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Self- Healing Composites: A Review on Material Characterization and Testing Methods

Danica Rayen, Siva Vigneshwaran U, Harini Vuppalapati, Arko Pravo Chakraborty, Kanu Priya Jhanji*, Amit Kumar R
School of Aeronautical Sciences, Hindustan Institute of Technology and Science, Chennai, Tamil Nadu, India 603103.
*Mail id: kanujhanji@gmail.com

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***Abstract*:**
The intent of this work is to give account of recent trends in self-healing composites since 2016. Self-healing materials are well known for their natural healing tendency, faster recovery when damaged. In this work, initially the various methods of healing are discussed. The main focus is on the healing agents that can be encapsulated in the micro/nano-capsules and micro/nano-fibers or vascular-based networks. It also summarizes the recent developments and applications of self-healing composites in aerospace industry. Subsequently, the review work was carried on core-shell nano-fibers and capsules. In-depth survey was conducted on the testing methods and fabrication techniques that have been utilized in recent years for fabrication and to determining response of self-healing materials to mechanical loads. This review will help the academic researchers to come up with new ideas on high performance materials.

Keywords- Self-healing; Healing agents; Nano-fibers; Micro-capsules, Hollow tubes, Vascular networks

I. INTRODUCTION

Self-healing mechanisms are most fascinating phenomenon occurs in most of the living organisms. For instance, when the skins are scratched or the blood vessels are torn and bleed; living things have the tendency to bind the tissues together. Similarly, the materials can also be fabricated to get self-healing properties **Error! Reference source not found.**.

Damages in material not only collapse the structure but also allay mechanical, thermal and electrical properties. The researchers made various experiments to get back the same properties in the materials **Error! Reference source not found.**.

Composite materials are widespread in various advance applications such as civil, aircraft and ships. The composite material has tremendous strength to weight ratio under normal operating conditions. In case of engineering materials, there are various methods to induce self-healing properties. Self- healing is divided into two categories, (i) Intrinsic healing and (ii) Extrinsic healing. Extrinsic recuperation is the addition of healing agent by disparate methods, whereas intrinsic healing involves a reversible molecular bond chemical reaction (supramolecular chemistry) in the structure of materials. This system does not need external source for healing process, it occurs simultaneously when the catastrophic failure happens. It eliminates the need to perform the temporary repairs on damaged structures **Error! Reference source not found.**.

The primary section of this work explains some basic self-healing processes which include Diels–Alder reaction (intrinsic), capsule-based healing (extrinsic) and vascular networks-based healing (extrinsic).

The second section describes about the various mechanical tests conducted on the composite specimens; ASTM standards used in various test methods are also discussed.

Finally, the last section includes the overall result and healing efficiencies of the tested materials. This work provides knowledge and creates awareness of the importance of self-healing nature in engineering material in young talents and future generations.

II. MATERIALS

Self-healing agents are selectively used according to the healing mechanism and base material. There were two main healing mechanisms used for the composite/nano-composite materials. They are (i) Intrinsic self-healing (ii) Extrinsic self-healing. In these systems, a variety of healing agents were used so the materials meet requirements of an application.

A. INTRINSIC SELF HEALING

Intrinsic mechanism often requires an external reaction to trigger the healing process. The process is depicted in Figure **1**.

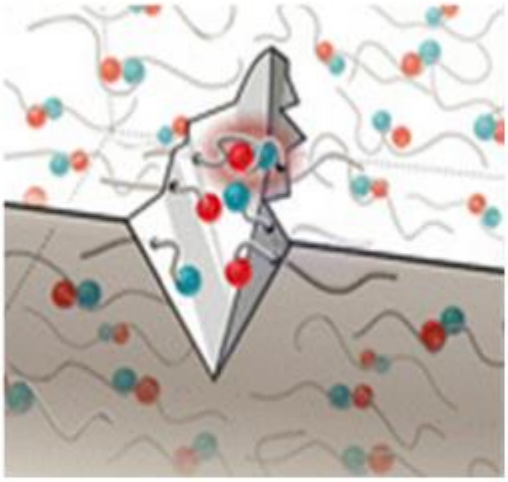

Figure 1.Intrinsic self-healing approach **Error! Reference source not found.**

It comprises of re-formable covalent bonds and dynamic inter-linkages. The most widely used method is the reversible Diels–Alder reaction **Error! Reference source not found.**. Wang et al. **Error! Reference source not found.** presented a novel self-healing material: Ultraviolet (UV) curable polyurethane resin. The synthesis of UV polyurethane resin was processed by Diels-Alder reaction. The uniform film of resin was applied on the glass slide which was cured by UV light. Polymer and graphene based self-healing material prepared via Diels–Alder (D-A) reaction was also investigated by various researchers **Error! Reference source not found. Error! Reference source not found. Error! Reference source not found. Error! Reference source not found.**.

Zhang et al. **Error! Reference source not found.** erected self-healing fiber reinforced plastic (SHFRP) by using intrinsically healable iso cyanurate-oxazolidone (ISOX) thermosetting matrix with carbon fibers. Uni-Directional (UD) carbon fiber/epoxy resin prepreg was used for manufacturing of composite laminate with stacking sequence $[45/\text{-}45/0/90]_{2S}$ and the laminate was modified by placing DA based BMI pp reversible cross linker (BMI prepolymer resin) between the −45° and 0° plies**Error! Reference source not found.**.

In another work, the formation of composite consisted of aromatic polyamide (fPA) with pendent furan groups and fullerene $C_{60}$was done. Diels-Alder (D-A) reaction of furan groups and C_{60} resulted in reformable cross-linking of fPA at normal temperature **Error! Reference source not found.**.

Guadagno et al. **Error! Reference source not found.** produced nano structure of polyhedral oligomericsilsesquioxane (POSS) compound by regenerable hydrogen bonds to start off self-healing mechanisms in thermosetting materials. Four different polyhedral oligomericsilsesquioxane (POSS) compounds were used in the epoxy matrix. It was found that matrix with multi-walled carbon nano-tubes (MWCNT) can enhance healing up to 400%. Similarly, Ren et al. **Error! Reference source not found.** prepared shape memory self-healing composites with polycaprolactone /thermoplastic polyurethane and carbon nano-tubes. Polycaprolactone and polyurethane mixture was utilized as shape memory polymer matrix, polycaprolactone as healing agent and carbon

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nano-tubes as reinforcement webs.

Khan et al. **Error! Reference source not found.** manufactured thermal based self-healing nano-composite by reinforcing graphite nano-platelets in DA reaction-based hybrid polymer matrix and its healing capacity was found to be 87%.

Deng et al. **Error! Reference source not found.** synthesized low temperature self-healing polymer resin via the condensation copolymerization. These polymers have the ability to harden themselves when impact force act on it.

B. EXTRINSIC SELF-HEALING

The extrinsic healing action is based on employment of a healing agent embodied in the matrix which was used as additional constituent in composite. The recuperating agent is generally in the liquid state. Sometimes catalyst that may or may not be encased is also used to speed up recovery process in the material **Error! Reference source not found.**. Extrinsic self-healing has following divisions (i) capsule-based healing (ii) vascular-based healing.

In capsule based healing approach, the material was synthesized by encapsulation of healing agent in the capsule. When damage occurred, capsule broke, the healing agent leaked and swept out to heal the gap in the material.

Kosarli et al. **Error! Reference source not found.** fabricated microcapsules using urea-formaldehyde which formed shell for encapsulation of epoxy healing agents. The system showed 68% of recovery. The effect of capsule size was also determined and it was observed that larger capsule size results in increase in percentage of healing.

Ahangaran et al. **Error! Reference source not found.** developed the self-healing epoxy resin composites using polymethyl methacrylate (PMMA) for microcapsule. They used epoxy and mercaptan as a curing agent which was encapsulated in the PMMA polymer material. 80% of recovery was noticed after keeping the material undisturbed for 24 hours at room temperature.

Ahmed et al.**Error! Reference source not found.** erected dicyclopentadiene (DCPD) capsules by using urea formaldehyde as shell material. The experimental work was compared with the two-dimensional finite element modeling (FEM) result. It was found that size and shell did not affect the modulus of elasticity.

Fan et al. **Error! Reference source not found.** designed the two kinds of microcapsules for encasing polyurethane polymer. Shape memory polyurethane polymer was prepared by solvent polymerization and was covered with microcapsules to reduce corrosion of the material. When heat was introduced to the material, it recovered its permanent shape. Isophoronediisocyanate and polycaprolactone were used as shell for microcapsules. It was discovered that the shape memory polyurethane can recover 75% of its original shape.

In vascular self-healing approach, vascular network was synthesized by encapsulating the curing agent in hollow channel network or core-shell nano-fibers. When any catastrophic failure occurs in material, the nano-fibers rupture and curing agent leaks in the material like blood in the blood vessels.

Muruzabal et al. **Error! Reference source not found.** created a nano-composite by vacuum assisted resin transfer molding (VARTM) and a nano-vascular network with nanofibers. The nanofibers were fabricated using electrospinning method. It was found that the mitigation of mechanical properties was diminished as compared to the microcapsules.

Rasoul et al.**Error! Reference source not found.** used PMMA polymer as shell of the nanofiber. Two types of nanofibers were built by using epoxy resin and curing agent as core. PMMA polymer solution was synthesized by dissolving it in DMF solution and stirring for 24 hours at room temperature. Carbon Fiber Reinforced Polymer (CFRP) laminate was fabricated by hand layup and VARTM technique for uniform resin transfer.

Zanjini et al. **Error! Reference source not found.** introduced tri-axial electro-spinning process which has three polymer solutions for hollow fiber production with self-healing capability. The composite laminate was fabricated with Teflon based mold. FBG sensor was integrated in the composite laminate to check and find out the changes occuring in the material. Usage of glass fiber was done to form the panels by vacuum infusion technique. The nanofibers were placed on the 0^{o} angle ply and bidirectional

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E-glass stitched fabrics were used to laminate three layers of $(90/0)_s$ plies. Lee et al. **Error! Reference source not found.** introduced a new technique called solution blown process similar to electro-spinning process but uses pressure to blow the nanofiber formation. They also prepared two types of nanofibers which have resin and curing agent as core materials. The solution blown process was used for 30 minutes. The processed nanofiber mats with resin and curing agent were cut into required dimensions for mechanical test. The researcher did tensile test to rupture the material and produce crack. It was found that material healed successfully.

Dong et al. **Error! Reference source not found.** developed the polyvinyl alcohol /polyvinylidene fluoride coaxial core-shell nanofiber by electrospinning process and the corrosion inhibitor 2–mercaptobenzothiazole (MBT) was encapsulated in the core shell nanofiber to find out the corrosion resistance of the material. The corrosion effect was investigated using electrochemical techniques. The electrodes were prepared for electrochemical process. It was found that the corrosion effect has decreased in the material.

Wook Lee et al. **Error! Reference source not found.** proposed electro spun core-shell PAN nanofibers containing healing agents for self healing. For creating three- dimensional bulk matrix, the PAN nanofiber mats were chopped and mixed well with the PDMS solution. The solution was left for 24 hours in a vacuum chamber for curing the material. Finally, it was found that the spongy matrix can recover 100% when compression damage occurs.

III. MECHANICAL TEST

Disparate destructive approaches, for instance flexural test, impact test, tensile test, and compression test can be employed to learn about numerous mechanical properties of self-healing composites by creating various damages **Error! Reference source not found.**. The summary of these test used in various research are listed in Table 1.

Table 1. Summary of Mechanical test conducted on composites

MATERIAL	MECHANICAL TEST	ASTM	EQUIPMENT	REF
CFRP	Impact Test	ASTM D7136	Drop-weight impact test tower	**Error! Reference**
GFRP	Impact Test	-	Drop-weight tower	**Error! Reference**
GFRP	Flexural test	ASTM D6272	Instron model 5984	**Error! Reference**
CFRP and GFRP	Tensile test	ASTM D638-03	Universal testing machine	**Error! Reference**
CFRP and GFRP	Flexural test	ASTM D790	Universal testing machine	**Error! Reference**
CFRP and GFRP	Impact test	ASTM D256	Charpy impact	**Error! Reference**

CFRP	Compression test	ASTM D3410 ASTM D695 ASTM D6641	Universal testing machine	**Error! Reference source not found.**
Carbon/epoxy prepreg	Mode I testing	ASTM D5528	Universal Testing Machine	**Error! Reference nce**
Unidirectional glassfibre	Impact test	-	Drop-weight impactor test tower	**Error! Reference nce**
GFRP	Mode I testing	ASTM D5528	Universal Testing Machine	**Error! Reference nce**
GFRP	Flexural test	ASTM D7264	Universal Testing Machine	**Error! Reference nce**
GFRP	Tensile test	ASTM D3039	Servo-hydraulic test machine (Instron Model 8500)	**Error! Reference nce**
GFRP	Flexural test	ASTM 7264	Universal testing machine	**Error! Reference nce**
GFRP	Flexural test	ASTM E976-10	Universal testing machine	**Error! Reference nce**
CFRP	Compression test	ASTM D7137(M)-07	Universal testing machine	**Error! Reference nce source**

A. IMPACT TEST

Nezhad et al. **Error! Reference source not found.** carried out impact test using a drop-weight machine on the carbon fiber-reinforced polymer (CFRP) composite panels keeping the dimension of each specimen based on the ASTM standard D7136. The impactor's weight was kept at 4.2 kg and was capable of giving a force of 22 KN. Testing was done on thermoplastic and thermosetting epoxy materials consisting of 17 plies and 25 plies respectively. First sample was tested at two impact energy levels while the second was tested at three impact energy levels and their impact response was scrutinized. Later, it was found that thermoplastic material can resist impact damage more compared to the thermosetting material. Hart et al.**Error! Reference source not found.** conducted an impact test on a Glass fiber panel that were clamped at a fixed position in the drop-weight tower (Instron model 8250). The specimen dimensions are shown in K. P. Jhanji et al. conducted drop weight impact test on CFRP & GFRP with and without resin and hardener filled microcapsule to show partial healing at 2.5% inclusion of microcapsule.

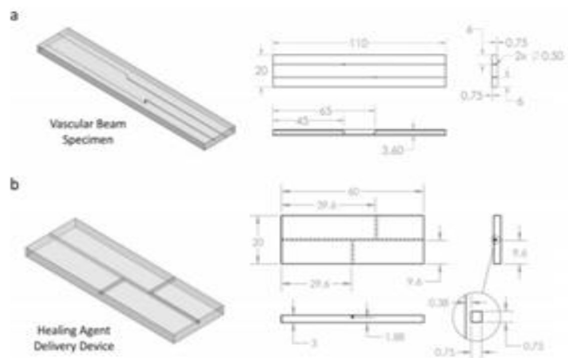

Figure 2. Schematic of vascular composite components for impact test **Error! Reference**

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. During the impact, 10 J and 15 J of impact energy delivered on the vascular based self-healing composite. It was observed that the material was able to recover its strength up to 47% after 48 hours of healing at room temperature. K. P. Jhanji et al. conducted drop weight impact test on CFRP & GFRP with and without resin and hardener filled microcapsule to show partial healing at 2.5% inclusion of microcapsule.

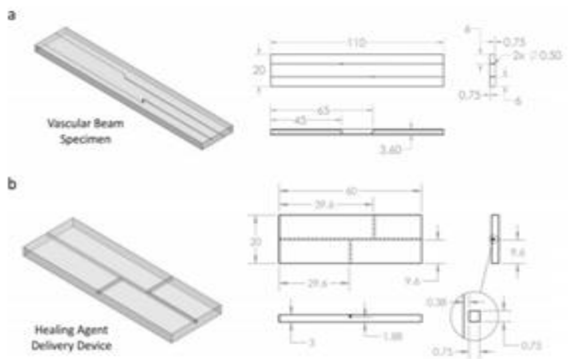

Figure 2. Schematic of vascular composite components for impact test **Error! Reference source not found.**

K Poyyathappan et al. **Error! Reference source not found.** found the maximum impact strength of 9.4 J on C-GFRP when a impact test was conducted by using a Charpy impact instrument on two different types of fiber reinforced panels which are C-GFRP (Carbon-Glass) and G-CFRP (Glass-Carbon) having a notch with the dimensions according to ASTM D256 standard.

B. COMPRESSION TESTING

K. Goto et al. **Error! Reference source not found.** used an INSTRON 5985 universal material testing apparatus of 250 kN for uniaxial compressive test as shown in Figure 3.

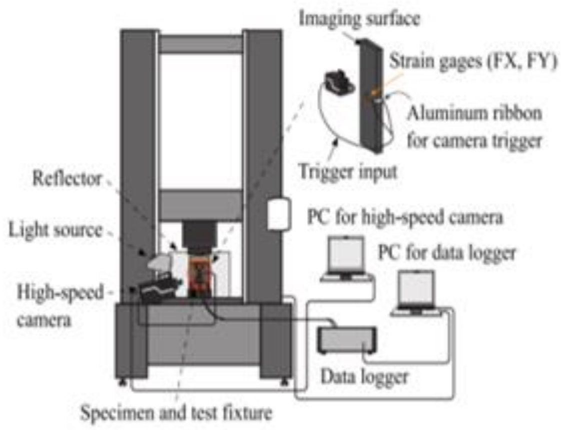

Figure 3. Compressive test system **Error! Reference source not found.**

Cross head travel was maintained at 1mm/min and the test fixture was made based on JISK7076 standards shown in Figure 4
.

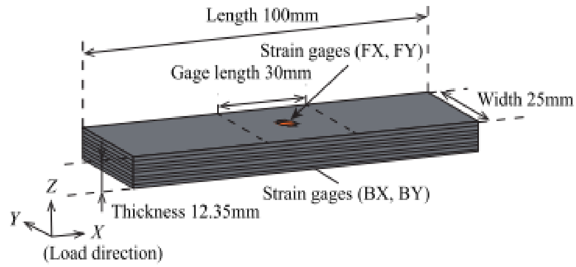

Figure 4 Compressive Test dimensions [33]

S. Manteghi, et al. **Error! Reference source not found.** presented a composite material with glass fibers and flax fibers as reinforcement and epoxy as matrix whose compressive properties were tested using universal testing apparatus .Three samples with two distinct configurations were tested according to ASTM D695 standard. The fatigue strength was found to be 53% of ultimate compressive strength.

C. TENSILE TEST

Tensile test for self-healing-based nano fibers composite materials are widely seen in many researches works. Different standards were used to conduct the test and the effect of self-healing was studied.

K. Molnár et al. **Error! Reference source not found.** performed tensile test on the composite with PAN-based carbon nano fibrous mat produced by electro spinning process with multi walled carbon nanotubes (MWCNT) dispersed in matrix. The test was conducted according to international standard EN ISO 527-2 using ZwickZ005 tensile tester. The thermal properties of these composite were noted to be twice than the normal composite and a improvement in mechanical properties were observed due to inclusion of CNT.

Dmitriy et al. **Error! Reference source not found.** conducted tensile tests on glass fiber with epoxy samples with ply orientation of [0º / 30º / 0º / 60º] by Instron 5882 universal testing machine.. The result showed that increase in temperature exposure of material, improved the strength of the composite material. Lee MW, et al **Error! Reference source not found.** disclosed that the application of tensile load damaged the nano fiber which resulted in leakage of curing agent leading to healing of the crack formed during testing. R. E Neisiany et al. **Error! Reference source not found.** found that the usage PAN based nanofiber self-healing carbon fiber compositeresulted in improvement of the tensile strength by maximum of 19%.

D. FLEXURAL TEST

C. Jie et al. **Error! Reference source not found.** conducted flexural test on the Al2O3f/SiO2 composites which resulted in a flexural strength of 55.6 MPa which indicated the reduction on mechanical properties.

Poyyathappan et al. **Error! Reference source not found.** carried out flexural test on the combination of glass fiber and carbon fiber. CFRP, GFRP, G-CFRP, C-GFRP and G-C-G-CFRP combination of specimens were used for flexural test that resulted in observation that CFRP can resist fracture damage with highest load of 2 KN than other composite materials.

E. DOUBLE-CANTILEVER BEAM (DCB) TEST

H. Saghafi et al. **Error! Reference source not found.** conducted mode-I fracture load (DCB) test on the carbon fiber prepregs with the addition of polyvinylidenefluoride (PVDF) nanofibers prepared by electrospinning process. The result revealed that the fracture toughness increases by 36% with the addition of PVDF nanofibers. Yademellat H et al. **Error! Reference source not found.** were conducted the DCB and ENF test on the electro spun nylon-based nanofiber composite laminates. Composite laminate fracture damages were simulated using FEM along with the experiment. On comparison it was revealed that the nanofibrous mat incorporated in the laminate reduce the fracture damage produced by the load testing. The size of the delamination and fracture also decreases. D. An et al. **Error! Reference source not found.** tested for the mode I fracture toughness on a glass fiber reinforced composite modified with nylon-based nanofiber fabricated by electrospinning and vacuum bagging technique. The result revealed that as the density of nanofiber increase, the material fracture toughness reduces.

IV. CHARACTERIZATION

A. Fourier transform infrared spectroscopy (FTIR)

FTIR spectroscopy is a technique that is used time and again to find the chemical composition of a material. A couple of radiation gets absorbed onto the material when the instrument sends infrared radiation towards the material. The composition, contamination of the material can be established with the help of absorption band level.

M.M. Sander et al. **Error! Reference source not found.** characterized the cross linked polymer composites PGS and PPy. Fourier transform infrared spectroscopy was used to determine the chemical bond between these two polymers and it was found that it form ester bond formation.

Gao and his peers **Error! Reference source not found.** used FTIR Spectrometer for testing the sandwich structure material which consist of carbon nanotubes as core layer and poly ethylene-co-methacrylic acid (EMAA) as outer layer. EMAA polymer was characterized to find the chemical reaction stability and it was revealed that pure EMAA consist of carboxyl group which increases the strength of material.

Doan T. Q. et al. **Error! Reference source not found.** used FTIR for chemical analysis of damaged area of steel that is coated with the nano fibers of polydimethylsiloxane and polydiethoxysiloxane fabricated by

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electrospinning process. The result showed that the healed region consists of various chemical reaction which changes the cross linking density. W. Zhang et al. **Error! Reference source not found.** fabricated the electro spun lignin/poly vinyl alcohol nanofibers to study the interaction between the lignin and PVA. Fourier transform infrared spectra determined the composition which showed that the hydroxyl group between lignin and PVA nanofibers formed strong hydrogen bond. Shalaby and his peers [45] used FTIR to show that the addition of antibacterial agents like as silver, zinc and copper that are added while preparing PAN based nanofiber, preserves and protect the PAN structures by forming chemical bonds. De Souza et al. **Error! Reference source not found.** confirmed that Diisocyanate microencapsulation contained TDI as a core agent, before and after trituration by comparing the FTIR spectra of microcapsule.

B. NEAR-INFRARED SPECTROSCOPY (NIR)

NIR spectroscopy is similar to FTIR method. It can identify many chemical compositions and its properties.

Y. Gao, et al.**Error! Reference source not found.** used this near-infrared spectrum (NIR) in the PMMA with carbon nanotubes on a glass fiber laminate to show the conversion of secondary amine group to tertiary amine group in the curing process in a self healing curing agent.

C. THERMOGRAVIMETRIC ANALYZER (TGA)

TGA is a thermal analysis process which computes the variation in thermal properties as a function of time.

Sander et al. **Error! Reference source not found.** concluded that the composite was stable up to 400 ^{o}C when the change in thermal properties of cross linked polymer composites PGS and PPy was measured by using Thermogravimetric analyzer. De Souza et al. determined the temperature degradation of each core material and each microcapsule.

C. Liu et al. **Error! Reference source not found.** developed graphene epoxy self-healing nanocomposite. The measurement of ethylenediamine-β-cyclodextrin (EDA-CD) on graphene surface was carried out on a Q500 Thermogravimetric Analyzer.

D. SCANNING ELECTRON MICROSCOPY (SEM)

SEM is a type of microscope that is used by various researchers to search evidence of healing in the materials.

Dai et al. **Error! Reference source not found.** used field emission scanning electron microscope (SU-8010) on the electromagnetic shielding composites with self-healing ability. It was revealed that the material healed fully after 24 hours. Similarly, the polymerization reaction of CFRP and GFRP were noticed by SEM investigations **Error! Reference source not found. Error! Reference source not found.**. Sander and Ferreira **Error! Reference source not found.** performed scanning electron microscopy (SEM) on the cross linked polymer composites poly glycerolsebacate (PGS) and polypyrrole (PPy). The samples were coated with gold to make conductive material. SEM images showed the cross section of the fractured area in polymer composite with different concentrations.

Doan et al. **Error! Reference source not found.** performed SEM on the electro spun PDMS nanofiber coating. It showed that fibers were distributed non-uniformly on the steel material. Shalaby et al. **Error! Reference source not found.** observed the morphology of the electro spun PAN nanofibers that consist of Silver, Zinc and Copper nanoparticles using a SEM (JSM-6360). The average diameter of nanoparticles was also found, it was 250 nm.

E. ACOUSTIC EMISSION

Acoustic emission is one of the non-destructive testing techniques used in many applications to detect damages in the material.

Gholizadeh et al. **Error! Reference source not found.** inspected the glass/epoxy composite laminate which was experiencing stress concentration due to the hole created for bolts and rivets. The stress concentration was reduced by placing nylon based nanofiber in the mid layer of the laminate. To analyse the damages,

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three Acoustic Emission (AE) analyses were used. The conventional method recorded only one type of damage (crack, breakage, debonding) but Wavelet Packet Transform (WPT) AE recorded all damages together. Sentry function was used to examine both mechanical and acoustic results. The results showed that damage was reduced by 51% with addition of nanofibers.

F. ULTRASONIC

Ultrasonic is another non-destructive testing technique which is very effective. This technique is widely used in almost all the areas. Post et al. **Error! Reference source not found.** monitored healing of delamination defect in CFRP. It was analyzed by two types of ultrasonic C-scanning i.e. water coupled and air coupled ultrasonic tests. The measuring time for water coupled ultrasonic test was 30 minutes and air coupled ultrasonic test was 1 hour. Both C scanning techniques were performed during different time interval of healing. From the results, it was evidenced that the material almost fully healed after 40 hours at 100°C.

V. APPLICATIONS

The heavily weighed metals are replaced by light composite materials in several advanced industries such as civil, aircraft, automobile, ships and submarines. The improvement in recent composite materials is self-healing technology which has been developing for past 20 years of research. The self-healing materials reduce repairing cost and maintenance time. The self-recovering composites can be used in distinct areas of aerospace industry as delineated in Figure 5.

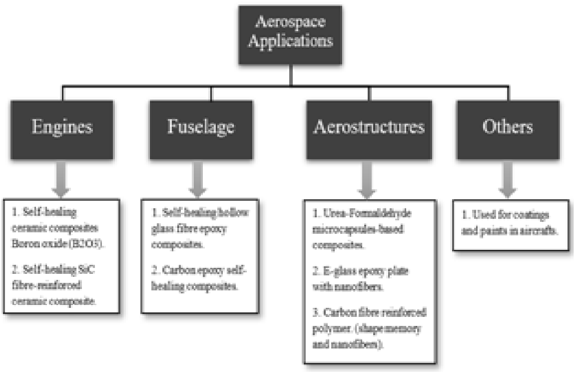

Figure 2 Aerospace Applications ***Error! Reference source not found.***

VI. CONCLUSION

The present work provided idea and knowledge on the trends and developments in self-healing composites since 2016. Various types of methods for inculcation of self –healing in composites were used. These methods includes replacement of old resin with new resin capable of regenerating its polymer chains, inclusion of vessels like capsules, hollow fibers and tubes with the capacity of storing and delivering healing agents. Various methods like electrospinning, in-situ polymerization were followed for fabrication of different self-healing mechanisms. A deep review was also conducted on various mechanical tests performed on the self-healing materials in recent years. Tests such as low-velocity impact, compression, tension and flexural were widely performed and self healing was measured in terms of restoration of mechanical properties during the tests. Self-healing was corroborated by various methods which includes Fourier transform infrared spectroscopy (FTIR), Near-infrared spectroscopy (NIR), Thermo-gravimetric analysis (TGA), Scanning Electron Microscopy (SEM), Acoustic Emission (AE) and Ultrasonic C-scan testing. Applications of self-healing materials in Aerospace Industry were also discussed.

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