Damping Capacities of Ti50Ni50–xCux Shape Memory Alloys Measured under Temperature, Strain, and Frequency Sweeps

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The damping capacities of Ti50Ni50–xCu SMAs with x = 0–20 at% were measured by DMA under temperature, strain and frequency sweep tests. The tanδ value exhibited at B2→B19 transformation is significantly higher than that at B2→B19 transformation under temperature/strain sweep tests. In the strain sweep test, the tanδ value decreases as the frequency increases, but it increases as the applied strain increases. The tanδ curves of the strain sweep tests can be fitted by tanδ = Kλn with n values being close to the friction type model. Adding Cu enhances the tanδ values of both B19 martensite and B2→B19 transformation in 0.1–10Hz, but that of B19′ martensite is enhanced only at 10Hz. From 0.1Hz to 1Hz, the decrement of the tanδ value in IF1 term is greater than that in IFPT term for SMA with x ≥ 5 at%. In the frequency sweep test, the tanδ value increases as the x value increases under the same applied frequency, no matter whether the martensite is B19′ or B19. [doi:10.2320/matertrans.M2014304]

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1. Introduction

TiNi shape memory alloys (SMAs), which undergo thermoelastic martensitic transformation, can exhibit a good shape memory effect (SME), pseudoelasticity (PE), and high damping capacity.1–8 Adding Cu into TiNi SMAs improves the SME and PE properties, and reduces the temperature hysteresis of the SMAs.9,10 It has been reported that the transformation sequences of Ti50Ni50–xCux (x = 0–30 at%) SMAs are B2→B19′, B2→B19→B19′ and B2→B19, when the Cu-contents are x ≤ 7.5, 10 ≤ x ≤ 15 and 18 ≤ x ≤ 30, respectively, by electrical resistance measurement, dynamic mechanical analyzer (DMA) test and theoretical calculation,12–15 but they are x ≤ 5, 7.5 ≤ x ≤ 15 and 20 ≤ x ≤ 30, respectively, by Seebeck coefficient measurement.12 Van Humbeeck et al.16 proposed that, for TiNi-based and Cu-based SMAs, the total internal friction, Q−1, can be decomposed into three terms, IFPT, IF1 and IFTr. The IFPT term is associated with phase transformation internal friction and is independent of the cooling/heating rate.16,17 The value of IFPT increases as the applied amplitude and the transformed volume per stress unit increase,8 and a small peak is exhibited when the mobility of the interfaces/boundaries reaches a maximum value.10 The IF1 term refers to intrinsic internal friction, responding to a single phase, i.e. martensite or austenite phase, and is strongly dependent on dislocation density and twin boundary.10–13 In general, the value of IF1 is greater for martensite than for austenite, which contributes to the reversible motion of twin boundaries between martensite variants. The IFTr term denotes transient internal friction and is much more significant than the IFPT and IF1 terms during martensitic transformation. Delorme et al.21 pointed out that, during martensitic transformation in Fe-Ni, Co-Ni, and Fe-Cr-Ni alloys, the IFTr term is proportional to the cooling/heating rate, the transformed volume rate, the applied frequency, and the strain amplitude, but it vanishes under the isothermal condition.23 Most damping materials are used at constant temperature, so the damping capacities, i.e., the IFPT and IF1 terms, under isothermal condition are important. However, very few reported studies have investigated the values of IFPT and IF1 in Ti50Ni50–xCu SMAs.24,25 The damping properties, especially the IFPT and IF1 at a low frequency, for Ti50Ni50–xCu SMAs under the strain sweep tests (tanδ values vs. strain under constant frequency and temperature) and the frequency sweep tests (tanδ values vs. applied frequency under constant strain amplitude and temperature) are still unclarified. Therefore, this study systematically investigates the damping properties of Ti50Ni50–xCu (x = 0–20 at%) SMAs by strain sweep and frequency sweep tests. The effects of the applied strain on the damping capacities exhibited in Ti50Ni50–xCu SMAs are also discussed.

2. Experimental Procedures

Ti50Ni50–xCu (x = 0, 5, 7.5, 10, 12.5, 15 and 20 at%) ingots were prepared by a vacuum arc remelting (VAR) with the raw materials of titanium (purity of 99.7 mass%), nickel (purity of 99.99 mass%), and copper (purity of 99.9 mass%), totaling about 120 g. The ingots were remelted six times in an argon atmosphere which had passed through a gas purifier to reduce its oxygen content. The weight loss during the remelting was less than 1 × 10−4. For Ti50Ni50–xCu SMAs with x ≤ 12.5 at%, the ingots were hot rolled at 900°C into plates of about 2 mm thickness by a rolling machine (DBR150x200 2HI-MILL, Daito Seiki Co, Japan), and then solution-heat-treated at 900°C for 1 h followed by quenching in water, but those with x = 15 and 20 at% were only solution-heat-treated at 900°C for 4 h, followed by quenching in water because these SMAs are intrinsically brittle. The surface oxide layer of the plate was removed using an etching
solution of HF : HNO₃ : H₂O = 1 : 5 : 20 in volume ratio. Thereafter, the plate was cut into specimens with dimensions of 40 × 7 × 2 mm³ for damping measurements. All specimens were prepared to the same size for the investigation.

The damping properties of the specimens were determined by a TA 2980 DMA instrument equipped with a single/dual cantilever and a liquid nitrogen cooling apparatus. In the strain sweep and frequency sweep experiments, the specimen was first cooled to −130°C and isothermally treated for 1 min, then heated to a specific temperature at a heating rate of 3°C/min and held at that temperature for 30 min to eliminate the βTₐ term. The applied amplitude and frequency range from 0.5 μm (7.1 × 10⁻⁶ strain) to 25 μm (3.5 × 10⁻⁴ strain) and 0.1 Hz to 10 Hz, respectively.

3. Results

3.1 The temperature sweep tests

Figures 1(a)–1(c) plot the curves of the tan δ value vs. temperature accompanied with those of the storage modulus vs. temperature for Ti₅₀Ni₅₀−ₓCuₓ SMAs with x = 5, 12.5 and 20 at%, respectively. More data of the curves obtained from the temperature sweep tests for Ti₅₀Ni₅₀−ₓCuₓ SMAs with x = 0, 7.5, 10 and 15 at%, including those plotted in Fig. 1, had been shown in our previous paper. From Fig. 1 and Ref. 15), transformation temperatures of Ti₅₀Ni₅₀−ₓCuₓ SMAs with x = 0–20 at%, including Mₙ, Mₚ, Mₙ', Mₚ', Mₙ, A₁ and A₄ are measured and listed in Table 1. Here, from the curves of the tan δ value vs. temperature, Mₙ and Mₚ are the starting and finishing temperatures of B₂→B₁₉ transformation, respectively; Mₙ' and Mₚ' are the starting and finishing temperatures of B₂→B₁₉ transformation, respectively; A₁ and A₄ are the starting and finishing temperatures of B₁₉→B₂ or B₁₉→B₂ transformation, respectively; and Mₙ and Mₚ are the peak temperatures of B₂→B₁₉' and B₂→B₁₉ transformations, respectively. From Fig. 1 and Ref. 15), for x = 0, 5 and 7.5 at%, there is a tan δ value peak corresponding to B₂→B₁₉' transformation with small storage modulus softening for each SMA, and the result of x = 5 at% is shown in Fig. 1(a); for x = 15 and 20 at%, there is also a tan δ value peak corresponding to B₂→B₁₉ transformation with large storage modulus softening, and the result of x = 20 at% is shown in Fig. 1(c), in which the tan δ value peak corresponding to B₁₉→B₁₉' transformation is about 100°C below Mₚ' temperature. It can be seen that Ti₅₀Ni₅₀−ₓCuₓ SMAs with x = 10, 12.5 and 15 at% exhibit a two-stage B₂→B₁₉→B₁₉' transformation, and the result of x = 12.5 at% is shown in Fig. 1(b). The transformation peaks of B₂→B₁₉ and B₁₉→B₁₉' transformations, in which can interfere the damping capacities exhibited in B₂→B₁₉ and B₁₉→B₁₉' transformations, so this SMA was not selected for the following strain sweep and frequency sweep tests. Although the low temperature tan δ value peaks in Figs. 1(a) and 1(b) look like the same, but when the applied frequency increases, the peaks in Fig. 1(a) shift to higher temperatures, but those in Fig. 1(b) do not. Therefore, the low temperature tan δ value peaks in Fig. 1(a) are regarded as relaxation peaks, and those in Fig. 1(b) are transformation peaks. According to Fig. 1 and Table 1, a full B₁₉ martensite for x = 0, 5 and 7.5 at% SMAs, and a full B₁₉ martensite for x = 12.5, 15 and 20 at% SMAs, when the specimens were cooled to −130°C and then heated up to room temperature. Therefore, from these SMAs, the

<table>
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<th>Mₙ'</th>
<th>Mₚ'</th>
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<td>54.8</td>
<td>39.8</td>
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</tr>
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*1: Here is the B₁₉→B₁₉' transformation, not the B₂→B₁₉' transformation.
*2: The two transformations are too close to identify the finishing temperature of B₂→B₁₉, and the starting temperature of B₁₉→B₂ transformations.
*3: The transformation temperature for B₁₉→B₂ transformation.
*4: Here is the transformation temperature for B₁₉→B₂ transformation.
the fact that, before the strain sweep test, the specimen is kept isothermally at room temperature for 30 min to eliminate the $IF_{PT}$ term. At the same time, no phase transformation appears in Fig. 2 at room temperature during the strain sweep test, so there is no $IF_{PT}$ term. As can be seen in Fig. 2, for the same SMA, the $\tan \delta$ value ($IF_1$ term) decreases as the frequency increases, but it increases as the applied strain increases. In addition, for B19 martensite in Ti$_{50}$Ni$_{50}$-Cu SMAs with $x = 12.5$ and 20 at%, the $\tan \delta$ value ($IF_1$ term) increases as the Cu-content ($x$ value) increases, especially when the applied strain is large. However, for B19' martensite in SMAs with $x = 0$ and 7.5 at% tested at 0.1 Hz and 1 Hz, the $\tan \delta$ value ($IF_1$ term) is almost the same in all testing ranges of the applied strain, but at 10 Hz, the $\tan \delta$ value is a little higher at $x = 7.5$ at% than at $x = 0$ at% under the same applied strain. Therefore, under the same testing condition, the Cu addition can enhance the $\tan \delta$ value ($IF_1$ term) for B19 martensite in the range of 0.1 Hz~10 Hz, but that for B19' martensite only at 10 Hz. The $\tan \delta$ value at the same applied strain with $x = 5$ at% is just in between $x = 0$ at% and $x = 7.5$ at%, and that with $x = 15$ at% is just in between $x = 12.5$ at% and $x = 20$ at%; therefore, the curves of the strain sweep tests for $x = 5$ and 15 at% SMAs are not shown in Fig. 2.

Figures 3(a) and 3(b) show the plots of the curves for the $\tan \delta$ value vs. applied amplitude (strain) at the $M_F$ temperature of B2$\rightarrow$B19' transformation for $x = 0$, 5 and 7.5 at% tested at 0.1 Hz and 1 Hz, respectively; Figs. 3(c) and 3(d) plot those at the $M_F$ temperature of B2$\rightarrow$B19 transformation for $x = 12.5$, 15 and 20 at% tested at 0.1 Hz and 1 Hz, respectively. The $\tan \delta$ value shown in Fig. 3 is contributed by both $IF_{PT}$ and $IF_1$ terms because the specimen was tested under the isothermal condition, in which the $IF_{PT}$ term is eliminated. At the same time, the test was also conducted at the transformation peak temperature, so the $\tan \delta$ value has the $IF_{PT}$ term. From Fig. 3, under the same applied strain, one can see that the $\tan \delta$ value exhibited at B2$\rightarrow$B19 transformation is significantly higher than that at B2$\rightarrow$B19' transformation. This characteristic comes from the fact that B2$\rightarrow$B19 transformation involves greater storage modulus softening than B2$\rightarrow$B19' transformation does, as shown in Figs. 1(a) and 1(c). Meanwhile, for B2$\rightarrow$B19 transformation, the higher the Cu-content ($x$ value) in the SMA is, the larger the $\tan \delta$ value it has, especially when the applied strain is higher than $1 \times 10^{-4}$. However, for B2$\rightarrow$B19' transformation under the same applied strain, the $\tan \delta$ values exhibited for SMAs with $x = 5$ and 7.5 at% are almost the same, and these values are a little higher than that for the SMA with $x = 0$ at%. Therefore, for B2$\rightarrow$B19' transformation, when a small amount of Cu element with $x \leq 7.5$ at% is added in the Ti$_{50}$Ni$_{50}$ SMA, its $\tan \delta$ value becomes a little higher.

The reported study investigated the $IF_1$ term of Ti$_{50}$Ni$_{50}$-Cu, SMAs with $x = 5$, 15 and 20 at% under the isothermal condition for 30 min, but only the condition at $8.5 \times 10^{-5}$ strain and 1 Hz frequency was measured. These reported data are almost the same as the $\tan \delta$ values ($IF_1$ term) obtained at $8.5 \times 10^{-5}$ strain and 1 Hz frequency shown in Figs. 3(b) and 3(d). This indicates that the approach of the strain sweep test to measure the $\tan \delta$ values ($IF_1$ term) is convincing. Furthermore, the parameters of the strain and

Fig. 2 The $\tan \delta$ curves of the strain sweep test for B19' and B19 martensites at room temperature, measured at (a) 0.1 Hz, (b) 1 Hz, and (c) 10 Hz.

damping properties of the full B19' and B19 martensites can be investigated individually at room temperature under the strain sweep and frequency sweep tests. These two sweep tests were also conducted at $M_P$ and at $M'_P$ temperatures to study the damping characteristics exhibited in B2$\rightarrow$B19' and B2$\rightarrow$B19 transformations, respectively.

3.2 The strain sweep tests

Figures 2(a)–(c) show the plots of the curves for $\tan \delta$ value vs. applied amplitude (strain) for B19' and B19 martensites at room temperature under the strain sweep test at 0.1 Hz, 1 Hz and 10 Hz, respectively. The $\tan \delta$ value shown in Fig. 2 is contributed only by the $IF_1$ term. This comes from

$x$ Cu, Shape Memory Alloys Measured under Temperature, Strain, and Frequency Sweeps
The frequency used in this study are varied in a wide range, thus the effects of the strain and frequency on the \( IF_I \) and \( IF_{PT} \) terms can be discussed, as shown in the following.

The decrement of the \( \tan\delta \) value from 0.1 Hz to 1 Hz under 3.5 \( \times \) 10\(^{-4}\) strain at room temperature, and that at \( M_p \) and \( M_p' \) temperatures, are measured from Figs. 2 and 3, respectively, and the results are shown in Fig. 4. As mentioned above, the \( \tan\delta \) value in Fig. 2 is contributed only by the \( IF_I \) term, and that in Fig. 3 is contributed by both \( IF_{PT} \) and \( IF_I \) terms. Therefore, from Fig. 4, one can find that the decrement of the \( \tan\delta \) value contributed by both \( IF_{PT} \) and \( IF_I \) terms is higher than that contributed only by the \( IF_I \) term. Besides, the magnitude of the decrement increases as the \( x \) value increases in the range of \( x \leq 12.5 \) at\%, and it roughly keeps the same for the further \( x \) value. This feature indicates that the frequency effect on the decrement of the \( \tan\delta \) value is more sensitive with Cu content (\( x \) value) for B19\textsuperscript{A} martensite than for B19 martensite, and also for B2\textsuperscript{A}B19\textsuperscript{A} transformation than for B2\textsuperscript{A}B19 transformation. In addition, from Ref. 13), one can find that the temperature effect on the \( \tan\delta \) value is insignificant under isothermal condition.

### 3.3 The Frequency Sweep Tests

Figure 5 plots the curves of the \( \tan\delta \) value vs. frequency for B19\textsuperscript{A} and B19 martensites tested at room temperature under a constant amplitude of 5 \( \mu \)m (7.1 \( \times \) 10\(^{-5}\) strain). Figures 6(a) and 6(b) show the plots of the curves for the \( \tan\delta \) value vs. frequency tested under 7.1 \( \times \) 10\(^{-5}\) strain at the \( M_p \) temperature of B2\textsuperscript{A}B19\textsuperscript{A} transformation for \( x = 0, 5 \) and 7.5 at\%, and at the \( M_p' \) temperature of B2\textsuperscript{A}B19\textsuperscript{A} transformation for \( x = 12.5, 15 \) and 20 at\%, respectively. As shown in Figs. 5 and 6, the \( \tan\delta \) value decreases as the frequency increases until a resonant frequency appears at around 50~60 Hz. Meanwhile, as can be seen in Fig. 5, under the same applied strain, the \( \tan\delta \) value of B19 martensite is remarkably higher than that of B19\textsuperscript{A} martensite, and the \( \tan\delta \) value increases as the \( x \) value increases under the same applied frequency, no matter whether the martensite is B19\textsuperscript{A} or B19.
4. Discussion on the damping characteristics exhibited by strain sweep and frequency sweep tests

4.1 The two regions of the tan δ curves in the strain sweep test

The tan δ curves shown in Figs. 2 and 3 can be fitted with a power function, \( \tan \delta = K \varepsilon^n \), where \( K \) is a constant, \( \varepsilon \) is the strain and \( n \) is the exponent value. The calculated \( n \) values for Figs. 2 and 3 are listed in Table 2 and 3, respectively, and these values are found in the range of 0.41–0.60. From Figs. 2 and 3, one can find that the \( \tan \delta \) value increases as the applied strain increases, but it decreases as the frequency increases, regardless of the specimen is B19 or B19 martensite. Figures 2 and 3 also indicate that the strain dependence of \( \tan \delta \) values can be divided into two regions. In the region of low strain, \( \tan \delta \) values are almost independent or weakly dependent on the strain. However, in the region of high strain, \( \tan \delta \) value increases rapidly as the strain increases. It is well known that \( Q^* = \tan \delta \) when the \( \delta \) value is small.\(^{26-28}\) According to the previous report,\(^{29}\) for the strain sweep test, \( Q^* \) is the sum of the strain independent or weakly dependent part, \( Q_0^* \), and the strain dependent part, \( Q_h^* \). As proposed by the Granato-Luecke break-away model\(^{24,29-31}\) at low strains, dislocations are pinned by the weak pinning points, so \( Q_0^* \) dominates \( \tan \delta \) value, and \( Q^* \) is independent or weakly dependent on the strain. However, at high strains, \( Q_h^* \) dominates the \( \tan \delta \) value which increases as the strain increases.

The Granato-Luecke break-away model assumes that the strain dependent part, \( Q_h^* \), is related to the dislocations break-away from “a single row” of pinning points. However, the friction type model was proposed recently which can explain this phenomenon more accurately.\(^{32-34}\) In this new model, the high strain dependent part comes from the dislocations overcome “several rows” of pinning points. In addition, the calculated \( n \) values in Granato-Luecke break-away model are around 2.5–3.0, but those in friction type model are around 0.5.\(^{35}\) Therefore, from the calculated \( n \) values shown in Tables 2 and 3, one can find that the characteristics shown in Figs. 2 and 3 are more close to the friction type model. Moreover, the calculated \( n \) values for B19 martensite and B2→B19 transformation are higher than those for B19’ martensite and B2→B19’ transformation, respectively. This feature may be attributed to the storage

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<th>( x=12.5^* )</th>
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</table>

*1 for B19’ martensite.
*2 for B19 martensite.

<table>
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<th>( x=15^1 )</th>
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</tr>
<tr>
<td>1 Hz</td>
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<td>0.50</td>
<td>0.50</td>
<td>0.58</td>
<td>0.54</td>
<td>0.60</td>
</tr>
</tbody>
</table>

*1 for B2→B19’ transformation.
*2 for B2→B19 transformation.

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**Table 2** The calculated exponent \( n \) values measured from Fig. 2 in the strain sweep tests for Ti\(_{50}\)Ni\(_{50}\)Cu SMAs at room temperature.

**Table 3** The calculated exponent \( n \) values measured from Fig. 3 in the strain sweep tests for Ti\(_{50}\)Ni\(_{50}\)Cu SMAs at \( M_p \) and \( M_p^A \) temperatures.

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Fig. 5 The tan δ curves vs. frequency of the frequency sweep test for B19’ and B19 martensites at room temperature for \( 7.1 \times 10^{-5} \) strain.

Fig. 6 The tan δ curves of the frequency sweep test, measured at 5 \( \mu \)m (\( 7.1 \times 10^{-5} \) strain) (a) at \( M_p \) temperature for B2→B19’ and (b) at \( M_p^A \) temperature for B2→B19’.

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modulus softening exhibited in B2→B19 transformation which is higher than that in B2→B19’ transformation, and may also be contributed by the lower storage modulus value exhibited in B19 martensite than that in B19’ martensite at room temperature.  

4.2 The frequency effect on $IF_I$ and $IF_{PT}$ terms

The result shown in Fig. 4 indicates that the effect of frequency on the tan δ value contributed by the $IF_I$ term is higher than that by the $IF_{PT}$ term in Ti$_{50}$Ni$_{50-x}$Cu$_x$ SMAs with $x = 5$ and 7.5 at%, but this effect is just the opposite in low Cu-content with $x < 5$ at%. This characteristic demonstrates that the higher Cu-content with the larger solid-solution strengthening in Ti$_{50}$Ni$_{50-x}$Cu$_x$ SMAs can enhance the frequency effect on the $IF_I$ term in B19’ martensite more obviously than that on the $IF_{PT}$ term in B2→B19’ transformation. In addition, from Fig. 4, considering the damping capacity of the $IF_I$ term from the movement of twin boundaries between the martensite variants, and that of the $IF_{PT}$ term associated with the movement of the B2/B19 or B2/B19’ phase interfaces, the frequency effect on the mobility of variant twin boundaries is higher than that on the movement of phase interfaces in SMAs with $x \geq 5$ at%.

Figure 6 shows that, under the same frequency, the tan δ value of B2→B19 transformation is higher than that of the B2→B19’ transformation. At the same time, the tan δ value associated with B2→B19’ or B2→B19 transformation increases as the Cu-content ($x$ value) increases. The explanation is that the transformation shear in B2→B19 transformation is less than that in B2→B19’ transformation, which allows the twin boundaries to move more easily in B19 variants than in B19’ variants, and also suggests the interfaces between the B2/B19 phases move more easily than those between B2/B19’ phases.

In Fig. 6(a), the Cu addition can enhance the tan δ values of Ti$_{50}$Ni$_{50-x}$Cu$_x$ SMA, but tan δ values of $x = 5$ and 7.5 at% are almost the same until a resonant frequency appears. The similar phenomenon also occurs in Fig. 3. It means that, in B19’ martensite, 5 at% Cu added in Ti$_{50}$Ni$_{50}$ SMA can increase the tan δ values obviously, but more Cu addition seems no obvious effect in both strain sweep and frequency sweep tests at the $M_P$ temperature.

5. Conclusions

The damping capacities of Ti$_{50}$Ni$_{50-x}$Cu$_x$ SMAs with $x = 0$~20 at% were measured under temperature, strain, and frequency sweep tests. In the strain sweep test at room temperature, the tan δ value decreases as the frequency increases, but it increases as the applied strain increases. In the strain sweep test at transformation peak temperature, the tan δ value exhibited at B2→B19 transformation is significantly higher than that at B2→B19’ transformation. This feature comes from the fact that B2→B19 transformation involves higher storage modulus softening than B2→B19’ transformation does. The added Cu enhances the tan δ value for B19 martensite and for B2→B19 transformation in 0.1~10 Hz, but the value for B19’ martensite is enhanced only at 10 Hz. The tan δ curves of the strain sweep tests can be fitted by tan δ $= Ke^n$ with $n$ values being close to the friction type model. In addition, the calculated $n$ values for B19 martensite and B2→B19 transformation are higher than those for B19’ martensite and B2→B19’ transformation, respectively. Also, the decrement of the tan δ value in $IF_{PT}$ term is greater than that in $IF_I$ term for SMA with $x = 0$ at%, but it is just the opposite for SMAs with $x \geq 5$ at%. In the frequency sweep test, the tan δ value decreases as the frequency increases until a resonant frequency appears. Meanwhile, the tan δ value of B19 martensite is remarkably higher than that of B19’ martensite, and this tan δ value increases as the $x$ value increases under the same applied frequency, regardless of the martensite is B19’ or B19. In addition, in B19’ martensite, a little Cu addition in Ti$_{50}$Ni$_{50}$ SMA can increase the tan δ value, but the higher Cu addition in Ti$_{50}$Ni$_{50}$ SMA seems no obvious effect in both strain sweep and frequency sweep tests at the $M_P$ temperature.

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