stable ceramic reinforcements, the MAX triggered in-situ MMCs are found to exhibit a superior combination of mechanical strengths and ductility. Based on the previous

investigation about MAX-MMCs, this review covers important research work that has led to advances in the preparation, reactive mechanism, microstructure, tribological, properties of these composite materials in the past decades.

# **Chapter 1: Composites**

## 1.1 Introduction:

Many of our modern technologies require materials with unusual combinations of properties that cannot be met by the conventional metal alloys, ceramics, and polymeric materials. This is especially true for materials that are needed for aerospace, underwater, and transportation applications.

A composite material can be defined as a combination of two or more materials that results in better properties than those of the individual components used alone. In contrast to metallic alloys, each material retains its separate chemical, physical, and mechanical properties.

the two constituents are a reinforcement and a matrix. The main advantages of composite materials are their high strength and stiffness, combined with low density, when compared with bulk materials, allowing for a weight reduction

The new composite material often displays many beneficial characteristics; in many cases, composites are stronger, of lower density, or less costly in comparison to established materials. Commonly, composites consist of two or more different components; the basic components are usually strongly fused at the interface. A variety of both natural and synthetic materials confirm to this picture, such as mortar and concrete, reinforced rubber, alloys, polymers containing fillers, aligned and chopped fiber composites, porous and cracked media, polycrystalline (metal) aggregates, and others [1].

Composite materials are composed of individual basic materials, which are referred to as so-called constituent materials. Two main categories of constituent materials are distinguished: the matrix and the reinforcement. At least one representative from each category is needed to create a composite. The matrix phase embeds, surrounds, and supports the reinforcements by preserving their relative locations. The reinforcements contribute their specific physical and mechanical assets, thus enhancing the properties of the matrix [2].

## **1.2 Advantages of composite materials:**

- Lower density (20 to 40%)
- Higher directional mechanical properties (specific tensile strength (ratio of material strength to density) 4 times greater than that of steel and aluminum.
- Higher Fatigue endurance.
- Higher toughness than ceramics and glasses.
- Versatility and tailoring by design.
- Easy to machine.
- Can combine other properties (damping, corrosion).
- Cost.

## **1.3 Disadvantages:**

- Not often environmentally friendly.
- Low recyclability.
- Cost can fluctuate.
- Can be damaged.
- Anisotropic properties.
- Matrix degrades.
- Low reusability.

## **1.4 Classifications of Composites:**

Composite materials are commonly classified at following two distinct levels:

The first level of classification is usually made with respect to the matrix constituent. The major composite classes include Organic Matrix Composites (OMCs), Metal Matrix Composites (MMCs) and Ceramic Matrix Composites (CMCs). The term organic matrix composite is generally assumed to include two classes of composites, namely Polymer Matrix Composites (PMCs) and carbon matrix composites commonly referred to as carbon-carbon composites.

The classifications of the composites depending mainly on the geometry of the reinforcement: whether it is particulate, flake, or fibers (Figure 1) or could be based on the nature of matrix phase: whether it is polymer, metal or ceramic [3].

### **1.4.1 Metal matrix composite:**

Metal matrix composites possess some attractive properties, when compared with organic matrices. These include strength retention at higher temperatures, higher transverse strength, better electrical conductivity, superior thermal conductivity, higher erosion resistance etc. However, the major disadvantage of metal matrix composites is their higher densities and consequently lower specific mechanical properties compared to polymer matrix composites. Another notable difficulty is the high-energy requirement for fabrication of such composites.

### **1.4.2 Polymer matrix composite:**

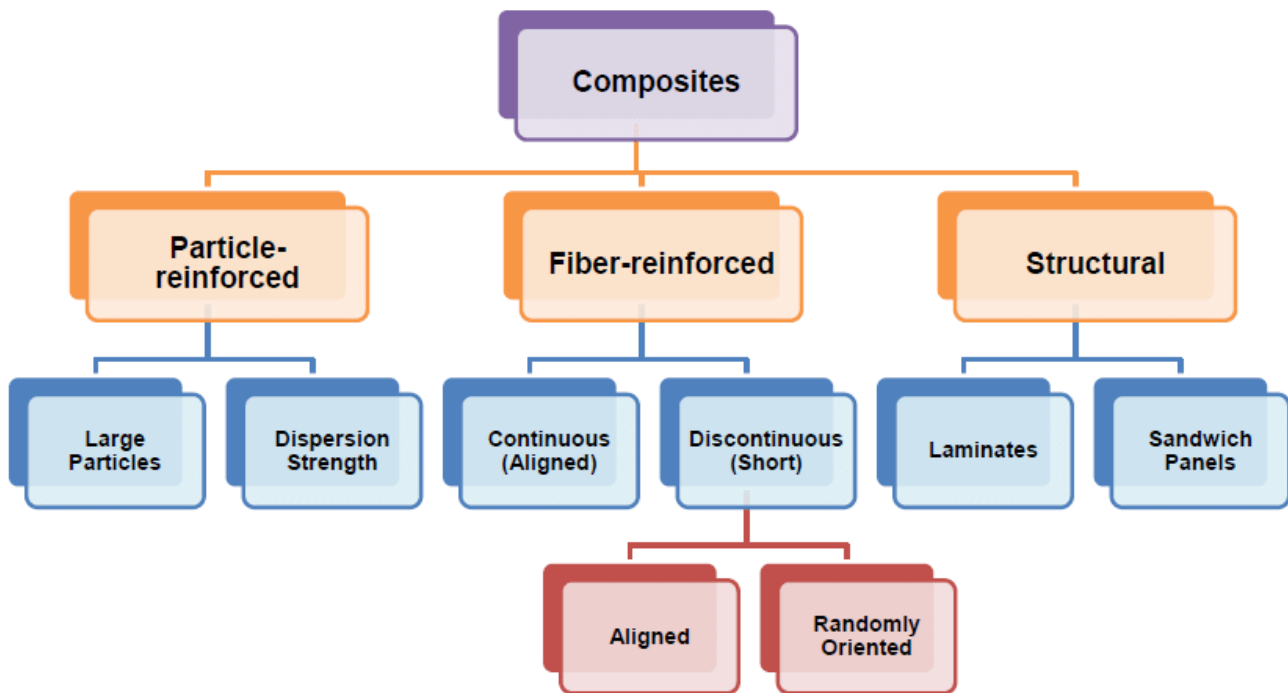
A very large number of polymeric materials, both thermosetting and thermoplastic, are used as matrix materials for the composites.

Generally speaking, the resinous binders (polymer matrices) are selected on the basis of adhesive strength, fatigue resistance, heat resistance, chemical and moisture resistance, etc.

### **1.4.3 Ceramic Matrix composite:**

Ceramic fibers, such as alumina and SiC (Silicon Carbide) are advantageous in very high temperature applications, and also where environment attack is an issue.

The second level of classification refers to the reinforcement form fibre reinforced composites, laminar composites and particulate composites. Fibre Reinforced composites (FRP) can be further divided into those containing discontinuous or continuous fibres.



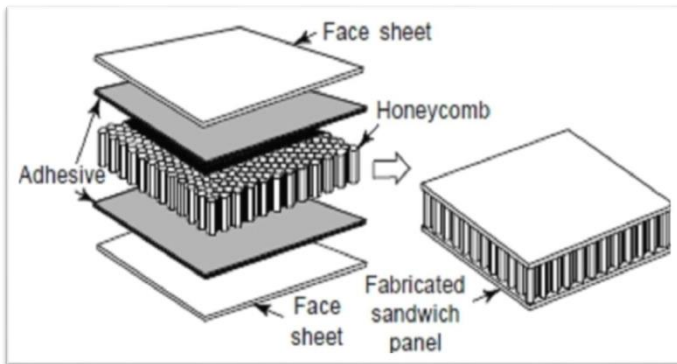
*Figure 1. 1: A simple classification schemes for the various composite types.*

## 1.5 Fiber Reinforced Composites:

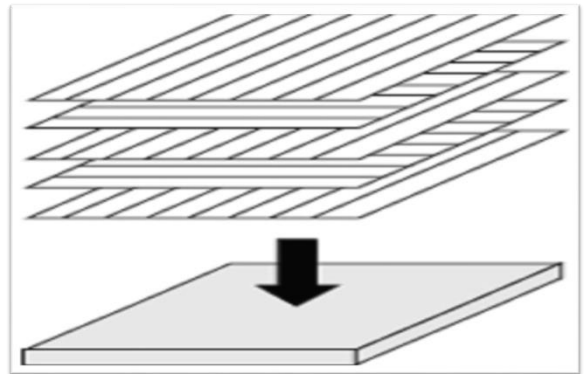
They are composed of fibres embedded in matrix material. Such a composite is considered to be a discontinuous fibre or short fibre composite if its properties vary with fibre length. On the other hand, when the length of the fibre is such that any further increase in length does not further increase the elastic modulus of the composite, the composite is considered to be continuous fibre reinforced. Fibres are small in diameter and when pushed axially, they bend easily although they have very good tensile properties. These fibres must be supported to keep individual fibres from bending and buckling.

### 1.5.1 Laminar Composites:

They are composed of layers of materials held together by matrix. Sandwich structures fall under this category.



**Figure 1.2: Honeycomb structure**



**Figure 1.3: Laminar composite**

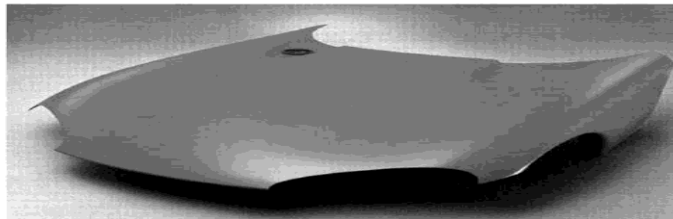
### 1.5.2 Particulate Composites:

They are composed of particles distributed or embedded in a matrix body. The particles may be flakes or in powder form. Concrete and wood particle boards are examples of this category.

#### Some applications of composite materials :



Composites are used in both trucks and cars to reduce weight and increase fuel efficiency.



Recreational vehicles have long used fiberglass composites, mostly for its durability and weight savings over metal.



**Figure 1.4: Transportation application.**

The major auto-makers (Fig1.4) are increasingly turning to composites to help them meet performance and weight requirements, thus improving fuel efficiency. Cost is a major driver for commercial transportation, and composites offer lower weight and lower maintenance costs.





Many of the world's roads and bridges are badly corroded and in need of constant maintenance or replacement.

Composites offer much longer life with less maintenance due to their corrosion resistance.

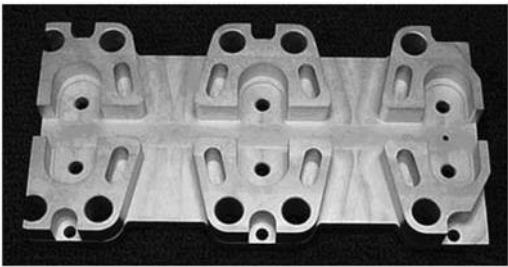


Repair, upgrading, and retrofit of bridges, buildings, and parking decks

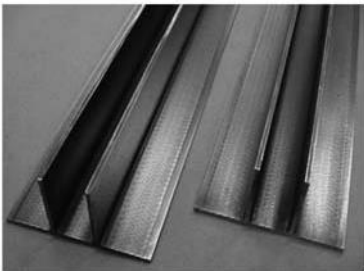
***Figure 1.5: Infrastructure applications.***

Using composites to improve the infrastructure (**Figure 1.5**) of our roads and bridges is a Relatively new, exciting application. Many of the world’s roads and bridges are badly corroded and in need of continual maintenance or replacement.

Although metal and ceramic matrix composites are normally very expensive, they have found uses in specialized applications such as those shown in (**Figure 1.6**). Frequently, they are used where high temperatures are involved. However, the much higher temperatures and pressures required for the fabrication of metal and ceramic matrix composites lead to very high costs, which severely limits their application.



Metal Matrix Composite  
Electronic Components



Metal Matrix Composite  
Structural Components



Ceramic Matrix Ceramic  
Exhaust Nozzles



Carbon-Carbon Brakes

**Figure 1.6: Metal and ceramic matrix composite applications.**

# Chapter 2: MMC

## **2.1 Introduction:**

### **2.1.1 Metal-matrix composites:**

As the name implies, for metal-matrix composites (*MMCs*) the matrix is a ductile metal. Metal matrix composites, at present though generating a wide interest in research fraternity, are not as widely in use as their plastic counter parts. High strength, fracture toughness and stiffness are offered by metal matrices than those offered by their polymer counterparts. They can withstand elevated temperature in corrosive environment than polymer composites. Most metals and alloys could be used as matrices and they require reinforcement materials which need to be stable over a range of temperature and non-reactive too. However the guiding aspect for the choice depends essentially on the matrix material. Light metals form the matrix for temperature application and the reinforcements in addition to the aforementioned reasons are characterized by high modulus.

Most metals and alloys make good matrices. However, practically, the choices for low temperature applications are not many. Only light metals are responsive, with their low density proving an advantage. Titanium, Aluminium and magnesium are the popular matrix metals currently in vogue, which are particularly useful for aircraft applications. If metallic matrix materials have to offer high strength, they require high modulus reinforcements. The strength-to-weight ratios of resulting composites can be higher than most alloys.

The melting point, physical and mechanical properties of the composite at various temperatures determine the service temperature of composites. Most metals, ceramics and compounds can be used with matrices of low melting point alloys. The choice of reinforcements becomes more stunted with increase in the melting temperature of matrix materials.

Here it would be worthwhile to mention that an important area of composite research are *MMCs*, in which a metallic host material is modified through the addition of reinforcement. These reinforcements may be in the form of particles or fibers. The goal is to supplement the desirable properties of the metal, such as ductility, by the addition of a reinforcement that can improve the performance of the material in its final application. Common goals in the creation of new *MMCs* are: to achieve increased strength, reduce friction, and wear or prevent corrosion.

## 2.2 Strengthening Methods in MMCs

### 2.2.1 Dislocation strengthening:

Dislocations' density is higher in the composite matrix than in unreinforced metal with the same history. In a composite, dislocations can be generated by straining in response to an applied load, straining to relax residual thermal stresses caused by CTE mismatches between matrix and reinforcement. The increase in dislocations' density can be written as Russell and Lee(2005):

### 2.2.2 Orowan strengthening:

This mechanism strengthens the composite by making sub-sequent dislocation motion more difficult. Although important in dispersion-strengthened systems, Orowan mechanism does not effectively strengthen other MMCs because in other MMCs there reinforcement phases (particles) are too large and too far to be considered as effective obstacle to the motion of dislocations. However, Orowan strengthening mechanism can be significant in discontinuously reinforced aluminum composites where the particles are finer and closer to get her and causes increase in the strength of the MMC.

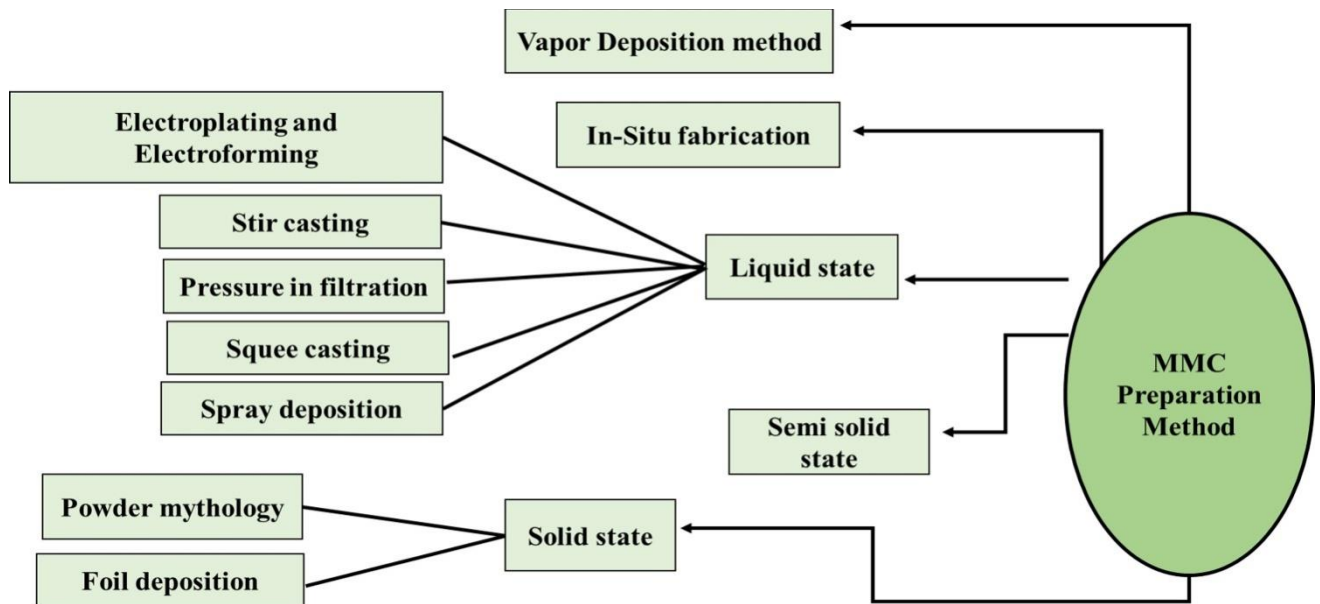
### 2.2.3 Grain refinement strengthening:

The MMCs usually possess much finer grain size than that of unreinforced matrix-metal. HallPetch relationship implies that the beginning of plastic flow in the matrix depends on the local magnification of stress at grain boundaries resulting from dislocation pile-ups. A small grain size results in fewer dislocations in the pile-ups, and hence the applied stress must be higher to cause yielding.

The matrix in a metal matrix composite (MMC) is usually an alloy, rather than a pure metal, and there are three types of such composites, namely, dispersion-strengthened, in which the matrix contains a uniform dispersion of very fine particles, particle-reinforced, in which particles of sizes greater than  $1\ \mu\text{m}$  are present, fiber-reinforced, where the fibers may be continuous throughout the length of the component, or less than a micrometer in length, and present at almost any volume fraction,[5].

### 2.3 Methods for fabricating MMCs:

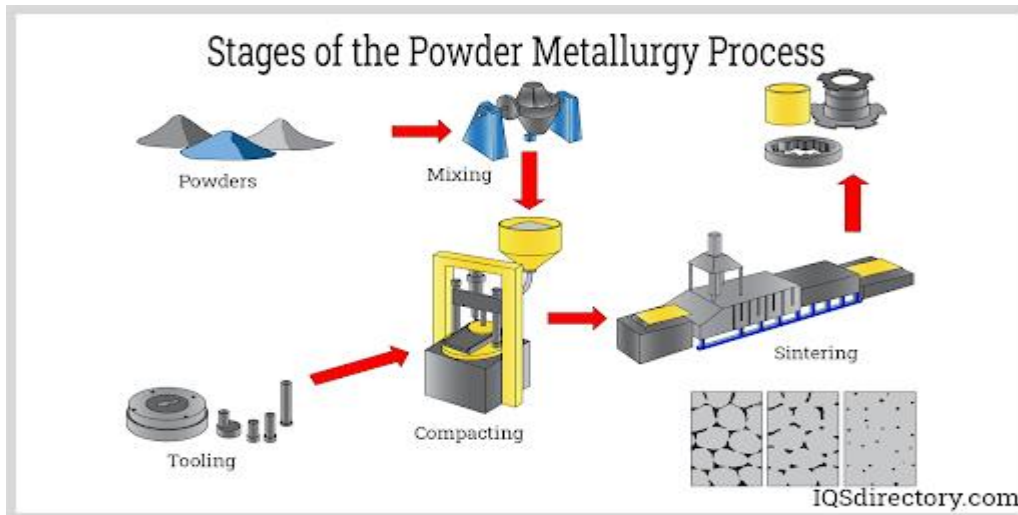
There are two basic techniques in making of metal matrix composite such as solid phase techniques and liquid phase techniques. The solid phase techniques such as powder metallurgy, diffusion bonding and plasma transferred. The liquid phase techniques are, squeeze casting, vacuum infiltration techniques, stir casting and compo casting.



*Figure 2.1: Mmc fabrication methods.*

### 2.4 Production of composite materials by powder metallurgy methods:

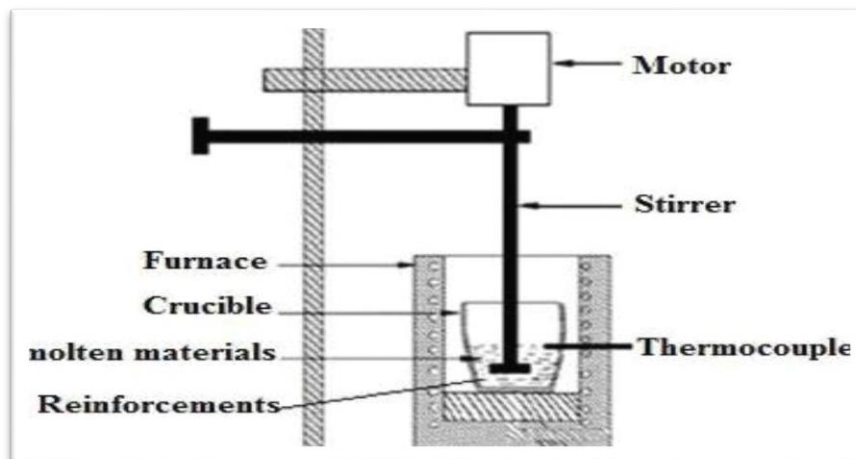
Powder metallurgy methods are based on the classical blending of matrix powders and reinforcing elements (dispersion powders, platelets and ceramic) and further cold pressing and sintering ; The method described above, on account of its simplicity, is applied widely for the production of composite materials with magnesium alloys matrix [6], aluminium alloys matrix [7], and copper matrices [8]. Mechanical alloying is nowadays one of the most widely applied methods for the production of composite materials reinforced by dispersion particles. The process is performed in a high-energy ball mill, making possible the introduction of hard dispersion particles into a relatively soft metal matrix [9]. The composite powders, so produced are then pressed and consolidated or cold pressed, sintered.



*Figure 2.0 .2: powder metallurgy process*

### 2.4.1 Stir casting:

The process of stir casting generally involves the admixture of ceramic particulate reinforcement with a molten metal matrix. The particulates are distributed and suspended in the molten metal via high energy mixing or another appropriate process. This process is widely used for applications that require high production volumes and low cost. [10]



*Figure 2. 3: Stir casting process set up.*

### 2.5 Wear behavior:

Perhaps the biggest in solving wear problems is that of anticipating the type(s) of wear to which components will be subjected. Material can be removed from a solid surface in only three ways: by

melting, by chemical dissolution, or by the physical separation of atoms from the surface. The last method can be accomplished either by the one-time application of a high strain or by cyclic straining at lower magnitudes. Mechanical and chemical processes may operate separately or together, such as abrasion in a corrosive medium. [11]

### **2.5.1 Types of wear:**

Depending on the nature of movement of the media following types wear has been classified:

- Abrasive wear
- Sliding and Adhesive wear
- Corrosive wear
- Oxidational wear

#### **2.5.1.1 Sliding and adhesive wear:**

Both refer to a type of wear generated by the sliding of one solid surface along another surface.

Adhesive wear is as ambiguously defined as is sliding wear, though the two are not strictly synonymous. Adhesive wear denotes a wearing action in which no specific agency can be identified as the cause of the wear.

#### **2.5.1.2 Abrasive wear:**

Abrasive wear, as defined by ASTM, is due to hard particles or hard protuberances that are forced against and move along a solid surface. Wear, in turn, is defined as damage to a solid surface that generally involves progressive loss of material and is due to relative motion between that surface and a contacting substance or substances.

#### **2.5.1.3 Corrosive wear:**

It is defined as the degradation of materials in which both corrosion and wear mechanisms are involved. The combined effects of wear and corrosion can result in total material losses that are much greater than the additive effects of each process taken alone, which indicates synergism between the two processes. This article focuses on the corrosion-wear synergism in aqueous slurry and grinding environments. Examples of corrosive wear problems encountered in industry are provided, and the effects of environmental factors on corrosive wear are discussed.

#### **2.5.1.4 Oxidational wear:**

It is a form of wear that primarily occurs during nominally unlubricated conditions of sliding. It is usually, but not always, a beneficial form of corrosion in which oxide films that are approximately 3 or 4 mm thick form on the real areas of contact at the relatively high "hot-spot" temperatures that



often occur between sliding interfaces. In many cases, tribological oxidation can reduce the wear rate by two orders of magnitude, compared with the wear of the same metals under an inert atmosphere. However, amelioration of the wear rate will only occur if the oxide layers are formed during sliding. It is not possible to artificially produce low-wear surfaces by pre-oxidizing the surfaces under static furnace conditions. . [11]

### **2.5.2 Properties for wear resistance:**

Wear resistance is an important function in the balance of properties provided by MMCs. The addition of hard reinforcements intrinsically improves the wear resistance of the host metal. Further, additions such as graphite can also provide intrinsic lubricity. MMC materials have been engineered to provide exceptional wear resistance and represent an important family of Applications .

### **2.6 Application of MMCs:**

The combined attributes of MMCs , together with the costs of fabrication, vary widely with the nature of the material, the processing methods, and the quality of the product. In engineering, the type of composite used and its application vary significantly, as do the attributes that drive the choice of MMCs in design. For example, high specific modulus, low cost, and high weldability of extruded aluminum oxide particle-reinforced aluminum are the properties desirable for bicycle frames. High wear resistance, low weight, low cost, improved high-temperature properties, and the possibility for incorporation in a larger part of unreinforced aluminum are the considerations for design of diesel engine pistons. MMCs are being used in various applications including, [Chawla and Chawla\(2013\)](#):

Aerospace

Transportation

Electronics

Electric power transmission Recreational products and sporting goods

Wear-resistance materials

The automotive market is a high volume and a high technology market, but costs should be as low as possible. However, there are still a lot of reasons to consider the use of light aluminum composites:

Reduction of the weight of engine parts.

Increase of the operation temperature of engines.

Improvement of the tribological properties of moving and contacting.

Components (wear resistance, lubrication).

Increase of stiffness and strength.

### **Example:**

Railroad brakes The driving force for lightweight railway vehicles has also prompted the use of high-performance MMCs, Chawla and Chawla (2013). A conventional brake system for a railway vehicle consists of four brake disks, calipers, hand brake, and electromagnetic track brake. This makes up about 20% of the total weight of the bogey. A particulate-reinforced aluminum–matrix composite (AlSi7Mg p SiC particulates, supplied by Duralcan) is fabricated by a multi-pouring process, where alternating layers of the unreinforced alloy and MMC is cast in successive layers ; This contributed to reduced cost by using less MMC and placing the composite in the strategically important region , in contact with the wearing surface). In this application, the weight is reduced from 115 kg for a spheroidal graphite iron disk to 65 kg for the MMC disk, a weight savings of 43%. The steel brake shows significant cracking, while the MMC brake is in relatively good condition.



*Figure 2.4: drums and brake rotors made of MMC.*

## **2.7 Current status of research and developments:**

Traditionally composites were produced by processing techniques e.g., powder metallurgy, rapid solidification , and diverse casting techniques. The reinforcing phase is first mixed with the matrix material. The scale of therein forcing phase is consequently constrained by the

starting powder size, which is typically of the order of few microns to that of few tens of microns and rarely below  $1\mu\text{m}$ . During the last ten years, the new in situ processing technology for fabricating metal and ceramic composites has emerged as a very promising technique. Some researchers regard in situ MMCs as the second-generation MMCs due to the potential advantages it has over the conventional MMCs. The following are the advantages of the in situ MMC fabrication technique over the metal matrix composites produced by ex situ method.

The in situ form reinforcement is thermo-dynamically stable in the matrix, leading to less degradation in high temperature service.

The reinforcement matrix interfaces are clean contributing to an improvement in wettability. Fabrication cost is lower, because it is the one-step process.

The in situ form reinforcing particles are finer in size and their distribution is more uniform resulting in better mechanical properties of the MMCs.

Because of the great potential that the in situ MMCs offer for wide spread application novel processing-techniques have been recently developed to produce them. A brief description of some of the important techniques is given in the following section.

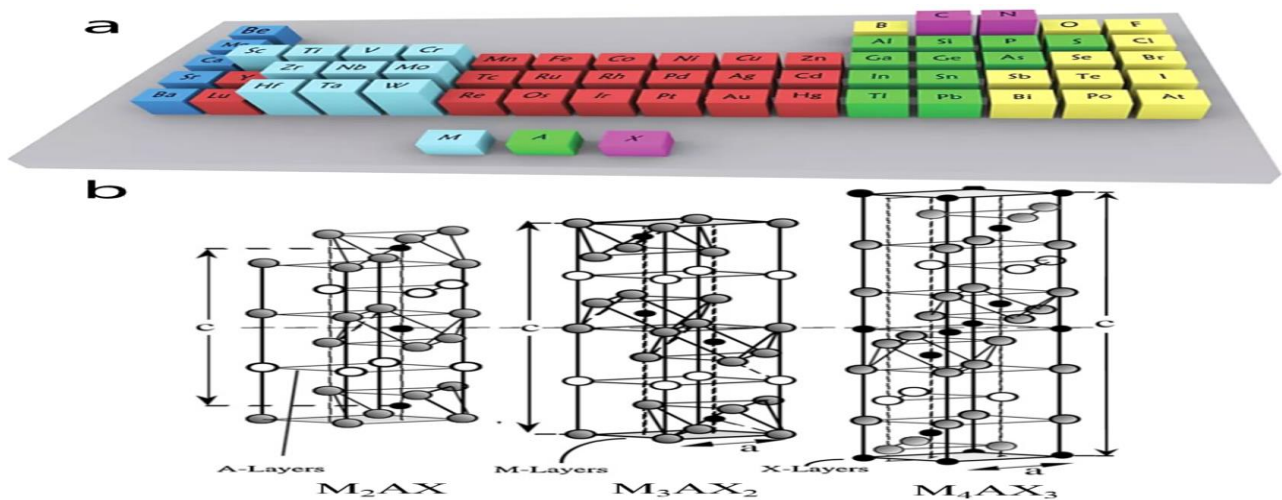
# **CHAPTER 3: MAX PHASE**

### 3.1 INTRODUCTION:

The  $M_{n+1}AX_n$  phases (where M is an early transition metal, A is an A-group element and X is C or N, are layered hexagonal solids with two formula units per unit cell, in which near close-packed layers of M, are interleaved with layers of pure A-group elements, with the X-atoms filling the octahedral sites between M layers. The different unit cell of MAX phases labeled as 211 phase, 312 phase, and 413 phase [12]. They combine the merits of both metals and ceramics. Like metals, they are an excellent conductor of electricity and heat and most readily machinable, with low susceptibility to thermal shock. Like ceramics, light weight and exceptionally oxidation and wear resistance [13].

The fabricated composite materials possess a relatively lower and stable friction coefficient meanwhile exhibiting an excellent wear resistance.

the layered hexagonal MAX phases are thermo-dynamically stable nano laminates suggesting they are favorable self-adjusting solid lubricant materials [14]. There is no doubt about this since reports on the properties of  $Ti_3SiC_2$ , which was identified that the material, just like layered graphite, felt lubricious. Many researchers have devoted to the study of the friction properties of MAX phases and their enhanced MMCs Their results indicated that the layered MAX phases greatly promote the formation of dense lubricating films covered on the matrix, which can effectively reduce the wear rate of the matrix material. The fabricated composite materials possess a relatively lower and stable friction coefficient meanwhile exhibiting an excellent wear resistance.



**Figure 3.1: MAX phase units.**

### 3.2 The Chemistry and Crystal Structure of MAX Phases:

The M-M distances in the MAX phases are strongly correlated with, and almost equal to, the M-M distances in the MX phases. As for the MX compounds (15), it is useful to consider the MAX compounds to be interstitial compounds in which the A atoms and the X atoms fill the interstitial sites between the M atoms. In such a scheme, the  $c$  parameter of the 211 phases—composed of four M layers per unit cell—should be  $\approx 4$  times the  $a$  parameter. Similar arguments for the 312 and 413 phases, with six and eight M layers per unit cell, respectively.

### 3.3 Atomic Bonding in the MAX Phases:

Currently, investigators are devoting much effort to theoretical modeling of the MAX phases (16). Not unlike the MX phases, bonding in the MAX phases is a combination of metallic, covalent, and ionic. The following conclusions of the theoretical work are noteworthy:

(a) As in the MX compounds, there is a strong overlap between the  $p$  levels of the X elements and the  $d$  levels of the M atoms, leading to strong covalent bonds that are comparable to those in the MX binaries (16). (b) The density of states at the Fermi level,  $N(E_F)$ —as in some MX binaries, but notably not TiC—is substantial. (c) The  $p$  orbitals of A atoms overlap the  $d$  orbitals of the M atoms. (d) The electronic states at the Fermi level are mostly  $d$ - $d$  M orbitals. (e) In the  $M_2AlC$  phases, there is a net transfer of charge from the A to the X atoms (17). Whether this is true of other MAX phases as well awaits more studies.

Because of the similarities of structure and atomic bonding between the MAX and their corresponding MX phases, these phases share many common attributes and properties. For example, both are metal-like conductors and stiff. Their mechanical properties, however, could not be more different. Some of the MX phases are some of the hardest solids known. They are also brittle, nonmachinable, and susceptible to thermal shock. In sharp contradistinction, as discussed herein, the MAX phases are quite damage tolerant, thermal shock resistant, and most readily machinable. Finally, the Raman modes for the MAX phases have been deciphered and are, for the most part, in excellent agreement with experimental results (18).

### **3.4 The transformation of MAX to MX in metal matrix:**

During the past decade, a great variety of processing techniques have been adopted to fabricate MAX decomposed MX particulate reinforced in situ MMCs. The feasibility of in-situ reactions involved in thermodynamics of decomposition of MAX phases has been considered by several researchers [19]. In the following text, the thermodynamic decomposition of MAX phases which paves the way for the fabrication of in-situ binary carbide/nitride reinforced MMC is introduced.

At high temperatures, the MAX phases do not melt congruently but decompose periodically into A-rich liquids and  $M_{n+1}X_n$  carbides or nitrides. Thermal decomposition occurs by the loss of the A elements and the formation of lower n-containing MAX phases and/or MX. For example, in  $Ti_3AlC_2$ , its decomposition into TiC and  $Ti_2AlC$  as lower order or intermediate phase was observed at  $\geq 1400^\circ C$  in the vacuum condition [20]. However, at higher temperatures, as in comparison with TiC, a gradual disappearance of  $Ti_2AlC$  also indicates that  $Ti_2AlC$  experienced further decomposition into TiC via the further sublimation of Al. However, in contrast to  $Ti_3AlC_2$ , no intermediate or lower order MAX phase but the final formation of  $TiC_x$  was observed for the decomposition of  $Ti_3SiC_2$ .

### **3.5 PROPERTIES:**

Some of the physical properties of the MAX phases, such as thermal expansion, elastic properties, and thermal conductivity, have much in common with their respective MX binaries (21). However, the electronic structure and transport properties of the MAX phases are more akin to those of the transition metals (21). The unique combination of properties possessed by the MAX phases—i.e., ease of machinability, low friction, thermal and structural stability, and good thermal and electrical conductivities—makes them attractive for many applications such as rotating electrical contacts and bearings, heating elements, nozzles, heat exchangers, and tools for die pressing. Many of these applications are currently being field tested and are at various stages of development. There are several hurdles to commercialization, including the relative high cost of the powders and sintered components compared with other structural ceramics such as SiC and alumina. Another hurdle is the novelty of these solids and the reluctance of some researchers to use them instead of solids with which they have been working with for many years and are more familiar. The lack of

pressureless sinterable powders until recently also slowed the development of these materials' use.

Tribological performance has been improved by adding secondary phases such as Al<sub>2</sub>O<sub>3</sub>, BN, B<sub>4</sub>C, SiC, chopped SiC fibers, Ti<sub>5</sub>Si<sub>3</sub> and Ag.(22), (23), (24), (25), (26) Ceramic particles improve the wear resistance due to their higher hardness and decentralization of shear stresses under the counter-body, meanwhile the addition of metallic particles modifies the tribochemistry reactions. Additionally, MAX phases have been recently added as reinforcing particles in metals (27), and polymers.(28),The composites present lower  $\mu$  and WRs due to the mitigation of the abrasive wear and modification of the tribochemistry reactions. Electrical properties: MAX phases are metallic electrical conductors in which the resistivity increases linearly with temperature.

### **3.5.1 Synthesis:**

The synthesis of MAX phases can be categorized in three main groups:

- i) Physical Vapor Deposition (PVD) techniques,
- ii) Solid state reactions
- iii) Molten processes. In general, the synthesis of MAX phases is relatively simple, but high yields ( $\sim > 95$  wt.%) are difficult because they coexist with other thermodynamically stable phases such as carbides or nitrides and intermetallic.

Unfortunately, M-A-X phase diagrams are only available for some systems. The main limitation is the synthesis of large quantities of highly pure MAX phase powders, which has restricted their transfer to industry. PVD methods usually lead highly pure MAX phase coatings, but they are not suitable processes for powder production. On the other hand, powders can be synthesized by liquid/solid state reaction approaches, but more investigation is required to improve purity, scalability, reproducibility and costs.

#### **Liquid/Solid State Reaction**

Liquid/Solid state reaction (SSR) is the most used method to synthesize MAX phases. This method is based on the reactions at high temperature between the elemental constituent starting powders or other precursors such as carbides (MX) or hydrides (MH). The synthesis temperature typically ranges between 1100 and 1700 °C and is carried out under protective



atmosphere to avoid the oxidation. As “M” source elemental powders such as Ti for Ti<sub>2</sub>AlC and Ti<sub>3</sub>AlC<sub>2</sub>, or Cr for Cr<sub>2</sub>AlC are more commonly used, although carbides such as TiC and Cr<sub>3</sub>C<sub>2</sub> are also good options

MAX phase composites MAX phases have been reinforced with ceramic particles to improve their mechanical and tribological performance, and they have also been used as reinforced phase in metallic and polymeric matrices. In general, the precursors are mixed in precise quantities to attain the maximum MAX phase yield, minimizing the content of carbides or nitrides. However, the content of these secondary phases can be controlled through the quantities of the starting powders to develop Ceramic Matrix Composites (CMCs).

### **3.6 Potential applications**

#### **3.6.1 High temperature structural material:**

The most likely potential application for Al<sub>2</sub>O<sub>3</sub> scale-forming MAX phases is as components that operate under aggressive environments at temperatures between 1100 and 1400 °C over long period of times. In addition to heating elements and gas nozzles, Ti<sub>2</sub>AlC, Ti<sub>3</sub>AlC<sub>2</sub> and Cr<sub>2</sub>AlC are potential candidates to replace Ni/Co super alloys in the hottest part of a gas turbine engine to enable operation at higher temperatures and thereby increase efficiency.

#### **3.6.2 Nuclear:**

Phases such as Ti<sub>3</sub>SiC<sub>2</sub>, Ti<sub>2</sub>AlC, Ti<sub>3</sub>AlC<sub>2</sub>, Cr<sub>2</sub>AlC, V<sub>2</sub>AlC, Zr<sub>2</sub>AlC, Zr<sub>3</sub>AlC<sub>2</sub> and Nb<sub>4</sub>AlC<sub>3</sub>, that combine excellent radiation tolerance with oxidation and corrosion resistance, mechanical properties and chemical stability have attracted considerable attention as potential accident tolerant fuel (ATF)

#### **3.6.3 Heat exchangers:**

One more application that has been considered is to use MAX phases as heat exchangers for higher temperatures (~ 850 °C) than conventional metallic systems,(270) where ceramics are the only alternative. Unfortunately, their poor mechanical properties, difficult machinability, low thermal conductivity,

#### **3.6.4 Concentrated Solar Power (CSP):**

CSP systems convert solar energy into electric power without greenhouse emission and offer the possibility of thermal energy storage. CSP designs concentrate solar radiation into a receiver using mirrors, and the heat is transported by a heat transfer fluid (HTF) to a steam turbine to produce electricity.

## **CHAPTER 4: EXPERIMENTAL DETAILS**

## 4.1 The process of bonding m50 with reinforcement (Ti2AlC, Ti2Al3C):

### 4.1 POWDER PREPARATION:

M50 and M50 matrix with different contents of Ti<sub>2</sub>AlC/Ti<sub>3</sub>AlC<sub>2</sub> were fabricated by natural sintering. The composite powders of M50 matrix were composed OF ( Carbides,Sulfur,Aluminum ,Nickel ,Copper, Cromium, Vanadium ) and iron powders by weight fractions 0.75, 0.01,0.3,0.2,0.5,5,4.2,89.04. The weight fractions of Ti<sub>3</sub>AlC in the composites were 5, 10, 15 and 20% respectively, the powders were mixed together by vibration milling for 30 minutes each. The in-situ formed composites were fabricated by preparing powder mixtures with 5%, 10 wt%, 15%, and 20 wt% of Ti<sub>2</sub>AlC in M50 matrix, respectively (see table IV.2.1).

It is well established that commercial Ti<sub>2</sub>AlC powders often contain a sizable amounts of Ti<sub>3</sub>AlC<sub>2</sub>. The Ti<sub>2</sub>AlC (Maxthal211) powder is henceforth referred to as TAC.

SAMPLE	Wt.% M50	M50 weight( gr)	Wt.% maxthall 211	Maxthall 211 weight (gr)	SAMPELS NAME
1	100%	4	0%	0	M50
2	95%	3.8	5%	0.2	5 TAC
3	90%	3.6	10%	0.4	10 TAC
4	85%	3.4	15%	0.6	15 TAC
5	80%	3.2	20%	0.8	20 TAC

*Table 1: Powder mixtures with 5%, 10 wt%, 15%, and 20 wt% of Ti<sub>2</sub>AlC in M50 matrix, respectively.*

### 4.2 XRD OF MAXTHALL211 POWDER:

- The analysis of the phases were made in ( USTHB LABORATORY ) obtained during the X-ray diffraction analysis (XRD) of the PHASE MAX powder, is accessible using the X'pert High score Plus software, which is accompanied by a PDF2 database allowing identification of the elements of each phase identified .



*Figure 4. 1: Different contains of modified M50 steel powder.*

### 4.3 Rotatory mixer:

We made this rotatory mixer manually to powder or pulverize solid materials without an associated liquid. The grinding for 30 min at a rotary speed of 250 rpm was performed for all the compositions, in a PM 400 planetary mill, with ball-to-powder ratio of 5:1. This experiment and results were established in (**LABORATOIRE DE RECHERCHE ET ETUDE INDUSTRIELLE TECHNOLOGIQUE**).



*Figure 4.2: Rotatory mixer.*

### 4.4 Compaction:

Compaction is a common operation across a range of industries. The process involves filling a powder formulation into a die, compression using rigid punches, and ejection from the die. Post-compaction operations such as sizing, sintering, coating, etc. are usually also applied depending on the application.

The powder mixtures of modified M50 steel and (5%, 10%, 15%, 20%)  $Ti_2AlC/Ti_3AlC_2$  were then cold pressed at 10 tons into a ~16 mm stainless steel die. This experiment and results were established in (**LABORATOIRE DE RECHERCHE ET ETUDE INDUSTRIELLE TECHNOLOGIQUE**)



*Figure 4.3: Powder compacting press.*

#### **4.5 Heat treatment:**

Heat-treating (or sintering) is a group of industrial, thermal and metalworking processes used to alter the physical, and sometimes chemical, properties of a material. The most common application is metallurgical.

We used this heat treatment argon atmosphere for 4hours at temperature of 1050°C **IN ALGESCO (general electric) – Guerouaou**

#### **4.6 Grinding:**

Grinding is the process of creating a smooth and shiny surface by rubbing it or by applying a chemical treatment, leaving a clean surface with a significant specular reflection, in some materials polishing is also able to reduce diffuse reflection to minimal values. We used different sizes of sandpaper starting with 220, passing to 400, 600, 800, 1200 and 4000, Grinding was made by diamante solution 3um passing to 1um, established in **(SDM LABORATORY OF MECHANICAL ENGINEERING DEPARTMENT)**



*Figure 4.4: Grinding machine.*

#### **4.7 Microscopy observation:**

Microscopy is the technical field of using microscopes to view objects and areas of objects that cannot be seen with the naked eye, there are three well-known branches of microscopy: optical electron, and scanning probe microscopy, along with the emerging field of X-ray microscopy, in our study we used an optical microscope (euromex), All of these observations were done in (**Laboratoire Etudes et Recherche en Technologie Industrielle – University of Blida 1** )



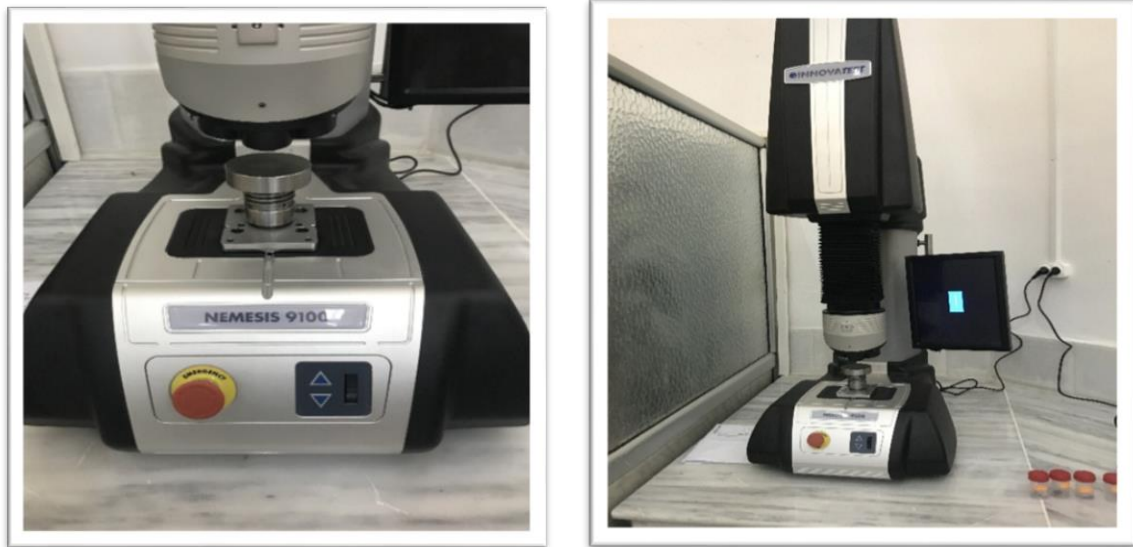
*Figure 4.5: Optical microscope*

#### **4.8 Hardness:**

The hardness of a material is defined as the mechanical resistance that a material opposes to penetration.

The hardness of the elaborated samples was measured using Vickers hardness Indenter (INOVATEST NEMESIS 9100) at room temperature for 10 s at a load of 5 Kgf. An average of 5 readings for each composition is reported in the text.

The hardness test was established in (**Laboratoire Etudes et Recherche en Technologie Industrielle – University of Blida 1** )



*Figure 4.6 Hardness tester*

#### **4.9 Tribology:**

Tribology is the science and engineering of interacting surfaces in relative motion. It includes the study and application of the principles of friction, lubrication, and wear.

The dry sliding wear tests were conducted in a ball-on-disk configuration (CSM Tribometer, Switzerland), in an open atmosphere. The ball was made of 100Cr6 steel ball (6 mm, Hv ~ 8GPa). The testing was performed under normal applied loads of 1, 5 N, and a relative sliding speed of 5cm/s for 100 m sliding distance. An average of the final 10 m data points of the friction coefficient ( $\mu$ ) was used to calculate steady state friction coefficient ( $\mu$ ).

The tribology tests were established in (**Laboratoire Etudes et Recherche en Technologie Industrielle – University of Blida 1** )





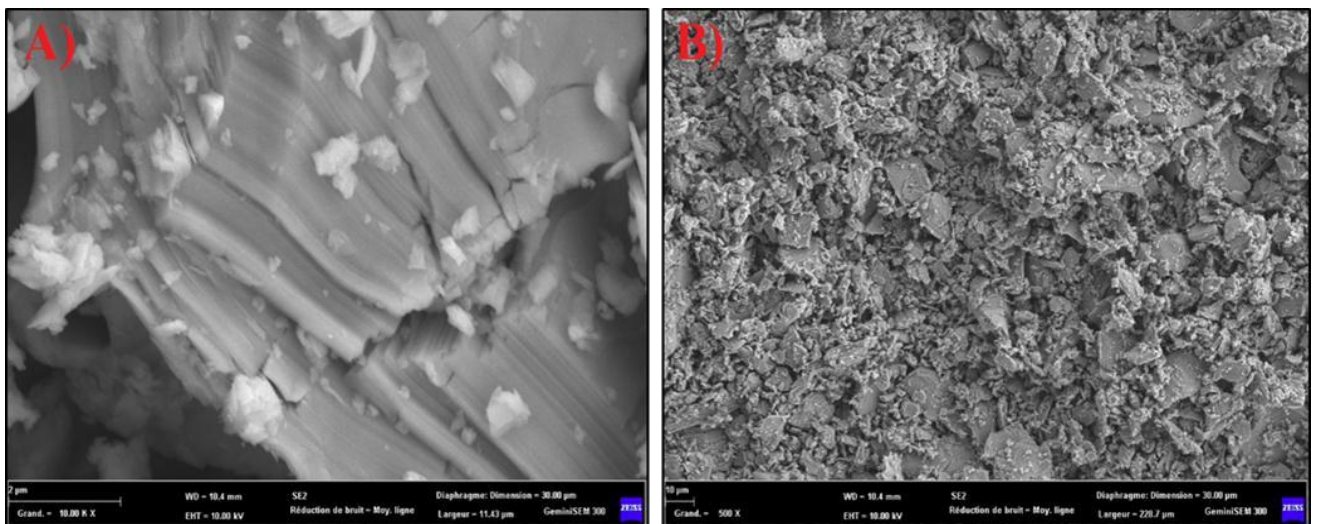
*Fig 4.7 Tribometer*

# **CHAPTER 5: DISCUSSION AND RESULTS**

## 5.1. Max phase characterization:

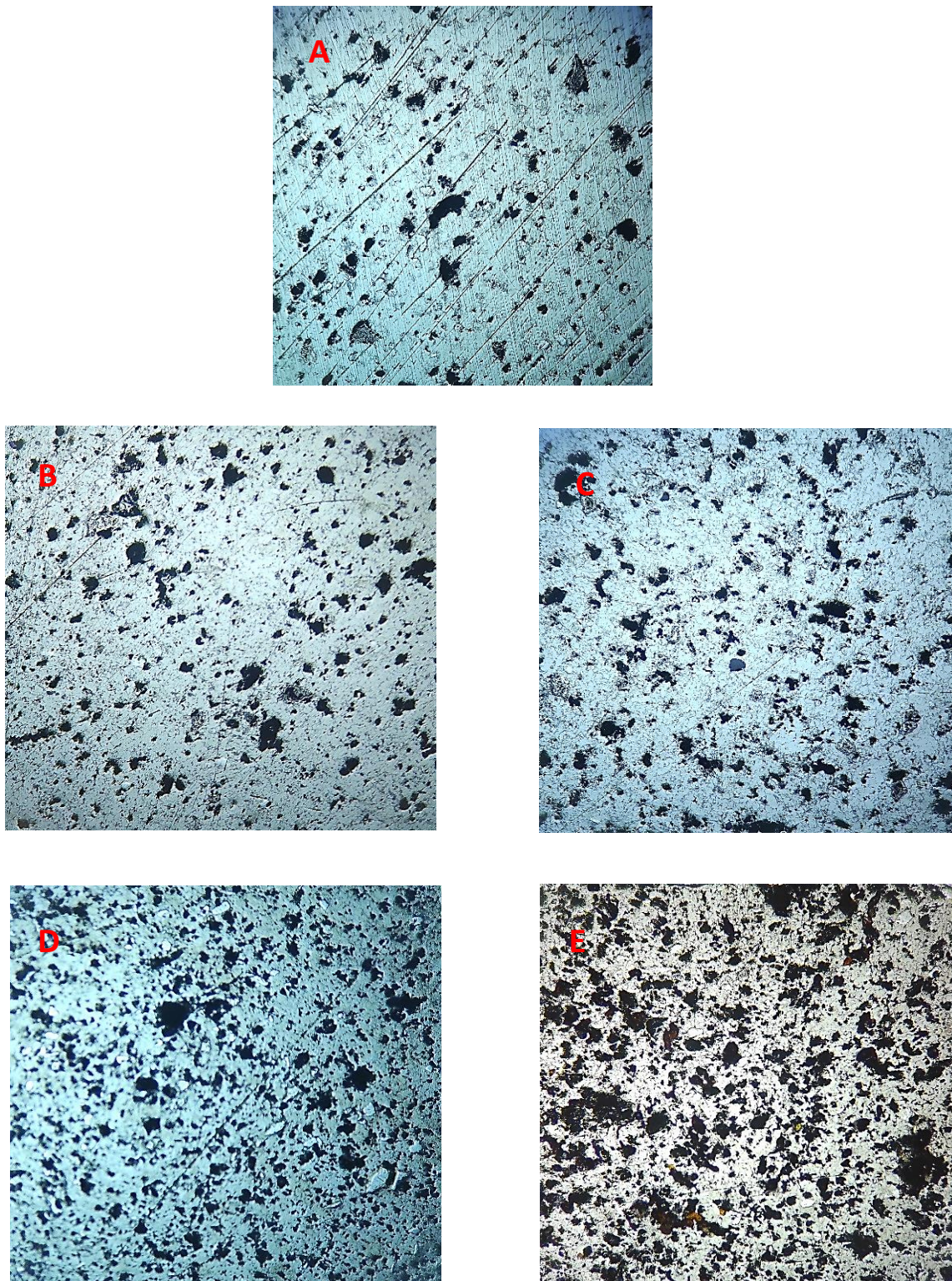
### Scanning electron microscope (SEM):

Figure 5.1 represents a scanning electron microscope (SEM) imaging of the PHASE MAX used in our work, where we can distinguish the lamellar appearance which corresponds to the typical structure of the MAX PHASES (Figure 5.1). It can be observed that the particles of TAC have irregular shapes (coarse and fine agglomerate) (Figure 5.1B).

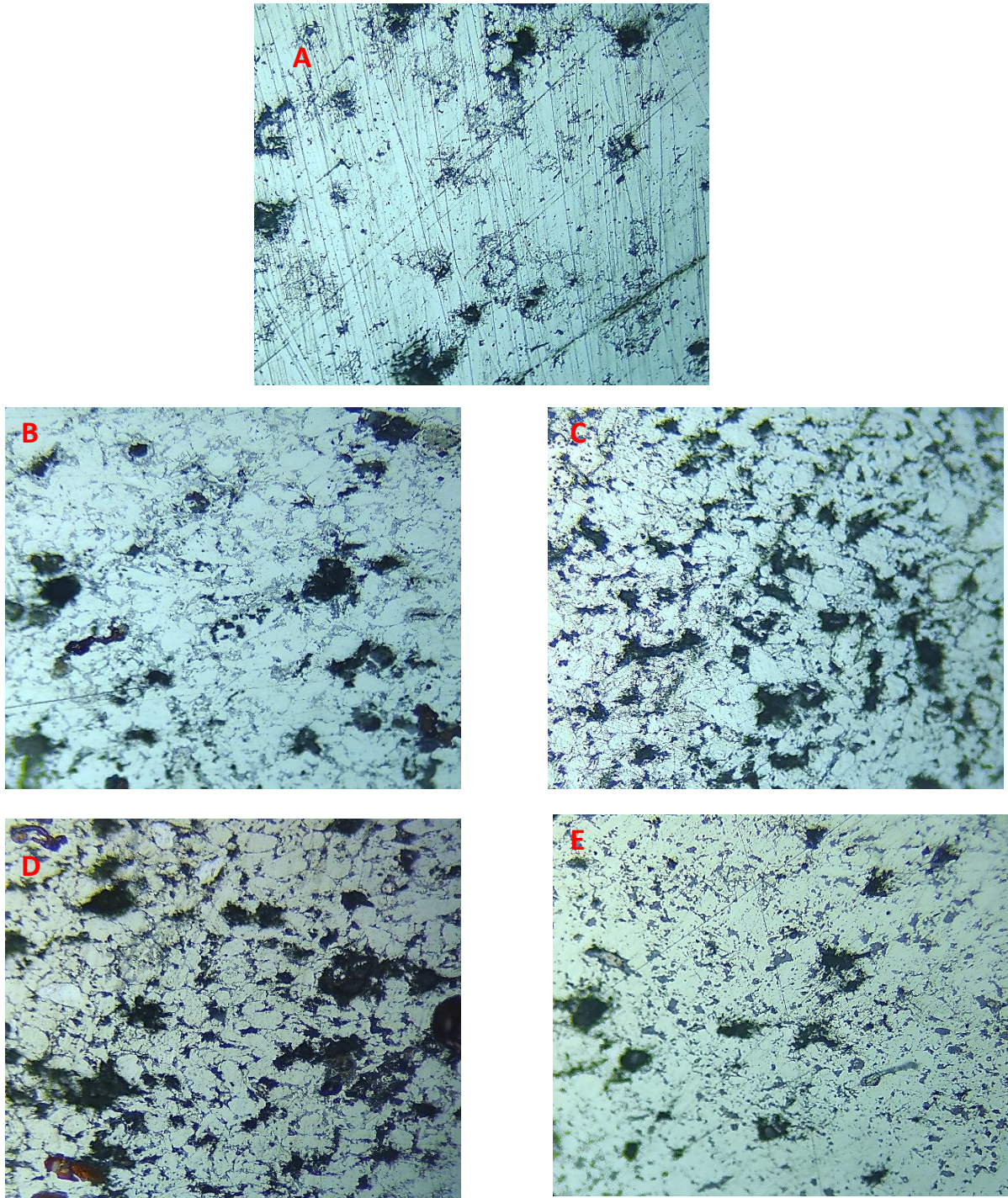


*Fig 5.1: MAX phase SEM imaging (Ti<sub>2</sub>AlC and Ti<sub>3</sub>AlC<sub>2</sub>).*

## 5.2 Optical Microscope Observation:



*Fig 5.2: Optical micrographs (x50) of : A) the modified M50 Steel sample; B, C, D and E represents the M50-TAC composites that contains 5, 10, 15 and 20% of MAX powder, respectively.*

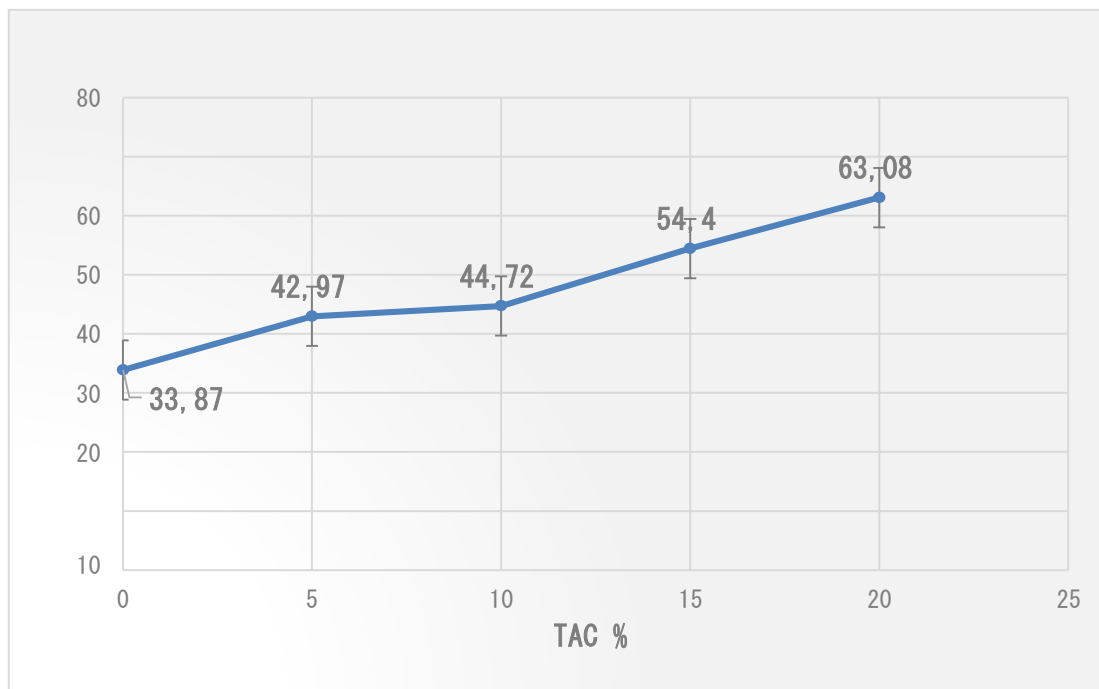


*Fig 5.3: Optical micrographs at higher magnification (x200) of : A) the modified M50 Steel sample; B, C, D and E represents the M50- TAC composites that contains 5, 10, 15 and 20% of MAX powder, respectively.*

**Fig 5.2** shows that the Open porosity varies with TAC additions. The reason for that could be two-fold : i) the volume shrinkage caused by the decomposition of MAX phases [64, 65–66], ii) Probably the fact that a new phase is created. Thus, tiny voids formation between the two is inevitable. Consequently, **Fig 5.3** shows that a new phases appeared (reinforcements) that have different contrasts and sizes along the surface.

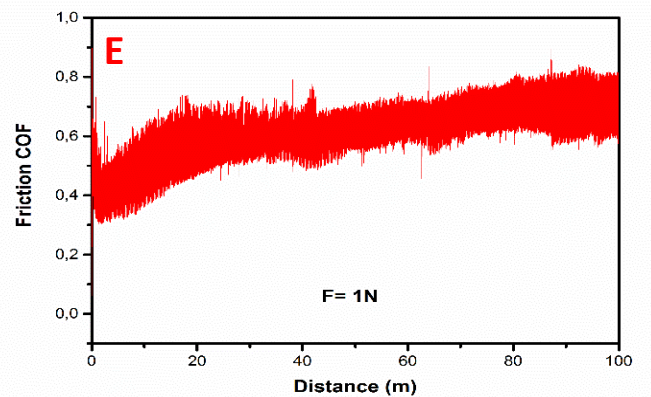
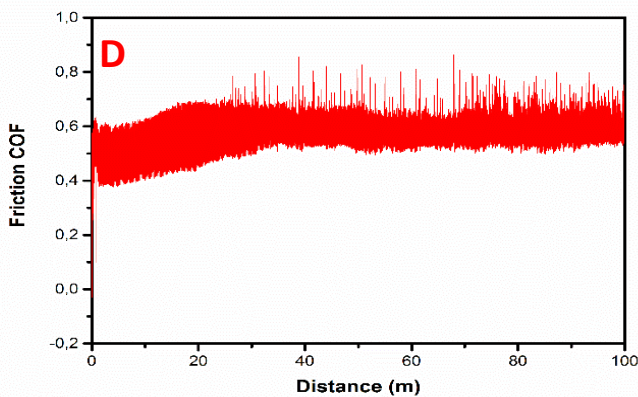
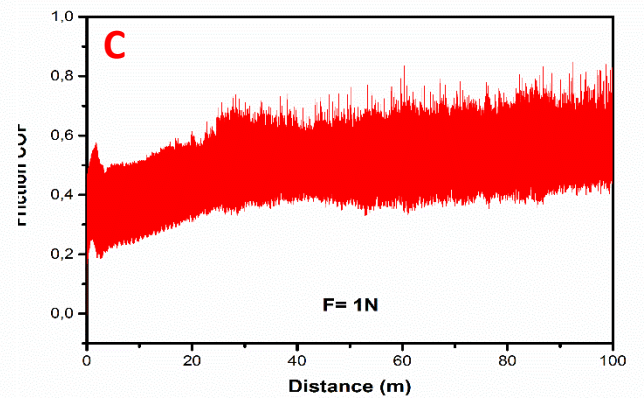
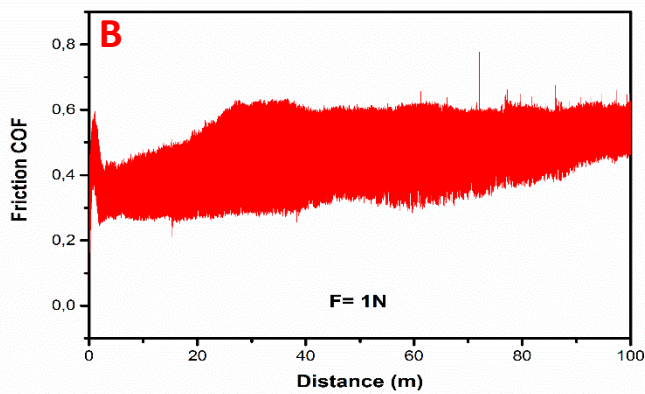
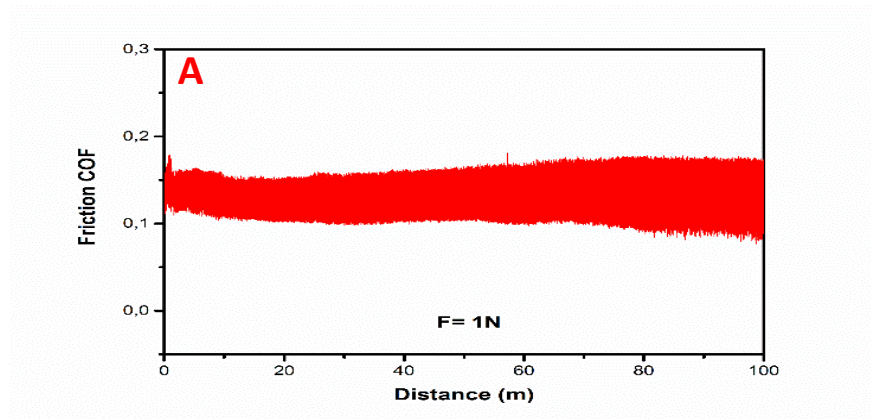
### 5.3. Hardness:

Vickers Hardness of all M50TAC composites exhibited much higher values compared to pure modified M50 steel (Fig.5.4). An addition of 5 wt% of TAC particles resulted in an increased hardness of M50 matrix. With further increase in TAC content (10, 15 and 20 wt %), a further increase in the composite hardness is observed, even though the amount of porosity increased. At certain percentage of TAC we suspect that the porosities will affect the hardness.

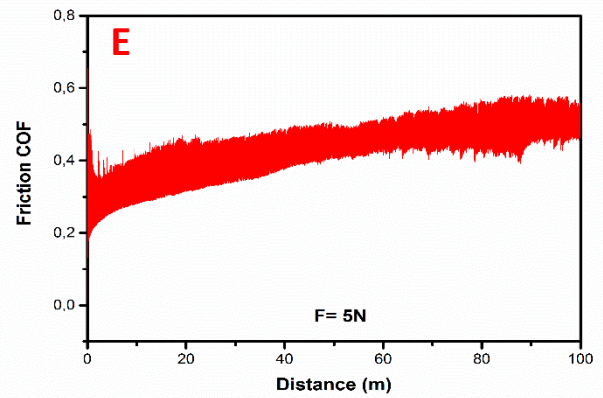
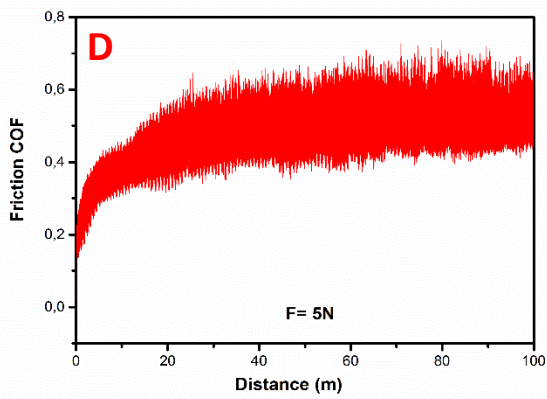
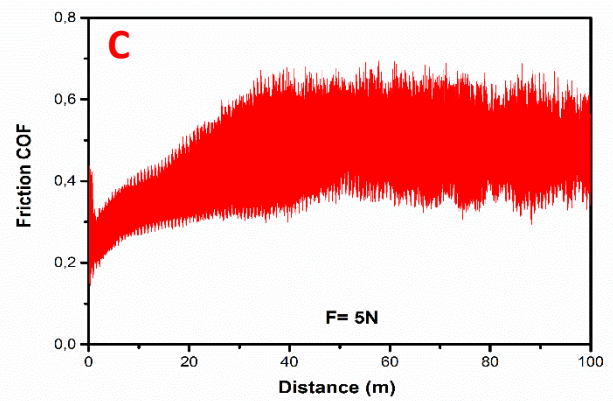
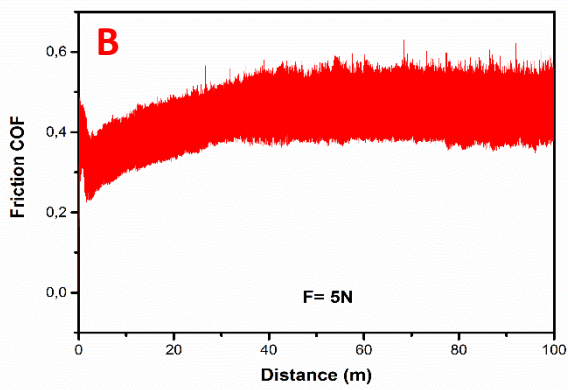
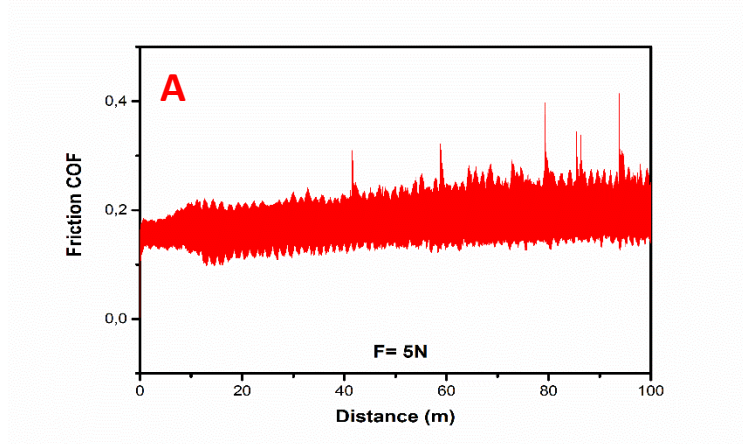


**Fig 5.4: Vickers hardness, of the modified M50 steel and M50-TAC Composites**

## 5.4. Tribology tests:

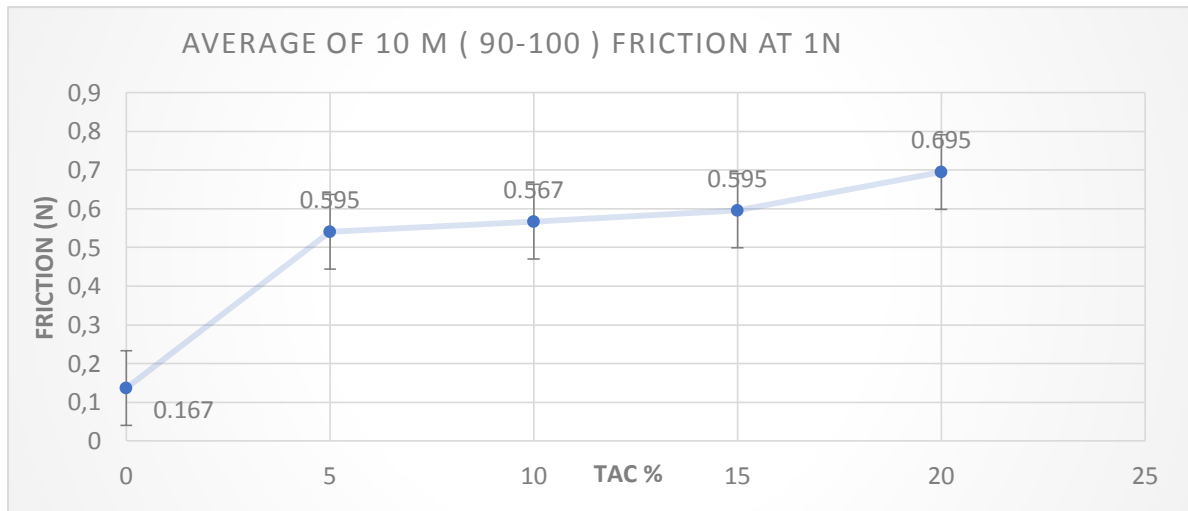


*Fig 5.5: Friction coefficient vs distance under 1N Load of: A) modified M50 steel; and M50-TAC composites (B: TAC5, C: TAC10, D: 1 TAC15, E: TAC20).*



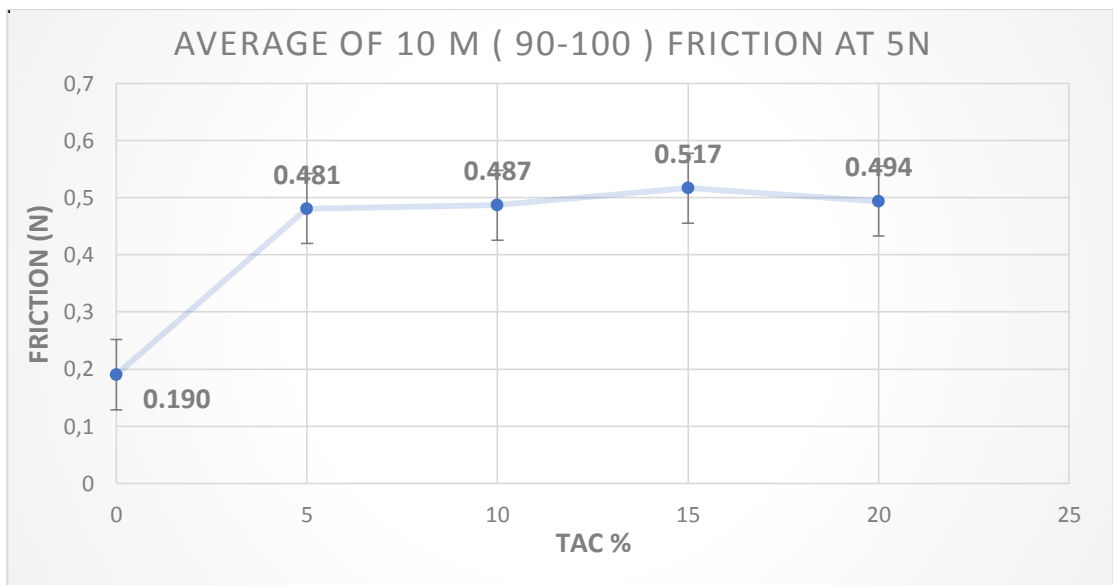
*Fig 5.6: Friction coefficient vs distance under 5N Load of: A) modified M50 steel; and M50-TAC composites (B: TAC5, C: TAC10, D: 1 TAC15, E: TAC20).*





**Fig 5.7: Average of the last 10m of fcof value at 1N of the different samples**

Figure 5.7 show the friction coefficient ( $\mu$ ) values under 1 N load of pure M50 and M50-TAC composites. M50-TAC composites exhibit a significant increase in the friction coefficient compared to pure modified M50 steel due to the amount of TAC added.



**Fig 5.8: Average of the last 10m of fcof value at 5N of the different samples**

After applying 5 N loads (**Figure 5.8**), for pure modified M50 steel we can notice almost the same values in the friction coefficient compared to 1N, on the other hand, for the elaborated composites,  $\mu$

increases with increasing of TAC percentage. It is well established that in MMC's, the reinforcement particles plays an obstacle role that lead to increase fcof values during sliding.

## 5.4 General conclusion:

In this work, the microstructure, hardness, and dry sliding, elaborated from M50 and MAX phases ( $\text{Ti}_2\text{AlC}$ - $\text{Ti}_3\text{AlC}_2$ ) powder mixture, were studied.

The main conclusions can be summarized as follow:

- Porosity variation in m50 composites as a function of reactive TAC volume fractions. It indicates that porosity varies linearly with the increase in TAC content. This result denotes that pressureless sintering of M50 composites becomes difficult as the concentration of TAC in the M50 matrix increases.
- The friction coefficient increases with the increase of TAC (2x higher than pure M50), meaning the reinforced material got higher hardness, this also could means it got better wear resistance.
- We are suspecting that new phases have been created after the heat treatment so we need an observation with SEM to detect them.

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