Review

Shape Memory Alloy Reinforced Self-Healing Metal Matrix Composites

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Abstract: This paper reviews the synthesis, characterization, healing assessment, and mechanics of NiTi and other shape memory alloy (SMA)-reinforced self-healing metal matrix composites (SHMMCs). Challenges to synthesizing and characterizing the SMA-reinforced SHMMCs and the strategies followed to overcome those challenges are discussed. To design the SMA-reinforced SHMMCs, it is necessary to understand their microstructural evolution during melting and solidification. This requires the knowledge of the thermodynamics of phase diagrams and nonequilibrium solidification, which are presented in this paper for a model self-healing composite system. Healing assessment provides information about the autonomous and multicycle healing capability of synthesized SHMMCs, which ultimately determines their success. Different techniques to assess the degree of healing of SHMMCs are discussed in this paper. Strategies are explored to find the optimum volume fraction of SMA wires needed to yield the matrix and prevent damage to the SMA wires for the most effective healing. Finally, major challenges, knowledge gaps, and future research directions, including the need for autonomous and multicycle healing capability in SMA-reinforced SHMMCs, are outlined.

Keywords: self-healing metal matrix composites; shape-memory alloys; crack closure; phase transformation

1. Introduction

Self-healing refers to the remarkable ability of a material to repair damage inflicted upon it, such as cracks or voids. The concept of self-healing in materials finds its inspiration from the regenerative properties found in biological tissues, which can heal themselves naturally following an injury or bleeding. This fascinating phenomenon has been observed in various living systems, ranging from plants and animals to humans. Scientists and engineers aim to mimic and harness these inherent healing mechanisms to design materials that can autonomously repair themselves when subjected to damage [1]. The concept of self-healing in materials represents the shift of the current materials design strategy from “damage prevention” to “damage management” [2]. Self-healing has so far been most successfully applied to polymeric [3–13], ceramics [14–18], and cementitious materials [19–26]. Developing self-healing properties in metals or metal composites is challenging due to their strong metallic bonds, which provide exceptional strength and stability. However, this strength limits the necessary atom mobility and rearrangement for effective self-healing. Additionally, the small size and low diffusion rate of potential healing atoms, particularly at operating temperatures, further hinder the achievement of self-healing properties in these materials [27].

Self-healing materials find applications in a wide range of industries, including automotive, electronics, aerospace, defense, manufacturing, construction, healthcare, and
consumer goods. Similarly, Self-healing Metal Matrix Composites (SHMMCs) are anticipated to play a crucial role in aerospace, automotive, electronics, energy conversion, and energy storage-related sectors [28–32]. SHMMCs are proposed for spacecraft, extraterrestrial planet surface rovers, and deep-sea applications that are currently impractical or impossible to repair while in service [33]. SHMMCs offer significant potential for application in a wide range of aircraft structural components, providing enhanced performance and durability in fuselage skin, stingers, frames, ribs, longerons, stiffeners, doors, fuel tanks, landing gears, wheel wells, fuel lines, shock struts, empennage structures, avionics enclosures, and floor beams [34]. These advanced materials hold promise in spacecraft structures, including hulls, frames, trusses, panels, and support structures, enabling extended missions with improved reliability. Furthermore, the utilization of SHMMCs in the oil and gas industry could revolutionize structures such as oil-well casings, pipelines, offshore platforms, and drilling equipment, delivering enhanced structural integrity and maintenance capabilities. Similarly, extraterrestrial planet surface rovers could greatly benefit from SHMMCs in various critical structures, encompassing chassis, body panels, wheels, suspension components, robotic arms, and other load-bearing and protective elements, enabling reliable exploration and operation. Additionally, deep-sea applications stand to benefit from the use of SHMMCs in structures like underwater vehicles, submersibles, remotely operated vehicles (ROVs), and offshore oil and gas exploration equipment, enhancing their durability and performance in challenging marine environments.

Shape-memory alloys (SMAs) are among the newest and most popular smart/intelligent materials that can recover their original shape when heated above a critical temperature [35,36]. This paper discusses the synthesis, characterization, healing assessment, and mechanism of NiTi and other SMA-reinforced SHMMCs. When heat is applied to fractured SMA-reinforced SHMMCs, the stretched SMA undergoes phase transformation and returns to its original shape. Compressive healing force exerted by the SMA and predetermined matrix liquefaction at healing temperature help heal cracked SMA-reinforced SHMMCs [37–39].

Various synthesis processes were used to fabricate SMA-reinforced composites, including liquid metallurgy [40–42], powder metallurgy [43–45], ultrasonic consolidation [46,47], hot pressing method [48], and friction stir processing [49]. This paper will provide a review of synthesis processes used to fabricate SMA-reinforced SHMMCs. Different characterization techniques such as optical microscopy (OM) [34,39,50–52], scanning electron microscopy (SEM) [40,53,54], X-ray diffraction (XRD) [55,56], transmission electron microscopy (TEM) [57,58], microindentation [59–61], dynamic mechanical analysis (DMA) [50], differential scanning calorimetry (DSC) [39,50,55,62], etc. were used to characterize SMA-reinforced metal matrix composites. This paper will provide a review of different characterization techniques that are used to characterize SMA-reinforced SHMMCs.

Healing assessment is vital for SMA-reinforced SHMMCs, as it validates their structural application. The evaluation of healing is required to investigate the performance of synthesized SHMMCs. Healing assessment gives an idea about the autonomous and multicycle healing capability of synthesized SHMMCs, which ultimately determines their success. This paper will present techniques to assess the degree of healing for SMA-reinforced SHMMCs. The challenges in conducting the healing assessment tests and strategies for overcoming these challenges will be discussed.

When designing SHMMCs, it is critical to determine the optimum volume fraction of SMA fibers required to yield the matrix, while avoiding damage to the SMA fibers. The effect of pre-strain and optimum orientation and distribution of SMA fibers in the metal matrix required for the most effective healing of SMA-fiber-reinforced SHMMCs are among the other major considerations for the designing purpose. This paper will review an analytical model that determines the minimum volume fraction of SMA fibers required to induce a clamping force at the crack interface during a healing cycle. Various challenges such as improving wetting between the reinforcement and matrix, overcoming oxidation, providing sufficient capillary pressure, and achieving healing of macroscopic damage
without compromising strength or functionality are encountered in the development of SMA-reinforced SHMMCs [37,63–65]. Major challenges, knowledge gaps, and future research directions, including the need for autonomous and multicycle healing capability to develop SMA-reinforced SHMMCs, will be outlined in this paper.

2. Classification of Self-Healing Metal Matrix Composites

SHMMCs are classified on the basis of self-healing mechanism, phase transformation involved during healing, characteristic length scale of the healed damage, and autonomy. SMA clamp-and-melt-based self-healing, solder tube/capsule-based self-healing, and nano-SMA dispersoid-based self-healing are among the self-healing mechanisms studied so far for metal matrix composites. SHMMCs are classified by Manuel [66] based on the type of phase transformation involved during healing, such as solid-state or liquid-assisted healing. According to this classification, SMA clamp and melt and solder tube/capsule-based self-healing are classified as liquid-assisted healing, whereas nano-SMA dispersoid-based self-healing is classified as solid-state healing. Tasan et al. [67] have classified the SHMMCs based on the characteristic length scale of the healed damage. According to their classification, nano-SMA dispersoid-based healing is classified as nanoscale healing, whereas SMA clamp and melt and solder tube/capsule-based healing is classified as macroscale healing. Based on autonomy, SHMMCs can be classified as autonomous or nonautonomous [67]. The autonomous SHMMCs do not require external intervention to repair the damage, whereas nonautonomous SHMMCs do require external intervention, such as the application of heat. According to this classification, nano-SMA dispersoid-based healing is classified as autonomous healing, whereas SMA clamp and melt and solder tube/capsule-based healing is classified as nonautonomous healing. Table 1 shows the classification of self-healing metal matrix composites. The SMA clamp-and-melt-based self-healing concept stands out as the most promising among the macroscale mechanisms because of its ability for limitless repetition. In contrast, the solder tube/capsule-based self-healing approach utilizes encapsulated solder materials that are released to fill and repair cracks. While offering localized healing, its success depends on the careful selection of host matrix and solder materials. The nano-SMA dispersoid-based self-healing mechanism offers a precise nanoscale healing potential but relies on specific material combinations to achieve the desired nanosized coherent particles with shape-memory effects [67].

3. Synthesis of Shape Memory Alloy Reinforced Self-Healing Metal Matrix Composites

This review will consider the following alloys analyzed as matrixes in previous studies:
- Sn-13at.%Bi [39]
- Mg-5.7at.%Zn-2.7at.%Al [39]
- Al-Si, Al-Cu, and Al-Cu-Si [34]
- Al-3at.%Si [68]
- Sn-20wt.%Bi [50,69]
- Sn-13wt.%Bi [70]
- Bi-10wt.%Sn [50,69]
- Zn-0.8Al-0.015Cu [71]
AA2014 (an aluminum alloy consisting of Al-Cu-Si-Mn-Mg-Fe) [72,73] The influence of design factors such as SMA volume fraction, pre-strain of SMA wires, and healing temperature on the healing of SHMMCs has not been thoroughly investigated. Poormir et al. [51,70] and Srivastava et al. [72] conducted experimental studies using the Taguchi method [74] to investigate the impact of design factors, including volume fraction, diameter and pre-strain of SMA, specimen size, and healing temperature, on the healing process of SMA-reinforced SHMMCs.

Manuel [38,39] developed proof-of-concept self-healing composites by using a Tin-based metal matrix (Sn-13at.%Bi) and equiatomic NiTi SMA wires. One percent volume fraction of continuous, uniaxially oriented NiTi SMA wires was used as reinforcement. Surface treatment techniques, including electropolishing of the SMA wires in a solution of 5% perchloric acid and 95% acetic acid, followed by a 5 nm gold sputter-coating, were employed to enhance the bonding between the NiTi SMA wires and the metal matrix. Specially designed clamps were used to maintain tension in the wires during casting, and the clamp/wire assembly was placed in a heated graphite mold coated with boron nitride (BN). BN spray treatment was applied to both the crucibles and molds to prevent carbide formation and facilitate the casting process. The specimens obtained from the casting process were subsequently cooled to room temperature through air-cooling.

Misra [50] fabricated SHMMC made of a Sn-20wt.%Bi matrix with 20% volume fraction pre-strained NiTi wires reinforcement. The NiTi wires underwent etching and flux treatment to enhance the bonding between the metal matrix and NiTi reinforcement. Following the fabrication of single fiber composites through the dip coating of NiTi wire in Sn-20wt.%Bi, the pressure infiltration technique was employed to introduce NiTi wires with a high-volume fraction into the metal matrix. The purpose of fabricating single fiber composites was to avoid wire charring and embrittlement during the pressure infiltration process. Charring can compromise wire properties and effectiveness as reinforcement, while high temperatures and pressures during infiltration can induce wire embrittlement, reducing overall strength, toughness, and durability of the composite material.

Poormir et al. [51] fabricated SHMMCs composed of a Sn-13wt.%Bi matrix with 0.78, 1.55, and 2.33% volume fraction NiTi strip reinforcement. Casting was used as a fabrication technique because of the equipment availability and ease of process control. A metallic mold with internal fixtures was designed and constructed to ensure proper placement of the continuous reinforcement uniaxially within the matrix. The casting process involved melting tin and bismuth in a stainless-steel crucible at 300 °C and pouring the molten alloy into the mold to create bending test samples. However, the presence of a stable titanium oxide layer on the surface of the NiTi strip reduced its wettability, leading to lower interface strength. To overcome this, the SMA strips were treated by immersing them in an aquatic acid solution of 4.8%HF-10.5%HNO₃ for 5 min to remove the oxide layer.

Ferguson et al. [71] fabricated NiTi-reinforced Zinc Alloy ZA-8 (Zn-0.8Al-0.015Cu) composite by Casting technique. Two types of samples, namely the “loop” and “rod” types, were fabricated to investigate the relative effectiveness of two methods for load transfer from the reinforcement to the matrix material. The first method involved transferring the load directly by establishing interfacial bonding between the matrix and reinforcement, while the second method relied on the indirect transfer through a mechanical connection to a bolt integrated within the matrix. For fabricating the “loop” sample, the NiTi wires were treated by fluxing in an aqueous solution of 4.8%HF-10.5%HNO₃ for 5 minutes, followed by washing in distilled water, drying, and coating with Indalloy Flux #2 for 3 min to enhance the bonding between the reinforcement and matrix. The NiTi wires were then trained by heating them in a preform-frame made of steel bars (100 mm × 20 mm × 2 mm) and threaded steel rods (5 mm diameter) to 500 °C for 1 h, followed by quenching in room temperature water for 5 times, in order to create a permanent looping structure that could act as structural support for load transfer. Before casting, the inner bolts were removed, and the frame was positioned in a permanent steel mold, both of which had been preheated to a temperature of 150 °C. Preheating helps to minimize thermal shocks, improve mold
filling, enhance the flowability of the molten alloy, and promote proper solidification of the material during casting. Additionally, preheating can also aid in reducing any potential defects or inconsistencies in the final casted product. The ZA-8 alloy was melted at 600 °C for 1 hour in a BN-coated graphite crucible before being poured into the preheated mold with the preform. The crucible was coated with BN to prevent carbide formation and enhance castability. The resulting synthesized looped sample had reported dimensions of 12.7 mm in thickness and 38.1 mm in width. For the “rod” type sample, threaded steel rods and steel bars of the same dimensions as those used for the looped sample were employed as a frame to fabricate the wound SMA preform. The as-received NiTi wires were used without any surface treatment for fabricating this type of sample. The rod samples were fabricated using identical melting and casting techniques as the looped samples. The dimensions of the synthesized rod sample were reported as 12.1 mm in thickness (or 12.7 mm for the second sample) and 34.9 mm in width.

Fisher et al. [68] fabricated NiTi-reinforced SHMMC by casting (Al-3at.%Si matrix reinforced with two vol% NiTi SMA wires). The fabrication process involved several steps. Initially, a master alloy was prepared by melting high-purity aluminum (99.99% purity, Alfa Aesar, Haverhill, MA, USA) and silicon (99.9999% purity, Alfa Aesar) at 850 °C until a homogenous liquid solution was formed. This master alloy was then cast into a graphite mold. Subsequently, the appropriate proportions of the master alloy and aluminum were melted at 750 °C to form a liquid solution, obtaining an Al-3at.%Si alloy matrix. To fabricate the SHMMCs, the molten matrix material was poured into a specially designed graphite mold that was coated with BN and equipped with a wire holder containing pre-aligned NiTi wires (Ni-49.3at.%Ti, Ø = 0.87 mm, Memry Corporation, Bethel, CT, USA). Prior to casting, the mold was heated to 350 °C to ensure proper temperature conditions. Finally, the cast SHMMC sample was allowed to cool, and subsequent heat treatment was conducted at 592 °C for 24 h to achieve the desired post-healing microstructure.

Srivastava et al. [72] fabricated a hybrid SHMMCs consisting of an AA2014 matrix reinforced with NiTi wires (Ni55Ti45) and solder alloy (Sn60Pb40). To fabricate the composite, a customized steel split die pattern was utilized. First, a steel frame was wound with pre-strained NiTi wires and annealed at 500 °C for 10 min. After that, quenching of the NiTi wires was conducted in cold water at 20 °C so that the NiTi wires memorized their cold state shape. Next, an AA2014 ingot was melted in the melting furnace at 800 °C. The melt was poured directly into the mold cavity, containing a steel frame and wound pre-aligned NiTi wires in the die. The die was designed to allow space for longitudinally filling the solder alloy reinforcement throughout the length of the sample. The solder alloy was loaded externally into the mold, and the opposite end of the hole was sealed with refractory cement to prevent solder alloy drainage during the subsequent healing heat treatment of the composite.

Sharma et al. [75] successfully fabricated a NiTi-reinforced SHMMC using the semi-solid metal process via the rapid slurry formation (RSF) technique, ensuring careful handling to preserve the self-healing effect. The synthesis involved several steps, starting with the preparation of A356 alloy using the RSF process, where a graphite crucible was filled with the alloy billet and subjected to an electrical resistance furnace. Equiatomic NiTi wires were etched, pickled, and coated with molten A356 alloy to enhance their integration with the matrix. These wires were randomly placed in the semi-solid slurry of A356 alloy using a scope, and the slurry–wire mixture was compressed inside a hydraulic press mold to form the composite material. Rapid quenching in water preserved the microstructure and improved the mechanical properties of the alloy. Testing and characterization were performed on the prepared self-healing material. The quenched samples were reheated below the recrystallization temperature to relieve internal stresses, rectify microvoids, and enhance the integration of the reinforcement with the matrix. Controlled heating at a specific temperature induced a phase transformation in the NiTi wire, effectively training it in a new orientation. Additionally, surface finishing processes were applied to enhance the alloy’s surface. The sample was then subjected to controlled loading, resulting in the
creation of the first crack. Maintaining the structural integrity allowed for subsequent recoverability analysis. By placing the cracked and bent sample in a muffle furnace at 363.15 K for 5 min, the activation temperature of the shape memory alloy was reached, causing austenite to transform into martensite and providing the necessary restoring force for partial closure of the crack. The synthesis process aimed to achieve an SHMMC with improved mechanical properties and crack closure capabilities through the incorporation of NiTi wires and the design of the A356 alloy matrix to undergo partial liquefaction at the healing temperature.

Rohatgi was granted three patents [76–78] that introduced the concept of incorporating macro, micro, and nano-sized SMA particles and fibers into metal matrices to induce self-healing characteristics in fabricated composites. The healing mechanism of SHMMCs involves the phase transformation of SMAs, which is associated with shape change that generates compressive forces on the matrix, effectively closing and healing cracks in the material. The patents proposed various methods for triggering self-healing in localized areas, such as electromagnetic induction, microwave, ultrasonic, ballistic, and laser energy. The patents also put forth a range of solidification techniques for the synthesis of SHMMCs, including stir mixing, squeeze casting, pressure and pressureless infiltration, powder metallurgy, and hybrid methods that incorporate stir mixing, wetting agents, ultrasonic mixing, and squeeze casting.

In the future manufacturing of SHMMCs, several techniques, ideas, and employed procedures can be explored to overcome existing challenges. These include additive manufacturing processes such as 3D printing, which offer precise control over material deposition and the ability to incorporate self-healing agents at specific locations. Advanced surface engineering techniques, such as surface modification and coatings, can be employed to enhance the bonding and interaction between the matrix and reinforcement materials, improving the overall healing performance. Nanotechnology can be utilized to design and synthesize nanostructured reinforcements with tailored properties, enabling better integration and interaction with the matrix. Smart manufacturing approaches, such as the integration of sensors and feedback systems, can be adopted to monitor and control the healing process in real time, ensuring optimal healing efficiency. Multi-scale reinforcements, combining different sizes and types of reinforcements, can be employed to achieve synergistic effects and enhance the overall healing capabilities of SHMMCs. Advanced characterization techniques, such as in situ imaging and analysis, can provide valuable insights into the healing mechanisms and help optimize the material design. Hybrid manufacturing processes that combine different fabrication techniques, such as casting, forging, and additive manufacturing, can be developed to leverage the advantages of each method and improve the overall manufacturing efficiency. Sustainable manufacturing practices, including the use of recycled materials and energy-efficient processes, can be implemented to reduce the environmental impact of SHMMC production. Computational modeling and simulation can be employed to predict and optimize the healing behavior of SHMMCs, enabling virtual testing and design optimization before physical fabrication.

3.1. Reinforcement-Metal Wettability

Wettability is a critical factor in the development of SMA-reinforced SHMMCs as it directly influences the interaction between the liquid matrix and SMA reinforcements. It determines the matrix’s ability to penetrate and wet the SMA reinforcements, which significantly affects the quality and performance of the composite. Achieving good wettability is crucial as it promotes effective bonding and facilitates load transfer between the matrix and reinforcements. However, it is important to note that wettability and bonding are not synonymous terms. While excellent wettability, characterized by a low contact angle, is an essential requirement, it does not guarantee a strong bond at the interface. Good wettability can coexist with a weak van der Waals-type low-energy bond [79,80]. Therefore, additional factors such as surface chemistry, interfacial reactions, and mechanical interlocking contribute to the overall bonding strength in SHMMCs.
The poor wettability of metal matrix with NiTi SMA is a significant concern in the development of SHMMCs, which has been addressed by various researchers through different approaches. Manuel [38,39] proposed electrochemical etching of the SMA followed by sputter coating it with gold (Au) to improve wettability. Electroless coating with copper (Cu) [81] or removal of the oxide with an etchant or a flux [82–84] have also been explored to enhance wettability. Ruzek [85] investigated pressure infiltration with an electroless copper coating to improve the wetting behavior of the NiTi surface. However, coating-based approaches have shown limitations because of diffusion of the metal coating into the alloy matrix, resulting in interface deterioration with aging and thermal cycling, making them less popular for solder matrices. Ruzek [85] reported that electroless Cu-coated SMAs exhibit persistent fiber pullout rather than crack bridging in the solder–SMA composite, indicating poor interfacial strength. Pressure infiltration alone can partially overcome non-wetting forces, but because of a lack of bonding, a good fiber–matrix interface cannot be obtained. Good bonding between the matrix and SMA reinforcement was achieved by Misra et al. [50,69], Poormir et al. [51,70], and Ferguson et al. [71] using an etchant (4.8%HF-10.5%HNO_3) to remove the oxide layer from SMA reinforcement. Misra et al. [50,69], and Ferguson et al. [71] conducted an additional step after etching the SMA reinforcement that further enhanced the wettability of the reinforcement with the metal matrix. A phosphoric acid-based flux was applied after etching the SMA reinforcement to remove surface oxides, perform pickling, and enhance surface energy. This flux aimed to prepare the reinforcement for subsequent processing and enhance bonding with the matrix material. The metal matrix and reinforcement bond most effectively when both etching and fluxing have been applied.

3.2. Negative Coefficient of Thermal Expansion (CTE) Materials as Reinforcements for SHMMCs

The use of negative coefficient of thermal expansion (CTE) materials as reinforcement SHMMCs, as proposed by Manuel in a patent [86], presents an interesting concept with potential benefits for crack repair. The two-stage crack repair process involving crack closure from the contraction of the contracting constituent, followed by crack repair during partial liquefaction of the matrix material, is intriguing. However, the application of this concept is currently in the developmental stage, and experimental demonstration of self-healing ability is lacking. The authors reported that the contracting constituent can be either SMA materials (nickel–titanium-based alloys, including high-temperature modifications such as Ti(NiPt), TiHfNi, Ti(NiPd), Ti(NiAu), NiTiSn, and the like, indium–titanium-based alloys, nickel–gallium-based alloys, nickel–aluminum-based alloys, copper-based alloys (e.g., copper–zn–aluminum alloys, copper–aluminum–nickel alloys, and copper–tin and copper–gold alloys), silver–cadmium-based alloys, gold–cadmium-based alloys, manganese–copper-based alloys, indium–cadmium-based alloys, iron-based alloys, (e.g., iron–palladium-based alloys, iron–platinum-based alloys, iron–chromium alloys, and iron–manganese alloys), and the like) or materials that have a negative CTE (examples of materials that exhibit negative CTE are cubic zirconium tungstate (ZrW_2O_8), members of the AM_2O_8 family of materials (where A = Zr or Hf, M = Mo or W), and ZrV_2O_7; A_2(MO_4)_3 is also an example of controllable negative thermal CTE). ALLVAR alloy 30 [87] and trifluoroscurandium (ScF_3) [88] also exhibit negative thermal expansion. Carbon fibers [89] show negative CTE between 20 °C and 500 °C. Quartz (SiO_2) and a number of zeolites [90,91] also show negative CTE over certain temperature ranges.

One limitation of the concept is the availability of suitable materials with negative CTE for reinforcement. While a variety of materials are listed as examples of negative CTE materials, the practicality of using these materials as reinforcement in SHMMCs needs to be further investigated. The availability, cost, and processability of these materials may pose challenges in their practical implementation. Another limitation is the lack of experimental demonstration of self-healing ability using negative CTE materials as reinforcement. Although the concept of exerting compressive force at the cracked surfaces during healing using negative CTE materials is intriguing, there is a need for rigorous experimental testing to validate the effectiveness of this approach. The mechanism and
extent of crack closure, partial liquefaction of the matrix material, and subsequent healing need to be systematically studied to understand the feasibility and limitations of this approach. Furthermore, the concept of using negative CTE materials for crack repair in SHMMCs may require careful consideration of the thermal properties and compatibility between the reinforcement and matrix material. Mismatch in thermal properties, such as CTE, between the reinforcement and matrix material may result in residual stresses, which could affect the overall performance and reliability of the composite. The potential for interface debonding, reduced mechanical properties, and premature failure because of residual stresses should be carefully evaluated.

3.3. Thermodynamics of Solidification and Healing

Understanding the thermodynamics of solidification and healing is crucial for the development and optimization of SMA-reinforced SHMMCs. Solidification transforms the molten metal matrix into a solid-state during cooling, facilitating the healing mechanism. By heating the composite above the SMA’s phase transformation temperature, partial melting of the matrix alloy occurs, allowing it to infiltrate cracks and initiate healing. It is crucial to maintain a specific volume ratio of dendrites to eutectic in the matrix alloy, which ensures that the crack cavities are adequately infiltrated by a liquid phase after heating and healing, thereby providing sufficient capillary pressure [1]. As the SMA returns to its original shape and clamps the crack, the partially molten matrix alloy solidifies, sealing the crack and enabling self-healing. In this section, we will delve into the thermodynamic principles governing the solidification and healing of a NiTi-reinforced Sn-Bi matrix.

(a) Partial Melting and Solidification

Equilibrium phase diagrams for multicomponent material mixtures are valuable tools for predicting the formation of thermodynamically stable phases across various temperature, pressure, and composition ranges. These diagrams provide insights into the phase behavior of the mixtures and aid in understanding the stability of different phases under different conditions. The renowned Gibbs phase rule [92–95], formulated by American physicist Josiah Willard Gibbs in his seminal work “On the Equilibrium of Heterogeneous Substances”, published in parts between 1875 and 1878 [96], relates the number of phases present (P), the degrees of freedom (F), and the number of components (C) under constant pressure as:

\[ P + F = C + 2 \]  

(1)

The Gibbs phase rule (given as Equation (1)) allows for the prediction of the degrees of freedom and the number of phases present in a binary system. The rule predicts that in the Sn-Bi system (C = 2, P + F = 4), two-phase regions (P = 2) with two degrees of freedom (F = 2) can exist. Figure 1 is an equilibrium phase diagram of Sn-Bi, which shows that a two-phase region exists between the liquid solution phase (L) and the solid solution of Sn and Bi. Consequently, the alloy experiences partial melting within the two-phase region for a given composition and temperature.

For a fixed composition in a binary eutectic phase diagram, upon increasing the temperature, melting in the alloy will initiate beyond the solidus temperature and completely melt just above the liquidus temperature. At a specific composition in the phase diagram, the liquidus and solidus temperatures of the alloy coincide and reach their minimum, resulting in congruent melting akin to pure metal. This composition is referred to as the eutectic composition, and the temperature is known as the eutectic melting point. In the Sn-Bi eutectic system, the eutectic composition is approximately Sn-57wt.%Bi, and the eutectic melting point is around 139 °C [97,98]. A binary eutectic alloy has a lower melting point in its eutectic composition than either of its constituent elements. Choosing an alloy with a eutectic phase is essential for designing the matrix of SMA-reinforced SHMMCs. Crystals of the primary phase initiate nucleation and growth when the matrix alloy is cooled from the liquid into a liquid–solid two-phase region, except at eutectic compositions. The lever rule [92,94,95] allows for the design of the matrix alloy for SHMMCs with a
predictable liquid fraction required for the healing of the composite at a given temperature. Conversely, the temperature corresponding to a fixed liquid fraction of the matrix alloy for a given composition can also be calculated by using the equations of the solidus and liquidus lines and the lever rule. It is worth noting that designing the matrix alloy for SHMMCs using the equilibrium phase diagram assumes ideal equilibrium conditions that are rarely found in real systems. Nevertheless, this strategy serves as a reliable starting point for designing the matrix alloy for SHMMCs.

![Tin–bismuth binary phase diagram. Reprinted/adapted with permission from Ref. [50]. Figure 1.](image)

Figure 1. Tin–bismuth binary phase diagram. Reprinted/adapted with permission from Ref. [50].

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(b) Nonequilibrium Solidification

The melting and solidification of matrix alloys, including Sn-Bi in SHMMCs, rarely achieve equilibrium conditions because of the time-dependent kinetic processes such as diffusion [95,99,100]. Micro-segregation occurs across dendrites because of the nonequilibrium solidification of the matrix alloy. During solidification, the resulting liquid becomes enriched in solute, leading to constitutional undercooling ahead of the solidification front. Initially, a spherical solid nucleus forms in the undercooled region of the melt, but as it grows, the spherical morphology becomes unstable, resulting in non-planar (dendritic, columnar) solidification fronts caused by perturbations. As dendrites continue to grow, the inter-dendritic liquid becomes progressively enriched in solute, approaching the eutectic composition. Assuming limited diffusion in the solid, complete mixing in the liquid phase, and equilibrium at the solid-liquid interface, Scheil’s equation (Equation (2)) can be employed to determine the solute distribution within a solidified bar [95,99–101]. Scheil’s equation, also known as the nonequilibrium lever rule, is given by:

\[
C_S^* = k C_O (1 - f_S) ^{k - 1}
\]  

(2)

where \(C_S^*\) is the concentration of the solute in the solid, \(C_O\) the initial concentration of the liquid, \(k\) the partition coefficient, and \(f_S\) the fractional distance (solidified) along the bar.

A nonequilibrium solidification microstructure is beneficial in designing the matrix alloy of SHMMCs. This is because, within the matrix alloy, there will always be inter-dendritic eutectic phases that possess a lower melting point compared to the surrounding matrix. These eutectic phases are capable of melting first, thereby providing a healing liquid that acts as a “welding” agent during the healing process of an off-eutectic composition of matrix alloy. This unique characteristic facilitates the effective repair of cracks or defects in the SHMMCs, enhancing their structural integrity.
(c) Thermodynamics of Healing

Self-healing is a nonequilibrium process in which the restoring thermodynamic force (e.g., diffusion) causes healing by shifting the system away from thermodynamic equilibrium [1]. The healing of the SMA-reinforced SHMMCs can be viewed as a self-organization process that results in increased orderliness or decreased entropy of the material. Placing the system in a metastable state causes it to deviate from thermodynamic equilibrium. When degradation occurs in a system, it breaks the fragile metastable equilibrium and drives the system to its new, most stable state. Metastability is achieved in an SMA-reinforced SHMMC by prestraining the SMA, by heating that causes the phase transformation, and by designing the matrix to partially liquefy at the healing temperature. When heat is applied to the system, the martensitic–austenitic phase transformation of the SMA recovers the strain, resulting in compressive force to the matrix and crack clamping. Furthermore, partial liquefaction of the matrix welds the crack/void via wetting and capillary interactions between the eutectic liquid and the crack/void. These two phenomena result in a more stable configuration for SMA-reinforced SHMMCs by decreasing the entropy (increasing the orderliness) at higher scales. During healing, the entropy production at a particular scale is compensated for by entropy production at another level. Though the orderliness of the SMA-reinforced SHMMC as observed during healing grows (entropy decreases), the excess entropy should be produced at the lower scale. Hence, the strain of the reinforced SMA and volume fraction of liquid during healing can be considered as healing parameters necessary for driving self-healing.

3.4. The Impact of Reinforcement on the Solidification Process in SMA-Reinforced SHMMCs

The impact of reinforcement on the solidification process in SMA-reinforced SHMMCs encompasses a range of factors, including how the wettability of the reinforcement affects the solidified microstructure of the composite [101,102], how the reinforcement affects the viscosity and fluidity of the melts during nucleation and growth [103–106], and how micro-segregation occurs during solidification [103]. When SMAs are presented in the liquid matrix, i.e., Sn-Bi, they can have several effects on the solidification of castings: acting as a solute and diffusion barrier, thereby altering the curvature of the solid–liquid interface; catalyzing heterogeneous nucleation of the solid phase from the melt on the SMA, which reduces the grain size; reducing the latent heat required for solidification, thereby increasing the solidification rate; decreasing the viscosity and fluidity of the melt and giving it thixotropic properties under certain conditions; restricting fluid convection because of the narrow interstices between the SMAs, which influences the solidification structure; affecting morphological instabilities such as planar-to-cellular dendritic solidification; influencing the dendrite structure; affecting grain size because of the heterogeneous nucleation or restricted spaces between the SMAs that restrict growth; and affecting micro-segregation in the matrix [102].

Table 2 shows the synopsis of research on the synthesis of SMA-reinforced SHMMCs.

<table>
<thead>
<tr>
<th>Year, Author</th>
<th>Matrix Alloy</th>
<th>Reinforcement (Pre-Strain)</th>
<th>Bonding and Special Distribution of SMA</th>
</tr>
</thead>
<tbody>
<tr>
<td>2007, Manuel [39]</td>
<td>Mg-5.7at.%Zn-2.7at.%Al</td>
<td>1% continuous, uniaxially orientated martensitic NiTi wire of 190.5 µm diameter (percentage of pre-strain is not given)</td>
<td>The wires were sputter coated with 5 nm of gold to increase the wettability of the wires. The wires were threaded through specially made clamps to hold them in tension during casting.</td>
</tr>
</tbody>
</table>
Table 2. Cont.

<table>
<thead>
<tr>
<th>Year, Author</th>
<th>Matrix Alloy</th>
<th>Reinforcement (Pre-Strain)</th>
<th>Bonding and Special Distribution of SMA</th>
</tr>
</thead>
<tbody>
<tr>
<td>2009, Ruzek [85]</td>
<td>Sn-20%Bi, Bi-10%Sn</td>
<td>NiTi SMA short fibers with a diameter of 100 microns (no pre-strain)</td>
<td>Poor wettability of Copper-coated NiTi; NiTi fibers were randomly oriented.</td>
</tr>
<tr>
<td>2013, Misra et al. [50,69]</td>
<td>Sn-20wt.%Bi, Bi-10wt.%Sn</td>
<td>20% by volume of NiTi SMA fibers (wires were etched and flux treated) of 100 µm and 500 µm diameter (4.5% pre-strain as provided by the manufacturer)</td>
<td>Bonding between matrix and NiTi wires was improved; NiTi wires were uniaxially oriented, but the pattern seems to be random; No proof of whether pre-strain was retained in the fabricated composites.</td>
</tr>
<tr>
<td>2015, Ferguson et al. [71]</td>
<td>Zn–0.8Al–0.015Cu</td>
<td>NiTi SMA strips (strips were etched and flux treated) with different reinforcement vol.% (0.78, 1.55, and 2.33); dimension of the strip is 0.762 mm × 0.254 mm. Different pre-strains (0%, 2%, or 6%) of the wires</td>
<td>The preform frame consisting of 100 mm × 20 mm × 2 mm steel bars and 5 mm diameter threaded steel rod was used to train the wires.</td>
</tr>
<tr>
<td>2018, Poormir et al. [70]</td>
<td>Sn-13wt.% Bi</td>
<td>0.92, 1.54, 2.01 vol% NiTi SMA wires (wires were etched and flux treated) of 0.38 mm diameter (no pre-strain)</td>
<td>Good bonding; a custom-made metallic mold was designed and constructed with internal fixtures to keep the SMA strips in desired position and direction.</td>
</tr>
<tr>
<td>2018, Fisher et al. [68]</td>
<td>Al-3at.%Si</td>
<td>2 vol% NiTi SMA of 0.87 mm diameter; knots were tied to act as mechanical anchors and compensate for the low chemical bonding between the reinforcement and matrix (no pre-strain)</td>
<td>A custom-designed graphite mold coated with BN and equipped with an end-wire holder containing pre-aligned NiTi wires (Ni-49.3at.%Ti, Ø = 0.87 mm).</td>
</tr>
<tr>
<td>2020, Srivastava et al. [72]</td>
<td>AA2014 matrix</td>
<td>NiTi wires vol% (0.5%, 1.3%) and solder alloy; wire diameters (0.47 mm, 0.96 mm)</td>
<td>A steel frame was wound with pre-strained NiTi wires and annealed at 500 °C for 10 min. After that, the NiTi wires were quenched in cold water at 20 °C so that they memorize their cold state shape.</td>
</tr>
</tbody>
</table>

4. Characterization of Shape Memory Alloy Reinforced Self-Healing Metal Matrix Composites

SMA-reinforced SHMMCs were characterized by different characterization techniques such as optical microscopy (OM) [33,34,39,50–52,68,70,86,107], scanning electron microscopy (SEM) [33,34,39,50,69,72,73,85,108], energy dispersive X-ray spectroscopy (EDS) [33,50,69], X-ray photoelectron spectroscopy (XPS) [50], inductively coupled plasma–atomic emission spectroscopy (ICP-AES) [33,39], X-ray diffraction (XRD) [50], microindentation testing [39,50,73], dynamic mechanical analysis (DMA) [50], and differential scanning calorimetry (DSC) [33,39,50]. Among these techniques, OM and SEM have been the most commonly used techniques for characterizing SMA-reinforced SHMMCs, indicating their significance in understanding the composite’s structure and properties. Evaluating the advantages and limitations of the different characterization techniques used to characterize SMA-reinforced SHMMCs is critical in providing comprehensive insights into these composites’ complex behavior and performance. This section will provide a critical analysis of the existing characterization techniques used in the field, shedding light on their strengths and weaknesses, and providing valuable guidance for a better understanding of the structure and properties of these composites.
Optical Microscopy (OM):

OM is the most commonly used characterization technique to characterize SMA-reinforced SHMMCs. It is performed on the specimens to confirm the expected microstructures, detect porosity, and inspect the interface between the matrix material and SMA. OM [39] was employed to investigate the microstructures of Sn-based proof-of-concept SHMMCs in both as-cast and heat-treated conditions. Heat treatment at 169 °C for 24 h was applied to homogenize the dendritic as-cast microstructure and achieve a microstructure resembling that of healed samples, enabling direct comparison without casting effects. The micrographs revealed that the SMA wires appeared as large circles, with dark regions indicating Sn-rich phases and lighter areas representing eutectic phases. The micrographs suggested that heat treatment resulted in a more uniform structure compared to the as-cast condition. Moreover, during annealing, eutectic liquid tended to form around the SMA wires, leading to insufficient bonding between the metal matrix and the SMA wires. This finding highlights the importance of using OM to identify and understand the microstructural changes that occur during the fabrication process in SHMMCs.

OM was used to evaluate the effectiveness of surface treatment methods in improving the interface bonding between the matrix and reinforcement in SHMMCs. Some studies [52,85] observed weak bonding between the matrix material and the NiTi SMA wires. This is because NiTi wires have stable nickel and titanium oxide on the surface, which reduces the wettability of the NiTi wires in the matrix, thus weakening the interface strength. Figure 2a–c [52] illustrate the regions surrounding the SMA wires in NiTi-reinforced self-healing Al-A380 matrix composites. The micrographs reveal the SMA wires as circular structures. It is evident from these figures that there is a significant lack of bonding between the Al matrix and the SMA wires. Etching the NiTi wire with an aqueous solution of 4.8%HF-10.5%HNO₃ and then fluxing with phosphoric-acid-based flux (Indalloy Flux #2, Indium Corporation of America, Clinton, NY, USA) was used [50,69,71] as an efficient method to remove the oxide layer from the NiTi surface.

![Figure 2](image_url)

Figure 2. (a–c) Optical micrograph of the area around NiTi SMA wires. Reprinted/adapted with permission from Ref. [52]. 2014, Pradeep Rohatgi.

An optical microscopic image of the impact of applying an etch solution on the strengthening of matrix–strip bonding is shown in Figure 3 [51]. The image shows that using the etch solution to remove the oxide layer from the surface of the reinforcement improved the bonding between the matrix alloy and reinforcement. These examples highlight the capability of OM to evaluate the effectiveness of surface treatment methods in improving the interface between the matrix and reinforcement in SHMMCs.

SHMMCs were investigated via optical microscope after they were tested under different loading conditions. The optical micrograph, as reported by [33], revealed debonding occurrences between an Al-3at.%Si matrix and NiTi wires (V_f = 4.43%) following tensile testing. Debonding was identified during the tensile testing through distinctive indicators such as cracking sounds and stress reduction at different strain levels. It is assumed that the decreased healing percentages observed in SHMMCs with higher volume fractions of NiTi wires are a result of this debonding phenomenon. Furthermore, debonding is expected to impede the wires from clamping the crack, particularly when the temperature rises during heat treatment for healing purposes.
was the more reactive element addition, and Cu was the thermodynamically less reactive
alloy element. This was attributed to the increased bond area and load-carrying capability of the
reactive alloy element, as depicted in Figure 5. This improvement was attributed to the increased bond area and load-carrying capability of the
alloy element addition with lower oxide formation free energy compared to the base element, in order
to promote effective healing. The presence of the reactive element in the base element
reduces the formation of the passivating surface oxide layer on the exposed surfaces of the
alloy when the alloy is in its molten state, leading to the formation of a strong chemical
bond. The reactive element hypothesis was tested using the model systems antimony-
copper (Sb-Cu) and antimony-zinc (Sb-Zn) (Sb served as the base element here, while Zn
was the more reactive element addition, and Cu was the thermodynamically less reactive
element addition based on the change in Gibbs Free Energy for the respective reaction). The
mechanical testing and microstructure analysis revealed that the incorporation of a highly
reactive alloy element led to enhanced bonding effectiveness, as depicted in Figure 5. This
improvement was attributed to the increased bond area and load-carrying capability of the

Figure 3. A cross-section optical micrograph of the interface between the Sn-Bi matrix and SMA
strips, with (a) 5× and (b) 20× magnification. Reprinted/adapted with permission from Ref. [51]. 2018, Poormir et al.

In Figure 4 [68], optical micrographs of the SHMMC samples subjected to fatigue
testing are presented. As depicted in Figure 4a-d, during the initial testing phase, crack
propagation primarily occurs along the intergranular region, following the eutectic zones
of the composites. However, after the healing process of the specimen, the crack path
becomes more convoluted. This change in crack path can be attributed to the morphological
alterations and growth of the Al-3at.%Si microstructure caused by heat treatment, which
reroutes the crack propagation. As a result of these modifications, the sample exhibits
improved toughness.

Figure 4. Optical micrographs of an untested SHMMC specimen (a), a post-fatigue cracking
specimen (b), a post-healing specimen (c), and a specimen after a second M(T) fatigue test (d).
Reprinted/adapted with permission from Ref. [68]. 2018, Fisher et al.

OM has been used to evaluate the bonding between the cracked surfaces after healing.
The bonding between the cracked surfaces after healing is critical in regaining the structural
integrity of the composites. Oxide layers that form immediately at the cracked surfaces
upon exposure to air can prevent bonding of the cracked surfaces. To address this issue,
Fisher et al. [107] proposed a thermodynamic approach to improve healing at the solid-
liquid interface. The approach involved designing alloys with a reactive element alloying
addition with lower oxide formation free energy compared to the base element, in order
to promote effective healing. The presence of the reactive element in the base element
reduces the formation of the passivating surface oxide layer on the exposed surfaces of the
alloy when the alloy is in its molten state, leading to the formation of a strong chemical
bond. The reactive element hypothesis was tested using the model systems antimony-
copper (Sb-Cu) and antimony-zinc (Sb-Zn) (Sb served as the base element here, while Zn
was the more reactive element addition, and Cu was the thermodynamically less reactive
element addition based on the change in Gibbs Free Energy for the respective reaction). The
mechanical testing and microstructure analysis revealed that the incorporation of a highly
reactive alloy element led to enhanced bonding effectiveness, as depicted in Figure 5. This
improvement was attributed to the increased bond area and load-carrying capability of the
metallic system. The formation of stable oxides through the reduction of the passive oxide layer on the parent metal surface played a crucial role in establishing a strong chemical bond across the interface, thereby contributing to the enhanced bonding observed. However, the effectiveness of this approach in SMA-reinforced SHMMCs has yet to be explored. This finding demonstrates the potential of OM in evaluating the effectiveness of healing mechanisms and bonding at the interface in SHMMCs.

OM plays a crucial role in characterizing SMA-reinforced SHMMCs, providing insights into microstructural changes, interface bonding, debonding, and crack propagation. However, OM has limitations that need to be considered. First, OM provides only surface-level information, relying on reflected light, which may not accurately represent the internal microstructure of SHMMCs, leading to incomplete or inaccurate characterization. Second, OM may lack sufficient resolution to clearly distinguish between phases or microstructural features in complex systems. Third, OM provides a two-dimensional view, limiting the characterization of complex three-dimensional microstructures and interface bonding in SHMMCs, potentially missing important information in the third dimension. Fourth, OM may have limitations in detecting and characterizing very small defects, such as microcracks or dislocations. Fifth, OM has limitations in studying dynamic or time-dependent behaviors of SHMMCs and may not capture in situ or real-time behavior during deformation or thermal cycling. Sixth, OM provides limited information about the chemical composition of materials, which is essential for understanding the atomic-level behavior and performance of SHMMCs. It is important to consider these limitations to obtain a comprehensive understanding of SMA-reinforced SHMMCs and complement OM with other advanced characterization techniques.

Scanning Electron Microscopy (SEM):

Scanning electron microscopy (SEM) offers superior performance compared to optical microscopy in terms of magnification, resolution, depth of focus, and observable features, making it a preferred choice for characterizing the microstructure of SHMMCs when cost is not a limiting factor. Figure 6a [69] shows the SEM microstructure of Sn-20wt.%Bi reinforced with 20 vol% of NiTi wires at a magnification of 300×. Figure 6b provides a closer view at a magnification of 1000×, highlighting a specific area within the microstructure marked in Figure 6a. The figures clearly depict that NiTi wires are well-bonded with the metal matrix, and the presence of a light phase in the matrix is enriched in bismuth.

Figure 7 [69] shows the elemental mapping of Sn-20wt.%Bi-NiTi SHMMC close to the interface. The magnified area under investigation captured a eutectic region in the Sn-20wt.%Bi matrix. A distinct interface was observed at the boundary of NiTi and the Sn-20wt.%Bi matrix. The presence of an inter-metallic zone consisting of Sn-Ti-Ni was found by mapping elemental pairs, which proves that good wetting had occurred at the interface after surface treatment of NiTi Wires. A good interface can transfer the load better from the fiber to the matrix during healing.
Scanning electron microscopy (SEM) offers significant advantages in terms of magnification, resolution, and elemental mapping for microstructure characterization of SHMMCs, making it a preferred choice when cost is not a limitation. The SEM images presented in Figures 6 and 7 provide clear evidence of the capabilities of SEM in revealing the microstructural details of Sn-20wt.%Bi-NiTi SHMMC, supporting the claim that SEM is a valuable tool for characterizing the microstructure of such materials.

Elemental mapping of Sn-20wt.%Bi-NiTi SHMMC close to fiber–matrix interface. Reprinted/adapted with permission from Ref. [69]. 2022, Salowitz et al.

SEM offers significant advantages in terms of magnification, resolution, and elemental mapping for microstructure characterization of SHMMCs, making it a preferred choice when cost is not a limitation. The SEM images presented in Figures 6 and 7 provide clear evidence of the capabilities of SEM in revealing the microstructural details of Sn-20wt.%Bi-NiTi SHMMC, supporting the claim that SEM is a valuable tool for characterizing the microstructure of such materials.

X-ray Diffraction (XRD):
X-ray diffraction (XRD) was employed for phase characterization, as reported in [50]. Qualitative analysis of XRD patterns revealed a gradual decrease in the intensity of peaks associated with tin, eventually disappearing with an increase in bismuth content. Moreover, certain compositions exhibited pronounced texturing effects. The unique crystal structures, lattice parameters, and XRD patterns of each phase enabled accurate identification of the composition of the phases through XRD characterization. Though XRD has limitations, including the need for careful sample preparation, and the potential cost of equipment and maintenance, XRD remains a powerful tool for phase analysis in material-characterization studies.

Differential Scanning Calorimetry (DSC):
Differential scanning calorimetry (DSC) was utilized to determine the transformation temperatures of the SMAs. The DSC test involved maintaining a constant heating and cooling rate during the measurement. The martensite finish temperature (M_s), martensite start temperature (M_t), austenite start temperature (A_s), and austenite finish temperature (A_f) of the SMA reinforcement were determined using the ASTM Standard F 2004-05 (2010) [39,50,109] approach, which involves identifying the intersection of tangent lines on the steepest slope of the DSC curve. As reported in [39], DSC test results for the TiNi SMA (Ti-49.4at.%Ni) annealed at 500 °C for 3 h revealed that the M_t, M_s, A_s, and A_f temperatures of the SMA reinforcement were measured as 58, 71, 88, and 105 °C, respectively. DSC, while a valuable technique for determining transformation temperatures and enthalpy...
changes in SMAs, has certain limitations. It primarily provides information about the thermodynamic properties, such as the transformation temperatures and enthalpy changes, without providing detailed insights into the microstructure or phase evolution of the material. Additionally, the accuracy and reproducibility of DSC results can be influenced by various experimental factors, including heating and cooling rates, sample size, and sample purity, which must be carefully controlled to ensure reliable results.

Findings from the studies reveal that OM, SEM, EDS, XRD, and DSC are viable characterization techniques for characterizing SMA-reinforced SHMMCs. Instrument costs, skilled workforce, and proper sample preparation strategies are critical factors to consider during the characterization of SMA-reinforced SHMMCs. It is clear from this study that different characterization techniques can be used to determine the structure of the matrix materials, SMA reinforcement, and interface of SMA wires and matrix materials. Characterization revealed how the structures are related to the macroscopic properties of the SMA-reinforced SHMMCs. For future characterization of SHMMCs, characterization of healing of microvoids and strains using strain imaging, non-destructive tests such as X-ray, CT scan, and ultrasonic testing need to be conducted. The above study shows that characterization of SMA-reinforced SHMMCs is essential to predict the performance of the different elements and estimate the relations among processing-structure-property-performance of the composites to achieve greater efficiency in synthesizing them.

5. Healing Assessment of Shape Memory Alloy Reinforced Self-Healing Metal Matrix Composites

5.1. Healing Assessment Techniques

The healing efficiency of a composite is assessed by comparing the mechanical properties of the virgin composite with the post-heal mechanical properties of the same composite, using the following relationship:

\[ \frac{MP_{\text{healed}}}{MP_{\text{virgin}}} \times 100\% = \% \text{ Healed} \]

Sample testing:

Various mechanical tests, including tensile, bending, and fatigue tests, were performed to study the healing behavior of SHMMCs, as detailed below.

Tensile testing:

Several earlier studies [52,71] on NiTi-reinforced SHMMCs did not adhere to standard methods for fabricating tensile test specimens to assess healing efficiency. Manuel [39], Wright et al. [34], Fisher et al. [68], and Poormir et al. [70] fabricated sub-size tensile test specimens to demonstrate healing efficiency, whose dimensions are specified in ASTM E8M: Standard Test Methods for Tension Testing of Metallic Materials [110]. The specifications of sub-size tensile test specimens are provided below (Figures 8 and 9). Table 3 presents the dimensions of the rectangular tension test specimen as shown in Figure 8, while Table 4 provides the dimensions of the round tension test specimen as shown in Figure 9.

Table 3. Different dimensions of the rectangular tension test specimen as illustrated in Figure 8.

<table>
<thead>
<tr>
<th>Dimensions (mm)</th>
<th>G—Gauge length</th>
<th>25.0 ( \pm ) 0.1</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>W—Width</td>
<td>6.0 ( \pm ) 0.1</td>
</tr>
<tr>
<td></td>
<td>T—Thickness</td>
<td>6</td>
</tr>
<tr>
<td></td>
<td>R—Radius of fillet, min</td>
<td>6</td>
</tr>
<tr>
<td></td>
<td>L—Overall length, min</td>
<td>100</td>
</tr>
<tr>
<td></td>
<td>A—Length of reduced section, min</td>
<td>32</td>
</tr>
<tr>
<td></td>
<td>B—Length of grip section, min</td>
<td>30</td>
</tr>
<tr>
<td></td>
<td>C—Width of grip section, approximate</td>
<td>10</td>
</tr>
</tbody>
</table>
Table 4. Different dimensions of the round tension test specimen as illustrated in Figure 9.

<table>
<thead>
<tr>
<th>Dimensions (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>G—Gauge length</td>
</tr>
<tr>
<td>D—Diameter</td>
</tr>
<tr>
<td>R—Radius of fillet, min</td>
</tr>
<tr>
<td>A—Length of reduced section, min</td>
</tr>
</tbody>
</table>

Rectangular Tension Test Specimen:


Round Tension Test Specimen:


Bending test:

Ruzek [85], Misra et al. [50,69], Poormir et al. [51], and Srivastava et al. [72] fabricated bending test [111–113] samples to assess the healing of SHMMCs. The bending test is advantageous as it provides an easily visible crack on the surface of the test sample and allows for partial damage without complete fracture. It also allows the performing of shape recovery tests to demonstrate healing in the samples [50,69,85]. Figure 10 shows the schematic of a three-point bend test setup.

Figure 10. The three-point bend test setup used by Ruzek and Misra. The grey bar represents the test specimen, the blue arrows represent the force direction during the test, and the red arrows represent the relevant measurements. Reprinted/adapted with permission from Refs. [50,85]. 2009, Andrew Ruzek, and 2013, Shobhit Misra.
Fatigue testing:
Fisher et al. [68] fabricated middle-tension (M(T)) fatigue-crack growth rate samples using geometry from ASTM E647: Standard Test Method for Measurement of Fatigue Crack Growth Rates [114]. The fatigue tests were performed on an MTS 321.12 hydraulic load frame with a strain-controlled setup and were terminated after 350,000 cycles. Healing cycles were carried out at 592 °C for 24 h before retesting to determine the percentage of healing, with crack size quantification performed optically at the midpoint of each sample.

5.2. Healing Assessment Results
Manuel [39] used Sn-13at.%Bi and Mg-5.7at.%Zn-2.7at.%Al as matrix materials, along with 1 vol% continuous, uniaxially oriented NiTi SMA wires as reinforcement, to synthesize SHMMCs. The NiTi SMA wires were sputter coated with 5 nm of gold to enhance matrix and fiber wettability. In the case of Sn-13at.%Bi-NiTi, the crack formed during tensile testing was successfully closed, and the healed specimen exhibited a recovery of over 94% of its original tensile strength. However, for Mg-5.7at.%Zn-2.7at.%Al-NiTi, the composite showed only partial crack closure after healing due to rough crack walls preventing complete closure. The limited fraction of SMA reinforcement wires in the Mg-based alloy was unable to generate sufficient force to overcome the strength of the matrix material.

Wright et al. [34] conducted research aimed at demonstrating a proof-of-concept shape-memory alloy self-healing (SMASH) technology for aeronautical applications, specifically targeting fatigue cracks propagating through the matrix. The team designed thermodynamic compositions for three different alloy systems, Al-Si, Al-Cu, and Al-Cu-Si, to produce aluminum alloy matrices with predetermined eutectic phases. The local microstructure near the crack-initiation site was found to have a significant impact on the fatigue crack growth rate, with the presence of a discontinuous eutectic phase favoring longer fatigue life. Interestingly, the behavior of the crack as it grew shifted from an intergranular to a transgranular failure mechanism after repeated healing. However, Al-Cu or Al-Cu-Si alloys showed minimal signs of healing, indicating the complexity of the self-healing process in these systems.

By conducting finite element simulations, Zhu et al. [115–117] investigated the effect of SMA reinforcement, the matrix’s softening property, and the SMA’s pre-strain on the healing of SHMMCs. The study found that prestraining of the SMA reinforcement and softening property of the matrix material at healing temperature is beneficial for crack closure and self-healing. SMA provides a more vital shape-recovery ability than non-transforming materials. Mohsen et al. [118] conducted a study using 3D FEM/XFEM modeling to examine the impact of NiTi SMA wires on crack closure and healing performance in a self-healing mechanism involving microcapsules with a glass shell and healing agent. They observed that a higher volume fraction of SMA wires improved the healing process and increased ultimate fracture stress. Applying a 1% pre-strain to the SMA wires resulted in complete crack closure and improved adhesion of the healing agent. Increasing the wire radius or volume fraction enhanced fracture stress and the ability to close the crack. The thickness of the microcapsule affected its likelihood of breaking, while the microcapsule radius did not have a significant impact. Moreover, a stronger interface increased the probability of microcapsule breakage.

Misra [50] fabricated an SHMMCS composed of Sn-20wt.%Bi and Bi-10wt.%Sn matrices reinforced with 20% volume fraction of NiTi wires. The Bi-10wt.%Sn-NiTi composite exhibited self-healing properties, with the ability to heal at 145 °C while providing a 23% eutectic healing liquid to close cracks. All bent samples fully recovered from flexural strain, as shown in Figure 11. Figure 11a illustrates crack closure upon the application of heat, while Figure 11b demonstrates the straightening of the specimen with the application of heat. As heat permeated through the samples and elevated their temperatures, the shape-memory effect of the NiTi was activated, resulting in the straightening of the specimens and the closure of the cracks. It is worth noting that the complete recovery of flexural strain occurred well before the temperature approached the healing temperature (T_H = 145 °C) for
the alloy. In addition, 92% of the flexural strength for Sn-20wt.%Bi-NiTi SHMMC and 88% for Bi-10wt.%Sn-NiTi SHMMC were recovered within an hour of healing. Figure 12 shows the snapshot of the healing L-shape restoration and crack closures of a bent Sn-20wt.%Bi-NiTi bar at equal intervals recorded by Misra. The image shows the progressive restoration of the bar’s shape and the closure of cracks. The restoration of the bar’s geometry took approximately 36 s and was fully accomplished within 74 s after placing the sample on a hot plate. As the temperature increased, the eutectic material within the matrix underwent melting, enabling its flow into the cracks and subsequent welding of the structure. The angular rate of recovery during this process was measured to be approximately 1 degree per second.

Figure 11. Bending Test Specimens tested by Rohatgi and Misra: (a) crack closure with applied heat and (b) straightening of specimen with applied heat.

Figure 12. Snapshots of healing L-shape restoration and crack closures of bent Sn-20wt.%Bi-NiTi bar at equal intervals taken by Misra. Reprinted/adapted with permission from Ref. [69]. 2022, Salowitz et al.

Poormir et al. [70] used the Taguchi method to study design factors (volume fraction of SMA strip, pre-strain of SMA NiTi strip, and healing temperature) on NiTi SMA-reinforced Sn-13wt.%Bi alloy SHMMCs. They aimed to optimize mechanical properties with minimal experiments. This study yielded the following four conclusions: (1) optimal conditions for ultimate tensile strength healing efficiency were 2.33% SMA volume fraction, 6% pre-strain, and 190 °C healing temperature; (2) ductility healing efficiency was optimized with 0.78% SMA reinforcement, 0% pre-strain, and 190 °C healing temperature; (3) higher SMA volume fraction improved ultimate tensile strength healing efficiency but decreased ductility healing efficiency; increased healing temperature improved both properties; pre-strain affected ultimate tensile strength only; and (4) >2.33% SMA volume fraction caused manufacturing difficulties and internal structural damage. Higher pre-strains and healing temperatures led to sample instability.

Ferguson et al. [71] synthesized Zinc based SHMMCs by embedding NiTi SMA wires into Zinc ZA-8 die-casting alloy (Zn-0.8Al-0.015Cu). The objective of the study was to assess the efficacy of two load transfer methods in the healing mechanism: direct load transfer through interfacial bonding between the metal matrix and SMA reinforcement, and indirect transfer via mechanical load transfer to a bolt embedded in the matrix. The findings revealed an increase in the volume fraction of SMA reinforcement resulted in a rise in ultimate tensile strength, but only mechanical load transfer samples with higher strain hardening showed significant regained strength after heat treatment for healing. Notably, the investigation identified two critical flaws in the healing system. Firstly, it was necessary
to encase the sample in sand to maintain structural stability during the healing process. Secondly, the healing process resulted in only up to a 30% recovery of the original ultimate tensile strength and ductility in the fabricated SHMMCs, indicating limitations in achieving full restorative potential.

Fisher et al. [68] synthesized SHMMCs by incorporating 2 vol% NiTi SMA wires reinforcement into Al-3 at.% Si matrix. Al-3at.%Si was chosen as the matrix material due to its composition, providing 20% liquid and 80% solid at the healing temperature, ensuring structural stability. The authors provided compelling proof for the healing mechanism of the SHMMCs under both tensile and fatigue conditions, through a combination of experimental and computational approaches. The SHMMCs samples were carefully heat-treated for 24 hours at 592 °C to achieve a post-heal microstructure. Their research revealed that the SHMMC could recover over 90% of its strength after a healing cycle, with an average healing efficiency of 91.6% under tensile conditions. Their investigation into fatigue behavior demonstrated promising outcomes in the healing of fatigue cracks, with consistent reductions in crack length observed across all conducted fatigue tests as a result of the healing cycle and the subsequent bonding of the matrix to itself. Additionally, the healing process led to a decrease in the fatigue crack growth rate.

Srivastava et al. [72] employed a combination of low melting point alloy and SMA as healing agents in the matrix to demonstrate self-healing in hybrid SHMMCs. The investigation focused on an AA2014 matrix reinforced with NiTi SMA wires and Sn60Pb40 solder alloy. They utilized the Taguchi L8 mixed orthogonal array technique to identify the influential design factors such as the volume percent of SMA wires, specimen size, and diameter of SMA wires on the healing response. The healing duration was found to be the most influential design factor. The researchers reported a 100% recovery in crack width, 96.95% recovery in crack depth, and 73.76% recovery in flexural strength. Another study [73,108] on the same hybrid SHMMC demonstrated a 100% recovery in crack width, 87.85% recovery in crack depth, and 75.40% recovery in flexural strength. The studies involved the utilization of a highly sensitive eddy current test at various frequencies to measure the crack depth in SHMMCs. Additionally, apart from employing the Taguchi L8 mixed orthogonal array and analysis of variance, the researchers developed a predictive Fuzzy model based on S/N ratio to identify the optimal design factors combination for effective healing in damaged SHMMCs.

Sharma et al. [75] investigated the self-healing behavior of a metal–matrix composite prepared using the semi-solid metal processing technique known as rapid slurry formation. They analyzed the recovery rate and microstructure of the self-healing material, confirming successful integration of the reinforcement within the matrix. Random dispersion of NiTi wires and the conversion of alpha aluminum’s dendritic microstructure into a globular form through RSF improved the alloy’s strength and facilitated partial crack closure. The integration of A356 alloy with NiTi wires enabled shape-memory effects, allowing the material to regain its original shape upon exposure to heat. Crack closure analysis showed a positive recovery of 44.277% on the deformed surface, highlighting the potential of this approach for designing self-healing metallic materials. However, optimizing wire distribution and orientation could further enhance mechanical properties such as strength, stiffness, and toughness.

The conclusion can be drawn that the utilization of SMA reinforcements holds promise in enhancing the healing capability of metal matrix composites, with certain composites exhibiting significant recovery of mechanical properties after healing. However, limitations exist, including challenges in manufacturing at higher volume fractions of SMA reinforcement and potential instability of the composite structure at higher healing temperatures. Other challenges include poor wettability between the reinforcement and matrix, oxidation, insufficient capillary pressure, and achieving healing of macroscopic damage without compromising strength or functionality. Further research is imperative to optimize the design and processing parameters of SMA-reinforced SHMMCs and to explore their potential for practical applications.

To date, no analytical models have been developed for designing and synthesizing the microstructure of SHMMCs with long, short, or nanosized NiTi and other SMA fibers, where the crack can be closed or its propagation stopped, or where the crack can be filled with a healing material. It is expected that an accurate analytical model will be able to successfully predict the optimum volume fraction of SMA fibers needed to yield the matrix and prevent damage to the SMA fibers during healing. Analytical models need to be developed to calculate the pre-strain of SMA fibers and predict the optimum orientation and distribution of SMA fibers in the metal matrix for the most effective healing of long SMA-reinforced SHMMCs. The importance of bonding and load transfer between SMA fibers and the matrix, effect of pre-strain in SMA fibers for load transfer between SMA fibers and the matrix, the rate of crack propagation, rate of crack closure, and shape recovery of SMA-reinforced SHMMCs need to be quantified. Manuel [39] developed an analytical model to determine the minimum volume fraction of SMA wires required to induce a clamping force at the crack interface during a healing cycle. The healing of the SMA-reinforced SHMMCs samples is greatly affected by the volume fraction of SMA reinforcement, compressive yield strength of the matrix, and parent phase flow stress of the SMA reinforcement. Manuel’s model presented in this section is based on the SMA wire’s transformation temperatures and the matrix’s compressive yield strength at elevated temperatures. The Johnson–Cook relationship is a mathematical equation that characterizes the response of materials when subjected to compressive loads at high temperatures. It mathematically represents the best fit curve through the temperature-dependent matrix compressive flow stress, which is presented in Equation (3) [119].

\[
\sigma_{\text{flow}} = [A + B\varepsilon^n] \left[1 + C\ln\dot{\varepsilon}^* \right] \left[1 - \left(\frac{T - T_{\text{room}}}{T_{\text{melt}} - T_{\text{room}}}\right)^m\right]
\]

where \(\sigma_{\text{flow}}\) = flow stress of matrix alloy, \(\varepsilon\) = equivalent plastic strain, \(\dot{\varepsilon}^*\) = normalized plastic strain rate, \(T_{\text{room}}\) = room temperature, \(T_{\text{melt}}\) = melting temperature of matrix alloy, and \(A, B, C, n, \text{ and } m\) are empirical constants that depend on the material being. The constants \(A, B, C, n, \text{ and } m\) are typically determined through experimental testing and calibration for each specific material of interest to accurately represent its behavior under such conditions. The flow stress of an alloy depends on work-hardening, strain rate, and thermal effects, which are presented in the above equation by the three groups of bracketed terms. However, if the focus is solely on the temperature effects on stress, Equation (3) can be simplified to the following expression:

\[
\sigma_{\text{flow}} = A \left[1 - \left(\frac{T - T_{\text{room}}}{T_{\text{melt}} - T_{\text{room}}}\right)^m\right]
\]

Flow stress will be zero at the melting temperature (\(T_{\text{melt}}\)). Since the matrix alloy contains the eutectic phase, the alloy’s eutectic temperature (\(T_{\text{eut}}\)) is used as a substitute for \(T_{\text{melt}}\) because of the alloy softening that takes place during healing in the partially liquefied alloy. Therefore, the Johnson–Cook relationship can be modified as Equation (5), considering the alloy’s melting temperature as the eutectic healing temperature.

\[
\sigma_{\text{flow}} = A \left[1 - \left(\frac{T - T_{\text{room}}}{T_{\text{eut}} - T_{\text{room}}}\right)^m\right]
\]

The matrix is clamped back together by the applied force of SMA wires at elevated temperatures at the equilibrium condition. The applied forces (\(F\)) between the matrix and SMA wires are equal at this condition. Equal forces can be represented by normalized stresses (\(\sigma\)) over the area (A) for the matrix and SMA wires:

\[
F_{\text{SMA}} = F_{\text{matrix}} \rightarrow \sigma_{\text{SMA}}A_{\text{SMA}} = \sigma_{\text{matrix}}A_{\text{matrix}}
\]
where $F_{SMA} = \text{applied force by the SMA wire}$, $F_{\text{matrix}} = \text{applied force by the matrix}$, $\sigma_{SMA} = \text{recovery stress SMA wire}$, $\sigma_{\text{matrix}} = \text{stress in the matrix during healing}$, $A_{SMA} = \text{cross-sectional area of SMA wire}$, and $A_{\text{matrix}} = \text{cross-sectional area of the matrix}$. Dividing Equation (6) by the total cross-sectional area, the following relationship can be obtained:

$$\sigma_{SMA} V_f = \sigma_{\text{matrix}} (1 - V_f)$$  \hspace{1cm} (7)

where $V_f = \text{volume fraction of SMA wires}$. During healing of the composite, the stress in the SMA wires is determined by the reversion stress ($\sigma_R$) of the SMA wires, and the stress in the matrix is governed by the matrix compressive yield stress ($\sigma_{MCYS}$) at elevated temperatures under compression. The SMA wires must yield the matrix to prevent plastic deformation during healing. Equation (7) can be modified as the following expression:

$$\sigma_R = \sigma_{MCYS} \left( \frac{V_f}{1 - V_f} \right)$$  \hspace{1cm} (8)

Equation (8) demonstrates how the volume fraction of the SMA wires in the SHMMC indirectly relates $\sigma_R$ to the $\sigma_{MCYS}$ of the matrix alloy. Since the flow stress of a matrix alloy is equal to its compressive yield strength (i.e., $\sigma_{\text{flow}} = \sigma_{MCYS}$), Equation (5) can be written as:

$$\sigma_{MCYS} = \Lambda \left[ 1 - \left( \frac{T - T_{\text{room}}}{T_{\text{melt}} - T_{\text{room}}} \right)^m \right]$$  \hspace{1cm} (9)

The analytical model for the optimal volume fraction of SMA wires in the SHMMC to induce a clamping force at the crack interface during a healing cycle is derived by substituting the value of $\sigma_{MCYS}$ from Equation (9) (the modified Johnson–Cook model) into Equation (8):

$$\sigma_R = \Lambda \left[ 1 - \left( \frac{T - T_{\text{room}}}{T_{\text{melt}} - T_{\text{room}}} \right)^m \right] \left( \frac{V_f}{1 - V_f} \right)$$  \hspace{1cm} (10)

Manuel [39] has found that for a Sn-based proof-of-concept composite, a 1% volume fraction of Ti-49.4 at.%Ni SMA wire is needed for healing the composite. However, a 30% volume fraction of Ti-49.4 at.%Ni SMA wire is needed for a Mg-based proof-of-concept composite. The reason for the higher volume fractions of SMA wires needed in healing the Mg-based composite than the Sn-based composite is that matrix strength increases for a Mg-based composite, which resists the recovery force of SMA wires.

Fisher [33] has verified the above model for Al-Cu-Si and Al-Si alloys. It is found that for an Al-4.1Cu-2Si (at%) composite, the minimum NiTi SMA wire volume percentage required is determined to be 55%. This implies that the SHMMC would need to contain more SMA wires than matrix alloy to achieve effective healing. Similar data is obtained for the SMA-wire-reinforced Al-3 at%Si composite. For this composite, the volume percentage of SMA wires needed for the healing is calculated to be 25%. However, Fisher has reported that healing occurred in Al-Si composites with less than 2.5 vol% SMA wires, demonstrating that the Manuel model is inadequate in determining minimum SMA volume fractions in Al-based composites. The large difference between the calculated value and experimental value of the minimum volume fractions of SMA wires required to induce a clamping force at the crack interface during a healing cycle was attributed to the assumptions made in the model, such as equal strain and no plasticity in the SHMMC [33]. In addition, increased toughness of synthesized SHMMC can also be a reason to decrease the required minimum volume fraction of SMA wires for effective healing. Therefore, a more accurate analytical model needs to be developed, which will be able to address the mentioned issues and minimize the difference between the calculated value and experimental value of the required volume fractions of SMA wires needed for the most effective healing of SHMMCs.
7. Future Research Directions

Over the last decade, the study of SMA-reinforced SHMMCs has emerged as a significant new research field in materials science and engineering. Self-healing properties offer numerous benefits, such as improved resilience, longer lifespan, enhanced safety, and reduced maintenance costs, making them desirable for various applications including building structures, biomedical implants, defense sectors, and transportation systems. Different matrix materials, such as low-melting alloys like tin-based alloys/solders, aluminum-, magnesium-, and zinc-based alloys, have been used to demonstrate self-healing in SHMMCs. However, there is a need to develop new SHMMCs using high-temperature alloys and alloys with high mechanical properties and explore new types of SMAs or materials with negative CTE as reinforcements. Incorporating reactive elements into the matrix of SHMMCs can also improve the bond at the interface and enhance healing efficiency.

Achieving optimal wetting between the reinforcement and matrix during synthesis is crucial for ensuring effective load transfer and bonding between the two materials. This can be challenging, as inadequate wetting can result in weak interfaces, reduced mechanical properties, and compromised healing performance. Therefore, more research is needed to develop effective techniques to enhance wetting and promote strong bonding between SMA fibers and the matrix. The oxidation of SMA fibers and metal matrices during synthesis and healing can significantly impact their functional properties. Oxidation can decrease the recovery stress and strain of SMA fibers and prevent bonding of the cracked surfaces of metal matrices, thereby limiting their healing capabilities. Investigating strategies to overcome oxidation and preserve the integrity of SMA fibers and metal matrices in SHMMCs is essential to ensure their optimal performance.

Providing adequate capillary pressure during the healing process is critical for promoting crack closure and enhancing the healing efficiency of SMA-reinforced SHMMCs. Capillary pressure plays a vital role in driving the healing agents into the cracks and promoting their closure, which is essential for effective healing. Therefore, further research is needed to understand the factors that influence capillary pressure in SHMMCs and develop strategies to optimize it for efficient healing. Preserving the strength and functionality of SHMMCs during the healing process is crucial for maintaining their structural integrity and performance. Healing mechanisms such as annealing or thermal treatment can affect the mechanical properties of the composite, including its strength, toughness, and fatigue resistance. Therefore, investigating methods to ensure that the healing process does not compromise the overall strength and functionality of the composite is critical for its successful application in practical scenarios.

Autonomous and multicycle healing techniques need to be developed for SHMMCs, as most current methods require external fields such as thermal heat or electric current. Remote structural health monitoring should also be integrated into SHMMCs and components for improved performance. Traditional methods for healing assessment, such as a hot plate, may need to be replaced with electric heating or a hot bath to achieve uniform heating. Characterization techniques such as strain imaging, X-ray, CT scan, and ultrasonic tests can be used to evaluate damaged samples and healing of micro voids and strains. Additionally, creep tests, low cycle fatigue tests, and compression-after-impact tests can provide valuable insights into the healing behavior of SHMMCs. Developing more complex and accurate models for the strain field, considering the shape-memory effect of SMAs, will also aid in experimentation.

Further research is needed to investigate the effect of random SMA fibers on healing and the potential of incorporating SMA nanoparticles into the metal matrix to induce autonomous self-healing. Determining the upper size limit of SMA particles or short fibers for inducing self-healing without external stimuli and understanding the role of nano- and micron-sized SMA short fibers or particles in autonomous self-healing require further exploration. Computational fluid dynamics analysis of healing liquid flow through the microstructure at various crack length scales, as well as quantitative modeling of crack filling and solidification processing of matrix and healing liquid in SHMMCs, taking into
account microstructure, surface tension, and capillary forces, can provide valuable insights into crack closure and healing mechanisms.

8. Conclusions

Research on SMA-reinforced SHMMCs is imperative because of their immense potential in the automotive, spacecraft, aerospace structures, and defense industries. This paper provides an in-depth analysis of the synthesis, characterization, and healing assessment techniques used by various researchers in the field of SMA-reinforced SHMMCs. The findings indicate that casting is the most commonly used synthesis process for fabricating SMA-reinforced SHMMCs, while SEM and optical microscopy are the preferred characterization techniques. Solidification processing of SMA-reinforced SHMMCs is presented in this paper; the scope of study of this topic is not fully explored. Future research is needed to understand the solidification processing of SMA-reinforced SHMMCs.

One of the major concerns in SMA-reinforced SHMMCs is the reinforcement-metal wettability. It is found that etching the SMA wire with an aqueous solution of 4.8%HF-10.5%HNO$_3$ and then fluxing with phosphoric-acid-based flux is the most efficient method to remove the oxide layer from SMA wires and improve the bonding between SMA wires and the matrix material. Mechanical tests such as tensile, bending, and fatigue tests have been used to assess the performance of SMA-reinforced SHMMCs, but impact properties are yet to be investigated. The impact properties of SMA-reinforced SHMMCs need to be investigated, as these composites are envisioned for use in spacecraft that may be impacted by meteoroids or space debris during their missions in space.

The autonomous and multicycle healing capability of SMA-reinforced SHMMCs is an area that requires further research, as no comprehensive studies have been conducted in this field. The paper presents an analytical model to determine the optimum volume fraction of SMA wires required for effective healing without damaging the SMA wires. However, the model is found to be insufficient for determining minimum SMA volume fractions in Al-based composites, and more precise analytical models need to be developed for this purpose. Additionally, a systematic study is needed to quantify the required pre-strain, optimum orientation and distribution of SMA fibers in the metal matrix to achieve the most effective healing in SMA-reinforced SHMMCs. The importance of bonding and load transfer between SMA fibers and the matrix, as well as the effect of pre-strain in SMA fibers on load transfer, should also be assessed.

It is imperative to accurately predict the rate of crack propagation, rate of crack closure, and shape recovery in SMA-reinforced SHMMCs to design them effectively. Further research should be conducted to explore new self-healing mechanisms for high-temperature and high-mechanical-property alloys, develop autonomous healing techniques, and design novel testing and characterization techniques for evaluating healing. Additionally, improving wetting between the reinforcement and matrix during synthesis, overcoming oxidation, providing adequate capillary pressure, and preserving strength and functionality during healing are recommended areas for further investigation.


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