DESIGN AND CHARACTERIZATION OF ANISOGRID LATTICE STRUCTURES WITH CARBON NANOTUBES

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Abstract

The unique carbon nanotubes properties (mechanical, electrical, thermal, etc.) are considered as a key factor for future improvement of many engineering macro and nano systems characteristics.
A possible field of application of advanced composite materials is represented by innovative and recently introduced "Anisogrid Lattice Structures", realized in the form of a thin-walled shell (cylindrical or conical) with a system of a helical and circumferential ribs (with or without internal and external skin). Using this typology of structures, it is possible to satisfy high requirements of static resistance and stability (local and global buckling) in the minimum mass condition (Vasiliev Model). Further improvements are expected by introducing nanotubes reinforced composites.
This new generation of structures and composite materials find concrete and interesting application in the aerospace technologies.
This paper reports the authors' studies on the synthesis (arc discharge and laser ablation methods), purification (oxidation, chemical etching, ultrasound) and morphological analysis (Optical, SEM, TEM, EDS) of carbon nanotubes. The mechanical behaviour of a cylindrical anisogrid lattice elements (using typical aerospace materials and nanotubes), simulated by both numerical and FEM analysis, are discussed. The mechanical test of the polymeric matrix reinforced with nanometric elements are shown. Besides, the activities are illustrated relevant to design and manufacturing of Anisogrid Lattice Structures (plate and cylindrical geometry) prototypes.

Key words: carbon nanotubes, anisogrid lattice structures.

1. Introduction

The carbon nanotubes (CN) [3] and the anisogrid lattice structures [4] allows to improve the performances and the characteristics of many Engineering Advanced Systems. In particular, the aerospace sector finds great interest in developing a structural elements with the properties of high mechanical resistance, stability, lightness. Besides, the electrical, thermal and magnetic properties of CN, find a concrete application in the space sub systems (nanoelectronic devices for example).
The primary problem is the high costs required in real applications of this new king of material and structure.
From the CN's synthesis to the prototype realization is necessary to follow the below scheme:

- CN's synthesis and purification
- CN's morphological analysis
- Study of the composite CN reinforced
- Mechanical test (static, dynamic)
- Prototypes numerical design
- Manufacturing and test of the prototypes.

This paper shows the activities of the research program above illustrated.

2. Synthesis and purification of Carbon Nanotubes

For the carbon nanotubes synthesis three facilities where developed:

- Arc discharge in inert environment [6]
- Water immersed arc discharge [10]
- Laser ablation CO_2 [7]

Every synthesis method allows to obtain different CN's morphologies. The quantity and the quality of CN synthesized are the primary parameters for their integration in an engineering device.
By the arc discharge it is possible to produce high quantity of CN with no good alignment and with a lot of impurities, but poor of crystallographic defects. Using the laser ablation, in correspondence of the same quantity produced, the purification degree is higher.
With the CVD (chemical vapour deposition, not studied in this paper) high alignment is obtained, but with low quantity produced and with much crystallographic defects [3].
The choice of the method is function of the engineering system typology in which the integration of the CN is necessary. In the structural applications always high CN quantity is requested (alignment and purity aren't the

principal requirements). On the contrary, the nanoelectronic devices require low CN's quantity, but very high alignment and purification degree.
All synthesis experiment are performed using graphite rods (diameter: 6.15 mm) with high purity (2 ppm)
Fig. 2.1 shows the arc discharge (inert environment) developed [8]. The followings parameters are employed:

- Dc voltage (20-25 V)
- Amperage: 50-60 A
- Inert environment (helium gas, flow pressure: 0.2 bar).

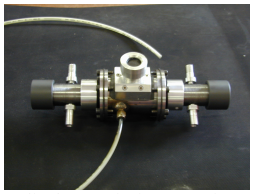

fig. 2.1 the arc discharge (in inert environment) facility

The examples of the material synthesized are shown in fig. 2.2 & 2.3

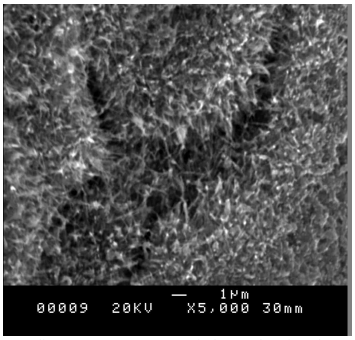

fig. 2.2 nano material synthesized

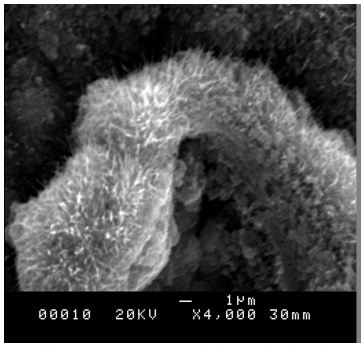

fig. 2.3 nano material synthesized

The water immersed arc discharge method [10] has been tested with the below parameters:

- Dc voltage (20-25 V)
- Amperage: 60-90 A
- De-ionized water.

In fig. 2.4 the arc is shown during a preliminary test

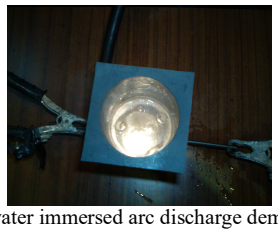

fig. 2.4 water immersed arc discharge demonstrator

Fig.s 2.5 & 2.6 illustrate the material produced with the water immersed arc discharge method.

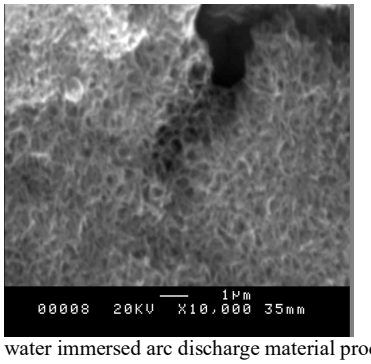

fig. 2.5 water immersed arc discharge material produced

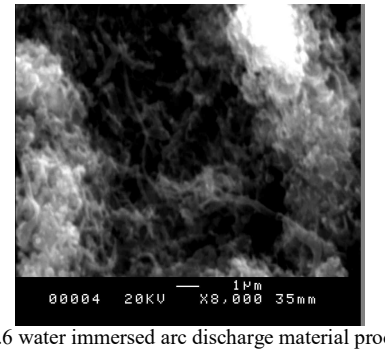

fig. 2.6 water immersed arc discharge material produced

Analysing the figures 2.2, 2,3, 2.5 and 2.6 it is possible to understand how the use of a different synthesis method and parameters gives a significant variation in the morphologies, quality and quantity of the material produced.
The capability to obtain the complete control, and reliability too, of the synthesis process is primary target for the use of CN. Besides quantity, quality and relative costs are direct consequence.
The CO_2 laser ablation tests (fig. 2.7) are performed with the following parameters [7]:

- Power: 900 W
- Wave length 10.6 μm
- Argon flow: 60 l/min

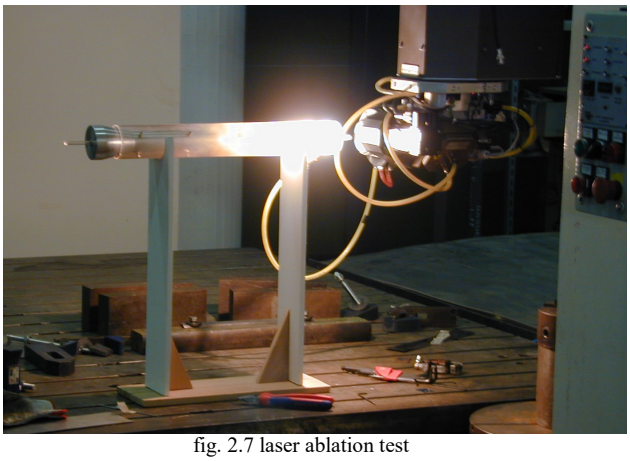

fig. 2.7 laser ablation test

Fig. 2.8 illustrates the deposit picked to the collector. The deposit contains a fibre like nanostructures. Again, the difference is evident among the various synthesis methods. Besides, it is possible to produce CN using the laser Nd-Yag. With the CO_2 the synthesis are performed without the use of infrared oven. Instead with the Nd-Yag the above oven is

requested. In fact, for a good CN's production it is necessary to reach high temperature to sublimate the carbon contained in the graphite target, and to build the tubular geometry characteristic of the carbon nanotubes.

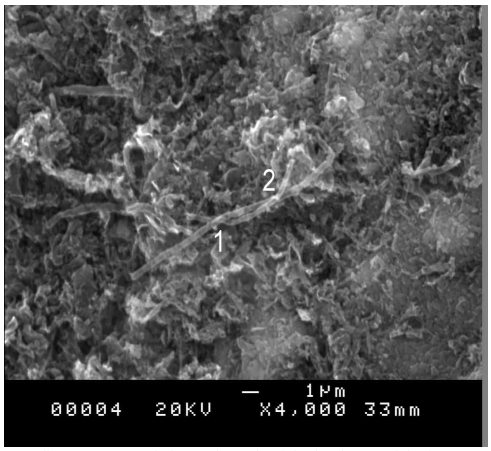

fig. 2.8 material produced with the laser ablation

After the synthesis a CN's, a purification process is required to maximise the intrinsic properties of the nanomaterial and of the device in which it is integrated. Three typical methods are available [3]:

- chemical etching
- oxidation
- ultrasound treatment

With the chemical etching it is possible to eliminate from the deposit the catalysts employed for the carbon nanotubes synthesis and growth. The better purification methodology is the oxidation. Thanks to the different chemical reactivates of the amorphous graphite and the CN, it is possible to obtain a deposit containing mainly nanotubes and a little quantity of impurity. The DTA & TG tests are performed to evaluate the purification degree of the deposit removed from the cathode electrode subject to the arc discharge in inert environment. The parameters used are:

- 50 mg of synthesized material
- oxidizing flow: N_2 90%, O_2 10 %
- T_{max}: 780 °C
- Time: 5 h.

Figure 2.9 shows the DTA-TG data experiment. Between 500 and 600 °C (DTA curve) it is possible to observe a chemical reaction of the material. The preferential oxidation of amorphous carbon is expected to occur.

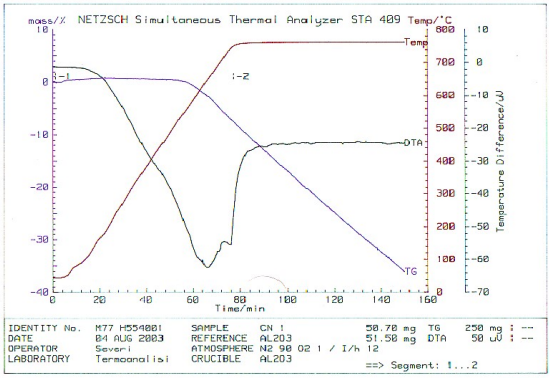

fig. 5.2 DTA – TG test results

Consequently, the purification process was successively performed with a new sample in the DTA apparatus using the following experimental conditions:

- fluxing in (slightly) oxidant atmosphere (N_2 90%, O_2 10%)
- T max = 530 °C
- Time: 2.5 hours.

In fig. 2.10 the morphologies are illustrated of the cathode deposit before the purification, and in fig. 2.11 the same deposit after the DTA test. Comparing the figures it is evident the major deposit purification degree after the purification treatment at 530 °C. Since the impurity are embedded in the CN's structure, using the oxidation method it is not possible to obtain a complete destruction of the undesired elements (amorphous graphite, catalysts, nanoparticles, etc.).

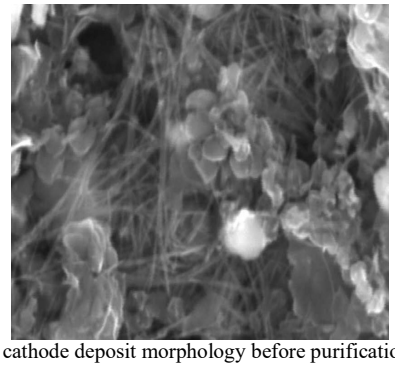
fig. 2.10 cathode deposit morphology before purification (DTA)

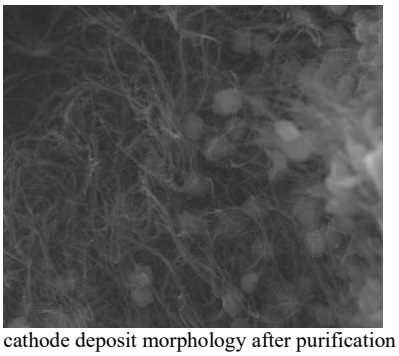
fig. 2.11 cathode deposit morphology after purification (DTA)

After the synthesis, and in particular in the case of the arc discharge method, the CN show a typical entangled configuration with a different quantity of impurity.
During the ultrasound treatment, by the vibration generated it is possible to deploy the CN bundles [5]. A ultrasound purification experiment is performed: 50 mg of cathode deposit is purified for 30 minutes. In fig. 2.12 and 2.13 the morphology differences between the deposit before and after the ultrasound test are shown.

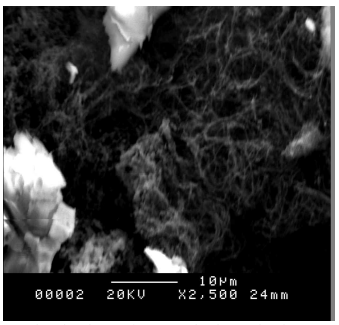

fig. 2.12 cathode deposit morphology before purification (ultrasound)

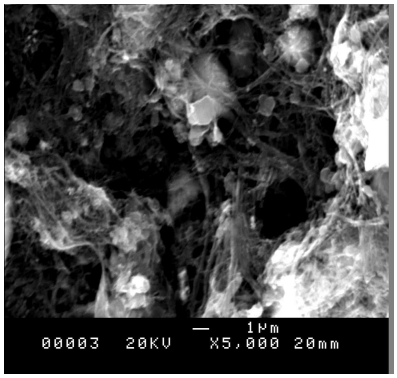

fig. 2.13 cathode deposit morphology after purification (ultrasound)

3. Morphological analysis of Carbon Nanotubes

The procedure applied for the qualification of CN production is described and discussed [1][2][3].
By simple optical microscopy it is possible to perform a first characterisation of the obtained product, after the arc discharge application. The morphology of the surface and the colour of the electrode (cathode) offer a means to select the region where the concentration of CN is higher.
The region characterised by grey colour (fig 3.1) is characteristic of high CN concentration. More in detail (fig. 3.2) a crystalline aspect determines where the CN concentration is maximum (fig. 3.2 position **B** and **C**).

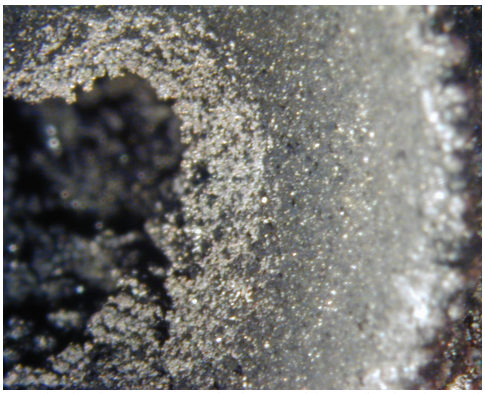
fig. 3.1 optical microscopy analysis, graphite cathode electrode after the arc discharge

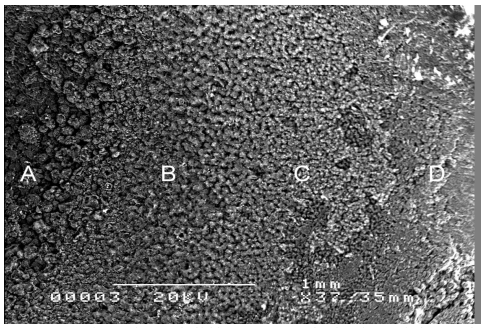

fig. 3.2 SEM imagine of different morphologies of the cathode

By means of the electronic microscopy (SEM), finally, it is possible to observe directly the formation of CN bundles and the efficiency of the adopted process methodology. It is possible also to have an indication of the grade of purity attained.
Figs. 3.3, 3.4 and 3.5 show SEM images of the regions **B**, **C** and **D**, respectively, marked on fig. 3.2. The presence of CN is clearly evidenced in figs. 3.3 and 3.4 by filament structures (regions **B** and **C**). The amorphous structures of fig. 3.4 (region **D**) evidence practical absence of CN.
The confirmation that the filament structures of figs. 3.3 and 3.4 can be referred to the formation of CN can be obtained only by TEM analysis (Transmission Electron Microscopy).
Literature data report that the preparation of CN samples for TEM observation needs not less than 3 hours.
The Authors developed a simple methodology for a rapid preliminary observation of CN, hereby described:

- positioning of a 100 mesh Ni grid on a filter containing CN powder
- mechanical deposition of powders by compression
- deposition of a C film (to increase the sample electrical conductivity).

Figs 3.6 and 3.7 show TEM images (200 KV) of carbon nanotubes, obtained using the described preparation.

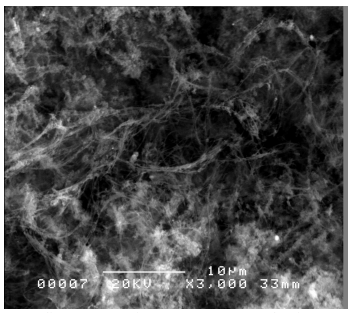

fig. 3.3 region **B,** SEM analysis

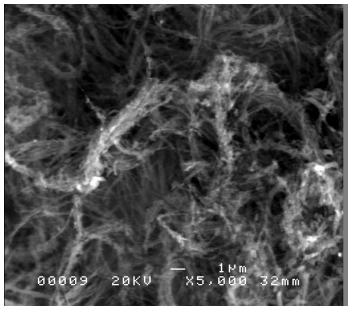

fig. 3.4 region **C,** SEM analysis

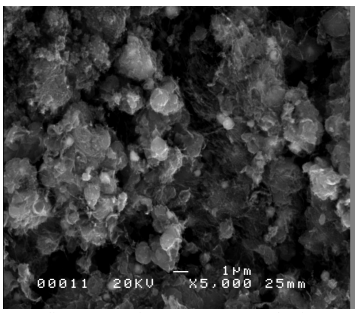

fig. 3.5 region **D,** SEM analysis

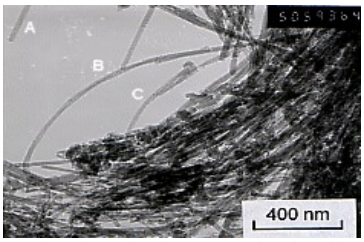

fig. 3.6 STEM imagine of carbon nanotubes bundles (50 000 x)

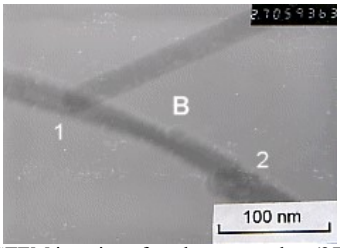

fig. 3.7 STEM imagine of carbon nanotubes (270 000 x)

Finally, EDS analysis can be utilised to evidence the presence of impurity in the final product, typically metals used as catalyst.
Fig. 3.8 shows EDS analysis of the cathode, after the arc discharge. The presence of the adopted catalysts (*Ni*, *Y*) is clearly evidenced.

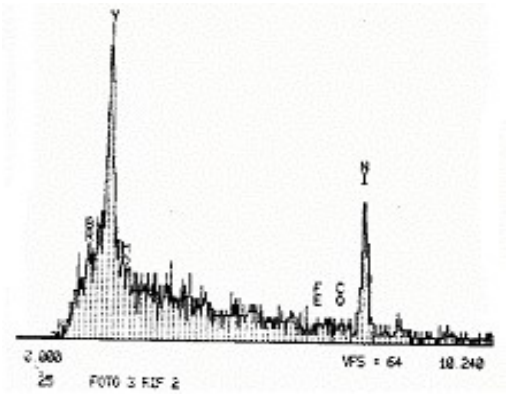
fig. 3.8 EDS analysis of the cathode electrode

4. Numerical and FEM analysis of the anisogrid lattice structures

The Anisogrid lattice structures (fig. 4.1) are characterized by helicoidal ribs, resistant to the applied load, and circumferential ribs, which ensure stability against local and/or global buckling [4]. With this new advanced structures it is possible to satisfy the followings requirements:

- static resistance
- dynamic stability (buckling behaviour)

in the minimum mass (**M**) condition as per Vasiliev Theory [4].

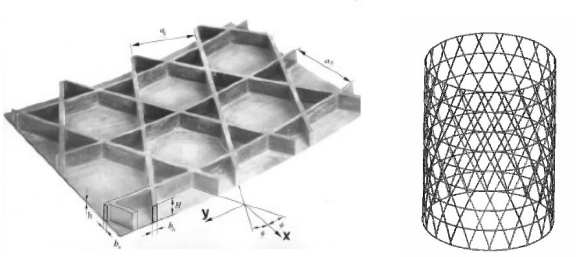
fig. 4.1 anisogrid lattice structure configuration

The procedure required to manufacture an anisogrid prototype is:

- fix radius **R**, height **H** of the anisogrid element; material employed (density, ultimate stress, Young modulus), applied load **P**
- determine the ribs dimension with Vasiliev model (in minimum mass condition)
- perform the FEM analysis
- verify the static resistance and the structure stability
- manufacture a prototype
- perform a mechanical test
- final verification of the mechanical behaviour.

To study the mechanical behaviour of Anisogrid Lattice Structures it is useful to develop a numerical program which determines the characteristic dimensions as a function of the initial parameters (**R**, **H**, **P**, **ribs number**, etc.).
Computer programs in MATLAB have been developed by the Authors, which can calculate the geometry of the anisogrid element, when fixed values of the dimensions of the structure are assumed: radius **R,** height **H** and the applied load **P**.
Moreover, a program has been developed, according to Vasilev theory, able to calculate the variation of the geometry of the anisogrid element, when radius **R** and height **H** of the structure are simultaneously varied (fig 4.2).

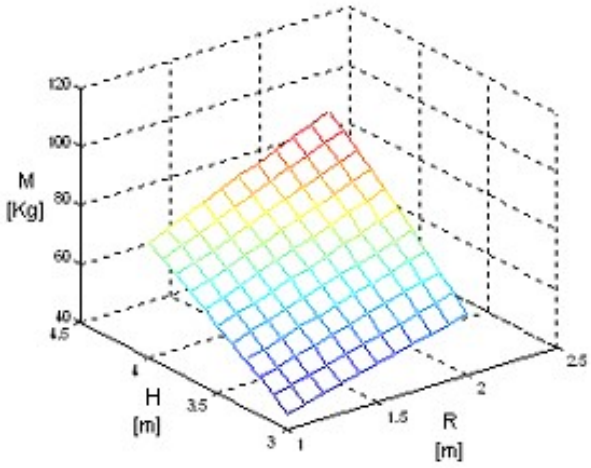

fig. 4.2 Structure Mass **M** vs Radius **R** and **L**ength **H**

The Vasiliev model [4] represents only a preliminary structure design. Employing the ribs dimension corresponding to the minimum mass condition, it is necessary to determine the loads and the constraints configuration that satisfy simultaneously the above requirements (static and stability behaviour). With the NASTRAN, the requested configuration has been determined and is reported below:

- The load is uniformly distributed on all the nodes of the FEM model
- The basement of the structure is constrained against translation
- All the remaining nodes of FEM model are constrained against rotation.

The load distribution (fig. 4.3) and the eigenvalue of 0.98 (fig. 4.4) demonstrate that the static resistance and the buckling stability of a design with minimum mass (Vasiliev model) is verified.

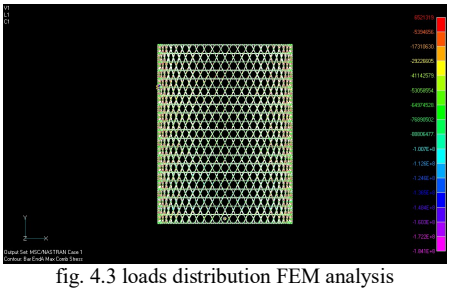
fig. 4.3 loads distribution FEM analysis

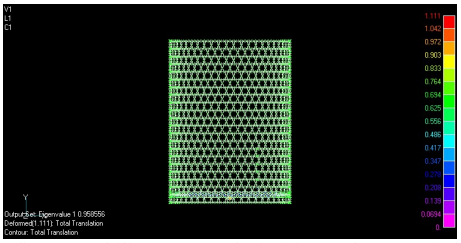

fig. 4.4 stability FEM analysis (eigenvalue = 0.98)

Besides, dynamic analysis are performed to study the structure vibration frequency (fig. 4.5).

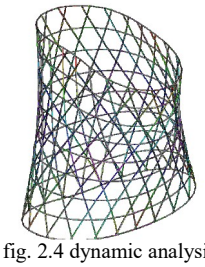

fig. 2.4 dynamic analysis

It's useful to evaluate the ribs dimension variation (as per Vasiliev model) using different materials: **a)** aluminium alloy Al 2024, **b)** composite epoxy resin carbon fibres reinforced (Hs/Ep), **c)** composite epoxy resin reinforced by carbon fibres and by a dispersion of carbon nanotubes, 5% by weight.
A numerical example is applied on a cylindrical anisogrid lattice structure (radius **R** = 1.5 m, height **H** = 4 m, applied load **P** = 3 MN, tab. 4.1).

MATERIAL	Al 2024	Hs/Ep	Hs/Ep + 5% CN
YOUNG'S MODULUS [Pa]	70E9	12E10	16E10
MASS [Kg]	206.3	84.1	69.7

tab.4.1 Structure Mass calculated for different materials

In this paragraph the studies that demonstrate the Vasiliev model [4] are illustrated. Using numerical and FEM analysis, the loads and constraints configurations have been determined that satisfy the structural requirements (static and of stability) in the minimum mass condition.

5. Study, design and manufacturing of composite reinforced with nanometric particles and of anisogrid lattice prototypes

In the design of carbon nanotubes reinforced composite the basically problem is to obtain a homogeneous distribution of the nanoparticles in the matrix [3]. The isotropic mechanical behaviour allows to improve the properties and the performances of an aerospace system manufactured with advanced composite materials. The nanometric chemical adhesion between the resin (matrix) and the carbon nanotubes determines the macroscopic characteristic of the composite element.
In the present work, composite samples are produced using the following basic elements:

- commercial epoxy resin
- curing agent: developed by Chemical Department of "La Sapienza" University – Rome
- nanometric graphite powder with carbon nanotubes addition
- graphite powder granulometry 20 µm.

Total concentration of dispersed powder realised was 10% and 20%. With different percentage of dispersed nanoparticles the morphologies of the material and the relative behaviour can vary drastically.
Samples dimensions are: 10x10x120 mm (fig. 5.1)

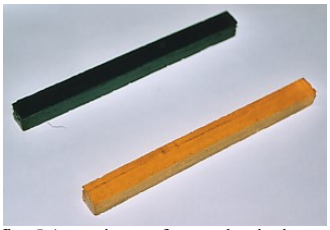

fig. 5.1 specimens for mechanical tests

Curing process adopted:

- room temperature x 24 hrs
- furnace curing 80 °C x 3 hrs.

Impact test where finally performed. Following considerations are driven:

1. the reduction of powders dimension increases the impact resistance properties
2. a good surface finishing improves the mechanical properties.

Fig. 5.2 shows the fracture surface morphology of a sample containing 20 % of powders. The pre-crack length is 2 mm. A brittle behaviour of crack propagation is evidenced.

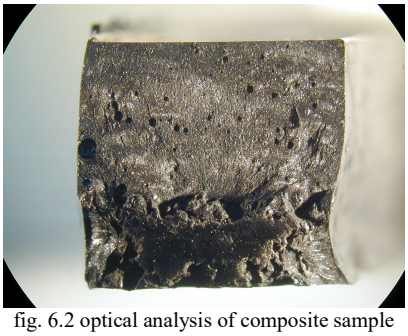

fig. 6.2 optical analysis of composite sample

To understand the fracture-mechanic behaviour of the composite, SEM characterisations of fracture surface were performed.
Fig. 5.3 shows the SEM images of the samples containing 10% of powders.
In the area **A** (crack initiation) and **B** (propagation) there is no presence of preferential directions of crack propagation.

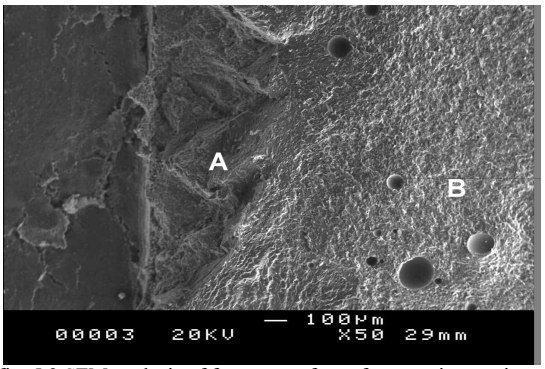

fig. 5.3 SEM analysis of fracture surface of composite specimen (10% powder addition)

On the contrary, the sample containing 20% powder, preferential directions of cracks propagation are observed (fig. 5.4, area **A**, **B**, **C** and **D**).
The presence of preferential direction is due to the non-uniformity of powders dispersion in the matrix and/or not completed curing.

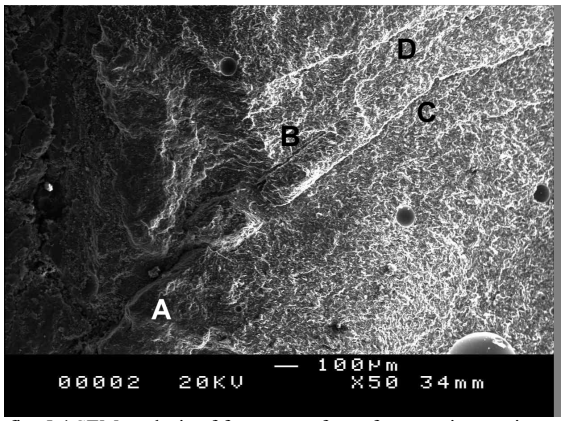

fig. 5.4 SEM analysis of fracture surface of composite specimen (20% powder addition)

A further observation is that the fracture lines change direction in correspondence of cavities (or voids).
In fig. 5.5 two fracture lines (**A** & **B**) are deviated by the presence of a void (see points **C** & **D**), and are stopped in point **E**.

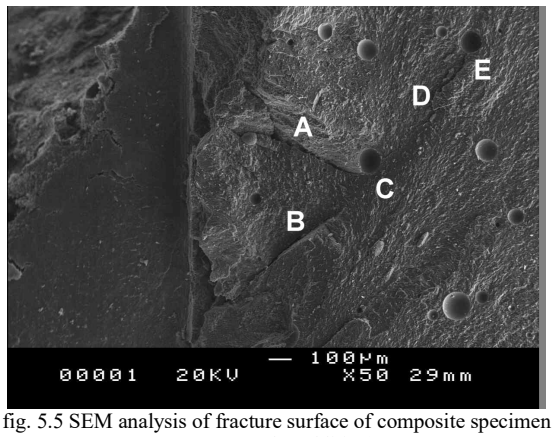

fig. 5.5 SEM analysis of fracture surface of composite specimen (20% powder addition)

To understand the mechanical variations that occurred when different powder quantities are employed, it is necessary to compare the impact test results with the traction experiments.
When different powder quantities are used, the fracture energy (impact test) remains practically the same, while in the traction test it is possible to observe a significant increment of the Young Modulus (*E*) (tab. 5.1).

MATERIAL	**RESIN EPOXY**	**RESIN EPOXY + GRAPHITE (10% WT)**
***E* [GPa]**	3.395	3.75
Δ*E*%	10.5%	

tab. 5.1 Young Modulus in function of different powder quantity (with CN) dispersed in a polymeric matrix

As shown in tab. 5.1 using a nanometric graphite particles (10% in wt) the Young's modulus improvements is 10.5 %. With a nanometric structured particles (only carbon nanotubes without impurity, catalysts, etc.) it is possible to obtain a major mechanical properties increase.

In fig. 5.6 a 5.7 are reported the graphics (σ-ε) of the traction tests in the case of matrix resin (without nanometric particles) and of the composite (epoxy resin + 10% wt of nanometric powder with CN).

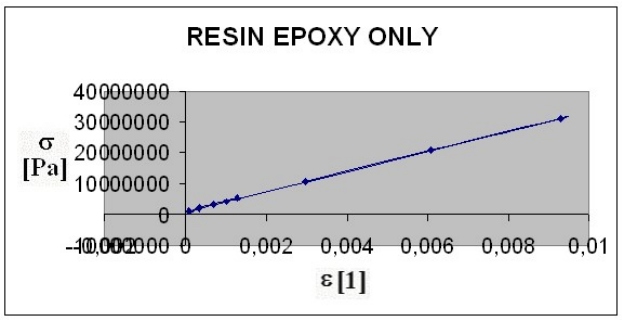

fig. 5.6 traction test graphic of epoxy resin

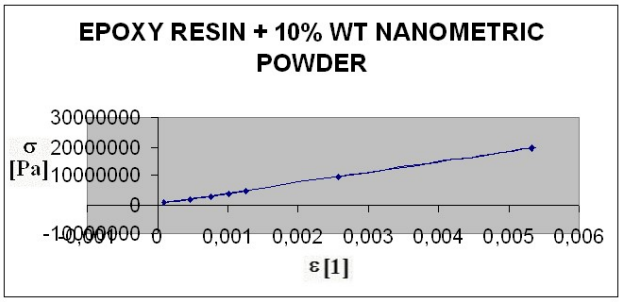

fig. 5.7 traction test graphic of composite (epoxy resin + nanometric powder)

After the preliminary mechanical test, a flat prototype of anisogrid lattice structure was realised, with the aim to understand process and practical procedures necessary to future realisation of full scale anisogrid elements and characterised by complex geometry, particularly cylindrical (as possible application for fuselages, launchers, satellites, rockets, etc.).
A preliminary design was calculated (see fig 5.8) to realise an under-scale prototype, dimensions 21x17 cm.
Successively the mould was prepared (fig. 5.9) by traditional mechanical tooling.
Following materials were utilised for the practical realisation of the composite prototype:

- epoxy resin
- curing agent (Triaethylentetramin)
- glass fibres (12000 per single filament)
- nanometric powder (graphite) + CN.

The following curing procedures were adopted:

- room temperature (21 °C) curing x 24 hours
- furnace curing at 80 °C x 3 hrs.

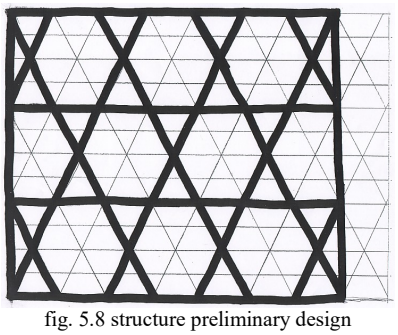
fig. 5.8 structure preliminary design

fig. 5.9 mould

fig. 5.10 flat anisogrid lattice prototype

Fig. 5.10 shows the produced prototype, after some preliminary and successful mechanical tests (vibration and tension tests).
More accurate and detailed investigations are necessary for a complete qualification of the prototype.

The present investigation is looking for procedures which can be used, possibly, during future industrial and automatic production. Therefore, a new CAD design was performed (see figs. 5.11 & 5.12), the positive simulacra of the lattice structure was realised by rapid prototyping, the final mould (negative) was obtained in silicon resin.

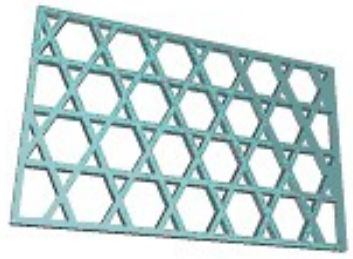

fig. 5.11 CAD design of the anisogrid lattice flat element

Finally, a 3D–CAD model was prepared to be used for rapid prototyping of a positive cylindrical geometry of an anisogrid lattice structure (fig. 5.14). Final silicon mould (negative) can be easily obtained.

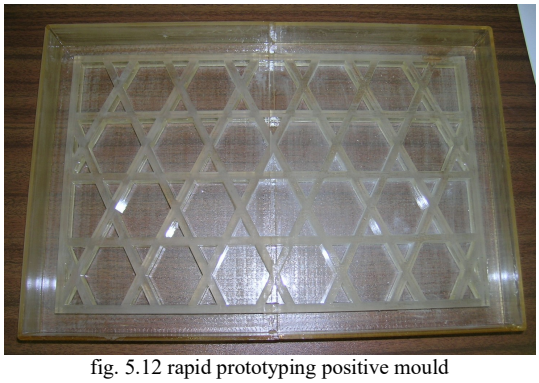

fig. 5.12 rapid prototyping positive mould

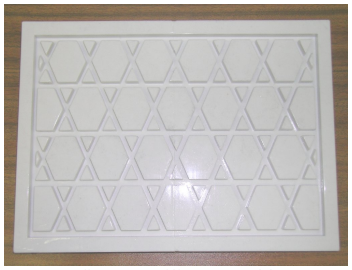

fig. 5.13 silicon mould

fig. 5.14 3D - CAD design of the anisogrid lattice cylindrical element and rapid prototyping positive mould

fig. 5.15 3D - CAD silicon mould during the solidification phase in the vacuum chamber

fig. 5.14 silicon mould

8. Conclusions

The utilisation of Vasiliev theory allows to design innovate structures, anisogrid lattice, satisfying the requirements of minimum mass in the global and local stability conditions. The load and constraint configuration that satisfy the stability conditions are determined by FEM analysis.
The addition of carbon nanotubes in a composite material (epoxy fibre reinforced polymers) improves the mechanical properties. In particular, the increase of the Young Modulus offers the possibility to further reduce the mass of the structure.
As final results, the combination of both the innovative design of anisogrid lattice structures and the utilisation of composite materials containing carbon nanotubes, allows to

obtain a strong overall mass reduction of the structural component, maintaining the stability requirements.

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