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Self Healing Polymers: Its Properties and Applications

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Abstract

Stimulated by biological systems, self-healing polymers proved its materiality in many Industries especially in the aviation sector. As these materials require less protection from indentation and provide better mechanical properties such as high fracture strength, high elastic modulus and ability to mend the crack, at the scale of micrometer make them promising material to avoid unexpected catastrophic failure. This paper reviews the different recuperating mechanisms within self-healing polymers including a brief discussion on composites. Consequences of self-healing on various mechanical properties such as fracture, fatigue, impact, tensile strength, elastic modulus, compression have been examined. Also, applications of self-healing material, especially in the aviation sector and its future prospects has been discussed.

Keywords: Self-healing polymers, Extrinsic and Intrinsic healing, Mechanical properties of self-healing material, Application of self-healing material

1. Introduction

In the past two decades, polymer composites have accelerated the evolution of different technologies namely aerospace, energy storage devices and sports goods have significant use of them. Thus, it is inevitable to make these materials more durable and safer, by reducing their failure modes such as nano scale cracks, crack propagation and fracture damage. These cracks are mainly developed at the time of manufacturing or servicing, and it is nearly impossible to mend them manually. Here, nature inspired self-healing mechanism plays a vital role to patch the damage and retard fracture propagation and increase the lifespan, efficiency and sustainability of a material. (Dry, 1996)

Composites provide some unique benefits over metal alloys, wood and concrete due to their characteristics such as corrosion resistance, fire resistance, etc. Composites are a hybrid of two or more insoluble constituents namely reinforcement and matrix. Matrix is the continuous phase of composites which surround the fiber while the reinforcement is the discontinuous phase that provides strength and stiffness to composites. On the basis of matrix material, they are classified into three types namely, metal, ceramics and polymeric composites (Hsissou et al., 2021). In this review paper we are only concerned with polymer based composite materials.

As the name implies polymer composites are the plastic in which fiber such as glass, carbon, aramid and Boron are embedded. These are further classified into two categories thermoplastic Matrix based and thermosetting matrix-

based polymer composites. Thermoplastics are linear or slightly branched long chain molecules that soften when heated due to weakening of secondary bond after glass transition temperature Tg and gain original strength when cooled due to reduction of molecular motion. On increasing temperature, their viscosity and elastic modulus decreases significantly. They offer high strength, shrink resistance, easy bendability and thermal and electrical insulation. According to type of resins, they can be used in low stress applications such as carry bags or highly stressed mechanical parts. Polymer composite thermoplastic Matrix materials includes: PPS, PEEK (Serope Kalpakjian et al.,**Manufacturing Engineering and technology**, 6th ed. Pearson Prentice Hall, 2010)

Thermosets are covalently cross-linked or heavily branch network polymers. Curing in a thermoset is Irreversible because the covalent bond anchors the change together to resist any kind of motion at high temperature. These are generally harder and stronger and have better mechanical, thermal properties and dynamic stability than thermoplastics. Common polymer composite thermosetting matrix materials include: Epoxies, Phenolic, Polyester, Polyimides.

Traditionally the damage was treated by the simplest external patches, tapered scarf or stepped-in repair mechanism. In most of them we establish the outer plaster to outstay energy and cease crack propagation, but this method ultimately decreases mechanical strength of material. Self-healing could be considered a better substitute that can heal cracks of micro level without any significant loss of mechanical power. (Hayes et al., 2007)

2. Self-Healing Conception

From an early-stage different traditional mending techniques of polymeric material were in existence. Mostly these techniques use a smearing of reinforcement over the surface to absorb shocks and stop further crack propagation. These approaches are effective in recovering surface damages and can mend only visible cracks. Furthermore, the expensive nature of these techniques and adverse impact on mechanical properties of polymers created demand for a more effective technique (Bond et al., 2008). Thus, the evolution of the self-healing concept in polymers, drives a new era of recuperating methodologies. The fascinating feature of self-healing is that it is not just clogging the gaps and hardened fracture in materials, but also restoring the mechanical, chemical, electrical and other functional properties of the materials. Self-healing materials retain their functional properties by mending micro-level cracks, either by external additives such as microcapsules or hollow fibers or by internal reversible bonding (Blaiszik et al., 2010). Today, many prestigious organizations such as NASA are researching self-healing materials to increase the Technology Readiness Level (TRL) of thermal protection system applications to use in space environments (NASA). Still, it is an evolving field so a limited number of commercial applications exist like Arkema's Reverlink™. (Brás et al., 2012) Undoubtedly this revolutionary concept has potential to influence numerous industries such as aerospace, sports, atomic power, automobile, bioengineering, construction, electronics and communication, marine technology and space exploration etc.

3. Classification of self-healing mechanism

Broadly polymeric material can heal in two fashions; some materials have movable phase that can patch the damage by flowing over the cracks and solidify again, consequently the material properties restore while others have reversibility of their primary covalent bond to regain their initial properties (Hager et al., 2020). Generally, there are two strategies for self-healing operation: extrinsic and intrinsic. Extrinsic self-healing approach consists of nano or microcapsules embedded in a polymer matrix as a separate phase. The matrix also has uniformly distributed catalysts either in the form of capsule or dissolved directly that combine with healing agents to form a solid product (Garcia, 2014). Conversely intrinsic self-healing materials are not required to have any additive and involve the supramolecular chemistry to retain their properties (Yang and Urban, 2013). The other type of classification is based on autonomic healing which requires no external trigger to initiate and non-autonomic healing in which external stimuli such as pressure and heat etc. must be applied to initiate the healing function (Blaiszik et al., 2010).

3.1 Extrinsic healing

3.1.1 Microencapsulation

Microcapsules can be considered as tiny glue packets spilled throughout the polymer matrix. When the material gets mechanically damaged these microcapsules bleeded the healing agent to patch the crack and stop its propagation. These microcapsules are mostly made of Urea formaldehyde, triethylenetetramine (TETA) and poly (methyl methacrylate) (PMMA) (Blaiszik et al., 2008; Li et al., 2013). These microcapsules are prepared by processes such as meltable dispersion, in situ and interfacial encapsulation. Mostly epoxies are used as healing agents due to its useful adhesive properties and also catalysts are used as polymerizer which reacts with a healing agent and makes a solid product. Sometime Matrix itself provides the functionality of a catalyst under external incitement. Also, the catalyst can be embedded in different capsules like a healing agent, or both can be placed in the matrix as a separate phase (Das et al., 2016). Generally, microcapsules are of micro level but Blaiszik et al. prepared nano capsules by ultrasonication technique over Urea Formaldehyde capsule shells having DCPD as healing agent. Further these nano capsules are stabilized by ultra hydrophobes such as hexadecane and octane that makes it possible to achieve nanoscale (Blaiszik et al., 2008). However, this method has its own limitations such as microcapsules must survive during large scale manufacturing that is a significant constraint on the design of capsule packets. Although repeated healing is possible, it is impossible to determine when the whole healing agent has been consumed (Hayes et al., 2007).

Nevertheless, it also gave birth to innovative approaches of self-healing such as **remote self-healing** which uses the fact that molten polymers manifest more interfacial diffusion. If this diffusion can be induced locally in a damaged area, it will aid self-healing. This can be achieved by super magnetic nanoparticles rapidly oscillating in a magnetic field generating localized heat, melting itself and repairing a polymer material matrix (Yang and Urban, 2013). The general method of encapsulation is shown in figure 1. Ultimately microcapsules are additives that influence Mechanical properties of polymers significantly.

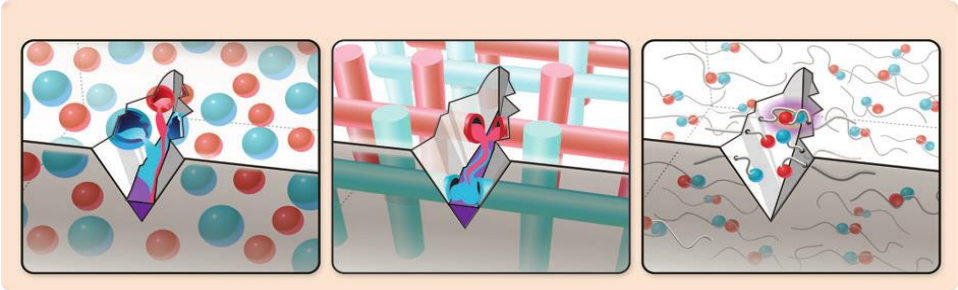
3.1.2 Mesoporous Network

It can be partitioned in a manner similar to encapsulation but consists of hollow fiber or mesoporous network filled with healing chemicals. These hollow channels are incorporated at the time of manufacturing by replacing some reinforcement fiber. Mesoporous networks enjoy several advantages over encapsulation such as a wide range of remote damage sites can be healed effectively due to ultra-high uniformity.

Unlike capsule based systems, here, healing chemicals are impregnated after embedment of network into matrix by capillary action therefore, the healing chemical should have favorable wetting and chemical properties to ensure proper stability of the system (Blaiszik et al., 2010) Self-healing, microvascular DCDC (Double Cleavage Drilled Compression) samples were manufactured by (Hamilton et al., 2010) in an epoxy matrix with channels 200 μm in diameter. (EponTM 8132) was injected as epoxy resin and (EpikureTM 3046) as aliphatic amidoamine hardener into alternating layers of vascular network.

There are three assembling methods of network structure for self-healing by using vascular networks. 1D, 2D and 3D. Dry and Sottos initially analyzed the recuperating functionality of the 1D system using glass pippete of mm diameter embedded in epoxy resin (Dry and Sottos, 1993). The 2D network has been developed by Williams et al. by assembling a hierarchical network using PVC tubes (Williams et al. 2007). The 3D system is a replica of the circulatory system of the living body and highly efficient and provides a long lifespan to composites (Aragón et al., 2008). Eventual blockage of microchannels caused by continuous buildup of healed material after repeated healing

is the crucial factor observed that limits the functionality of the network. Vascular network measurably influences the mechanical properties of composite that is discussed in later sections.



(*a*) capsule-based (*b*) vascular method (*c*) intrinsic methods.

Figure 1 : (Blaiszik et al., 2010).

3.2 Intrinsic healing

3.2.1 Diffused Thermoplastic Additives

This approach relies on modification of the polymer matrix system. Based on modified traditional thermosetting matrix, which can reform on heating, a new alternative technology has been developed in which thermoplastics are dissolved to aid the self-healing in thermoset resin. (Chen et al., 2003) The moment the physical state of resin has been altered by cracks or scratches, the dissolved thermoplastic material gets into action and diffuses through the thermoset matrix to stitch the cracks by chain bridging and execute self-healing upon heating and applying pressure. S.A. Hayes researched this field in detail. He added poly(bisphenol-A-co-epichlorohydrin) as thermoplastic as selected thermoset resin in three proportions. 5wt %, 10wt % and 20wt % and due to correlation in between viscosity and healing agent 10wt% mixture was preferred as it provides acceptable healing (Hayes et al., 2007). It might be a better alternative to overcome the limitation of capsule based healing, but implementation of temperature and pressure limit its own applications.

3.2.2 Reversible Chemical Reaction

This new class of self-healing involves such cross linked polymers which are capable of healing internal cracks by reversible covalent bond mostly triggered by heat aur photo irradiation. There is no remarkable difference in mechanical properties of these materials to epoxy resin. Therefore, these can be used for a structural application. Although reversible covalent bonds knock out the need of capsule packet and catalyst but now heat is essential to trigger and assist the healing process. In 2004 Chen et al. evolved a transparent, repeatedly remendable organic material and studied it using NMR spectroscopy and SEM. It was found that upon heating above 120° Celsius monomer linkage break and remend themselves again on cooling and restore cracked area.

Chen et al. conducted a detailed study on thermal reversibility of Diels-Alders (DA) such as 2ME4F and 2MEP4F highly cross-linked polymers using solid state NMR. Mechanism of the Diels-Alder (DA) reaction for highly cross-linked polymers is shown in figure 2. It is very well known that disulfide groups dissociate into two thiol groups by a reduction reaction and can re-associate by an Oxidation reaction. This fact was used to achieve self-Healing by introducing disulfide groups in a rubber network which can remend the material at moderate temperature (Candell et al., 2011). The governing exchange reaction of the disulfide group leads the cross links across the damaged surface. Reversible acyl hydrazone bond based covalent dynamic gels are another example of reversible reaction studied by Deng. (Deng et al., 2010)

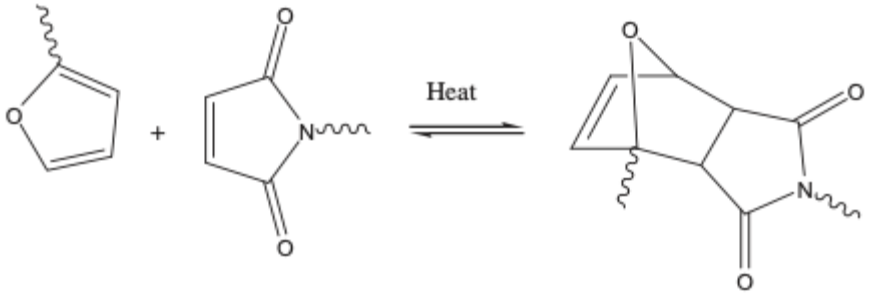


Figure 2 : Diels-Alder (DA) reaction mechanism (Park et al. 2010)

3.2.3 Ionic Interaction

While the presence of reversible covalent bond is one aspect of self-healing another aspect is reversible ionic interaction. This approach incorporates the use of ionomeric copolymers with ionic segments that can form reversible link clusters activated under elevated temperature or UV radiation. For example, projectile puncture test performed by Kalista et al. in 2007 on poly (ethylene-co-methacrylic acid) (EMAA) occurs in two steps: in first step the heat produced during projectile impact testing is absorbed by polymer surrounding that create a local mobile phase, in second step the mobile interface fused together to heal the damage. In 2013 a comparative study performed by Francesconi et al. of self-healing ionomer to aluminum alloy bumper. The impact behavior of EMAA copolymer with acid group neutralized with Sodium ion was examined and it was found an increment in self-healing nature with increasing impact speed that make it suitable to be included as inner layer of composites shield in spacecraft for debris protection.

3.2.4 Supramolecular Chemistry

Unlike covalent bonds the features of supramolecular interaction namely rapid reversibility, directionality and sensitivity make it unique among all self-healing approaches. Like other intrinsic approaches this also required an external stimulus such as heat, pressure and light etc. to trigger self-healing. Basically, these are electrostatic interactions including hydrogen bonding, **π-π** stacking interactions and metal legend coordination (Yang and Urban, 2013).

In 2008, Cordier et al. discussed the design and synthesis of thermoreversible rubber associated together to form cross linked via hydrogen bond. Fatty dimer or trimer carboxylic acid were used as a starting material to avoid crystallization and enhance glass behavior. Undoubtedly more time provides better healing, but a 15-minute healed sample can be deformed up to 200% without breaking. Like other intrinsic procedures, nearly all reversible supramolecular interactions need an external provocation to activate healing mechanisms, like pressure, radiation or heat.

4. Mechanical Properties of Self-Healing Polymers

Effectively, the presence of healing agents in a polymer can increase the resistance of the polymer to fracture propagation. When the crack due to fracture commences propagation, the healing agent will heal and repair the area of the crack, thus increasing the overall lifespan of the polymer. In 2011 JinMiller et al. investigated the fracture and fatigue response when a healing agent was infused within the thin epoxy film generally used in the aerospace industry. In the crack propagation stage of fracture, it was found that the crack healing efficiency increased by 51% when the healing agent was added. Subsequently, this also resulted in an increase in the fatigue life of the film.

In 2002 Brown et al. examined the effect of addition of catalyst towards self-healing and its subsequent relation to the fracture toughness properties of the self-healing polymer. The maximum crack healing efficiency was obtained when the size of the catalyst particles ranged from 180 μm to 355 μm. If the concentration of catalyst in the self-healing polymer exceeded 2.5% the fracture toughness began to decrease. Beyond a 3% concentration of catalyst the fracture toughness decreased massively. It was also determined that the addition of microcapsules up to a 15% concentration resulted in an increased fracture toughness.

In the case of PDMS (polydimethylsiloxane) composite, it was established that when self-healing PRC (Polyacrylonitrile Resin Cure) fibers were impregnated into the composite the young's modulus value increased by a factor of 1.4 on tensile testing (4 cycles) when compared to its initial value. It can thus be inferred that the addition of a healing agent resulted in increase in stiffness in the material and thus fortified the composite post healing. In comparison the original PDMS composite without the healing agent (PRC) experienced a waning in its stiffness properties on completion of the tensile test. (MinWook et al., 2015).

For composites with an implanted microvascular network as the healing agent, it was discovered that the mechanical properties degraded when the network was introduced. The tensile strength of the composite with microvascular network was found to have degraded by 38% and its compressive strength by 13-70% depending on the volume percentage of the microvascular network when compared to a composite without the network. On subjecting the composite to a low velocity impact test, it was found that within 7 days the healing efficiency reached 89%. For a creep test (at 12.3 MPa constant stress and 90° C), the healing efficiency reached 83% in 7 days. (Eslami-Farsani et al., 2019).

Self-healing polymers, while exhibiting a small degradation in mechanical properties in most of the cases (in certain rare cases it may improve them), increases the overall life period of the composite and thus can be implemented in most applications where long life and low weight is of paramount importance.

5. Applications of self-healing polymer in aerospace Technology

The introduction of polymer composite in the aeronautic industry is primarily concerned about weight reduction (Kesearvani, 2017; Mangalgiri, 1999) and later interested in better design flexibility, easy production, no corrosion and ease of processing. Weight reduction is directly associated with lower fuel consumption thereby less environmental pollution and lower manufacturing cost (Mohan et al., 2017; Ghori et al., 2018). One of the major issues that material scientists are facing is how to incorporate the self-healing mechanism concept in the aviation sector into action. This problem can be ruled out by introducing the epoxy resin composites to fabricate giant components in the aviation industry that are able to sustain aerodynamic loads (Guadagno et al. 2010).
The operating temperature range of space vehicles or satellites is drastic; it varies from -150°C to 150° C (Toohey et al. 2009). It is critical that the self-healing mechanism be initiated in this extreme fluctuation of temperature range. For this reason the fundamental requirement of the effective aerospace self-healing composite has to satisfy the physical and chemical stability and high healing efficiency at both higher and lower temperature (Dry et al., 1997; Brown et al., 2002; Guadagno et al. 2011). In 2008 Wilson et al. have worked on the thermal stability of different catalysts such as Grubbs' first-generation catalyst (G_1), second-generation Grubbs' catalyst (G_2) Hoveyda–Grubbs' second generation catalyst (HG_2) with various monomeric healing agents in variety of epoxy matrices. It is observed that epoxy systems containing HG_2 catalysts have exceptional stability at 180 °C with 57% of retention of healing performance (Guadagno et al. 2011). In 2007 Merely et al. proposed a patent to use 5-ethylidene-2-norbornene (ENB) as a healing agent that functions at low temperature. To account for this, Guadagno et al. have examined the ring opening metathesis polymerization (ROMP) of G_1, G_2, HG_2 catalysts embedded in ENB healing agents in 2009. The obtained results reported in Literature (Guadagno et al. 2011) says that a system containing HG1 catalyst with ENB

as healing agent the metathesis reaction is active even at -50°C. These results contribute to the foundation for advanced self-healing mechanisms in aerospace components. Recently Yuye Zhu et al. synthesized a UV responsive self-healing microcapsule so that UV radiation in space is transferred into a favorable one. (Zhu et al., 2019)

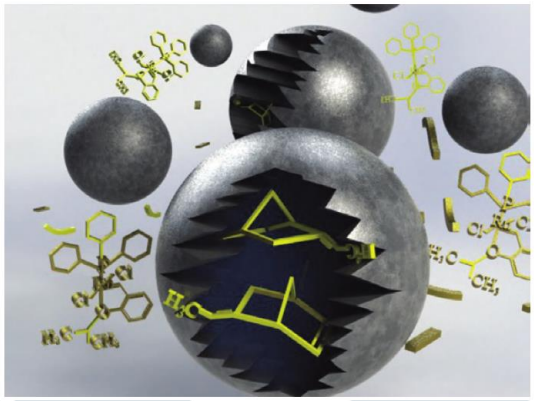


Figure 3 : Schematic representation of self-healing formulation.

5.1 Other applications

- Nowadays self-healing polymers are extensively used in the biomedical field mainly in tissue engineering. Self-healing hydrogels have a wide range of applications in tissue engineering especially as an artificial bone, artificial teeth and regeneration of cartilage, central nervous system and skin (Audelo et al., 2021).
- Soft robots are vulnerable to cuts, tears and different kinds of damages caused by sharp objects, tendon cut, overloading, fatigue and interfacial debonding. All these effects collectively curb the efficient use of soft robots. Introducing self-healing elastomers in soft robotics find solutions to the vulnerability of the soft stem system and also the self-healing mechanism considerably improves the economic crisis in robotic systems by weight reduction and reducing maintenance cost (Terryn et al., 2021).
- After applying excess force to the self-healing rubber rather than cracking, the material incorporated reversible polymer bonds that will return to their original form when the stress is removed. A recent discovery finds application of self-healing rubber in the toy industry as they promote the strategy to reuse and reduce the waste (Utrera-Barrios et al., 2021)..
- Application of self-healing material finds a solution to a smashed screen. Some LG phones like G flex already include self-healing materials on its back cover that can heal scratches. There are certain drawbacks in potential application (Terryn et al. 2021).
- In 2001 automobile industry witnessed a self-healing polymeric paint that can withstand minor scratches and resists corrosion this simple solution has the potential to extend the use life of vehicles. Nissan Motor Co. Ltd came with the world's first self-healing paint coat for car surfaces (Li. et al., 2009) "scratch shields" is the trademark of this product.

6. Future Prospects

Undoubtedly, the self-healing concept is escalating mankind towards science fiction inspired futuristic world. Researchers have already developed such techniques of self-healing which are able to restore not only mechanical properties, but also chemical, electrical and optical properties of material. In the past few years some unimaginable achievement has been noticed in this domain. Conductive self-healing material has been researched by Tan et al. in 2018 for electronics and bioelectronics systems. These materials are developed either by using conducting polymers having reversible bonds or making the self-healing material conductive by using Fillers. Also, NASA reported a novel polyimide film matrix-based self-healing wiring system for orbiters. Some material having healing time as low as 20 seconds has been reported so far, at ambient temperature leaving no scars behind (NASA). It can be envisaged that bio engineering and artificial intelligence (AI) will play a crucial role to lead a wave of further growth of self-healing technology.

By noticing rapid development, it can be anticipated that in near future self-healing material will rule every sphere of life and will be capable of working not only in ambient conditions that will be beneficial for normal consumers, but also in harsh environments such as deep ocean and cosmic space under extreme temperature and pressure that will assist scientists to explore untouched area of earth and space.

7. Conclusion

Advances of the past few decades in the field of self-healing material can pave the way to several industrial or non-industrial applications. The objective of this review paper was to discuss different mechanisms of self-healing such as microencapsulation, vascular network, dissolved thermoplastic, reversible interaction and others which have been developed so far. These materials have attributes to recover not only mechanical properties, but also optical and electrical properties that make them useful for many industries specifically for the Aerospace sector. In summary, Self-healing materials have a very glittering future ahead that will make mankind capable of exploring unseen spheres of universe.

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