



**STUDY AND ANALYSIS OF MANUFACTURING  
CAR BODY BY CARBOTANIUM MATERIAL  
A PROJECT REPORT**

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**BONAFIDE CERTIFICATE**

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**INTERNAL EXAMINER**

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## ABSTRACT

The reaction of liquid titanium with carbon was investigated according to two different reactive melt infiltration experiments. In the first one, the titanium plate on the surface of C/C composite was rapidly melted in an arc melting furnace to investigate the reaction in a short period (less than 3 minutes). In the second one, the reactive melt infiltration was conducted in a vacuum welding furnace to investigate the reaction in a long period. The results show that a TiC layer is firstly formed on the C/C composite in a very short period by the in situ reaction of liquid titanium with carbon. The liquid titanium and carbon are separated by the reaction-formed TiC layer. The formation of the initial TiC layer is of a solution-precipitation mechanism. The further growth of the TiC layer depends on the diffusivity of carbon through TiC, which is of a diffusion control mechanism..

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## LIST OF ABBREVIATIONS

CNT	Carbon Nano Tube
TI	Titanium
GP	Giga Pascal
C/C	Carbon Copy
MD	Molecular Dynamics
CF	Carbon Fiber
CNP	Carbon Nano Particals
PEO	Poly Ethylene Oxide
PVA	Poly vinyl alcohol
PAN	Poly Acrylo Nitrile
UHMWPE	<u>Ultra High Molecular Weight Polyethylene</u>
GFRP	Graphite Nano-Reinforced Polymer
MVA	Micro Air Vehicles
ASTM	American Society For Testing and Materials
ASME	American Society of Mechanical Engineers
UTS	Ultimate Tensile Strength
TMC	Titanium Matrix Composites
CVD	Chemical Vapor Deposition
PMMA	Poly Methyl Meta Crylate
PTT	Pressure Temperature Time
AES	Advanced Encryption Standard

# **CHAPTER 1**

## **INTRODUCTION**

### **1.1 INTRODUCTION**

CNTs have been widely used as reinforcements for various metals such as aluminum, nickel, copper, magnesium, and also their alloys. Ti and its alloys are also used widely in industrial, automotive, and space applications because of their high specific strength and Young's modulus. Because of their high corrosion resistance, Ti and its alloys are widely used for chemical and petrochemical applications.

CNT reinforced Ti composites can be used in space and defense applications because these composites are lightweight and at the same time exhibit exceptional mechanical properties. The mechanical properties of Ti and its alloys can be further improved by adding CNTs. To the best of the knowledge of the authors, this will be the first study using MD for predicting the mechanical properties of the CNT–Ti composites.

MD method is based on Newton's equations of motion in which the atoms are imparted certain velocity and the system is allowed to evolve under a particular ensemble such as, constant number of atoms, volume, and temperature or a constant number of atoms, pressure, and temperature. The number of atoms selected for a particular system depends on the accuracy required in the results. Higher the number of atoms, better will be the accuracy of the results obtained. But at the same time computational cost also increases.

## **CHAPTER 2**

### **LITERATURE REVIEW**

#### **2.1 ELASTIC PROPERTIES OF CARBON NANOTUBES**

Antunes et al.,[1] explained Carbon nanotubes and their heterojunctions are efficient components for reinforcement of composites, for constructing micro- and nanodevices, and for designing new materials with required electronic and mechanical properties. This review attempts to classify the accomplishments in predicting of the elastic properties of carbon nanotubes and their heterojunctions by analytical and computational approaches. The literature results concerning Young's modulus, shear modulus and Poisson's ratio of perfect and with defects single-walled and multi-walled carbon nanotubes and their heterojunction are analysed and systematized.

#### **2.2 MG REINFORCED WITH AL<sub>2</sub>O<sub>3</sub> AND C NANOTUBES**

Schaller et al.,[2]. Discussed the high damping magnesium matrix has been reinforced either with multi-wall carbon nanotubes or with Al<sub>2</sub>O<sub>3</sub> SAFFIL short nanos (40–100 μm), the interface of which was modified by chemical vapor deposition of carbon nanotubes before their infiltration with magnesium. It was found that the addition of the carbon nanotubes increases the high temperature (500 K) shear modulus of the composite by about 20%. A thermal hysteresis is observable in the mechanical loss spectra, which may be associated with the transitory effects generally observed in the metal matrix composites. These transitory effects are strongly reduced when the reinforcements are carbon nanotubes only, smaller interface thermal stresses.

## 2.3 STRENGTHENING IN (CNT/AL) COMPOSITES

Rahul.R et al.,[3] proposed Carbon nanotubes (CNT) have a Young's modulus of 1 TPa, making them ideal reinforcements for composite materials. It is important to understand the relevant strengthening mechanisms involved in CNT/Al composites, in order to produce optimized composites. Three major mechanisms are analyzed along with experimental procedure for making CNT/Al composites.

## 2.4 TITANIUM CARBIDE NANOFIBERS-REINFORCED ALUMINUM COMPACTS

A. M. Nabawy et al.,[4] explained High Frequency Induction Heat Sintering (HFIHS) have been used to produce nanocomposites of TiC nanofibers reinforced Al matrix composites. The titanium carbide nanofibers were first synthesized by sol-gel followed by electrospinning technique and high temperature calcination process. The average diameter of the calcined nanofibers was 90 nm. The formation of the as-synthesized titanium carbide nanofibers was confirmed by X-ray diffraction pattern and transmission electron microscopy. The TiC nanofibers is then mixed with the aluminum powders and introduced into (HFIHS) to produce composites of TiC nanofibers reinforced aluminum matrix. Compaction of the mixture was at a temperature of 580 °C under the pressure of 50 MPa. A high dense Al-TiC of 99.5% relative density and around 85 HV (833 MPa) hardness has been obtained in the Al reinforced with 5 wt % TiC nanofiber. With respect to mechanical properties, the samples of Al contains 5 wt % TiC, have as high as 415 and 350 MPa, respectively value of compressive and yield strength.

## 2.5 ATTRIBUTES, CHARACTERISTICS, AND APPLICATIONS OF TITANIUM

Seattle et al.,[5] presented Titanium is an attractive material for numerous industries, but its utilization has been restricted. A broad range of activities are underway to reduce this cost. Significant cost reductions could greatly expand the industrial base. The U.S. Army would like to use it for reduced weight of armored vehicles, the U.S. Navy would like to use it for the superstructure of some of its surface ships as they tend to get top heavy, the chemical/petrochemical industry could take greater advantage of its corrosion resistance, and the aerospace industry would use more for weight savings if the price can be driven down. At present, with the only volume users being the chemical and aerospace industries, when the aerospace industry has a significant pickup in orders, such as when the Boeing 787 gets up to production rate, the Boeing requirements will be very high, and the price will go up.

## 2.6 APPLICATION OF TI - ALLOY IN AUTOMOBILE INDUSTRY

A. M. Nabawy et al.,[6] proposed to improve the safety and reliability of vehicles, consideration needs to be given to the design and manufacture, especially the manufacturing of materials. Titanium is a very good material for making bodywork frames, which not only have a high strength but also have good toughness. In Japan, automakers chose pure titanium welded pipe to make a body frame that would give motorists enough safety when driving.

## CHAPTER 3

### METHODS&MATERIAL

#### 3.1 CARBON NANOS

Carbon nanos (graphite nano or graphite fiber) are nanos about 5–10 micrometres in diameter and composed mostly of carbon atoms. Carbon nanos have several advantages including high stiffness, high tensile strength, low weight, high chemical resistance, high temperature tolerance and low thermal expansion. These properties have made carbon nano very popular in aerospace, civil engineering, military, and motorsports, along with other competition sports. However, they are relatively expensive when compared with similar nanos, such as glass nanos or plastic nanos.

To produce a carbon nano, the carbon atoms are bonded together in crystals that are more or less aligned parallel to the long axis of the nano as the crystal alignment gives the nano high strength-to-volume ratio (in other words, it is strong for its size). Several thousand carbon nanos are bundled together to form a tow, which may be used by itself or woven into a fabric.

Carbon nanos are usually combined with other materials to form a composite. When impregnated with a plastic resin and baked it forms carbon-nano-reinforced polymer (often referred to as carbon nano) which has a very high strength-to-weight ratio, and is extremely rigid although somewhat brittle. Carbon nanos are also composited with other materials, such as graphite, to form reinforced carbon-carbon composites, which have a very high heat tolerance.

### 3.1.1 Structure and Properties

Carbon nano is frequently supplied in the form of a continuous tow wound onto a reel. The tow is a bundle of thousands of continuous individual carbon filaments held together and protected by an organic coating, size such a PEO or PVA. The tow can be conveniently unwound from the reel for use. Each carbon filament in the tow is a continuous cylinder with a diameter of 5–10 micrometers and consists almost exclusively of carbon. The earliest generation (e.g. T300, HTA and AS4) had diameters of 16–22 micrometers. Later nanos (e.g. IM6 or IM600) have diameters that are approximately 5 micrometers.

The atomic structure of carbon nano is similar to that of graphite, consisting of sheets of carbon atoms arranged in a regular hexagonal pattern (graphene sheets), the difference being in the way these sheets interlock. Graphite is a crystalline material in which the sheets are stacked parallel to one another in regular fashion. The intermolecular forces between the sheets are relatively weak Van der Waals forces, giving graphite its soft and brittle characteristics.

Depending upon the precursor to make the nano, carbon nano may be turbostratic or graphitic, or have a hybrid structure with both graphitic and turbostratic parts present. In turbostratic carbon nano the sheets of carbon atoms are haphazardly folded, or crumpled, together. Carbon nanos derived from PAN are turbostratic, whereas carbon nanos derived from mesophase pitch are graphitic after heat treatment at temperatures exceeding 2200 °C. Turbostratic carbon nanos tend to have high tensile



strength, whereas heat-treated mesophase-pitch-derived carbon nanos have high Young's modulus.

### 3.1.2 Application

The strongest demand for carbon nano come from aircraft and aerospace, wind energy, as well as the automotive industry with optimized resin systems. Carbon nano can have higher cost than other materials which has been one of the limiting factors of adoption. In a comparison between steel and carbon nano materials for automotive materials, carbon nano may be 10-12x more expensive.

## 3.2 COMPOSITE MATERIALS

Carbon nano is most notably used to reinforce composite materials, particularly the class of materials known as carbon nano or graphite reinforced polymers. Non-polymer materials can also be used as the matrix for carbon nanos. Due to the formation of metal carbides and corrosion considerations, carbon has seen limited success in metal matrix composite applications. Reinforced carbon-carbon (RCC) consists of carbon nano-reinforced graphite, and is used structurally in high-temperature applications.

The nano also finds use in filtration of high-temperature gases, as an electrode with high surface area and impeccable corrosion resistance, and as an anti-static component. Molding a thin layer of carbon nanos significantly improves fire resistance of polymers or thermoset composites because a dense, compact layer of carbon nanos efficiently reflects heat. The increasing use of carbon nano composites is displacing aluminum from

aerospace applications in favor of other metals because of galvanic corrosion issues.

### 3.2.1 Carbon Nano Protector

Carbon nano filament yarns are used in several processing techniques: the direct uses are for prepregging, filament winding, pultrusion, weaving, braiding, etc. Carbon nano yarn is rated by the linear density (weight per unit length (i.e)  $1 \text{ g}/1000 \text{ m} = 1 \text{ tex}$ ) or by number of filaments per yarn count, in thousands.

. For example, 200 tex for 3,000 filaments of carbon nano is three times as strong as 1,000 carbon filament yarn, but is also three times as heavy. This thread can then be used to weave a carbon nano filament fabric or cloth. The appearance of this fabric generally depends on the linear density of the yarn and the weave chosen. Some commonly used types of weave are twill, satin and plain. Carbon filament yarns can also be knitted or braided.

### 3.3 MICROELECTRODES

Carbon nanos are used for fabrication of carbon-nano microelectrodes. In this application typically a single carbon nano with diameter of  $5\text{--}7 \mu\text{m}$  is sealed in a glass capillary.<sup>[16]</sup> At the tip the capillary is either sealed with epoxy and polished to make carbon-nano disk microelectrode or the nano is cut to a length of  $75\text{--}150 \mu\text{m}$  to make carbon-nano cylinder electrode. Carbon-nano microelectrodes are used either in amperometry or fast-scan cyclic voltammetry for detection of biochemical signaling.

### 3.4 FLEXIBLE HEATING

carbon nanos can carry very low currents on their own. When woven into larger fabrics, they can be used to reliably deliver infrared heating in applications requiring flexible heating elements and can easily sustain temperatures past 100 °C due to their physical properties.

Many examples of this type of application can be seen in DIY heated articles of clothing and blankets. Due to its chemical inertness, it can be used relatively safely amongst most fabrics and materials; however, shorts caused by the material folding back on itself will lead to increased heat production and can lead to a fire.

#### 3.4.1 Carbon Nano Reinforced Polymer

Carbon nano reinforced polymer (American English), Carbon fibre reinforced polymer (Commonwealth English), or carbon nano reinforced plastic, or carbon nano reinforced thermoplastic (CNRP, CRP, CNRTP, or often simply carbon nano, carbon composite, or even carbon), is an extremely strong and light nano-reinforced plastic which contains carbon nanos.

The spelling 'fibre' is usual outside the USA. CNRPs can be expensive to produce but are commonly used wherever high strength-to-weight ratio and stiffness (rigidity) are required, such as aerospace, superstructure of ships, automotive, civil engineering, sports equipment, and an increasing number of consumer and technical applications.

The properties of the final CNRP product can also be affected by the type of additives introduced to the binding matrix (resin). The most common additive is silica, but other additives such as rubber and carbon nanotubes can

be used. The material is also referred to as graphite-reinforced polymer or (GFRP is less common, as it clashes with glass-(nano)-reinforced polymer).

The binding polymer is often a thermoset resin such as epoxy, but other thermoset or thermoplastic polymers, such as polyester, vinyl ester, or nylon, are sometimes used.

### 3.4.2 Properties

CNRPs are composite materials. In this case the composite consists of two parts: a matrix and a reinforcement. In CNRP the reinforcement is carbon nano, which provides the strength. The matrix is usually a polymer resin, such as epoxy, to bind the reinforcements together.<sup>[1]</sup> Because CNRP consists of two distinct elements, the material properties depend on these two elements.

Unlike isotropic materials like steel and aluminum, CNRP has directional strength properties. The properties of CNRP depend on the layouts of the carbon nano and the proportion of the carbon nanos relative to the polymer. The two different equations governing the net elastic modulus of composite materials using the properties of the carbon nanos and the polymer matrix can also be applied to carbon nano reinforced plastics.

The fracture toughness of carbon nano reinforced plastics is governed by the following mechanisms: 1) debonding between the carbon nano and polymer matrix, 2) nano pull-out, and 3) delamination between the CNRP sheets.<sup>[4]</sup> Typical epoxy-based CNRPs exhibit virtually no plasticity, with less than 0.5% strain to failure. Although CNRPs with epoxy have high strength and elastic modulus, the brittle fracture mechanics present unique challenges to engineers in failure detection since failure occurs catastrophically.

Environmental effects such as temperature and humidity can have profound effects on the polymer-based composites, including most CNRPs. While CNRPs demonstrate excellent corrosion resistance, the effect of moisture at wide ranges of temperatures can lead to degradation of the mechanical properties of CNRPs, particularly at the matrix-nano interface.

## **CHAPTER 4**

### **MANUFACTURE**

#### **4.1 CARBON NANO REINFORCED POLYMER**

Primary element of CNRP is a carbon filament; this is produced from a precursor polymer such as PAN , rayon, or petroleum pitch. For synthetic polymers such as PAN or rayon, the precursor is first spun into filament yarns, using chemical and mechanical processes to initially align the polymer chains in a way to enhance the final physical properties of the completed carbon nano.

Precursor compositions and mechanical processes used during spinning filament yarns may vary among manufacturers. After drawing or spinning, the polymer filament yarns are then heated to drive off non-carbon atoms (carbonization), producing the final carbon nano. The carbon nanos filament yarns may be further treated to improve handling qualities, then wound on to bobbins.

Many CNRP parts are created with a single layer of carbon fabric that is backed with nanoglass. A tool called a chopper gun is used to quickly create these composite parts. Once a thin shell is created out of carbon nano, the chopper gun cuts rolls of nanoglass into short lengths and sprays resin at the same time, so that the nanoglass and resin are mixed on the spot. The resin is either external mix, wherein the hardener and resin are sprayed separately, or internal mixed, which requires cleaning after every use. Manufacturing methods may include the following:

#### 4.1.1 Modeling

The mold is then filled with epoxy and is heated or air-cured. The resulting part is very corrosion-resistant, stiff, and strong for its weight. Parts used in less critical areas are manufactured by draping cloth over a mold, with epoxy either pre impregnated into the nanos (also known as pre-preg) or "painted" over it.

High-performance parts using single molds are often vacuum-bagged and/or autoclave-cured, because even small air bubbles in the material will reduce strength. An alternative to the autoclave method is to use internal pressure via inflatable air bladders or EPS foam inside the non-cured laid-up carbon nano.

#### 4.1.2 Vacuum Bagging

A nano glass, carbon nano, or aluminum mold is polished and waxed, and has a release agent applied before the fabric and resin are applied, and the vacuum is pulled and set aside to allow the piece to cure (harden). There are three ways to apply the resin to the fabric in a vacuum mold.

First method is manual and called a wet layup, where the two-part resin is mixed and applied before being laid in the mold and placed in the bag. The other one is done by infusion, where the dry fabric and mold are placed inside the bag while the vacuum pulls the resin through a small tube into the bag, then through a tube with holes or something similar to evenly spread the resin throughout the fabric.

Wire loom works perfectly for a tube that requires holes inside the bag. Both of these methods of applying resin require hand work to spread the resin evenly for a glossy finish with very small pin-holes.

A third method of constructing composite materials is known as a dry layup. Here, the carbon nano material is already impregnated with resin (pre-preg) and is applied to the mold in a similar fashion to adhesive film. The assembly is then placed in a vacuum to cure.

#### 4.1.3 Compression Modeling

A quicker method uses a compression mold. This is a two-piece (male and female) mold usually made out of aluminum or steel that is pressed together with the fabric and resin between the two. The benefit is the speed of the entire process. Some car manufacturers, such as BMW, claimed to be able to cycle a new part every 80 seconds. However, this technique has a very high initial cost since the molds require CNC machining of very high precision.

#### 4.1.4 Filament Winding

For difficult or convoluted shapes, a filament winder can be used to make CNRP parts by winding filaments around a mandrel or a core.

### 4.2 APPLICATION

- Aerospace engineering
- Automotive engineering
- Carbon nano micro electrodes
- Sports



## CHAPTER 5

### TITANIUM ALLOY

#### 5.1 TITANIUM ALLOY

Titanium alloys are metals that contain a mixture of titanium and other chemical elements. Such alloys have very high tensile strength and toughness (even at extreme temperatures). They are light in weight, have extraordinary corrosion resistance and the ability to withstand extreme temperatures.

##### 5.1.1 Properties

Titanium dioxide dissolves in the metal at high temperatures, and its formation is very energetic. These two factors mean that all titanium except the most carefully purified has a significant amount of dissolved oxygen, and so may be considered a Ti – O alloy.

#### 5.2 TITANIUM GRADES

The ASTM International standard on titanium and titanium alloy the following alloys, requiring the following treatment:

"Alloys may be supplied in the following conditions: Grades 5, 23, 24, 25, 29, 35, or 36 annealed or aged; Grades 9, 18, 28, or 38 cold-worked and stress-relieved or annealed; Grades 9, 18, 23, 28, or 29 transformed-beta condition; and Grades 19, 20, or 21 solution-treated or solution-treated and aged.

### 5.2.1 Grade 1

The most ductile and softest titanium alloy. It is a good solution for cold forming and corrosive environments. ASTM/ASME SB-265 provides the standards for commercially pure titanium sheet and plate.

### 5.2.2 Grade 2

Un alloyed titanium, standard oxygen.

### 5.2.3 Grade 2H

Un alloyed titanium (Grade 2 with 58 ksi minimum UTS).

### 5.2.4 Grade 3

Un alloyed titanium, medium oxygen.

Grades 1-4 are unalloyed and considered commercially pure or "CP". Generally the tensile and yield strength goes up with grade number for these "pure" grades. The difference in their physical properties is primarily due to the quantity of interstitial elements. They are used for corrosion resistance applications where cost, ease of fabrication, and welding are important.

## 5.3 HEAT TREATMENT

Titanium alloys are heat treated for a number of reasons, the main ones being to increase strength by solution treatment and aging as well as to optimize special properties, such as fracture toughness, fatigue strength and high temperature creep strength. Alpha and near-alpha alloys cannot be dramatically changed by heat treatment. Stress relief and annealing are the processes that can be employed for this class of titanium alloys. The heat

treatment cycles for beta alloys differ significantly from those for the alpha and alpha-beta alloys.

Beta alloys can not only be stress relieved or annealed, but also can be solution treated and aged. The alpha-beta alloys are two-phase alloys, comprising both alpha and beta phases at room temperature. Phase compositions, sizes, and distributions of phases in alpha-beta alloys can be manipulated within certain limits by heat treatment, thus permitting tailoring of properties.

### 5.3.1 Alpha and Near-Alpha Alloys

The micro-structure of alpha alloys cannot be strongly manipulated by heat treatment since alpha alloys undergo no significant phase change. As a result, high strength can not be acquired for the alpha alloys by heat treatment. Yet, alpha and near-alpha titanium alloys can be stress relieved and annealed.

### 5.3.2 Alpha-Beta Alloys

By working as well as heat treatment of alpha-beta alloys below or above the alpha-beta transition temperature, large micro-structural changes can be achieved. This may give a substantial hardening of the material. Solution treatment plus aging is used to produce maximum strengths in alpha-beta alloys. Also, other heat treatments, including stress-relief heat treatments, are practiced for this group of titanium alloys as well.

### 5.3.3 Beta Alloys

In commercial beta alloys, stress-relieving and aging treatments can be combined.

## CHAPTER 6

### EXPERIMENTAL MATERIALS AND METHODS

#### 6.1 COMPOSITE MAKING

During the last three decades, most studies related to TMC's have been dedicated to the reinforcement of titanium or aluminide alloys by large diameter filaments (100-140  $\mu\text{m}$ ) processed by CVD. However, the cost of this type of filamentary reinforcement, the related conditions of composite manufacturing and also the large size of the reinforcement, impede their development and their use in applications requiring a finer subdivision of the reinforcement and moderate manufacturing costs.

Among the available fibrous reinforcement, carbon nanos are particularly interesting, since their stiffness can be so high that a rather small volume fraction of reinforcement (20 % of high modulus graphite nanos) is able to give an unidirectional composite the rigidity of steel without inducing an unavoidable embrittlement of the composite metallic component.

Thus, carbon nanos reinforced TMC's exhibit interesting perspectives provided to limit the nano/matrix (F/M) interaction during composites processing. As a matter of fact, the high temperatures generally required for TMC manufacturing (800-900°C) and the particularly strong chemical reactivity of the Ti/C couple, inevitably give rise to the formation of a brittle carbide zone at the nano/matrix interface resulting in undesirable notch effects and nanos degradation.

Various ways can be followed to overcome the difficulty of incorporating fine carbon nanos within titanium base alloys while limiting the F/M interaction:

- The use of a liquid infiltration route requires techniques which are not yet sufficiently rapid to avoid any excessive F/M interaction [7, 8].
- The vapor route based for instance on the physical vapor deposition, and followed by complementary operations of consolidation is rather expensive despite the flexibility of using impregnated nanos yarns or fabrics for manufacturing parts.
- The solid route can be used provided to subdivide finely the titanium base matrix in order to realize its coupling with fine nanos. Such a subdivision is possible in the case of filaments about a hundred microns in diameter thanks to the use of titanium foils or powders commercially available, but cannot be adapted to nanos about ten microns in diameter.

In the case of most titanium rich alloys, not only their significant ductility and reactivity makes much more difficult their subdivision without contamination, but also their densification by hot pressing in presence of carbon nanos requires particular conditions to control the highly reactive F/M interaction.

Thus, processing TMC through powder metallurgy techniques meets the following impeding difficulties, which are of concern in the present contribution :

- The preparation of fine titanium powders with grain size ranging between 1 and 10  $\mu\text{m}$ ,
- The protection of carbon nanos against the titanium chemical aggressiveness,
- The choice of a polymeric binder and the penetration of matrix powders within various fibrous architectures,
- The composite densification.

## 6.2 PROCESS OF A NON CONTAMINATED FINE TITANIUM POWDER

The method generally used for producing fine titanium powder from titanium hydride has been improved to decrease grinding duration and impurities contamination. The method consists in combining hydriding/dehydriding cycles and grinding, which enables a faster subdivision of titanium. The procedure and efficiency of the method are detailed elsewhere.

It is noteworthy that a spontaneous subdivision of titanium hydrides by cycling hydrogen pressure at suitable temperature (400-500°C) is not possible contrary to many intermetallic hydrides [10, 11]. The titanium hydride decrepitation is impeded by the titanium ductility, which also impedes the direct subdivision of titanium powders. In this module, user details are register into database.

After 20-30 hydriding/dehydriding cycles at 500°C under maximum and minimum hydrogen pressures of  $10^5$  Pa and  $10^{-1}$  Pa followed by only 7 min of grinding, the resulting titanium hydride powder exhibits a granulometry of about 5  $\mu\text{m}$  and a low contamination (300 ppm of iron).

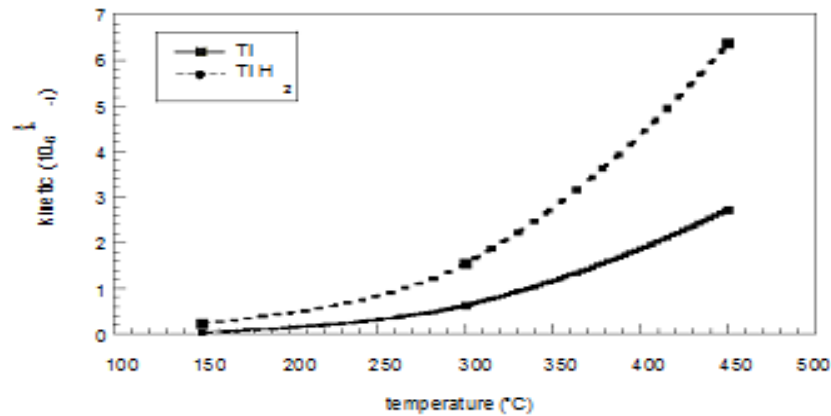


Fig. 1: Oxidation kinetics of hydride and unhydride d titanium powders

Figure 6.2 Hydride And Unhydrided Titanium Powder

### 6.3 PROTECTION OF CARBON NANOS

Various types of carbon nanos protection were tested: (1) the interposition of an ephemeral metallic barrier at the F/M interface, (2) the coating of carbon nanos by a less reactive pyrolytic carbon layer, (3) the deposition on nanos of a quasi chemically inert boron nitride layer.

In the first case, the great stability of the C/Cu/W system, also the slow and favorable W/Ti interaction (W helps ductile Ti<sub>2</sub>O<sub>3</sub> formation) led to consider Cu/W coatings as interesting candidates for nanos protection. The study of this system has been detailed elsewhere and shows its rather low efficiency although the presence of a W layer impedes copper spreading within matrix and TiC formation [13].

The second case, corresponding to the deposition on nanos of 70 nm of pyrolytic carbon, aims at decreasing the reactivity at the F/M interface thanks to a more favorable orientation of graphitic planes on nanos surface. That allows the formation of carbide interphase without partly consuming nanos.

As shown by AES analyses presented elsewhere, this type of pyrocarbon protection leads to the formation of 250 and 150 nm of TiC after isothermal exposures at respectively 750 and 700°C during half an hour [14].

Concerning the third case, the interposition of 0.8-1.1  $\mu\text{m}$  of BN between carbon and titanium appears to be efficient to protect carbon nanos. As a matter of fact, processing TMC reinforced by BN coated carbon nanos, at 700 °C during half an hour leads to F/M interfacial zones quasi free of TiC.

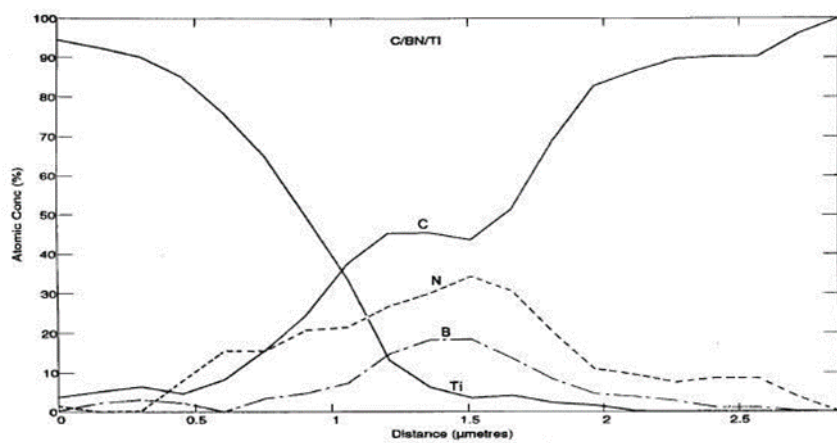


Figure 6.3 AES Concentration Profiles Related To Tmc Reinforced By Bn Coated Carbon Nanos

Anyway, the various tests of carbon nano protection whose main illustrative results are gathered in Table 1, show that processing carbon nanos reinforced TMC's at temperatures of about 700°C, is not inevitably accompanied by the formation of brittle interphases in such thicknesses that carbon nanos could not undergo the related notch effects.



M40 C nano coating	None	1.7 $\mu\text{m}$ of Cu		0.7 $\mu\text{m}$ of pyr.C		0.9 $\mu\text{m}$ of BN
Processing time (min)	30	30	20	30		30
Pressure P (MPa)	200	15	20	200	200	200
Processing T ( $^{\circ}\text{C}$ )	700	830	750	700	700	700
Interfacial zone ( $\mu\text{m}$ )	0.5	4	1.5	1.2	1.2	1.2
TiC thickness ( $\mu\text{m}$ )	0.5	1.4	0.4	< 0.1	< 0.1	<0.1

Table 6.2 F/M interaction zone

#### 6.4 PENETRATING THE MATRIX POWDER

In the case of the PMMA, a lower temperature ( $250^{\circ}\text{C}$ ) enables the decomposition and the complete elimination of the polymer after 5 hours in air without leaving any residual binder. Furthermore, the use of a PMMA solvent allows its viscosity to be easily adjusted for facilitating fibrous preform impregnation.

It is worthy of note the binder elimination time is obviously strongly dependent on structure shapes. Higher the structure surfaces for a given volume, shorter the binder elimination time. That is particularly the case of thin wall structures.

After testing various polymers, two thermoplastics were selected for thermogravimetric investigations concerning the conditions of their elimination at moderate temperatures.

## 6.5 COMPOSITES COMPACTION

short carbon nanos were used for manufacturing composites with reinforcement volume fraction of about 20-30 %. Lowering the densification temperatures and therefore limiting the F/M interaction, can be expected by increasing the compaction pressures of the matrix powder / carbon nanos mixture.

These PTT cycles aim at generating the following sequences of composite densification:

- Titanium base particles surrounding carbon nanos are plastically deformed as the compaction pressure increases. Although the plastic flow stress of the matrix remains significant at the densification temperatures, this mechanism of plastic deformation is important because of the small contact surfaces between particles. Also, this step is capable of causing mechanical nano degradations.
- Then, particles creep is able to carry on the densification thanks to diffusion mechanisms within particles. This step is not expected to provoke nano mechanical degradations, although, during short times (half an hour), the driving force of the related mechanism is as much pressure as temperature.
- The sintering mechanism involving inter particles diffusion is able to close residual pores and to induce particles bonding, but also it must be related to F/M interaction. Thus, more efficient the nano protection, more complete can be the matrix densification and consolidation.

It remains from the previous considerations, the necessity of optimizing PTT cycles to prevent any nano breakage while reaching the full matrix density.

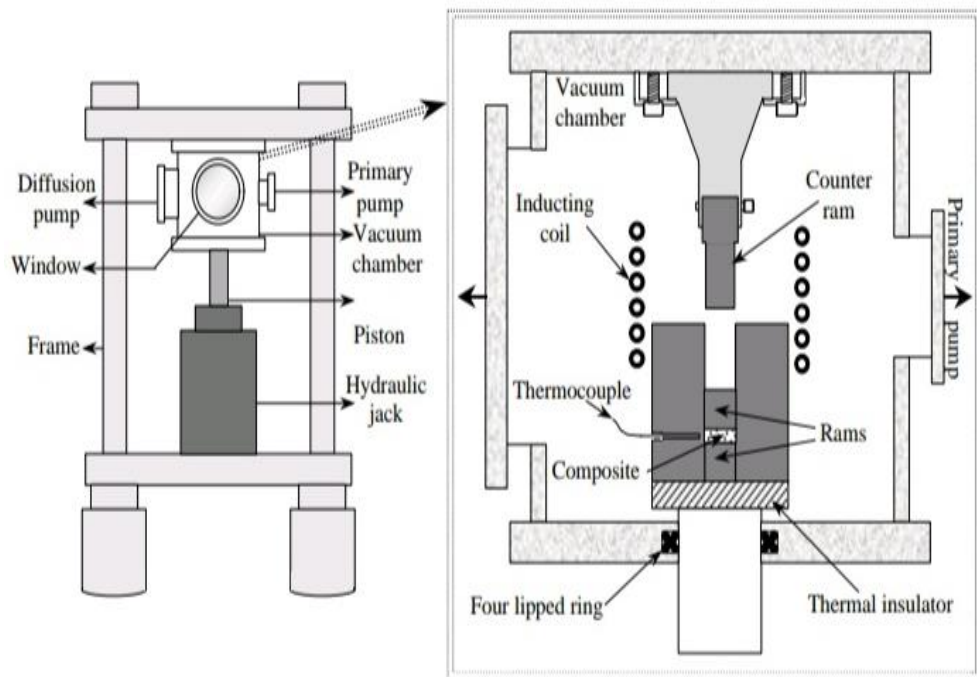


Fig. 3: Schematic representation of the hot pressing apparatus used for TMC processing

Figure 6.5.1 Schematic Representation of the Hot Pressing Apparatus Used for TMC Processing

The apparatus enables first the elimination of binder and then the titanium dehydrogenating under vacuum of  $5 \cdot 10^{-3}$ - $10^{-4}$  Pa, which allows the use of moderate temperatures and consequently only induces very small oxygen contamination. At higher temperatures, related to the matrix densification, the confinement of the titanium powder in the die also limits this matrix contamination by interstitial elements.

Besides, in any case, with the conditions of processing reported in Table 1, the matrix microstructure was observed to be very fine and free of pores as illustrated in Fig. 4. Below temperatures of 800°C, the microstructure size is close to the nano diameter, which is like expected since coarsening effects have not enough time to develop at the densification temperature.

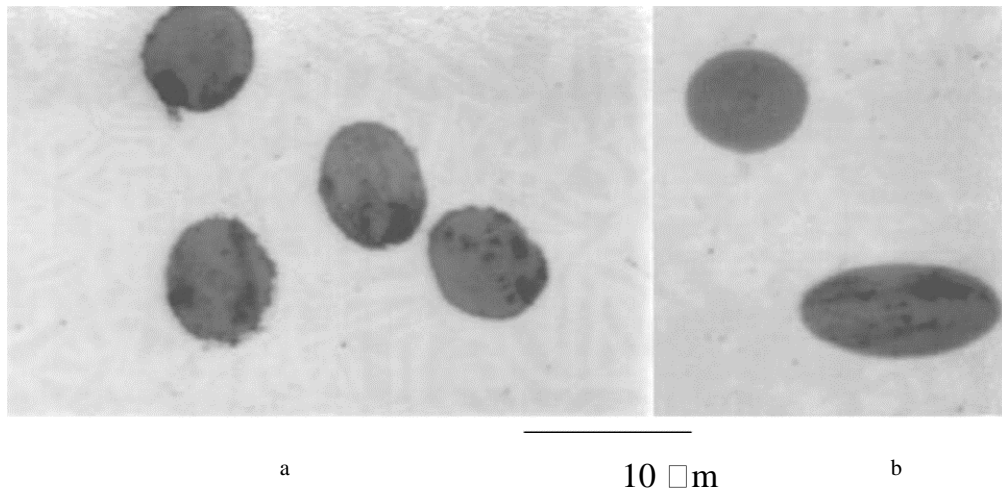


Figure 6.5.2 Optical micrographies of carbon nanos reinforced CMT's

(a)  $P = 200 \text{ MPa}$ ;  $T = 700 \text{ }^\circ\text{C}$ ;  $t = 30 \text{ min}$

(b)  $P = 650 \text{ MPa}$ ;  $T = 600 \text{ }^\circ\text{C}$ ;  $t = 30 \text{ min}$

Also Fig. 4 shows how profitable is a decrease in the processing temperature for the F/M interface. In the case of C coated carbon nanos, a reduction of 100 °C of the processing temperature enables a quasi elimination of the F/M interaction zone (Fig. 4 b), but the corresponding processing pressure (650 MPa) is uneasy to use on large arts in an industrial context. Furthermore, very high pressures require an optimization of the PTT cycle in order to prevent excessive nanos breakage.

## 6.6 MATERIALS

The matrix was an Titanium alloy with 6% magnesium in the composition AMg6. There were also 0.4% Si, 0.4% Fe, 0.1% Cu, 0.6% Mn, 0.2% Zn in the alloy.

## 6.7 COATING

Titanium n-butoxide ( $\text{Ti}(\text{C}_4\text{H}_9\text{O})_4$ , 99%, Acros) was used as a source of titanium alloy. The preparation of sol was carried out according to the following procedure: titanium but oxide was added to the mixture of solutions (butanol, acetic acid, acetylacetone) with stirring. The resulting solution was stirred 1 hour at room temperature. Then a mixture of distilled water and butanol were adding slowly dropwise while vigorously stirring for 1 hour. The prepared sol-gel solution was aged for 3 days to achieve completed hydrolysis.

## 6.8 THE FABRICATION METHOD

Composites reinforced with continuous uncoated and Ti alloy-composited carbon nanos were fabricated by one of the varieties of the liquid-phase infiltration method under pressure - the shell molding process. The fabrication technology of the CN/Al plates was described. In the present study, the pressure applied during pressing was 70 MPa. The nano volume fraction was 30%.

## 6.9 THREE-POINT BENDING TEST AND CHARACTERIZATION

For three-point test plane-parallel samples of composites with dimensions 70x10x2 were prepared. The loading was carried out before the failure of the sample. The maximum strength values were obtained by the following equation:

$$\sigma_b = \frac{3F_{\max} L}{2bh^2}, \quad (1)$$

where  $F$  is the maximum load preceding failure;  $b$  is specimen width;  $h$  is specimen thickness;  $L$  is the distance between the supports.

## CHAPTER 7

### RESULT AND DISCUSSIONS

#### 7.1 TI ALLOY-CARBON NANO COMPOSITE

Using scanning electron microscopy (SEM), images of the surface of the nanos before (figure 1a) and after coating (figure 1b) were obtained. In the initial state, carbon nanos contain many longitudinal strips. After coating, the nano surface is less banded, individual particles on the surface of different shape are observed.

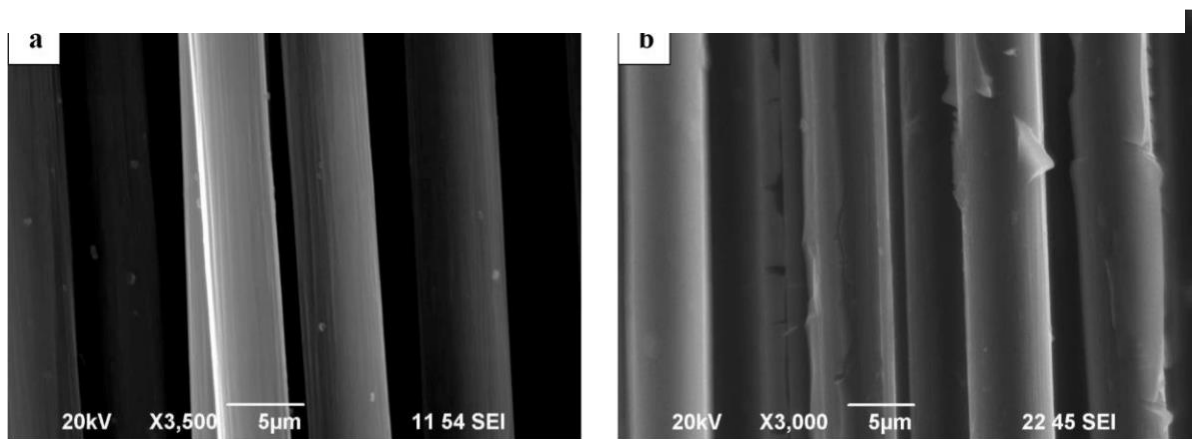


Figure 7.1 Sem Images Of Ti Alloy Carbon Nano Composite

#### 7.2 EVALUATION OF COATING PROTECTIVE PROPERTIES

To evaluate the protective properties of coating, the nanos were heated to temperature 600°C and 700°C [11, 13, 14]. After 6 hours at a temperature of 600°C, the weight loss of the coated carbon nano was about 35%, while the mass loss of uncoated carbon nano was about 67%.

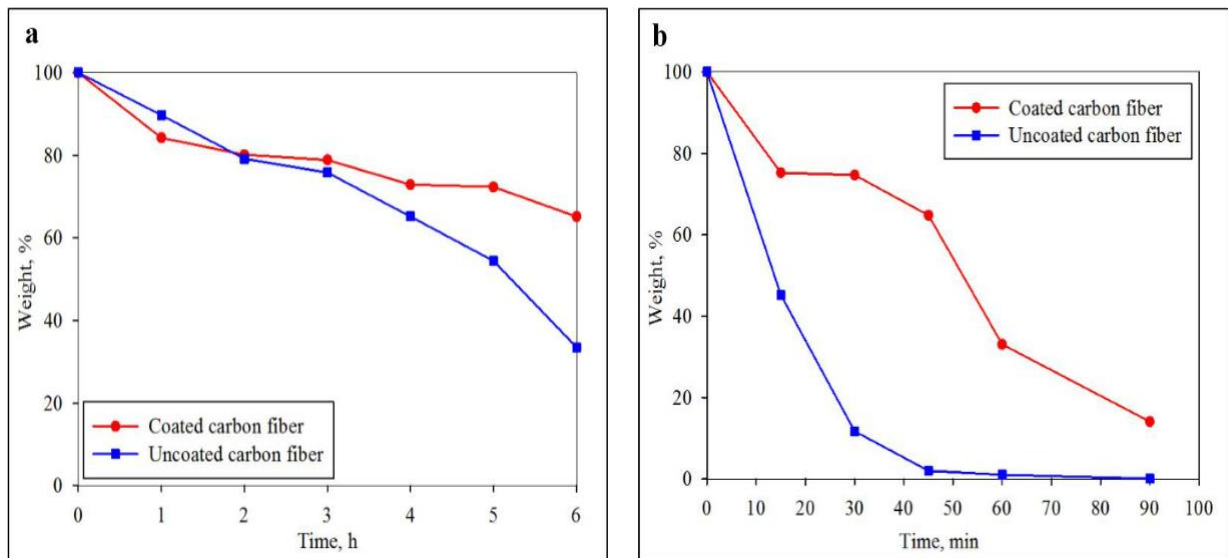


Figure 7.2 Plots Of The Carbon Nanos Weight Loss The Temperature

(a) 600°C and 700°C

(b) At a temperature of 700 °C

The burnout process is accelerated. After 90 minutes of exposure, the weight loss of the coated nano is about 86%. At the same time, uncoated nano is completely burned out. Thus, the TiO<sub>2</sub> coating applied by the sol-gel method according to the specified earlier regime significantly slows down of the nano oxidation process in the studied temperature range. This is a premise for using TiO<sub>2</sub> as a protective coating in the fabrication of CN/Al composite.

### 7.3 MICROSTRUCTURE OF COMPOSITES

Typical microstructure of CN/Al composites in transverse section of the direction of nano presented.

That the uncoated nanos composite has areas not infiltrated by aluminum. This is most likely due to the insufficient pressure of infiltration because of the almost complete absence of wetting of the surface of high-modulus carbon nano by matrix melt.



Under the same fabrication conditions, in the composite with the coated nanos in figure 3b there are no voids. This fact suggests that the TiO<sub>2</sub> coating improves wetting with an Al-6Mg melt, which means that less pressure is required to infiltrate the carbon nano preform with the alloy.

Bright inclusions can be observed in the matrix. Using local chemical analysis, it was established that these are particles of titanium oxide that were not in direct contact with the nano, and which were mentioned earlier in paragraph 3.1. It should also be noted that in the composite with the coated nanos no contacting nanos are observed in the images, which is an additional advantage of using the TiO<sub>2</sub> coating.

#### 7.4 BENDING TEST

The average bending strength of the composite with uncoated carbon nanos was 254 MPa, with titanium oxide coated carbon nanos was 667 MPa. In addition, the fracture behavior of the composites correlates with the strength values [4, 5, 15, 16]. The flat fracture surface of the uncoated nanos composite hints at a brittle fracture, and in areas without a matrix, rare individual nanos are pull-out. In the TiO<sub>2</sub>-coated nanos composite bundle fracture takes precedence, but rare single-nano fracture is also found here.

Thus, it can be concluded that in the case of using uncoated nanos, brittle fracture with low strength is most likely due to a strong bond at the nano-matrix interface due to the formation of aluminum carbides [15, 16]. In the case of using TiO<sub>2</sub>-coated nanos, the bond at the interface is less strong, as evidenced by the results of mechanical tests and the fracture behavior.

## CHAPTER 8

### CONCLUSION

In this paper, the possibility of using carbon nanos titanium alloy composites matrix was studied and the following conclusions were made:

- Carbon nanos with a Ti alloy in air at elevated temperatures much slower than uncoated nanos. So the weight loss of the coated nano at 600°C (6 hours) and 700°C (1.5 hours) turned out to be about 50% and 15% less respectively. Although the coating on the nanos was fairly uniform, it had a few cracks, which could negatively affect the protective properties.
- Under the same conditions of fabrication for infiltration of the entire Tialloy-coated carbon nano preform by aluminum alloy melt, it is necessary to apply less pressure in comparison with the uncoated nanos preform.
- The average bending strength of the uncoated carbon nanos composite was 254 MPa, while the strength of the Tialloy coating carbon nanos composite was more than 2.5 times higher — 667 MPa. This phenomenon can be explained by a strong bond between the matrix and the nano, resulting from the formation of aluminum carbides during brittle fracture, and the lower strength of such a bond in the absence of carbides, which does not allow the crack to free propagate in the material.

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