

A novel strategy for fabricating phase transforming NiTi shape memory alloy via multiple processes of severe plastic deformation

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Abstract

NiTi shape memory alloy was successfully fabricated from Ni/Ti multilayers via an innovatively combinative route consisting of accumulative roll bonding (ARB), high-pressure torsion (HPT) and annealing. Due to the severe plastic deformation induced by ARB and HPT, the subsequently post annealing time was significantly reduced. Differential scanning calorimetry (DSC) analysis confirms the occurrence of austenitic and martensitic transformations during heating and cooling process, implying that the shape memory effect was obtained.

Key words

metals and alloys, microstructure, NiTi shape memory alloy, accumulative roll bonding, high-pressure torsion.

Introduction

Equiatomic NiTi shape memory alloy (SMA) is the most commonly used shape memory material due to its good features such as high strength, superelasticity and excellent shape memory effect [1-3]. Generally, NiTi alloys are fabricated by ingot wrought process and powder metallurgy. In the former process, multiple annealing is needed due to the insufficient ductility of NiTi alloys. In the latter one, it is difficult to avoid interstitial contamination during powder consolidation processing [4].

An alternative manufacturing method for NiTi sheets was via multilayered thin Ni/Ti laminates. A stack of multiple layers of alternating Ti and Ni are subjected to cold or warm rolling, and then the as-rolled multilayered laminates are annealed to achieve alloying and further consolidation [5-7]. In our previous work [8], accumulative roll bonding (ARB) and hot isostatic pressing (HIP) process was utilized to fabricate the NiTi alloys.

Severe plastic deformation (SPD) has been applied to NiTi SMAs to enhance the properties through grain refinement [9-13]. There are also some other intensive plastic deformation methods to alter the properties of NiTi shape memory alloys, e.g. hot rotary swagging [14,15]. Among SPD processes, high-pressure torsion (HPT) is the most efficient way to achieve ultrafine-grained materials [11-13]. In the present work, HPT method was applied to the Ni/Ti laminates prepared by ARB. To our best knowledge, such kind of work has never been reported. The purpose of this work is to fabricate SMA based on the Ni/Ti laminates in micron-scale and to find a way to reduce annealing time in the fabrication of NiTi shape memory alloys for energy saving and higher efficiency.

1. Materials and methods

Commercially pure Ti (99.95 wt.%) and Ni (99.97 wt.%) foils with a rectangular shape (35×9.55 mm) were used as the starting materials. The Ni foils (30 μm in thickness) and the Ti foils (50 μm in thickness) were stacked alternately. The initially stacked Ni/Ti sample consisted of 19 layers of Ti foils and 18 layers of Ni foils, based on the designed composition of 50.8 at.% Ti and 49.2 at.% Ni. The surfaces of the Ni

and Ti were ultrasonically cleaned by acetone to remove the grease. The stacked Ni/Ti foils were sealed in a steel container and then subjected to ARB processing. A reduction of 50% in thickness was adopted and two passes were processed.

During the ARB process, the relationship between the equivalent plastic strain and rolling reduction was described as the following [16],

$$\varepsilon = \frac{2}{\sqrt{3}} \ln (H_0/h) \quad (1)$$

where H_0 and h are the initial layer thickness and nominal average layer thickness after each ARB cycle, respectively. The strain in Ti and Ni layers can be calculated as 2.63 after two ARB cycles.

The thickness of the Ni/Ti multilayered sheet is 0.45 mm after ARB processing. Ni/Ti discs with a diameter of 10 mm were cut from the Ni/Ti sheet for further HPT processing. HPT processing was conducted with a pressure of 6 GPa and 15 turns. The equivalent strain induced by HPT processing can be calculated according to equation (2) and (3) [17].

$$\gamma = \frac{2N\pi r}{h} \quad (2)$$

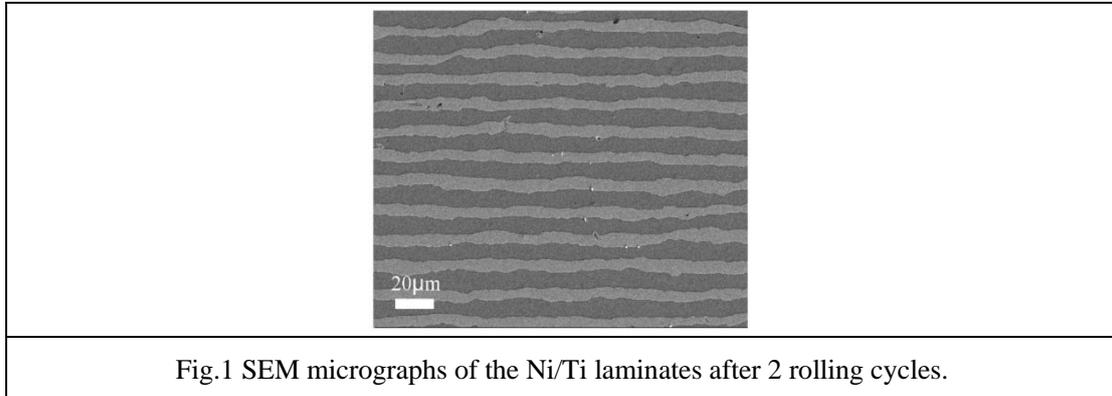
$$\varepsilon = \frac{2}{\sqrt{3}} \ln \left(\frac{\gamma}{2} + \sqrt{1 + \frac{\gamma^2}{4}} \right) \quad (3)$$

where r is the distance from the disk sample center, N is the number of turns, γ is the shear strain and h is the final thickness of the sample. The value for 15 turns ($N=15$) at the location of maximum radius is about 7.23.

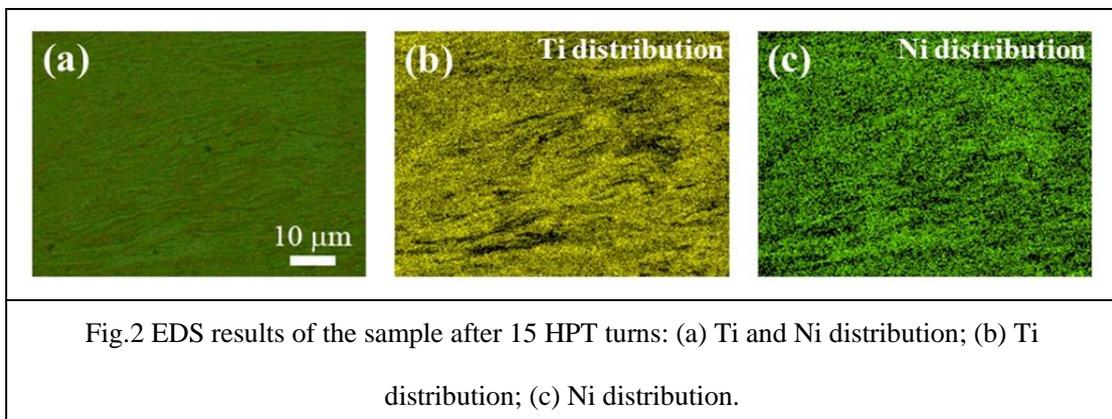
The HPT-processed samples were annealed at 900°C for 0.5 and 1 hour. The microstructure of the HPT-processed sample was characterized by scanning electron microscopy (SEM) equipped with an energy-dispersive X-ray spectrometer (EDS), and the microstructure of samples subjected to post-HPT annealing was observed by transmission electron microscopy (TEM). The phase transformation behaviors of the samples were examined by differential scanning calorimetry (DSC).

3.Results and discussion

Figure 1 show the SEM micrograph of the Ni/Ti laminates after 2 passes of ARB. It can be observed that the thickness of both the Ni and Ti layers are refined to about 10 μm .



EDS results of the ARB sample after 15 turns HPT processing are shown in Figure 2. It is revealed that the boundaries of Ni and Ti layers become unclear. Both Ni and Ti element distribution is relatively homogeneous, indicating Ni and Ti are mixed sufficiently during the HPT process.



The TEM microstructures of the samples subjected to post-HPT annealing at 900°C for 1h is shown in Figure 3a. Nanostructured martensite is obtained after post-HPT annealing, indicating that the NiTi SMA has been successfully fabricated.

Fig.3b shows the shape memory effect, namely, the transformation behavior by DSC measurements on the samples after post-HPT annealing. The specimen exhibits one exothermic peak in the cooling process and one endothermic peak in the subsequent

heating process, indicating the appearance of B2-B 19' martensitic transformation. The transformation temperatures and transition hysteresis are shown in Table 1, showing a M_s temperature of about 62.5°C, and a transition hysteresis width of about 37°C, which are comparable with the results of NiTi alloys made from conventional ingot-metallurgy method [18,19].

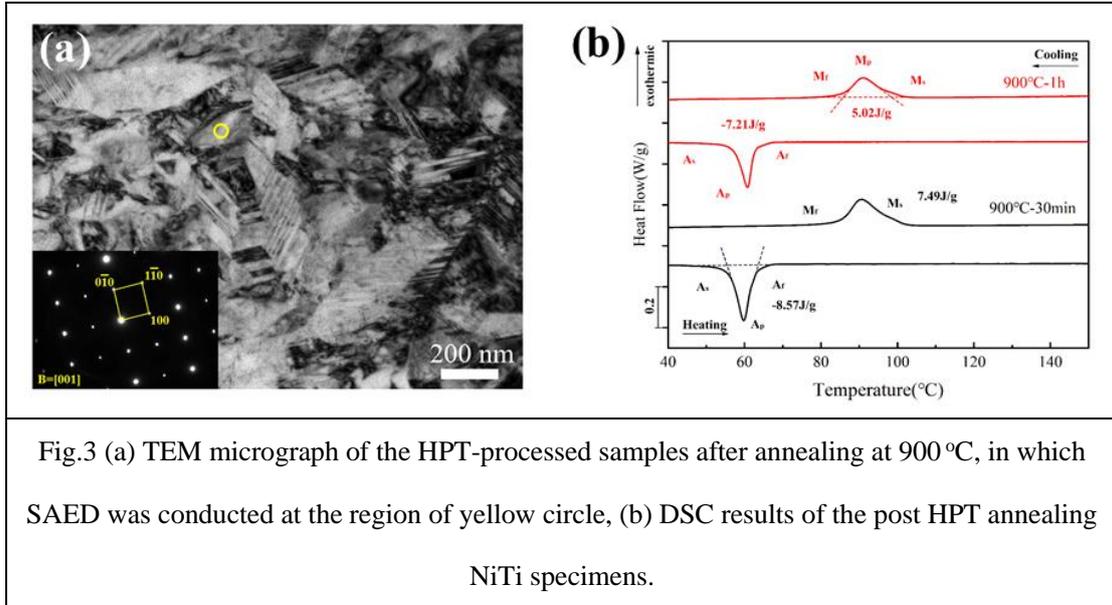


Table 1 Transformation behaviors of NiTi specimens after post HPT annealing

Time (hour)	Temperature(°C)						Transition
	A_f	A_s	M_f	M_s	A_p	M_p	hysteresis (°C)
0.5	99.8	85.1	56.1	62.5	90.7	59.7	37.3
1	99.1	85.3	57.3	62.7	90.9	60.8	36.4

There are several published reports on the process of NiTi SMAs through the fabrication of Ti/Ni laminate and subsequent annealing. One report showed that a Ti/Ni laminate with the average thickness of 0.68μm in Ti layer and 0.15μm in Ni layer was attained after hot and cold rolling of the Ti/Ni stack with the reductions of

92% and 99% respectively and NiTi SMA was obtained after subsequent annealing at 800°C for 10 h [6]. It was also reported that the cold rolling with a total reduction of 97% was conducted in the hot-press sintered multilayered Ti-Ni stackers and the Ti/Ni laminate with the average thickness of 1.9 μm in Ni layer and 3.6 μm in Ti layer was produced. Then the SMA can be fabricated via further annealing for 18 h at 800°C [4]. In our previous study [8], the multilayered Ni/Ti laminates were refined by accumulative roll bonding (ARB) with 6 passes, with an average individual layer thickness of < 45 nm being attained. The Ni/Ti laminates were subjected to hot isostatic pressing at 900 °C for 1 h to obtain a fully dense and homogeneous NiTi shape memory alloy. In the present study, only two passes of ARB were adopted to produce the average thickness of the Ni and Ti layers with a thickness of ~10 μm. During subsequent HPT processing, the laminate structure was destroyed, and a large number of defects could be introduced, which is beneficial to the promotion of the diffusion in the subsequent annealing process. Therefore, a very short time (900°C/0.5 h) for post HPT annealing was applied to obtain the SMA.

In our present work only two passes of ARB were applied and the effect of the combination of ARB with HPT processing for reducing the annealing time is obvious, which will reduce manufacturing time and relevant energy consumption. Our present work shows that combining two SPD methods (ARB and HPT) and subsequent annealing could provide a new route for the fabrication of NiTi SMA.

4. Conclusions

In summary, phase transforming NiTi shape memory alloy was attained by combining ARB, HPT and post-HPT annealing. The multilayered Ni/Ti laminates were attained by two pass accumulative roll bonding. After 15 turns HPT processing, the laminate structure was destroyed, and the Ni and Ti elements were homogeneously distributed within the matrix. NiTi SMA can be successfully fabricated after a short time annealing at 900°C. This work provides an alternative way to produce SMA by severe plastic deformation and post-deformation annealing.

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