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Aluminum-based composites reinforced with SiC particles and NiTi fibers: influence of fiber dimensions and aging time on mechanical properties

Zariff Chaudhury · Mohammad Hailat · Yi Liu · Golam Newaz

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Abstract NiTi short fiber and SiC particle reinforced 6061 Al alloy composites have been prepared by pressure-assisted induction heating method in ambient atmosphere. Two different composites with significant difference in NiTi fiber diameter (127 and 51 μm) have been prepared. It is found that 51-μm NiTi fibers are better bonded with the Al matrix; de-bonding has seldom been observed. There is better microstructural compatibility, i.e., fiber diameter is of the same order as the Al matrix grain size for this case. However, when 127-μm NiTi fibers are used, de-bonding and "pull-out" mechanism can be frequently observed. Aging effect on the properties of the composite has also been investigated. TEM and EDS experiments indicate that there is no evidence that the bonding of NiTi/Al is affected by either aging or size of NiTi fiber.

Z. Chaudhury
Metallurgical Technology, Technology Program, College of Agriculture and Technology, Arkansas State University, Jonesboro, AR 72467, USA

M. Hailat · G. Newaz
Department of Mechanical Engineering and Institute for Manufacturing Research, Wayne State University, Detroit, MI 48202, USA

Y. Liu
Department of Chemistry, Wayne State University, Detroit, MI 48202, USA

Y. Liu (✉)
Electron Microscopy Facility, Oregon State University, Corvallis, OR 97331, USA
e-mail: yi.liu@oregonstate.edu

Introduction

Shape memory alloy (SMA) reinforced composites have been of interest during the past two decades [1–27]. NiTi SMA in the form of either fibers [1, 2, 4, 7, 11, 15–20, 22, 28] or particles [13, 14, 23, 24, 29] were selected in most of the studies in the literature although CuZnAl SMA has also been considered [12, 25–27]. The unique shape memory effect (SME) of NiTi SMA has been utilized to apply a compressive stress to the matrix, so that the ductility and fatigue resistance of the composite are enhanced [1, 4, 7, 13–17, 29]. The compressive stress generated by NiTi SMA results from the reversible thermo-elastic phase transformation between austenite with *B2* structure and martensite with monoclinic *B19′* structure. There are temperature ranges for the phase transformation of SMA, i.e., austenization start and finish temperatures denoted by A_s and A_f, and martensite transformation start and finish temperatures denoted as M_s and M_f. By a certain heat-treatment procedure, the SMA can remember and recover its previous geometric shape via phase transformation when temperature changes are applied. The temperature range of the transformation is usually lower than 100 °C depending on ways of heat treatment and alloy processing history [1, 29–32], which makes SMA very operational and applicable.

The diameter of the SMA fibers employed in most studies is too large ranging from 190 to 500 μm [1, 4, 11, 19, 21, 28, 33] compared with the grain size of the metal matrix, which is usually around 10–20 μm for aluminum alloys [1, 4, 21]. As a result, the composites are not reinforced "microscopically" and the coarse NiTi fibers undertake much load than the matrix when the composite is deformed. Early de-bonding at the interface happens, and "pull-out" mechanism of the fiber has been frequently

observed from the fracture surface of the composite [1, 15, 33]. One interesting study conducted by Zhu and co-workers [34] obtained an improved tensile strength from Ni fiber reinforced polyester matrix composite. In their study, the diameter of the fiber was 76.2 μm, and the long fibers were cut into 2.5-mm short fibers by a flame from a mini-hydrogen torch. The flame melted the cutting locations on the fibers, and the melted ends of the short fibers formed two balls with an average diameter of 183 μm. The bone-shaped short fiber resisted the fiber be "pull out" during failure and made a great contribution to the improved tensile strength. However, de-bonding at the interface and "pull-out" mechanism were still observed [34].

Porter et al. [14] first came to the idea that the NiTi particles should be as small as possible in the composite and pointed out several potential problems in the production of the NiTi-reinforced aluminum composite, such as poor bonding between NiTi particles and Al matrix and interface reaction by diffusion. They fabricated a NiTi SMA particle-reinforced aluminum composite by mixing Al with 99 wt% in purity with 10 vol.% NiTi particles with about 5 μm in diameter obtained by mechanical milling from 40 μm [14, 29]. They found that the yield stress and ultimate strength increased about 43 and 54%, respectively. The smaller and mechanically milled NiTi particles also greatly enhanced the fatigue peak stress and fatigue life [13].

The present investigation is aimed to study the size effects of short NiTi fiber and aging conditions on microstructure and mechanical properties in NiTi short-fiber reinforced 6061 Al/SiC composite. The interface bonding quality between the NiTi fibers and the matrix of the aluminum alloy composite has also been studied by scanning electron microscope (SEM) and transmission electron microscope (TEM). It has been identified that the mechanical properties is degraded when the coarser (127 μm in diameter) short NiTi fibers is used. SME of NiTi SMA short fiber on the matrix has not been investigated in this current work. It will be addressed in our other research work.

Table 1 Chemical composition of the as-received 6061 Al powder (wt%)

Cr	Cu	Fe	Mg	Mn	Si	Ti	Zn	Al
0.08	0.27	0.26	0.97	0.02	0.56	0.02	0.05	Balance

Table 2 Typical chemical composition of the as-received SiC powder (wt%)

SiC	SiO_2	Si	Fe	Al	C
98.5	0.5	0.3	0.08	0.1	0.3

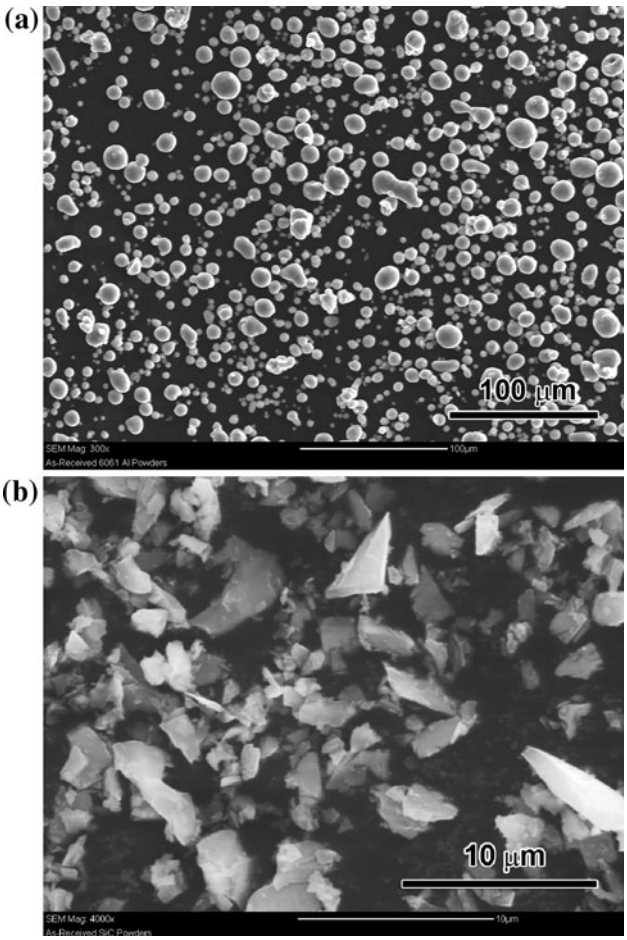

Fig. 1 SEM images of (**a**) 6061 Al alloy powders and (**b**) SiC powders

Experimental

The NiTi short fiber-reinforced 6061 Al—5 wt% SiC composites were prepared by pressure-assisted sintering technique in ambient atmosphere as described in details by Xie et al. [1]. 6061 Al atomized uncoated powders with the average size of 30 μm were mixed with 5 wt% SiC powders with the average size of 2.5 μm in a ball mill for about 6 h (Tumbler Model A), the rotary speed is 1,550 rpm.

Table 3 Nominal chemical composition (wt%) of the as-received NiTi fiber

Ni	Ti	O	H	C	Cu	Fe	Co	Cr	Nb
56.2	Balance	0.026	0.001	0.0033	<0.005	<0.005	<0.005	<0.005	<0.005

No ball was used during mixing. The 6061 Al powders were obtained from Valimet Inc. (Stockton, CA) and its chemical composition is listed in Table 1. The SiC powders were obtained from Electro Abrasive Corporation (Buffalo, NY) and its chemical composition is listed in Table 2. The NiTi SMA fibers used in the present investigation were super elastic grade SMA (SE508) having diameters of 127 and 51 μm. The NiTi fiber was obtained from Nitinol Devices & Components (Fremont, CA) and the chemical composition (in wt%) is shown in Table 3. It contains 56.2% Ni, 0.026 wt% oxygen, 0.001 wt% H, 0.0033 wt% C, and <0.005 wt% Cu, Fe, Co, Cr, Nb, and balanced Ti. The long NiTi fibers were chopped into 8–10 mm in length. The as-received fiber was in cold-worked condition. Based on the vendor, the start and end temperatures of martensite transformation M_s and M_f in the as-received are below 0 °C. The surface of the NiTi fiber was etched in a 40% nitric acid solution for 48 h followed by washing in alcohol and drying to remove any possible oxide layer which may hamper the reaction bonding quality at the interface. NiTi fibers with both 127 and 51 μm in diameters were selected to make the composite. Figure 1 shows SEM images of the as-received 6061 Al alloy powders (Fig. 1a) and SiC powders (Fig. 1b).

The mixed powders containing 6061 Al alloy and SiC were further mixed with the NiTi fibers. The volume fraction of NiTi fibers is 0.4%, which corresponds to 1 wt%. The final mixture was placed in a die mold with 57 mm in diameter by using a spatula spoon to make sure that the fibers are distributed uniformly with the powders and compacted at about 160–170 kN on a MTS 801 Test System to form a solid green compact as shown in Fig. 2. The maximum load of the test system is 220 kN. Boron nitride spray was applied to the inner surface of the die mold to prevent the composite from sticking to the mold after sintering. A 5 kW Ameritherm induction unit was employed to heat the mold to the expected temperature. The green compact solid was sintered at 585 °C with a

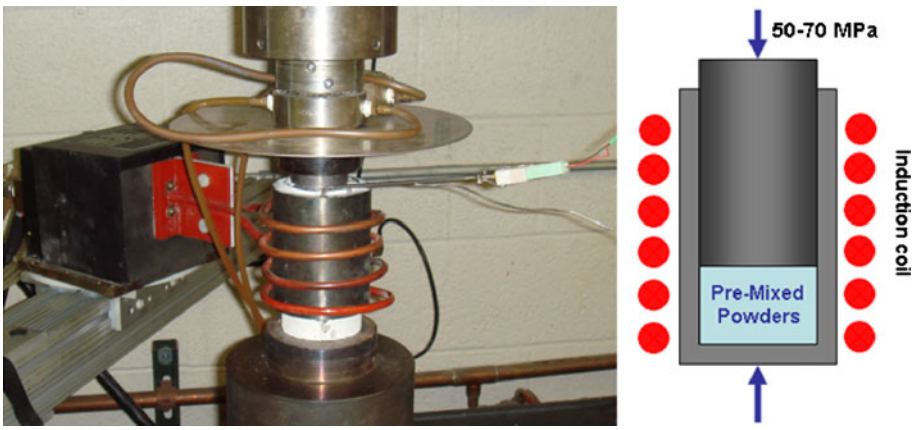

Fig. 2 The setup of the pressure-assisted sintering technique used in the present study

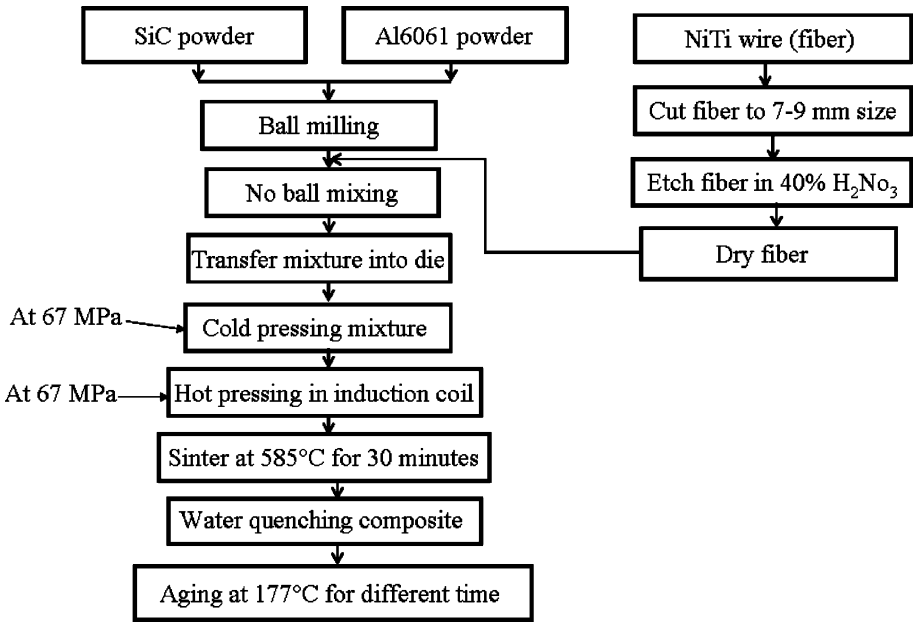

Fig. 3 Procedures for the preparation of the 6061 Al composites reinforced by SiC and NiTi fiber

tolerance of ±3 °C under a pressure of 67–70 MPa. The temperature 585 °C for sintering was selected in order to conduct solid solution treatment of the composite.

Then the final sintered composite was quenched in water immediately after the pressure-assisted sintering. It was reported that four factors are critical for optimal fabrication condition, i.e., heating rate, sintering temperature, pressure, and time [1]. Good mechanical properties have been obtained at a heating rate of 20–25 °C/min, a sintering temperature of 585 °C and a hot uniaxial pressure of 65–70 MPa for 25–30 min [1]. A 6061 Al—5 wt% SiC composite without NiTi fiber was also prepared in the same way as described above for comparison purpose. The whole procedure of the composite preparation is shown in Fig. 3.

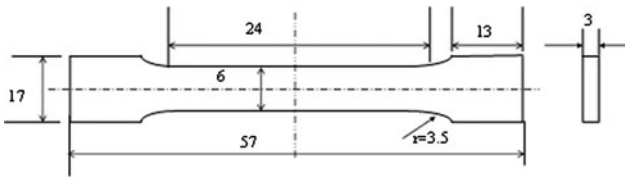

Fig. 4 Specimen for the tensile test (in mm)

Flat dog-bone tensile test samples were cut from the as-prepared composites by electric discharge machining (EMD). Figure 4 shows the dimension and the shape of the tensile test specimens. Since the composites were quenched from 585 °C which was actually a solid solution treatment, in order to improve the mechanical properties of the 6061 Al alloy matrix, the samples were aged at 177 ± 3 °C for 30, 60, and 180 min for precipitation followed by air cooling. The complete heat treatment cycles from solid solution treatment to aging treatment at 177 °C is shown in Fig. 5. Some tensile samples were kept without aging, after the solid solutions heat treatment, for comparison with the aged samples.

Samples were cut by a low speed diamond saw from all the as-prepared and heat-treated composites. The samples were then polished by using various grades of metallographic sand papers to see the distribution of fibers in the 6061 Al alloy matrix. The NiTi fiber-reinforced composite samples used for hardness tests contain 0.5 wt% 51-μm NiTi fibers and 0.5 wt% 127-μm NiTi fibers. Vickers microhardness tests were conducted on Leco LM100

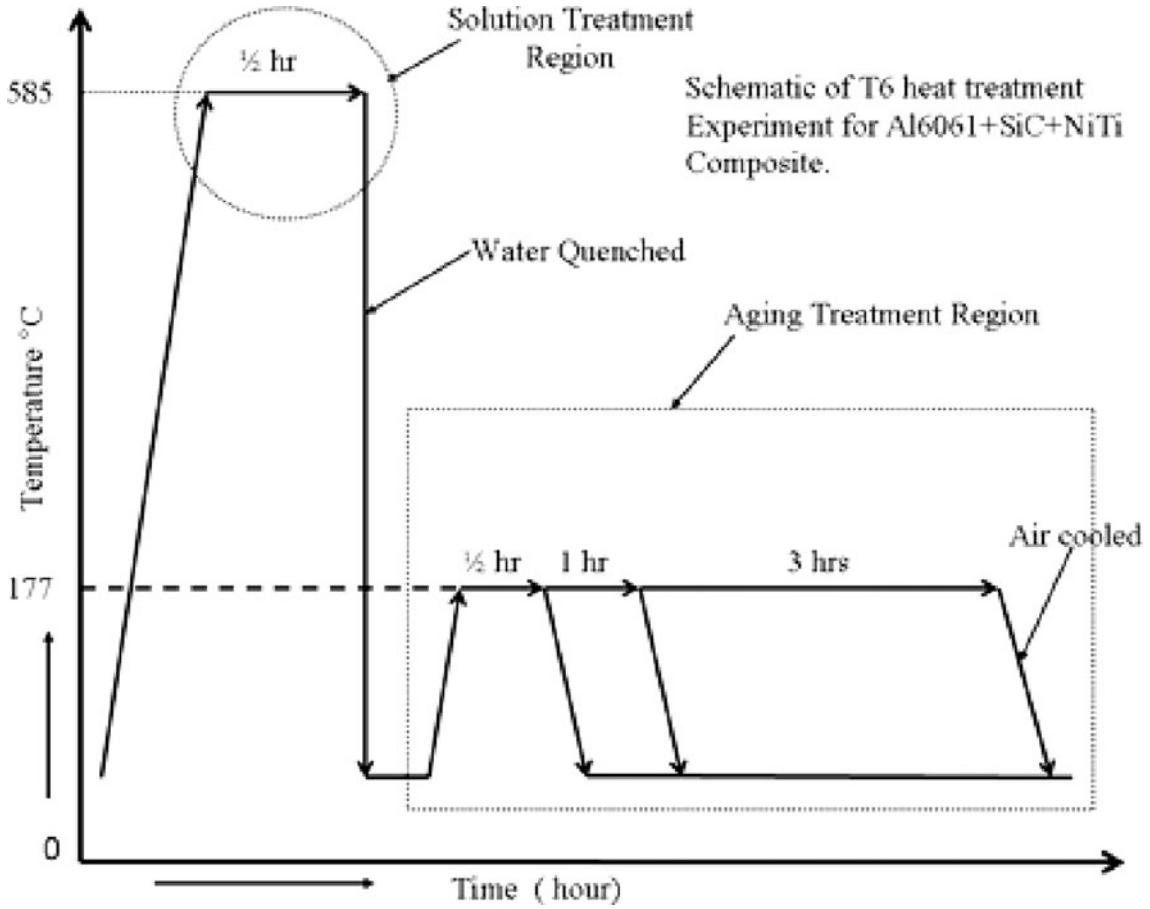

Fig. 5 Schematic diagram of the heat-treatment experiments for 6061 Al + SiC + NiTi fiber composite

microhardness tester. In most of the tests, 200 g load for 15 s was used, while some of the data were collected under 100 g load for 15 s. No obvious difference was found by using different load in the present study. The microhardness was measured 10 times on each sample. The uniaxial tensile tests were conducted on MTS 810 test machine with hydraulic wedge grips. The strain rate was 2.3×10^{-3}/s. Nikon Eclipse LV100D-U optical microscope was used to check the microstructure of the samples at low magnification. The samples for optical microscope examinations were etched by using Keller's agent, which contains 2 mL HF, 3 mL HCl, 5 mL HNO_3, and 190 mL distilled water.

The fractured tensile specimens were characterized by using a Hitachi S-2400 SEM operating at 25 kV. The interface between NiTi fibers and Al alloy matrix were studied by using a JEOL-2010 FasTEM TEM operating at 200 kV. Energy dispersive spectrometry (EDS, EDAX, Inc.) was also used to detect local chemical composition near the NiTi/Al interface. TEM foils of the composites

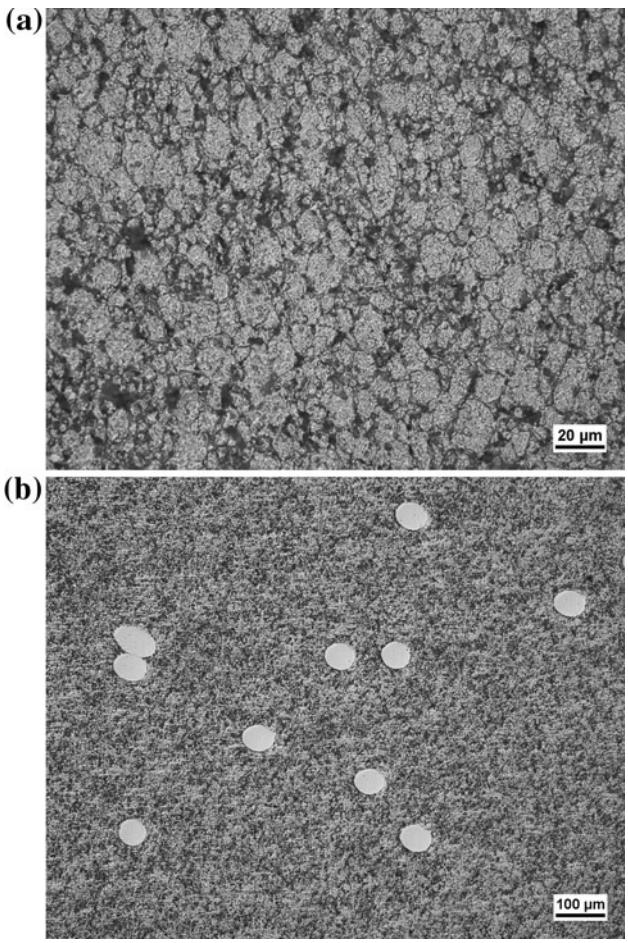

Fig. 6 Typical optical microscope images of (**a**) SiC distributed in 6061 Al Alloy matrix and (**b**) 51-μm NiTi fibers in 6061 Al alloy matrix with 5 wt% SiC powders

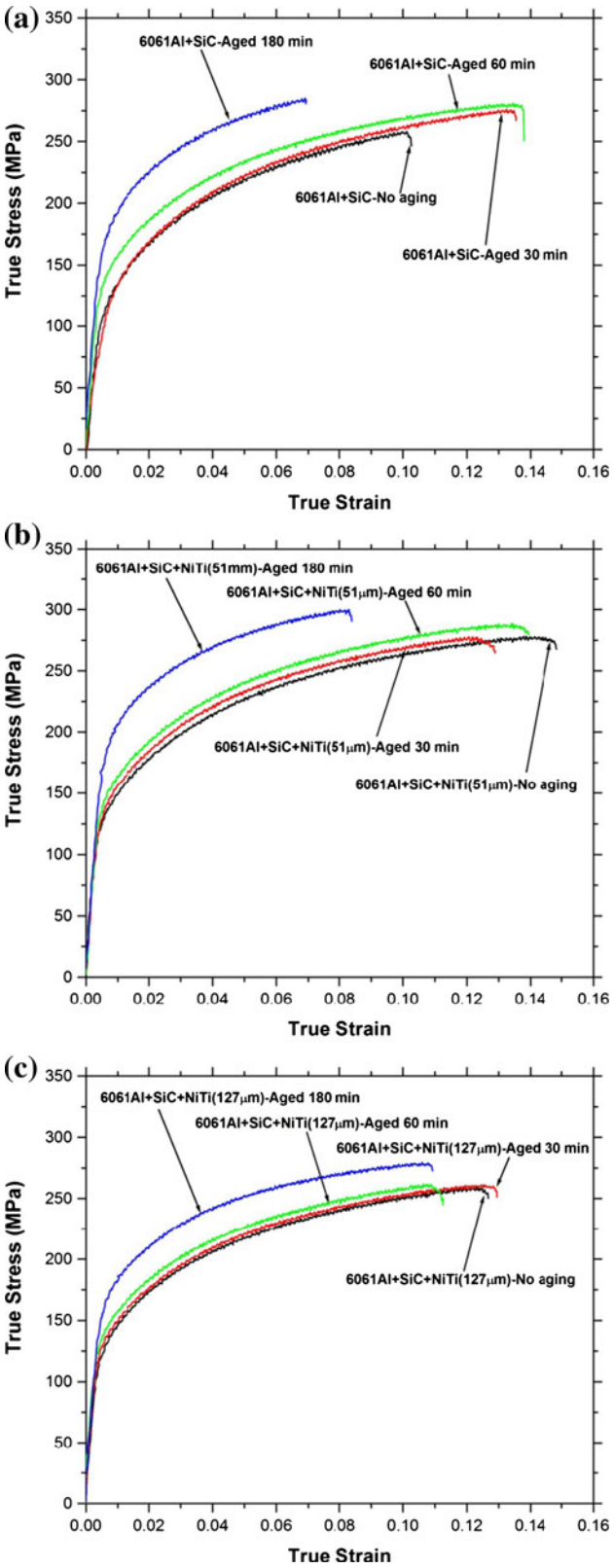

Fig. 7 Aging effect on the mechanical properties of (**a**) 6061 Al—5 wt% SiC composite, (**b**) 6061 Al—5 wt% SiC–1 wt% NiTi (51 μm) composite, and (**c**) 6061 Al—5 wt% SiC–1 wt% NiTi (127 μm) composite

were prepared by mechanically grinding followed by ion-beam milling. Gatan 691 Precision Ion Polishing System (PIPs) was used under 4.5–5 kV with a beam incident angle of 5–7°.

Results and discussion

Figure 6 shows typical optical micrographs of 6061 Al alloy matrix reinforced by SiC particles (Fig. 6a), and NiTi fibers in 6061 Al alloy matrix with SiC particles (Fig. 6b). In the areas with fewer SiC particles, the grain size of the Al alloy matrix is estimated as 15–20 μm, however, in the areas with more SiC particles, the grain size is usually smaller than 5 μm.

Figure 7 shows the aging effect on 6061 Al—5 wt% SiC composite (Fig. 7a), 6061 Al—5 wt% SiC–1 wt% 51 μm–NiTi composite (Fig. 7b) and 6061 Al—5 wt% SiC–1 wt% 127-μm NiTi composite (Fig. 7c). When the aging time is <60 min, very limited strength enhancement of the composite can be obtained; however, when the aging time is increased to 180 min, the strength enhancement is quite obvious. About 30–50 MPa enhancement of strength has been achieved for all the samples. The increase in microhardness due to precipitates during the solid solution treatment has been well-documented in the literature, for example [35]. Since the precipitates hinder the movement of dislocations, the strength of materials increases [36]. However, no difference can be found from the fracture surfaces of these samples as shown in Fig. 8. On the other hand, as shown in Fig. 9, when there is no aging applied, or when aging time is <60 min, there is almost no observable difference in tensile properties among the composites 6061 Al—5 wt% SiC (Fig. 9a), 6061 Al—5 wt% SiC–1 wt% 51-μm NiTi (Fig. 9b) and 6061 Al—5 wt% SiC–1 wt% 127-μm NiTi (Fig. 9c). Strengthening by the addition of NiTi fiber is very limited as shown in Fig. 9a. This indicates that 1 wt% (0.4% volume fraction) addition of NiTi fiber does not obviously enhance the mechanical properties of the composite if the SME is not activated. With increasing aging time, the difference in mechanical properties between the composites 6061 Al—5 wt% SiC–1 wt% 51-μm NiTi and 6061 Al—5 wt% SiC–1 wt% 127-μm NiTi is also increased as shown in Fig. 9c and d. About 30–40 MPa difference was achieved at the ultimate strength of the sample aged for 180 min as shown in Fig. 9d. The strength of the composite reinforced by 51-μm NiTi fiber is higher than that of the composite reinforced by

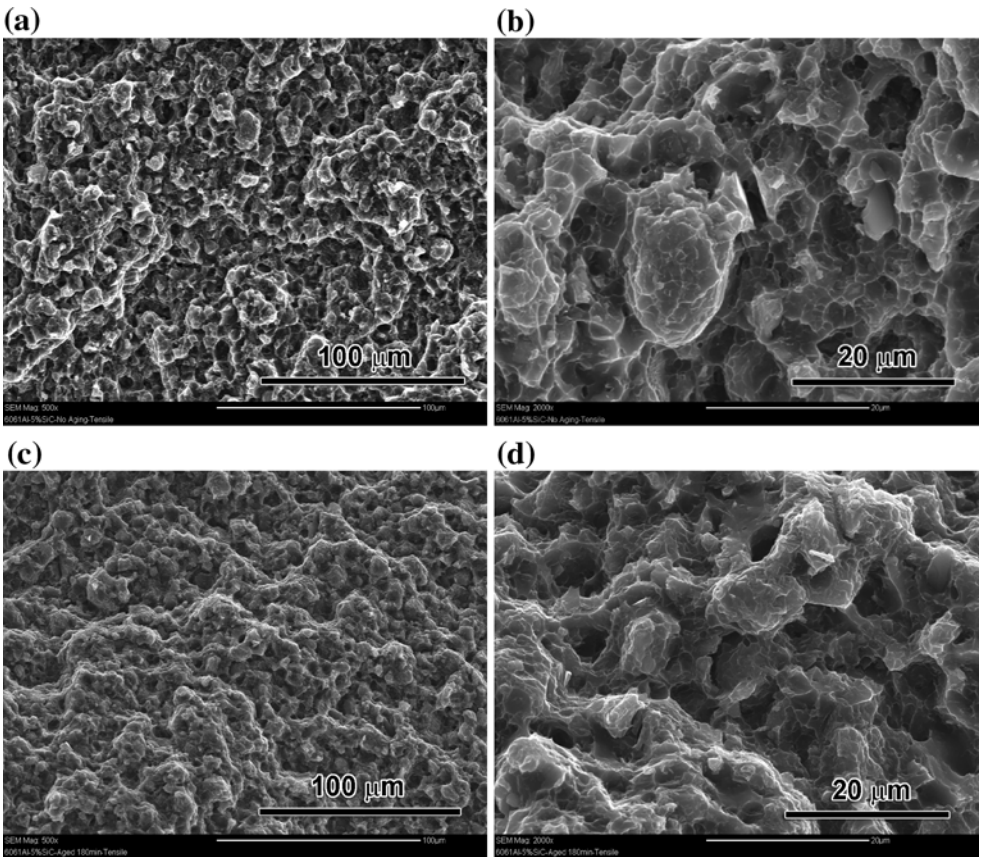

Fig. 8 Effect of aging on the fracture surface of 6061 Al + 5 wt% SiC composite. (**a**), (**b**) No aging and (**c**), (**d**) aged for 180 min at 177 °C

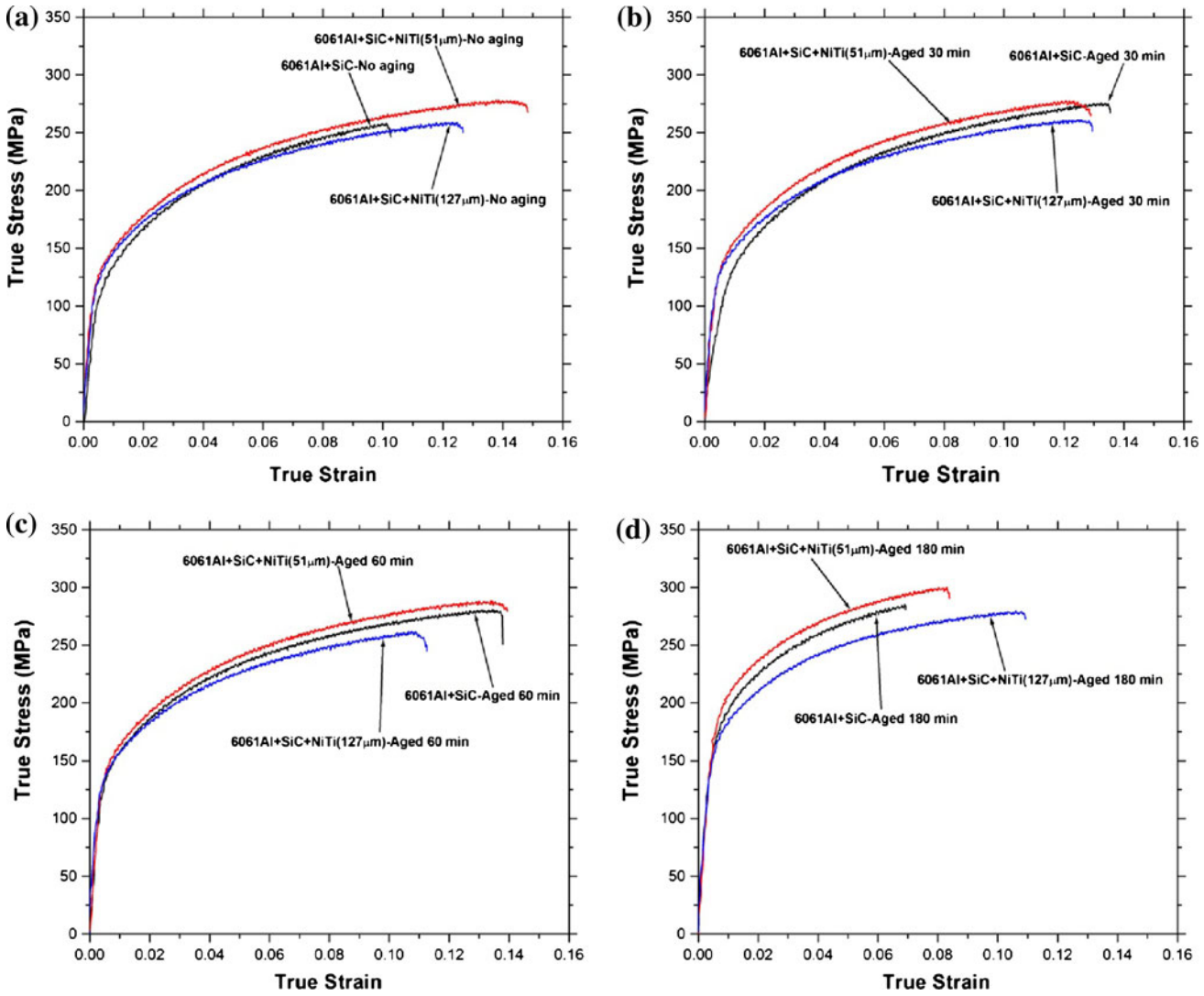

Fig. 9 Aging effect on the mechanical properties of 6061 Al—5 wt% SiC composite, 6061 Al—5 wt% SiC–1 wt% NiTi (51 μm) composite, and 6061 Al—5 wt% SiC–1 wt% NiTi (127 μm) composite. (**a**) No aging, (**b**) aged for 30 min, (**c**) aged for 60 min, and (**d**) aged for 180 min

127-μm NiTi reinforcement, but just a little higher than that of the composite without NiTi fibers. This implies that the bonding between 6061 Al matrix and 51-μm NiTi fiber is better and the interface has no negative effect on the mechanical properties of the composite, while the bonding between the 6061 Al matrix and 127-μm NiTi fiber might not be so good, since the mechanical properties becomes worse than the composite without NiTi fiber. This is consistent with the microhardness measurements on the matrix of these samples as shown in Fig. 10, where the microhardness kept almost unchanged until the aging time was increased to 180 min. Therefore, the strength enhancement shown in Figs. 7 and 9 is mainly due to the precipitates induced in the aluminum matrix during aging.

Fracture surface observations on the 6061 Al—5 wt% SiC–1 wt% NiTi composites indicate that the diameter

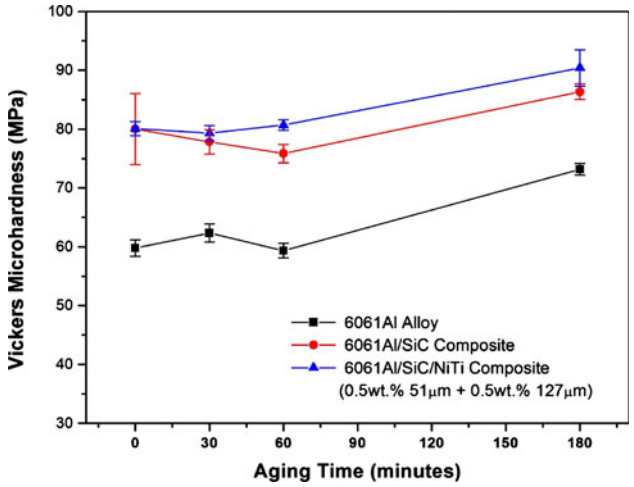

Fig. 10 Vickers microhardness versus aging time

of NiTi fiber is a very important factor affecting the mechanical properties of the composites. Coarser NiTi fibers are detrimental to the mechanical properties of the composites. As shown in Figs. 11 and 12, the bonding between 6061 Al matrix and 51-μm NiTi fiber is much better than that between Al matrix and 127-μm NiTi fiber. When 127-μm NiTi fiber is used, the bonding is actually not good at all, cracks can be frequently found. Part of the bonding between 6061 Al matrix and 127-μm NiTi fiber was destroyed when the composite deformed (Fig. 12a), and sometimes the bonding was fully destroyed (Fig. 12d). "Pull-out" mechanism can be observed frequently as shown in Fig. 12b and c, and as indicated before [1]. When 51-μm NiTi fiber is employed, most of the bonding was not destroyed by deformation as shown in Fig. 11a, b, and d. Sometimes, when the fiber is parallel to the fracture surface as shown in Fig. 11c, instead of de-bonding at the interface between NiTi fiber and 6061 Al matrix, the bonding is so good that the NiTi fiber itself was fractured due to deformation. "Pull-out" mechanism has not been observed. Since the diameter of the 51-μm NiTi fiber is much closer to the grain size of the matrix (15–20 μm), the mismatch in microstructure is lowered. Better bonding quality between Al and NiTi has been achieved. This explains the reason that the composite with 127-μm NiTi fiber has lower mechanical properties than that with 51-μm NiTi fiber.

The 6061 Al/SiC composites reinforced by both 51-μm and 127-μm NiTi fibers before and after aging for 180 min were further examined on TEM equipped with EDS. Chemical composition was explored by probing different locations near the interface between NiTi fibers and 6061 Al alloy matrix. No evidence has been observed that the chemical composition distribution near the interface is affected by either aging or size of NiTi fiber. This implies that interface reaction completed during the preparation of the composite and aging only affected the mechanical properties of the matrix in the composite.

Figure 13a shows some locations where EDS analyses were conducted near the NiTi/Al interface in 6061 Al/SiC composite reinforced by 51-μm NiTi fiber after aging for 180 min. As listed in Table 4, along the NiTi/Al interface, points "a" and "b" show similar chemical compositions containing Al, Ti, Ni and small amount of Cu and C with different percentage combinations, while point "d" shows a chemical composition similar to point "c," which is close to the composition of 6061 Al alloy matrix. The carbon

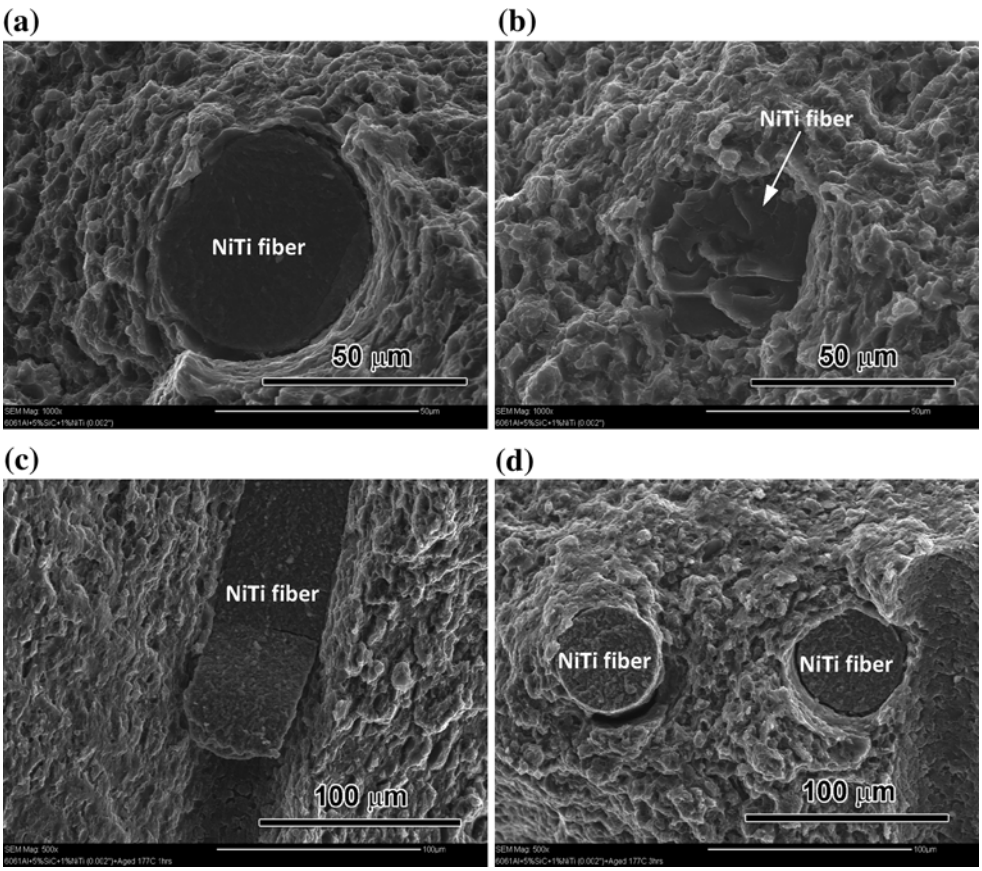

Fig. 11 Fractured surface of 6061 Al + 5 wt% SiC + 1 wt% NiTi with 51 μm in diameter, (**a**), (**b**) without aging, (**c**) aged for 60 min, and (**d**) aged for 180 min at 177 °C

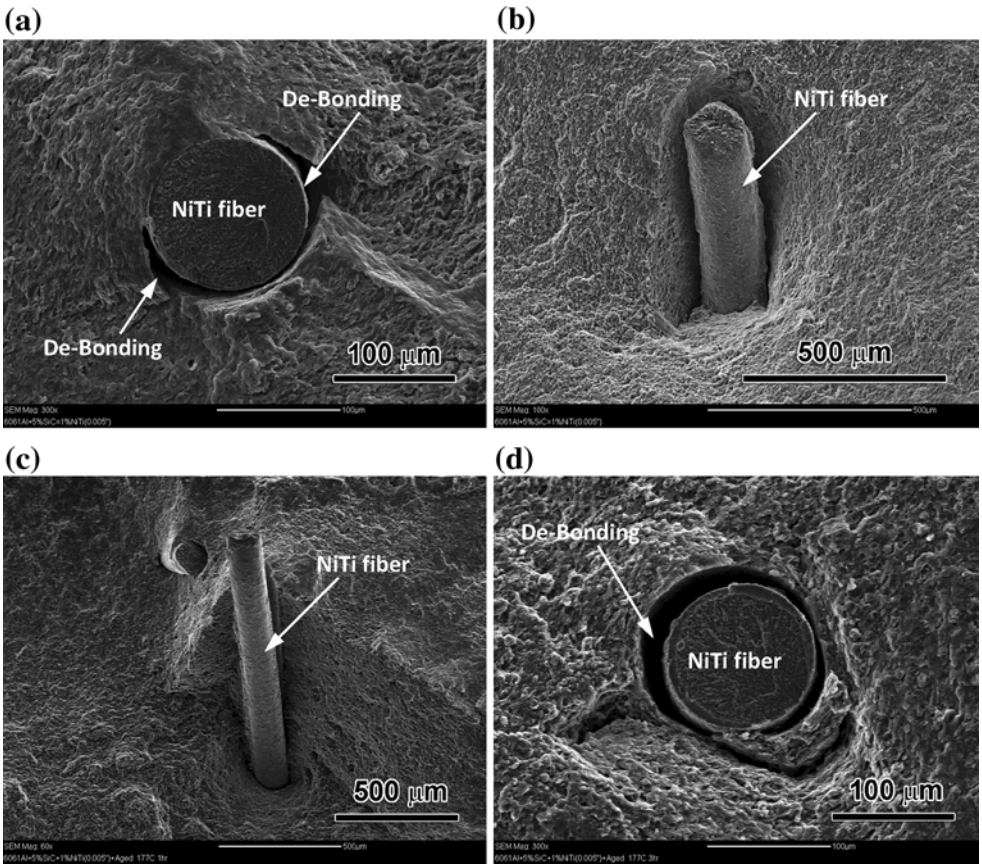

Fig. 12 Fractured surface of 6061 Al + 5 wt% SiC + 1 wt% NiTi with 127 μm in diameter, (**a**), (**b**) without aging, (**c**) aged for 60 min, and (**d**) aged for 180 min at 177 °C

content was not included when quantitative analyses were conducted due to the inaccuracy. This indicates that the chemical composition along the NiTi/Al interface is not homogenous. A continuous product of interface reaction similar to our previous report [21] was not formed. In our previous report on 6061 Al alloy composite reinforced by 300-μm NiTi fiber, three layers, such as Al_3Ti, Al_9FeNi, and Mg–O between these two layers along the NiTi/Al interface were found [21]. The composition at points "a" and "b" in Fig. 13a belongs to a compound containing Al, Ti, and Ni elements, most likely $Al_3Ti(Ni)$. The different interface reaction found in the present study from our previous report [21] is probably due to the addition of SiC particles into the composite. In Fig. 13a, point "f" shows a high percentage of Si which is probably related with a SiC particle near the NiTi fiber, at the interface of NiTi/SiC pointed by point "e," carbon and high percentage of Mg were found. The addition of SiC particles made the chemical composition distribution near the NiTi/Al interface more complicated.

In the composite 6061 Al/SiC reinforced by 127-μm NiTi fiber before aging as shown in Fig. 13b and listed in Table 5, points "a" and "c" correspond to 6061 Al alloy matrix and NiTi fiber, respectively, while point "f" contains Al, Ti, and Ni and small amount of Cu and Mg which is close to point "a" and "b" shown in Fig. 13a, and is supposed to form a compound among Al, Ti, and Ni. Point "b" contains high percentage of Al and Si, indicating that there is SiC particle nearby. Interestingly, point "d" contains Al, Fe, Ni, which is close to Al_9FeNi found in our previous report [21]. Point "e" contains higher percentage of Ti, this is probably because point "e" is closer to the NiTi/Al interface.

Based on the above EDS measurements along NiTi/Al interface, it is clear that the chemical composition along the interface is not homogeneous, and chemical composition fluctuation exists along the interface. Possibly compounds of Al–Ti–Ni as shown by point "a" and "b" in Fig. 13a, and Al–Fe–Ni as shown by points "d" and "e" in Fig. 13b have formed during preparation of the composites. However, the compounds possibly formed along the interface are not continuous, probably due to the large amount of addition of SiC particles which frequently interrupt the formation of the compounds. In any case, no macroscopic defect can be found along the NiTi/Al interface, which is good for the mechanical properties of the composites.

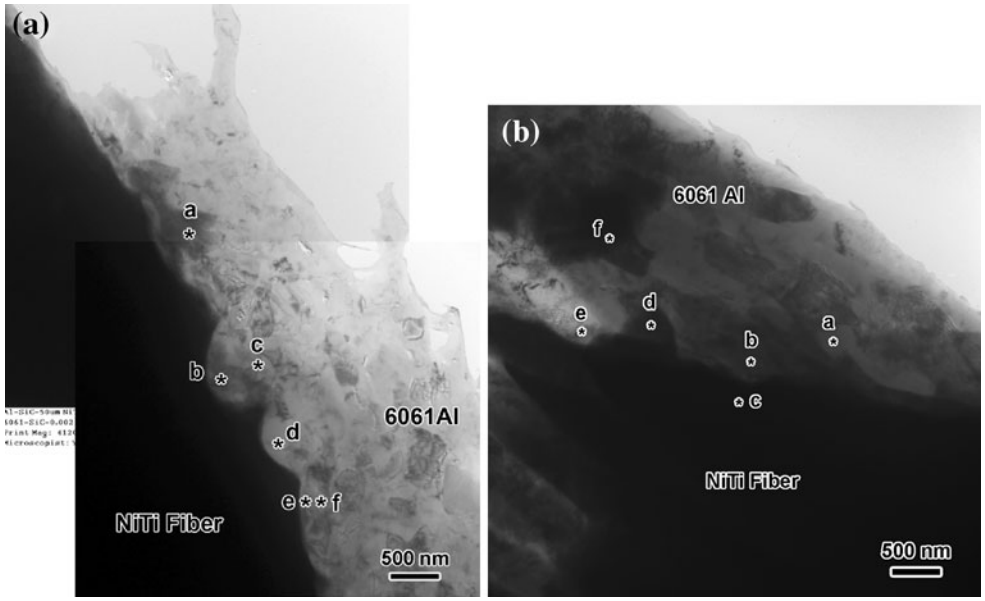

Fig. 13 Typical EDS analyses along the NiTi/Al interface in the 6061 Al/SiC composite reinforced by (**a**) 51-μm NiTi fiber after aging for 180 min and (**b**) 127-μm NiTi fiber before aging

Table 4 Typical chemical composition along NiTi/Al interface in the composite reinforced by 51-μm NiTi fiber after aging for 180 min (in at.%)

Locations in Fig. 13a	Mg	Al	Si	Ti	Ni	Cu
a	1.7	44.6	0.6	24.9	25.1	3.1
b	1.4	73.2	0.4	11.2	11.9	2.0
c	1.5	94.8	0.7	0.6	0.4	1.9
d	1.7	95.1	0.7	0.7	0.4	1.4
e	56.4	5.5	22.7	10.3	1.6	3.5
f	0.9	46.8	49.6	0.6	0.5	1.5

Table 5 Typical chemical composition along NiTi/Al interface in the composite reinforced by 127-μm NiTi fiber before aging (in at.%)

Locations in Fig. 13b	Mg	Al	Si	Ti	Fe	Ni	Cu
a	1.7	94.3	0.2	0.7	n/a	0.4	2.6
b	2.4	48.3	47.1	0.3	n/a	0.2	1.7
c	n/a	1.4	0.2	44.9	n/a	49.1	4.3
d	n/a	68.8	1.5	n/a	11.6	15.6	2.2
e	3.7	42.7	0.6	34.5	3.4	7.5	8.1
f	2.2	52.9	0.2	19.9	n/a	22.6	2.2

Conclusions

6061 Al composite containing 5 wt% SiC particle and 1 wt% (0.4% volume fraction) NiTi short fiber has been fabricated by pressure-assisted induction heating method. In order to study the size effect of NiTi fibers on the bonding between NiTi fibers and Al matrix, NiTi fibers with both 51 and 127 μm in diameters were used in the composite. SME was not activated in order to only reveal the size effect.

It is found that coarser NiTi fibers have low quality of bonding with the matrix, and are detrimental to the mechanical properties of the composites. Fracture surface observations indicate that the bonding between 6061 Al matrix and NiTi fiber is much better when 51-μm NiTi fiber is selected. There is better microstructural compatibility, i.e., fiber diameter is of the same order as the Al matrix grain size for this case. "Pull-out" mechanism is seldom observed. However, when 127-μm NiTi fiber is selected, de-bonding phenomenon and "pull-out" mechanism can be frequently observed. Aging only affects the mechanical properties of Al alloy matrix. EM and EDS analyses indicate that the chemical composition is different near the interface from that in the matrix, and Al–Ti–Ni and Al–Fe–Ni compounds are most likely formed along the interface, however, the interface reaction layer is not continuous, and is often disturbed by SiC particles. Mg is very easy to segregate to the interface. No macroscopic defect has been found along the NiTi/Al interface. Since no evidence shows that the bonding of NiTi/Al is affected by either aging or size of NiTi fiber, the improved bonding quality is attributed to the selection of NiTi fiber with smaller size.

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