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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 $\tan\delta$ values of $B2 \rightarrow R$ and $R \rightarrow B19'$ internal friction peaks measured by step cooling method are lessening with prolonging the t value. This is because the $\tan\delta$ 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 ($\tan\delta$) peaks with storage modulus (E_0) minimums corresponding to martensitic (premartensitic) transformations [4]. It has been reported that the damping characteristics of $\tan\delta$ 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 $\tan\delta$ 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 $\tan\delta$ 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.

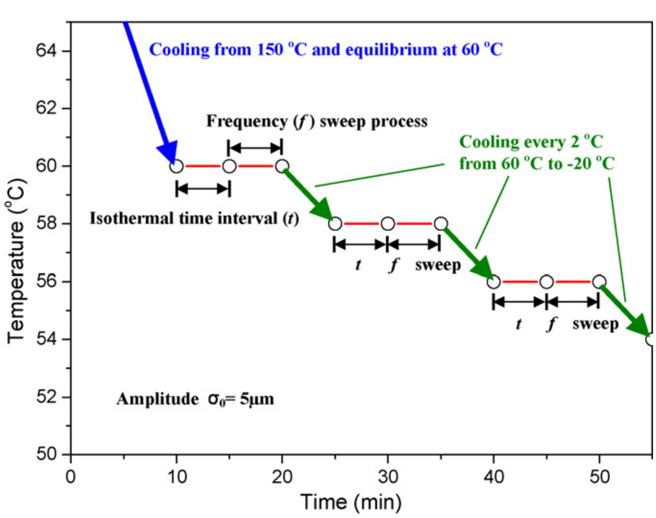

Fig. 1. Schematic representation of the step cooling method.

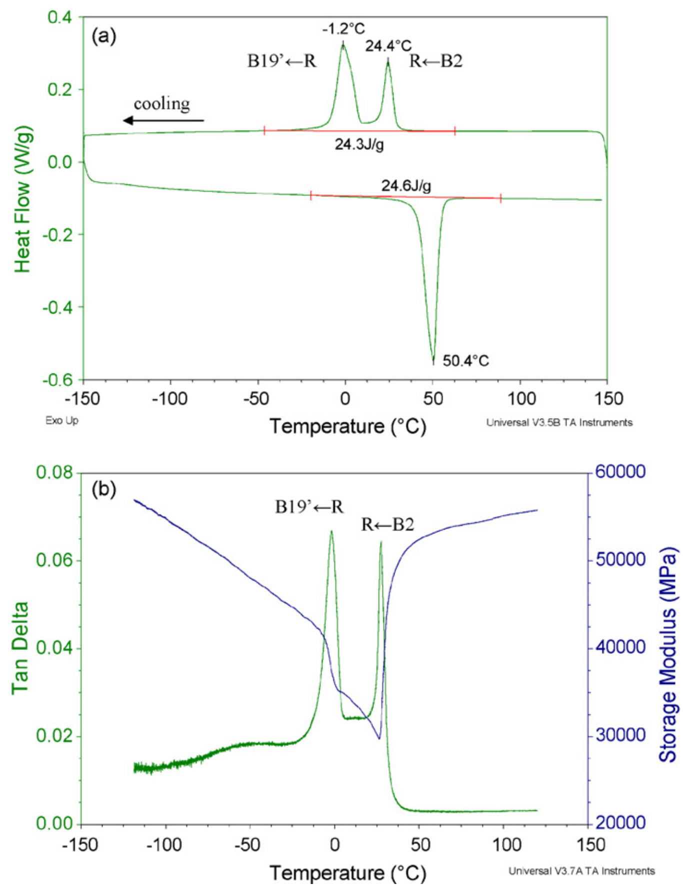

Fig. 2. (a) DSC and (b) DMA cooling curves for 30% thickness-reduced and 650 °C × 2 min annealed $Ti_{50}Ni_{50}$ SMA.

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.

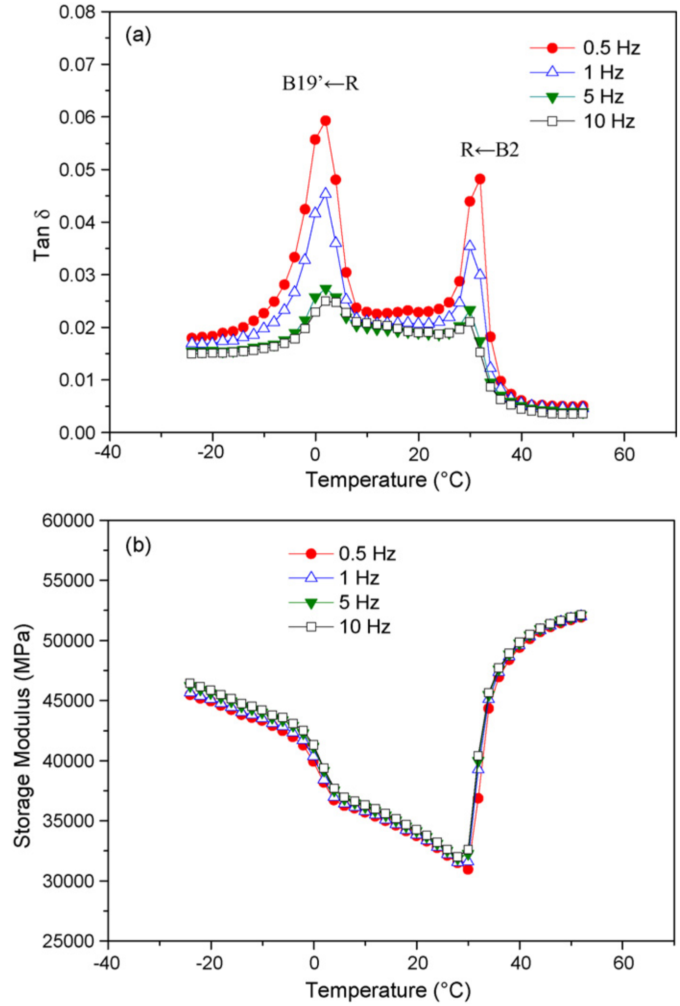

Fig. 3. (a) tan δ and (b) E_0 cooling curves for Fig. 2(b) specimen measured by step cooling method with isothermal time interval $t = 1$ min.

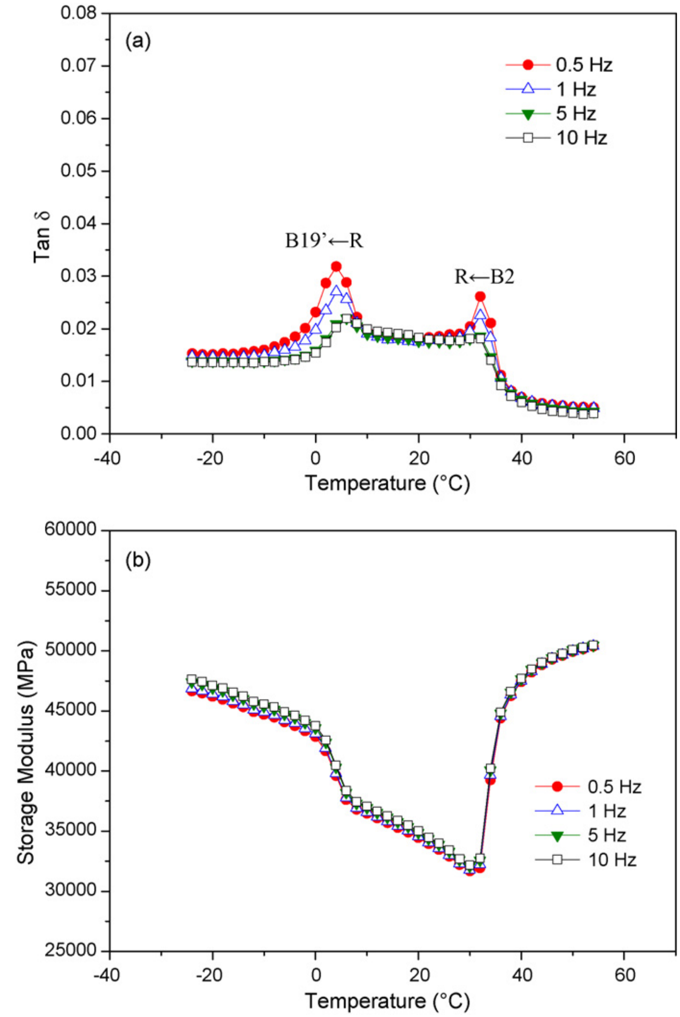

Fig. 4. (a) tan δ and (b) E_0 cooling curves for Fig. 2(b) specimen measured by step cooling method with isothermal time interval $t = 10$ min.

Fig. 5(a and b) plot the tan δ cooling curves measured by step cooling method under $t = 1$ min, 5 min, 10 min and 30 min when the applied frequency is 1 Hz and 10 Hz, respectively. As shown in Fig. 5(a), at $\nu = 1$ Hz, tan δ values of B2 → R and R → B19′ internal friction peaks decrease from 0.035 to 0.016 (54.3% reduction) and from 0.045 to 0.019 (57.8% reduction), respectively, when the t is increased from 1 min to 30 min. On the other hand, as shown in Fig. 5(b), tan δ values of B2 → R and R → B19′ internal friction peaks measured at $\nu = 10$ Hz only decrease from 0.021 to 0.016 (23.8% reduction) and from 0.025 to 0.019 (24.0% reduction), respectively, with increasing $t = 1$–30 min. Fig. 6 plots the tan δ values versus t of B2 → R and R → B19′ internal friction peaks under different frequencies. As shown in Figs. 5 and 6, both tan δ values of B2 → R and R → B19′ peaks decrease with increasing the t. Besides, the tan δ values measured at lower frequency decrease more significantly than those at higher one. This feature indicates that the t effect is more obvious at lower frequency when the specimen is measured by step cooing method.

4. Discussion

It has been proposed that the internal friction associated with the first-order phase transformation is composed of IF_{Tr}, IF_{PT} and IF_I [19–23]. The first term IF_{Tr} which appears at low ν and non-zero $\dot{T}$ is the transitory internal friction. Besides, according to Delorme's theory [19], the tan δ value of IF_{Tr} is proportional to the volume transformed per unit time. Therefore, IF_{Tr} should diminish when the specimen is kept isothermally at a constant temperature. The second term IF_{PT} is the internal friction due to the phase transformation and is independent on $\dot{T}$. The third term IF_I is the intrinsic internal friction contributed from the single phase such as austenitic or martensitic phase. In the low frequency range, the internal friction peak observed during martensitic transformation is mainly ascribed to the first term IF_{Tr}, instead of IF_{PT} and IF_I. Since IF_{Tr} is a non-steady term which diminishes with the isothermal time interval t, it is important to observe the relation between IF_{Tr} tan δ value and t during isothermal condition. Fig. 7 plots tan δ values versus t (0–30 min) when Fig. 2(b) specimen is kept isothermal ($\dot{T} = 0\,^{\circ}$C/min) at the B2 → R and R → B19′ internal friction peak temperatures. As shown in Fig. 7, both tan δ values of B2 → R and R → B19′ transformations decrease significantly in the initial state of isotherm and then gradually reach a steady value. According to reported studies [19–23], the decayed tan δ values during isotherm represent the transitory internal friction IF_{Tr}, and the steady tan δ values after 30 min isotherm for B2 → R and R → B19′ transformations are the inherent internal friction $(IF_{PT} + IF_I)^{B2\to R}$ and $(IF_{PT} + IF_I)^{R\to B19'}$, respectively,

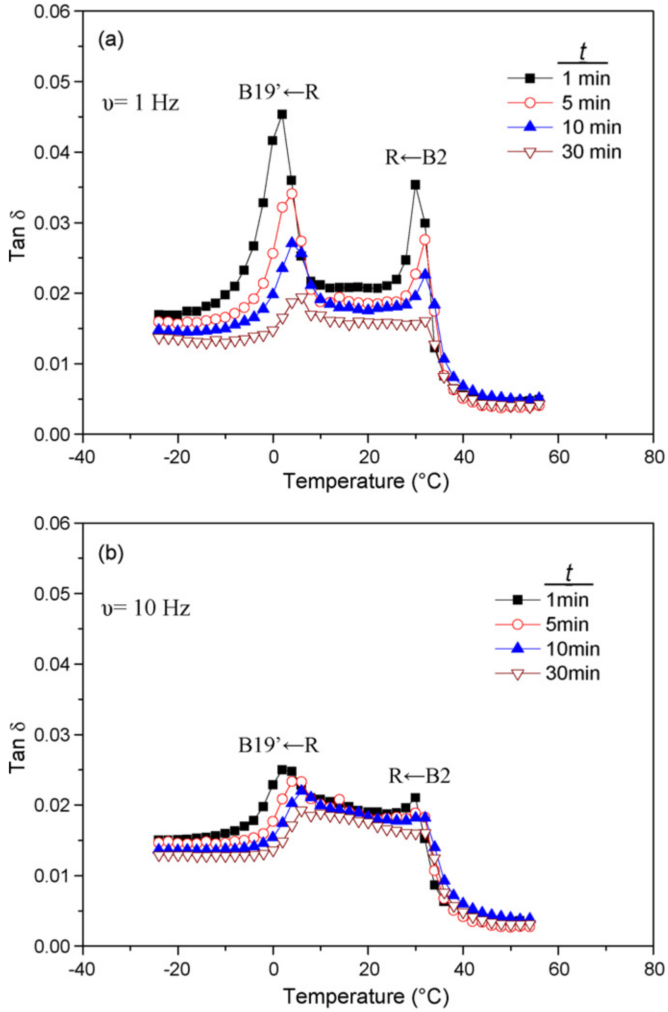

Fig. 5. tan δ cooling curves for Fig. 2(b) specimen measured by step cooling method at (a) $\nu = 1$ Hz and (b) $\nu = 10$ Hz.

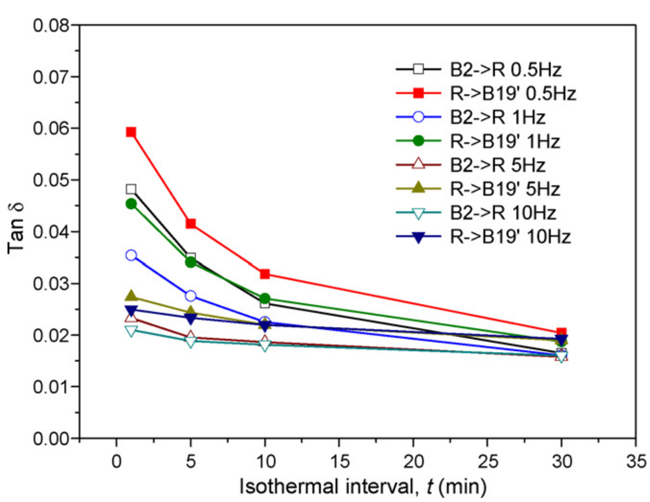

Fig. 6. tan δ values vs. isothermal time interval t of B2 → R and R → B19′ peaks measured by step cooling method.

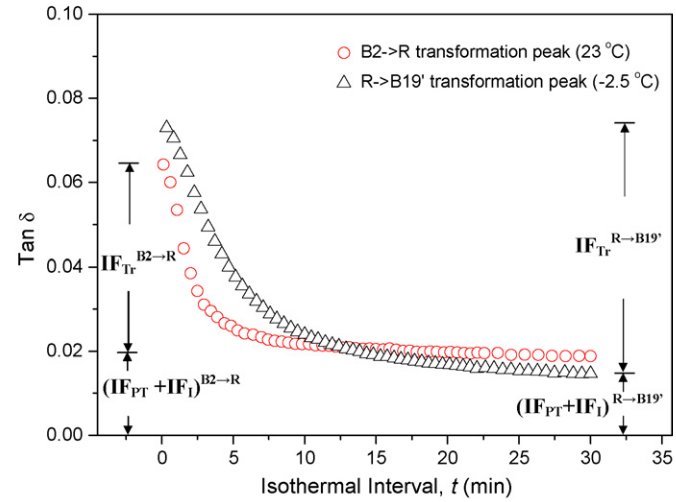

Fig. 7. tan δ values vs. isothermal time interval t when specimen is kept isothermally at B2 → R and R → B19′ peak temperatures with $\nu = 1$ Hz.

as illustrated in Fig. 7. Consequently, the tan δ value of IF_{Tr} is indeed 0 when the specimen is kept at a constant temperature after a sufficient time and the residual tan δ value is only originated by IF_{PT} and IF_I. In other words, the measured tan δ value during martensitic transformation is a combined contribution of ($IF_{PT} + IF_I$) and IF_{Tr} before it achieves a steady state. Therefore, it is difficult to measure the internal friction properties of TiNi-based SMAs precisely by step cooling method. This is because the tan δ value does not achieve a steady state before the insufficient t value and thus the residual IF_{Tr} still subsists and decreases continuously during the following frequency sweep process. This characteristic explicates tan δ values of B2 → R and R → B19′ internal friction peaks measured by step cooling method are lessening with prolonging the t value, as shown in Figs. 5 and 6, due to the diminishing IF_{Tr} under isothermal treatment. Besides, it has been reported that the tan δ value of IF_{Tr} is a function of $\dot{T}/\nu$ in the low frequency range ($\nu < 10$ Hz) [19–23]. This characteristic reveals the tan δ value of IF_{Tr} is lower and thus the t effect is less significant when the step cooling method is measured at higher frequency, as shown in Figs. 5 and 6.

Though IF_{Tr} is a non-steady term which is diminishing during isothermal treatment, it should be very carefully to choose the t value when internal friction properties of TiNi-based SMAs are measured by step cooling method. If the chosen t value is insufficient, the measured tan δ value is still decreasing during the following frequency sweep process and causes the unreasonable results, such as the tan δ value measured at higher frequency can be greater than that measured at lower one [16,17]. This feature is unreasonable because the tan δ value of IF_{Tr} and ($IF_{PT} + IF_I$) is a function of $\dot{T}/\nu$ and $\sigma_0/\nu^{1/2}$, respectively, in the low frequency range ($\nu < 10$ Hz) [13–15,19–23]. Nevertheless, from the experimental results of Fig. 7, the tan δ value of IF_{Tr} achieves a 95% reduction after the specimen was kept isothermally at B2 → R and R → B19′ peak temperatures for 12.7 min and 17.7 min, respectively. Consequently, it is acceptable to measure the internal friction properties of TiNi-based SMAs by step cooling method with a sufficient t value, such as chosen $t = 20$ min, to eliminate most of the influence of the non-steady IF_{Tr} during the following frequency sweep process. At $t = 20$ min, the tan δ reduction of IF_{Tr} at B2 → R and R → B19′ peak reaches 97.8% and 96.3%, respectively.

5. Conclusions

The internal friction of TiNi-based SMAs measured by step cooling method in DMA may be misled if 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} does not achieve its steady state after the insufficient t and still subsists and decreases continuously during the following frequency sweep process. The t effect is more significant when the step cooling method is measured at lower frequency because IF_{Tr} is a function of $\dot{T}/\nu$. Since IF_{Tr} is a non-steady term which is diminishing during isothermal treatment, it is important to measure the internal friction of TiNi-based SMAs by step cooling method with a sufficient t value. 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}.

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