Structural designation and mechanical properties of TiNi/Ti2Ni laminated composites

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Abstract. TiNi/Ti2Ni laminated composites were fabricated using hot isostatic pressing (HIP) furnace together with structural designation by the control of Ti and Ni foil thicknesses. During HIP processing, Ti reacts with Ni, leading to the formation of softer TiNi and harder Ti2Ni. Through adjusting the thickness ratio of Ti and Ni foils, the ratio of TiNi and Ti2Ni fraction can be controlled. Hard-soft-hard, soft-hard-soft and hard-intermediate-soft laminated composites were designed, where hard layer was made by 50 μm Ti and 20 μm Ni, soft layer was made by 40 μm Ti and 20 μm Ni, and the intermediate structure between hard and soft ones was made by 45 μm Ti and 20 μm Ni. The mechanical properties were characterized using uniaxial tension, uniaxial compression, single edge-notched beam and split-Hopkinson pressure bar. The results show that all three laminated composites have compression strength of 1690 ~ 1853 MPa and fracture toughness of 27.2 ~ 37.9 MPa·m1/2 due to the same constituents of TiNi and Ti2Ni. However, the soft-hard-soft structure shows the best response under high strain rate because it was fractured into larger pieces while the other two structures were fractured into smaller pieces together with smaller fracture strain.

1. Introduction

Laminated composite is developed on the basis of in-depth study of the microstructure and mechanical behavior of shell nacre [1, 2]. Laminated composite is generally formed by arranging two or more foils with different physical or chemical properties associated with certain layer spacing and layer thickness ratio. Because of small layer spacing and multi-interface effect, the performance of laminated composite is better than that of the corresponding monomer material [3].

It is reported that advanced composite armors have a front that deforms the projectile tip and, in turn, has the capability to reduce penetration when improving mechanical properties such as high hardness, thermal resistance, and compressive strength [4]. Although these properties can be achieved by intermetallics alone, intermetallics cannot be used for armors on their own because of brittle fracture. On the other hand, ductile materials can provide structural integrity to prevent complete penetration of projectiles and absorb projectile energy [5, 6]. Thus, the combination of hard and ductile materials is a feasible way to produce armor materials, which promotes the development of laminated structures [7]. We have studied homogeneous TiNi/Ti2Ni intermetallic laminated composites [8]. Following this, in this paper, inhomogeneous TiNi/Ti2Ni laminated composites were
fabricated using hot isostatic pressing (HIP) furnace and the effect of designed inhomogeneous structures on mechanical properties was systematically investigated.

2. Materials and methods

Commercial Ti foils and Ni foils were cut into 50 × 40 mm2 rectangular sheets, cleaned in the alcohol using ultrasonic vibration and naturally dried in the air. The Ti foil thickness included 40, 45 and 50 μm, referring as to Ti40, Ti45 and Ti50, respectively; Ni foil thickness was 20 μm, referring as to Ni20. Figure 1 shows the stacking strategy. As shown in figure 1(a), from top to bottom, 16 layers of Ti50 and 16 layers of Ni20, 16 layers of Ti45 and 16 layers of Ni20, 16 layers of Ti40 and 17 layers of Ni20 were laid alternately in sample 1. From both top/bottom to the middle of sample 2, 16 layers of 50 μm Ti foil and 16 layers of Ni20 foil, 16 layers of Ti45 foil and 16 layers of Ni20 foil, 16 layers of Ti40 foil and 16 layers of Ni20 foil were laid alternately, with addition layer of Ni20 in the middle. From both top/bottom to the middle of sample 3, 16 layers of Ti40 foil and 16 layers of Ni20 foil, 16 layers of Ti45 foil and 16 layers of Ni20 foil, 16 layers of Ti50 foil and 16 layers of Ni20 foil were laid alternately, with additional layer of Ni20 foil in the middle. These three samples were sintered in a vacuum hot-pressing furnace at 900 °C under 8 MPa for 8 h. Sintering details were referred to [7].

Microstructures were characterized using a scanning electron microscopy (SEM) equipped with a backscattered electron (BSE) detector. The fracture toughness was measured by three-point bending (TPB) tests at room temperature, which was carried out using an electro-mechanical universal testing machine at a loading speed of 0.5 mm/min. The size of sample 1 was 1.5×3×17 mm3 with a 1.5 mm notch (in the middle on 17 mm side), the size of sample 2 and sample 3 was 3×6×30 mm3 with a 3 mm notch (in the middle on 30 mm side). Uniaxial compression tests were performed at room temperature and at a strain rate of 1×10−3 s−1. Split-Hopkinson pressure bar (SHPB) was used to test the dynamic compressive properties. For compression, the size of sample 1 was φ 3×3 mm2 and the size of sample 2 and sample 3 was φ 4×6 mm2. All the loading direction was along the laminate thickness. Fracture surfaces were examined using SEM.

![Figure 1](image)

**Figure 1.** Schematic diagram of three stacking schemes.

3. Results and discussion

Figures 2(a-c) show the overview of three different stacking strategies while figures (d-e) indicate the representative microstructures for different layers. According to our previous investigation [9], the
reaction between Ti and Ni during 8 h holding at 900 °C resulted in the formation of TiNi and Ti$_2$Ni. TiNi was in bright and Ti$_2$Ni was in grey in the BSE mode because of higher atomic weight in the former. From Ti50/Ni20 layer, Ti45/Ni20 layer to Ti40/Ni20 layer, the content of Ti$_2$Ni decreased because the thickness of Ti decreased, while the content of TiNi increased (figures 2(d, e)). According to previous research [7], Ti$_2$Ni is brittle and TiNi is ductile. Thus, Ti50/Ni20 layer is the hardest with high strength, while Ti40/Ni20 layer is the softest with good toughness. As a result, sample 1 exhibited hard-intermediate-soft laminated composites (figure 1(a)), where hard, intermediate and soft layers were sintered by Ti50/Ni20, Ti45/Ni20 and Ti40/Ti20, respectively. As shown in figure 2(c), sample 2 shows symmetrical gradient structure, which was formed by sintering Ti50/Ni20, Ti45/Ni20 and Ti40/Ni20 from both top/bottom to the middle. This is called as soft-hard-soft structure. Figure 3(b) indicates a symmetrical gradient structure in the sample 3, which was formed by sintering Ti40/Ni20, Ti45/Ni20 and Ti50/Ni20 from both top/bottom to the middle. This is named as hard-soft-hard structure.

Figure 2. (a-c) Full view of (a) sample 1, (b) sample 3 and (c) sample 2 in BSE mode; (d, e) representative transition microstructures in sample 2 (d) from Ti50/Ni20 layer to Ti45/Ni20 layer and (e) from Ti45/Ni20 layer to Ti40/Ni20 layer.

Figure 3 shows the compressive engineering stress-strain curves for three different structures. The elastic modulus of sample 2 and sample 3 were basically the same, while that of sample 1 was relatively small. The compressive strength of sample 3 is the highest (1853±4 MPa), while sample 2
has the smallest compressive strength (1690 ± 20 MPa). The fracture strain of sample 1 was 23%, while sample 2 and sample 3 had fracture strain of 19% and 16%, respectively. Figures 4-6 show the microstructures parallel to the cylinder axis. All three samples exhibited a typical main crack having an angle of 45° with compression direction due to the largest shear stress in this direction (figure 4a, figure 5a and figure 6a). Cracks between soft TiNi and hard Ti2Ni were detected (figures 4-6). For sample 1, many cracks were observed in Ti2Ni because it was brittle (figure 4c). The cracks in the Ti45/Ni20 layer was larger than Ti40/Ni20 (c.f. figure 4c and 4b) due to small fraction of Ti2Ni in the latter. Because of the symmetrical gradient structure, the top and bottom of sample 2 were seriously broken (figure 5(a)) due to large fraction of brittle Ti2Ni in Ti50/Ni20 layer; while many small cracks were observed along the interfaces between soft TiNi and hard Ti2Ni (figure 5b). For sample 3, similar fracture behavior was observed, showing a main crack and many small cracks. The sample 3 was fracture earlier than sample 2 because harder parts of sample 3 were in the middle.

**Figure 3.** Compressive engineering stress-strain curves at static condition.
Figure 4. Fracture behaviour of sample 1.

Figure 5. Fracture behaviour of sample 2.

Figure 6. Fracture behaviour of sample 3.

Figure 7 shows the load-displacement curves of different samples after three-point bending.
According to our previous analysis [8], the serrations in the curves indicated the deflection of cracks between soft TiNi and hard Ti2Ni. The thickness of ductile layer in the middle of sample 2 was the largest due to hard-soft-hard structure. This hindered the crack propagation and delayed the fracture, leading to the largest fracture toughness (37.9±2.7 MPa·m$^{1/2}$) in comparison with sample 1 (27.2±5.1 MPa·m$^{1/2}$) and sample 2 (30.6±1.8 MPa·m$^{1/2}$).

![Figure 7](image.png)

**Figure 7.** Load-displacement curves of three-point bending tests.

Figure 8 shows compressive engineering stress-strain curves under high strain rates. As shown in figure 8(a), the compressive strength of sample 1 at strain rates of $2.2 \times 10^3$ s$^{-1}$, $2.9 \times 10^3$ s$^{-1}$ and $3.9 \times 10^3$ s$^{-1}$ were 1856 MPa, 1910 MPa and 1935 MPa respectively. As shown in figure 8(b), the compressive strength of sample 2 at strain rates of $1.4 \times 10^3$ s$^{-1}$, $1.9 \times 10^3$ s$^{-1}$ and $2.2 \times 10^3$ s$^{-1}$ are 1791 MPa, 1815 MPa and 1819 MPa respectively. As shown in figure 8(c), the compressive strength of sample 3 at strain rates of $1.4 \times 10^3$ s$^{-1}$, $1.8 \times 10^3$ s$^{-1}$ and $2.2 \times 10^3$ s$^{-1}$ are 1828 MPa, 1940 MPa and 1947 MPa respectively. With increasing strain rate, the strength of all three samples increased, indicating strain rate hardening. As shown in figure 8(d), under the strain rate of $2.2 \times 10^3$ s$^{-1}$, sample 3 showed the best dynamic compression performance while the compression performance of sample 1 and sample 2 were basically the same. Fracture characterizations were shown in figures 9-11 after compression at a strain rate of $2.2 \times 10^3$ s$^{-1}$. A large number of cleavage steps and ductile shear tear cracks were observed in the sample 1 (figure 9). The sample 2 was seriously broken into very small pieces. In addition, there was obvious delamination at the fracture surfaces (figure 10), indicating ductile fracture of TiNi and brittle fracture of Ti2Ni [7]. Interestingly, sample 3 had a relatively small degree of fragmentation and retained a relatively complete part of bottom (figure 11(a)). This fracture behavior was related to soft-hard-soft structure. Due to the top soft layer, it can be deformed and absorb energy. The middle layer was hard and also absorbed energy in the form of cracks and fragments (figure 11(b)). The bottom layer was soft and absorbed energy in the form of plastic deformation.
Figure 8. (a-c) Compressive engineering stress-strain curves at high strain rates of (a) sample 1, (b) sample 2 and (c) sample 3. (d) Compressive engineering stress-strain curves at a strain rate of $2.2 \times 10^3$ s$^{-1}$ for different structures.

Figure 9. Fracture behaviour of sample 1 after dynamic compression at a strain rate of $2.2 \times 10^3$ s$^{-1}$. 
4. Conclusions

In this study, TiNi/Ti$_2$Ni laminated composites were fabricated using hot isostatic pressing (HIP) together with structural designation by the control of Ti and Ni foil thicknesses. During HIP processing, Ti reacted with Ni, leading to the formation of softer TiNi and harder Ti$_2$Ni.

(a) Through adjusting the thickness ratio of Ti and Ni foils, the ratio of TiNi and Ti$_2$Ni fraction can be controlled. Hard-soft-hard, soft-hard-soft and hard-intermediate-soft laminated composites were designed, where hard layer was made by 50 µm Ti and 20 µm Ni, soft layer was made by 40 µm Ti and 20 µm Ni, and the intermediate structure between hard and soft ones was made by 45 µm Ti and 20 µm Ni.

(2) All three laminated composites had compression strength of 1690 ~ 1853 MPa and fracture toughness of 27.2 ~ 37.9 MPa m$^{1/2}$ due to the same constituents of TiNi and Ti$_2$Ni. However, the soft-hard-soft structure showed the best response under high strain rate because it was fractured into larger pieces while the other two structures were fractured into smaller pieces together with smaller...
fracture strain.

(3) This study demonstrated that the mechanical performance of laminated composites can be tuned by arranging soft layer and hard layer in different modes.

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