Development of Environmental Chamber for Testing of Composite Materials at Cryogenic Conditions

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ABSTRACT

Composite materials are widely used in different areas of science for many applications. They are being used prominently in aerospace industry. There is significant importance of testing of these composite materials, as they could be liable to failure due to temperature variation, which can range up to cryogenic temperatures and that must be sustain by material exposed to that condition. In this work, tensile strength of composite materials has been observed by using double walled vacuum chamber, which is designed for the Universal Testing Machine (UTM). A chamber with two walls, which are separated by vacuum in between, made up of material SS316. The chamber has been designed in such a way that it can accommodate a moving shaft, at the bottom part of the chamber that can apply a tensile load on the specimen whereas the upper shaft connected to the chamber has been fixed. The one end from the cryogenic chamber has been fixed to the jaws of the UTM and to another end, chuck is fitted, so that it can hold the specimen at the time of test. The chamber provides a cryogenic environment while testing of the specimen. Cryogenic temperature can be obtained in the chamber by the spraying of liquid nitrogen (LN2) into the chamber. To create an isolated environment, a vacuum pump having capacity 10^{-3} mbar has been used to create vacuum between the walls of the chamber. In the chamber for the collection of data pertaining to temperature, two digital thermocouples have fitted. A high-resolution camera incorporated inside the chamber which confirm us the condition of occurrence of fracture. Design of this cryogenic chamber has been done in the designing module of CATIA. Further, thermos mechanical analysis is done on the chamber to investigate the stresses developed in SS316 LN under cryogenic conditions. The testing of composite materials is an important aspect to be considered for the development of aerospace compatible materials. These materials experience cryogenic conditions which affect the mechanical and thermal properties. Further, there is a chance that the failure of these materials may occur at cryogenic conditions. Hence, in this work the testing of composite materials has been done in cryogenic conditions with the help of an environmental chamber.

Keywords: Cryogenics, Environmental chamber, Testing, cryogenic condition, Composite material**.**

1 Introduction

In the coming era new materials has been introduced for various applications such as space applications, building and bridges are introducing in industry with different mechanical thermo-mechanical and physical properties. These materials are generally composite materials. The important purpose for the use of composite materials in today's industry is ultra-light weight and high stiffness and high strength and its other thermomechanical and mechanical properties which are achievable as per required for the specific application. It's a challenging job to find out these thermomechanical and mechanical properties of composite materials, especially for the applications in space purpose because in space the temperature is very low i.e. up to cryogenic level and the probability for the failure of material is more. Hence for testing purpose of such materials we need to create an environmental chamber in which such conditions, similar to space conditions, can be attained and we can test those materials and find out their thermo-mechanical and physical properties. In this research work we will study that till date how these cryogenic conditions can be achieved and testing done and how we create a chamber with cryogenic conditions for testing of composite materials where we can find its thermomechanical and mechanical properties. The basics of some terminologies used in this are as follows:

1.1 Materials and composite materials

1.1.1 Material:

These are the purest form of solid matter or mixture of two different solid matters known as alloys exhibits some specified properties exists under some certain conditions. Materials having some specified crystalline & atomic structures which express their bonding strength between atoms to atom.

According to scientific definition, materials are non-living matter, either it is natural material or man-made material. It can be classified on the basis of physical, geological chemical properties and in some cases biological properties also. Materials are studied under material science.

In industries materials are being used for production and manufacturing of products. Generally it is used as raw that is not any process implemented on it but sometimes there are some processes implemented on materials before use.

Some types of materials are as follows:

- Biomaterial
- Composite materials made up of two or more materials having different physical properties.

1.1.2 Composite Materials:

Composite material composed of two or more constituents that are different in shape and in chemical properties/ composition and which are insoluble in each other. The new product material is stronger in strength, lighter in weight or less expensive in cost. The composite materials are made up of different layers known as laminates arranges in different patterns with different orientations. Some common applications of composite materials are in Aerospace industries, Sport's Goods Industries, Automotive Industries, Home Appliance Industries, Buildings and bridges, Bath tubs, storage tanks etc.

Contrived composite materials includes Concrete, Reinforced plastics such as Fiber-reinforced plastics, Ceramic composites.

Composite materials manufactured by fibres and matrix. Fibres are oriented with different orientations in matrix. Cross section of fibres is much less than matrix. Reinforcing phase materials are in form of fibers i.e. thread or in form of flakes.

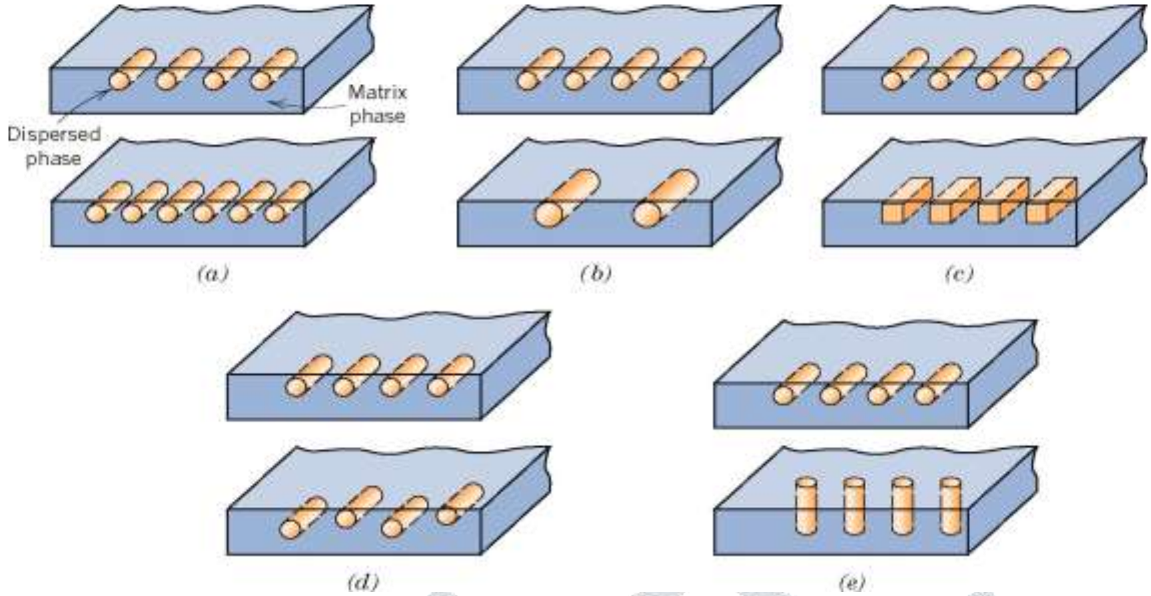


Figure 1: Different types of orientations of reinforcement fibers in matrix for composite materials.

1.2 Classification of composite materials:

Composite materials are classified according to the reinforced fibers used in matrix. Classifications of composite materials are as follows:

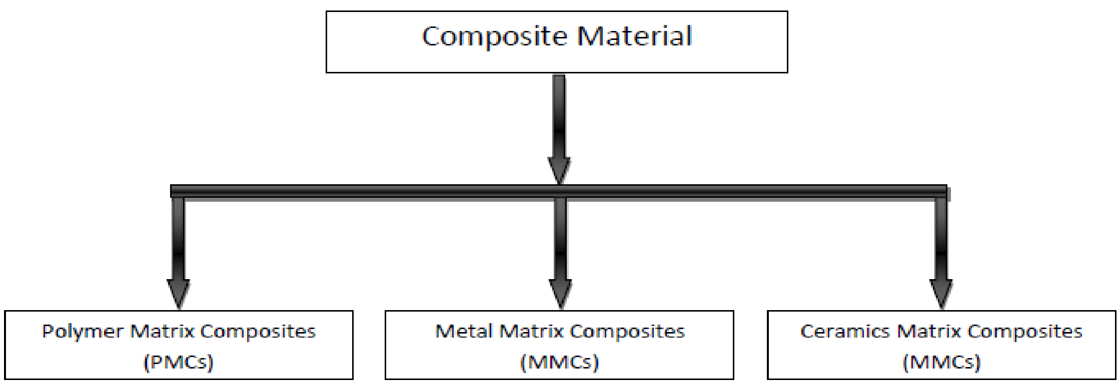


Figure 2: Different types of composite materials

1.2.1 Polymer Matrix Composites (PMCs)

Polymers of thin diameter are arranged in a particular design in matrix so as increase the strength of composite materials. Some drawbacks of PMC are Low operating temperatures, High coefficients of thermal and moisture expansion and Low elastic properties in certain directions. Some common examples of fiber are glass fiber, graphite fiber and kevlar fiber.

1.2.2 Metal Matrix Composites (MMCs)

In these composites metals are used as matrix. Some metals like aluminum and titanium are used as matrix and carbon and silicon carbide are used as fibers. With addition of fibers like silicon carbide, thermal conductivity and electric conductivity of metals can be reduced and elastic stiffness can be increased. Some advantages of MMCs are high elastic properties, high service temperature, moisture insensitivity, high electric and thermal conductivity, better wear resistance, good fatigue strength, and flaw resistances. The major disadvantage of MMCs is that it is less ductility and having less fracture toughness.

1.2.3 Ceramic Matrix Composites (CMCs)

In ceramic matrix composites, ceramics like alumina calcium alumina silicate is used and fibre of carbon/ silicon carbide has been used. The advantage of CMCs is that it has high strength and hardness.

1.3 Thermo-mechanical properties

Thermo-mechanical properties of a material is consists of Thermal Properties (Specific heat capacity, Thermal conductivity, Melting point, Latent heat), Thermo-elastic properties (Young's modulus, Poisson's ratio, Coefficient of thermal expansion, Elasticity), Plastic Properties (Plasticity, Yield stress, Creep, Recrystallization), Fracture properties (Ductile, Brittle, Shock viscosity, Tensile strength, Bending strength, Shearing strength, Fatigue strength).

The real behavior of materials under mechanical and thermal loading is described by a set of physical characteristics. It can be separated in four groups conditionally. The first group includes specific heat capacities, melting point and thermal conductivity which describes the behavior of materials, irrespective of their mechanical properties, under thermal loading conditions. Second group includes elastic modulus, viscosity, yield constants etc. that describes rheological behavior of materials under mechanical loading condition without its fracture. Third group includes properties such as coefficients of thermal expansion, density and activation energy which states thermo-mechanical behavior of materials. Forth group describes the fracture behavior of materials. The dependencies of all these characteristics from the temperature, describes thermo-mechanical properties of materials.

1.4 Cryogenics

Cryogenics is the branch of science which deals in or study the change in behavior in chemical or physical properties of materials at cryogenic temperatures. Generally not any solid point of temperature is proved for start of cryogenic temperature or cryogenic conditions. But scientist assumes to be cryogenics starts below 93.15K [1].

There are many methods such as heat conduction, Joule Thompson Effect that is cooling by rapid expansion and adiabatic demagnetization by which we can generate cryogenic environments with lowest temperatures. Out of these, the heat conduction and evaporative cooling are most commonly used processes. Similarly Joule Thompson Effect is commonly used in domestic and industrial refrigeration and air conditioning system. Adiabatic demagnetization process is mainly used for cryogenic applications which help in attaining absolute zero.

1.4.1 Heat conduction

Heat conduction is the process of transfer of heat from one solid matter to another solid matter when they are in contact with each other and also there should be a temperature difference between them. That is heat travels from higher temperature body to lower temperature body.

1.4.2 Evaporative cooling

Evaporative cooling is the process when the liquid fluid carries the heat in vapour form and transfer the heat from one medium to another fluid flowing in another medium through convection process. HVAC industries follow this process.

1.4.3 Joule Thompson Effect

The Joule–Thomson effect is also called as Joule–Kelvin effect or Kelvin–Joule effect. Joule Thomson expansion is the change in temperature of a gas or liquid when it is passes through a valve or porous plug with insulation with environment so that there is no heat exchanged with surroundings [2].

2 Literature review

Till date the testing methodology used for testing the composite materials for space applications is initially treat it with refrigerants (liquid nitrogen, ammonia etc.) then it tested under normal atmospheric conditions on testing machines. But when it place on testing machine in normal atmospheric conditions its properties might have been changed which could affect the properties of composite materials. Hence to eliminate this affect the same environmental conditions are required during the testing also. Therefore it is essential to develop a chamber which can provide the same conditions in which composite material is treated so that its properties may not get changed and further more we can analyze the thermomechanical and mechanical properties of composite materials under cryogenic conditions.

Weiwei Wu et al done experiment uses the Si_3N_4 ceramics with SiC whisker to prepare a composite material by taking Si_3N_4 a powder with sintering additives (4 wt.% Al_2O_3 and 4 wt.% Y2O3) for 10 h in a polyethylene bottle using Si3N4 balls and ethanol as the grinding media and prepared powders were hot-pressed at 2073 K, 30 MPa. and concluded that fracture toughness (at 77 K 8.79±0.64 MPa $m^{1/2}$), hardness, flexural strength of Si3N4-SiCw composite increases with decrease in temperature. The fracture toughness is approximately 41% is higher than at 293 K [3].

Sai et al performs an experiment in which they used the Y_2O_3 powder with AIN powder for making composite material. 5 wt% Y_2O_3 powder is mixed with AIN powder and pressed by cold isostatic pressing process and found that as temperature decreases from 293k to 77k the flexural strength increases.

Sukjoo Choi et al performs an analytical and Finite Element analysis of graphite/epoxy composite material for cryogenic storage for space vehicles that carries liquid hydrogen (LH_2) because generally composite material has high specific stiffness and strength as compared to pure materials. As cryogenic tanks have to face much variation in temperatures during and after the re-entry in earth's atmosphere. They have initially immersed specimen in Liquid nitrogen for approximately 5 min due to which the composite material acquires the cryogenic temp and its thermo-mechanical properties, mechanical properties attains the level of cryogenic conditions and then load is applied and analyzed the change in properties.

Md S. Islam et al manufacture the composite material specimen with carbon and Kevlar fiber and metal as matrix at room temperature and thus cryogenic treatment is applied by submerging it in liquid nitrogen for 6 hours and then placed at room temperature. Now tensile and flexural load were applied to specimen at room temperature and thermomechanical properties and mechanical properties were analyzed while three point bending test and found that when specimen were submerged in liquid nitrogen its mechanical properties may get affected. Then failure specimens were again exposed to cryogenic conditions and analyzed with SEM in which researcher observe that no more cracks arises [4].

Weiwei Wu et al prepare a composite material and carried out a test for fracture toughness during cryogenic treatment by keeping specimen in liquid refrigerant, and found that its properties have not changed [5].

Takefumi Horiuchi et al study about the required properties of composite materials for cryogenic applications and found that Carbon Fiber Reinforced Polymer (CFRP) is ideal for cryogenic application with high carbon contents because of its good ratio of strength to thermal conductivity below 77K [6].

Hei-lam Ma et al have done their studies on mechanical behavior of Glass fiber/epoxy composites in woven form. They prepared 18 samples of composite material, which were conditioned at three different temperatures i.e. room temperature (295K), dry ice temperature (199K) and liquid nitrogen temperature (199K) for 15 minutes, and then impact test was performed on impact tester INSTRON Dynatup 9250NV and observe with the result that with decrease in temperature, depth of damage of composite material decreases [7]. **Y. Shindo et al** worked on tensile and damage behavior of a plain wave glass/ epoxy composite at cryogenic temperature. The specimen was tested at Room Temperature, 77K, 20K, 4K. The test was performed on a servo hydraulic testing machine. Load frame, extensometer system and specimen is encased in a Dewar of Liquid Nitrogen, Helium gas and Liquid Helium to attain the temperature 77K, 20K, 4K respectively. For recording of stress strain behavior sensor were mounted on specimen for Room temperature testing and for liquid nitrogen testing sensors were placed at the end of stainless steel waveguides, outside the Dewar of liquid nitrogen. At room temperature tensile stress as reaches its maximum value the specimen gets failed immoderately. At 77K specimen shows the knee point in stress strain measurement. Following table shows the stress strain results of samples at Room temperature, 77K, 20K, and 4K. [8].

Tensile test results

	L (mm)	E_w (GPa)	ν_w	σ_{ult} (MPa)	σ_w^k (MPa)	ε_w^k (MPa)
RT	250	25.3	0.18	273	–	–
	200	26.4	0.17	294	–	–
		24.8	0.19	278	–	–
77 K	250	30.4	0.23	653	259	0.93
		30.5	–	611	258	0.94
	200	31.6	0.23	556	264	0.91
		30.9	0.22	535	261	0.93
20 K	200	32.5	0.23	550	255	0.82
4 K	250	31.2	0.22	593	240	0.83
	200	32.9	0.24	565	256	0.83
		33.1	0.25	541	257	0.83
		32.3	0.24	557	235	0.76

Table 2: Results of Tensile Test at Different temperatures. [9]

G. Z. Ma et al performs an experiment on Cu-Zr-Al metallic glass composite by immersing it in liquid nitrogen for 7 hours. They investigate the microstructure of composite material with XRD (X-ray diffraction) and SEM (scanning electron microscope) and conclude that due to cryogenic treatment of Cu_{46} Zr_{46} Al_8 composite material for 72 hours, micro-hardness increases up to 18.55% and similarly ultimate compression fracture increases up to 37.5%. [10]. **Takeda et al** performs an experiment in which they study the glass fiber reinforced polymer composite material specimens behavior at room temperature and after cryo-treatment of specimen at 77K. Also they choose to cross verify their result with finite element method to determine the stress strain generated in specimen and found that its strength improved after cryogenic treatment [11]. **Xu et al** study the behaviour of Kevlar 129 at cryogenic conditions. For testing they treat the specimen of kevlar 129 with cryogenic liquid and perform the testing at room temperature and found one drawback that as test were performed at room temperature due to which the desired results may get effected because specimen tries to attain the temperature from its surrounding of room temperature due to the heat transfer law. [12]

Zhyang et al performs an experiment on carbon fiber T300 by two different methods i.e.by Temperature programmed controlled method (TPCM) and Quenching method. In TPCM specimen was kept in temperature programmable controlled chamber for 12 hours in which temperature was cooled to cryogenic temperature (77K) at the rate of 2°C/min. and specimen gradually attains the cryogenic properties. Then for testing specimen take out from chamber and load was applied on it and its physical and mechanical properties were measure. In quenching method, specimen was immediately submerged in liquid nitrogen for 12 hours and then load was applied on specimen at room temperature to analyze the physical and mechanical properties. [13].

3 Design and Fabrication of Environmental Chamber

Drawings of environmental chamber in 2D & 3D. Dimensions are taken from UTM present in laboratory in university. Then material SS316 is arranged for the fabrication of chamber with following composition & properties:

Composition: Fe/< .03C/16-18.5 Cr/10-14 Ni/2-3 Mo/<2 Mn/<1 Si/< .045 P/< .03S

Property	Minimum Value (S.I.)	Maximum Value (S.I.)	Units (S.I.)
Density	7.87	8.07	Mg/m^3
Bulk Modulus	134	152	GPa
Compressive Strength	170	310	MPa
Ductility	0.3	0.51	N/A
Elastic Limit	170	30	MPa

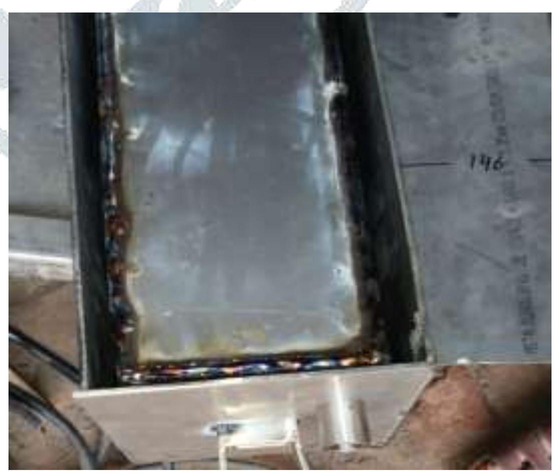
Endurance Limit	256	307	MPa
Fracture Toughness	112	278	$MPa.m^{1/2}$
Hardness	1700	2200	MPa
Modulus of Rupture	170	310	MPa
Poisson's Ratio	0.265	0.275	N/A
Shear Modulus	74	82	GPa
Tensile Strength	480	620	MPa
Young's Modulus	190	205	GPa
Specific Heat	490	530	J/Kg.K
Thermal Conductivity	13	17	W/m.K

Table 1: Properties of SS316 [14]

Fabricate rectangular chamber of 2 no's with different dimensions and different height measurements. Weld the top and bottom plates having the holes for holding the specimen into the jaws of UTM and make the necessary holes into the chamber for the attachment of pressure control and measurement gauges and vacuum pump inlet valve. Attach the valves (pressure measurement gauges and control valves etc.). Now insulate the smaller box with insulation and wrap it in aluminium foil. Place the insulated chamber concentrically inside the chamber with bigger diamensions. Weld the top & bottom plates of bigger chamber. A compressor used to create vacuum inside the chamber. Place the specimen inside the chamber and place the chamber between the jaws of the UTM. Now supply the liquid nitrogen to chamber through one way inlet valve. Apply the load and analyse the results. The step by step formation of chamber illustrated with the help of pictures below.

When composite material is cryogenically tested in an environmental chamber then we come to know that either composite material specimen used for testing exhibits the desired thermomechanical properties, mechanical properties and physical properties or not which are required for the space applications.

Formation of inner chamber



Formation of outer chamber with air gap

Assembly of One way valve

Finished Product

Vacuum has done between two layers of chamber

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