Fatigue Resistance of Liquid-Assisted Self-Repairing Aluminum Alloys Reinforced with Shape Memory Alloys

Shape Memory Alloy Self-Healing (SMASH) Technology for Aeronautical Applications

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Abstract

A self-repairing aluminum-based composite system has been developed using a liquid-assisted healing theory in conjunction with the shape memory effect of wire reinforcements. The metal-metal composite was thermodynamically designed to have a matrix with a relatively even dispersion of a low-melting eutectic phase, allowing for repair of cracks at a pre-determined temperature. Additionally, shape memory alloy (SMA) wire reinforcements were used within the composite to provide crack closure. Investigators focused the research on fatigue cracks propagating through the matrix in order to show a proof-of-concept Shape Memory Alloy Self-Healing (SMASH) technology for aeronautical applications.

1.0 Introduction

1.1   Fatigue Life and Repair of Aeronautical Structural Members

Aerospace-grade aluminum materials subject to cyclic loading are susceptible to failure by fatigue at loads well below their yield strength. Numerous cases of catastrophic fatigue failures are documented in engineering fracture references and fatigue endurance has been an ongoing topic of interest for civilian and military aging aircraft, or airworthiness, conferences [1]. In aeronautical applications, wrought and cast aluminum alloys have commonly been used in structural and non-structural parts throughout aircraft, such as fuselage skin/stringers/frames, ribs, longerons, stiffeners, wing spars, fuel tanks, landing gears, wheel wells, fuel lines, shock struts, cargo doors, floor beams and seat tracks [2]. Even with the increased popularity of carbon-reinforced composite materials for structural applications, commercial companies continue to develop advanced aluminum alloys for damage tolerance at critical areas in aircraft, including primary fuselage and wing structures [3]. Besides improving upon conventional aerospace grade materials such as 2XXX-series (Al-Cu) and 7XXX-series (primarily Al-Zn), companies continue to look at utilization of Al-Li alloys to take advantage of aluminum alloys' strength to weight ratio while improving upon fatigue life, crack propagation, and fracture toughness. In addition, research is being conducted in advanced hybrid systems with selective reinforcements, where fiber metal laminate or fiberglass pre-preg composite materials are bonded to aluminum structures at locations where damage tolerance is a driving design factor [3,4]. According to one reference, these advanced concepts can more than double the inspection intervals of current aircraft [3]. For both aging aircraft and new aeronautical concepts, consideration of material performance and failure mechanisms must be taken into account to ensure reliability of the aircraft and mission success.

It is known that 90% of moving part failures occur due to fatigue [5]. A characteristic of fatigue fracture is that it is time dependent, providing an opportunity to detect and correct cracks that have initiated and started to propagate (Stage I and II) before full failure (Stage III) occurs. When a crack is detected on an aero-structure or component during routine maintenance or other inspections, the options to maintain airworthiness of the aircraft are to repair the crack or replace the part. There are ways to externally repair the crack using mechanically fastened or adhesively bonded techniques [6]. Conventional repair techniques on metal structures include using doublers to reinforce the material surrounding the crack, using selective plating over the crack, filling the separated material using polymeric fillers, crack tip blunting by drilling the crack tip to prevent further propagation, grinding out the crack and using weld overlays for the required part thickness, and thermal spraying the crack. Investigations have also focused on

advanced repair techniques using composite patch repairs over metal structures [6,7]. Doublers are commonly used to fix aeroframes but surface preparation and bonding can be problematic and additional stress concentrations may be introduced into the damaged areas. Weld overlays work well in steel-based alloys, but produce a region of decreased strength in aluminum structures. Composite patch repairs are sensitive to surface preparation and may introduce material compatibility problems in terms of thermal expansion and corrosion. In all, the techniques that are available to repair aluminum alloy aerostructures require using an external material for repair, skilled application of the repair by trained personnel, and direct access to the damaged area in question. At times when the materials or skills are not available, these techniques are not an option.

1.2   Shape Memory Alloy Self-Healing Technology

Taking advantage of the memory effect of shape memory alloy (SMA) reinforcements, and a liquid-assisted healing concept, metal-metal composite systems are being developed by researchers to be used in high-stress locations where fatigue crack initiation could be problematic [8]. This research uses liquid-assisted healing to create Shape Memory Alloys Self-Healing (SMASH) technology. Although the SMASH technology being developed can be used for any type of failure mechanism that results in a tight crack in the metal, the focus of the investigation is fatigue fractures due to their prevalence in industry and their potential for catastrophic events. The materials system being designed would allow for repair of detected fatigue cracks without the need for additional external materials or human interaction, which are both needed with conventional repair techniques.

SMASH technology uses a two-step crack repair method: crack closure from contraction of the SMA reinforcements and crack repair during partial liquefaction of the matrix (liquid-based healing). Both steps are accomplished by heating the crack area to a pre-determined healing temperature. Researchers start by using a thermodynamic approach to design a metal matrix with a certain fraction of a low melting phase that liquefies at the pre-determined temperature to ensure liquid-based healing. Liquid-based healing alone is limited by flaw size and healing large flaws is difficult because the flaws can lead to weakening of capillary forces and incomplete healing. The use of a crack-closure mechanism overcomes this limitation, and the SMA reinforcements provide a clamping force. Figure 1 shows the concept of liquid-assisted healing in detail [9]. A force is applied to the composite parallel to the SMA reinforcements and large enough to initiate and propagate a crack in the matrix. When a crack is present in the matrix material, local stresses induce phase transformations in the SMA reinforcements, causing the SMA reinforcements to stretch and bridge the crack. Martensite variants within the SMA locally reorient to accommodate the applied stress. Heating the metal-metal composite above the reversion temperature of the SMA reinforcements causes SMA contraction and forces crack closure. While held at the reversion temperature, the low melting phase of the matrix partially liquefies and acts as a healing agent filling in the crack and solidifying when brought back to room temperature. Liquid-assisted healing results in the healing agent being constantly available and damage can be repaired multiple times over the lifetime of the part.
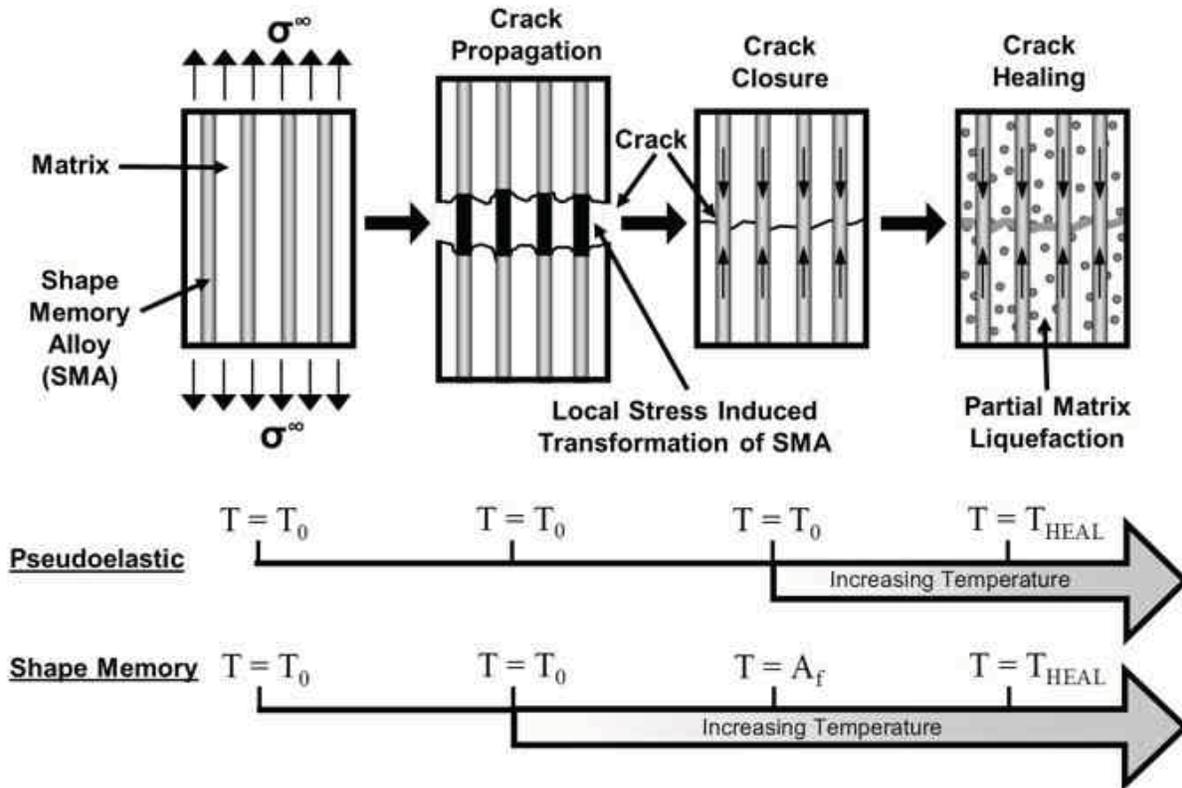
Figure 1. Schematic representation of liquid-assisted healing of SMA reinforced metal matrix composites [9].

A proof-of-concept (POC) tin-bismuth (Sn-Bi) matrix with SMA reinforcements displayed successful crack closure and healing of overload cracks using the liquid-assisted approach [9, 10].   However, questions remained about the ability to design a higher-strength aluminum-based matrix that could eventually be used for aircraft applications. The knowledge gap also extended to the ability of liquid-assisted healing to repair fatigue cracks from cyclic loading and the effects of healing on the fatigue life of the composite.

This investigation has shown that there is an alternative to common repair techniques of fatigued structures. This new option would utilize a heating source to drive the material to repair itself by forcing crack closure and affording liquid-assisted crack healing at the crack front. The implications of a successful materials system would improve the damage tolerance and fatigue life of metals at critical structural locations.

2.0 Experimental Procedure

2.1   Matrix Alloy Design and Fabrication

In order to design the optimal matrix microstructure, a thermodynamic approach was utilized such that the aluminum matrix microstructure contained a pre-determined fraction of a low melting eutectic constituent. The test material was fabricated with about 2 volume percent of SMA wire reinforcements to create samples in bar, dogbone, and compact test geometries

using casting and hot pressing techniques. Details of the matrix alloy design and fabrication are reserved due to intellectual property rights.

2.2   *Mechanical Testing*

Once the optimal aluminum alloy matrix composition was established, two materials systems were tested: the POC metal matrix composite that had shown self-repairing capabilities, and the newly developed aluminum matrix composite. Post fabrication, the samples were tested in a universal test machine to show initial tensile strength and/or induce cracks depending on the test. A tensile test was performed after healing to show the retained tensile strength. Sub-size tensile specimens were created in accordance with ASTM E8. Fatigue stress-life testing was carried out on samples fabricated according to ASTM specifications for tensile testing (E8) and fatigue testing (E466). Stress-life fatigue testing was performed with a stress amplitude ratio of R=0.1 (tension-tension), sinusoidal waveform, and test frequency of 5 Hz. Fatigue crack growth tests were carried out on middle-tension, M(T), and single edge notch tension, ESE(T), specimens made to ASTM E647. Middle tension specimen testing was conducted with a stress amplitude ratio of R=0.1, maximum stress at 0.8 of the yield strength, and 80 Hz frequency. Crack growth measurements were taken optically from the center of the specimens. Single edge notch tension specimen testing was carried out at a stress amplitude ratio of R=0.1, 5 Hz frequency and with a stress intensity factor range of $\Delta K=3.6$ ksi$\sqrt{}$in.

3.0 Results and Discussion

3.1   *Aluminum-Based Self-Healing Alloys*

Aluminum alloy matrices with the pre-determined eutectic phase were thermodynamically designed for three different systems: Al-Si, Al-Cu, and Al-Cu-Si [11]. Figure 2 shows the sectional microstructure of an Al-Si alloy. The uniform spheroidal distribution of the eutectic is ideal for liquid-assisted healing and the SMA wire bonding to the matrix was adequate. The Al-Si composite was heat-treated and tested in tension until a crack formed, heat-treated to heal cracks that formed during tensile testing, and tested again. The stress-strain curve in Figure 3 shows effective healing with over 90% retained ultimate tensile strength (UTS) in the healed composite, where "monolithic" refers to the original composite and "post heal" refers to the specimen after the crack had been healed. The tensile testing on the Al-Si composite proved self-healing in a binary matrix alloy with pre-determined eutectic phase and 2 vol% SMA wires.
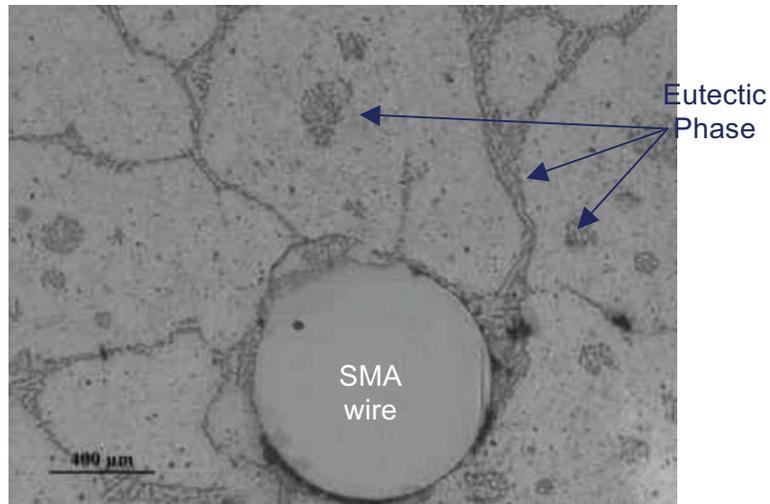
Figure 2: Representative microstructure of binary Al-Si composite showing adequate SMA wire bonding and proper distribution of eutectic phase [11].
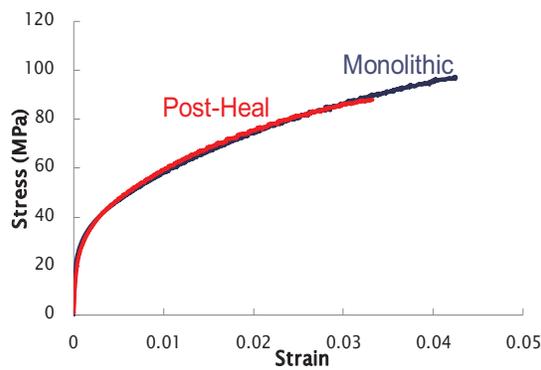

Figure 3. Tensile testing of single Al-Si composite before and after healing showing 90% retained UTS [11].

Matrices were also designed and cast using Al-Cu and Al-Cu-Si which thermodynamically should exhibit liquid-assisted healing behavior. Each alloy was heat-treated, tensile tested, heat-treated again, and tensile tested a second time. These alloys were found to be much more brittle than the Al-Si in tension (Figure 4). Almost no healing was evident in either Al-Cu or Al-Cu-Si alloys. It is theorized that the lack of ductility in the matrix prevented the SMA wire from undergoing a phase transformation and thus could not provide a clamping force on the crack during healing. Without a clamping force to close the fracture faces, healing was unable to occur.
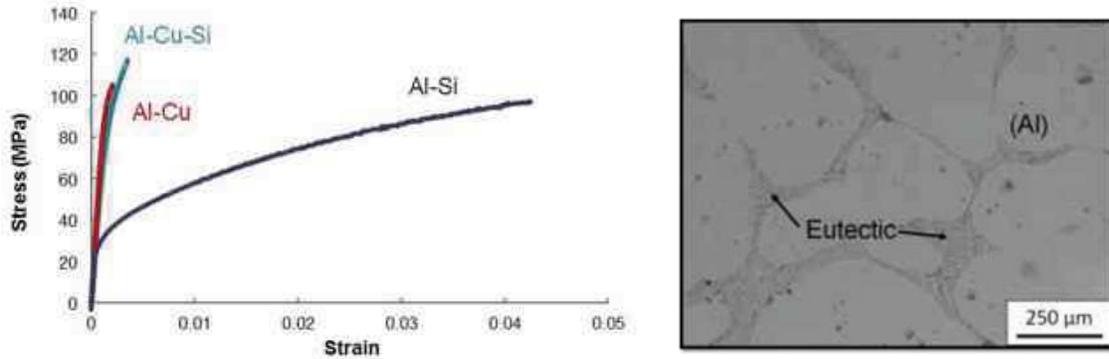
Figure 4. Comparison of tensile behavior for the Al-Si, Al-Cu, and Al-Cu-Si alloys pre-heal and representative microstructure of Al-Cu-Si (right) [11].

In order to overcome issues with incorporating complex wire geometries, and to attempt to improve ductility in order to achieve healing in the ternary Al-Cu-Si alloy, other fabrication techniques were investigated. A two-step high temperature pressing and diffusion bonding technique was developed. Initial compression testing of the matrix alloy at temperatures ranging from 200-550°C was conducted to determine the temperature dependence of the yield strength as well as the ductility at elevated temperature. This information was used as a first approximation for determining the temperature necessary to achieve adequate ductility for flow of the matrix around the wires, and how much pressure could be applied without exceeding the strength of the material. Once the plies were pressed together, a diffusion bonding step was carried out to enable the healing of the matrix across the previous interface. For an initial optimization of the process, the effect of pressure and diffusion bonding time were explored by a small design of experiments, and it was determined what pressure and temperature produced the most favorable composite microstructure (Figure 5a). Using this newly developed pressing technique, it was applied to more complex wire placement producing a multi-ply composite. Up to four plies with three reinforcement layers at the interfaces were successfully fabricated (Figure 5b). This composite was then machined and tensile tested, showing an increase in apparent ductility of the composite compared to the cast composite (Figure 5c).
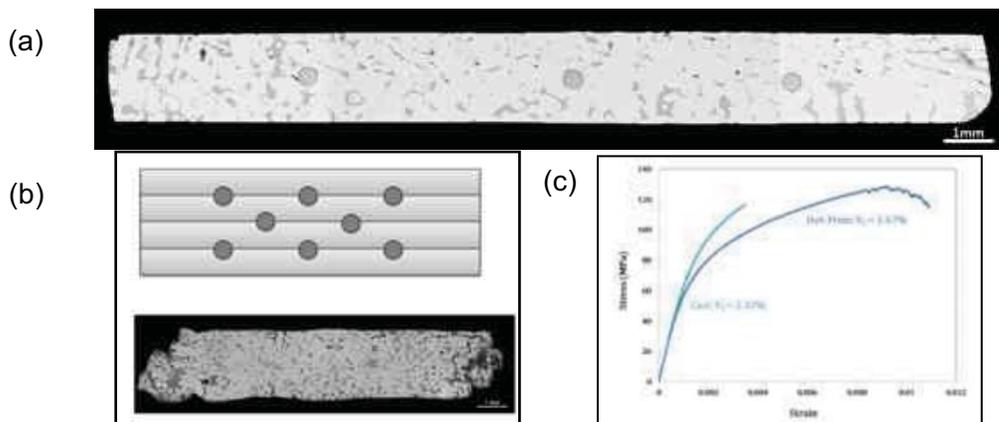

Figure 5: Representative cross section of diffusion bonded composite (a). Application of technique to fabrication of multi-ply composite (b) and comparison of mechanical properties of hot-pressed samples to cast sample (c) showing improvements in ductility achieved [11].

3.2 Fatigue Properties and Healing of POC Sn-Bi Alloy

The proof-of-concept (POC) Sn-Bi matrix with SMA reinforcements had already displayed successful crack closure and healing of overload cracks using the liquid-assisted approach. Investigators wanted to test the ability to repair fatigue cracks in the POC material system using the liquid- assisted crack repair approach. Initially only the Sn-Bi matrix was tested to obtain high-stress, low-cycle fatigue data. In order to establish the experimental methodology that would eventually be used on the aluminum-based SMASH alloys, and due to the availability of Sn-Bi specimens in the ASTM E-8 geometry, these non-ideal E-8 dog bone specimens were tested prior to using the ideal ASTM E466 specimen geometry. Figure 6 shows the stress life fatigue curve (S-N) for the POC Sn-Bi matrix and the Sn-Bi with SMA reinforcements of both geometries. The fatigue life of the POC composite was greatly improved with the addition of the SMA reinforcements. As expected, the fatigue life was further improved when using the E466 samples. Although the Sn-Bi POC composite material system was not under consideration for future aeronautical applications, investigators used the tested samples to determine fatigue crack healing ability. One of the samples that had undergone low-stress, high-cycle fatigue to failure was given a healing treatment. Figure 7 shows that the healing treatment on the POC composite resulted in crack closure.   X-ray computed tomography (CT) non-destructive evaluation (NDE) was used to verify that the through-thickness fatigue crack was no longer detectable and proved that liquid-assisted healing was possible on fatigue cracks.
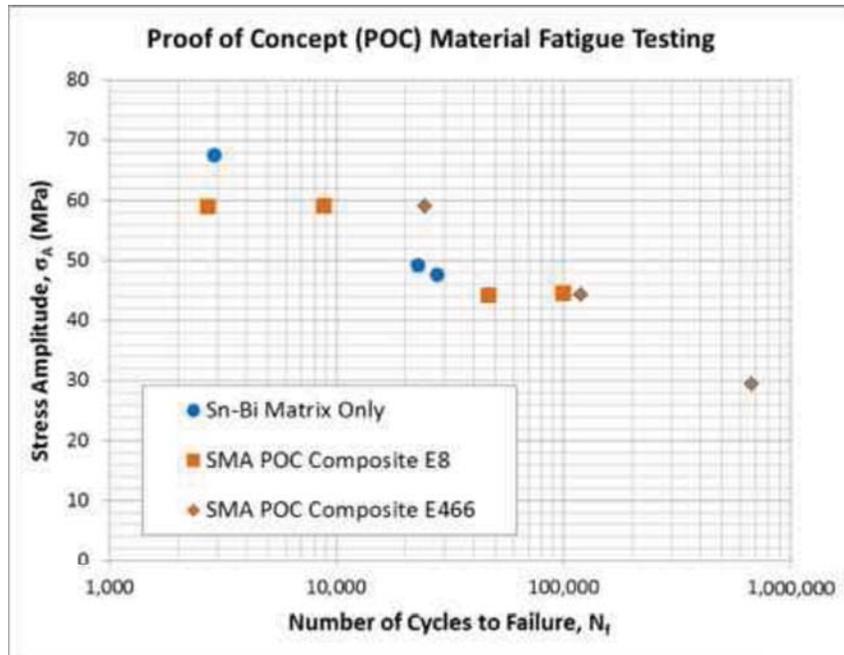

Figure 6. Stress-life fatigue data for the Sn-Bi POC matrix and composite system.
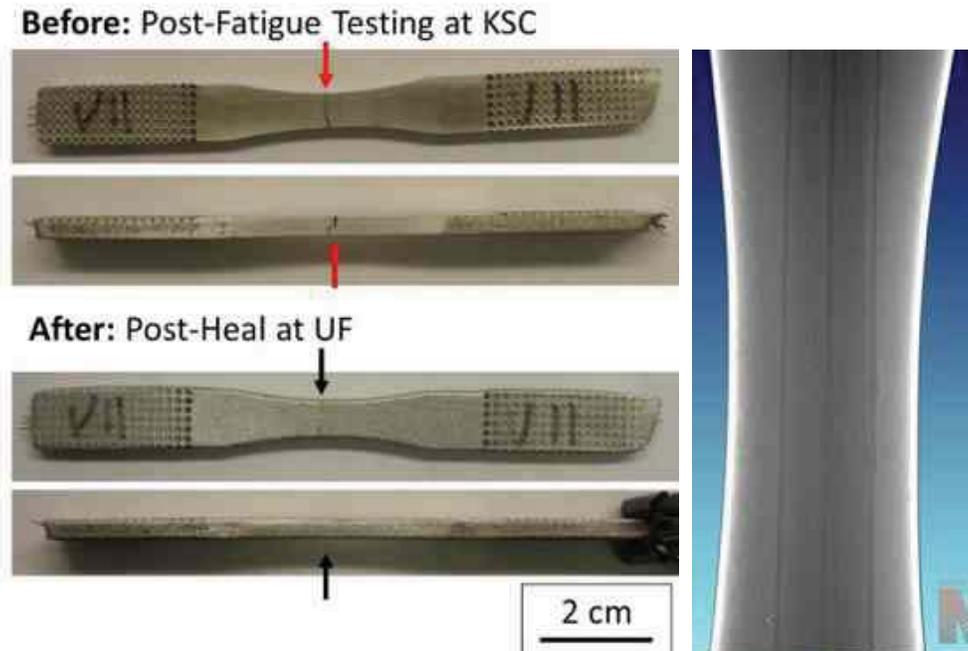
Figure 7.   Low-stress, high-cycle fatigue cracked POC specimen underwent the healing treatment resulting in crack closure (left).   NDE image shows absence of crack through thickness after healing (right).

3.3   Fatigue Properties of SMASH Aluminum-Based Alloy

Fatigue testing of cast Al-Si composite specimens made to E466 dogbone geometry specifications was carried out to create a stress-life curve of one of the aluminum-based SMASH alloys. Figure 8 shows that the S-N curve exhibits significant variability in the data, attributed to the gas and shrinkage porosity that was present in the samples due to the fabrication technique. Gas evolution during casting was not well controlled and resulted in each sample having a random distribution of spherical voids. Additionally, cooling caused shrinkage porosity in various regions of the casting. In some cases, the porosity was observed within the gage section of the test specimens. Figure 9 shows the CT slices taken through thickness prior to any mechanical testing of one sample. The slices show the density differences between the SMA wire reinforcements and the Al-Si matrix as well as significant amounts of shrinkage porosity internally near the SMA wires.   Since fatigue life is highly dependent on surface asperities, any shrinkage cavity that extended to the surface could have accelerated fatigue crack initiation. Once fatigue crack(s) initiated, internal flaws would have provided a preferred path for crack propagation.
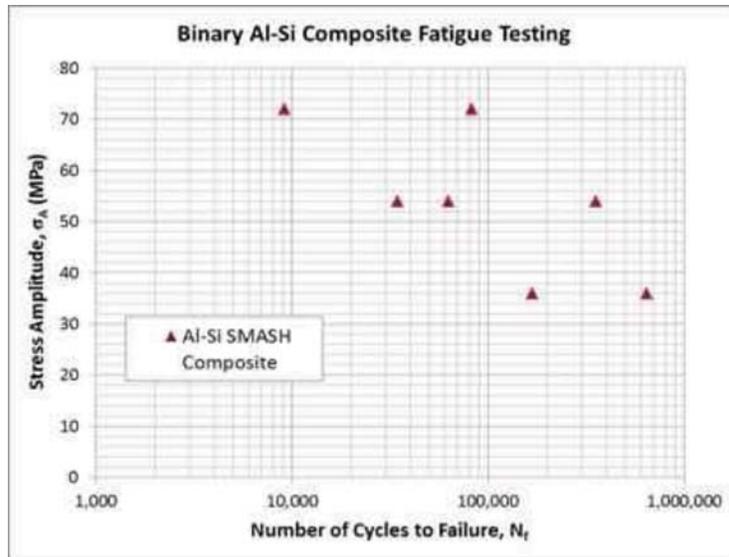
Figure 8. Stress life Al-Si composite data showing variability due to porosity in the samples.
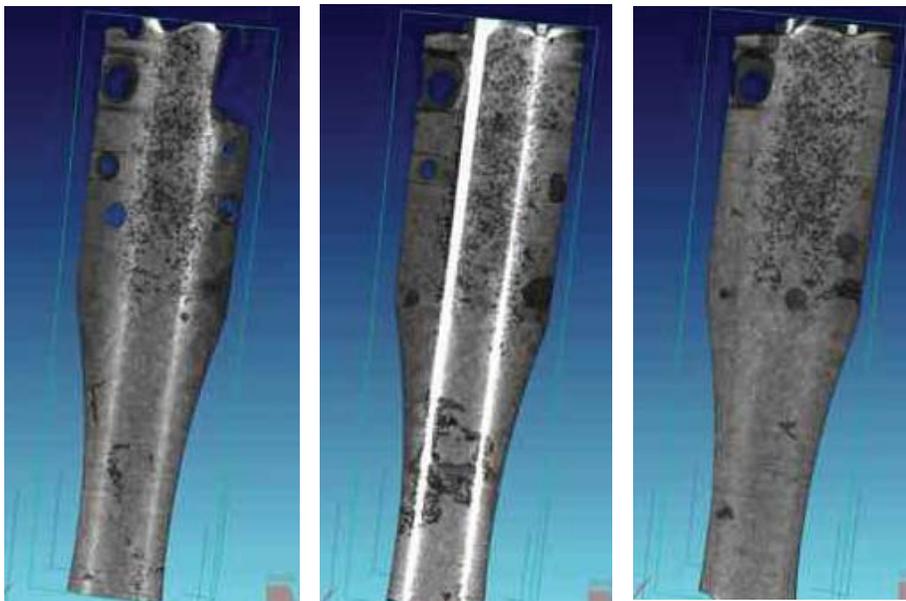

Figure 9. Sequential CT slices of an Al-Si composite dog bone specimen showing gas porosity at the grip section and shrinkage porosity within the gage length and near SMA wire reinforcements, most evident in the middle image. It is expected that the porosity from the casting technique degraded the fatigue properties of the materials system.

A scanning electron microscope (SEM) was used to image the fracture surface of the Al-Si fatigue tested SMASH composite specimens. It was observed that crack deflection was occurring around the SMA wires as shown in Figure 10. The crack initiated at the lower left corner of that particular sample and propagated linearly through the matrix thickness until it encountered the SMA wire, at which point the crack changed direction until final fracture. It was evidence that the SMA reinforcements were not affected by cyclic loading. In determining the

relevance of the fatigue behavior of the cast Al-Si composite, the data was normalized with respect to the ultimate tensile strength and compared to normalized data from a conventional cast aluminum alloy, A201 [12]. Trending of the two alloys shows similar fatigue life behavior, an indication that the SMASH alloy has the capacity of rivaling conventional alloys if the porosity can be controlled and strength can be increased. It is expected that the hot-pressing diffusion bonding technique described in section 3.1 would resolve the variability in fatigue life data by eliminating the amount of porosity and improving the mechanical properties.
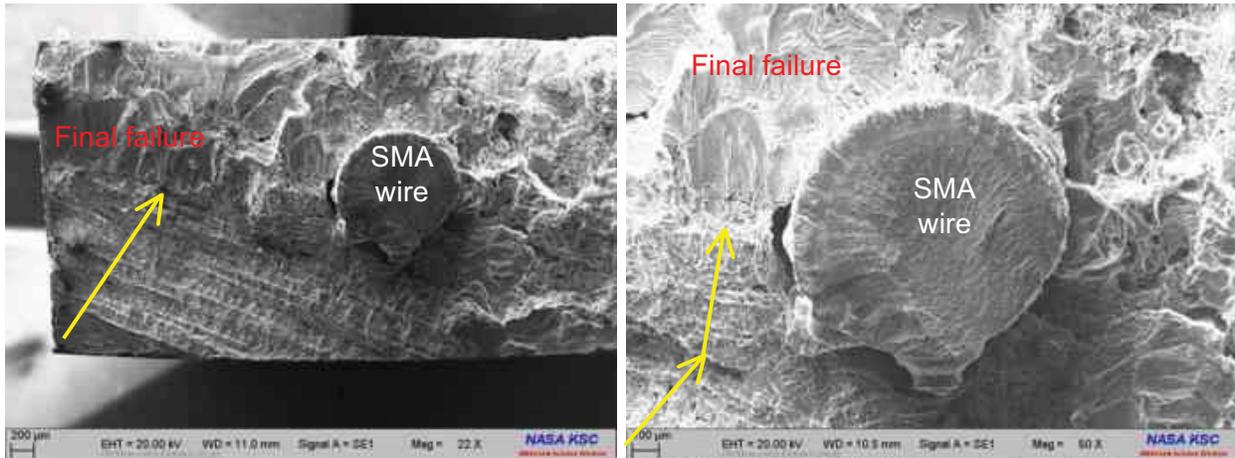

Figure 10. SEM images of Al-Si composite cyclically loaded to failure showing crack deflection around SMA wire. Crack initiated at lower left corner (arrow, left image) and propagated linearly through thickness until it encountered the SMA wire, at which point the crack changed direction (arrows, right image) until final fracture.
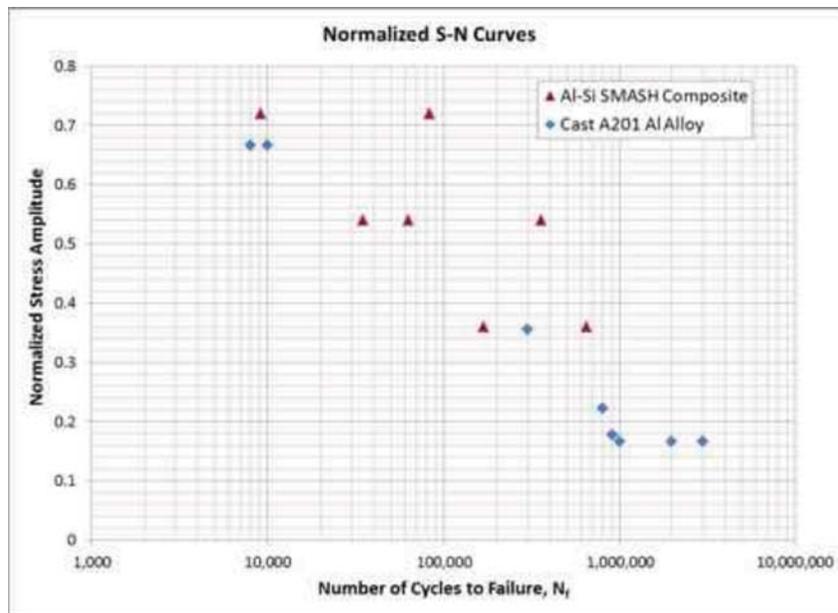

Figure 11. S-N data from the Al-Si SMASH alloy was normalized with respect to the UTS and compared to normalized data of a conventional cast aluminum alloy, A201. Notwithstanding the variability in the SMASH alloy data, both alloys had similar trending of fatigue behavior, an indication that the SMASH alloy has the capacity of rivaling conventional alloys. Low-stress, high-cycle run-out behavior is still to be determined for SMASH alloys.

3.4   Healing Fatigue Cracks in SMASH Aluminum-Based Alloy

Fatigue crack growth tests were conducted on middle tension M(T) and singe edge notch tension ESE(T) specimens to show initiation, propagation and healing of a small fatigue crack on the Al-Si matrix and Al-Si SMASH composites. The M(T) samples were machined based on ASTM Standard E647 from a larger casting of the Al-Si alloy with embedded SMA wires. Each sample had two embedded wires, one on either side of the machined notch, and testing was carried out under parameters described in section 2.2. It was observed that cracking initiated from the notch and propagated in a mostly intergranular manner, following the regions of eutectic phase. Testing was stopped, samples were heat treated to heal the fatigue cracking, and imaged using optical microscopy along the healed crack region. The samples were then re-tested in fatigue using the same parameters as the initial testing. Figure 12 shows the original crack propagation path, the healed crack, and the re-tested crack path. Most notably, the eutectic distribution was modified by heat treatment during the healing process. Although the original crack may have led to a faster crack initiation, and the presence of eutectic near the machined crack still provided a preferred crack path, the local modification of the microstructure proved to be beneficial. Due to the eutectic redistribution near the crack tip, the crack path was altered during subsequent fatigue testing. This secondary crack is characterized by a higher degree of crack deflection, resulting in an overall lower crack growth rate, as summarized in Figure 13. This also indicates that fatigue crack growth rate is highly dependent on the local microstructure surrounding a crack initiation site, and discontinuous eutectic is beneficial for improved fatigue life. It should be noted that the first recorded crack length was at approximately 100k cycles, where the first measureable fatigue crack was seen, not the machined notch length. Variance in post-heal crack length can be attributed to differences in amount of crack healing, as well as the amount of local microstructural changes that occurred during healing.
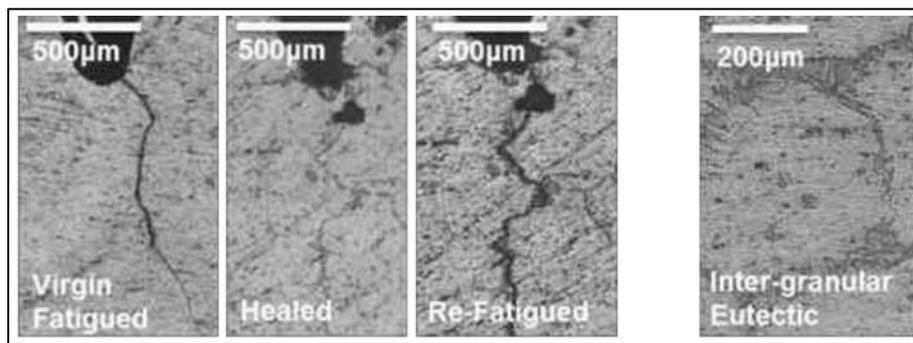

Figure 12. Optical micrographs of cracks extending from a pre-machined notch in M(T) Al-Si SMASH sample after initial testing (left), after healing (middle left) and re-testing after healing (middle right). Healing of the sample led to crack closure as well as altered crack path upon further fatigue cracking. Right image shows a typical intergranular eutectic microstructure of this M(T) sample.
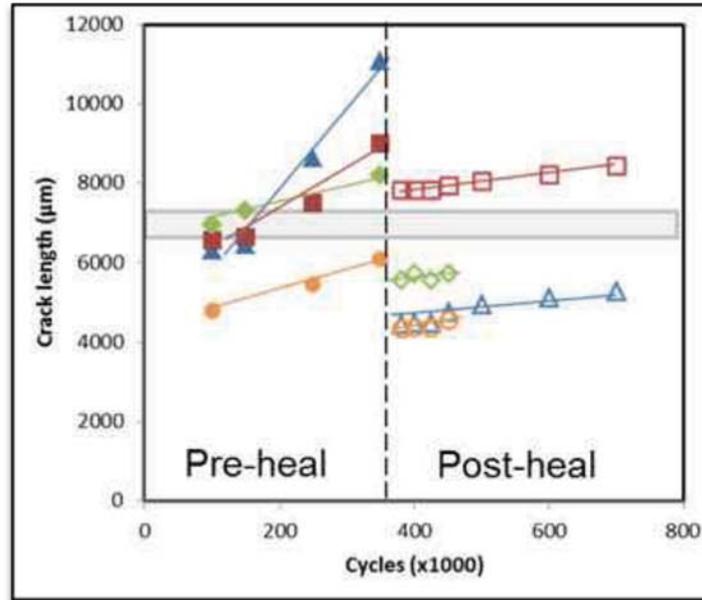
Figure 13. Fatigue crack growth rate from M(T) testing of Al-Si SMASH alloy. All cracks showed some crack closure, as well as reduction in crack growth rate after healing. The grey area represents the location of SMA reinforcement.

To further characterize the fatigue properties of the Al-Si alloy matrix only, ESE(T) samples were machined to ASTM E647. These samples were made using the Al-Si matrix material and did not contain any SMA wires. Sample fabrication and testing procedures are outlined in Section 2.2. Similarly to the M(T) samples, cracking on the ESE(T) samples was observed to occur from the notch, propagating primarily intergranularly through the matrix. After initial testing resulted in crack initiation and propagation, the heat treatment was applied to heal fatigue damage. Samples were re-tested after microstructural evaluation via optical microscopy in order to determine healing efficiency. Figure 14 shows how the crack propagated after healing. While the crack initially propagates intergranularly following the eutectic along the grain boundaries, it changes to transgranular propagation once the crack extended beyond previously healed sections of the matrix material. This behavior is seen due to the liquid-assisted nature of the healing process, in which the liquidus phase is supplied via preferential melting of the eutectic. This liquid phase flows to the cracked region and solidifies there upon cooling. As with the M(T) samples, fatigue crack growth rates were highly dependent on the local microstructure. One ESE(T) sample was re-tested and re-healed after the first initial healing treatment to determine properties after multiple fracture and healing cycles. Figure 15 illustrates crack growth rates in an ESE(T) sample for the Al-Si matrix after two cracking/healing cycles. It was observed that there are changes in crack growth behavior after each repeated healing, indicating a transition from intergranular to transgranular failure mechanism, as seen in the microstructure at point "A" in Figure 14. It can also be noted that each healing event heals less material than previous healing events. This may be due to the absence of SMA wires, which would provide pull-back force in the composite, allowing for more complete reduction in crack length. Fatigue cracks usually propagate transgranularly and it is likely that the liquid-assisted healing treatment creates a network of eutectic phase within the microstructure that affects crack propagation. It was shown that cracks propagate intergranularly along the eutectic network through healed regions and then transgranularly through material that had not been damaged previously. It is an indication that the healing treatment will close existing fatigue

cracks and may deter future crack propagation, but the healed regions are not as effective as virgin matrix regions in crack retardation. This decrease in fatigue damage tolerance may be alleviated by a secondary heat treatment after the healing treatment.
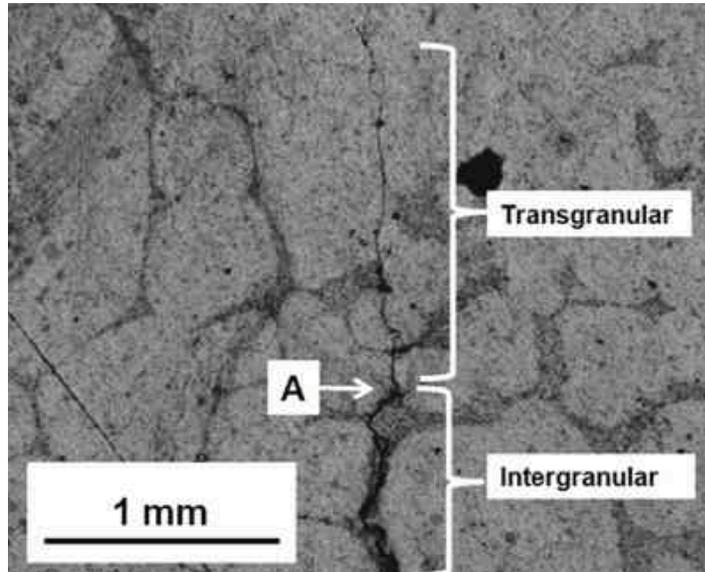

Figure 14: Optical micrographs of crack extending from a pre-machined notch in ESE(T) Al-Si matrix sample. The sample was tested, healed, and re-tested. Healing treatment led to crack closure and after continued testing, resulted in an altered crack path changing from intergranular propagation along the eutectic phase to transgranular propagation through the matrix (point "A").
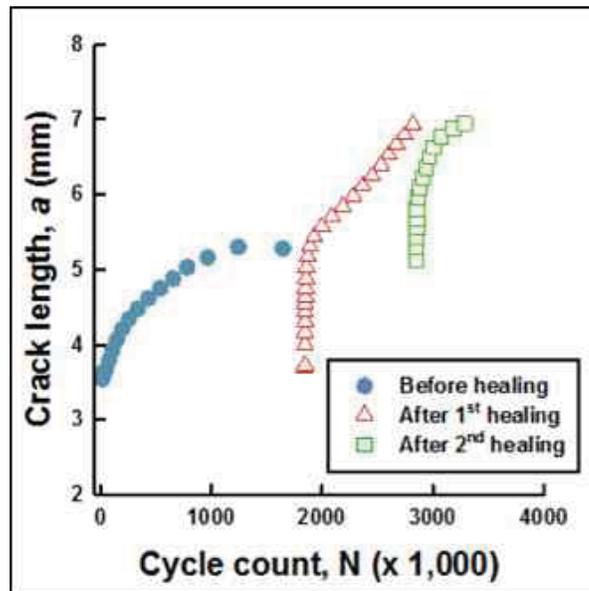

Figure 15: ESE(T) crack growth rate of Al-Si matrix after two healing treatment cycles. All cracks showed some crack closure despite the lack of SMA wires, in addition to a reduction in crack length.

4.0 Conclusions and Future Work

Investigators were able to show that the liquid-assisted healing in aluminum-based SMASH technology could be used to repair fatigue cracks propagating through the matrix. The metal-metal composite materials under development could prove to be candidates to improve damage tolerance of aeronautical structures.

Specific conclusions from this investigation included:
- Demonstrated self-healing of a low-stress high-cycle fatigue crack in a proof-of-concept Sn-Bi matrix composite using the SMA reinforcements for crack closure and low-melting eutectic phase for liquid-assisted healing.
- Proved self-healing in a binary Al-Si matrix alloy with pre-determined eutectic phase and 2 vol% SMA wires. This alloy retained more than 90% ultimate tensile strength (UTS) post-healing and showed adequate wire bonding.
- Fatigue tested the self-healing binary Al-Si composite to create an S-N curve. Porosity within the cast samples attributed to scatter in the data.
- Determined the fatigue behavior of the Al-Si composite and matrix to determine crack propagation after multiple healing cycles.
  - Observed that fatigue crack growth rate is dependent on the local microstructure surrounding the crack initiation site and discontinuous eutectic is beneficial for improved fatigue life.
  - Observed that there are changes in crack growth behavior after repeated healing, transitioning from an intergranular to a transgranular failure mechanism.
- Developed diffusion-bonded fabrication techniques to make multi-ply specimens by isostatically hot pressing thin slices of the matrix and sandwiching SMA reinforcements at the interface. With this method, up to four plies with three reinforcement layers at the interfaces were successfully fabricated.

Future work includes optimizing the processing technique, heat treatment, and fiber reinforcement orientation to improve the fatigue behavior of the aluminum-based SMASH alloy. This investigation has shown that there is an alternative to common repair techniques of fatigued structures, utilizing an external heating source to drive the material to repair itself by forcing crack closure and affording liquid-assisted crack healing at the crack front. The implications of a successful materials system could revolutionize the industry and NASA programs by improving damage tolerance and fatigue life of metals at critical structural locations. The integrated self-repairing approach would improve durability and sustainability of the aerospace material to ensure vehicle safety.

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