

# MULTIFUNCTIONAL SELF-HEALING AND MORPHING COMPOSITES

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## ABSTRACT

Highly innovative work towards development of a new class of materials called “Multifunctional Composites (MCs)” for multi-role structural aircraft skin applications possessing both self-healing and morphing functionality has been achieved. Proof of concept was demonstrated showing that a low volume-fraction (5-10%) of magnetic particles is sufficient for enabling self-healing of an approximate 150 micron x 5000 micron crack in a mendomer polymer using inductive heating. It was also demonstrated that a carbon-fiber-composites can be fabricated to morph using an apparent shape memory effect of the same mendomer that was used to demonstrate the self-healing. Studies of particle composition and mendomer were performed to determine the relative optimal material components for self-healing and morphing functionality. Department of Defense applications of the technology were articulated in collaboration with a major ballistic missile defense integrator. Future work is also briefly discussed.

## 1. Introduction to MCs

In this paper we investigate a novel combination of load carrying, morphing and self-healing composite structures for missile interceptor applications. The fabricated structures combine conventional carbon fiber-composite technology with magnetic-particle composites to form a smart material system that will both morph and self-heal. These material systems can confer multifunctional benefits such as self-healing of microcracks and delamination to large area shape optimization. Obvious advantages are to vehicular structures where self-healing of otherwise potentially catastrophic damage can save not only in expensive equipment, but also in lives. Where minor shape changes can improve vehicle performance, fuel economy is an additional benefit. In the following subsections we

first introduce self-healing materials following by a brief description of morphing aircraft. Finally the concept of a multifunctional composite is described which merges the capabilities of self-healing and induced deformation.

### 1.1 Self-healing material systems

Self-healing can be loosely defined as the ability of a material to self-repair from damages inflicted on it. This often refers to the ability of a material to heal small cracks autonomously--though large cracks, bullet holes, and even cleaved surfaces have been shown to heal due to the energy associated with the damage event or through manual intervention and application of heat to stimulate the mechanism of self-healing. “Self-repair” is often used synonymously with self-healing.

In their pioneering work, White and his associates [Dry and Sottos, 1993; White et al., 2001] reported the development of a polymeric material with such a healing property. The autonomous crack healing is accomplished by dispersing microspheres containing a healing chemical called dicyclopentadiene (DCPD) and a polymerizing agent known as Grubbs’ catalyst. When a crack is initiated in the material, the high stresses associated with it cause the nearest microspheres to break, releasing the chemical, which after interacting with the catalyst, initiates a chemical polymerization reaction of the DCPD that heals the crack. Similarly fibers storing healing resin have also demonstrated where, when fractured, the resin flows into the damage sites within the structure [Pang et al., 2005]. A second family of polymers (polystyrene, poly-vinyl ester) has been developed with much more diminished crack healing properties [Raghavan and Wool, 1999].

Since these materials are at a very early stage of their development, they suffer from a number of deficiencies. The catalyst and the healing agent degrade at high temperatures and the chemical reaction time diminishes

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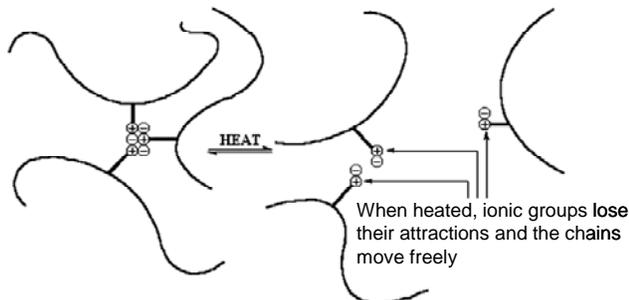
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at very low temperatures. The material reported in [Dry and Sottos, 1993; White et al., 2001] can be healed only once, since once the microcapsules are broken, there is no healing agent left to perform its function. Also, the poly-DCPD “healant” may not be fully compatible with the matrix to be healed, resulting in reduced mechanical properties. In the case of the second type of thermoset material [Raghavan and Wool, 1999], researchers found their crack repair capability to be “poor” and the recovered mechanical strength was only 1.7% of the original value. Although improvements in the healing properties of these polymers are being made through continued research, for the microsphere approach in particular, it is of great interest to search for alternate materials that have the capability to repair cracks multiple times without the addition of external agents.

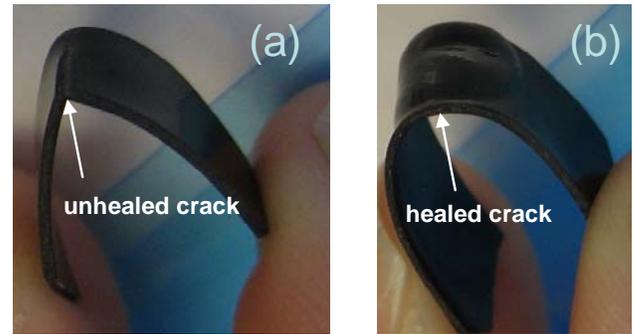
The “mendomer” developed by UCLA [Chen et al, 2002] is such a material that is both structural and can repair after repeated damage. These materials have been so named due to their mending properties. The mendomer material is a highly cross-linked polymer (Furan-Maleimide crosslinked solid) with thermally reversible linkages, resulting from multiple Diels-Alder (DA) connections. Preliminary research indicates that the activation energy to break this reversible linkage is dramatically lower than all the other non-reversible covalent connections that characterize the polymers reported [Dry and Sottos, 1993; White et al., 2001; Raghavan and Wool, 1999]. This implies that the retro-DA reaction is an accessible reaction pathway that is preferred over (non-reversible) bond-breaking reactions for crack propagation in the polymer network. When the mendomer material is heated, its polymer chains gain enough mobility and the terminals of the broken linkages appear to find their counterparts to reconnect. Upon cooling, the connection remains intact and over time, the cracks are healed. This process is fully reversible and can be used to repair the cracks multiple times.



**Fig. 1** Depiction of self-healing ionomers

Other materials have been shown to heal when punctured where energy in the form of heating is attributed to the resealing of ionomeric membranes after bullet penetration [Fall, 2001; Kalista, 2003]. Figure 1 depicts the self-healing of ionomers in terms of ionic group re-attachment. These ionomers have been

demonstrated to self-heal using embedded magnetic particles in a related approach for wire insulation applications as shown in Figure 2. These ionomeric-magnetic-particle composites are suitable for compliant self-healing applications requiring a Young’s Modulus in the 200 MPa range whereas the mendomer-magnet-particle composites currently investigated in this paper are suited to structural applications requiring a Modulus greater than 2 GPa.



**Fig. 2** Response of previously adjacent strips of soft polymers containing a low-volume fraction of magnetic particles and showing site-specific healing (a) cleaved surface remains unhealed where heating is not applied, (b) cleaved surface has healed where site specific healing has been applied (scarring is also evident)

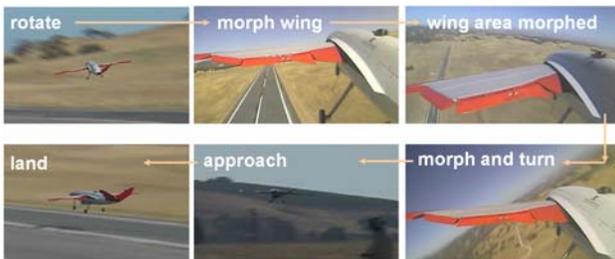
Due to the self-healing activation by heat, mendomer and ionomer self-healing materials may have anti-fatigue properties. For the mendomer, during cyclic loading the heat that is generated in the fatigue zone may be adequate to heal cracks after they are formed. At low temperatures, even though the polymer chains cannot move freely as they do at reversal temperatures, the mobility may still help to reconnect linkages. Both the mendomer and ionomer materials are expected to be highly resistant to damage caused by explosive loading since the cracks formed during loading are likely to heal while they are subjected to the temperature field generated by the explosion.

## 1.2 Morphing structures and material systems

A morphing aircraft can be defined as an aircraft that changes configuration to maximize its performance at radically different flight conditions [Bowman et al., 2002]. These configuration changes can take place in any part of the aircraft, e.g. fuselage, wing, engine, and tail. Starting from the first successful powered flight by Wright Brothers, who used differential twisting of the wing to control the airplane, most of the morphing technologies have been historically performed at a small scale. The small-scale morphing, such as aileron, elevators, flaps, landing gear, etc. is used either to enable a controlled flight or to improve the aerodynamic performances of an aircraft. The large-scale morphing

designs have been attempted in the past as well. Most notable among is the wing design, which was successfully utilized in several airplane design including F-111, MiG-23, F-14, B-1B etc. However, due to the weight penalty, the fixed-wing designs have been preferred in the past.

With the advent of new technologies such as smart structures and materials, a more serious design and development effort in the area of morphing aircraft was initiated under DARPA and NASA leaderships [McGowan et al, 2002]. This effort culminated in SMA (Shape Memory Alloy)-based smooth cambering of Northrop UCAV test model under the Smart wing Phase II program [Kudva et al., 2002]. A more recent research efforts (under DARPA, NASA, and AFRL sponsorships) focused on more dramatic configuration changes such as 200% change in aspect ratio, 50% change in the wing area, 5° change in the wing twist, and 20° change in the wing sweep to lay the ground work for a truly multi-mission morphing aircraft. This led to the design and development of two morphing wings, which were tested in 2005 in NASA Transonic Wind Tunnel by NextGen Aeronautics and Lockheed Martin under separate contracts [Joshi et al., 2004; Love et al., 2004]. Such large-scale wing geometry changes, while extremely challenging, can be conceptually achieved in a variety of ways including folding, hiding, telescoping, expanding, and contracting a wing, coupling and decoupling multiple wing segments.



**Fig. 3 NextGen Morphing UAV during flight**

The main concerns facing a successful morphing design is how to 1) design a wing skin that can undergo deformation of more than 100% strain while maintaining the load bearing capability and requiring minimal actuation energy for deformation 2) reduce the weight penalty arising from the morphing mechanisms and actuators, and 3) develop supporting sub-systems of the morphing aircraft in order to accommodate the large-scale wing morphing. Figure 3 shows snapshots of a recently concluded flight of a morphing UAV by NextGen Aeronautics. However, a rigorous development of a morphing aircraft would require investigation in the area of morphing mechanisms, flexible load bearing skin, aeroelasticity, controls, aerodynamics, structural optimization, and engine morphing. Research studies in these areas are currently

being conducted in industries and academia to make a truly successful morphing aircraft, which is expected to increase the warfare lethality at the reduced system-level production and operational costs [Inman et al., 2001].

As mentioned earlier, wing skin is one of the most critical technologies that needs to be matured for a successful structurally morphing aircraft design. Its design has the conflicting requirements of large deformation, high fatigue life, load carrying capability, and low actuation energy. Therefore, the self-healing material in conjunction with deformation mechanism can be very useful for the design of morphing wing. It is found that, in addition to demonstrating self-healing properties, the mendomer polymer is also observed to demonstrate shape memory characteristics which can be used to design morphing functionality into a composite structure. The shape memory effect (SME) can be defined as the ability of a material to be deformed, and then, upon application of a stimuli, return to its original shape. The most common stimulus being currently used is direct heat, though light of a specific wavelength may be used as well. Indirect heat caused by an electric field has been also demonstrated as the activation mechanism. The amount of deformation that can be accommodated is highly dependant upon the material being strained. For instance, some shape memory alloys can be strained to nearly 10%, while shape memory polymers can be strained to nearly 400%. The main advantage of this type of deformation is that it does not need an extra set of actuators. In other words, the induced deformation is an intrinsic characteristic of the material. This has the potential of weight and space savings for a morphing wing design.

### 1.3 Multifunctional Composite (MC) structures

While morphing and self-healing functionality merit their own individual advantages, the combination of these functions promise even greater benefits for high-performance multi-role structures used in not only weapon systems, but in aerospace and commercial applications. To be sure, the authors' experience with self-healing and shape memory materials has revealed reflected properties of one material in the other. Our final goal of this and the consecutive work is to further develop this new class of multifunctional composite structure that merges both self-healing materials and morphing technology (i.e., morphing structures and shape memory materials). Such a confluence of technologies will revolutionize the way structures are designed and built. While the combination of smart materials and structures to support multifunctionality is a familiar territory, this research appears to mark the first effort into specifically combining morphing and self-healing onto a single platform. How these MC structures have been fabricated, characterized are described in the

following sections—as well as tests results and figures demonstrating self-healing and morphing proofs of concept.

## 2.0 MANUFACTURING OF MCS

To facilitate the controlled self-healing and morphing of the material, a low-volume fraction (<10%) of microscale and nanoscale magnetic particles are designed into the composite to be coupled later to a damage event such as delamination. By application of a magnetic field at a specified frequency the particles inductively respond. The associated ferromagnetic switching of domains gives rise to eddy current and hysteretic loss which in turn facilitates self-healing by heating of the mendomer above its glass transition temperature in selected areas.

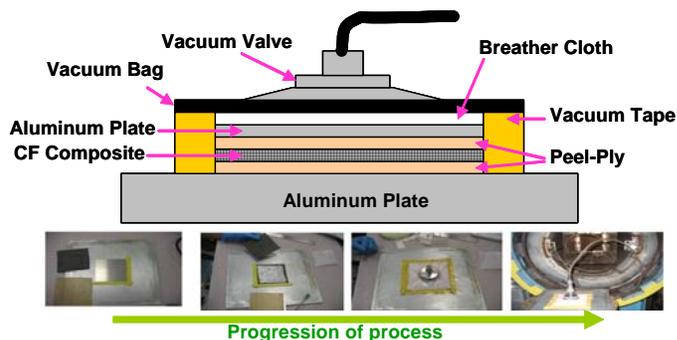
When the magnetic particles are added into the mendomer during its fabrication, the result is a mendomer material that can be heated to its glass transition temperature by an applied magnetic field. Samples are made of mendomers, which are prepared in several steps from commercially available DCPD. The resulting product is converted directly to Mendomer-400. The mendomer is mixed with a small volume fraction of magnetic particles alternatively pressurizing and venting the composite using a vacuum bag technique and autoclave approach.

This methodology is completely modular and can be easily altered by changing the tether to improve the polymer's toughness. With the aid of molecular modeling other candidate tethers can be selected to further modulate polymer properties. This mendomer-400 polymer is expected to possess mechanical properties rivaling other commercial epoxy resins, with the added exception of being self-healing. As mentioned previously, an alternate mendomer-401 was synthesized to verify that that a lower healing temperature ( $T_g$ ) could be designed by modifying the molecular structure.

In order to investigate a built-in upper-temperature-limit switch, composites using a ferrite compound of magnetic particle were fabricated. These were in addition to the metal oxide mendomer composites that were fabricated that contained either gamma or alpha phase iron oxide particles. Once the Curie temperature of the ferrite compound is reached, the composites cease to experience further heating. This is not expected for iron oxide particles, where a low-temperature cut-off temperature was previously verified as absent. Another benefit to the use of the particles made of the ferrite compound stems from the fact that thermal degradation of polymers by metal oxides has been widely studied. Iron oxide is known to accelerate the degradation of certain polymer systems [DeBarros et al., 1991, Kashiwagi, 2003]. Specifically, poly(vinyl chloride)

(PVC) is known to eliminate hydrogen chloride at elevated temperatures (~180 C) in the presence of iron oxide [Uegaki et al., 1977]. Alternatively, iron containing metallic complexes can be reversibly oxidized by peroxides yielding free radicals catalyzing the degradation of polymeric systems. The ferrite compound particles that were used have not been shown to catalyze the degradation of polymeric materials and can be used as an alternative to iron oxide. Moreover, because of their higher relative magnetic susceptibility of the ferrite compound, the component can be used in lower concentrations to achieve the desired effect.

The Carbon Fiber (CF) composite panel is prepared by traditional vacuum bagging technique as depicted in Figure 4, incorporating pure mendomer (and in the future premixed mendomer-nanoparticle composites) and cross weave. A square aluminum plate is used as the base plate on which the panel is to be processed. Vacuum tape (yellow in the photos of Fig. 4), which prevents the liquid from flowing away from the cross weave, is placed around the area where the panel is to be cast as shown in Fig. 4. Next a layer of peel-ply is used as a releasing agent to prevent the panel from adhering to the aluminum plate. The mendomer (or mendomer-particle mixture for future procedures) is evenly spread over the carbon fiber cloth layers (2), and covered with another layer of peel-ply. The top aluminum plate is placed above the peel-ply, followed by a layer of breather cloth, which allows the complete evacuation of the panel. Next, the vacuum valve is inserted through a section of vacuum bag material, and the bag material is adhered to the vacuum tape to completely seal the system and preventing vacuum leaks. This assembly is then further processed in an autoclave by polymerizing and curing under vacuum. The assembly is slowly cooled and carefully disassembled to obtain the composite panel.



**Fig. 4 Vacuum bag technique for manufacturing carbon-fiber composites**

The resin particle composite is prepared by thoroughly mixing the desired mendomer with the appropriate percentage of metal nanoparticles. This mixture is then transferred to the desired specimen mold, and evacuated in a vacuum oven. The oven is purged

with argon to remove oxygen, and heated to 125 °C. Some out gassing will occur during the melting/polymerization, which occur simultaneously upon melting. The out gassing can be controlled by regulating the pressure in the vacuum oven. Increasing the pressure inside the oven as the polymerization nears completion, which is characterized by an increase in viscosity, eliminates the presence of bubbles in the specimen.

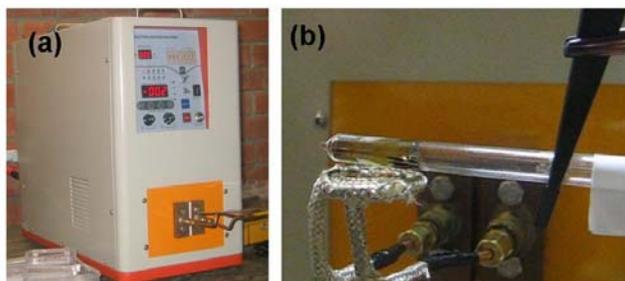
### 3.0 CHARACTERIZATION OF MCS

The mendomer-400 was fully characterized by Fourier Transform Infrared Spectroscopy (FTIR), Nuclear Magnetic Resonance (NMR) both proton (1H) and carbon (13C), Matrix Assisted Laser Desorption/Ionization (MALDI-MS), and Elemental Analysis. The mendomer-400 is converted to Polymend-400 by heating to its “cracking” temperature which is determined by modulated Differential Scanning Calorimetry (DSC), which typically ranges from 100-150 degrees Celsius.

The physical properties of Polymend-400 were empirically determined. The glass transition (T<sub>g</sub>) and healing temperature was determined by Dynamic Mechanical Analysis (DMA) and DSC. Its thermal stability was measured by Thermo-Gravimetric Analysis (TGA). The mechanical properties; i.e. compression modulus, flexural modulus, and Young’s modulus of Polymend-400 was determined as dictated by ASTM standards. The shape memory properties were determined by measuring the original dimensions of the specimen, then programming a new shape into the specimen by heating to its T<sub>g</sub>. The new shape is retained after cooling to room temperature. The original shape can be recovered by heating once again to its T<sub>g</sub>. The self-healing properties of the polymer were measured by measuring the strength of a virgin specimen in tension, then stressing this specimen until fractured. After being heated to 150 °C to induce healing, the specimen was retested in a similar manner as before. The second measurement was compared with the first and a % strength recovery percentage was calculated to quantify the healing properties of this material.

Various magnetic particle compositions were investigated to serve as the inductive heating component for the mendomer/magnetic particle composites including alpha and gamma iron oxide as well as a proprietary ferrite compound. While different particle distributions were inherently part of the study, further investigation must be executed—including a particle distribution and average size study where the composition is fixed--before conclusions can be drawn about the nanoscale response versus microscale response of the magnetic particles.

The self-healing mendomer/magnetic particles samples were characterized using a Superior Induction SI-7kW HF System used in constant voltage manual mode (Fig 5a). The slow-cooled self-healing composite showed healing at 3.4 amps, 78 volts and 1.6 kW after 2 minutes of exposure. A “pancake” coil was used to isolate the area of healing as shown in Fig. 5b. The mendomer-particle sample was kept in the test tube for ease of crack identification.



**Fig. 5 Self-healing set-up using (a) SI-7kW HF System (b) showing “pancake” coil with sample**

Mendomer/carbon-fiber samples were heated using a handheld heat gun to demonstrate morphing using the SME. The following section describes the results of the characterization that was described in this section.

### 4.0 RESULTS OF MC CHARACTERIZATION

This section discusses the characterization results for the mendomer-400, mendomer-401 and proof-of-concept tests for a mendomer-magnetic-particle composite and a mendomer-carbon-fiber composite. Magnetic particle parametric study results have not been included in this publication. The NextGen-UCLA team successfully demonstrated the ability of a mendomer-magnetic-particle composite to self heal with the application of a magnetic field. The team also demonstrated the ability of a mendomer-carbon-fiber composite to morph under applied heat by relying on the SME.

**Table 1: Mechanical properties of mendomer-400 and mendomer-401**

Polymer	Compressive Modulus GPa	Compressive Strength MPa	Flexural Modulus GPa	Maximum Fiber Stress MPa
Mendomer-401	1.95	104	3.26	116.8
Mendomer-400	1.65	83.5	2.63	109.3

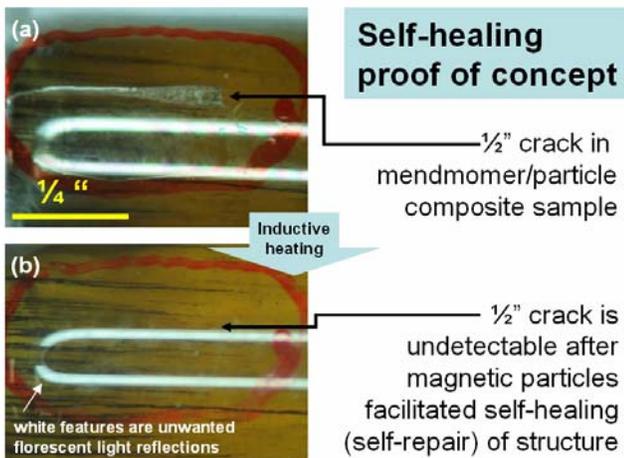
The measured mendomer-400 polymer properties are given below. Values for PMMA (Poly(methyl methacrylate)) are also given to provide a comparison to a polymer known to be tough and impact resistant.

- Thermally Remendable at 150 °C
- Poisson Ratio is 0.306
- Density is 1.31 g/ml (PMMA is 1.19 g/ml)

- Thermally Stable where for Tg measurements, a 5% Weight Loss at 410 °C indicates the beginning of sample decomposition
- Glass Transition (Tg) is 150 °C
- Flexural Modulus is 2.63 GPa (PMMA is 3.0 GPa)
- Compression Modulus is 1.65 GPa
- Young's Modulus is 2.4 GPa (PMMA is 2.6 GPa)
- Yield Strength is 136 MPa (PMMA is 86 MPa)
- Shape Memory Effect is demonstrated for complete strain recovery after 2 min at 150 °C.
- Chemically resistant to oxidizing solutions, hydrofluoric and sulfuric acid

Two mendomers, mendomer-400 and mendomer-401, were investigating to validate theory supporting the adjustment of glass transition temperatures (self-healing temperature) and mechanical properties as shown in Table 1. The Tg of mendomer-401 was verified to be lower than the mendomer-400.

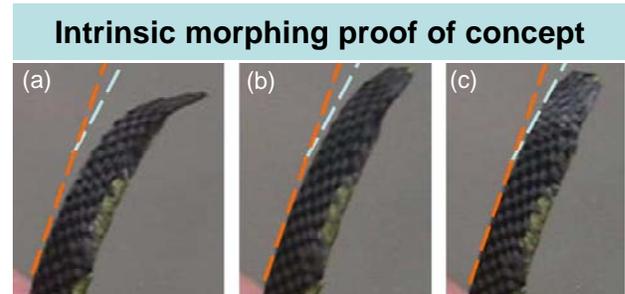
Figure 6a shows a mendomer-401/ferrite compound samples with a 0.5" length crack prior to healing. Figure 6b shows the same specimen after it was subjected to inductive heating from an external magnetic field; it can be observed in the micrograph that the crack was healed. The red pen markings helped identify the location of the crack on the specimen. The white wide line features are artifacts of the fluorescent lighting that was present when the micrographs were being taken. Magnetic particles were aligned in a magnetic field to demonstrate site specific healing in the mendomer. As such, the total volume fraction of the sample is different than the relative volume fraction near the healed area.



**Fig. 6 Proof-of-concept showing self-healing of mendomer using a low-volume fraction of magnetic particles (a) the sample with a crack prior to self-healing (b) the sample after self-healing**

What has demonstrated is that once the mendomer is heated above its glass transition temperature, cracks in the composite visibly heal. While quantification of the

degree of self-repair must be performed as was performed for the pure mendomer, the similar absence of diffracted light in the healed sample indicates a sealed crack. Also, further investigation at 100X magnification failed to uncover any evidence of the previous presence of a crack. Admittedly lack of sufficient registration of the initial crack location rendered production of a comparison micrograph at 100x infeasible.

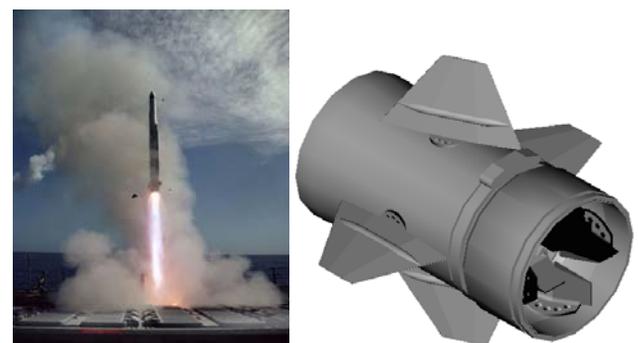


**Fig. 7 Carbon-fiber composite demonstrates morphing using SME—i.e., uncurling of sample (a) deformed from original linear shape prior to heating (b) during heating (c) after heating to 150 °C**

Figure 7 demonstrates the shape memory effect and associated morphing of a mendomer fiber composite as the composite tip unfolds and returns to its original shape. This sample was fabricated using mendomer-401 and a woven carbon-fiber composite.

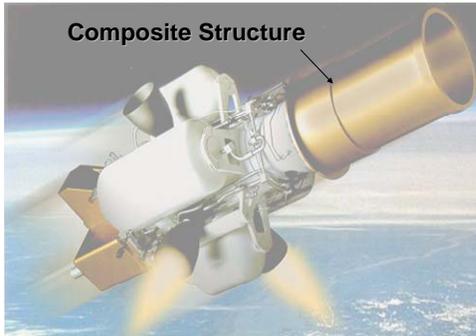
## 5.0 POTENTIAL APPLICATIONS OF MCS

The success of this new class of multifunctional material for near term Department of Defense (DoD) technology insertion is highly dependent on its integration into an army Ballistic Missile Defense System (BMDS). NextGen is currently collaborating with Raytheon Missile Systems to discuss insertion of this self-healing morphing technology into several of their programs including their Kinetic Energy Interceptor (KEI) and Exo-atmospheric Kill Vehicle (EKV) Programs.



**Fig. 8 Air missile control surface showing spar and hinge mechanism**

An example application of the technology includes multifunctional morphing and self-healing carbon-fiber replacement of air missile control surfaces (Fig. 8) where a deformed control surface could change the aerodynamic properties of the missile to achieve a desired maneuver while at the same time healing from damages sustained during transport or flight.



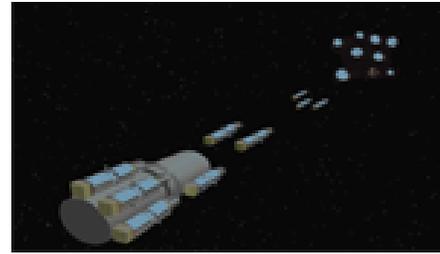
**Fig. 9 Exo-atmospheric Kill Vehicle (EKV) Structure is made of composite materials**

Additionally, tactical cruise missiles would benefit where morphing could significantly improve system performance characteristics such as range and cruise speed, decreased payload and increased endurance. Self-healing skins and control surfaces would extend the overall useful lifetime of the missile, for example repairing missile damage due to corrosion. Where stiffness is critical self-healing composite structures would have direct benefits to interceptor structures such as the one shown in Fig. 9 of an EKV. The KEI is the weapon component of the Ballistic Missile Defense System designed to defeat intermediate and long-range ballistic missile threats. The structural self-healing properties of the composite would serve as an immediate benefit to the interceptor and launcher parts of the KEI shown in Fig. 10.



**Fig. 10 Interceptor and launcher parts of the KEI**

Other missile related applications include hypergolic fuel tanks, phalanx structures, kinetic energy interceptor canisters. The Multiple Kill Vehicle (MKV) system allows for more than one kill vehicle to be launched from the same booster as depicted in Figure 10. In the same way EKV and KEI programs will benefit, Future Multiple Kill Vehicle (MKV) components will also improve from the MC technology discussed in this paper.



**Fig. 11 Missile Defense Agency MKV**

## CONCLUSIONS

This work described the development towards a new class of multifunctional materials that possess both self-healing and morphing properties. While proof of concept was demonstrated to show that a small volume-fraction of magnetic particles is sufficient for self-healing of a 360 micron x 5000 micron thick crack in mendomer-401, more quantitative analysis is necessary to determine extent of scarring and associated degradation of properties. Proof of concept was also demonstrated to show that carbon-fiber composites could be fabricated to morph using the apparent SME properties of the mendomer. Studies of magnetic particle composition (gamma phase iron oxide versus a ferrite compound) and mendomer (400 versus 401) were performed to determine a relative optimal choice of material system components for self-healing and morphing functionality. Future work will focus on combining the self-healing and morphing properties into one composite as well as coupling these functions to a failure event. Multiscale modeling of the micromechanical level of healing reconciled with predictive continuum mechanics models is also of interest.

Multifunctional material systems and structures possess the added advantage of combining properties to optimize both material and structural design to in turn optimize for performance and behavior. It is in the sum of the parts that the greater benefit is achieved whose final structural performance has been optimized through coupled optimized design. Morphing at a minimum can increase vehicle performance, but in its most beneficial use can potentially enable an air vehicle (aircraft or missile) to change its mission. The added functionality of self-healing would not only extend the life of these multifunctional structures but also prevent material failures that can be catastrophic. Moreover, today's warfighter has the potential to benefit from the self-healing of protective wear and personal gear as well as potentially benefit from increased survival from any stealth-related benefits of morphing for camouflage.

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