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Isothermal effect on internal friction of $Ti_{50}Ni_{50}$ alloy measured by step cooling method in dynamic mechanical analyzer

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Abstract

The internal friction of TiNi-based SMAs measured by step cooling method in DMA may be misled when the used isothermal time interval t is insufficient. For $Ti_{50}Ni_{50}$ SMA, the tan δ values of B2 → R and R → B19′ internal friction peaks measured by step cooling method are lessening with prolonging the t value. This is because the tan δ value of IF_{Tr} still subsists and decreases continuously during the frequency sweep process. The t effect is more significant when the step cooling method is measured at lower frequency. According to the experimental results of $Ti_{50}Ni_{50}$ SMA, it is acceptable to choose $t = 20$ min to eliminate most of the influence of IF_{Tr}.

Keywords: Shape memory alloys (SMA); Internal friction; Dynamic mechanical analysis; Step cooling method; Isothermal time interval

1. Introduction

TiNi-based alloys are known as the most important shape memory alloys (SMAs) because of their excellent properties in shape memory effect, superelasticity and high damping characteristics [1–12]. When heating/cooling TiNi-based SMAs, there are internal friction (tan δ) peaks with storage modulus (E_0) minimums corresponding to martensitic (premartensitic) transformations [4]. It has been reported that the damping characteristics of tan δ peak(s) during martensitic transformation are closely related to experimental parameters such as heating/cooling rate $\dot{T}$, frequency ν and amplitude σ_0. Most of the reported studies investigated the damping characteristics of TiNi-based SMAs at an abiding cooling/heating rate [2–12]. Only a few works discussed the inherent internal friction of TiNi-based SMAs under isothermal conditions ($\dot{T} = 0$) [13–15]. Dynamic mechanical analyzer (DMA) can measure the tan δ and E_0 values of the specimen by accurately controlling these experimental parameters and is suitable for precisely studying the damping characteristics of internal friction peak(s) in TiNi-based SMAs [12–15]. Recently, Fan et al. reported the internal friction of $Ti_{50}Ni_{48}Fe_2$ [16] and $Ti_{50}Ni_{50-x}Cu_x$ [17] SMAs measured by DMA using a process termed "step cooling/heating method", i.e., the DMA specimen was first kept isothermally at different set temperature for 5 min to reach thermal equilibrium and followed by a series of frequency sweep. In step cooling/heating method, the cooling and heating processes are the same from the viewpoint of isothermal condition, thus the "heating" was removed afterward from the terminology of the "step cooling/heating method" in this study. The step cooling method has the benefits of easily determine the frequency-related properties such as the frequency effect on the damping capacity because the specimen can be measured with different frequencies discretely and sequentially when isothermal at each set temperature. However, for TiNi-based SMAs, the tan δ value is strongly associated with the isothermal time interval [13–15] and this effect should be considered carefully when the specimen was measured by the step cooling method. The main purpose of this study is to investigate the influence of isothermal time interval on the internal friction of equiatomic TiNi SMA measured by DMA with the step cooling method. Besides, we also discuss how to perform the step cooling method appropriately for TiNi-based SMAs in DMA tests to avoid the misleading experimental results.

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2. Experimental procedures

Equiatomic TiNi SMA was prepared by conventional vacuum arc remelter. The as-melted ingot was hot-rolled at 850 °C into a 2-mm-thick plate and then solution-treated at 850 °C for 2 h followed by quenching in water. Thereafter, the plate was cold-rolled at room temperature along the hot-rolling direction to eliminate the influence of rolling texture [18] and reached a final 30% thickness reduction. Subsequently, the cold-rolled plate was cut along the hot-rolling direction into test specimens with the dimension of 37.8 mm × 6.7 mm × 1.3 mm for DMA test and about 30 mg for DSC test. The cut specimens were sealed in evacuated quartz tubes and annealed at 650 °C for 2 min. Transformation temperatures of cold-rolled and annealed specimen were determined by DSC test using a TA Q10 DSC equipment with a constant cooling rate of 10 °C/min. tan δ and E_0 values of cold-rolled and annealed specimen were measured by a TA 2980 DMA instrument equipped with a single cantilever with a constant cooling rate of 1 °C/min and an amplitude of 5 μm (strain amplitude = 5.7×10^{-5}). Thereafter, the same specimen was conducted in the same DMA equipment by the step cooling method as follows. The DMA specimen was kept isothermal at temperatures every 2 °C from 60 °C to −20 °C for a certain time interval t and subsequently followed by frequency sweep from 0.5 Hz to 10 Hz discretely at each isothermal temperature. Fig. 1 plots the schematic representation of the aforementioned step cooling method in which the time interval t used in this study is 1 min, 5 min, 10 min and 30 min.

3. Experimental results

3.1. DSC and DMA results measured at a constant $\dot{T}$

Fig. 2(a and b) show DSC and DMA curves, respectively, of 30% thickness-reduced $Ti_{50}Ni_{50}$ alloy annealed at 650 °C for 2 min. In Fig. 2(a), there are B2 → R and R → B19′ two transformation peaks in the forward transformation and there is B19′ → B2 one transformation peak in the reverse. Fig. 2(b) illustrates the tan δ and E_0 curves of the specimen with the same thickness-reducing and annealing as Fig. 2(a). Only the cooling curves with $\dot{T}$ = 1 °C/min, ν = 1 Hz and σ_0 = 5 μm are shown in Fig. 2(b) for conspicuous description. There are also two internal friction peaks appearing in the tan δ curve which correspond to B2 → R and R → B19′ transformation peaks observed in DSC curve shown in Fig. 2(a). The peak temperatures measured by DSC and DMA tests show a small shift due to different cooling rates and specimen sizes used in these two tests. Except for these two tan δ peaks, an extra broad peak is also observed in Fig. 2(b) at about −50 °C. This extra peak which is not observed in the DSC curve is known as the relaxation peak [4]. Besides, as shown Fig. 2(b), the E_0 cooling curve declines gently in B2 parent phase, then drops drastically and exhibits a deeper minimum during B2 → R premartensitic transformation and a shallower minimum during R → B19′ martensitic transformation.

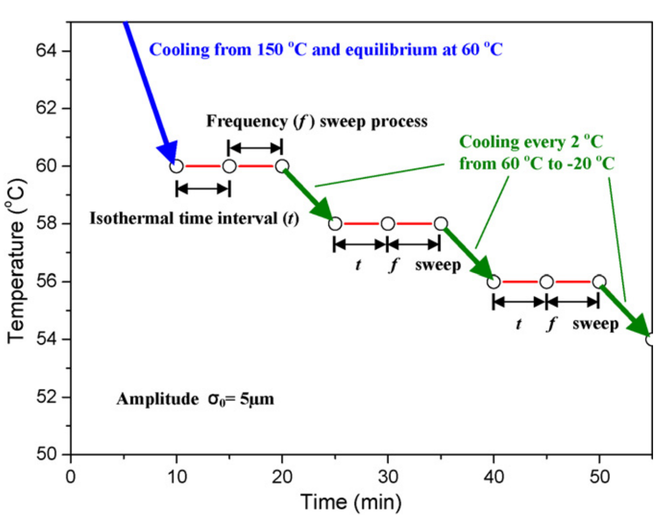

Fig. 1. Schematic representation of the step cooling method.

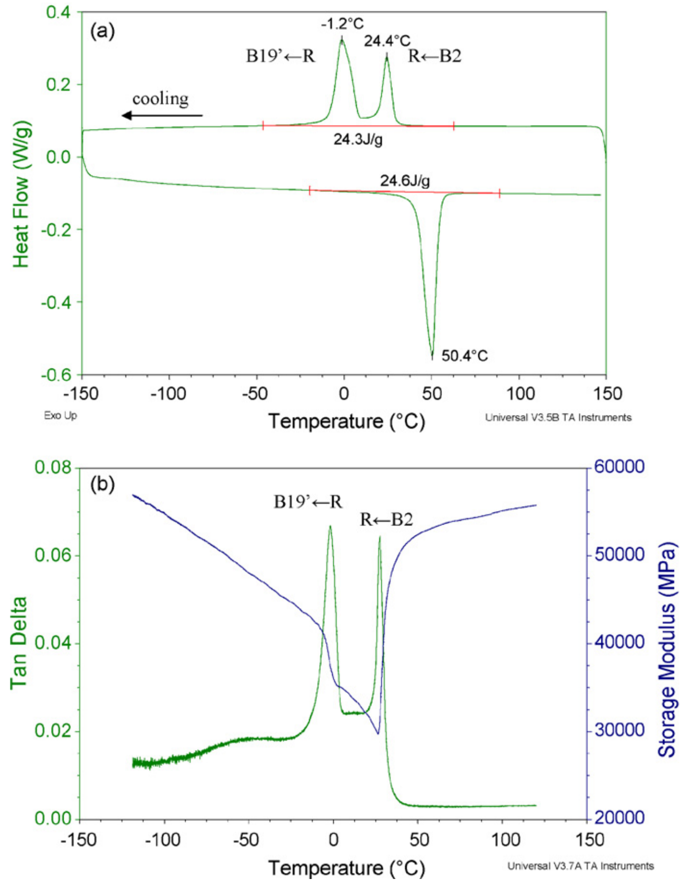

Fig. 2. (a) DSC and (b) DMA cooling curves for 30% thickness-reduced and 650 °C × 2 min annealed $Ti_{50}Ni_{50}$ SMA.

3.2. DMA results measured by step cooling method

Fig. 3(a and b) show the tan δ and E_0 cooling curves, respectively, for the Fig. 2(b) specimen measured by step-cooling method of Fig. 1. The specimen was kept isothermal at temperatures every 2 °C from 60 °C to −20 °C for t = 1 min and then the frequency was swept from 0.5 Hz to 10 Hz discretely. As shown in Fig. 3(a), both tan δ values of B2 → R and R → B19′ internal friction peaks decrease when the frequency is increased from 0.5 Hz to 10 Hz. On the other hand, as can be seen in Fig. 3(b), the E_0 curves measured at different frequencies between 0.5 Hz and 10 Hz are almost the same. This feature implies that the E_0 value does not show strong frequency dependence in the frequency range between 0.5 Hz and 10 Hz.

Fig. 4(a and b) show the tan δ and E_0 cooling curves, respectively, for the Fig. 2(b) specimen measured by step cooling method with the same experimental parameters as Fig. 3 except that t is 10 min, instead of 1 min, at each set temperature. As can be seen in Fig. 4(a), both tan δ values of B2 → R and R → B19′ internal friction peaks also decrease with increasing the frequency. Besides, as shown in Fig. 4(b), the E_0 value measured at different frequencies dose not show frequency dependence either. This phenomenon is similar to the results shown in Fig. 3. However, comparing Fig. 4(a) to Fig. 3(a), under the same frequency, tan δ values of B2 → R and R → B19′ internal friction peaks measured at t = 1 min are higher than those measured at t = 10 min. This phenomenon indicates that the measured tan δ values of the transformation peaks are closely related to the isothermal time interval t used in the step cooling method.

3.3. Isothermal effect in step cooling method

In order to investigate the effect of isothermal time interval t on the internal friction measurement of $Ti_{50}Ni_{50}$ SMAs, tan δ tests using step cooling method with different t values were conducted and the results are exhibited in Fig. 5.