The Influence of Cold-Rolling on the Internal Friction Behavior of TiNi Shape Memory Alloy

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This work is focused on the influence of cold-rolling on the transformation and internal friction behavior of TiNi shape memory alloy. The DSC results show the endothermic peaks disappeared with the increasing of cold-rolling reduction and there was an exothermic peak around 330°C on the curve during the first heating process. The exothermic peak is associated with recrystallization of amorphous phase induced by cold-rolled work. During the second heating process, the endothermic peak showed up but the exothermic peak didn’t. These phenomena are confirmed by the internal friction results. During the first heating process, the transformation internal friction decreased with the increasing of the cold-rolling reduction and the internal friction augmentation appeared around 330°C which is related to recrystallization.


(Keywords: TiNi alloy, internal friction, differential scanning calorimetry (DSC), cold-rolled work, amorphous)

1. Introduction

With the extensive research of shape memory alloys, people find that shape memory alloys possess the high damping capacity, besides shape memory effect, excellent pseudoelasticity and corrosion resistance.¹–⁴ As a measurement, internal friction is known to be sensitive to the structure and has been used to investigate the structural relaxation and crystallization of amorphous alloys. So far, the influences of frequency, amplitude, compositions and heat treatment on the internal friction of shape memory alloys have been investigated systemically. But, the effect of cold-rolled on the internal friction of shape memory alloys has been seldom studied. There are some researchers⁵–⁹ only working on the internal friction investigations of as-cold reduced shape memory alloys after different anneal conditions. To our knowledge, the researches on internal friction of as-cold reduced shape memory alloys without any anneal treatment are very few for now. Hereinto, Lin et al.⁴ investigated the internal friction behavior of as-cold reduced TiNi alloys and the measuring temperatures were −150~250°C. Cai et al.² only studied the effect of cold-rolled work on internal friction of TiNi alloys at room temperature. So, they didn’t find out the effects of amorphous phase, which was induced by cold-rolled work, on the internal friction behavior of TiNi alloys.

In this work, the as-cold reduced TiNi specimens weren’t subjected to any heat treatment. The DSC and internal friction measurements were carried out during the first and second heating processes. The recrystallization of amorphous phase induced by cold-rolled work was found and the effects of amorphous phase on the transformation and internal friction behaviors were discussed.

2. Experimental Procedure

Ni₅₅.₅Ti₴₈.₅ alloy sheets with 2 mm in thickness were obtained from the General Research Institute for Nonferrous Metals, China. Specimens (4 × 100 mm) were spark cut from the sheets for cold rolling. The as-cut specimens were annealed in vacuum at 800°C for 30 min and then aged at 500°C for 30 min with cooling in water. To eliminate the shape change during the heat treatment, the specimens were sandwiched between the two flat stainless steel plates. At room temperature, the as-anealed specimens were cold-rolled to the extent of 11, 18 and 28% reduction in thickness and then subjected to the internal friction test. The internal friction test was conducted on a LMR-1 Low Frequency Mechanical Relaxation Spectrum Analyzers. This apparatus consists of an inverted torsion pendulum, a temperature controller, a photoelectron transformer and an industrial computer 610 which controls all measurements and the data can be processed in real time. The internal friction measurements of the specimens were carried out as a function of temperature with a heating rate of 1°C/min and a frequency of 1 Hz. The testing temperature range is from −95°C to 400°C and the N₂ is full of the specimen chamber as a protecting gas. The strain amplitude is 50 × 10⁻⁶. All measurements of the internal friction were conducted on the specimens by forced oscillation. Small samples of length 4 mm for DSC test were cut from the specimens using a low speed diamond saw. DSC experiments were conducted from −45°C to 400°C with the heating and cooling rate of 10°C/min by using a Netzsch DSC 2004 Phoenix.

3. Results and Discussion

Figure 1 shows the DSC results of TiNi alloy with different cold-rolling reduction during the first heating process. One can see that an obvious endothermic peak appeared on the DSC curve of unrolled specimen around 30°C and this peak consisted of two independent peaks corresponding to M → R and R → P transformations. However the endothermic peaks disappeared with the cold-rolling reduction increasing, which is associated with martensite stability of as-rolled specimens. On the other
words, the reverse martensite transformation is getting too slow to be observed by the DSC equipment under the constraint of the deformation dislocations. Meanwhile, there was an exothermic peak around 330°C on the curves of the as-rolled specimens and the area of the exothermic peak increased with the increasing of cold-rolling reduction.

Figure 2 shows the DSC curves of the specimens with different cold-rolling reduction during the second heating process. It can be seen that the endothermic peaks also appeared around 50°C on the DSC curves of as-rolled specimens after the complete transformation. The endothermic peak consisted of two independent peaks as well and the area of the low-temperature peak decreased with the increasing of cold-rolling reduction.

Figure 3 shows the internal friction curves of TiNi specimens with different cold-rolling reduction during the first heating process. It can be seen that the unrolled specimen had a conspicuous internal friction peak and the peak was split into two peaks at the top. This phenomenon is consistent with the DSC result. Furthermore, the transformation internal friction peaks of the cold-rolled specimens appeared within the range from 100 to 300°C. The transformation internal friction peaks shifted towards high temperature and the maximum value of peaks decreased as the cold-rolling reduction increased. The shift of the internal friction peaks is associated with the stabilization of the thermo-elastic martensite, which is investigated extensively by lots of researchers.4,10–14) For the decrease of the value of the internal friction peak, it could be explained from two ways. First, it could be explained by Dejonghe-Delorme model15,16) and the specific explanation could refer to our previous work.17) Second, it is associated with the reorientation of martensite variants during the cold-rolled work process. The Schematic illustration of the reorientation of martensite variants shown in Fig. 4 was given to understand the mechanism well. It can be seen that the unrolled martensite consists of different orientation variants as shown in Fig. 4(a). After slight deformation, martensite variants are reoriented along the cold-rolled direction incompletely as shown in Fig. 4(b). As the cold-rolling reduction increases further, reoriented variants merge each other and the amount of variants declines. Finally, variants are reoriented completely and form the parallel streaky structures with different orientation, shown in Fig. 4(c). When the cold-rolling reduction keeps increasing further, the parallel streak structures are staggered in Fig. 4(d). On the basis of these considerations, the amount of variants interfaces would decrease dramatically with the increasing of the cold-rolled
reduction. Furthermore, the mobility of variants interfaces would be weakened when the reoriented variants get staggered. Thus, these result in the decrease of internal friction of as-rolled specimens. Meanwhile, one can see that there was an obvious augmentation around 330°C on the curves of the as-rolled specimens.

Figure 5 indicates that the internal friction curves of the as-rolled specimens during the second heating process. One can see that the temperatures of the transformation internal friction peaks shifted down to 40°C, compared with the first heating process. The internal friction augmentation of the as-rolled specimens still exists after one thermal cycle. As shown in Fig. 1, there were the exothermic peaks around 330°C on the DSC curves of the as-rolled specimens and the area of the peak increased as the increasing of the cold rolling reduction. It can be seen clearly from Fig. 6.

Recently, the effects of severe plastic deformation on the microstructure and the properties of shape memory alloy have been researched widely.\textsuperscript{18–23} It was found that an amorphous phase was produced as a result of the accumulation of high density dislocations and depended on the amount of cold work. According to H. Nakayama \textit{et al.}\textsuperscript{19} and K. Tsuchiya \textit{et al.}\textsuperscript{22} the amorphous phase was only found in the 40% or bigger cold rolled samples. However, the differences between their work and our work are transformation temperatures and heat treatment state of samples. At the room temperature, the samples in Ref. 19, 22) were homogenized at 900°C for 60 min, while the samples used in this work were annealed at 800°C for 30 min and aged at 500°C for 30 min. The grain size of the samples used in this work might be smaller and more precipitation particles exist than those in Ref. 19, 22). During the cold rolling process, the deformation microstructure and defects might be more prominent in our research, which results in a more severe accumulation of high density dislocations in the sample. Based on these factors, the amorphous phase could be produced more easily in our samples during the cold rolling process.

Furthermore, G. B. Cho \textit{et al.}\textsuperscript{23} found that the volume fraction of amorphous phase increases from 17% to 43% with increasing working ratio from 30% to 70%. It suggests that amorphous phase might appear in a less than 30% cold rolling ratio. Ewert \textit{et al.}\textsuperscript{24} found an exothermic peak around 350°C on the DSC curve of the as-rolled TiNi alloy, and the area of the peak increased with the increasing of cold-rolling reduction, when they focused on the transformation behavior of TiNi alloy with the different cold rolling reduction. The same phenomenon was found by A. K. Srivastava \textit{et al.}\textsuperscript{25} and K. Inaekyan \textit{et al.}\textsuperscript{26} They found this exothermic peak is associated with the recrystallization of the amorphous phase.
induced by the cold-rolling. So, it could be a reasonable explanation for the exothermic peak in our present work.

Compared Fig. 1 with Fig. 3, the temperature of the exothermic peak on the DSC curve is corresponding to that of the internal friction augmentation on the internal friction curve. So, it could conclude that they are related to the recrystallization of the amorphous phase. During the recrystallizing process, the atoms diffuse more actively and those as-crushed grains re-nucleate and grow into fine and homogeneous equiaxed crystals. These processes consume lots of energies so that the internal friction increases.

From Fig. 2 to Fig. 5, one can see that the exothermic peak on the DSC curve disappeared but the internal friction augmentation didn’t. This could be associated with the appearance on the DSC curve disappeared but the internal friction behaviors of TiNi alloy has been investigated. During the first heating process, the martensitic reverse transformation peaks shifted towards high temperatures of the transformation internal friction peaks shifted down to 40°C, compared with the first heating process. The internal friction augmentation of the as-rolled specimens still exists after one thermal cycle.

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