Microstructure and mechanical properties of superelastic NiTi fiber reinforced NiTi/(Al₃Ti+Al₃Ni) metal-intermetallic laminated (SFR-MIL) composites

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Abstract
In this work, a novel superelastic NiTi fiber reinforced NiTi/(Al₃Ti+Al₃Ni) metal-intermetallic laminated (SFR-MIL) composites was fabricated via vacuum hot pressing method. In order to accurately determine the reaction products of the fabrication process, the microstructure and phase constituents were analyzed and determined using scanning electron microscopy (SEM), energy dispersive spectroscope (EDS) and x-ray diffraction (XRD). Results showed that Al₃Ti and Al₃Ni were formed during fabrication process. In addition, the quasi-static tensile testing results indicated the average UTS and failure strain of SFR-MIL composite are 377.0 MPa and 15.4%, respectively. The compressive tests of SFR-MIL composites manifested that the average peak compressive strength of this class of materials with load perpendicular to layers is 1114.3 MPa, equivalent to or higher than that of some other laminated composites. Then, the fracture mechanisms are discussed, and the results showed that the reaction band is the possible crack initiation due to the stress concentration, and the major fracture mechanisms are intergranular and transgranular fracture modes.

1. Introduction

Intermetallics are always rated as a kind of promising materials due to their excellent properties, such as high modulus, high stiffness, superior wear resistance and stability in high temperature [1–4]. However, intermetallics are brittle in nature, extremely limiting their application [5–7]. Therefore, efforts have been made to improve the ductility of intermetallics, among which a new structural material, Ti/Al₃Ti laminated composite, was developed to endow these brittle intermetallics with plasticity and various functionalities [8, 9].

Inspired by abalone shells, Ti/Al₃Ti laminated composite possesses a layered structure combining the ductile layer Ti with the brittle layer Al₃Ti, and the fabrication and performance of this material have been thoroughly studied. For example, Vecchio fabricated the Ti-Al₃Ti laminated composite by hot pressing method, proving that the mechanical properties of the MIL composites can be varied and tailored by simply varying the individual foil thicknesses and layering sequence [10]. In Rohatgi’s study, it was found that crack bridging and crack deflection by the Ti layers were primarily responsible for the large-scale bridging conditions leading to the R-curve behavior and enhanced fracture toughness [11]. Zhou and his co-workers studied the interface tensile behavior of the Ti/Al₃Ti metal-intermetallic laminate (MIL) composite under quasi-static and high strain rates, indicating that the interface tensile strength of the Ti/Al₃Ti MIL composite increases with increasing strain rate [12]. Moreover, in Zelepugin’s work, a computational model was presented for the materials subjected to high velocity impact, showing that a multilayered metal-intermetallic composite has higher strength characteristics.
compared with a uniform Al$_3$Ti intermetallic target, which was in good qualitative and quantitative agreement with the experimental data [13].

Considering that Ti/Al$_3$Ti laminated composite possess so many superior properties, a lot of work has been done to improve the overall performance of this kind of materials [14, 15]. On the whole, there are two main approaches to achieve this purpose. On the one hand, Ti$_x$Al$_{3-x}$Ti laminated composite can be reinforced by introducing SiC fiber, Al$_2$O$_3$ fiber and NiTi fiber, etc [16–18], to enhance its specific properties. For example, Han found that both of the compressive strength and failure strain increase compared to that of Ti$_x$Al$_{3-x}$Ti after the introduction of Al$_2$O$_3$ ceramic fiber [19]. Tian et al studied the improved fracture toughness of NiTi shape memory alloy, showing that the improved toughness of the shape memory alloy NiTi fiber reinforced Ti$_x$Al$_{3-x}$Ti metal-intermetallic laminated (SMAFR MIL) composites is mainly attributed to the plastic deformation and breakage of the NiTi fibers [2]. On the other hand, on the basis of the ideas that the laminated composites are fabricated via alternate stacking of tough layer and stiff layer can achieve the desired properties, a number of novel laminated composites have been designed and fabricated, manifesting that this kind of materials can possess excellent mechanical performance [20–23]. In addition, some Fe-based and Ni-based laminated composites were designed and investigated [24, 25].

Based on the aforementioned idea, a novel material, i.e. NiTi/(Al$_3$Ti+Al$_3$Ni) laminated composite, has been developed using hot pressing sintering, and it proved that this composite has superior mechanical properties [26]. Shape memory alloy NiTi has been widely employed due to its shape memory effects, superelasticity and high damping capacity [20, 21, 27]. In addition, a reaction layer without crack can form when it meets Al layer at the temperature above 650 °C. However, the centerline formed unavoidably in the intermetallic layer, which become the initial crack location, leading to the decrease of the mechanical properties [26]. In light of this, the superelastic NiTi fiber was chosen to fabricate a new kind of laminated composites in the hope of obtaining a material with better performance.

The objective of this research is to investigate the microstructure and basic mechanical behavior of the superelastic NiTi fiber reinforced NiTi/(Al$_3$Ti+Al$_3$Ni) metal-intermetallic laminated (SFR-MIL) composites. Initially, the SFR-MIL composite was fabricated using hot press sintering technique. Then, the microstructure and phase constituents were analyzed and determined using scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS) and x-ray diffraction (XRD). Finally, the mechanical performance of this composite was discussed.

2. Experimental procedures

2.1. Materials fabrication

The superelastic NiTi fiber reinforced NiTi/(Al$_3$Ti+Al$_3$Ni) metal-intermetallic laminated (SFR-MIL) composite investigated in this work were fabricated by hot pressing sintering in vacuum using NiTi alloy sheet (100 mm × 100 mm × 0.8 mm), 1060 Al sheet (100 mm × 100 mm × 1.1 mm) and continuous NiTi fiber.

| Materials        | Composition (wt.%) |
|------------------|--------------------|
| Al 1060          | Al: balance, Si:0.25, Cu:0.05, Zn:0.05, Mn:0.03, Mg:0.03, Fe:0.35, V:0.05 |
| NiTi sheet/fiber | Ti: balance, Ni: 55.89, C:0.043, N:0.003, O:0.043, H:0.002 |
with a diameter of 0.8/1 mm. The chemical composition of starting materials used in this work is shown in table 1. The sheets and fibers were polished using silicon carbide paper in order to remove surface oxide layers and contaminations, and then rinsed in water for 5 min. After that, all the raw materials were cleaned ultrasonically in alcohol for 15 min and then dried rapidly. In order to ensure the chemical compositions of the product are approximately identical in each fabrication process, the NiTi sheets and fibers were bought from the same manufacturer and they have the same component.

Before sintering, the pretreated NiTi sheets and Al sheets were alternately stacked by the sequence of NiTi/Al/NiTi, and the NiTi fibers were fastened to the Al sheets with an interval of 2.5 mm. The ‘NiTi foil - NiTi fiber - Al foil - NiTi foil’ was defined as a ‘unit’. The schematic diagram of stacked materials is presented in figure 1. During the hot pressing process, the temperature was raised to 600 °C in 2 h, then maintained in this temperature for 1 h; during this low temperature stage, a 2.5 MPa loading pressure was applied on the stack for the sake of the compaction of the foils and fibers. The following step was to raise the temperature to 645 °C within 40 min and kept it for 4 h. Details for the parameters can also be found elsewhere [26, 27].

2.2. Microstructure characterization
The microstructure characterization of SMAFR MIL composite was observed by Sirion 200 scanning electron microscopy (SEM) equipped with an EDS system designed by FEI with which the number of atoms of each element could be determined and the corresponding phase be also likely to be determined approximately. The phase constituent analysis was performed by X’Pert PRO x-ray diffraction (XRD) with the scanning speed of 2° min⁻¹ from 10° to 90°. The fracture morphology of SFR-MIL after bending was characterized by FEI Quanta 200 scanning electron microscopy (SEM).

2.3. Mechanical testing
Quasi-static tensile and compressive tests of SFR-MIL composite were carried out on Instron 5500 R load frame at the strain rate of 0.001/s under room temperature (23 °C). In the tensile tests, all the three samples were machined from the same SFR-MIL composites (38 vol.% NiTi layer, 6 vol.% NiTi fiber). As for the compression tests, five samples that were also obtained from the same composites (volume fraction: 37 vol.% NiTi layer, 5 vol.% NiTi fiber) were tested under the condition of loading perpendicular to the layers, respectively. The stress-strain curves were plotted in the form of engineering stress and engineering strain data, and the same convention was used throughout this work. Detailed information of mechanical test samples can be found in figure 7 and 8.

3. Results and discussion

3.1. Microstructure characterization
As shown in figure 2, the x-ray diffractograms reveal that in addition to the original NiTi phase, the superelastic NiTi fiber reinforced NiTi/(Al3Ti+Al3Ni) metal-intermetallic laminated (SFR-MIL) composite mainly consists of two newly formed intermetallics, i.e. Al3Ti and Al3Ni phases. Due to the sufficient reaction time, the
aluminum phase has been consumed completely. Therefore, the SFR-MIL composite contains two important components—the ductile NiTi plate/ﬁber and the brittle intermetallic phase (Al3Ti and Al3Ni).

As illustrated in Figure 3, the general microstructure exhibits multi-layer features, in which several unique components are included. It can be found that many cell-like blocks were formed during the fabrication process, and most of the NiTi ﬁbers can maintain their basic shape. As expected, the reaction band was formed between the NiTi phase and the intermetallic phase, corresponding to our previous investigation with respect to the shape memory alloy ﬁber reinforced metal-intermetallic Ti/Al3Ti laminated (SMAFR) composite, whose reaction band exists beside NiTi ﬁber as well [27]. Moreover, due to the appropriate parameters in the fabrication process, the interface between the phases, especially the one between the ﬁber and the two-phase area, can bond well in the SFR-MIL composite. Figure 4 shows the EDS analysis between NiTi ﬁber and the intermetallic layer. It is found that the Area A, also known as reaction band, is a kind of a multiple phase mixture, including various Ti-Al and Ni-Al intermetallics [28, 29]. Area B and Area C are composed of Al-based intermetallics. It can be seen that the ﬁber is bonded well with the intermetallic layer because of the metallurgical reaction.

Based on the EDS element mapping, the distribution of Al, Ni and Ti elements near the NiTi ﬁber is shown in Figure 5. It is clearly observed that the Al atoms spread over the intermetallic layer, making this layer ﬁlled with Al-based intermetallics. In terms of Figures 5(c) and (d), an obvious circle with Ti-rich area around the ﬁber can be detected, which proves that Al3Ti is a preferred formed phase when it is far away from the NiTi ﬁber/plate.

**Figure 3.** SEM image of the microstructure of SFR-MIL composite, including ductile NiTi layer, NiTi ﬁber and intermetallic layer (reaction band, two-phase area and pure Al3Ti phase).

**Figure 4.** EDS analysis between NiTi ﬁber and the intermetallic layer, showing that the Area A, is a kind of a multiple phase mixture, including various Ti-Al and Ni-Al intermetallics; Area B and Area C are composed of Al-based intermetallics.
Figure 6 shows the SEM images of the area near the reaction band and the pure Al₃Ti phase around the fiber with high magnification, and EDS analysis were performed in spectra 1-2 and Areas 3-4, as marked in figures 6(a) and (b), the results listed in table 2. EDS analysis showed that the whole intermetallic layer is composed of Al-rich compounds: The atom ratio of Spectrum 1 (light grey phase) mainly consists of Al and Ni atoms, only with a small amount of Ti atoms; the same phenomenon can be found in Spectrum 2 (dark phase), whose Ti atoms are predominant as compared with Ni. Therefore, it can be concluded that the newly formed products are Al₃Ti and Al₃Ni, corresponding to the XRD analysis. It is noted that in Area 3, the ratio of Al/Ti is approximately 3/1 regardless of the small amount of Ni element, Al₃Ti is a preferred phase in the region far away from the NiTi fiber, as mentioned above. Moreover, a centerline is observed in the middle of the pure Al₃Ti phase, which is a typical phenomenon due to the diffusion reaction [11]. Area 4 is a multiple phase zone, consisting of the same kind of dark phase and white (or grey) phase as that is shown in figure 6(a). The ratio of
Al/X (X stands for Ti or Ni) is about 6/1, and the ratio of Al/(Ni+Ti) is nearly 3/1, indicating that Al$_3$Ti and Al$_3$Ni are two basic intermetallics in the SFR-MIL composite.

In summary, experiencing the vacuum hot pressing sintering, the raw materials in one unit - NiTi foil, Al foil and NiTi fiber - have been replaced by a new unit, i.e. 'NiTi layer - reaction band - intermetallic layer - NiTi fiber - intermetallic layer - reaction band - NiTi layer'. The overall structure basically keeps the ductile/brittle structure with the reinforcement NiTi fiber in the brittle intermetallic layer. The parameters used in this fabrication are appropriate, making sure that there are no or less cracks in the SFR-MIL composite.

### 3.2. Tensile behavior of SFR-MIL composites

In order to explore the possible application of superelastic NiTi fiber reinforced NiTi/(Al$_3$Ti+Al$_3$Ni) laminated (SFR-MIL) composite, tensile tests were performed with three samples cut from the identical SFR-MIL composite (38 vol.% NiTi layer, 6 vol.% NiTi fiber). Figure 7(a) exhibits the typical macro morphology of SFR-MIL composite after tensile tests. It can be found (upper image) that the delamination occurred between the NiTi layer and the intermetallic layer. In addition, the left bottom image of figure 7(a) manifests that a tearing phenomenon (front side) took place during the loading stage, implying that the outer NiTi layer was likely to bear relatively high load and fractured initially compared with the inner NiTi layer. Moreover, it is significant to note from the right bottom of figure 7(a) that a great many intermetallic blocks (Al$_3$Ti and Al$_3$Ni) stringed by NiTi fiber did not crack into smaller pieces, which is extremely different from those of NiTi/(Al$_3$Ti+Al$_3$Ni) laminated composite, whose intermetallic layer almost fragmented into small pieces and then fell off, making the original intermetallic layer of composite without NiTi fiber reinforcement nearly empty. As seen in figure 7(b),

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**Table 2.** Typical chemical composition of each layer and phase detected by EDS.

|                | Al          | Ti          | Ni          |
|----------------|-------------|-------------|-------------|
|                | Wt%         | At%         | Wt%         | At%         | Wt%         | At%         |
| Spectrum 1     | 60.21       | 76.28       | 4.14        | 2.96        | 35.65       | 20.76       |
| Spectrum 2     | 60.55       | 74.18       | 28.40       | 19.60       | 11.05       | 6.22        |
| Area 3         | 62.38       | 74.68       | 37.19       | 25.08       | 0.43        | 0.24        |
| Area 4         | 60.00       | 74.87       | 16.92       | 11.90       | 23.08       | 13.24       |
the samples first experienced elastic stage, and then pseudoelastic stage until it reached the ultimate tensile strength (UTS), which is consistent with the investigation of NiTi/(Al$_3$Ti + Al$_3$Ni) laminated composite [26]. It is worth noting that in the pseudoelastic stage, only a few fluctuations occurred, implying that in this stage the intermetallics actually cracked but not severely. This phenomenon is quite distinct with that of NiTi/(Al$_3$Ti + Al$_3$Ni) laminated composite, whose intermetallic layer cracked to small pieces. Moreover, the ultimate tensile stress (UTS) and failure strain of each sample of SFR-MIL composites are listed in table 3. It can

Figure 8. Loading scheme and configuration of compressive specimen (a) and the compression stress-strain curves of the SFR-MIL samples tested with the loading perpendicular to the layers at the strain rate of 0.001/s (b).

| Number | UTS, MPa | Failure Strain |
|--------|----------|----------------|
| 1      | 381.3    | 14.7%          |
| 2      | 368.4    | 16.4%          |
| 3      | 351.2    | 15.0%          |
| Average| 377.0    | 15.4%          |

Figure 9. SEM images of the typical fracture characteristics observed from the intermetallic and NiTi fractured sample surface (a), near the NiTi fiber (b), the cross section parallel to the layers (c), intermetallic layer with high magnification (d).
be seen that the average UTS of SFR-MIL composite is 377.0 MPa, 47.1% higher than that of NiTi/(Al₃Ti + Al₃Ni) laminated composite (266.1 MPa). The average failure strain of SFR-MIL composite is 15.4%, slightly less than that of NiTi/(Al₃Ti + Al₃Ni) laminated composite (16.1%) due to the larger fraction of NiTi layer (47 vol.%).

Considering the fact that SFR-MIL composite possesses superior performance compared with the NiTi/(Al₃Ti + Al₃Ni) laminated composite, it is reasonable to conclude that NiTi fiber plays an imperative role in the tensile process because it can not only provide the similar pseudoelastic effect as the NiTi layer, but also strengthen the composite by offering the bonding interface between the fiber and intermetallic layer. NiTi fiber exhibits its advantages, proving that it is an appropriate reinforcement.

### 3.3. Compressive response of SFR-MIL composites

Like the traditional Ti/Al₃Ti laminated composite, it is appealing to investigate the basic compressive properties of SFR-MIL composite. The compression tests were conducted using 5 samples with loading perpendicular to layers at the strain rate of 0.001/s. The stress-strain curves and the results of the compression tests are shown in figure 8 and table 4, respectively. As seen in figure 8, the compressive curves of the five samples have similar compressive behavior, going through the elastic stage and then failed. It is noted that some curves fluctuated when they nearly reached the peak, showing that some of the intermetallics broke away from the NiTi layer (proved by the following section) due to the load applied on the samples. The experimental results from table 4 show that the average peak compressive strength of SFR-MIL with load perpendicular to the layers is 1114.3 MPa with the average failure strain 1.1%, both of which are equivalent to or even higher than most of laminated composites, such as SMAFR, SMAPR and Ti/Al₃Ti laminated some other fiber reinforced laminated composites [8, 26, 27, 30]. Other laminated composites with reinforcement possess similar characteristics, but this material is still a promising candidate considering NiTi can provide unique damping capacity [17–19].

After the quasi-static compression test, the fractured surface was observed by SEM for thoroughly understanding the fracture mechanism of SFR-MIL laminated composites, shown in figure 9. It can be seen in figure 9(a) that the delamination occurred between the NiTi layer and the intermetallic layer (right top). In addition, from the cross-section morphology, it can be observed that the crack propagation is mainly parallel and perpendicular to the layers, with the joint point being reaction band near the NiTi fiber, which is because the stress concentration is likely to take place in the reaction band, making this area the possible crack initiation [26]. The fractured surface near the NiTi fiber is shown in figure 9(b), indicating that the cracks near the fiber exhibit typical brittle fracture features. Moreover, since the delamination took place, the fractured characteristics can be observed from other angles, as shown in figure 9(c), which presents the fractured surface parallel to the layers. From figure 9(c), it is found that the crack path is along the fiber direction, and the NiTi fiber did not fracture, meaning that the fiber still holds its elasticity. The detailed features of the intermetallic layer with high magnification are shown in figure 9(d), from which we can find that regardless of some impurities, some typical transgranular fracture and intergranular fracture features can be found, showing a representative brittle fracture trait. All in all, the mechanical properties of SFR-MIL composites are closely relevant to each component, especially to the crack propagation paths, which can release the energy.

| Number | Compressive Stress (MPa) | Failure Strain (%) |
|--------|--------------------------|--------------------|
| 1      | 1016.6                   | 1.1                |
| 2      | 1146.1                   | 1.1                |
| 3      | 1238.6                   | 1.2                |
| 4      | 1007.7                   | 1.0                |
| 5      | 1162.3                   | 1.2                |
| Average| 1114.3                   | 1.1                |

Table 4. Results of SFR-MIL composites after quasi-static compression tests.
4. Conclusion

In the present study, a novel kind of superelastic NiTi fiber reinforced NiTi/(Al\textsubscript{3}Ti+Al\textsubscript{3}Ni) metal-intermetallic laminated (SFR-MIL) composites were successfully fabricated via vacuum hot pressing process. The detailed constituents have been characterized, and the mechanical properties have been discussed. The main conclusions drawn from the current work are summarized as follows:

(1) After the vacuum hot pressing sintering, a new structure of 'NiTi layer - reaction band - intermetallic layer - NiTi fiber - intermetallic layer - reaction band - NiTi layer' was formed. The overall structure basically keeps the ductile/brittle structure with the reinforcement NiTi fiber in the brittle intermetallic layer. EDS analysis showed that the intermetallic layer includes Al\textsubscript{3}Ti phase and Al\textsubscript{3}Ni phase, and an almost pure Al\textsubscript{3}Ti circle band was found with a centerline in it.

(2) The quasi-static tensile testing results indicated the average UTS and failure strain of SFR-MIL composite are 377.0 MPa and 15.4%, respectively. The obvious improvement compared with the NiTi/(Al\textsubscript{3}Ti+Al\textsubscript{3}Ni) laminated composite without fiber is attributed to the NiTi fiber, which can provide the similar pseudoelastic effect as the NiTi layer, and strengthen the composite by offering the bonding interface between the fiber and intermetallic layer at the same time.

(3) The compressive tests of SFR-MIL composites manifested that the average peak compressive strength of this class of materials with load perpendicular to layers is 1114.3 MPa, equivalent to or higher than that of some other laminated composites. The delamination of the NiTi layer and the intermetallic layer can account for the fluctuations of the stress-strain curves before the failure of this material.

(4) The compression fracture of SFR-MIL composites was investigated via SEM observation, and the fracture mechanisms are determined. The reaction band is the possible crack initiation due to the stress concentration, thus making the crack path parallel and perpendicular to the layers with the reaction band near NiTi fiber the joint point. Moreover, the intermetallic layer shows brittle fracture features, in which the major fracture mechanisms are intergranular and transgranular fracture modes.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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