IJSRM
INTERNATIONAL JOURNAL OF SCIENCE AND RESEARCH METHODOLOGY
An Official Publication of Human Journals

Human Journals
**Research Article**
March 2019 Vol.:12, Issue:1


# Carbon-Epoxy Composite Having Thermal Responsive Self-Healing Properties for Structural Applications

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An Official Publication of Human Journals

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**Submission:** 22 February 2019
**Accepted:** 28 February 2019
**Published:** 30 March 2019



**Keywords:** Biomimetic, Shape Memory Polymer, Close-then-heal, MFIP technique

## ABSTRACT

Self-healing materials are artificial or synthetically-created substances which have the built-in ability to automatically repair damage to themselves. Generally, materials will degrade over time due to fatigue, environmental conditions, or damage incurred during usage. Cracks and other types of damage on a microscopic level have been shown to change thermal, electrical, and acoustical properties of materials, and the propagation of cracks can lead to eventual failure of the material. Formation of microscopic cracks which leads to macroscopic cracks of damages is a persistent problem in most structural and space applications. A biomimetic Shape Memory Polymer (SMP), the ability of a self-healing composite to heal, and to repair and restore structural-length scale damage using a Close-Then-Heal (CTH) self-healing mechanism. The related tools have been used for shape memory self-healing thermosets epoxies with different epoxide equivalent, cured with phenol formaldehyde, and reinforced with carbon woven fabric cloth. The blend of different epoxies and phenol formaldehyde was prepared using MFIP technique, i.e. Multi Functionality Introduced at Prepreg (MFIP) stage. The Prepreg of carbon fiber was be prepared by this technique for high temperature resistant composite material for aerospace application. The main emphasis of the investigation was to evaluate mechanical thermal and self-healing properties. As per the concept of self-healing composite high MW epoxy resin being used to act as a thermoplastics material which on temperature change will flow in damaged area and heal the composite material.

## INTRODUCTION

Formation of microscopic cracks which leads to macroscopic cracks of damages is a persistent problem in most structural and space applications. A biomimetic shape memory polymer (SMP), the ability of a self-healing composite to heal, and to repair and restore structural-length scale damage using a close-then-heal (CTH) self-healing mechanism. The related tools has been used for shape memory self-healing thermo sets epoxies with different epoxide equivalent, cured with phenol formaldehyde and reinforced with carbon woven fabric cloth. The blend of different epoxies and phenol formaldehyde was prepared using MFIP technique, i.e. Multi Functionality Introduced at Prepreg *(*MFIP*)* stage. The Prepreg of carbon fiber was be prepared by this technique for high temperature resistant composite material for aerospace application. The main emphasis of theon was to evaluate mechanical thermal and self-healing properties. As per the concept of self-healing composite high MW epoxy resin being used to act as a thermoplastics material which on temperature change will flow in damaged area and heal the composite material.

### Definition of self-healing

Self-healing materials exhibit the ability to repair themselves and to recover functionality using the resources inherently available to them. Self-healing can be defined as the ability of a material to heal (recover/repair) damages automatically and autonomously, that is, without any external intervention. Many common terms such as self-repairing, autonomic-healing, and autonomic-repairing are used to define such property in materials. Incorporation of self-healing properties in manmade materials very often cannot perform the self-healing action without an external trigger. Self-healing materials offer a new route toward safer, longer-lasting products and components. Self-healing materials offer a new route toward safer, longer-lasting products and components. For thermal responsive shape memory polymers, the shape memory effect is observed by performing a typical thermo-mechanical cycle.

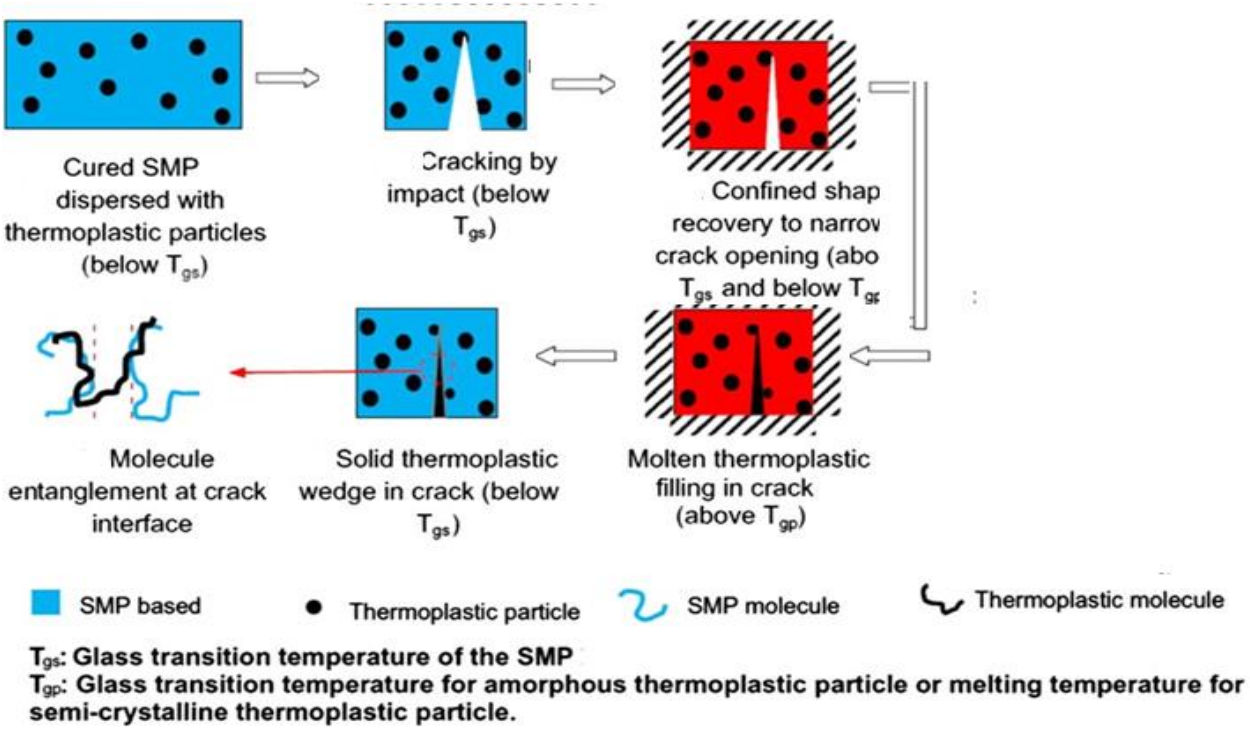


**Figure 1: Shape memory effect**

The concept of self-healing assisted shape memory effect composite high MW epoxy resin being used to act as a thermoplastics material which on temperature change will flow in damaged area and heal the composite material.

## MATERIALS AND METHODS

### Materials

The raw materials used in this research are:

**Table 1: Details of the raw material**

| S No | Material | Material detail & Function |
|---|---|---|
| 01 | High molecular weight epoxy | 5.6 equivalent/kg( healing) |
| 02 | Low molecular weight epoxy | 2.2 equivalent/kg( matrix) |
| 03 | Phenol formaldehyde | Resol( curing agent ) |
| 04 | Plain weave carbon fiber cloth | 12k( reinforcement) |

**High molecular weight epoxy** was in powder form and the chemical name of epoxy is (poly(bisphenol-A-co-epichlorohydrin) procured from M/s Atul Ltd, Mumbai, India. **Low**

**molecular weight epoxy** was in liquid form, and the chemical name is epoxy (diglycidyl ether of bisphenol-A) was obtained from M/S Mitsubishi chemical corporation under the trade name of JER828. **Phenol formaldehyde** resin used in this study was TPF/W/1617, commercially available from Tipco Industries Ltd., GUJRAT, India, which is a solventless resole type phenolic resin, having the solid content 81.70%. The physiochemical characteristics of resin were liquid with reddish brown in color having viscosity of 4800Cps at $25^0$C, pH 7.3 and gel time 7.6sec at $160^0$C. **Plain weave carbon fiber cloth** originated for aerospace use, but as it has become more usable this material expanded into the automobile industry.

In this research plain (1×1) weave carbon fiber is used 12k bundle size woven carbon fabric procured from M/S Adorn Engineers, Ahmedabad, Gujarat. According to their name weave pattern goes up one and down one. Plain weave then makes a tighter knit fabric and is easier to handle without making any distortions.

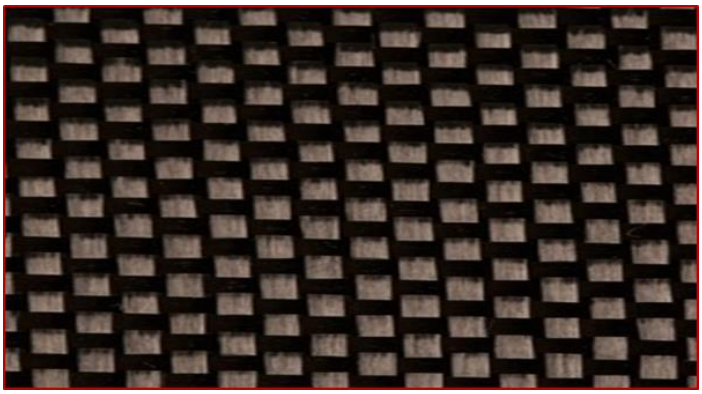

**Figure 2: 1×1 plain weave carbon fiber**

**Methodology**

Carbon fiber was cut into different sheets of same dimensions (21cm×15cm), with the help of hand layup process the resin according to composition shown in table no. 02 was applied on it. Total three samples were prepared with this technique. Eight layers were stacked together to form a composite. After applying resin on the sheets of fabric cloth, sheets were placed in oven for 5 minutes to 7 minutes at 80 ºC. Due to this tackiness of the resin was reduced. Cool the sheets at room temperature and stored in plastic sheets.

**Preparation of carbon fiber Prepreg with MFIP (Multi Functionality Introduce at Prepreg) technique:**

This blend system was used in Prepreg mode, i.e. half reaction was allowed to form and multi functionality was introduced at this stage. The chain extension was done using this system and prepolymer with chain extended epoxy has been formed by further curing. The resin matrix was heated to 150ºC for 2 hours to obtain multi functionality incorporated prepolymer. This was again cured at 200 ºC for two hours. Curing of the resin was established by using FT–IR.

In the present investigation, the phenol formaldehyde of resole type has been chosen for blending due to flame retardency characteristic and ability to form multi functionality adduct to eliminate the curing of epoxy hardening system. In the proposed molecular design a multifunctional epoxy (diglycidyl ether of bisphenol-A) was converted into intermediate adduct by reaction of phenolic resin. The intermediate adduct formation has been found due to varying reactivity's associated with two reactions viz. phenolic hydroxyl functionality with epoxy resin and self-condensation of methylol functionality of resol, i.e. curing of phenolic resin. is used as a chain extender of epoxy resin, thereby decreasing the cross linking density of the resultant matrix and increase the thermal stability with increase in other properties., for which the reaction is shown in Figure 3. Thermogravimetric analysis has been done for DEBA –PR compositions.

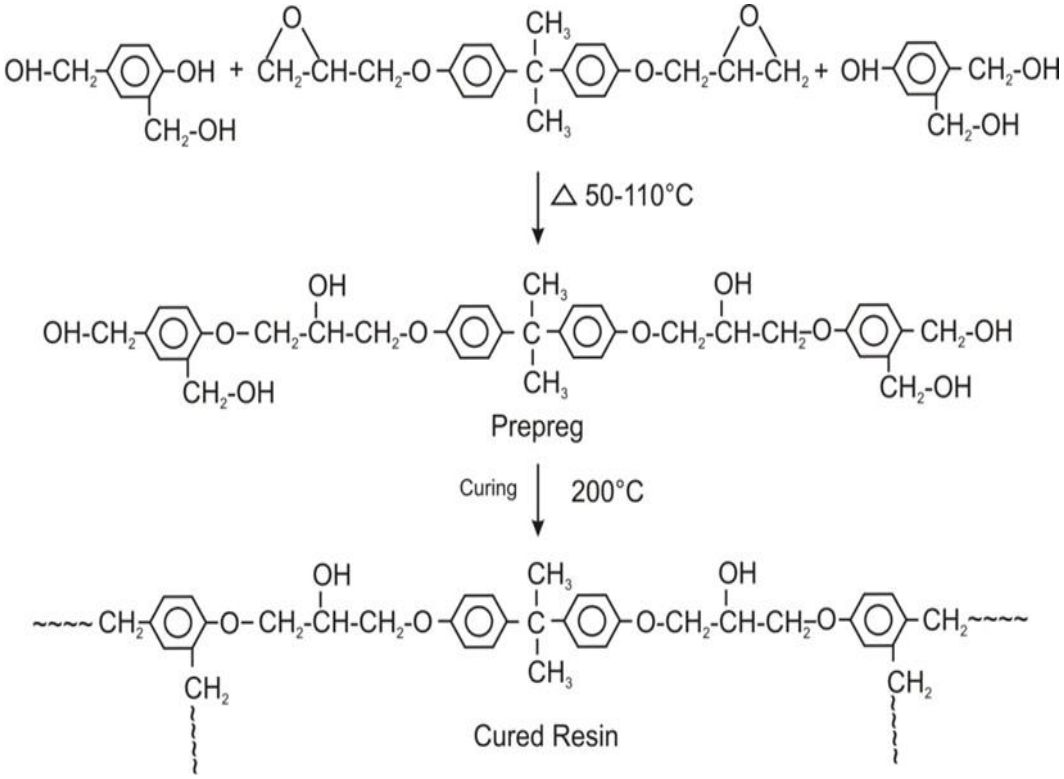


**Figure 3: Reaction at Prepreg mode and at curing mode**

The high molecular weight epoxy does takes part in the curing reaction but it forms a semi interpenetrating polymer network (a polymer comprising one or more networks and one or more linear or branched polymer characterized by penetration on a molecular scale of at least one of the networks by at least some of linear or branched macromolecules. In this network, in short the linear or branched polymers can, in principle, be separated from constituent polymer networks without breaking chemical bonds). The evidence of this phenomenon is shown in DSC thermal analysis.

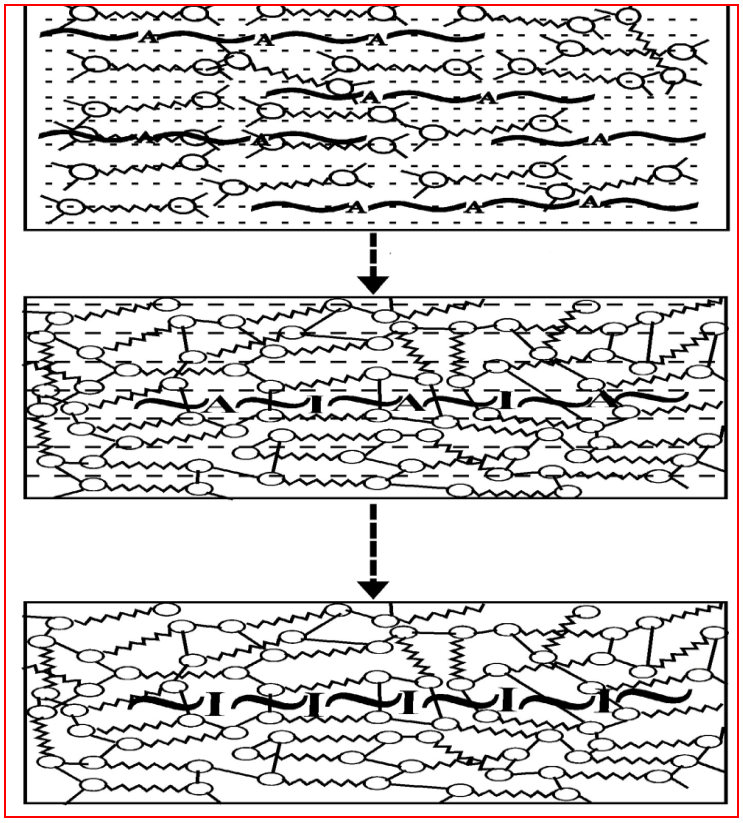

**Figure 4: Molecular arrangement of epoxies and curing agent (- - - lines shows the phenol formaldehyde curing agent, chain with rings shows the low molecular weight epoxy and dark line shows the high molecular weight epoxy)**

Three types of compositions are prepared by using 100 parts of epoxy with constant proportion of phenol formaldehyde.

**Table 2: Different compositions of the material**

| Designation | High MW epoxy (%) | Low MW Epoxy (%) | Phenol formaldehyde (gms) |
|---|---|---|---|
| Sample1 | 90 | 10 | 33 |
| Sample2 | 85 | 15 | 33 |
| Sample3 | 80 | 20 | 33 |

**Figure 5: Prepreg sheets of carbon fiber cloth**

In next step these Prepreg sheets which was stacked together was placed in mold of compression molding machine for 4hours. The curing reaction is divided into two parts shown in figure 5 and 6.


**Figure 6: Epoxy/carbon fiber composite**

***Based on the above work the testing and characterization of the composite prepared has been done and one paper is n progress.***

## Characterization

### Fourier Transform Infrared Spectroscopy:

Fourier Transform Infrared Spectroscopy (FTIR) is a technique which is used to obtain an infrared spectrum of absorption/ transmission of solid, liquid or gas. From FTIR analysis we can get the following information

- ✓ It can identify unknown materials
- ✓ It can give the information of chemical groups present in the sample
- ✓ It can determine the amount of components in a mixture

Infrared region is mainly divided into three types:

- ➢ Near IR : 14000-4000 $cm^{-1}$ ( 0.8-2.5 μm wavelength )
- ➢ Mid IR : 4000-400 $cm^{-1}$ ( 2.5 -25μm wavelength )
- ➢ Far IR : 400-10 $cm^{-1}$ (25-1000 μm wavelength )

In FTIR, most of the data are analyzed in Mid IR region. This region is again divided into two categories:

- ✓ Functional group region: 4000-1500$cm^{-1}$ wavelength
- ✓ Fingerprint region: 1500-400 $cm^{-1}$ wavelength

### Thermal analysis

There are numerous ways of characterizing a polymer/ composites, thermal analysis is one of them. Thermal analysis consists of a family of analytical techniques in which a property of the sample is monitored against time or temperature while the temperature of the sampleis programmed. Three different types of thermal analysis test are performed on the sample, they are:

- o DSC (Differential Scanning Calorimeter)
- o TGA (Thermogravimetric Analysis)

- DMA (Dynamic Mechanical Analysis)

## RESULTS AND DISCUSSIONS

### Mechanical Analysis of *Carbon Fibre Self-Healing Polymer Composites*

The mechanical properties, among all the properties, are often the most important properties because virtually all service conditions and the majority of end-use applications involve some degree of mechanical loading. The two main tests are performed in this research.

***Tensile strength***; tensile elongation and tensile modulus measurements are among the most important indication of strength in a material and are the most widely specified properties of plastic materials. Tensile strength, in a broad sense, is a measurement of the ability of a material to withstand forces that tends to pull it apart and to determine in what extend the material stretches before breaking.

Tensile strength at break = load recorded at break / cross- section area

The tensile strength of the composite made is given in ***Table-3***:

***Table-3: Tensile Strength of Self-Healing Composite***

| Batch No | Area($mm^2$) | Breaking Load(KN) | Tensile Strength at break (MPa) |
|---|---|---|---|
| S1 | 40.18 | 8.61 | 214.2 |
| S2 | 36.26 | 9.94 | 274.1 |
| S3 | 37.87 | 8.92 | 235.5 |

The samples were subjected to test for tensile strength using Universal Testing Machine (UTM) and the broken sample is shown in ***Figure-7***.

***Figure 7: Sample of Self-Healing composite after tensile strength***

It was observed from ***Table -3*** and ***Figure8*** that formulation S-2 showing highest tensile strength (improvement of 28%) could be due to favorable adduct formation and highest reinforcement with carbon fiber and as the high molecular weight epoxy increases the mechanical properties were also reduced almost 14%. This phenomenon indicates that high molecular weight which will be acting as plastic in nature and does not taking part in the cross-liking reaction surely will flow on Tg of the materials when further heated.

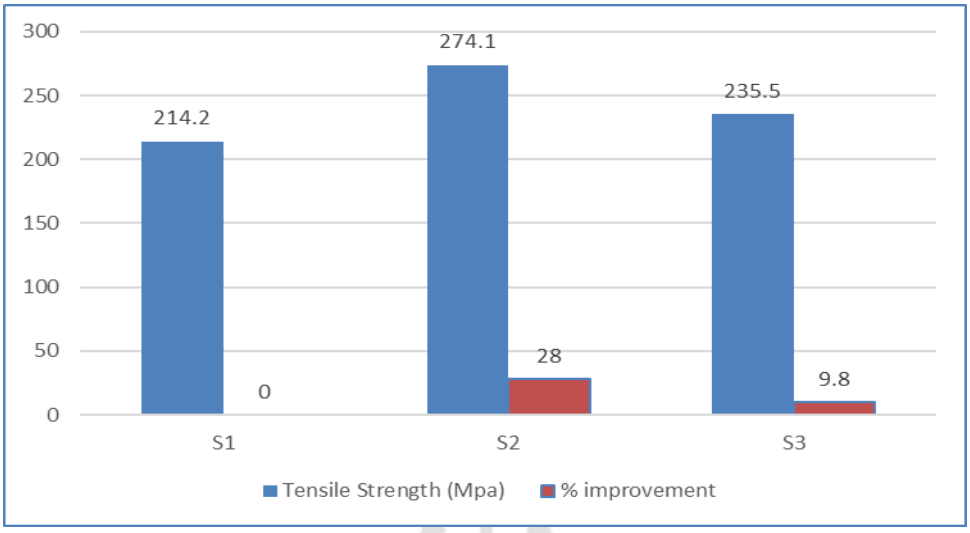


***Figure-8: Tensile Strength and Percentage improvement of Carbon Fibre Self-Healing Polymer Composites***

**Flexural Strength and Modulus**

Flexural strength is the ability of the material withstands bending forces applied perpendicular to its longitudinal axis. The stresses induced by the flexural load are a combination of compressive and tensile stresses. Flexural properties are calculated in the terms of maximum stress and strain that occur at outside surface of test sample. The flexural Properties of self-healing composite is given in ***Table-4***.

***Table-4: Flexural strength and flexural Modulus of self-healing composite.***

| Batch No | Flexural Strength (MPa) | Flexural Modulus (MPa) |
|---|---|---|
| S1 | 157.63 | 20959.36 |
| S2 | 239.56 | 35728.07 |
| S3 | 211.20 | 25121.30 |

In case of Flexural strength and percentage, improvement is depicted in ***Figure 9*** and Flexural modulus and percentage improvement is depicted in ***Figure 9 and Figure 10.***

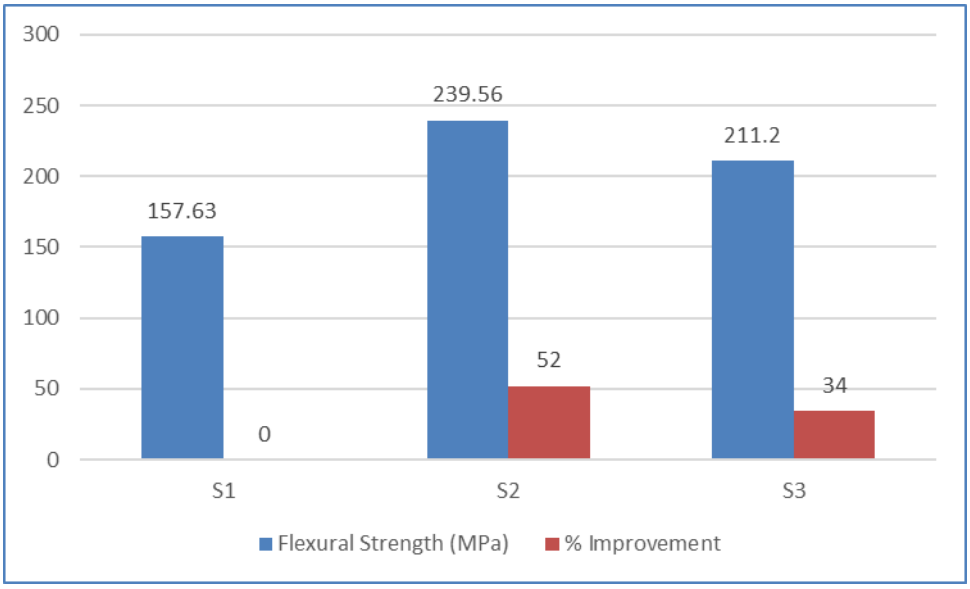


***Figure-9: Flexural Strength and Percentage improvement of Carbon Fibre Self-Healing Polymer Composites***

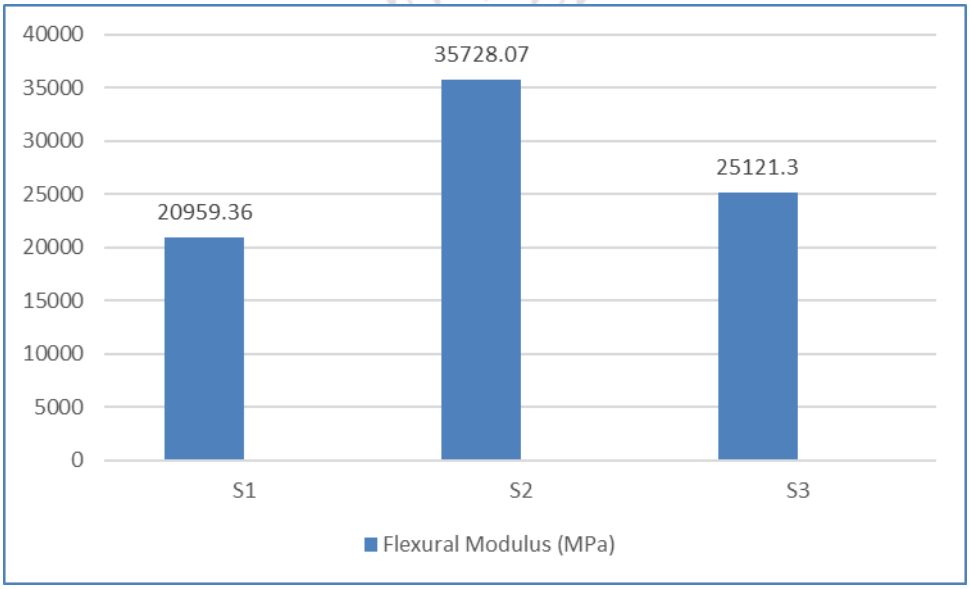


***Figure-10: Flexural Modulus of Carbon Fibre Self-Healing Polymer Composites***

Flexural strength also showing similar results like tensile strength and there is 52% improvement in flexural strength in case of S2 formulations. Flexural Modulus values showing 70% improvement in case of S2 which proved that there is good interface and there is favorable bonding formation in case of 85% of epoxy resin samples.

FTIR(Fourier Transform Infrared Spectroscopy) is an effective analytical tool for screening and profiling polymer samples. FTIR identifies chemical bonds in a molecule by producing an infrared absorption spectrum. The resulting spectra produces a profile of the sample, a distinctive molecular fingerprint that can be used to easily screen and scan samples for many different components. FTIR is an effective analytical instrument for detecting functional groups and characterizing covalent bonding information. FTIR analytical testing and data interpretation for polymers are available globally.

In order to facilitate the study of quantitative conversion of the resin to the intermediate adduct, FTIR analysis has been chosen here. The consumption of the epoxy was monitored from the peak ratio at 914 $cm^{-1}$(characteristics of epoxide ring stretch ) and 1500$cm^{-1}$ (assigned to semicirde stretching of benzene ring).

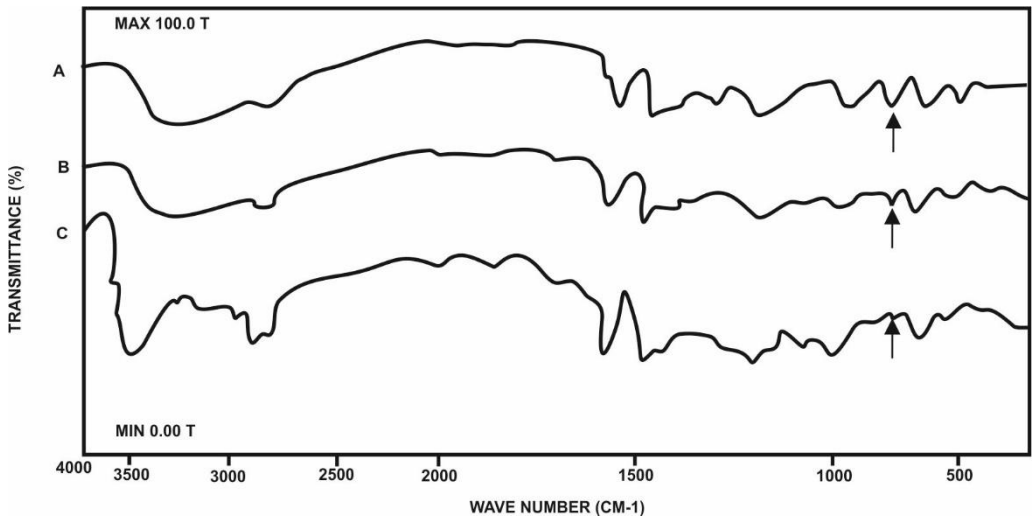


**Figure 11: FTIR Graph of sample S1**

**A shows curve at room temperature, B shows curve at prepreg mode, C shows curve for curing**

Most of the epoxy groups are consumed at prepreg preparation. The facts that only adduct formation takes place and not the primary phenolic resin curing was established only qualitatively by comparing the absorption peak ratio at 2900 $cm^{-1}$(the methylene- CH stretching) and 1500$cm^{-1}$. Since there is no change in the absorption peak ratio (2900/1500) during prepreg preparation, it is indicated that the curing reaction (characteristic of phenolic ring curing) did not takes place. The figure shows the typical FTIR spectra for matrix compositions at different stages i.e. from time of mixing up of two resins at room

temperature, prepreg preparation stage and full curing of the matrix. The disappearance of the characteristics absorption band at 914 $cm^{-1}$ for epoxy after preprag preparation provides the basis for the PR-EP adduct formation.

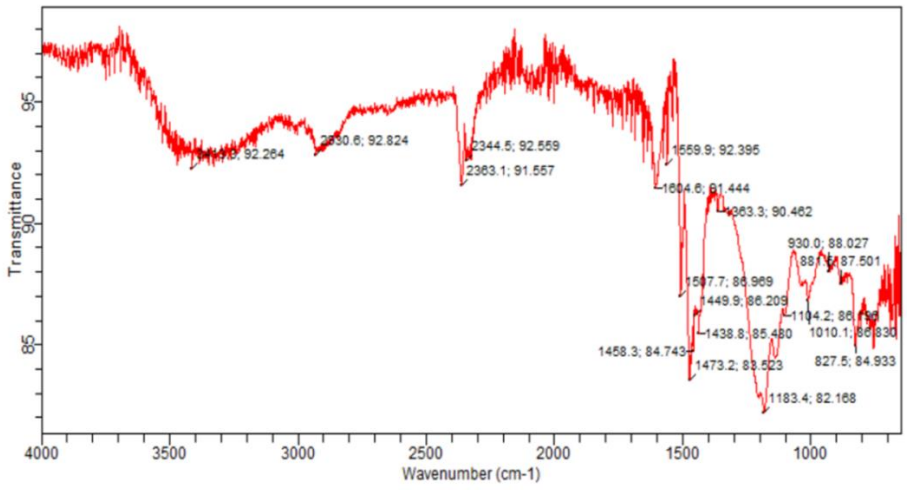


**Figure 12: FTIR sample 1**

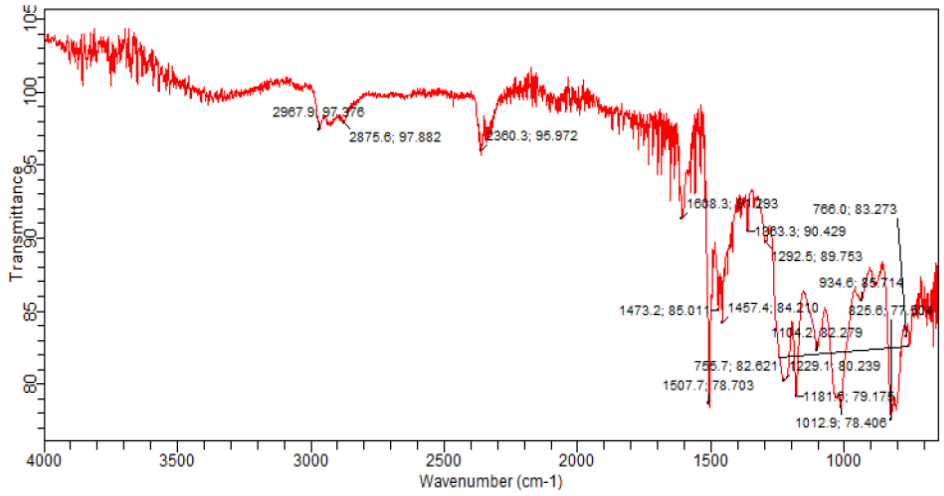


**Figure 13: FTIR sample 2**

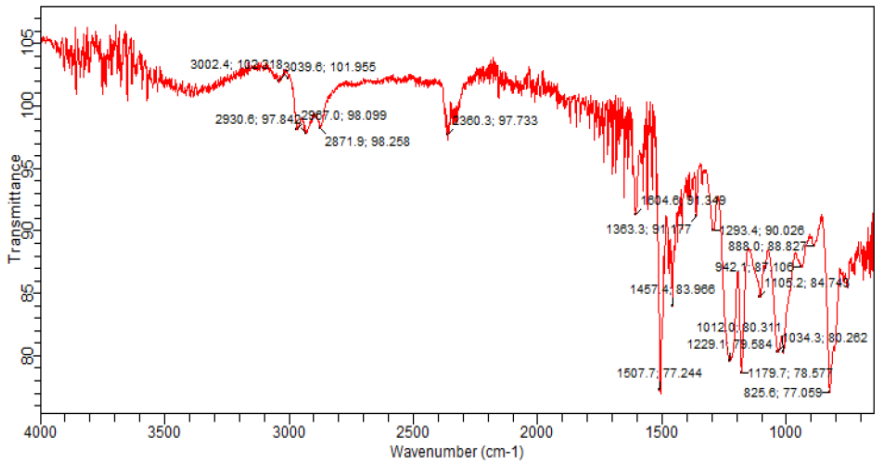


**Figure 14: FTIR sample 3**

**Differential scanning calorimeter**

Perkin Elmer, Dimond DSC was applied to observe the effect of high molecular weight epoxy resin added in epoxy matrix and to get the information of curing of the rein. The sample containing higher molecular weight epoxy resin showing melting peak at 257.04 ˚c which indicates that the high molecular weight epoxy resin acting as thermoplastic material and does not take part in the curing reaction. This resin will heal the polymer when cracking in the composite happened when subjected to heal it near to their Tg value.

The other sample S1 and S3 does not showing any thermoplastic behavior hence S2 will be the right choice to use as selfheal polymer. The Tg of the matrix determined by DMA and will be discussed in next section.

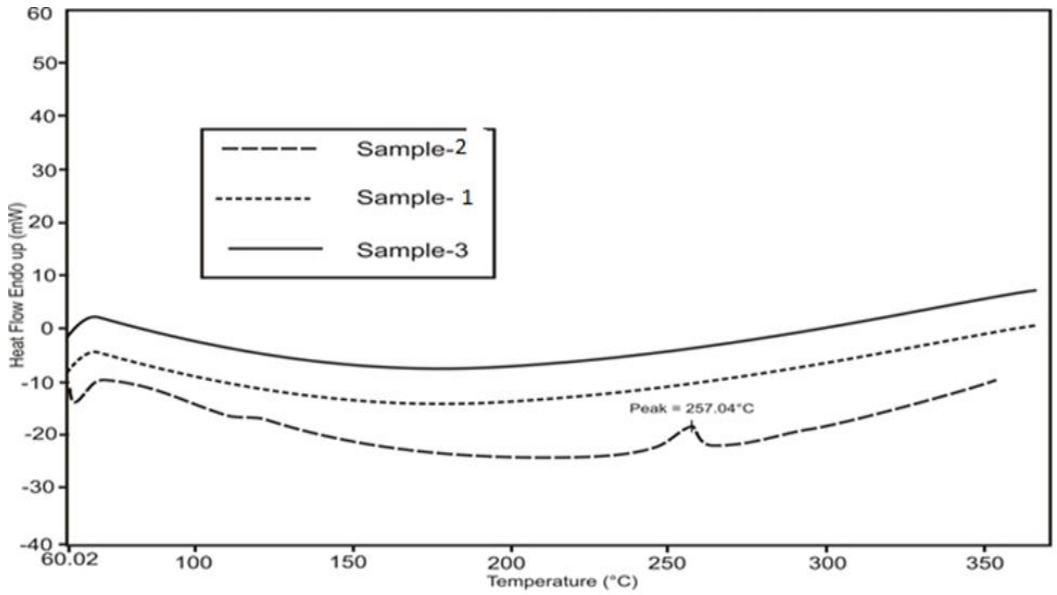


**Figure 15: DSC results of three different samples.**

**Thermogravimatric analysis (TGA)**

Pyris TGA, temperature rise from -50 ˚c to 850˚ c and rate of rise 20 c/min is used to determine the degradation pattern of resin matrix. On comparison it was found that sample S2 degrading with two step mechanisms. The first step degradation indicates degradation of thermoplastic epoxy resin which actually doesn't cure and does not acted as part and parcel of cured resin. Further, the sample degrade which indicate the degradation of cured resin, whereas other samples S1 and S3 showing single step degradation which again proves that the sample containing High molecular weight epoxy resin acted as self-heal polymer. This thermoplastic material suspended in cross linked structure of EP- Phenolic resin.

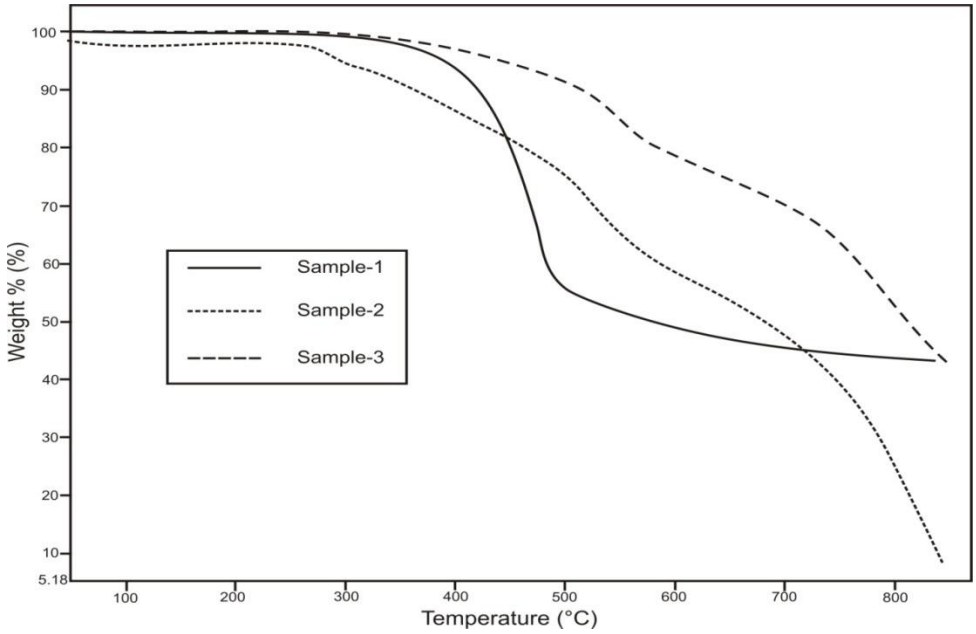


**Figure 16: TGA results of three different samples**

**Dynamic Mechanical Analysis (DMA)**

The Heating rate is from -40 ˚c to 250˚ c and Common rate of rise is used. The DMA is used to determine the mechanical deformation of a sample when a constant or fixed rate of stress is applied with respect to the function of the temperature. The information on DMA is general damping factor and stiffness reported. The storage modulus (E')used to measure the sample elastic modulus. The ratio of loss to storage is tan delta and is often called damping. It is a measure of energy dissipation of material. Sample S2 showing two Tg temperature, one is at 54˚C and other one is at 108˚C which again indicates the thermoplastic behavior of high molecular weight epoxy resin. The tan delta value is correlated with storage modulus value which again showing 103˚C and loss modulus is at 90˚C confirming that the load bearing characteristics is approx. 100˚c and if any reason crack happened in the composite self healing property can be achieved by again heating up to 60-70˚C.

In other samples like S1 and S3 shows only one Tg and obtained tan delta value is approx. 60˚c which shows single matrix behavior.

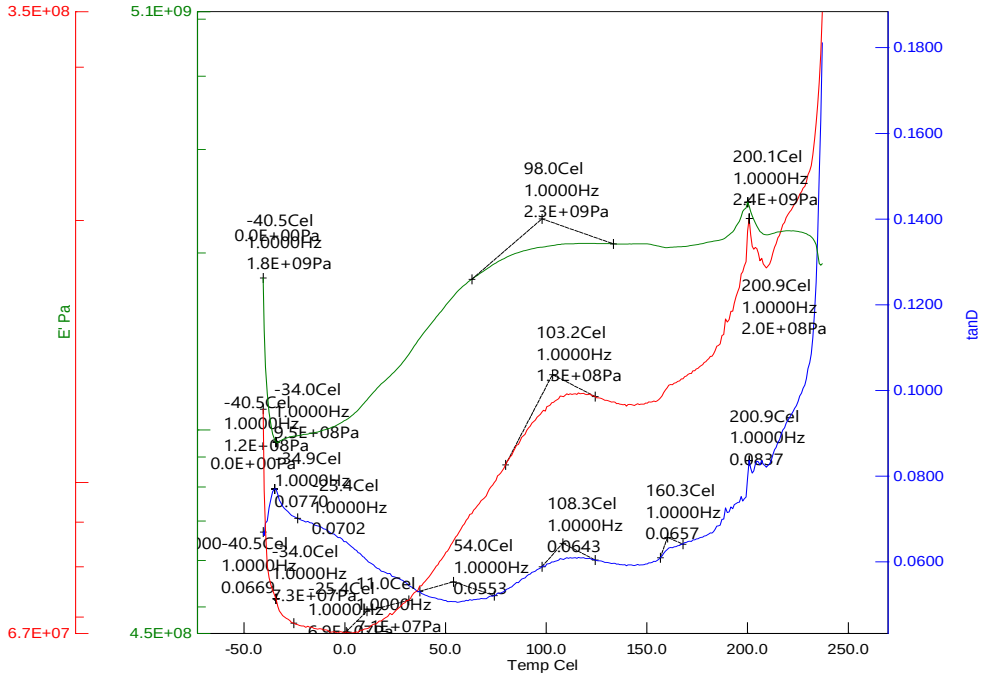


**Figure 17: DMA result of sample 1**

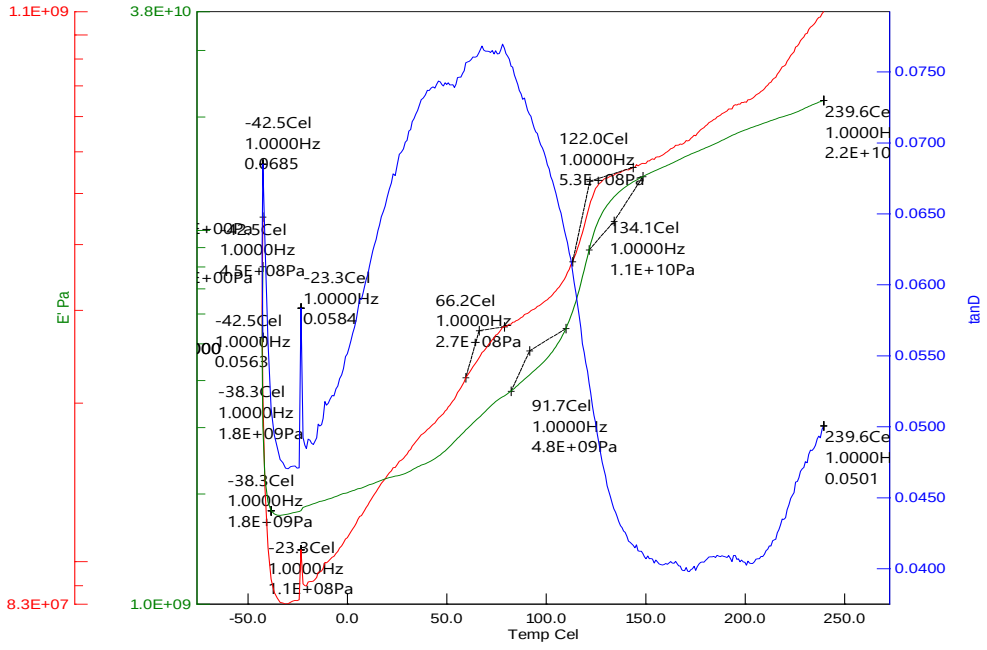


**Figure 18: DMA result of sample 2**

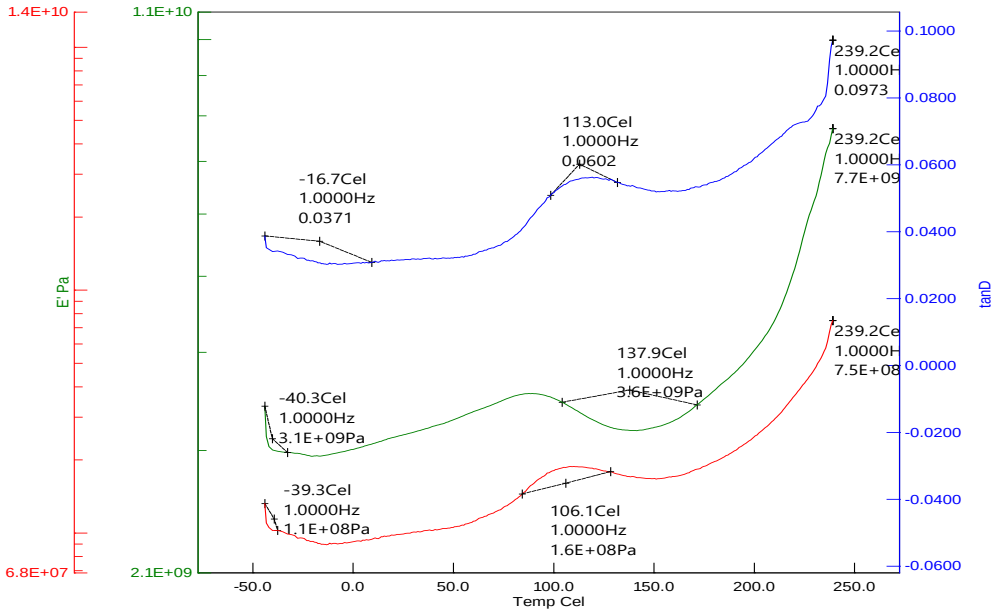


**Figure 19: DMA results of sample 3**

**Scanning Electron Microscopy (SEM)**

A SEM is a type of electron microscope that produces image of a sample by scanning it with a focused beam of electrons. The electrons interact with atoms in the sample, producing various signals that can be detected and that contain information out the sample surface topography and composition. The electron beam is generally scanned in a raster scan pattern, and the beam position is combined with the detected signals to produce an image. SEM can achieve resolution better than 1 nanometer. Specimens can be observed in high vacuum, in low vacuum, in wet conditions and at a wide range of cryogenic or elevated temperatures. The photograph of SEM is given in ***Figure-20***.

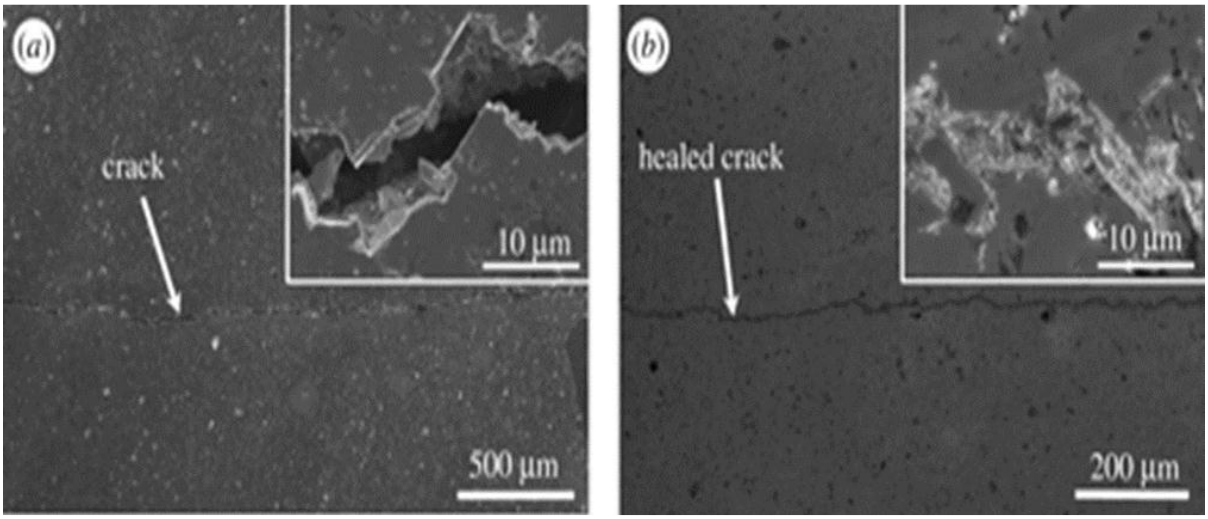


***Figure 20: SEM Photographs of Self-Healing Composites***

**Optical Microscope**

A simple microscope is a microscope that uses only one lens for magnification, and is the original light microscope. Van Leeuwenhoek's microscopes consisted of a single, small, convex lens mounted on a plate with a mechanism to hold the material to be examined (the sample or specimen). Demonstrations by British microscopist Brian J. Ford have produced surprisingly detailed images from such basic instruments. The use of a single, convex lens to magnify objects for viewing is found today only in the magnifying glass, the hand-lens. The optical photographs of self-healing composite are depicted in ***Figure 21***.


***Figure 21: Optical microscope pictures of crack and crack healed sample***

The self-healing behavior of the matrix depicted in ***Figure-21***. Sample S2 the thermoplastic epoxy suspended in cross linked network structure of epoxy phenolic resin which is forming a semi interpenetrating network basically responsible for self-healing behavior.

On cooling sample, S 2 thermoplastic epoxy resin and their Tg flow to this crack area and heal the polymer matrix and thereby this system is self-heal polymer matrix system with prepregable technology. This is prepared by DSC analysis that there is a prominent role of thermoplastic epoxy resin for self-healing property and there is clear cut evidence in SEM and optical microscope that this thermoplastic resin is taking part to self-heal the polymer.

## CONCLUSIONS

The results of mechanical properties proved that S2 samples showing highest improvement in mechanical properties and can be used for structural applications. The results of FTIR proved that there is an adduct formation is taking place between phenolic resin and epoxy resin and high molecular weight epoxy resin suspended in the matrix act as self-healing agent.

The result of DSC shows that these high molecular weight epoxy does not take part in curing reaction and suspended in cured matrix of low MW epoxy & phenolic. DMA results also matching with the results obtained for mechanical properties and S2 composition showing highest improvement in mechanical properties. DMA analysis also indicating that there is high molecular weight epoxy showing two Th value which actually work as self-healing agent during damage of these composite during heat treatment. The TGA once again proved the above results high molecule weight epoxy does not take part in curing reaction and suspended in cured matrix of low MW epoxy & phenolic .Schematically which is also present in result and discussion section.SEM and Optical micrograph of these damaged

composite material showed how the cracking healing mechanism happening. The desired mechanism property like tensile strength and modulus also achieved by making these C -fiber prepreg composite.

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