The Fabrication and Thermomechanical Behavior of Al and Ti SMA Composites

Kiyoshi Mizuuchi

Two kinds of composites—Ti-Ni shape-memory alloy (SMA) fiber-reinforced 6061 aluminum matrix composite and Ti-Pd(Ni) SMA-reinforced titanium-matrix composite—were fabricated by various processing methods and their microstructure and thermomechanical behavior were investigated. In both sets of composites, an increase in yield stress at high temperature was mainly caused by residual compressive stress created in the matrix in association with shape-memory effects of the embedded SMAs.

INTRODUCTION

A "smart" material is one that can spontaneously respond in proportion to the intensity of external stimuli to compensate for the resulting changes or enhance desired effects. Since the introduction of smart materials in 1989 in Japan,[1] these systems have been the subject of considerable attention and research. The ultimate objective of the research is to provide the load-bearing systems of structures with functions similar to those of biological systems, which maintain viability by using energy resources at the highest efficiency. Achieving this objective will lead to greater energy conservation and other benefits.

Early studies of the mechanical properties of aluminum- and polymer-matrix composites showed an improvement of mechanical properties by continuous reinforcement with Ti-Ni SMA fiber.[2,3] Those improved properties mean increases in both yield stress and fracture toughness at elevated temperatures. Considering this and other expected functions such as sensing, SMA-fiber-reinforced composites may be classified as smart materials.

Recently, we fabricated two kinds of composites: Ti-Ni SMA-fiber-reinforced 6061 aluminum-matrix composite[4–9] and Ti-Pd(Ni) SMA-reinforced titanium-matrix composite.[1–12] Their processing, thermomechanical properties and metallography also were investigated.

DESIGN CONCEPT

Figure 1 shows a design concept of smart composites using an SMA reinforcement. The embedded SMA is loaded at room temperature to transform the austenite phase to martensite. Then it is heated to induce its reverse transformation. This SMA shrinks in a composite during the reverse transformation and induces tensile stress in the SMA and compressive stress in the matrix. The compressive stress in the matrix is a key factor that enhances the tensile properties of such a smart composite.

Figure 2 depicts the residual compressive stress in the matrix by showing stress balance in the smart composite. The SMA in the composite is assumed to have a volume fraction of 50%. First, at room temperature, when the composite is loaded in tension up to ε_{ep}, the flow stress of the SMA and the matrix in the composite change along fine dotted and solid lines, respectively. Next, when the tensile stress for the composite is released to 0, the stress for the SMA and matrix decrease. They balance at ε_1 due to the difference between Young's modulus for the SMA and matrix. The composite is then heated beyond A_f where the austenite transformation concludes. At a temperature greater than A_f, the SMA embedded in the composite can largely shrink back to its original length due to the constraint from the matrix. Since the stress-strain curve of the SMA changes to the thick dotted line by heating above A_f from the fine dotted line, the stresses in the SMA and matrix are balanced at ε_2. At this stage, the composite is deformed in tension to its yield point and both the SMA and matrix of the composite can be deformed to a strain ranging from ε_2 to ε_{ep}. As a result, the yield stress of the composite becomes large because of the increase in the elastic limit of the matrix. The dark hatched area in the white thick arrow is the yield stress increase for the composite caused by the residual compressive stress formed in the matrix.

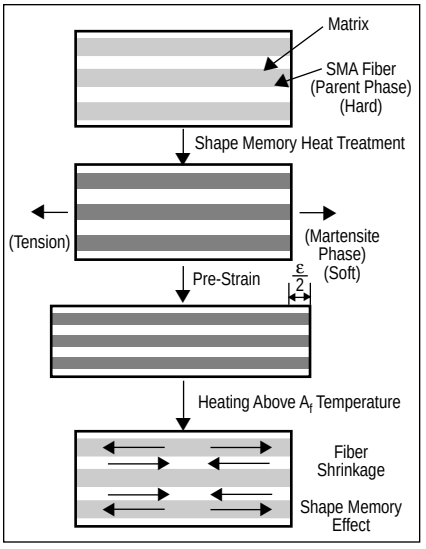


Figure 1. Design concept of a smart composite with SMA.

EXPERIMENTAL PROCEDURE

Four processing methods were used to fabricate SMA-reinforced composites: vacuum hot pressing, hot extrusion, spark plasma sintering, and sheath rolling. The first three were used to fabricate the 6061 aluminum matrix composite reinforced by Ti-50.3Ni (at.%) SMA fiber in short and continuous forms. The fourth process was used to make the Ti-matrix composite reinforced by Ti-25 Pd 24 Ni-1 W (at.%). From this point, the aluminum alloy is referred to as "aluminum," the 50.3 at.% nickel alloy is called "Ti-Ni," and the palladium-containing alloy is cited as "Ti-Pd(Ni)."

To increase the yield stress of the aluminum matrix, T6 heat treatment was carried out for all Ti-Ni-reinforced aluminum-composite specimens after processing. That is, specimens were heat treated up to 803 K in air and held at the temperature for 1.8 ks

for solution treatment, quenched into water, and then aged at 443 K in air for 64.8 ks followed by another water quenching. The water temperature was greater than 293 K to keep the Ti-Ni fibers in the austenite phase after quenching. This T6 heat treatment also applies to the shape-memorization treatment of Ti-Ni fibers embedded in the composites.

In the case of Ti-Pd(Ni)-reinforced composites, shape memorization of Ti-Pd(Ni) SMA embedded in titanium matrix was carried out by heating it to 873 K, holding it for 0.9 ks, and then quenching in water.

Vacuum Hot Pressing

Ti-Ni continuous fiber (200 μm in diameter) and aluminum alloy in a sheet form (0.5 mm thick) were used. Transformation temperatures for Ti-Ni fiber were M_s = 289 K, M_f = 280 K, A_s = 318 K, and A_f = 329 K. The chemical composition of the aluminum sheet was Al-bulk, Cu-0.15-0.40, Mg-0.80-1.2, Si-0.4-0.8, Fe-0.7, Mn-0.15, Cr-0.04-0.35, Zn-0.25, and Ti-0.15. As seen in Figure 3, the aluminum sheet was first sheared into a rectangular shape 10 mm wide. Then, a low-speed diamond wheel cutter was used to make slits measuring 0.3 mm wide and 1 mm long in both narrow sides of the sheet with an interval of 1 mm. Ti-Ni fiber was wound around the sheets through slits, and several prepreg sheets were stacked on the lower part of hot press dies. Then, composite specimens were fabricated by a custom-made vacuum hot press system, (Figure 4). Fabrication of the composite was performed under a vacuum of 2 Pa by heating to 823 K for 12.0 ks at a pressure of 1.4 MPa, followed by heating at 823 K for 1.8 ks at a pressure of 7.0 MPa and furnace cooling without loading. Composite specimens approximately 80 mm wide and 100 mm long were fabricated. The reinforcement volume fraction of the composite was 5.3%.

Hot Extrusion

An aluminum-matrix composite with a short-fiber Ti-Ni reinforcement was also fabricated, where the short fibers were 200 μm in diameter and 3 mm to 6 mm long; the aluminum matrix material was powder. The powder and short fiber were mixed at a ratio of four by volume percent. The chemical compositions of the as-received 6061 aluminum alloy powder were Al = 97.68, Mg = 1.10, Si = 0.62, Cu = 0.28, Cr = 0.19, Fe = 0.10, and Mn = 0.03. The particle size distributions of the as-received 6061 aluminum alloy powder were Al -44μm , 58.6 (wt.%); Mg 44 μm to 63 μm, 6.7 (wt.%); Si 63μm to 74 μm, 6.7 (wt.%); Cu 74 μm to 105 μm, 9.1 (wt.%); Cr 105μm to 149 μm, 7.4 (wt.%); Fe 149 μm to 350 μm, 4.2 (wt.%); and Mn 350 μm, trace. The short fiber has the same transformation temperatures as those used for the vacuum hot-pressing. Figure 5 shows the hot extrusion process. After mixing the powder and short fiber thoroughly, the mixed material was poured in an aluminum can (28 mm inside diameter, 50 mm height and 5 mm wall thickness). The can was then tapped, and cold compacted to make the mixture into a 45-mm high cylindrical shape. The relative packing density of the compacted mixture was about 50%. The can was capped with an aluminum lid (28 mm in diameter and 5 mm high), heated to 773 K and hot extruded in air to a rod with a diameter of 6 mm. After hot extrusion, the material was reheated without removing the aluminum can at 873 K for 1.8 ks to make an aluminum-matrix composite containing Ti-Ni short fiber.

During reheating, the reaction region was formed along the boundary between the matrix and fiber. After removing the aluminum can, the composite (in a rod shape) was machined to make tensile specimens with a gauge size of 4 mm in diameter and 10 mm long. Before machining and after removal of the aluminum can, the composite was roughly 4.4 mm in diameter. The extrusion ratio, R, was about 40.

Spark Plasma Sintering

Aluminum alloy powder and Ti-Ni fiber (0.5 mm in diameter and 58 mm long) as reinforcement were used to fabricate a composite via a spark plasma sintering. First, the lower punch was inserted into the die from one end and 4.9 g of aluminum alloy powder was added. Then, Ti-Ni fiber was placed on the top of the leveled powder with a constant spacing. Another 4.9 g of the powder covered the aligned Ti-Ni fiber and the upper punch was placed on the powder. The powder and Ti-Ni fiber were pressed at

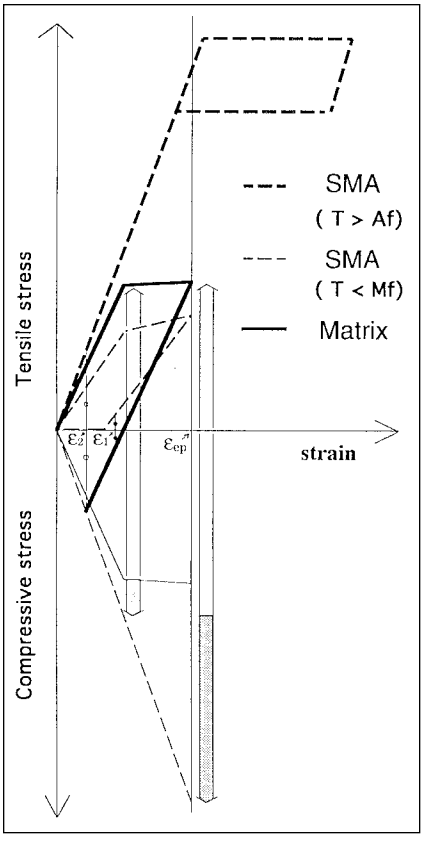


Figure 2. Stress balance in a smart composite with SMA at the temperatures below M_f and above A_f.

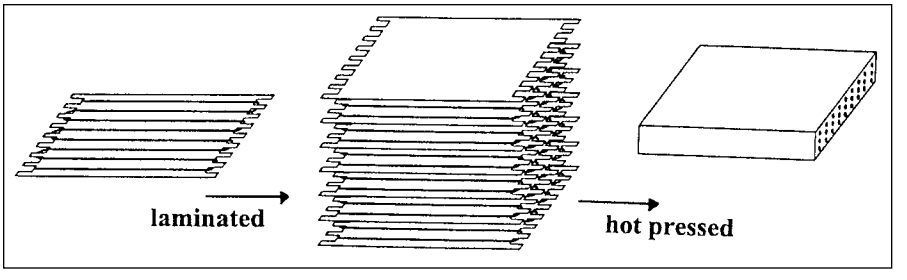
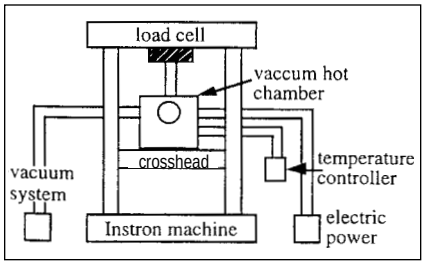
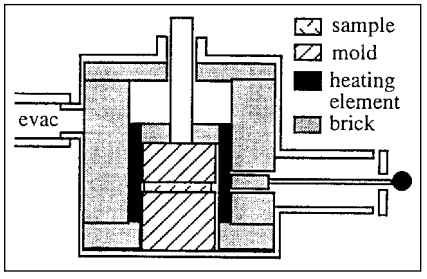


Figure 3. Configuration of smart-metal matrix composite with continuous SMA fiber reinforcement.



a



b

Figure 4. Schematic drawing of a custom-made vacuum hot press system and side view of sectioned vacuum hot chamber.

a pressure of 32 MPa through the upper and lower punches. Spark plasma sintering was performed after evacuation to a vacuum of 2 Pa. Sintering was done at 813 K at a heating rate of 2 K/s. The holding time was 0.3 ks. Longitudinal displacement between the upper and lower punches was monitored, along with the temperature of the die. Discharged pulsed current used was about 800 A, and spark was generated for a given interval of time.

After processing, the plate-shaped composite was machined to make tensile specimens with gauge dimensions of 9 mm wide, 1.5 mm thick, and 60 mm long. The Ti-Ni fiber volume fraction was 20%.

Sheath Rolling

Ti-Pd(Ni) SMA-reinforced composites were fabricated by a sheath-rolling process (Figure 7) using pure titanium as the matrix and plate-shaped Ti-Pd(Ni) SMA as the reinforcement. This SMA plate was made by hot rolling after levitation melting in an argon atmosphere (0.1 MPa) using a copper crucible in the shape of buttons 30 mm in diameter and 20 mm high. The nominal composition (at.9%) of this SMA plate was 50Ti-25Pd-24Ni-1W. Transformation temperatures were M_s (0) = 450 K, M_f (0) = 426 K, A_s (0) = 441 K, and A_f (0) = 465 K

Two SMA plates and three titanium plates were alternatively laminated and inserted in a SUS304 stainless steel pipe with a 20 mm inside diameter and 1 mm of wall thickness, followed by cold rolling to a flat, 7-mm thick pipe. Both ends of the pipe were spot-welded in a vacuum of 0.1 Pa. The pipe was heated to 1,123 K and sheath rolled to 6 mm thick to form a roughly 4-mm thick material inside the pipe from 5 mm thick.

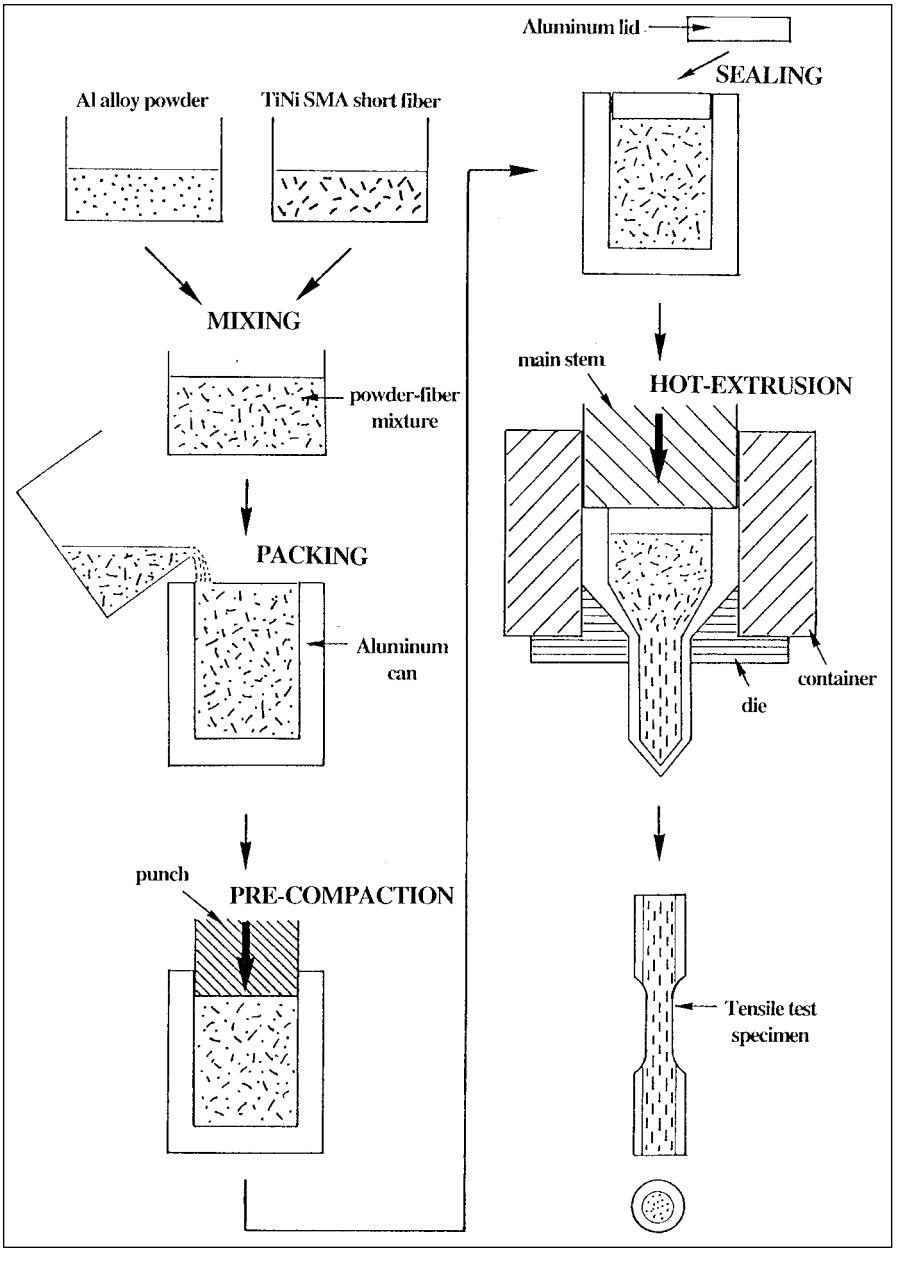


Figure 5. Schematic illustration of hot extrusion process.

After sheath rolling, the pipe was reheated at 1,123 K for 3.6 ks in a furnace and opened to remove a sheath-rolled composite. By machining, tensile specimens were prepared with a gauge size of approximately 10 mm × 4 mm × 40 mm.

Ti-Ni Reinforcement

Scanning electron microscopy (SEM) of specimens made via vacuum hot pressing, hot extrusion, and spark plasma sintering revealed no porosity on any of the interfaces between the Ti-Ni fiber and aluminum matrix. In addition, the Ti-Ni fibers are circular, indicating no heavy deformation occurred during fabrication. This could be caused by the combined effect of the soft aluminum matrix and hard Ti-Ni SMA fiber and suitable processing parameters. Further analysis seems to reveal a roughly 5 μm-thick interfacial reaction layer in the boundary between the fiber and aluminum matrix in all cases.

The interface formed by vacuum hot pressing and hot extrusion is a duplex-layered structure consisting of Al_3Ti and Al_3Ni layers. The Al_3Ti layer was formed next to the Ti-Ni fiber and the Al_3Ni layer next to the aluminum matrix. In the composite fabricated by spark plasma sintering, a triple-layered interface composed of Ni_3Ti, Ti_2Ni, and Al_3Ni was formed between the matrix and fiber. The Ni_3Ti layer was formed next to the Ti-Ni fiber, and the Al_3Ni layer was formed next to the aluminum matrix.

The difference of the interfacial reaction between composites fabricated by the spark plasma sintering and vacuum hot pressing or hot extrusion may be related for the following reasons: The spark plasma sintering process was performed in a short time (within 0.6 ks); the bonding of particles occurs mainly by instant evaporation of particle surface followed by condensation by high current passage due to skin effects by plasma sparking, which cleans the surface of particles and facilitates bonding especially under compression; and the interface growth rate may decrease sharply after bonding, because the main sintering mechanism changes from evaporation and condensation to interdiffusion.

Ti-Pd(Ni) Reinforcement

Ti-Pd(Ni) SMA-reinforced titanium matrix composites produced by sheath rolling were cut with a SiC wheel and polished mechanically using emery paper and alumina powders. Bonding between the titanium matrix and the Ti-Pd(Ni) SMA reinforcement was homogeneous. No de-bonding occurred along the boundary between the SMA and matrix. An interfacial reaction layer about 30-μm thick exists between the matrix and SMA plates. Additional analysis revealed that the interfacial layer has a triple-layered structure composed of palladium-poor, nickel-poor, and titanium-rich layers. The palladium-poor layer was next to the Ti-Pd(Ni) SMA plate, and titanium-rich layer was formed next to the titanium matrix. In the titanium-rich layer, titanium

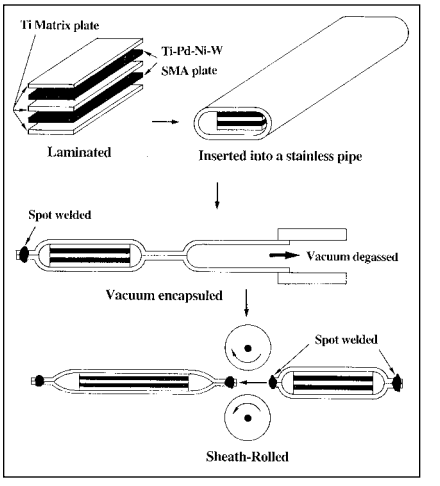


Figure 7. Schematic illustration of sheath rolling process.

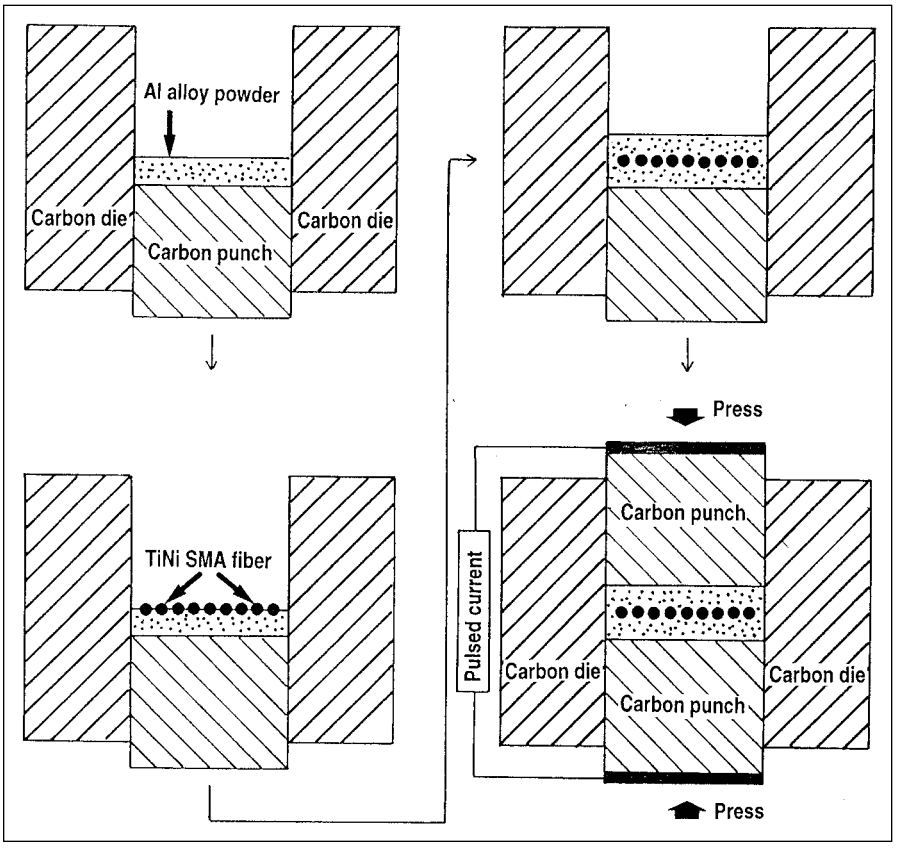


Figure 6. Schematic illustration of spark plasma sintering process.

content increased with increasing distance from the SMA. The formation of these three interfacial layers may be explained by the diffusion behavior of palladium and nickel into the titanium matrix.

Ti-Ni Reinforcement Composites

Tensile tests were carried out for Ti-Ni SMA fibers with a gauge size of 100 mm diameter and 100 mm long. For a Ti-Ni SMA fiber deformed at 293 K and deformed further at 373 K, the yield stress of the fiber is about 500 MPa at 373 K, approximately twice as high as that (about 250 MPa) at 293 K. This result indicates that Ti-Ni fiber can be effective as composite reinforcement if used at temperatures of at least 373 K.

Tensile tests were performed at 293 K (between M_S and A_s of Ti-Ni) and 373 K (above A_f) for Ti-Ni fiber-reinforced aluminum-matrix composites fabricated by spark plasma sintering, vacuum hot pressing, and hot extrusion. In all cases, tensile yield and flow stresses were lower at 293 K than at 373 K. For example, the gauge dimensions of tensile test specimens fabricated by spark plasma sintering were 9 mm wide, 1.5 mm thick, and 60 mm long. For details on the deformation processing, see References 4 through 9. The yield stress of the continuous-fiber-reinforced composites at 373 K was 42 MPa higher than that at 293 k. For the short-fiber-reinforced composites, the yield stress at 373 K was only 25 MPa higher than the flow stress of the composite deformed to 1.10% at 293 K. This difference of yield stress increase between continuous-fiber-reinforced composites and short-fiber-reinforced composites would be caused by the difference of the fiber aspect ratio between continuous and short fibers.

This stress increase at higher temperatures is attributed to the following: the compressive stress generated in the composite matrix due to the shape recovery of the Ti-Ni fiber after heating to a temperature higher than A_f; the compressive stress generated in the matrix caused by the coefficient of thermal expansion mismatch between fiber and matrix after heating; and the stress enhancement of the fiber at a temperature higher than A_f.

Ti-Pd(Ni)-Reinforcement

Tensile tests were performed for a Ti-Pd(Ni) SMA with a gauge size of 10 mm wide, 1 mm thick, and 40 mm long. For a Ti-Pd(Ni) SMA specimen obtained in tension at 293 K and deformed further at 433 K (slightly below A_s) and 473 K (slightly above A_f), the yield stress of the Ti-Pd(Ni) specimen is about 400 MPa at 473 K, much higher than that at 433 K (about 280 MPa) and 293 K (about 320 Mpa). This indicates that high-temperature Ti-Pd(Ni) SMA can be used as a reinforcement of composites at temperatures at least up to 473 K.

As for the Ti-Pd(Ni)-reinforced Ti composite made by sheath rolling, the gauge dimensions of the tensile test specimens were 10 mm wide, 4 mm thick, and 40 mm long. The yield stress of the composite is 285 MPa at 433 K and 335 MPa at 473 K. That is, the yield stress is about 50 MPa higher at 473 K than at 433 K. The increase in the yield stress at a higher temperature is significant, because the yield stress of pure titanium decreases monotonically from 373 K to 473 K.[10–12] The stress level is also increased significantly for the titanium-matrix composite between 297 K and 473 K, as compared with that of pure titanium. For example, the yield stress of the composite is 285 MPa at 433 K, while pure titanium is 180 MPa.[10–12] This strength increase of the composite can be attributed to the same three reasons mentioned earlier. It should be emphasized that titanium-matrix composite becomes as strong as about 300 MPa in an intermediate temperature range at least up to 473 K by using high-temperature Ti-Pd(Ni) SMA. This can be compared with the result obtained from an aluminum-matrix composite reinforced by lower-temperature Ti-Ni SMA.

FRACTURE

Ti-Ni-Reinforcement

Figure 8 shows the fracture surface of Ti-Ni short-fiber-reinforced aluminum-matrix composite produced by hot extrusion. Figure 8a shows short fibers of Ti-Ni

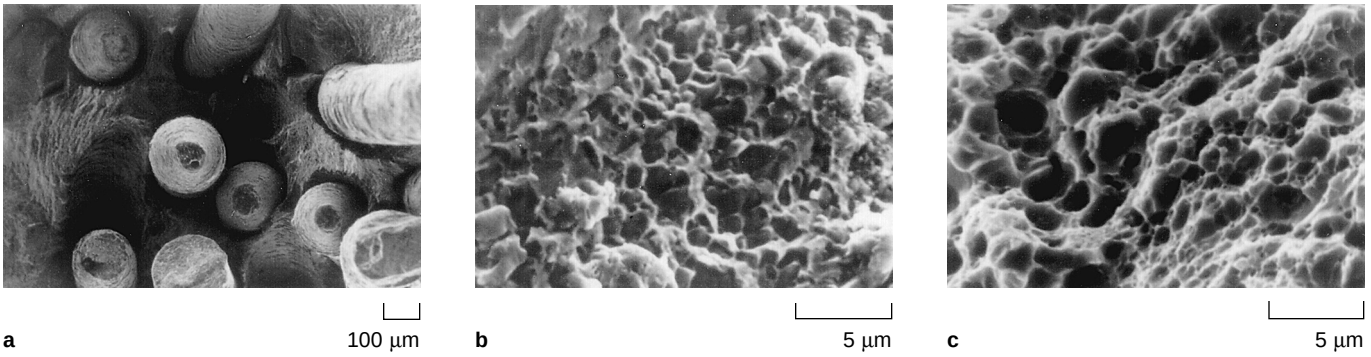
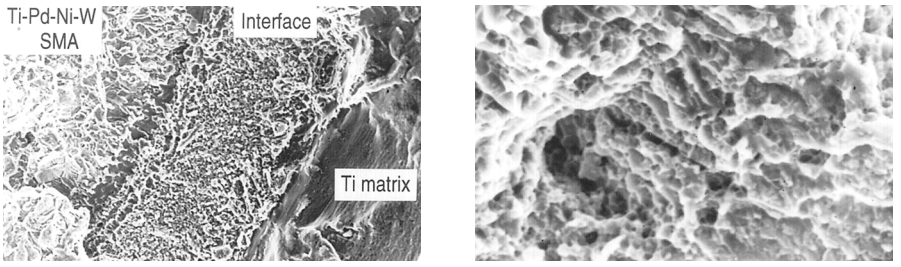


Figure 8. SEM images of fracture surfaces for a Ti-Ni reinforced aluminum-matrix composite reinforced by Ti-Ni short fiber, heat treated at 873 K for 1.8 ks after hot extrusion: (a) composite, (b) fiber, and (c) matrix.



a **b**

Figure 9. SEM images of fracture surfaces for a (a) Ti-Pd(Ni) reinforced titanium-matrix composite showing ductile dimple fracture surfaces at the (b) interface between the matrix and fiber. There is no de-bonding between the matrix and fiber.

pulled out from the aluminum matrix. Closer views of the fracture surface in Figure 8b and Figure 8c reveal that both the fiber and matrix show typical ductile dimple structure. Similar tendencies were also observed for the fracture surface of Ti-Ni long-fiber-reinforced aluminum-matrix composite specimens produced by vacuum hot pressing and spark plasma sintering. In all cases, Ti-Ni fibers were pulled out and the interfacial region formed between the Ti-Ni fiber and aluminum matrix seems not to be ductile. As referenced earlier, the interface region is a multi-layered structure consisting of Al_3Ti and Al_3Ni intermetallic phases. These phases are of DO_{22} structure and known to be brittle. To increase the ductility of this interfacial layer, the crystal structural modification, say from DO_{22} to $L1_2$, would be effective.[13,14] If improvement in the mechanical properties of the interfacial layer are made, stresses generated between the matrix and Ti-Ni SMA fiber can be more effectively transferred, resulting in a further increase in stress at 373 K.

Ti-Pd(Ni)-Reinforcement

Figure 9 shows SEM images of a fractured Ti-Pd(Ni) SMA reinforced titanium-matrix composite fabricated by sheath rolling. No apparent de-bonding between the matrix and SMA reinforcement was observed, even when deformed several percents in tension at 473 K (Figure 9a). This indicates that the Ti-Pd(Ni)-reinforced titanium composite evidently has good bonding between the titanium-matrix and reinforcement plate. Good bonding can be more readily seen in higher-magnification micrographs (Figure 9b). Clearly, there are typical ductile dimples on the fracture surface in the interfacial layer of the composite. Namely, the interfacial layer is ductile, and stresses generated between the titanium-matrix and Ti-Pd(Ni) reinforcement are considered to be effectively transferred, resulting in large increases in yield stress in the temperature range between 293 K and 473 K, when compared with the offset stress of pure titanium.

References

1. *Proc. Int. Workshop on Intelligent Materials* (Warrendale, PA: Technomic Publishing Co., 1989).
2. M. Taya et al., *Proc. Smart Materials,* ed. V.K. Varadon (Bellingham, WA: SPIE, 1993), p. 373.
3. A. Shimamoto, Y. Furuya, and M. Taya, *Proc. Int. Symp. Microsystems, Intelligent Materials and Robots,* ed. J. Tani (Bellingham, WA: SPIE, 1995), p. 463.
4. K. Mizuuchi et al., *US-Japan Workshop on Smart Materials and Structures*, ed. K. Inoue, S.I.Y. Shen, and M. Taya (Warrendale, PA: TMS, 1997), p. 75.
5. K. Hamada et al., *MRS Proc. Materials for Smart Systems II*, 459 (1997), p. 143.
6. J.H. Lee et al., *MRS Proc. Materials for Smart Systems II*, 459 (1997), p. 419.
7. K. Hamada et al., *Metall. and Mat Trans A,* 29A (1998), pp. 1127–1135.
8. K. Mizuuchi et al., *9th Cimtec-World Forum on New Materials Symposium VIII: Smart Materials Systems*, ed. P. Vincentini (Faenza, Italy: Techna Srl, 1999), p. 61.
9. K. Mizuuchi et al., *Kagaku to Kogyo,* 74 (2000), pp. 100–105.
10. K. Mizuuchi et al., *J. Jpn. Inst. Metals,* 61 (1997), p. 727.
11. K. Mizuuchi et al. *Third Pacific Rim Int. Conf. Adv. Mater. and Processing (PRICM-3),* vol. II (1998), p. 2051.
12. K. Mizuuchi et al., *Proc. Int. Conf. Solid-Solid Phase Transformations'99 (JIMIC-3),* vol. II, ed. M. Koiwa, K. Otsuka, and T. Miyazaki (1999), pp. 1044–1047.
13. H.R.P. Inoue et al., *MRS Symp. Proc. High Temperature Ordered Intermetallic Alloys III,* 213 (1991), p. 493.
14. K. Mizuuchi et al., *MRS Symp. Proc. High Temperature Ordered Intermetallic Alloys V*, 288 (1993), p. 1191.

Kiyoshi Mizuuchi is with Osaka Municipal Technical Research Institute.

For more information, contact Kiyoshi Mizuuchi, Osaka Municipal Technical Research Institute, Osaka 536-8553, Japan; e-mail: mizuuchi@omtri.city.osaka.jp.