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THEME

**Elaboration of few-layers Bismuthene and its behaviors
as a reinforcement in Aluminum matrix composite**

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To my best friend Reyane.

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Abstract

The 2D (two-dimensional) materials of Bismuthene are considered as new materials, they show promising properties like low thermal conductivity, semiconducting nature (from quantum-mechanical studies), high power factors, small diverse energy band gap, low carrier density, a structural configuration like carbon nanotubes, large spin-orbit pairing. Using the sonochemical exfoliation method, we attempt for our main objective of this work, which is the elaboration of Bismuthene powder. Secondly, and for better understanding we study his behaviors in a metal matrix composite as a reinforcement, in our case we choosed aluminum. After sintering our composite, while trying to polish it, we noticed that it has undergone through an exothermically reaction. After researching of oxides possibilities that could formed, we suspected that bismuth oxide hydrate was the cause of the pervious reaction.

Résumé

Les matériaux 2D (bidimensionnels) de la bismuthine sont considérés comme de nouveaux matériaux, ils présentent des propriétés prometteuses telles qu'une faible conductivité thermique, une nature semi-conductrice (issue d'études de mécanique quantique), des facteurs de puissance élevés, une petite bande interdite d'énergie diversifiée, une faible densité de porteurs, une configuration structurelle comme les nanotubes de carbone, grand couple spin-orbite. En utilisant la méthode d'exfoliation sonochimique, nous tentons d'atteindre notre objectif principal de ce travail, qui est l'élaboration de poudre de Bismuthene. Dans un second temps, et pour mieux comprendre nous étudions ses comportements dans un composite à matrice métallique comme renfort, dans notre cas nous avons choisi l'aluminium. Après avoir fritté notre composite, en essayant de le polir, nous avons remarqué qu'il avait subi une réaction exothermique. Après avoir recherché des possibilités d'oxydes qui pourraient se former, on s'en doutait que l'hydrate d'oxyde de bismuth était la cause de la réaction perméable.

تعتبر المواد ثنائية الأبعاد (ثنائية الأبعاد) للبزموتين مواد جديدة ، فهي تُظهر خصائص واعدة مثل الموصلية الحرارية المنخفضة وطبيعة أشباه الموصلات (من دراسات ميكانيكا الكم) وعوامل الطاقة العالية ، وفجوة صغيرة متنوعة في نطاق الطاقة ، وكثافة منخفضة للحامل ، و التكوين الهيكلي مثل الأنابيب النانوية الكربونية ، اقتران كبير في مدار الدوران. باستخدام طريقة التقشير الكيميائي .

نحاول تحقيق هدفنا الرئيسي من هذا العمل ، وهو إعداد مسحوق البزموتين. ثانيًا ، من أجل فهم أفضل ، ندرس سلوكياته في مركب مصفوفة معدنية مقوي

في حالتنا اخترنا الألومنيوم. بعد تلييد المركب الخاص بنا ، أثناء محاولتنا تلميعه ، لاحظنا أنه قد خضع لتفاعل طارد للحرارة ، بعد البحث عن احتمالات الأكاسيد التي يمكن أن تتشكل ، نتوقع أن هيدرات أكسيد البزموت كان سبب التفاعل المنفذ

General Introduction:

In materials science, our focus devoted to the design, creation and fundamental understanding of materials by understanding the relationships between processing, structure, properties and performance in engineering applications, and the science is constantly evolving and one of his recent discoveries is 2D materials.

2D materials have historically been a big part of science research materials, because of their extraordinary properties. The discovery of graphene in 2004, by NOVOSELOV and GEIM, and its exceptional properties called into question these materials in current research issues. Indeed, among these 2D materials, Bismuthene has incredible properties such as low thermal conductivity, semiconducting nature (from quantum-mechanical studies), high power factors, small diverse energy band gap, low carrier density, a structural configuration like carbon nanotubes, large spin-orbit pairing. These extraordinary properties pave the way for many applications, research on 2D materials has greatly accelerated in recent years and many new 2D materials have was discovered. Mention may be made, for example, stanene, germanium, germanene or even silicon, silicene.[1]

Powder metallurgy is a term covering a wide range of ways in which materials or components are made from metal powders. PM processes can avoid or greatly reduce the need to use metal removal processes, thereby significantly reducing yield losses in manufacturing and often leading to lower costs [1].

A metal matrix composite is a composite material with fibers or particles disseminated in a metallic matrix, such as copper, aluminum, or steel. The secondary phase is typically a ceramic or another metal. Compared to monolithic metals, MMCs have: Higher strength-to-density ratios, Higher stiffness-to-density ratios, better fatigue resistance, better elevated temperature properties, Higher strength, Lower creep rate, Lower coefficients of thermal expansion, better wear resistance.[2]

Our graduation project consists on elaboration of few layers Bismuthene, and studying his behavior in a metal matrix composite.

CHAPTER I: Introduction of powder metallurgy

I.1. Introduction:

The history of powder metallurgy and the art of metal and ceramic sintering are intimately related to each other. Sintering involves the production of a hard solid metal or ceramic piece from a starting powder. The ancient Incas made jewelry and other artifacts from precious metal powders, though mass manufacturing of PM products did not begin until the mid or late 19th century [3]. Modern powder metallurgy technology commenced in the 1920s with the production of tungsten carbides and the mass production of porous bronze bushes for compartments. During the Second World War, extra growth took a place in manufacture of a great variety ferrous nonferrous materials, including many composites and a steady growth period advanced during the postwar year until the early 1960s. Since then, growth of P/M has extended more rapidly mainly because of three potential reasons- Economical processing, unique properties and captive processes. So, by definition P/M is a process for forming metal parts by heating compacted metal powders to just below their melting points. In other words, PM is a metal shaping process that creates near-net parts from powdered metal.



I.1. Figure All of these components were made using powder metallurgy.

I.2. Why Powder Metallurgy?

The powder metallurgy process provides a host of advantages over competing metalworking technologies. These all add up to part-to-part uniformity for improved product quality, shape and material flexibility, application versatility, and cost effectiveness.

➤ Advantages of the Powder Metallurgy Process

- Minimizes machining by producing parts at, or close to, final dimensions;
- Minimizes scrap losses by typically using more than 97% of the starting raw material in the finished part;
- Permits a wide variety of alloy systems;
- Produces good surface finish;
- Provides materials which may be heat treated for increased strength or increased wear resistance;
- Provides controlled porosity for self-lubrication or filtration;
- Facilitates manufacture of complex or unique shapes which would be impractical or impossible with other metalworking processes;
- Is suited to moderate- to high-volume component production requirements;
- Offers long-term performance reliability in critical applications;
- Is cost effective.

I.3. The Powder Metallurgy Process:

The basic Powder Metallurgy process for structural press and sintered components. Powder Metallurgy comprises a family of production technologies, which process a feedstock in powder form to manufacture components of various types. These production technologies generally involve all or most of the following process steps:

- **Powder production**

Virtually all iron powders for PM structural part production are manufactured using either the sponge iron process or water atomization. Nonferrous metal powders used for other PM applications can be produced via a number of methods.

- **Mixing of powders**

This can often involve the introduction of alloying additions in elemental powder form or the incorporation of a pressing lubricant.

- **Forming of the mixed powder into a compact**

The dominant consolidation process involves pressing in a rigid toolset, comprising a die, punches and, possibly, mandrels or core rods. However, there are several other consolidation processes that are used in niche applications.

- **Sintering**

Sintering is defined as a heat treatment with the aim of consolidating the particles of the powders without reaching the melting temperature of one of the constituents which can be accompanied by a pressure in order to have a density almost at the same Theoretical density. This heat treatment carried out at a temperature below the melting point of the main component of the material allows the generation of strong bonds between the particles by diffusion of the material. In most cases, sintering is accompanied by a reduction in the dimensions of the part and therefore a reduction in the volume of the part, known as densification; is this a technique used to obtain experimental results approach to theoretical results.

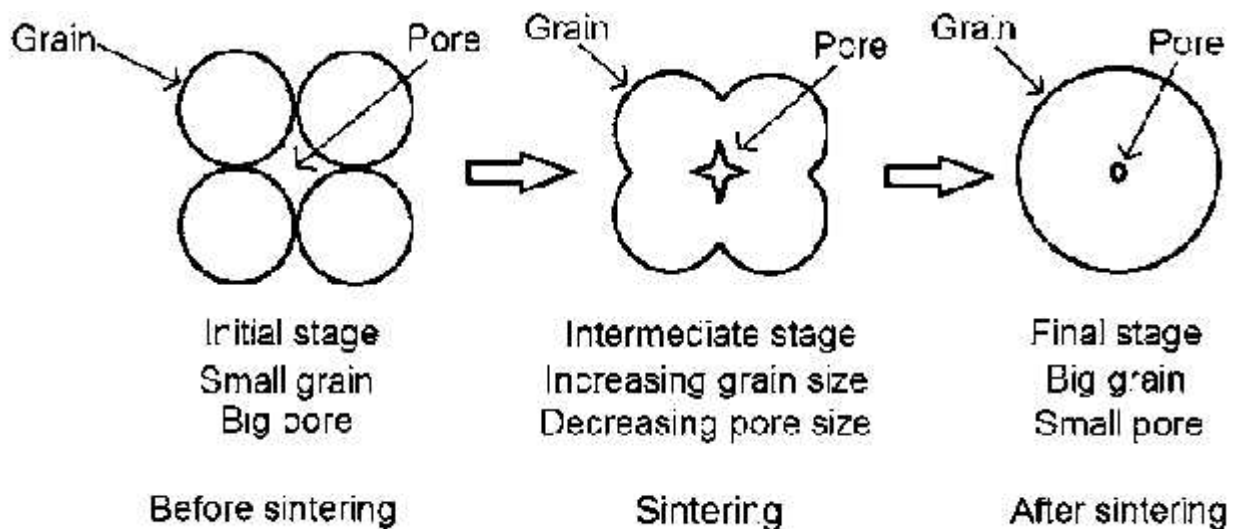


Figure I.2. A model explaining the sintering process

I.4. Types of sintering:

I.4.1 Solid phase sintering:

During solid phase sintering, all phases are in the solid state at the start of the investigations into the theory of sintering, the main research effort has been concentrated on model experiments, such as sintering of a sphere to another, a sphere has a plate a number of wires against each other.

I.4.2. liquid phase sintering:

Liquid phase sintering for which the quantity of liquid which appears is just sufficient to allow the rearrangement of the particles, to form liquid bridges between the grains and thus to bring them into contact with each other. This type of sintering is polyphaser; it involves the reaction between several chemical constituents. The properties of the ceramics obtained by these last two sintering processes are conditioned by the nature of the liquid phase which forms the grain boundaries on cooling. Densification, if it occurs, requires other mechanisms which will be detailed later.

If the material after sintering has the same chemical composition as the initial powder, this is referred to as non-reactive sintering; this is the most general case. In some special cases, the initial powder may be composed of a mixture of two (or more) chemical compounds; in this case, the heat treatment allows, on the one hand to form a new chemical compound, on the other hand to densify this new material; this is then referred to as reactive sintering. [4]

I.5. Sintering processes:

I.5.1. Hot isostatic pressing technique:

The hot isostatic pressing (HIP) technique, is based on the PASCAL principle, "the pressure applied to a static fluid or a gaseous medium is distributed uniformly in all directions and the forces exerted on the surfaces."

It makes it possible to densify or synthesize powders in order to have massive pieces of more or less complex sizes and shapes with a uniform microstructure as well as grain enlargement.

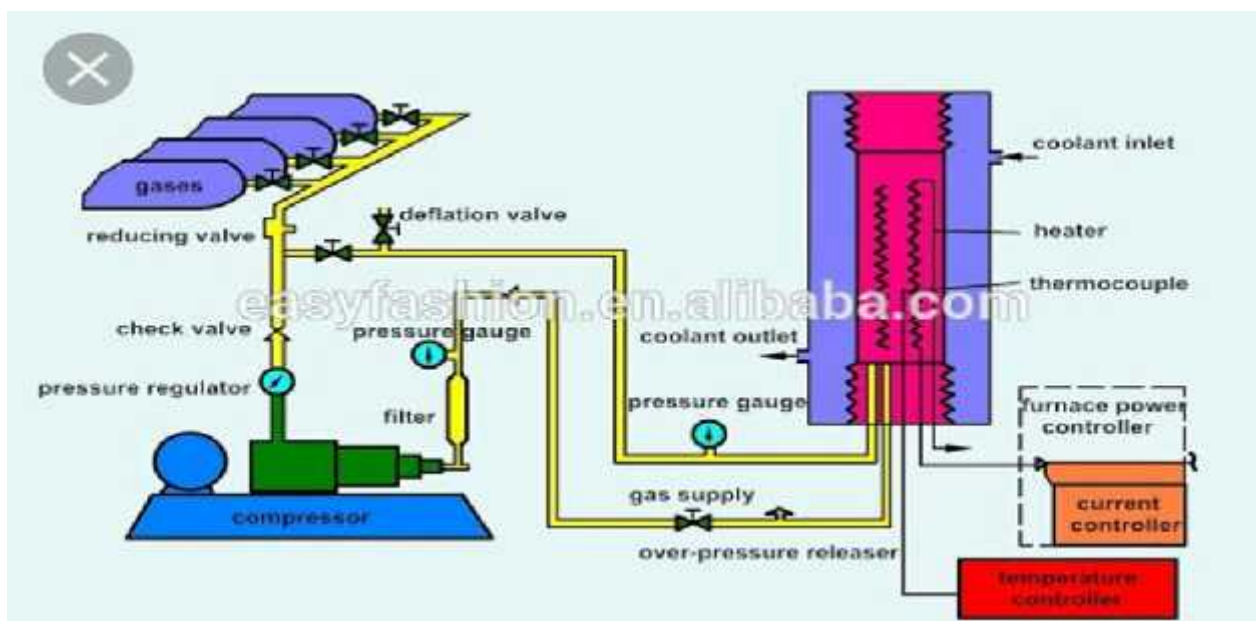


Figure I.3 hot isostatic press machine

I.5.2. SPS spark plasma sintering technique:

During SPS, heating is not imparted from the outside like HP, but rather generated by the Joule effect on the "mold-piston-sample" assembly. Depending on the intensity of the pulsed current, it is possible to perform very short thermal cycles of the order of a few minutes. Furthermore, the application of pressure which can reach 100mpa makes it possible to reduce the thermal cycles of sintering, which considerably reduces the growth phenomenon and maintains nanometric grain sizes.[4]

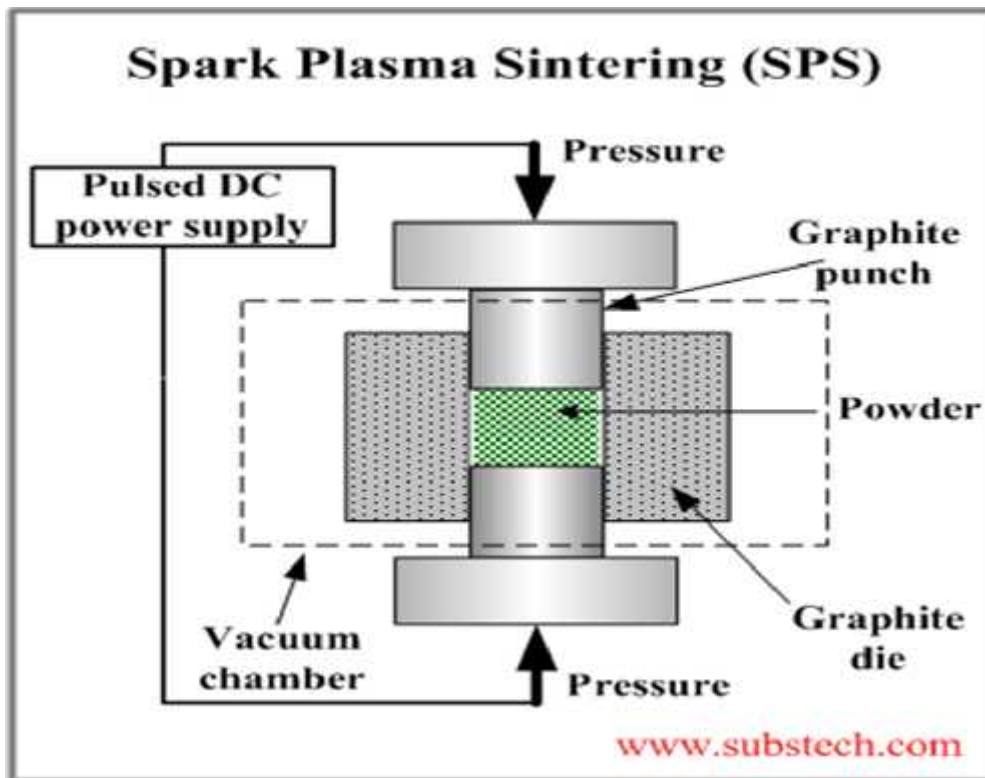


Figure I.4 SPS MACHINE

I.5.3. Technique of unidirectional hot pressing:

The unidirectional hot pressing (HP, "hot pressing"), is a technique in which the material is compressed at high temperature, in an axial uniform manner, in a simple mold.

After mixing it, the powder is cold compacted beforehand in order to have a solid that is easier to handle, then placed in a mold closed by a piston and it is the rise in temperature which takes place at a certain speed and the pressure applied mechanically on the piston, in particular at a well-defined speed (chosen value) which results in a material in its final state. The hot-pressing technique is particularly useful for materials with high granular growth rate.[4]

I.6. Introduction of Aluminum powder metallurgy:

Aluminum (Al), also spelled aluminium, chemical element, a lightweight silvery white metal of main Group 13 (III a, or boron group) of the periodic table. Aluminum is the most abundant metallic element in Earth's crust and the most widely used nonferrous metal. Because of its chemical activity, aluminum never occurs in the metallic form in nature, but its compounds are present to a greater or lesser extent in almost all rocks, vegetation, and animals. Aluminum is concentrated in the outer 16 km (10 miles) of Earth's crust, of which it constitutes about 8 percent by weight; it is exceeded in amount only by oxygen and silicon. The name aluminum is derived from the Latin word *Alumen*, used to describe potash alum, or aluminum potassium sulfate, $KAl(SO_4)_2 \cdot 12H_2O$.

I.6.1. History of aluminum powder metallurgy:

The United States government regulations have compelled automobile manufacturers improve fuel efficiencies and reduce emissions. To help meet these regulations, the automotive manufactures are exploring the use of powdered aluminum alloys and powdered aluminum metal matrix composites as substitutes for some incumbent ferrous materials in drive train components. The intrinsic properties of aluminum, for example, density and melting temperature, offer attractive incentives for weight reduction and lower metal working temperatures, which translates to lower cost. However, the automotive makers are hesitant to be solely dependent on aluminum P/M processing. For instance, in order for aluminum P/M parts to be practical for use in automobile drive train components, adequate mechanical properties, e.g., fatigue properties, are necessary. Another major issue reducing current costs of aluminum powder production and consolidation. Since the early 1960's, aluminum P/M processing includes aluminum powder production by gas (air) atomization and powder consolidation into billets for extruding, forging, or rolling into plate or sheet [2]. A major problem with the consolidation of the aluminum powder, particularly during pressing and sintering operations, was due to the obtrusive and intrinsic oxide film on aluminum powder particle surfaces. In the early to mid 1980's, when high-strength aluminum P/M alloys were consolidated into aerospace components, changes transpired in gas atomization practices to reduce oxide film thicknesses on aluminum powders. Inert gas atomization was deemed an alternate method to air atomization and successfully reduced surface oxide film thicknesses.

Today, in addition to aerospace uses, aluminum P/M has the opportunity to penetrate broadly into high volume production of automotive components. To enhance the viability of the aluminum powder production and consolidation technologies, the process needs further refinement to reduce manufacturing costs. An investment of time and money should lead to improvements of the current aluminum P/M technology. The expected outcome of these advances would be improvements in the quality of P/M components and reduction of manufacturing costs. To facilitate the necessary research, consortia and conferences provide a forum whereby technical information can be exchanged between the powder producers, part manufactures, and the automotive industry. Throughout my research, personal active involvement within the United States Automotive Manufacturers Partnership (USAMP) consortium and powder metallurgy conferences have facilitated the opportunity to present

various aspects of the aluminum P/M research presented in this dissertation. A literature survey of aluminum P/M shows that many researchers have explored aluminum alloy powder production practices, aluminum alloy powder characteristics, and aluminum alloy powder consolidation methods. However, there is significantly less information available about pure aluminum powders. Yet, some researchers discuss their results with the assumption that the alloy powders behave as pure aluminum powders and do not fully consider the resultant effects of the alloy elements. A benefit of studying pure aluminum P/M is that the results are unambiguous. The outcome from this research is an extensive database upon which one can more accurately assess the results observed in the aluminum alloy powders. Therefore, the objective of this research was to broaden the knowledge base of aluminum P/M through a fundamental study of processing pure aluminum powder. To achieve this objective, four topics were investigated: assessment of current gas atomization methods used to produce aluminum powder; powder characterizations of the resulting pure aluminum powders; consolidation of the pure aluminum powders; microstructure and mechanical property analysis of the consolidated powder samples.[5]

CHAPTER II: 2D Materials

II.1. Introduction :

The isolation of graphene in 2004 from graphite was a significant moment for the “birth” of a field: Two-dimensional (2D) materials. However, the term single-layer materials or 2D materials refers to crystalline solids consisting of a single layer of atoms. First 2D material graphene was discovered [06] in 2004 by two researchers at the University of Manchester, Prof. Andre Geim and Prof. Kostya Novoselov. They used the scotch-tape method to separate a single layer of graphene from graphite. 2D Materials have fascinated widespread attention due to their extraordinary physical and chemical properties. The rapid and prosperous development of graphene stimulates numerous research interests on other 2D materials. More than one thousand structures of 2D materials have been predicted to be easily exfoliated to monolayers or multilayers with fascinating physical properties, forming a large family of 2D materials [07]. The booming synthetic methods established from graphene have brought experimental realizations of dozens of novel 2D crystals. Monolayer MoS₂ and hexagonal boron nitride (h-BN) have been extracted at an early stage and have recently received much attention. Some graphene analogs such as black phosphorene [08], borophene, silicene, germanane, stanene, antimonene, bismuthene and tellurene have been synthesized in the past few years. Although these 2D materials have an atomic layer structure similar to that of graphene, their physical properties are distinct from those of graphene. Thus, these 2D materials can act as complementary materials and have the potential for broader applications. For example, unlike graphene, phosphorene has a strong in-plane structural anisotropy, leading to a significant dependence of the material properties on its orientation [09]. For electronic properties, graphene has a direct zero band gap and exhibits a certain metallicity. Other 2D crystals have a large variety of band structures. The direct band gaps of h-BN, MoS₂, and WSe₂ allow them to be promising materials for optical devices, transistors, phototransistors, and photodetectors. The metallic electronic character possessed by borophene and VS is essential for electronic and energy storage applications. In addition, stanene, as a 2D topological insulator, is theoretically predicted to display superconductivity at the edges. A large number of 2D material family members could satisfy variant requirements for a huge diversity of applications. The structure and mechanics of 2D materials play important roles in manufacturing, integration, and performance for their potential applications.[10]

During the synthesis of 2D materials, several types of defects are unavoidably generated. For example, during the chemical vapor deposition (CVD) process for the large-area growth of graphene, many isolated grains from different nucleation sites sew up into uniform structures, leading to the formation of grain boundaries (GBs) between neighboring grains with a misorientation. Additionally, graphene’s irradiation or chemical treatment can generate various point defects, such as dislocations, vacancies, and functionalized groups. The majority of experimental studies have shown that these defects in 2D crystals significantly affect their physical, chemical, and mechanical properties. In particular, it has been demonstrated that defects can tailor the properties of 2D materials via the controlled arrangement of defects. Therefore, the concepts of defect engineering and topological design have emerged and been used to achieve tunable properties of 2D materials.

(Figure II.2) shows the structural models of the experimentally known 2D graphene analogues of group-III, IV and V. All these post-graphene layered materials have a corrugated, puckered or buckled structure. Some of these materials have also been realized as graphene-like flat sheets by epitaxial growth. Experimental investigation of many of these elemental 2D materials is still in their infancy, but the available theoretical and the experimental reports are encouraging. In this review, we present the recent progress on the synthesis, structure, properties and applications of these elemental 2D materials.

II.2. Classification and Atomic Structures:

The 2D material family has prolonged to more than one thousand members based on theoretical predictions. Up to the present time, tens of these materials have been synthesized experimentally [11]. Generally, 2D materials can be categorized into four types (including graphene family, Xenes, chalcogenides, and 2D oxides) according to their components and atomic structures, as shown in (Figure II.3).

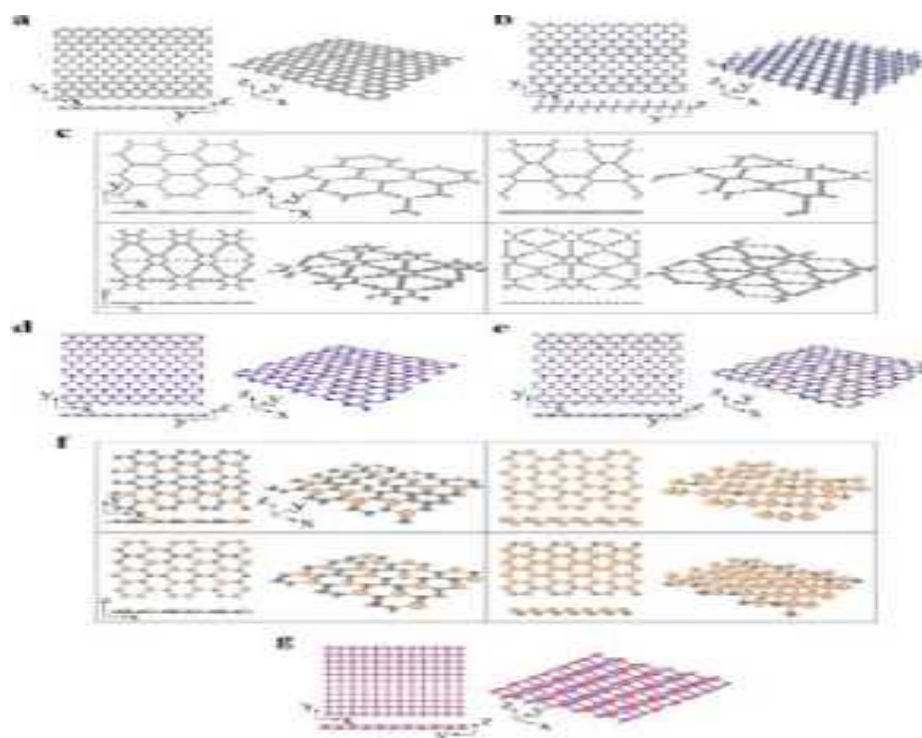


Figure II.3. The graphene family: (a) graphene (gray atom represents C), (b) CX ($X = \text{H}, \text{F}, \text{Cl}$; gray and blue atoms represent C and X, respectively), (c) graphyne (-graphyne, -graphyne, -graphyne and 6,6,12-graphyne from left to right, top to bottom; gray atom represents C), (d) h-BN (red and blue atoms represent B and N, respectively), (e) BCN; red,

gray and blue atoms represent B, C and N, respectively), (f) SixC1-x(x = 2/10, 5/6, 2/6, 14/18 from left to right, top to bottom; gray and yellow atoms represent C and Si, respectively), (g) TiC; red and blue atoms represent C and Ti, respectively).

II.2.1. Graphene and hexagonal boron nitride:

Graphene is a covalently-bonded hexagonal lattice of carbon atoms just one atom thick (about 0.14 nm). It is a semimetal (its conduction and valence bands both touch). Graphene's unique band structure means that electrons move through it at extremely high speeds (about 1/300 the speed of light), giving it fascinating properties - such as unparalleled thermal conductivity. Optically transparent, graphene absorbs only 2% of incident visible light and has the highest tensile strength of any material. A single monolayer of graphene, just 0.3 nm thick, would be able to support the weight of a football [12]. (Fig of football on monolayer) Hexagonal boron nitride (h-BN) is an isomorph of graphene (has the same crystallographic appearance), but has boron and nitrogen atoms instead of carbon. In contrast to graphene, it is a wide-bandgap insulator.

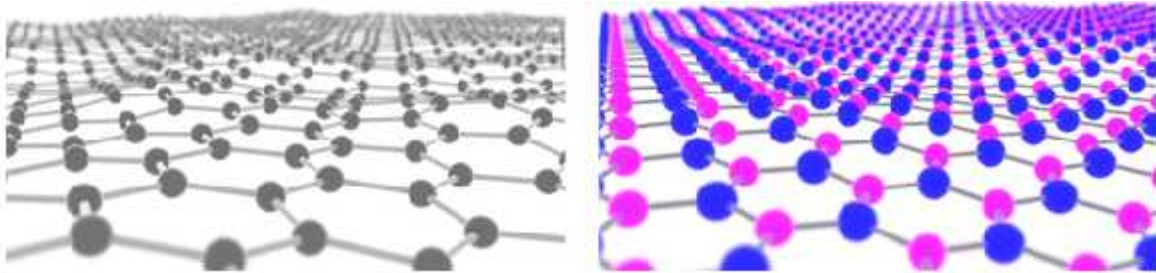


Figure II.4: Graphene (left) and hexagonal boron nitride (right) are both 2D materials. Despite both having a hexagonal lattice structure, they have very different physical properties. Graphene is an excellent electrical conductor while hexagonal boron nitride is a wide-bandgap insulator.

II.2.2. Transition Metal Dichalcogenides (TMDCs):

Transition metal dichalcogenides (commonly referred to as TMDCs) have the chemical formula MX_2 , where M is a transition metal (such as molybdenum (Mo) or tungsten (W)) and X is a chalcogen (such as sulfur (S), selenium (Se) or Tellurium (Te)). Bulk TMDCs are van der Waals materials with each layer being three atoms thick, consisting of the metal layer sandwiched between two chalcogenide layers. TMDCs can take various crystal structures. The most common is the 2H-phase with trigonal symmetry, which results in semiconducting characteristics such as possessed by MoS_2 , WS_2 , $MoSe_2$. These semiconductors have an indirect bandgap when in the bulk. For monolayers, the bandgap becomes direct and in the visible spectrum, making them attractive for optoelectronics. Charge mobilities of ~ 100 - 1000 $cm^2V^{-1}s^{-1}$ make them a popular choice for 2D transistors. Another possible structure is the metallic 1T phase, which is the most stable polymorph of WTe_2 .

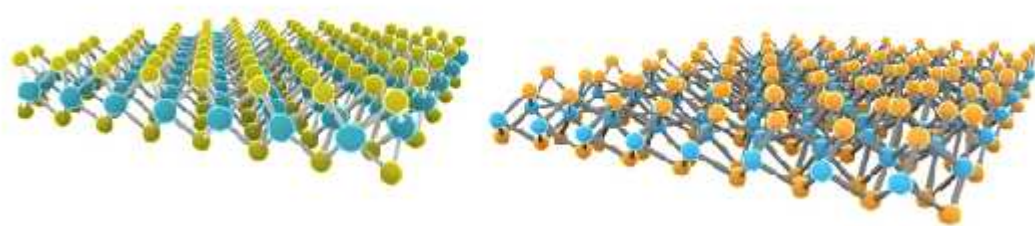


Figure II.5: *Left: Molybdenum disulphide (MoS_2). Right: Tungsten ditelluride (WTe_2). Both are 2-dimensional transition metal dichalcogenides. MoS_2 is most commonly found in the semiconducting 2H phase, while WTe_2 prefers to adopt the metallic 1T phase.*

II.2.3. Phosphorene:

Phosphorene is a single layer of black phosphorus – a layered, stable allotrope of elemental phosphorus. It is a direct bandgap semiconductor with a puckered honeycomb structure. The bandgap can be tuned throughout the visible region by stacking layers on top of each other. It has good charge mobility ($\sim 1000 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$), therefore making it suitable for optoelectronic devices and transistors. Phosphorene's corrugated structure means that its properties can vary significantly, depending on which direction the material is measured along.

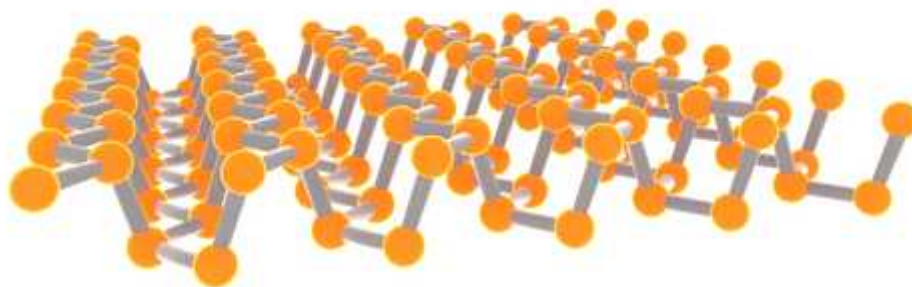


Figure II.6: *Phosphorene (also known as 2D black phosphorus) is a 2D semiconductor that is a promising material for transistors.*

II.2.4. Xenes:

Monolayers of silicon (silicene), germanium (germanene) and tin (stanene), are collectively known as Xenes (following the naming convention of graphene). They have a hexagonal structure similar to graphene, but are buckled to varying degrees. Unlike graphene, they cannot be exfoliated from bulk material and must be epitaxially grown on a substrate, and generally retain a strong interaction with that substrate. While still very much in their infancy, potential applications range from field-effect transistors to topological insulators.

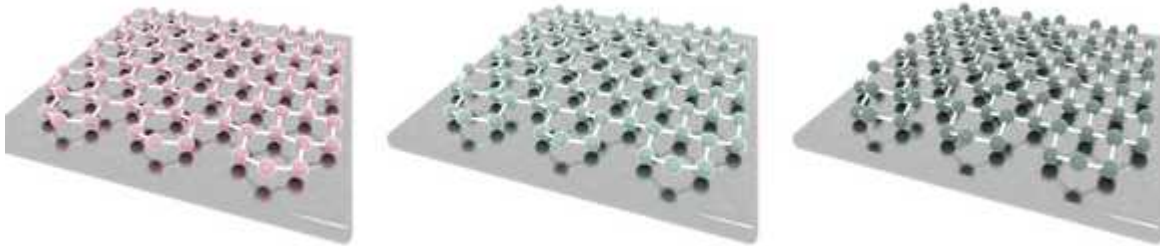


Figure II.7: Silicene (left), germanene (middle) and stanene (right) have buckled hexagonal structure

Recently, 2D analogues of antimony and bismuth (antimomene and bismuthine respectively) have also been grown. Bismuth shows potential for magneto-electronic applications [13].

II.3. Bismuth:

Bismuth (Bi), the most metallic and the least abundant of the elements in the nitrogen group (Group 15 [Va] of the periodic table). Bismuth is hard, brittle, lustrous, and coarsely crystalline. It can be distinguished from all other metals by its colour gray-white with a reddish tinge [17].

ElementProperties:

- Atomic number: 83
- Atomic weight ; 208.98040
- Melting point : 271.3 °C (520.3 °F)
- Boiling point : 1,560 °C (2,840 °F)
- Density: 9.747 gram/cm³ at 20 °C (68 °F)

II.3.1. History of Bismuth:

Bismuth metal has been known since ancient times; it was one of the first 10 metals to have been discovered. The name bismuth dates from around the 1660s and is of uncertain etymology; it possibly comes from obsolete German Bismuth, Wismut, Wissmuth (early 16th century), perhaps related to Old High German hwiz ("white"). [13]. Bismuth was accepted as a specific metal by the middle of the 18th century, and the Frenchman Claude-François Geoffroy published works on its chemistry in 1739 by the German chemist Johann Heinrich Pott and in 1753 [17].

II.3.2. Properties of Bismuth:

Bismuth is a rather brittle metal with a somewhat pinkish, silvery metallic lustre. Bismuth is the most diamagnetic of all metals (i.e., it exhibits the greatest opposition to being magnetized). It is hard and coarsely crystalline. It undergoes a 3.3 percent expansion when it solidifies from the molten state. Its electrical conductivity is very poor, but somewhat better in

the liquid state than in the solid. With respect to thermal conductivity, it is the poorest of all metals except mercury [17].

II.3.4. Introduction of Bismuthene:

Bismuth is currently the heaviest element used in the creation of a 2D material. Bismuthene is a single atomically thin layer of bismuth atoms arranged in a hexagonal array. Like many 2D materials, bismuthene has been grown on other substrates. In the case of bismuthene, it is grown onto silicon carbide substrates. Silicon carbide substrates have shown the most growth promise because the structure of the silicon carbide molecules directs the growth into the regular hexagon structure seen by many 2D materials. However, unlike graphene, the deposition process bonds the bismuth atoms to the silicon carbide sheet, giving rise to some valuable electronic properties. Unlike graphene and other 2D materials, bismuthene is a room-temperature topological insulator, not a conductor. However, when graphene is layered onto substrates, it can also act in a similar insulating manner. Bismuthene is a novel material because most topological insulators operate at temperatures well below freezing. As such, bismuthene is considered to have a high stability compared to many other 2D materials.

How do we make 2D materials?

It is possible to take any material and thin it down (until it has a thickness of only a few atoms) to create a 2D material. However, many materials (e.g. diamonds) have chemical bonds oriented in 3-dimensions, so thinning the material requires cutting these bonds – leaving them ‘dangling’. A 2D material created in this way will have a high density of dangling bonds, which are chemically and energetically unstable, and can force the material to rearrange its structure to lower its surface energy. Another allotrope of carbon – graphite – has strong chemical bonds only along planes within the bulk material. These planes are stacked on top of each other and held together by weak van der Waals interaction, and so can be separated without leaving any dangling bonds. In the case of graphite, a single plane is called graphene. Most of the 2D materials being studied therefore belong to the broader class of layered materials (or van der Waals materials).

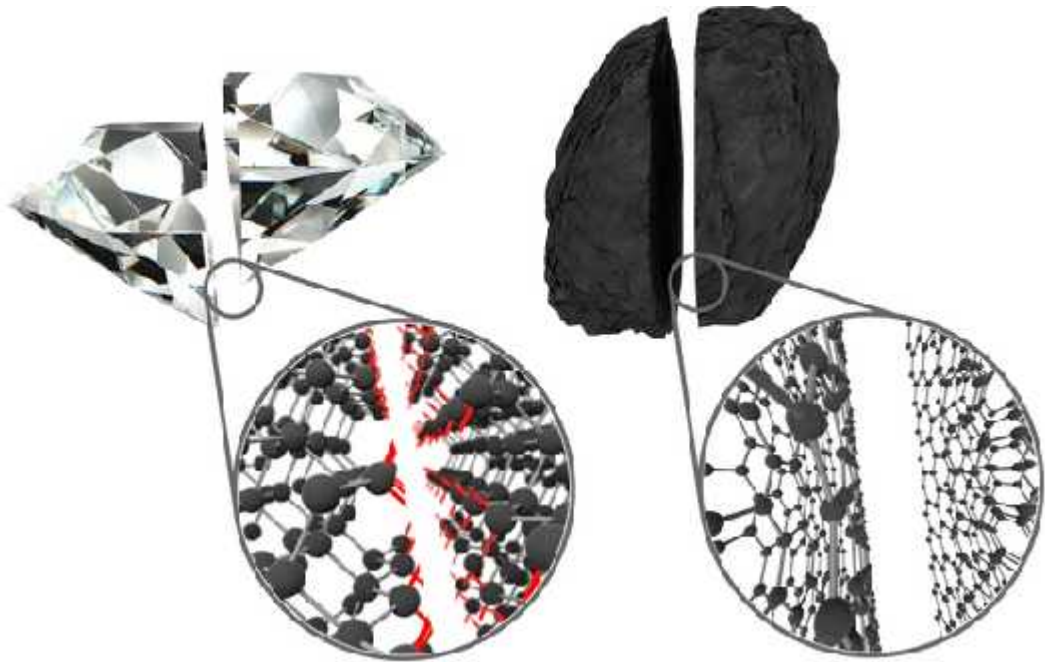


Figure II.8: Each carbon atom in diamond (left) has bonds extending in 3 dimensions - meaning that when diamond is cut in any orientation, some of these bonds must be broken and are left 'dangling' (shown in red). The atoms in graphite (right) have bonds extending in only 2 dimensions, so when it is cut in an orientation parallel to the bonds, none of them are broken.

There are two methods for making 2D materials:

- a) Top-down (start with a bulk material and make it thinner)
- b) Bottom-up (start with the atomic ingredients and assemble them together)

Within each of these approaches are several subcategories, each with their own advantages and disadvantages - explained below.

A) Top-down:

Mechanical exfoliation – Commonly known as the ‘Scotch-tape method’, it was first used to create monolayer graphene. A piece of sticky tape is applied to the surface of a layered material and then peeled off, taking flakes (consisting of a small number of layers) with it. The tape can then be pressed onto a substrate to transfer the flakes for study. The monolayer yield of this process is low (the flakes obtained are mostly multilayer), with no control over the size and shape. However, the size of monolayer flakes that can be produced is reasonable (from a few microns up to ~100 microns) and the quality of monolayers is excellent - with very few defects due to the lack of chemical processing involved. It is also a suitable technique for all van der Waals materials. For these reasons, mechanical exfoliation remains popular for lab-based studies, but it is not scalable for integration into new technologies.

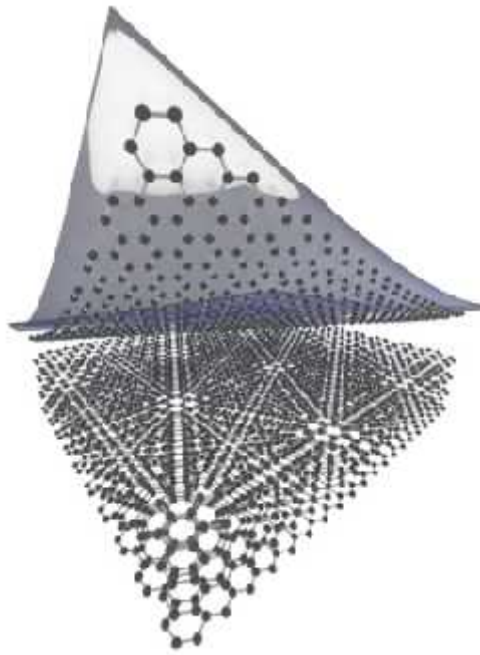


Figure II.09: Mechanical exfoliation involves peeling successive layers from a Van der Waals material using a tape.

B) Liquid exfoliation:

Another mechanical method, liquid exfoliation involves using an organic solvent as a medium to transfer mechanical force to the layered material (often in the form of a powder) suspended in the liquid. Sonication causes tensile stress to be applied to the layers, forcing them apart. To improve monolayer yield, variations exist - such as introducing reactive ions (between the material layers that create hydrogen bubbles) that push the layers apart, or that rapidly mix the solution to create additional shear force on the layers. This method is highly scalable but has several drawbacks. The monolayer yield is again generally low, and the flakes are often less than 100 nm in size (due to the applied forces breaking them apart). The resultant flakes may also potentially have a high density of defects and residual solvent when removed from solution, making them unsuitable for many optoelectronic applications.

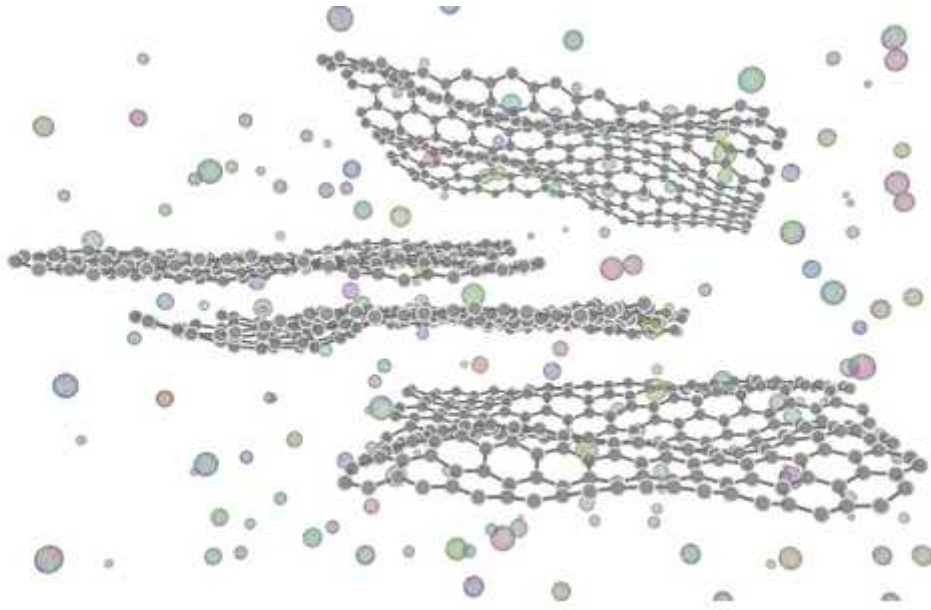


Figure II.10: Liquid exfoliation often uses bubbles to force layers apart.

Why are 2D materials different from bulk materials?

This comes down to three reasons:

a-Removal of van der Waals interactions:

A layered bulk material consists of many covalently-bonded planes held together by weak van der Waals interactions. When a force is applied to a material, these van der Waals forces can be easily overcome and the material breaks making it seem weak. Conversely, the covalent bonds that hold the atoms together in the layers are actually very strong. A monolayer will only have covalent bonds. By removing the ‘weak links’ from the material, it appears to become much stronger. For example, graphene has a tensile strength 1000 times greater than graphite, and while a graphite pencil can be easily broken, graphene is over 100 times stronger than steel.

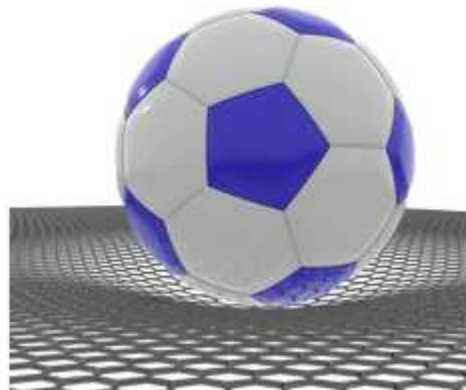
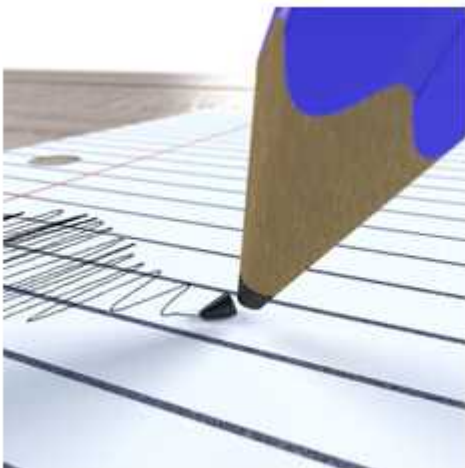


Figure II. 11: Graphite (left) can be easily broken because of its weak interplane Van der Waals forces, while graphene (right) has only covalent bonds and so it extremely strong - a monolayer is strong enough to support a football.

b- An increase in the ratio of surface area-to-volume:

The surface area-to-volume ratio of a material defines how much of it is exposed to its environment. This is important for chemical reactions – the more reactant that is in contact with the material, the faster the reaction can occur, so 2D materials tend to be more reactive their bulk counterparts. It also makes 2D materials more sensitive to their surroundings, an effect that is exploited for sensors based on 2D materials.

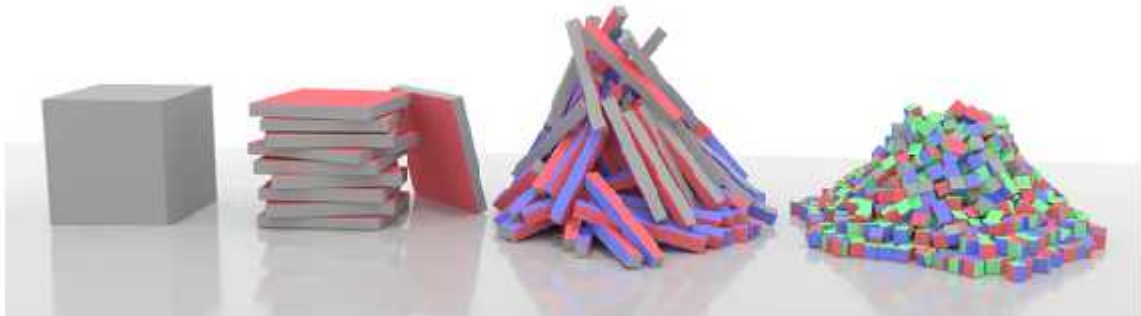


Figure II.12: When an object is divided into smaller components, its total surface area increases. From left to right: a bulk object is divided into 2D sheets - exposing the red surfaces, and the sheets are divided into 1D rods - further exposing the blue surfaces. Finally, the rods are divided into dots - exposing additional green surfaces.

c- Confinement of electrons in a plane:

The electronic and optical properties of a material depend upon its electronic band structure. This describes how electrons move through the material, and is a result of the periodicity of its crystal structure. When a material goes from bulk to 2D, the periodicity is removed in the direction perpendicular to the plane, which can greatly change the band structure. The modified band structures are responsible for the extremely high conductivity of graphene and the fluorescence of monolayer MoS₂.

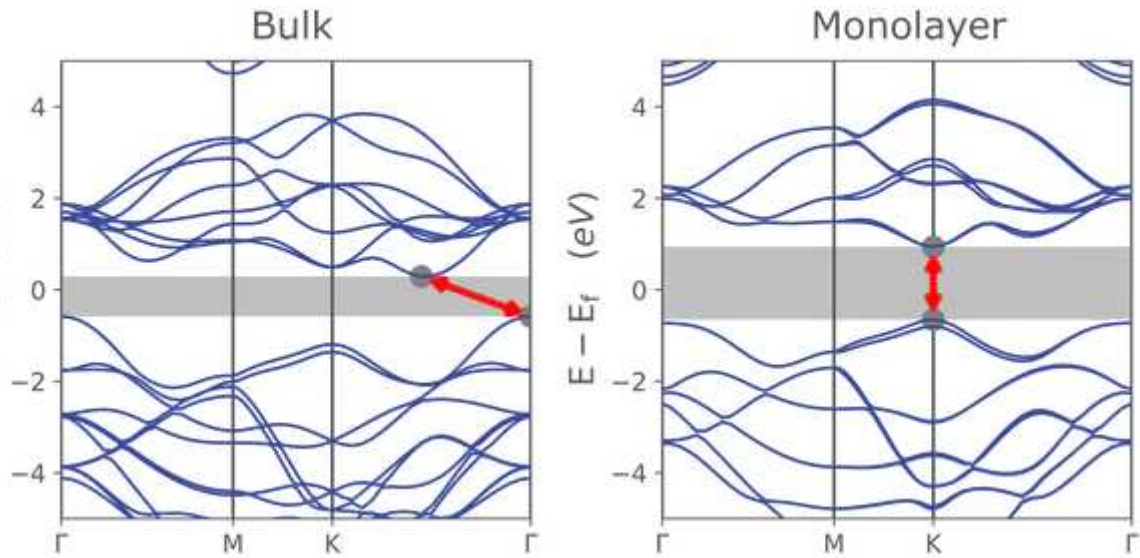


Figure II.13: Band structure diagram of (left) bulk and (right) monolayer MoS_2 showing the crossover from indirect to direct bandgap accompanied by a widening of the bandgap.

II.5. Applications of 2D materials:

The change in properties, caused by a reduction in the dimensionality of 2D materials, means that they are often well-suited to applications where the bulk material would be unsuitable. Below is a list of some of the most promising applications.

II.5.1. Transistors and sensors:

Field-effect transistors (FETs) have been fabricated from a variety of semiconducting 2D materials such as TMDCs [14] and black phosphorus. Their good charge mobility and moderate bandgaps make them good candidates for this application. Hexagonal boron nitride is often used as the gate dielectric. Despite its intrinsic lack of bandgap, graphene has also been used as the active channel in transistors by opening up a bandgap with methods such as engineering edge states, chemical doping, or applying electric fields.

An advantage of 2D materials over conventional silicon is their inherent flexibility. In combination with suitable substrates, 2D materials can be used to create flexible circuits [07]. While there are still some issues with large-scale production of high-quality 2D layers that would be required by the electronics industry, transistors remain one of the most promising applications. FET-based sensors have been fabricated from 2D TMDCs that can detect a range of chemicals in the parts-per-million range or better, including triethylamine [08], nitric oxide, ammonia, and nitrogen dioxide by measuring changes in conductance when exposed to these chemicals.

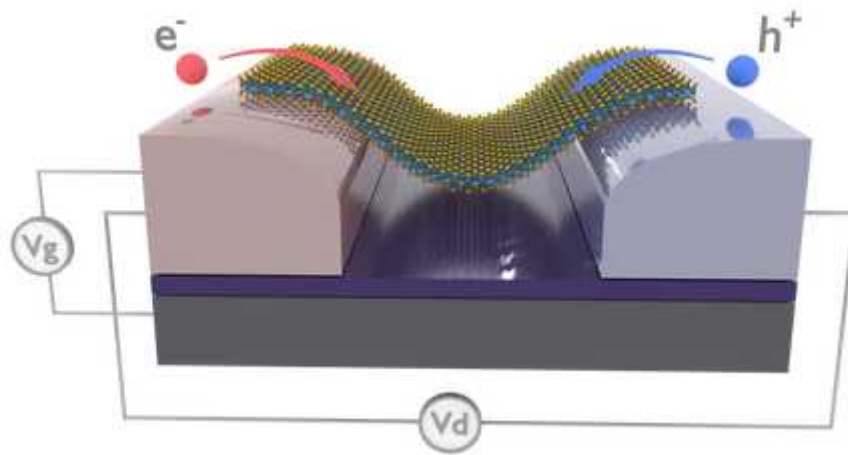


Figure II.14: A transistor formed from a monolayer of TDMC spanning metal electrodes.

II.5.2. 2D transistor:

A transistor formed from a monolayer of TDMC spanning metal electrodes.

II.5.3. Photodetectors:

Many TMDCs (e.g., MoS₂, MoSe₂, WS₂ and WSe₂) and black phosphorus have a bandgap in the optical or near infrared region and good charge transport characteristics. A single monolayer of TMDC (< 1nm thick) can absorb up to 10% of incident visible light [11], equivalent to approximately 100nm of silicon. Due to the thinness of the monolayers, this absorption is not enough for high efficiency photovoltaics. However, they can be fabricated into high efficiency photodetectors. A device fabricated from a mechanically exfoliated MoS₂ monolayer achieved a sensitivity of ~10³ A/W over the spectral range 400nm-680nm [15], while combining MoS₂ with graphene into a heterostructure the sensitivity reached 10⁸ [16].

II.5.4. Battery electrodes:

Electrodes for ion batteries and supercapacitors require electrically conductive materials with a high surface area to store high densities of ions. Graphene has received some attention as a possible successor to graphite electrodes [17, 18] due to its higher surface-to-mass ratio, higher conductivity, greater mechanical strength, and flexibility [19] which could lead to stronger, lighter batteries with greater power densities and faster charging times. 2D MoS₂ has also received a lot of attention as a possible electrode. While it is semiconducting in its most stable 2H crystal structure, it can be prepared in such a way (usually by chemical exfoliation) such that it adopts a metallic 1T phase. Electrodes formed from stacked 1T monolayers have shown higher power and energy densities than graphene-based electrodes [20].

II.5.5. Topological insulators:

Topological insulators (TIs) are materials that behave as insulators, except for at their edges where they can conduct electrons with high efficiency. Electrons with opposite spins travel around the edges in opposite directions. TIs may find practical applications in low-power electronic and spintronic devices (where electron spin is used to encode information or improve device performance). The Xenes (in particular bismuthene and stanine) are expected to act as TIs [21]. The TMDC WTe_2 can be switched between topological insulator and superconductor with an electric field [22, 23].

II.5.6. Valleytronics:

Valleytronics utilises a property of charge carriers in some TDMCs (e.g., MoS_2 , WS_2) called 'valley polarisation'. This property is linked to the spin and angular momentum of the electron and hole. Polarised optical excitation allows selective excitation of a particular valley, and hence carriers of a specific spin. This additional degree of freedom opens up the potential to create novel optoelectronic devices for data processing and storage. Valleytronics is covered in more detail here.

CHAPTER III: Metal Matrix Composite

III.1. Introduction:

In general, metals and alloys are brought forth and cast in bulk form, but we can additionally combine them with another material that will help us improve their performances: The material created is called metal matrix composite (MMC). Such composites include many different materials, which can be distinguished by their base metals (e.g., aluminum, copper, titanium); on the other hand, reinforcement, phases (e.g., fibers, particles, whiskers); or their manufacturing process (such as powder metallurgy, diffusion bonding, infiltration, stir casting)[24].

Bulk metal and alloys are cheaper materials and got high performances in the field. Then, why, fuse these materials with another phase, knowing that it will add cost and cause complications (in recycling, particularly)?

The successive facts explain the reasons that drive us toward the need for MMC technology.

The first is that the composite approach to material design allows it to go beyond Boundaries drawn in property space by the basic properties of the main material classes. An example is the specific modulus of a metal, defined as Young's modulus E divided by the density ρ : (E/ρ) . This parameter is a measure of performance limitation at critical deflections of weight Structural applications of linear elastic elements subjected to uniaxial loads. Well, the most important thing technical metals and alloys have roughly the same $(E/\rho) \approx 26 \text{ MJ kg}^{-1}$. So essentially there is only one way to go beyond this limit in metallic materials is to replace a significant fraction of the metal atoms with a phase made of atoms that are situated in the top rows of Mendeleev's Periodic Table and solidly bound together. For example, ceramics such as Al_2O_3 , B_4C , or SiC , and some Variants of carbon (eg high modulus carbon fiber or diamond) [24].

The second reason MMC is interesting has to do with processing: making composites is the only method that can introduce large amounts of oxides or carbides into some important metals. Iron is common - and easily - extensively fortified Carbides, nitrides or (rarely) oxides, since carbon, nitrogen and oxygen are soluble in this molten metal. On the other hand, liquid aluminum, magnesium, and copper basically have Insoluble in carbon. Therefore, the only way to introduce carbides into these metals is through Synthesis; the same applies to aluminum with oxides or nitrides. In other words, MMC Technology is key to the widespread use of aluminum, magnesium or copper. Microstructure and properties obtainable with iron [24].

The third reason for my interest in MMC has little to do with metals. Some phases, especially ceramics, have much better properties in their finely divided form. Especially micron-sized ceramic Fibers or ceramic particles are much stronger than bulk ceramics. In addition, small single-crystal ceramic particles can be excellent thermal conductors. Finely dispersed carbon is very hard, hard, and can also have good thermal conductivity (such as diamond)[24].

Therefore, in order to take advantage of this in macroscopic materials, it is necessary to incorporate such finely divided non-metal phases (fibers, discs, films or particles, etc.) into the composite, the matrix of which is advantageous from metals. Can be. In fact, metals are better conductors than polymers, stronger, stronger and more environmentally friendly. Of course, they are much stronger than ceramic [24].

These basic considerations have facilitated ongoing research on MMC that has been undertaken for decades. Especially through pioneering research such as the important contributions of Anthony Kelly and his colleagues. Research on this topic reached high intensity in the late 1980s and early 1990s. And as it continues to this day, it has a wider spectrum in a clearer direction and is, therefore, less visible. MMC is also entering "real world" engineering applications in many ways.[24]

III.2. Metal matrix composite :

III.2.1. Basics of metal matrix composite:

For many researchers, the term metal matrix composites are often equated with the term light metal matrix composites (MMC). Significant progress has been made in the development of light metal matrix composites in recent decades, enabling their introduction into major application areas. In traffic engineering, especially in the automotive industry, MMC is used commercially for fiber-reinforced pistons and aluminum crankcases, with reinforced cylinder surfaces and particle-reinforced brake discs [25].

These innovative materials open up endless possibilities for modern materials science and development; depending on the application, the properties of MMC can be integrated into the material on a customer-specific basis. Based on this potential, metal matrix composites meet all the expectations of designers. The advantages of composites are Realized when the component has an appropriate cost-performance ratio Manufacture. The use of composites is absolutely necessary for special properties Profiles can only be achieved by applying these materials. The possibility to combine different material systems (metal-ceramic-non-metal) offers unlimited variation possibilities. The properties of these new materials are basically determined by the properties of the individual components [25].

Reinforcing metal has many purposes. Reinforcement of the use of light metals opens up the possibility of using these materials locally when weight reduction is paramount.

The prerequisite here is the improvement of Component properties. Light metal composite materials development goals are:

- Increase in yield strength and tensile strength at room temperature and above while maintaining the minimum ductility or rather toughness.
- Increase in creep resistance at higher temperatures compared to that of conventional alloys.

- Increase in fatigue strength, especially at higher temperatures.
- Improvement of thermal shock resistance.
- Improvement of corrosion resistance.
- Increase in Young's modulus.
- Reduction of thermal elongation.

In summary, improvements in weight-specific properties can be provided for an expansion of application range, and the possibility of alternatives to general materials component property optimization. With functional materials there another goal is the prerequisites for maintaining the corresponding functionality material. The goals are:

- Increase in strength of conducting materials while maintaining the high conductivity.
- Improvement in low temperature creep resistance (reactionless materials).
- Improvement of burnout behavior (switching contact).
- Improvement of wear behavior (sliding contact).
- Production of layer composite materials for electronic components.
- Production of ductile composite superconductors.
- Production of magnetic materials with special properties [25].

For other applications, different development objectives are given, which differ from those mentioned before. For example, in medical technology, mechanical properties, like extreme corrosion resistance and low degradation as well as biocompatibility are expected [25].

Increased development activities have resulted in system solutions using metal composites, but the use of innovative systems has not been realized, especially in the field of light metals. The reason for this is in addition to inadequate process stability and security, production and processing issues and inadequate profitability. Applications such as traffic engineering are very cost-sensitive and conservative, and the industry is not willing to pay additional costs for the use of such materials. For all these reasons, metal matrix composites are just the beginning of the evolutionary curve of modern materials [25].

III.2.2. Types of metal matrix composites:

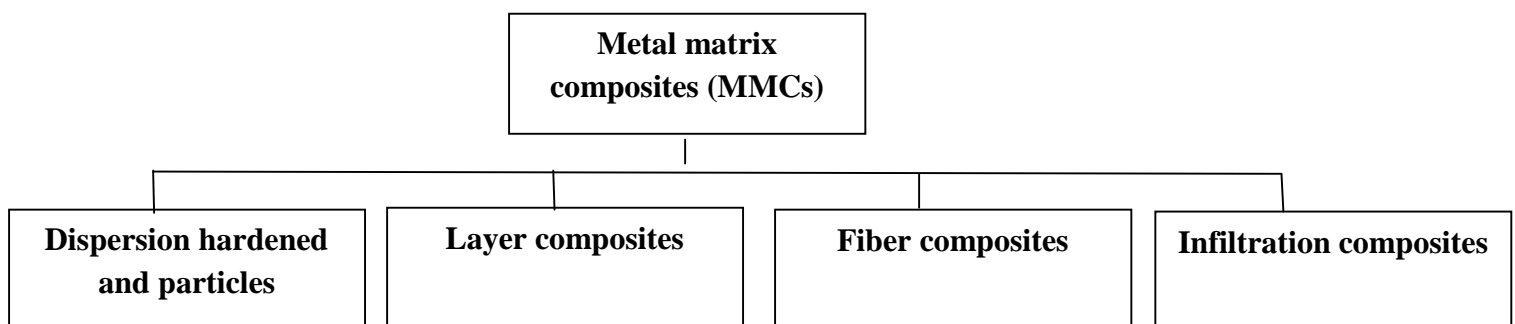


Figure III.1: Classification of composite materials with metal matrices.

Metal matrix composites can be classified in several ways. One classification is to consider the properties and contributions of particulate, layered, fibrous, and osmotic composite reinforced components (see figure III.1). Fiber composites can be further divided into continuous fiber composites (multifilaments and monofilaments) and short fiber or whisker composites, (see figure III.2).

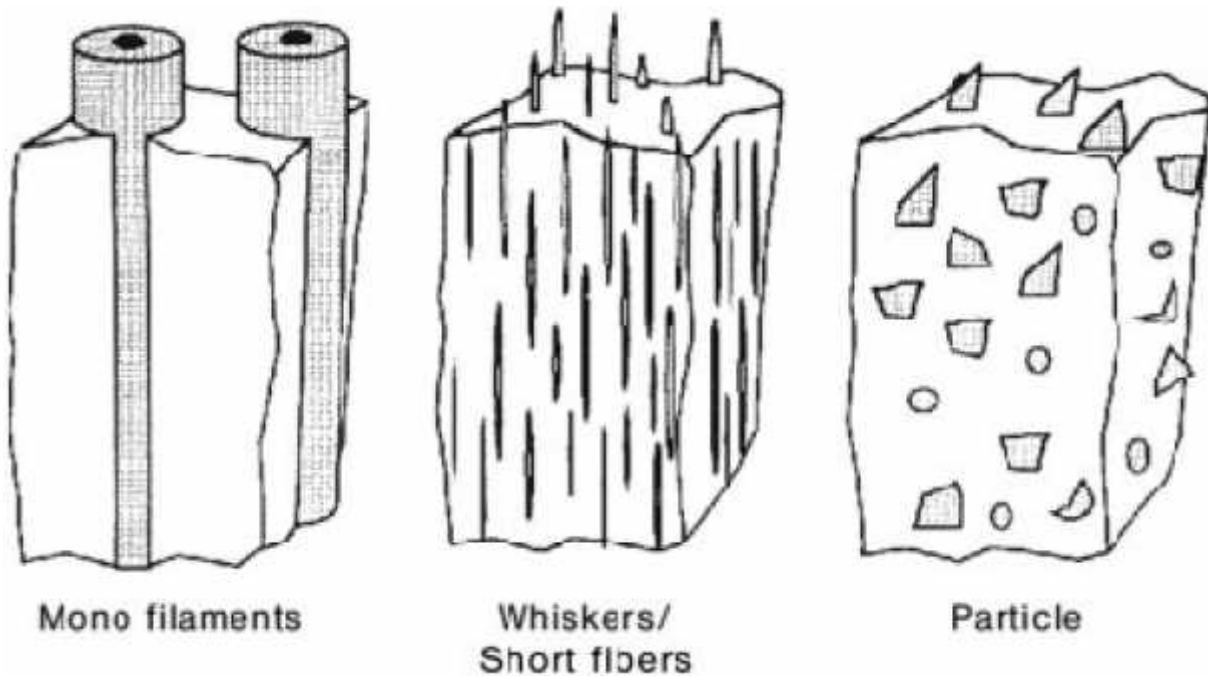


Figure III.2: Schematic presentation of three shapes of metal matrix composite materials.

III.2.3. Important metallic matrices:

A variety of metals and their alloys can be used as matrix materials. We describe briefly the important characteristics of some of the more common ones.

III.2.3.1. Aluminum matrix composite:

Most of today's metal matrix composites are based on aluminum and its alloys, both in research and development and in a variety of industrial applications. This is because aluminum is lightweight. This is the first requirement for most applications of today's metal matrix composites. It is also cheaper than other light metals such as titanium and magnesium. Traditional aluminum alloys are used in large quantities in many applications, from automobiles and aircraft to sports and leisure, so their excellent strength, ductility, and corrosion properties are well known for their versatility. You can change it. Of all the

excellent Al alloys, precipitation hardening alloys such as **Al-Cu-Mg** and **Al-Zn-Mg-Cu** are of particular importance. However, the most important and recent improvement in precipitation hardening aluminum alloys is the **Al-Li** alloy. The special effect of Li is that when alloyed to form Al, it simultaneously reduces the density and increases the modulus of elasticity of the alloy. So, it's not surprising that the aerospace industry is interested in the potential of **Al-Li** matrix composite material [26].

Table III.1 Ultimate tensile strength and elastic modulus of selected light metal matrix composites.

Matrix	Reinforcement	Vol fraction (%)	Longitudinal tensile strength (MPa)	Longitudinal elastic Modulus (GPa)
Al-7Zn	Graphite fibre	40	90	190
Al 2024	Boron fibre	60	1500	270
Al 6061	SiC fibre	50	1500	205
Al 2024	Al ₂ O ₃	50	450	175
Al 6061	SiC whisker	15	480	100
Al 6061	SiC particulate	15	370	100
Ti-6Al-4V	BORSIC	40	900	205
Ti-6Al-4V	SCS-6	35	1600	240
Ti-Al-4V	Sigma SM1240	35	1550	230
Mg	Graphite	40	560	230
Mg	SiC fibre	50	1300	230

The early expectations on the properties of aluminum matrix composites have been mostly fulfilled until now (Table III.1). For instance, the tensile strength of continuous boron-fiber reinforced aluminum matrix composites can exceed 1500 MPa. The Young's modulus of the same material is approx. 280 GPa. Accordingly, tensile strength similar to and stiffness superior to high-strength steel can be achieved with a much lower weight. Although such exceptional figures can be presented it should be mentioned that the best properties are only reached in the direction of fibers. Moreover, the production costs of continuous fiber materials are very high and their applications are rare. Therefore, a great deal of the aluminum matrix composites today are particulate reinforced and their properties are far from those presented above [26].

However, also particulate reinforced materials are expensive in comparison to monolithic materials which still restricts their utilisation in everyday life. Consequently, the development

work of aluminum matrix composites is currently concentrated on two sectors:(1) continuous fiber-reinforced composites with superior properties for very specific applications and (2) mass production technologies of inexpensive discontinuously reinforced composites with moderate properties for a wider range of applications [26].

III.2.3.1.1. Continuous fiber reinforced aluminum matrix composites:

Fiber-reinforced special composites are typically manufactured by liquid penetration of preforms or filament arrays and diffusion bonding of preforms. These processes contain many steps that are also difficult to automate. The size and shape of the components are limited and standard metalworking and machining methods are not possible.

Usually applied. As a result, the manufacturing costs of these materials remain high (Figure 2.3). Some applications, such as the military and aerospace industries, require extreme characteristics and cost efficiency is less important. Therefore, some future development work will continue to focus on further improving the material properties of state-of-the-art fiber-reinforced Al-matrix composites. Carbon, boron, SiC and Al₂O₃ are the most thoroughly researched endless reinforcements [26].

The density of carbon fiber is the lowest of them, so it can provide significant weight savings. In addition to being impressive. To improve strength, carbon and boron reinforcements can be used to adjust the coefficient of thermal expansion of the Al matrix composite. This option was used the microchip heatsink adjusts the heat coefficient of the Al/B composite to match the microchip [26].

The wetting and bonding or, on the other hand, excessive reactions between the matrix and ceramic are generally regarded as the major issue in most metal matrix composite materials. The problem of carbon, boron and SiC fibers are the potential heavy reactions during processing or in use at high temperatures which may degrade the mechanical properties. In the case of Al matrices much of these problems have been overcome by using suitable fibre coatings [26].

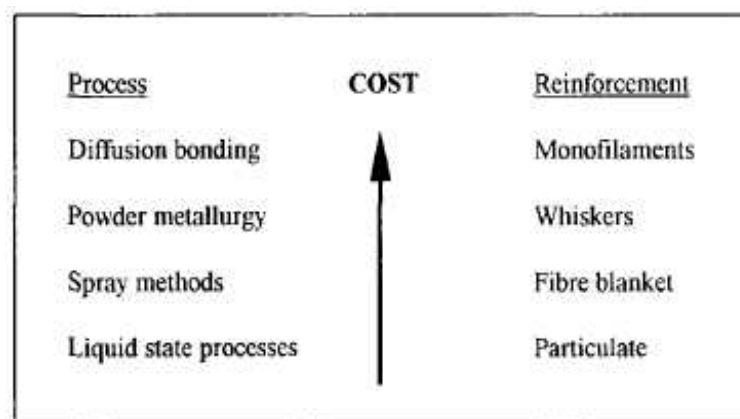


Figure III.3: The relative cost effectiveness of various processing methods and reinforcements

III.2.3.1.2 Particulate reinforced aluminum matrix composites:

Another trend in the study of Al-matrix composites is to develop more cost-effective manufacturing techniques, especially for discontinuously enhanced MMCs.

The development of monolithic aluminum alloys has made it possible to develop highly efficient and reliable levels of production technology. Many of these techniques Also aluminum matrix composite material. For example, you can use common molding methods such as extrusion, forging, and rolling. Traditionally, liquid state processes such as various casting and powder metallurgy processes have been used to manufacture particle-enhanced Al-matrix composites. However, new deposition and processes are being actively studied because they are potentially very efficient and economical. Since it is difficult to automate the powder metallurgy route, it may not be the correct answer for economically manufacturing Al-matrix composites. Therefore, the most promising processes are found in liquid states, deposition, and processes. The simplest, cheapest and most widely used method for monolithic AI parts is a variety of casting techniques. Therefore, it is not surprising that much value is placed on the development of these technologies [26].

Table III.2: Applications of Al matrix composites in Japan

Product	MMC system	Method of manufacture	Characteristics of applied MMC	Year (maker)
Vane, pressure side plate of oil pressure vane pump	Al ₂ O ₃ - SiO ₂ / AC4C	Squeeze casting(S.C.)	Wear resistance, noise damping	1987 (Hiroshima Aluminum)
Ring groove reinforced piston	Al ₂ O ₃ / Al-alloy	"	Light weight, wear resistance at high temperature	1983 (Toyota)
Golf goods Face of screwdriver	SiC _{pcs} / Al-alloy	"	Light weight, abrasion resistance	1984 (Nippon Carbon)
Connecting rod of gasoline engine	SUS fiber/ Al-alloy	"	Specific strength	1985 (Honda)
M6~8 bolt	SiC _w /6061	S.C. Extrusion Tread rolling	Neutron absorption high temperature strength, little degasing	1986 (Toshiba)
Joint of aerospace structure	SiC _w /7075	S.C. Rolling	Specific strength, low thermal expansion	1988 (Mitubishi)
Rotary compressor vane	SiC _w /Al-17%Si -4%Cu alloy	S.C.	Specific strength, wear resistance, low thermal expansion	1989 (Sanyo)
Shock absorber cylinder	SiC _p / Al-alloy	Compo-casting S.C. Extrusion	Light weight, wear resistance, thermal diffusivity	1989 (Mitubishi Aluminum)
Bicycle frame	SiC _w /6061	Powder metallurgy HIP, Extrusion	Light weight, high specific rigidity	1989 (Kobe steel)
Diesel engine piston	SiC _w / Al-alloy	S.C.	Light weight, wear resistance	1989 (Niigata)
Cylinder liner	Al ₂ O ₃ , CF/ Al-alloy	Low pressure S.C.	Wear resistance, light weight	1991 (Honda)

Although the density of SiC is slightly higher than that of Al, SiC particles are the most common discontinuous reinforcement in Al-matrix composites (Table III.2). This is to give the composite high strength and high modulus, even though it is cheap and readily available. In many cases, improving wear resistance is also a priority. Like SiC continuous fibers, the potential for chemical reactions limits high temperature applications and can cause manufacturing problems. Excess Si significantly reduces the reactivity of SiC in Al. Another broad particle reinforcement for Al-matrix composites is Al₂O₃. It is much more inert to Al and more resistant to oxidation than SiC. Therefore, it is much more suitable for high temperature manufacturing and use. In particular, stirring the liquid interferes with the problem of poor wettability of Al₂O₃ by Al [26].

The production route, matrix is alloyed or the reinforcement is surface coated. As mentioned above, Li has been found to be an advantageous alloying element. MgO on the surface of Al₂O₃ also improves wettability. On the other hand, in squeeze casting, wettability is not as important as in liquid agitation. Therefore, this efficient technique is widely used in the production of Al-matrix composites reinforced with particle Al₂O₃. In fact, this combination of manufacturing processes and composites is currently the most promising candidate for large-scale production of relatively inexpensive MMCs for general applications. Recently, several other particulate matter have also been investigated. For example, graphite can give composites specific tribological properties, and B₄C reinforced materials may have

nuclear applications due to the neutron capture properties of boron. Research on ZrO_2 reinforced materials has been conducted with similar uses in mind as Al_2O_3 reinforced materials. Previously, little or no attention was paid to the secondary operations required to manufacture functional parts from metal matrix composites. However, correct machining and welding the process is essential for the widespread use of composites. In the future, environmental factors will play an increasingly important role in the use of materials [26].

Therefore, Composite material recovery and recycling need to be developed and well organized. Duralcan, for example, has recently made significant efforts to improve these areas. Several deposition techniques are available for the production of continuous fiber composites, including: chemical vapor deposition (CVD), physical vapor deposition (PVD), electroplating, dip plating, and spray deposition. In this process, the individual fibers in the cable are coated with matrix metal. The materials are then integrated by diffusion joining to form a composite panel or structure. Although these steps have some advantages, the Manufacture of continuous fiber reinforced preforms is time consuming and has the same drawbacks as other methods of continuous fiber MMC. However, a fairly new and highly promising deposition process specifically developed for particle-enhanced materials is the Osprey (or Cospray) process [26].

An industrial scale example of exothermic reaction synthesis of reinforcements in metal matrix composites is Martin Marietta's XD process. In this case, the master alloy is included. Most of the reinforcements are produced by reaction synthesis. A sufficient amount of master alloy is mixed with the matrix metal to provide the desired level of reinforcement. Redissolved. Typical reinforcements for Al Matrix are SiC, TiC and TiB_2 . This method can also be applied to various other matrix metals [26].

III.3. Processing:

Many methods are available for manufacturing metal matrix composites. These processes are primarily liquid and solid processing. Some processes may include various deposition techniques or in-situ processes for incorporating the strengthening phase. It provides an overview of these manufacturing processes.

III.3.1. Liquid state processes:

Metals that do not have too high a melting temperature, such as aluminum, can be easily incorporated as a matrix in a liquid way. This section describes some important processes in the liquid state.

Casting or liquid infiltration includes infiltration of fiber bundles by liquid metal. It is not easy to manufacture MMC by simple liquid phase infiltration, mainly because it is difficult to wet the ceramic reinforcement with molten metal. If the fiber preform penetrates easily, the reaction between the fiber and the molten metal can be large. Fiber properties deteriorate. Fiber coatings that are applied before penetration to improve wetting and control responses have been developed and may lead to some improvements. However, the drawback here is the

fiber coating. Do not expose to air prior to penetration as surface oxidation will change the positive effects of coating[27].

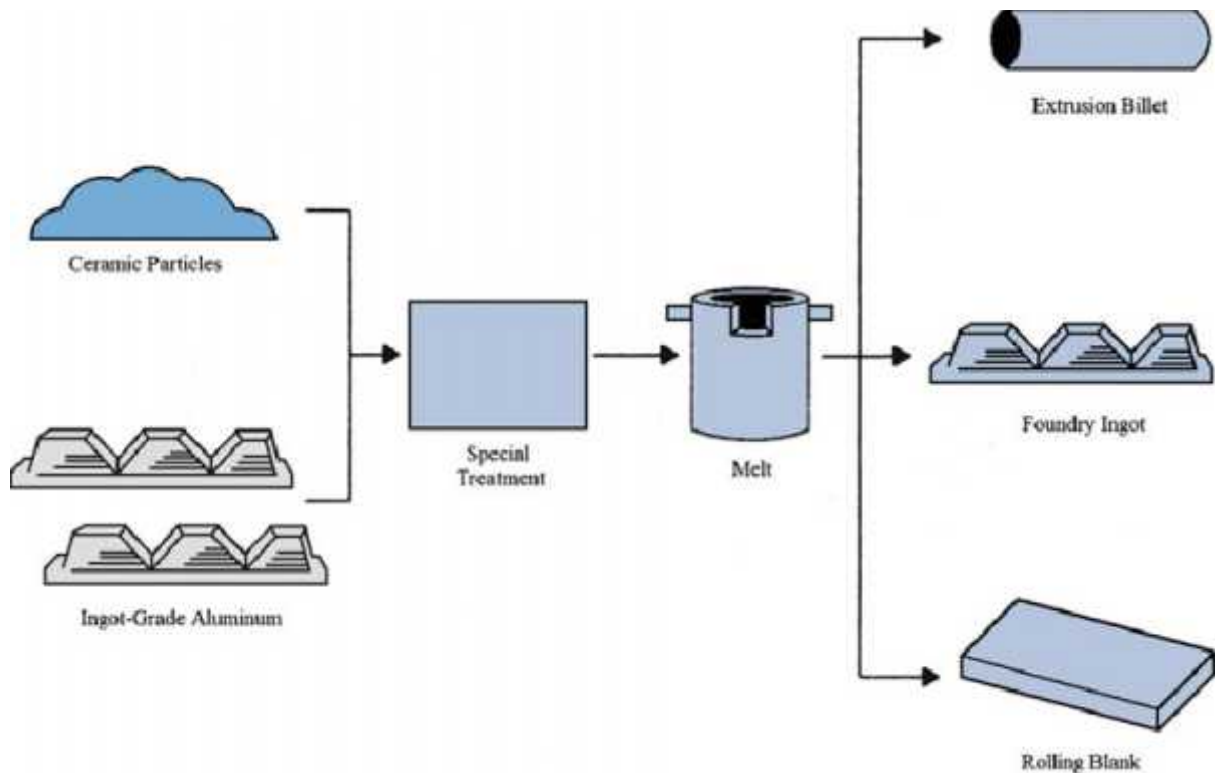


Figure III.5: Schematic of the Duralcan process

One commercially successful liquid infiltration process involving particulate reinforcement is the Duralcan process. (Figure III.5) shows a schematic of this process. Ceramic particles and ingot-grade aluminum are mixed and melted. The ceramic particles are given a proprietary treatment. The melt is stirred just above the liquidus temperature—generally between 600 and 700_C. The melt is then converted into one of the following four forms: extrusion blank, foundry ingot, rolling bloom, or rolling ingot. The Duralcan process of making particulate composites by liquid metal casting involves the use of 8–12 mm particles. Too small particles, e.g., 2–3 mm, will result in a very large interface region and thus a very viscous melt. In foundry-grade MMCs, high Si aluminum alloys (e.g., A356) are used, while in wrought MMC, Al–Mg type alloys (e.g., 6061) are used. Alumina particles are typically used in foundry alloys, while silicon carbide particles are used in the wrought aluminum alloys. For making continuous fiber reinforced MMCs, tows of fibers are passed through a liquid metal bath, where the individual fibers are wet by the molten metal, excess metal is wiped off, and a composite wire is produced. (Figure III.6) shows a micrograph of one such wire made of SiC fibers in an aluminum matrix. Note the multifiber cross sections in the broken composite wire. A bundle of such wires can be consolidated by extrusion to make a composite. Another pressureless liquid metal infiltration process of making MMCs is Lanxide’s Primex™ process, which can be used with certain reactive metal alloys such Al–Mg to infiltrate ceramic preforms. For an Al–Mg alloy, the process takes place between 750 and 1,000_C in a

nitrogen-rich atmosphere (Aghajanian et al. 1989). Typical infiltration rates are less than 25 cm/h[27].

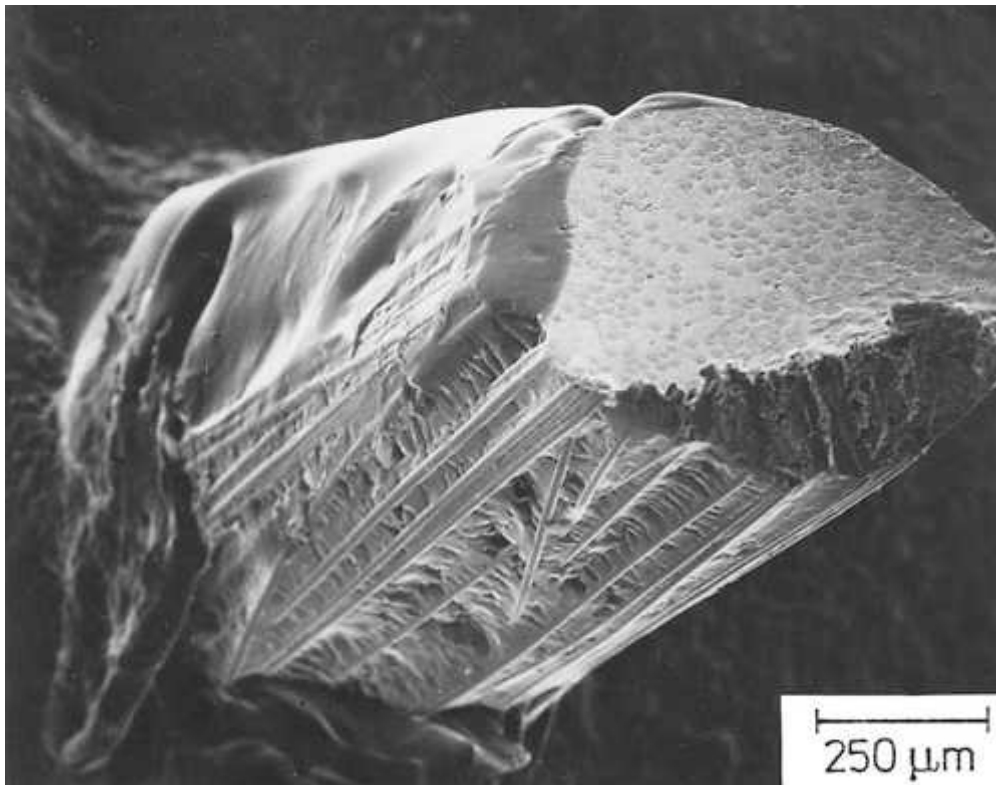


Figure III.6: *A silicon carbide fiber/aluminum wire preform. SiC fibers can be seen in the transverse section as well as along the length the wire preforms*

Squeeze casting, or pressure infiltration, involves forcing the liquid metal into a fibrous preform. (Figure III.7) shows two processes of making a fibrous preform. In the press forming process, an aqueous slurry of fibers is well agitated and poured into a mold, pressure is applied to squeeze the water out, and the preform is dried (Figure III.7a). In other process, suction is applied to a well-agitated mixture of whisker, binder, and water. This is followed by demolding and drying of the fiber preform (Figure III.7b). A schematic of the squeeze casting process is shown in (Figure III.8a) Pressure is applied until the solidification is complete. By forcing the molten metal through small pores of fibrous preform, this method obviates the requirement of good wettability of the reinforcement by the molten metal. (Figure III.8b) shows the microstructure of Saffil alumina fiber/aluminum matrix composite made by squeeze casting. Composites fabricated with this method have minimal reaction between the reinforcement and molten metal because of short dwell time at high temperature and are free from common casting defects such as porosity and shrinkage cavities. Squeeze casting is really an old process, also called liquid metal forging in earlier versions. It was developed to obtain pore-free, fine-grained aluminum alloy components with superior properties than conventional permanent mold casting. In particular, the process has been used in the case of aluminum alloys that are difficult to cast by conventional methods, for example, silicon-free

alloys used in diesel engine pistons where hightemperature strength is required. Inserts of nickel-containing cast iron, called Ni-resist, in the upper groove area of pistons have also been produced by the squeeze casting technique to provide wear resistance. The use of ceramic fiber reinforced metal matrix composites at locations of high wear and high thermal stress has resulted in a product much superior to the Ni-resist cast iron inserts [27].

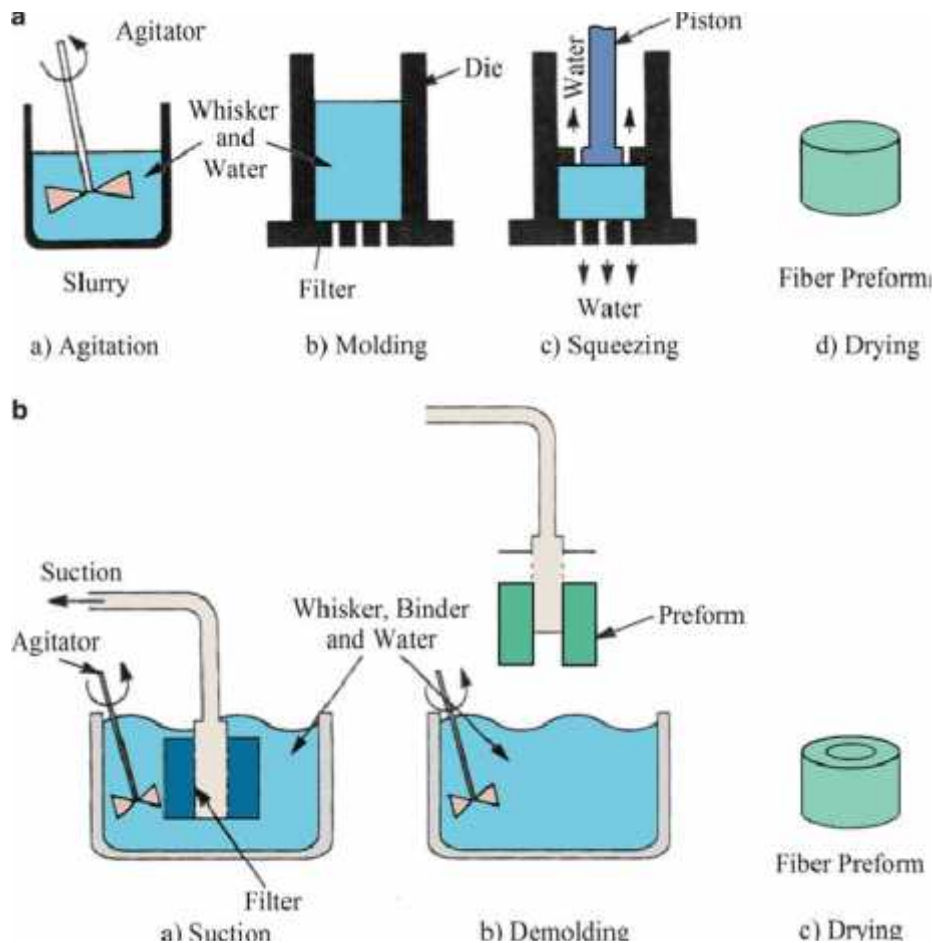


Figure III.7: (a) Press forming of a preform. (b) Suction forming of a preform

The squeeze casting technique, shown in (Figure III.8), has been quite popular in making composites with selective reinforcement. A porous fiber preform (generally of discontinuous Saffil-type Al_2O_3 fibers) is inserted into the die. Molten metal (aluminum) is poured into the preheated die located on the bed of a hydraulic press. The applied pressure (70–100 MPa) makes the molten aluminum penetrate the fiber preform and bond the fibers. Infiltration of a fibrous preform by means of a pressurized inert gas is another variant of liquid metal infiltration technique. The process is conducted in the controlled environment of a pressure vessel and rather high fiber volume fractions; complex-shaped structures are obtainable. Although commonly, aluminum matrix composites are made by this technique, alumina fiber reinforced intermetallic matrix composites (e.g., TiAl, Ni_3Al , and Fe_3Al matrix materials) have been prepared by pressure casting. The technique involves melting the matrix alloy in a crucible in vacuum, while the fibrous preform is heated separately. The molten matrix material (at about 100_C above the T_m) is poured onto the fibers, and argon gas is introduced

simultaneously. Argon gas pressure forces the melt to infiltrate the preform. The melt generally contains additives to aid in wetting the fibers[27].

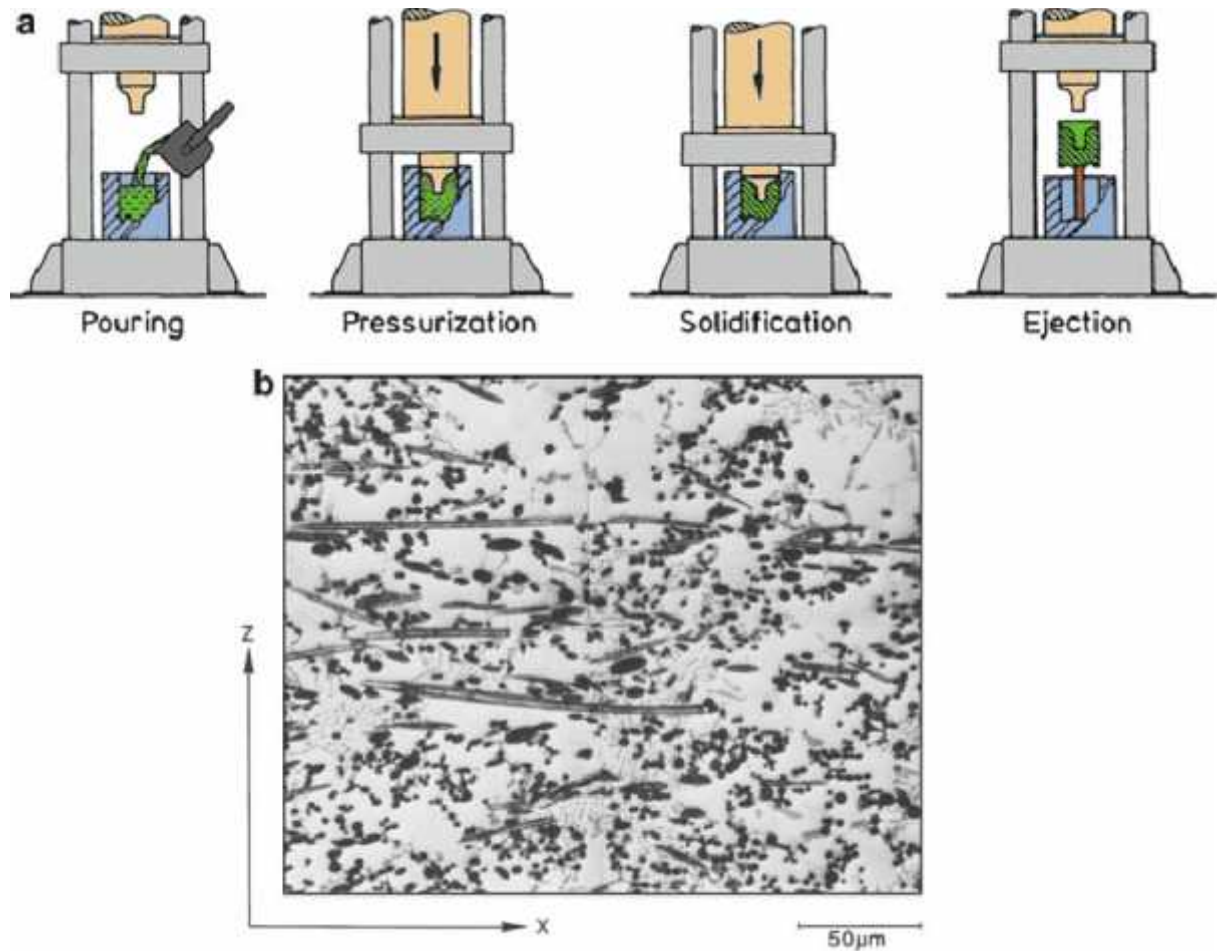


Figure III.8: (a) Squeeze casting technique for making a metal matrix composite. (b) The microstructure of Saffil alumina fiber/aluminum matrix composite made by squeeze casting.

III.3.2. Solid state processes:

Many solid-state techniques are available (Ghosh 1993). We describe some of the important ones.

Diffusion welding is a common solid-state welding technique used to join similar or dissimilar metals. Mutual diffusion of atoms from clean metal surfaces that are in contact at high temperatures results in welding. There are many variations of Basic diffusion bonding process. However, all include the step of applying pressure and high temperature at the same time. Matrix alloy foil and fiber assemblies, composite wires or single layer flakes are stacked in a given order. (Figure III.9a) shows a schematic of one such diffusion bonding process, also called the foil-fiber-foil process. (Figure III.9b) shows the microstructure of SiC fiber/titanium matrix composite made by diffusion bonding. The starting material in this

case was a titanium sputter coated SiC fiber. Using filament winding, I got a panel about 250mm thick. Four such plates were stacked and hot pressed at 90 ° C. for 3 hours under a pressure of 105 MPa. Careful examination of (Figure III.9b) reveals the leader interface between the layers of the Ti alloy matrix. Vacuum hot press is a very important step in the metal matrix composite diffusion bonding process. The main advantages of this technique are the ability to process a variety of matrix metals and the ability to control fiber orientation and volume fraction. The disadvantages are: A few hours of processing time, high processing temperature and pressure, all of which make the process very expensive. In addition, you can only create objects of limited size[27].

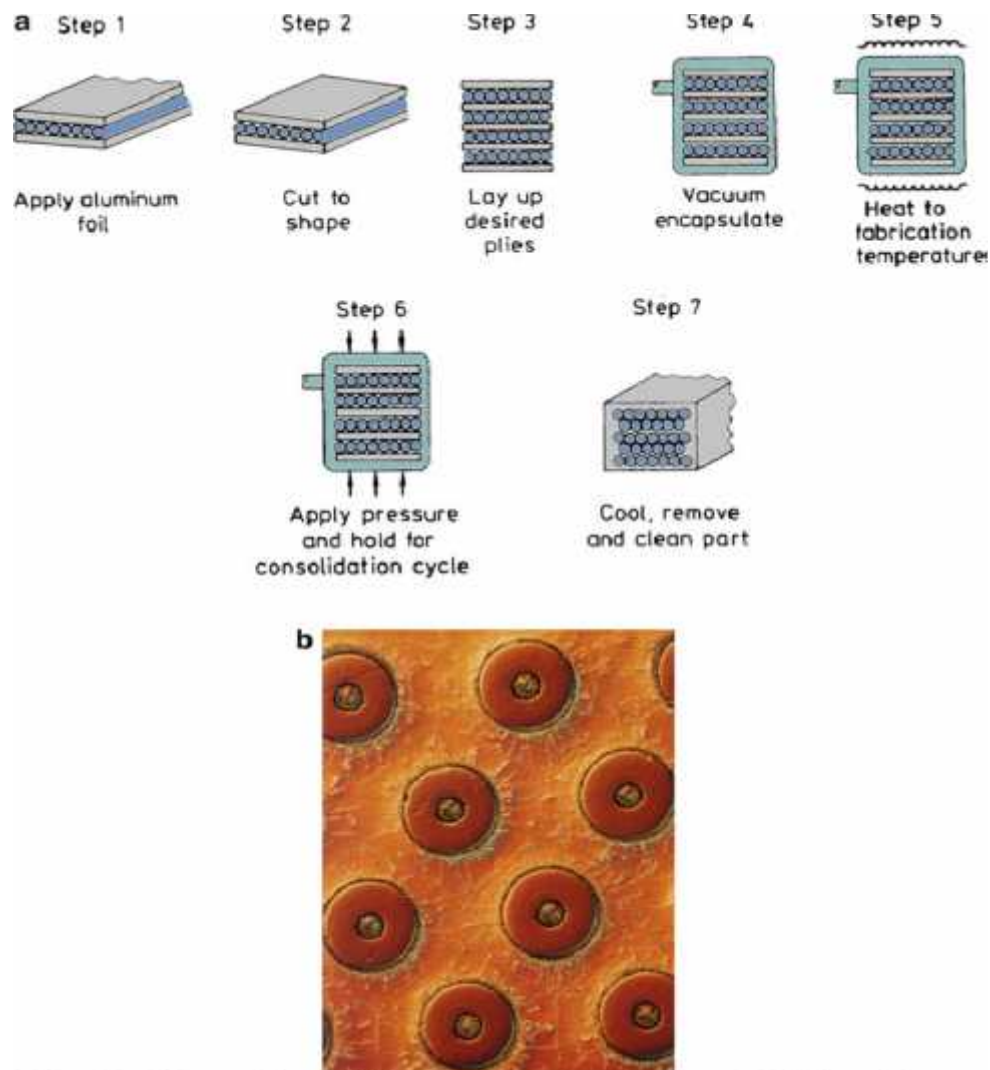


Figure III.9: (a) Schematic of diffusion bonding process. (b) The microstructure of SiC fiber/titanium matrix composite made by diffusion. Each fiber is 142 mm in diameter.

A hot hydrostatic press (HIP) can also be used instead of the uniaxial press. In HIP, the gas pressure on the can integrates the fittings contained within the can. With HIP, it is relatively easy to apply high temperature and high pressure over various shapes[27].

Deformation processing of metal / metal composites involves mechanical processing (implantation, extrusion, extraction, or rolling) of ductile two-phase materials. The two phases

match, the minor phase elongates and becomes essentially fibrous in the matrix. These materials are sometimes referred to as composite materials. The properties of the deformed composite are highly dependent on the properties of the starting material. The starting material is usually a two-phase alloy billet manufactured by a casting or powder metallurgy process. Roll bonding is a common technique for producing sheet-like laminated composites of dissimilar metals. Such composites are called laminated metal matrix composites. Other examples of deformed metal matrix composites are niobium-based conventional filamentary superconductors and high T_c superconductors [27].

The deposition technique for producing metal matrix composites coats the individual fibers in the strands with the matrix material required to form the composite, followed by diffusion bonding to form an integrated composite sheet or structure. Including doing. The main drawback of using sedimentation techniques is that they are very time consuming. However, there are some advantages:

- The degree of interfacial bonding is easily controllable; interfacial diffusion barriers and compliant coatings can be formed on the fiber prior to matrix deposition or graded interfaces can be formed.
- Thin, monolayer tapes can be produced by filament winding; these are easier to handle and mold into structural shapes than other precursor forms—unidirectional or angle-ply composites can be easily fabricated in this way [27].

Several vapor deposition techniques are available: dip plating, electroplating, spray deposition, chemical vapor deposition (CVD), and physical vapor deposition (PVD) (Partridge and Ward-Close1993). Immersion plating or dip plating is similar to permeation casting, except that the fiber tow continuously passes through a bath of molten metal, slurry, sol, or organic metal precursor. Electroplating creates a coating from a solution containing ions of the material of interest in the presence of an electric current. The fibers are wound around a mandrel that acts as a cathode and placed in an electroplating bath with the anode of the desired matrix material. The advantage of this method is that the temperature involved is moderate and no damage occurs. fiber. Electroplating problems can include the formation of voids between fibers and between fiber layers, inadequate adhesion of deposits to the fibers, and the limited number of alloy matrices available for this treatment. included. The spray deposition process usually consists of winding a fiber around a foil-coated drum and spraying molten metal onto it to form a monotape. The source of molten metal can be a flame, arc, or powder or wire material that melts in a plasma torch. The advantage of spray deposition is that the fiber orientation is easy to control and the molten matrix solidifies rapidly. In the CVD process, the vaporized component decomposes or reacts with another vaporized chemical on the substrate to form a coating on that substrate. Processing is generally done at high temperatures [27].

CHAPTER IV: Experimental part elaboration

IV.1. Introduction:

The purpose of this experimental part is the elaboration of 2D materials (Bismuthene) and studying his behaviors in aluminum matrix composite.

This chapter will focus on the experimental protocol as well as on the techniques of elaboration,

analysis and characterization undertaken on the powders used and the materials synthesized in our investigation.

According to the work that has been carried out, several experimental techniques such as hardness test, have been applied on samples in order to determine the coefficient of friction and the rate of wear.

The techniques that have been used are: scanning electron microscopy (SEM), Vickers hardness test. Optical microscope.

IV.2. Synthesis of Bismuthene:

Bismuthene is a two-dimensional material that has attracted considerable attention for a wide range of potential applications due to its excellent properties.

As a first step to elaborate and study its properties and possibly to apply them in the real world, different methods have been proposed to prepare bismuthene among them sonochemical exfoliation because it is possible to easily prepare bismuthene at low cost at large scale for applications.

IV.2.1. Bismuthene powder preparation:

-The powder was weighed three times by a precision balance of 10^{-4}



Figure IV.1 Precision balance of 10^{-4}

- Bulk bismuth was first grinded into bismuth powder with isopropyl alcohol.



Figure IV.2 Mortar and Pestle *Figure IV.3 isopropylalcohol*

- Afterward, 0.5 mL of bismuth isopropyl alcohol solution and 9.5 mL of isopropyl alcohol were sited in bottle and kept under ice bath sonication.



Figure IV. 4 Ultrasonic Bath

- Our bottle sited in ice bath probe sonication (ultrasonic homogenizer) for 10 h

Process of working

- The operation took us 20 days to finish our 10 h



Figure IV.5 probe sonication (ultrasonic homogenizer)

- Respectively, a centrifugation of the bismuthene dispersion mixture was made with the parameter following:
 - 10000 Tr/min
 - Centrifugation time of 3 minutes



Figure IV.6. Centrifugation of isopropyl and Bismuthene mixtures

- After this centrifugation treatment we used an oven to put the tubes of the centrifuge which contains the isopropyl and bismuthene in a steam room at 40°C for 2 hours so that the isopropyl evaporates and so that we can recover the bismuthene.



Figure IV.7. the drying oven tubes

IV.3. Elaboration of the control sample

- Chosing of powder :

Pure aluminium 99%



Figure IV.7. Aluminium powder

- Weight the powder : 3g
- Compacting pure aluminium powder at different presion
 - 10 T (417.34 Mpa)
 - 15 T (626 Mpa)
- Cold Compaction :
After we done weighing and mixing the powders, we put it in a device with a 16.5mm diameter right before we do the cold uniaxial compaction using a hydraulic press (HYDRAULIC SHOP PRESS 50TON), while applying 10 tons pressure during 10 min.
This operation was elaborated in LTSM laboratory in University of Blida 1.



Figure IV.8. Compactage machine

- Polishing is an operation aimed at refining the scratches in order to be able to observe the microstructure of materials the latter was carried out with carbide abrasive papers silicon (SiC) of different grain sizes: 400, 600, 1200,4000 to get a flat surface.



Figure IV.9. Control samples



Figure IV.10. polishing machine

- Sintering :

The pellets obtained by the cold compaction were placed in an oven. The thermal cycle of sintering is represented ($T_f = 600\text{ }^\circ\text{C}$ with a dwell time $t = 2$ hours); after treatment the pellets were oven cooled firm.



Figure IV.11. sintering machine

IV.3.1. Hardness Machine:

Hardness is the resistance of a material to localised plastic deformation. Hardness ranges from super hard materials such as diamond, boron-carbide to other ceramics and hard metals to soft metals and down to plastics and soft tissues. Hardness is just one mechanical measurement and properties such as toughness and strength need to be considered, as hard materials tend to have low toughness and can easily fracture.

- We made a comparison between different pressures and their impact on the hardness of materials.

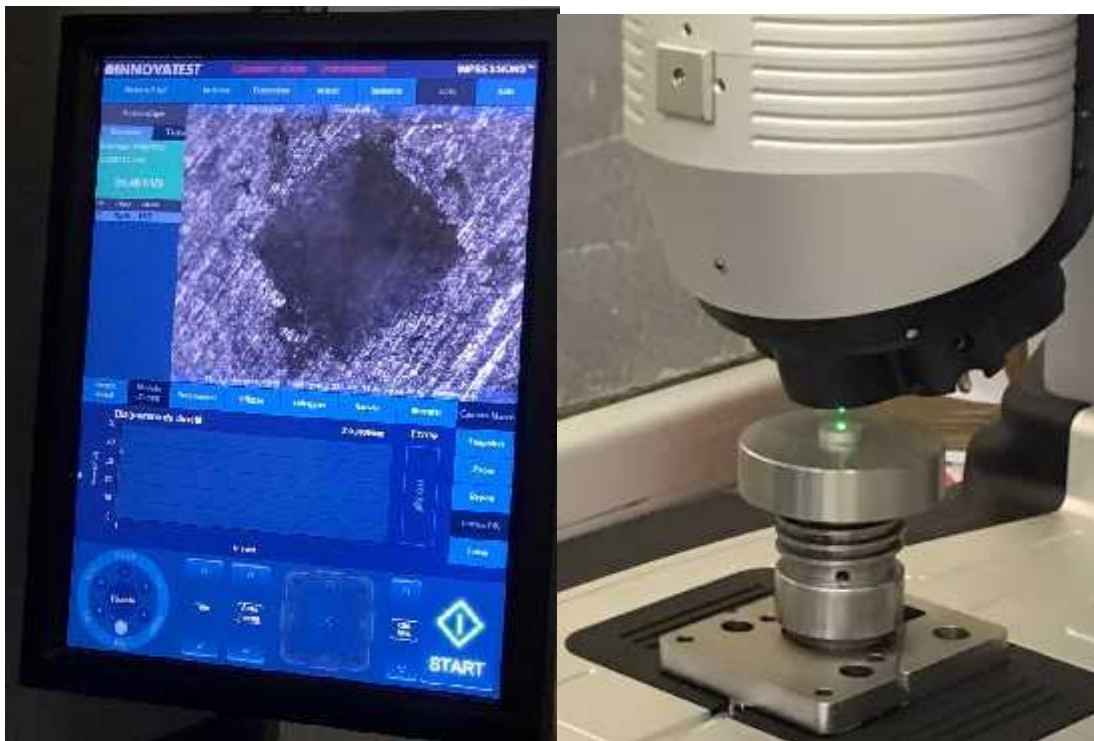


Figure IV.11. DUROMETER

- Theoretical Porosity calculations :
d = 17,3
S=235,06
m₁=3,014 g
m₂=3,074 g
e₁=5,03
e₂=5,03
V₁= 1182,35 mm²
V₂= 1182,35 mm²
10 T : $\rho = \frac{m}{V} = 2,549 \text{ kg/m}^3$
15 T : $\rho = 2,599 \text{ kg/m}^3$

10T : Density = $\frac{\rho}{\rho_n} = 0,94 = 94\%$; Porosity: 6%

15 T : Density = 0,96 = 96 % ; Porosity: 4%

From the previous calculations we noticed that the porosity in our sample's volume decrease relatively when we increase the pressure.

IV.4. Elaboration of composite (Aluminum \ bismuthene):

- Bismuthene weight : 0.02g =20mg



Figure IV.1 Precision balance of 10-4

- Aluminum powder weight : 2,98 g



Figure IV.12. Aluminuim fine powder

IV.5. Scanning Electronic Microscopy:

The operation of the scanning electron microscope is based on the emission of electrons produced by a cathode and the detection of signals resulting from the interaction of these electrons with the sample. Those electrons that irradiate the surface of the sample penetrate deep into the material and affect a volume called "interaction pear". The volume of this pear depends the average atomic number, the sample and the energy of the incident electrons.

- Characterization of the SEM we used :
 - Quanta 650 \FEI
 - Spectrometre bruker X flash 61no
 - Secondary electron detector (ETD)
 - Acceleration tension 30 KV
 - Zoom 150 K
 - Spot 1-10



Figure IV.13. Scanning electronic microscope (SEM)

IV.6. Optical microscope :

The microscope is a technique we used to inspect our samples in microstructural scale. We used an optical microscope called Nikon Eclipse LV150N equipped with a camera connected to a computer, which allowing us to record the micrograph observed.

The optical microscopic was elaborated in LTSM laboratory in University of Blida 1.



Figure IV.14. Optical microscope

V. Results and discussion:

V.1. Hardness test results of our control samples:

The following table represents the hardness averages of each pressure:

hardness	Hv 1	Hv2	Hv3	Moyenne
10 T	44.2	43.04	43.83	43.69
15 T	52.55	51.98	51.19	51.9066667

Table 01: the average of the results

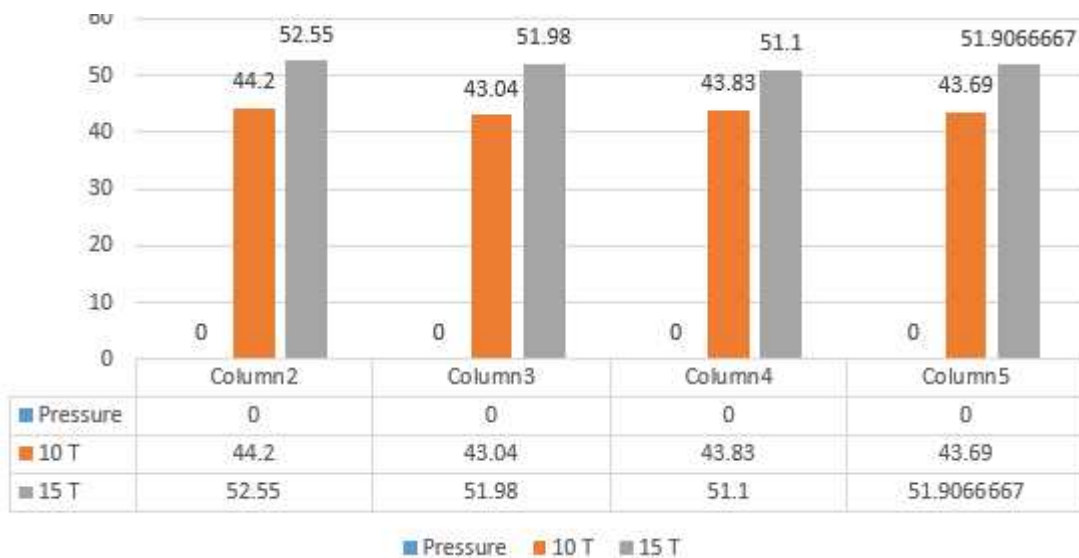


Figure V.1. histogram represents hardness as a function of pressure

The histogram in figure V.1 represents the impact of pressure to improve the hardness after these tests it was possible to deduce that there is a relationship between the compaction pressure and the hardness.

V.2. Characterization of Bismuthene:

V.2.1. Observation with A Scanning Electronic Microscope (SEM):

Figure V.2 represents a scanning electron microscope (SEM) imagery of the Bismuthene used in our work, where we can distinguish the two-dimensional aspect which corresponds to the typical Bismuthene structure. It can be observed that the few layers (figure V.2)

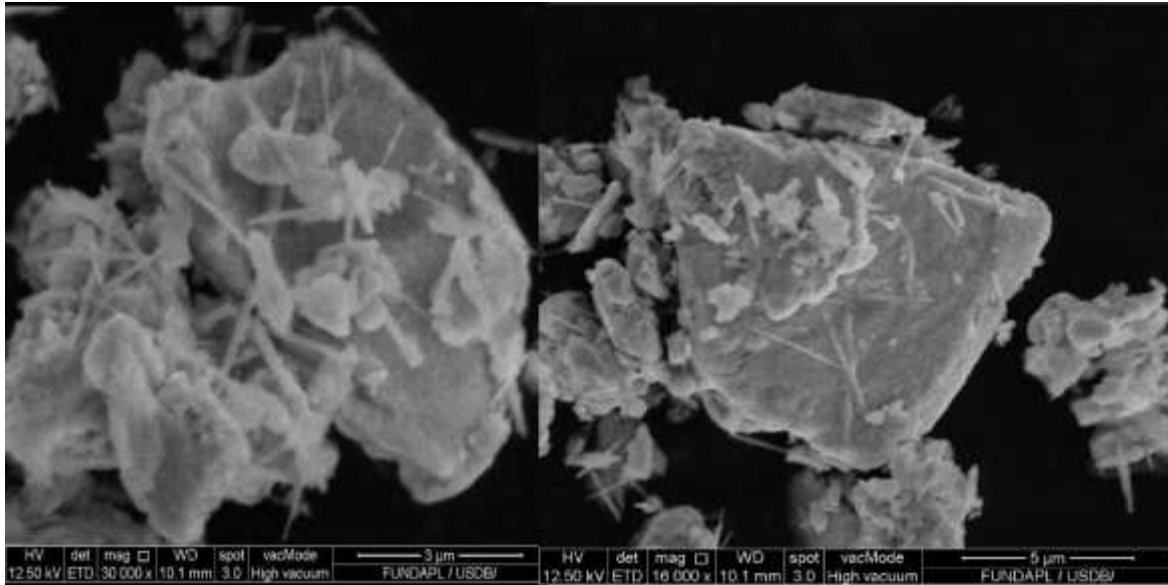


Figure V.2. a scanning electron microscope (SEM) imagery of Bismuthene

V.3. Optical microscope:

V.3.1 Before sintering:

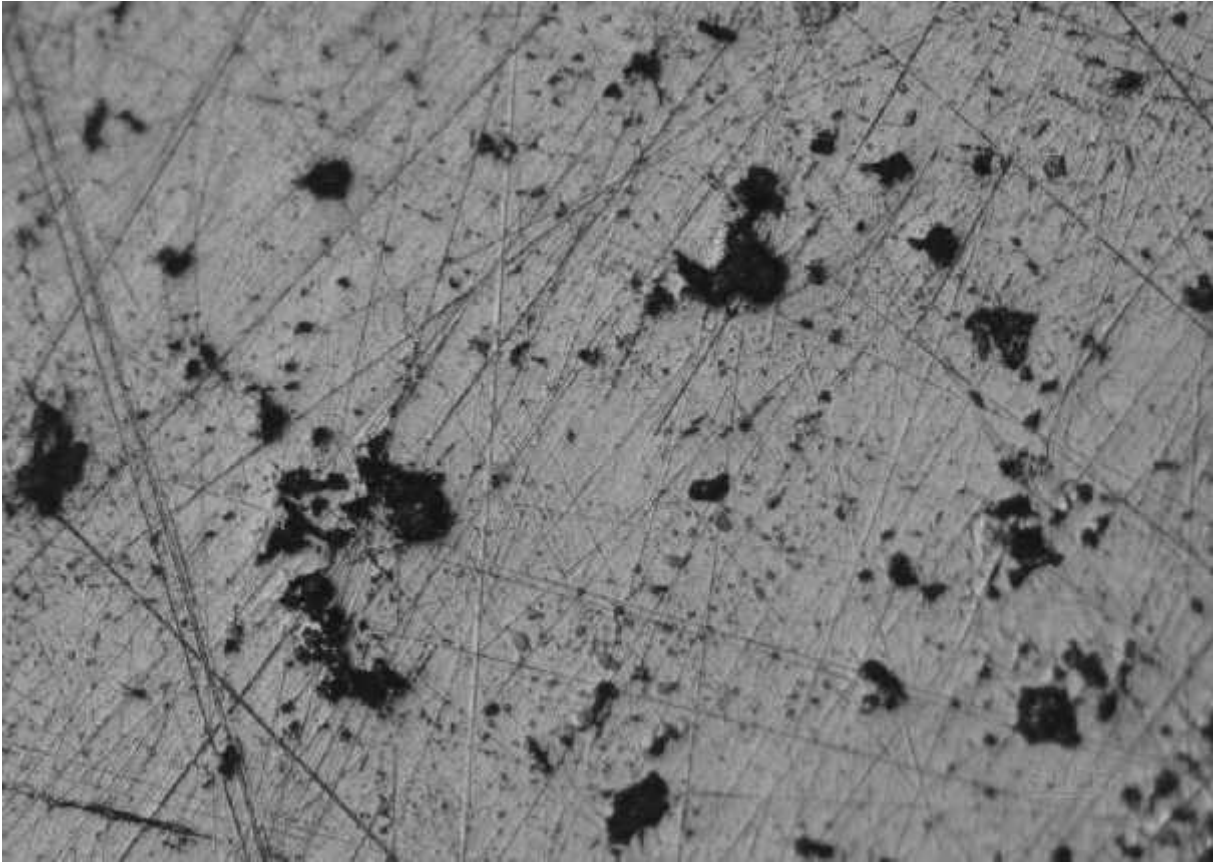


Figure V.3. Optical Microscopic imagery of Al-Bi composite before sintering

In the previous image we notice:

- A homogeneous distribution of Bismuthene particles in the aluminum matrix;
- absence of agglomeration of the reinforcement.

V.3.2. After sintering:

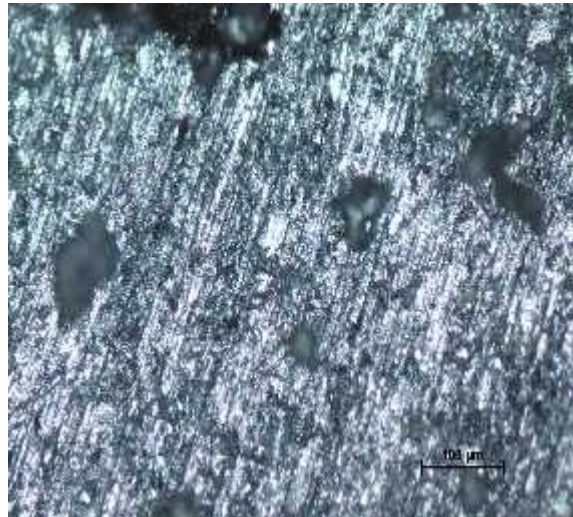
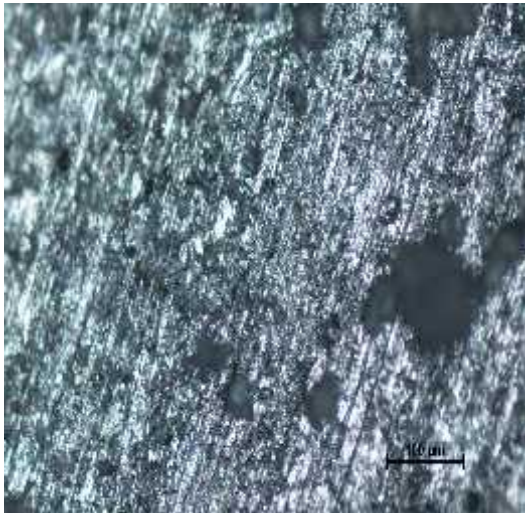
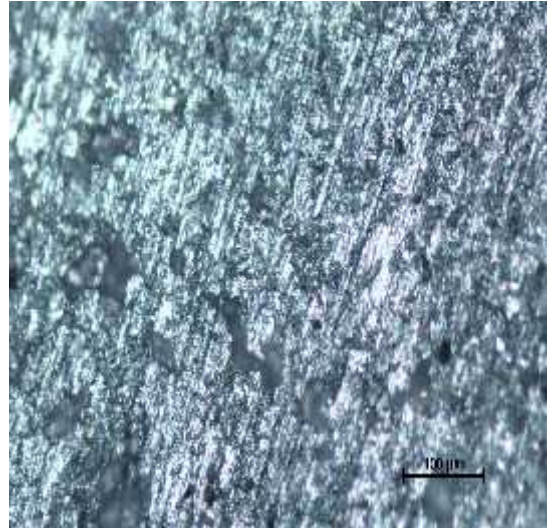
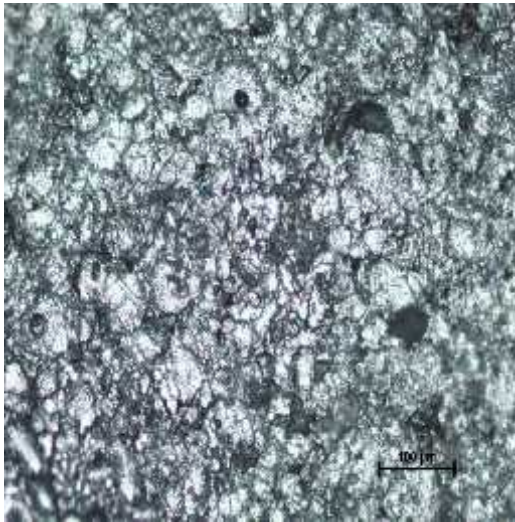


Figure V.4. Microscopic imagery of Al-Bi composite after sintering

From (Figure V.4) we suspect the creation of bismuth hydroxide or bismuth trihydroxide.

- We can see two area:
Dark area represents the bismuthine particles which where the reaction happened and the white area represents our aluminum matrix.

General Conclusion:

The objective of this work is to elaborate a two-dimensional Bismuthene material and to study its properties in a metal matrix composite.

As a first step in sample analysis, we used Scanning Electronic Microscope (SEM) to determine few layer Bismuthene. The results of the analysis showed that we successfully manufactured Bismuthene.

The optical microscope image was processed with the software (Image J) before sintering the results have showed a homogeneous distribution of Bismuthene particles, and also an absence of agglomeration.

After sintering our sample has reacted an exothermic reaction with water, we can say hydrophilicity of the Bismuthene powder is better than bismuth, we suspect the formation of bismuth hydroxide ($\text{Bi}(\text{OH})_3$), or bismuth trihydroxide, is predicted to occur, it is an incompletely characterized chemical compound of bismuth, it is generally described as bismuth oxide hydrate or bismuth hydrate, it has an alkaline behavior[28].

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[28]Bismuth hydroxide (Bi(OH)₃) (10361-43-0)