Development and Thermomechanical Analysis of Environmental Chamber for Testing of Composite Materials at Cryogenic Conditions

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MECHANICAL ENGINEERING

by

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DEPARTMENT OF MECHANICAL ENGINEERING

LOVELY PROFESSIONAL UNIVERSITY

PUNJAB

(July 2017)

CERTIFICATE

I hereby certify that the work being presented in the dissertation entitled "**Development and Thermomechanical Analysis of Environmental Chamber for Testing of Composite Materials at Cryogenic Conditions**" in partial fulfillment of the requirement of the award of the Degree of master of technology and submitted to the Department of Mechanical Engineering of Lovely Professional University, Phagwara, is an authentic record of my own work carried out under the supervision of Mr. Vishnu Saini, Assistant Professor Department of Mechanical Engineering, Lovely Professional University. The matter embodied in this dissertation has not been submitted in part or full to any other University or Institute for the award of any degree.

25-07-2017 Rajesh Kumar

Regd no.: 41400127

This is to certify that the above statement made by the candidate is correct to the best of my knowledge.

25-07-2017 Asst. Prof. Vishnu Saini

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The external viva-voce examination of the student was held on successfully

Signature of Examiner

Declaration

I, **Rajesh Kumar**, student of Master **of Technology (Part Time)** in **Mechanical Engineering** under the school of Mechanical Engineering of **Lovely Professional University, Punjab**, and Hereby declare that all the information furnished in this dissertation report is based on my own intensive research and is genuine. This dissertation does to the best of my knowledge contain part of my work which has been submitted for the award of my degree either of this university without proper citation.

Rajesh Kumar
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Date: 25-07-2017

Acknowledgment

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Last but not least, I express my sincere gratitude to my parents who have always support me throughout my studies at university and encouraged me with their best wishes.

Rajesh Kumar

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ABSTRACT

Composite materials are being used extensively in different realms of science for various applications. Their use is prominent in the aerospace industry. The testing of these composite materials are of significant importance, as they might be susceptible to failure due to the variation in the temperature ranges and the cryogenic temperatures which the material have to endure. In the present work, the tensile strength of composite materials are observed using double walled vacuum chamber which is designed for the Universal Testing Machine (UTM). The chamber with two walls, each separated by vacuum, is made of SS316.The chamber is designed in such a way that, so as to accommodate a moving shaft on the bottom part which applies tensile load on the specimen while the upper shaft connected to the chamber is fixed. One end of both shafts from the cryogenic chamber is fixed to the jaws of the UTM and has a chuck fitted on the other end to hold the specimen during the test. The chamber acts in such a way so as to provide a cryogenic environment for the testing of the materials. The cryogenic temperature in the chamber is obtained by the use of liquid nitrogen (LN2) which is sprayed into the chamber. A vacuum pump of capacity 10^{-3} mbar is used to create vacuum between the walls of the chamber, to create an isolated environment. Two digital thermocouples are fitted in the chamber to collect data pertaining to temperature. The high resolution camera incorporated inside the chamber gives an insight on how and when the fracture occurs. The design of the cryogenic chamber is done in the designing module 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 the present work, the composite materials are tested under cryogenic conditions in the environmental chamber.

1 Introduction

In the coming era new materials, composite materials for various applications such as space applications, building and bridges are introducing in industry with different mechanical thermo mechanical and physical properties. 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. To find out these thermomechanical and mechanical properties of composite materials is itself a challenging job specially for the applications in space purpose because in space the temperature is very low at cryogenic level and the chances of material failure is more. So for testing of these types of composite materials we need to create such type of environmental chamber with cryogenic conditions similar to space conditions where 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. Before further proceed I would like to explain some terms which 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 any non-living matter, either it is natural or man-made. They are given a classification based on the different properties defined as physical, chemical, geological, and in some cases, biological. Materials are studied under material science.

In industrial definitions materials are used for production and manufacturing processes. 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:

A Composite material is a material system composed of two or more constituents that are different in shape and 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:-

- Aerospace industry
- Sporting Goods Industry
- Automotive Industry
- Home Appliance Industry
- Buildings and bridges
- Bath tubs, storage tanks etc.

Engineered composite materials include:

- 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. The reinforcing phase materials are in the form of fibers (thread) or in the form of flakes.

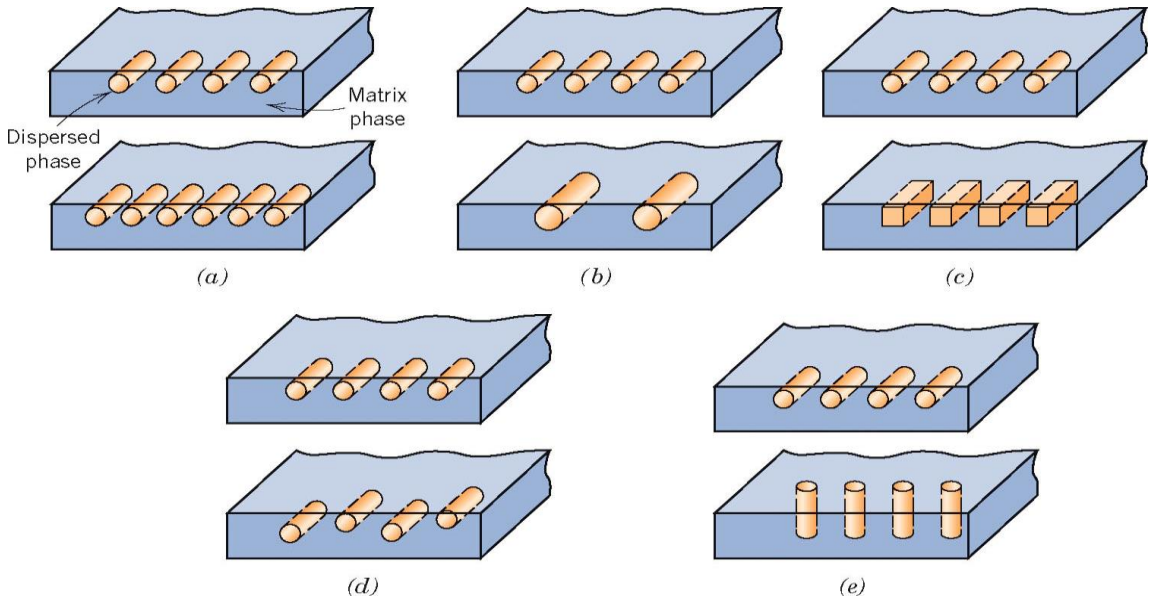

Figure 1 Different types of orientations of reinforcement fibers in matrix for composite materials.

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:

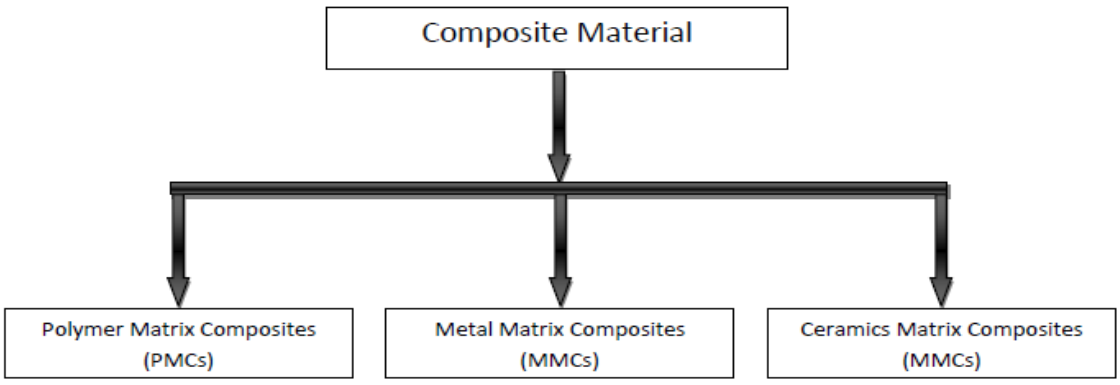

Figure 2 Different types of composite materials

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.

The drawbacks of PMC are:

a) Low operating temperatures
b) High coefficients of thermal and moisture expansion
c) Low elastic properties in certain directions

Common fiber examples are:

a) Glass
b) Graphite
c) Kevlar

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 conductivities of metals can be reduced and elastic stiffness of metal can be increased. Advantages of MMCs are as:

a) Higher elastic properties
b) Higher service temperature
c) Insensitivity to moisture
d) Higher electric and thermal conductivities
e) Better wear
f) Fatigue
g) 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)

Ceramic matrix is used in these composites. In these composites ceramics like alumina calcium alumina silicate is used and fibre is of carbon/silicon carbide used. The advantages of CMCs are that it has high strength and hardness.

1.3 Thermo-mechanical properties

Following figure shows the thoughness test and creep testings in different composite mterials like ceramic metal composite, polymer metal composite and metal metal composite and observed that while increasing load during test how specimen behaves.

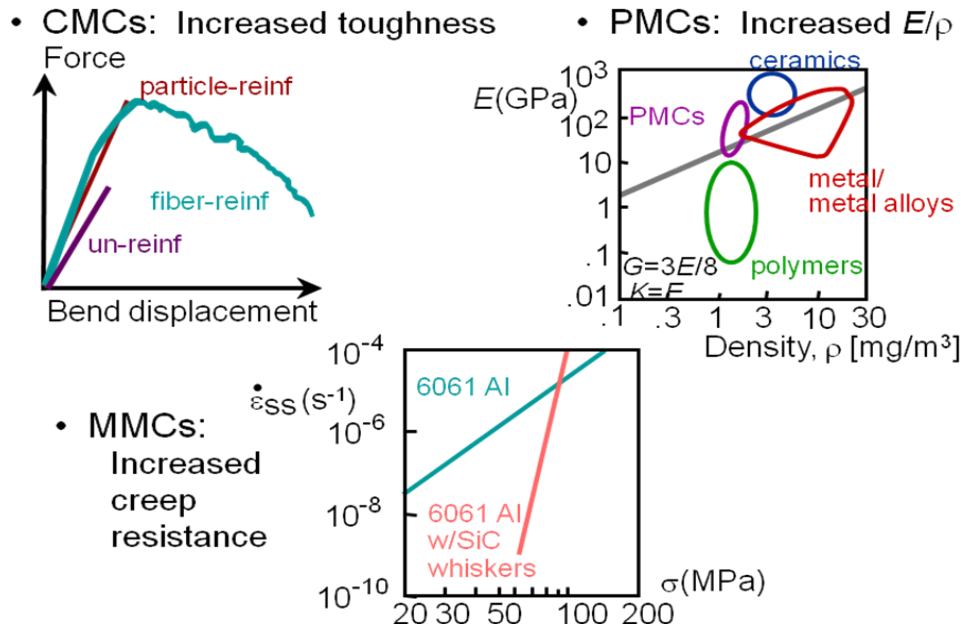

Figure 3 Creep rupture of a silicon-carbide reinforced aluminum composite [1]

2 Terminology

2.1 Classification of composite materials:

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

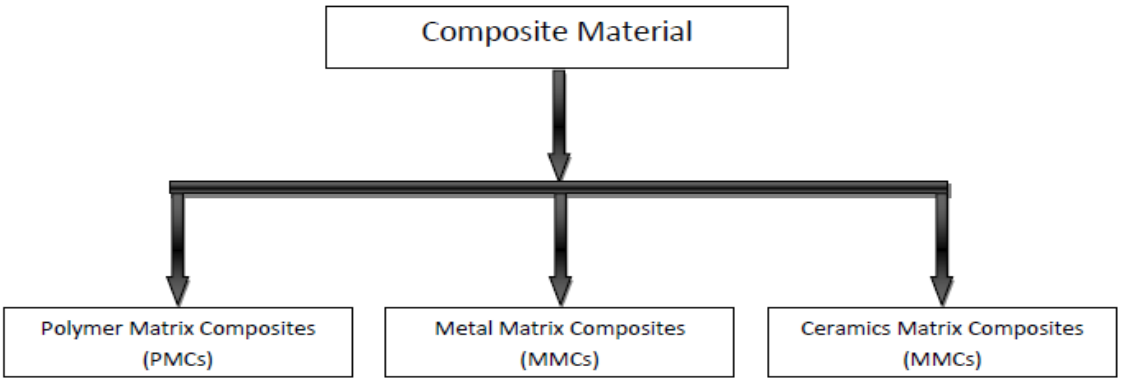

Figure 4 Different types of composite materials

2.2 Polymer Matrix Composites (PMCs)

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2.5 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 [2].

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. Heat conduction and evaporative cooling are commonly used processes. Similarly Joule Thompson Effect is commonly used in domestic and industrial refrigeration and air conditioning systems. The Adiabatic Demagnetization process is mainly used for cryogenic applications which help in attaining absolute zero.

2.5.1 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.

2.5.2 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.

2.5.3 Joule Thompson Effect

The **Joule–Thomson effect**, also known as the **Joule–Kelvin effect**, **Kelvin–Joule effect**, or **Joule–Thomson expansion**, is the temperature change of a gas or liquid when it is forced through a valve or porous plug while kept insulated so that no heat is exchanged with the environment [3]. This procedure is called a throttling process or Joule–Thomson process.

3 Literature review

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.

Then author performs the Vickers hardness (HV) test on the polished surface at 293 K, 195 K and 77 K with a load of 98 N for 15 s. Three-point bending method is used for Flexural strength(σ) test on the specimens of 4 mm × 3 mm × 36 mm, using a span of 30 mm and a crosshead speed of 0.5 mm/min. Specimens were & ground polished with diamond slurries down to 1 μm finish, and the edges should be chamfered to minimize the effect of stress concentration test bars of 2 mm × 4 mm × 22 mm size is used for fracture toughness evaluation by a single-edge notched beam test with a span of 16 mm. Before cryogenic tests, specimens were kept in the refrigerants.

Raman spectroscopic apparatus is used to observe the RAMAN SPECTRA. X-ray diffraction scanning electron microscopy coupled with Energy Dispersive Spectroscopy (EDS), Transmission Electron Microscopy (TEM) testing techniques were used to measure the microscopic level changes in grain structure.

In results it was observed that at 77 K (cryogenic temperature) there is stronger resistance to crack propagation which is due to higher residual stresses at room temperature which may contribute to fracture toughness.

In conclusion it is observed 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 approx 41% is higher than at 293 K. this can be possible by Sic whisker at cryogenic temperature [4].

Weiwei Wu et al done experiment uses the Al_2O_3 ceramics with SiC whisker to prepare a composite material by taking Al_2O_3 as powder. By Archimedes method was used to measure the density of the composites. Then performs the Vickers hardness (HV) test on the polished surface at 293 K, 195 K and 77 K with a load of 98 N for 15 sec. Three-point bending

method is used for Flexural strength (σ) test on the specimens of 4 mm × 3 mm × 36 mm, using a span of 30 mm and a crosshead speed of 0.5 mm/min. Specimens were & ground polished with diamond slurries down to 1μm finish, and the edges should be chamfered to minimize the effect of stress concentration test bars of 4 mm × 3 mm × 36 mm size is used for fracture toughness evaluation by a single-edge notched beam test with a span of 30 mm. Before cryogenic tests, specimens were kept in the refrigerants.

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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. The resulting material was cut into the pieces having dimensions 3mm X 4 mm X 36 mm. These specimens were tested at 293k, 195k & 77k by universal testing machine respectively. Weibull statistical analysis used to investigate the flexural strength. The equation used for investigation is as follows:

$\ln \ln [1/ (1-Pn)] = m \ln \sigma - m \ln \sigma_o$

& the failure probability estimator is:

Pn = 9n-0.5)/N

Where n is ranking

N is no. of tested specimens

M is Weibull modulus

σ is the flexural strength of n^{th} bar & σ_o is the characteristic flexural strength.

In situ X-Ray diffraction was carried out at different temperatures such as 293k, 195k & 77k. Similarly Vickers hardness testing is carried out at polished surface of specimens at 293k, 195k & 77k under load of 10 Kg in refrigerants.

As a result as temperature decreases from 293k to 77k the flexural strength increases. The Weibull modulus is also noted as:

At 293k is 15.13 and at 18.09 at 77k i.e. Weibull modulus increases with decrease in temperature. Flexural strength also increases from 346.6±29.2 MPa to 415.21±21.7 MPa as temperature decreases from 293k to 77k.

As a result lattice constants decreases and there exists the stronger bonds and increase in elastic modulus at 77k than 293k. Also the Viker indentation crack length reduced from 538.7±33.3μm at 293k to 412.9±26.5μm at 77k which shows the increase of fracture surface energy and elastic Modulus at cryogenic field [6].

S Watanabe et al study the behavior of CFRP behavior for use in propellant tanks of Reusable Launch Vehicles (RLVs) to reduce the gross lift weight. Due to cryogenic temperatures many thermal contraction gives rise to residual stresses in composite material and when mechanical load is applied then micro cracks may get start to exists just before ultimate strength of specimen, due to which fuel get leaks which is very much dangerous.

The behavior of CFRP will be analyze by finite element method similarly young's modulus and stress distribution are calculated numerically.

Researcher uses the Mathematical Analyses & ANSYS software for analyses. Considering cryogenic temperature 20K. The geometry of satin weave fabric laminate is $l=4h/3 \tan \theta$ and $d = l/8$.

Where $2h= 0.35$mm and $l= 1.48$mm,

And weave angle $\theta= \arctan (4h/3l)$ i.e. $\theta = 90$ deg.

It is studied that young's modulus is affected by cracks in surface layer except for two layer woven laminates & tensile stresses at crack tips [7].

Sukjoo Choi et al performs an analytical and Finite Element analysis of graphite/epoxy composite material for cryogenic storage for space vehicles to carry the liquid hydrogen (LH_2) because generally composite material has high specific stiffness and strength as compared to pure materials.

Because cryogenic tanks have to face much variation in temperatures during and and after the re-entry in earth's atmosphere. The scientist has performed the analysis in two ways to cross check the results i.e. analytically and by Finite Element Analysis. When prototypes were tested then it comes to know that at cryogenic conditions then micro cracks were introduced in transverse layers of laminations due to which de-lamination get starts due to thermal contraction starts in between the fiber and matrix phases of composite materials. This delamination provides the path to cryogenic fuel or cryogen to leak out which is very dangerous.

When specimen was tested under cryogenic conditions then specimen was initially immersed 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 while the composite material immersed in LN_2 load is applied to specimen and analyzed the change in properties.

The software used in this experiment was MATLAB. [8]

Md S. Islam et al performs an experiment in which they study the behavior of composite material consisting of carbon and Kevlar as fiber along with material for the preparation of cryogenic tanks which will carry the cryogenic fuel like Liquid oxygen (-183°C) liquid methane (-161°C) or liquid hydrogen (-252°C) etc.. These types of tanks are properly insulated with insulators to external & ambient temperature due to which there is temperature difference arises between the inside and outside of the tank know as temperature gradient. Because of this temperature gradient differential expansion and contraction occurs in the tank walls which will cause the failure of tank structure and this failure results in leakage of cryogenic fuel which is very dangerous for space vehicles.

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. For cryogenic treatment specimen is submerged in liquid nitrogen (LN_2) at -196°C temperature. Specimens were dipped in liquid nitrogen for 6 hours. Then specimens were removed from liquid nitrogen and placed in room temperature. After removal from liquid nitrogen tensile and flexural load were applied to specimen at room temperature and thermomechanical properties and mechanical properties were analyzed while three point bending test. As a

result it is noted 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 [9] **Weiwei Wu et al** prepare a composite material from GNPs/Al2O3 in distilled water. Then after number of process specimens were prepared with polished surfaces where the testing will be carried out. Dimensions of prepared specimen are 3 mm X 4 mm X 22 mm. Density of specimen is calculated by Archimedes principle and Vickers hardness is identified from Vickers indentation with load of 9.8 N for 15 sec at temperature 293 K and 77 K.

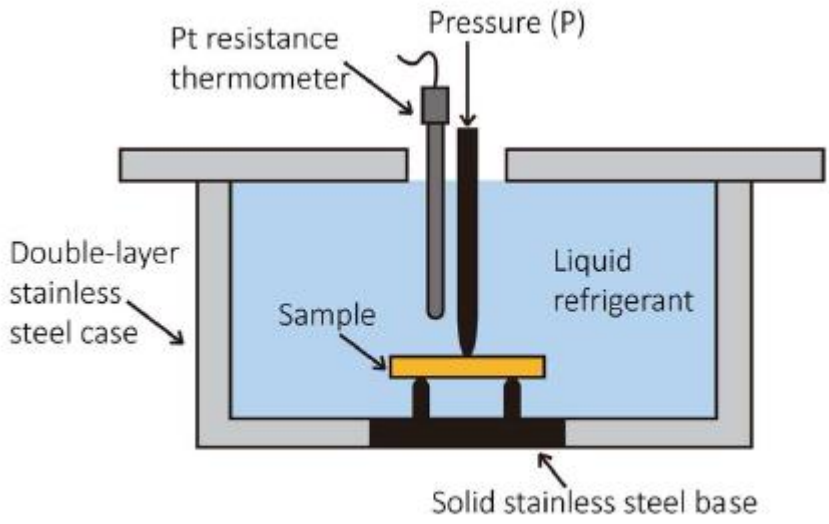

Figure 4 schematic diagram of testing on composite material in refrigerant.

Now fracture toughness test is carried out. For cryogenic testing specimen is keep in liquid refrigerant and tested over there as shown in figure 4 so that its properties cannot be changed [10].

Takefumi Horiuchi et al study about the required properties of composite materials for cryogenic applications. During their stud they found that properties of FRP material can be controlled by desired combination of fiber and matrix , also they possesses the high specific strength and having low thermal conductivity so they are considered good for cryogenic applications. The main properties required for cryogenic applications are as follows:

1. Heat inlet to support should be small

2. The total load of support should be the sum of gravity of cold mass and load due to thermal contraction.

CFRP is ideal for cryogenic application with high carbon contents because of its good ratio of strength to thermal conductivity below 77K. The successful example of this is CFRP used in cold transportable NMR cryostat [11].

Hei-lam Ma et al has done the study the mechanical behavior of Glass fiber/epoxy composites in woven form. They prepare the 18 samples of composite material for impact test. The samples were conditioned at three different temperatures at 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. This test was having result with decrease in temperature depth of damage of composite material decreases [12].

Y. Shindo et al study the tensile and damage behavior of plain wave glass/epoxy composite at cryogenic temperature. For this they adopt ASTM D3039 method for testing, in which specimen is of dog bone shaped having dimensions length 250mm width 25mm.

The specimen was tested at Room Temperature, 77K, 20K, 4K. The test was performed on a servo hydraulic testing machine which is adapted for cryogenic service. Load frame, specimen and extensometer system is ecased 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 wavegides, 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. [13].

Table 1 Results of Tensile Test at Different temperatures. [14]

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

G. Z. Ma et al performs an experiment on Cu-Zr-Al bulk metallic glass composite for introduction of martensitic transformation o cryogenic treatment (CT). They prepared the specimens in cylindrical rod shaped having dimensions 4mm diameter and 50mm in length. Then for cryogenic treatment specimen were immersed in liquid nitrogen for 7 hours. Then microstructure of composite material is investigated with X-ray diffraction (XRD) and scanning electron machine (SEM). To check the difference between microstructure of as-casted composite material and cryogenic treated composite material 5-5 specimens of both as-cast and CT composite material is investigated under XRD and SEM. Then for mechanical properties specimens of both as-cast composite material and CT composite material were tested under Vickers microhardness test and uniaxial compression test, which shows the result that after CT of composite material there is a change in microstructure of composite material. During their experiment they conclude that due to CT of Cu_{46} Zr_{46} Al_8 composite material for 72 hours microhardness increases upto 18.55% and similarly ultimate compression fracture increases upto 37.5%. [15]

Takeda et al performs an experiment in which they study the glass fiber reinforced polymer composite material specimens behaviour at room temperature as well as cryogenic temperature (77K) during testing. For room temperature testing load was directly applied to specimen without any temperature pre-treatment. Similarly for cryogenic temperature testing specmen was prior kept in liquid nitrogen for few hours and then take it out from liquid nitrogen and load was applied in room teperature after immediately take it out from the liquid

nitrogen. Another method they choose to cross verify their result is they perform the same analysis through finite element method to determine the stress strain generated in specimen and to know its strength improvement during cryogenic treatment. [16]

Xu et al study the behaviour of Kevlar 129 at cryogenic conditions. For testing at these cryogenic conditions they treat the specimen of kevlar 129 with cryogenic treatment in two ways which are as follows:

1. They kept the specimen in programmable controlled chamber for few hours in which specimen was cooled from RT to cyogenic temperature (77K) gradually at the rate of 2° C per minute. After this treatmen specimen take out from programmable controlled chamber and load was applied on this specimen for testing so that its stess stain results and its strength changes can be find out.

2. For another specimen the dip the specimen into liquid nitrogen for cryogenic treatment upto 12 hours so that specimen can attain the cryogenic properties and then specimen take out from liquid nitrogen and testing was performed on this.

In both testing technique theie is one draw back that both test were performed in room temperature due to which somewhere how the desired results may get effectedbecause specimen tries to attain the temperature from its surrounding of room teperature due to the heat transfer law. [17]

Zhyang et al performs an experiment on carbon fiber T300. They follow the two different methods for testing which are as follows:

1. Temperature programmed controlled method (TPCM).

2. Quenching method.

In technique 1 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 per minute. In this way 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 technique 2 specimen was immiditely submerged in liquid nitrogen so that its temperature go down and attains the cryogenic temperature. Specimen was kept in liquid nitrogen for 12 hours. After 12 hours specimen take out from liquid nitrogen and load was applied on it n room temperature to analyse the physical and mechanical prperties of composite material specimen. [18].

4 Scope of the study

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.

5 Objectives of the study

The objective is to develop the environmental chamber in which we can provide the cryogenic conditions to the composite material and test that composite material under same conditions so that the properties of the composite materials not to be change, due to change in environment, and the outcomes or end results of the experiment should be same as material placed in space and tested over there. So that we can analyze its thermomechanical properties, mechanical properties and physical properties and to check whether there is any difference in outcome results between the testing procedures.

6 Research Methodology

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:

Material: Stainless Steel Grade 316

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

Table 2: Properties of SS316 [19]

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

Fabricate the cylindrical chamber of 2 no's with different diameter 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 cylinder with insulation and wrap it in aluminium foil. Place the insulated cylindrical chamber concentrically inside the cylindrical chamber with bigger diameter. Weld the top & bottom plates of bigger cylinder. 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. This is the overview of Research methodology.

7 Experimental Setup and Results

7.1 Experimental Setup

A SS316 material sheet is purchased from market and cutt it through shearing machine as per required sizes in drawing as shown in pics:

The sheet purchased from market is cut into required sizes pieces is cut through shearing machine whose pictures are attached.

Figure 5 shearing machine

Figure 6 Cutting of SS316 sheet on shearing machine

Figure 4 & 5 shows the shearing of SS316 sheet. Then after cutting of sheet welding of chamber process carried on. Firstly passways of specimen and inlet hole of liquid nitrogen is fabricated as shows in below figure:

Figure 7 Preperation of specimen pockets and LN_2 inlet hole.

Figure 8 Preperation of Pocket for specimen.

Figure 6 & 7 shows the fabrication of specimen pockets in both top and bottom covers of internal as well as external chambers.

Figure 9 Fabrication of Internal Chamber (1)

Figure 10 Fabrication of Internal Chamber (2)

Figure 11 Fabrication of Internal Chamber (3)

Figure 8,9 & 10 shows the fabrication of side walls and both top & bottom covers of internal chamber. After competion of internal chamber external chamber manugacturing is to done whose picture are shown below.

Figure 11 shows the side wall welding of external chamber.

Figure 12 Fabrication of external Chamber (1)

Figure 12 also shows the fabrication of side walls as well as top and bottom covers of external chamber.

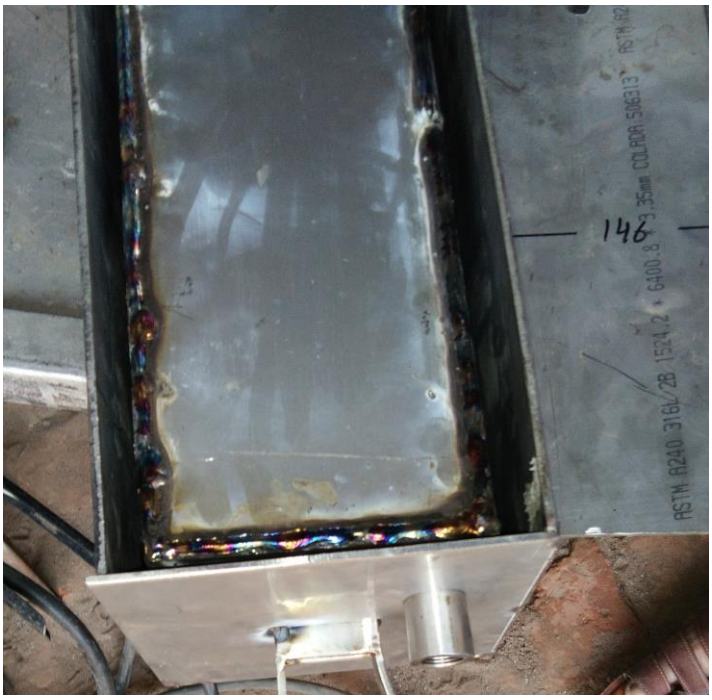

Figure 13 Fabrication of external Chamber (2)

Figure 13 shows the fabrication of attachment which will be connected with suction pump to creat vacuum between the walls of internal and external chambers.

Figure 14 Fabrication of suction pumpattachment asscessory in external Chamber

Figure 15 Fabrication of external Chamber (3)

Figure 14 shows the final welding of outer chamber after which chamber will get complete and looks as shown in figure 15.

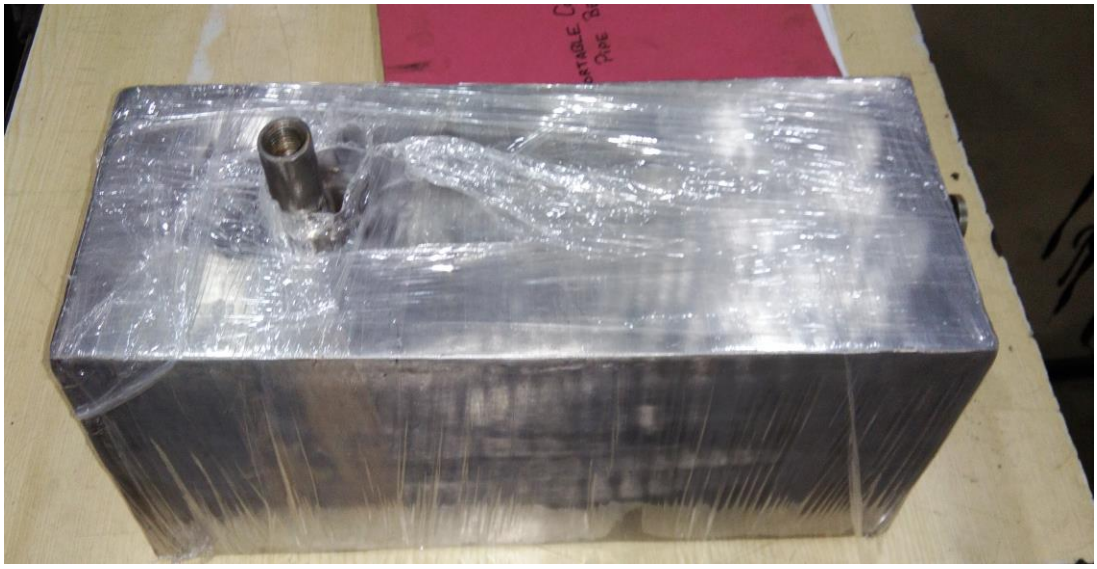

Figure 16 Ready Chamber

Chamber is connected with other equipments in assembly for testing as shown in picture below:

Figure 17 Assembled setup image

Suction pump sucks the air present in between the external chamber and internal chamber walls and maintains the vacuum pressure there for the specified time. Pictures of readings at vacuum gauge are shown below:

Figure 18 Reading of vacuum pressure at initial stage of experiment.

Figure 17 shows the initial reading of vacuum gauge after creating vacuum in between the chamber walls with the help of suction pump.

Figure 19 Reading of vacuum pressure after 10 min from start of experiment

Figure 18 shows the vacuum gauge reading after 10 minutes of start of experiment after stop the suction pump.

Figure 20 vacuum pressure reading after 20 min

Figure 19 shows the vacuum gauge reading after 20 minutes of start of experiment after stop the suction pump.

Figure 21 vacuum pressure reading after 30 minutes.

Figure 20 shows the vacuum gauge reading after 30 minutes of start of experiment after stop the suction pump.

Figure 22 vacuum gauge reading after 40 minutes

Figure 21 shows the vacuum gauge reading after 40 minutes of start of experiment after stop the suction pump.

At this stage vacuum remains constant for long time. We have observe this vacuum for upto 1 hour and takes different readings while observing it for 1 hour.

Now can close the all outways of the internal chamber except the inlet hole for liquid nitrogen. Close the both sample passway upper and lower with rubber and fill the liquid nitrgen upto a certain level into the internal chamber and leave it for few minutes approxximately 30 minutes so that internal chamber walls attains the cryogenic temperature. At this stage some amount of liquid nitrogen will get exausted into the environment. So again fill the liquid nitrogen into the internal chamber and close the lid of inlet hole. Suspend the temperature measuring instrument into the internal chamber so that internal temperature can be measured. Leave it for 1 hour and observe the readings of temperature variation.

7.2 Results

7.2.1 Reading 1

After connecting the suction pump with chamber pump start suction the air from the internal and external chamber walls then after few minutes suction is attain at 933.257 mbar. Then pump switch off the pump and observe the following readings at different time laps which are graphically shown below.

Time Lap	time in minutes	vaccum Reading	Vaccum Reading
0	0	700 mmhg	933.257 mbar
13 min	13	620 mmhg	826.599 mbar
25 min	38	450 mmhg	599.951 mbar
09 min	47	390 mmhg	519.957 mbar
13 min	60	220 mmhg	293.309 mbar

Once 700 mmhg achieved then motor shut down and keep watching for vaccum for 1 hour.

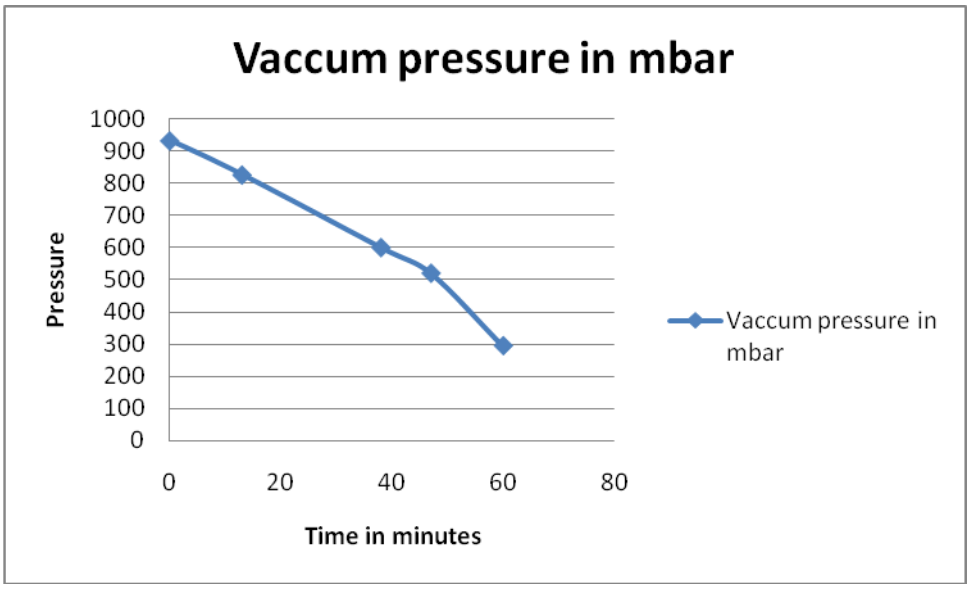

Figure 23 Graphical representation of readings observed from vacuum gauge.

Total drop in vaccum pressure in one hour is 640.218 mbar

7.2.2 Reading 2

After connecting the suction pump with chamber pump start suction the air from the internal and external chamber walls then after few minutes suction is attain at 919.924mbar. Then pump switch off the pump and observe the following readings at different time laps which are graphically shown below.

Time Lap	time in minutes	vaccum Reading	Vaccum Reading
0	0	690 mmhg	919.924 mbar
10 min	10	590 mmhg	786.602 mbar
10 min	20	490 mmhg	653.28 mbar
10 min	30	400 mmhg	533.29 mbar
10 min	40	310 mmhg	413.299 mbar
10 min	50	260 mmhg	346.638 mbar
10 min	60	200 mmhg	266.645 mbar
10 min	70	160 mmhg	213.316 mbar

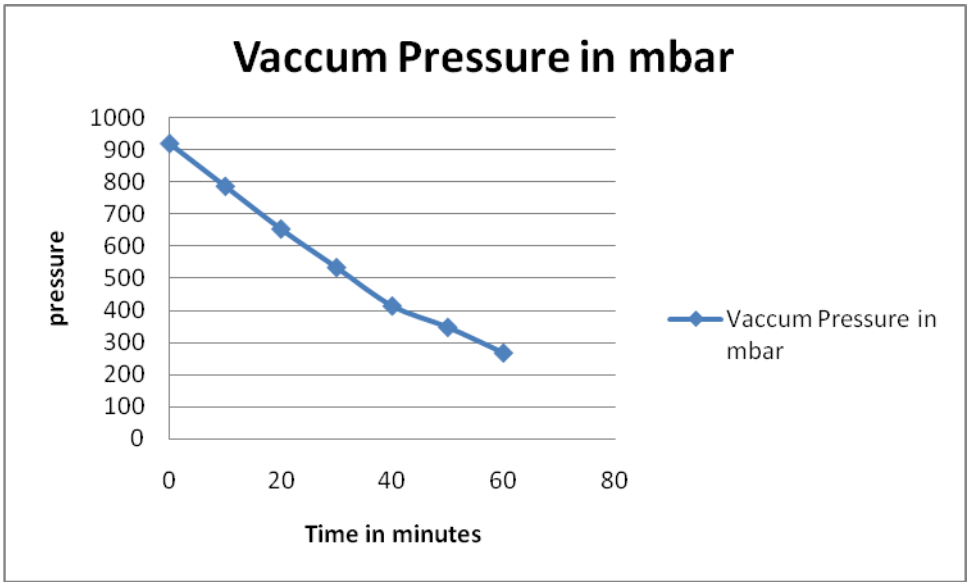

Figure 24 Graphical representation of readings observed from vacuum gauge

Drop in vaccum pressure in one hour is 653.279 mbar

7.2.3 Reading 3

After connecting the suction pump with chamber pump start suction the air from the internal and external chamber walls then after few minutes suction is attain at 919.924mbar. Then pump switch off the pump and observe the following readings at different time laps which are graphically shown below.

Time Lap	Time in minutes	vaccum Reading	Vaccum Reading
0 min	0	690 mmhg	919.924 mbar
5 min	5	640 mmhg	853.263 mbar
5 min	10	560 mmhg	746.605 mbar
5 min	15	510 mmhg	679.944 mbar
5 min	20	470 mmhg	626.615 mbar
5 min	25	420 mmhg	559.954 mbar
5 min	30	380 mmhg	506.625 mbar
5 min	35	320 mmhg	426.632 mbar
5 min	40	300 mmhg	399.967 mbar
5 min	45	260 mmhg	346.638 mbar
5 min	50	190 mmhg	253.313 mbar
5 min	55	190 mmhg	253.313 mbar
5 min	60	180 mmhg	239.98 mbar

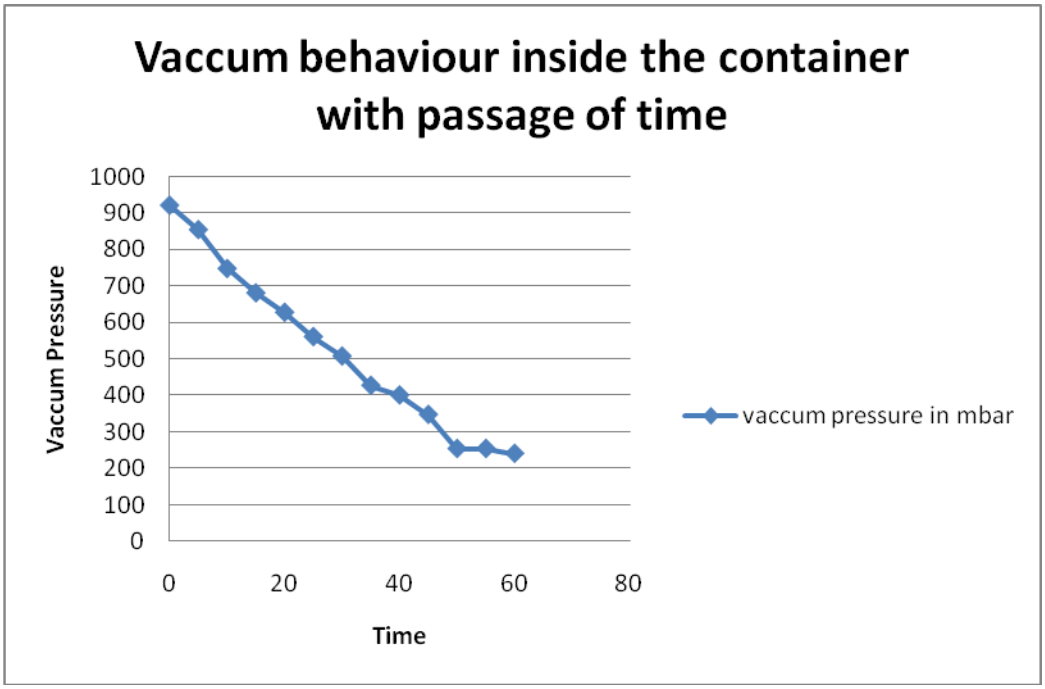

Figure 25 Graphical representation of readings observed from vacuum gauge

7.2.4 Reading 4

After connecting the suction pump with chamber pump start suction the air from the internal and external chamber walls then after few minutes suction is attain at 919.924mbar. Then pump switch off the pump and observe the following readings at different time laps which are graphically shown below.

Time Lap	Time in minutes	vaccum Reading	Vaccum Reading
0 min	0	685 mmhg	913.258 mbar
5 min	5	630 mmhg	839.931 mbar
5 min	10	565 mmhg	753.271 mbar
5 min	15	513 mmhg	683.944 mbar
5 min	20	475 mmhg	633.281 mbar
5 min	25	413 mmhg	550.621 mbar
5 min	30	390 mmhg	519.957 mbar

5 min	35	335 mmhg	426.632 mbar
5 min	40	305 mmhg	406.633 mbar
5 min	45	275 mmhg	366.637 mbar
5 min	50	200 mmhg	266.645 mbar
5 min	55	200 mmhg	266.645 mbar
5 min	60	200 mmhg	266.645 mbar

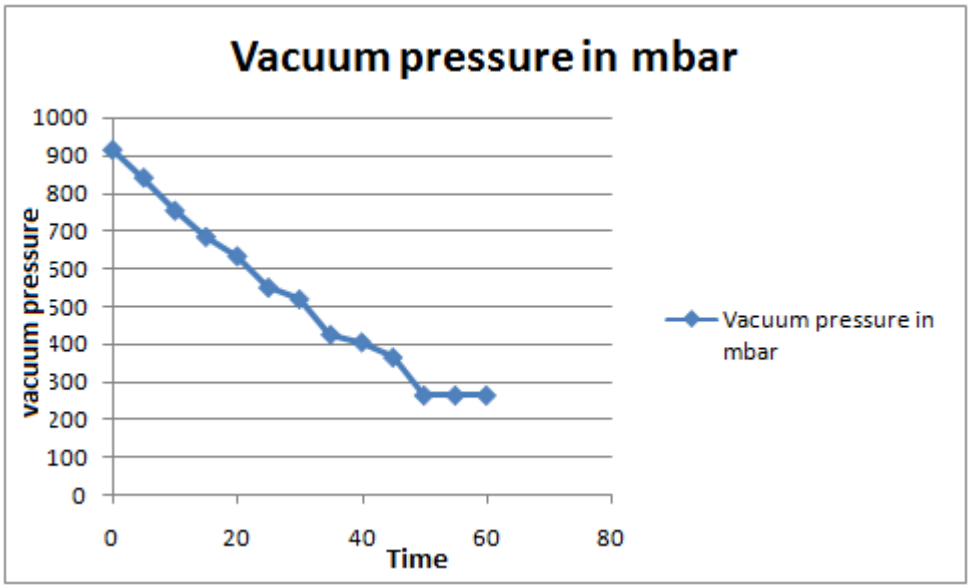

Figure 26 Representation of readings observed from vacuum gauge

8 Expected outcomes

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.

9 Conclusion and Future scope

9.1 Conclusion

It is observed that vacuum can be achieved for only less than 1 hour which means that there is some leakage of vacuum pressure either through assembly attachments or through the welding done on the edge of the internal or external chamber. To rectify this we can do the leakage test of chamber with hydrogen gas whose atomic size is very very small.

9.2 Future scope

We can do the leakage test of chamber with hydrogen gas whose atomic size is very very small.

When it passes the leakage test again vacuum testing can be carried out on this chamber. After which we can close the all outways of the internal chamber except the inlet hole for liquid nitrogen. Close the both sample passway upper and lower with rubber and fill the liquid nitrogen upto a certain level into the internal chamber and leave it for few minutes approximately 30 minutes so that internal chamber walls attains the cryogenic temperature. At this stage some amount of liquid nitrogen will get exhausted into the environment. So again fill the liquid nitrogen into the internal chamber and close the lid of inlet hole. Suspend the temperature measuring instrument into the internal chamber so that internal temperature can be measured. Leave it for 1 hour and observe the readings of temperature variation.

10 References

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