Multi-Axial Constitutive Equations of Polycrystalline Shape Memory Alloy* (Experimental Background)

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The inelastic behavior of shape memory alloy under multi-axial loading conditions was investigated by applying combined loads of axial force and torque to the thin-walled tube specimen made of Cu-based shape memory alloy. In this paper, unique behaviors revealed by the experiments which are not observed in the uniaxial experiments, are shown and discussed. These include the path dependency created by the change in microstructure (especially when variants operate simultaneously). The results obtained here manifest the significance of constitutive equations formulated on the basis of the semi-microscopic approach in the 1st paper.

Key Words: Constitutive Equations, Inelasticity, Combined Stress, Shape Memory Alloy, Multi-Axial Loading Experiments, Path Dependency, Change of Microstructure,

1. Introduction

The aim of this paper is to describe in detail the experimental results which support the formulation of semi-microscopic constitutive equations of shape memory alloy (SMA) proposed in the previous paper^{(1),(2)}, and to discuss the deformation mechanism of SMA from the theoretical as well as the experimental points of view.

SMA is one of the materials most expected to be used as the fundamental element of intelligent material systems or smart structures. The reason is that SMA has an actuating function with high power, a sensor function reacting sharply to thermal and stress changes, as well as sufficient strength and rigidity as a

structural material. Moreover, these functions (structural, sensor, and actuating) are retained at an atomic structural level, and thus this material can be integrated into micro-machines or micro-intelligent structures with high density. The development of SMA thin films is carried on these lines⁽³⁾.

SMA's are widely used not only in the engineering field but also in our daily life⁽⁴⁾. However, SMA is expected to have more potential applicability, and active research works have continued. Under this circumstance, some researchers have noted that the shape memory alloy may behave in a more complex and elegant manner under combined loading conditions, judging from the fundamental deformation mechanism of SMA; stress and temperature induced martensite phase forward and reverse transformations. That is, the performances of SMA are activated and controlled by six independent stress components and temperature. This fact suggests that we can realize a more elegant three-dimensional behavior of SMA by using the changes of these seven parameters in the proper way. We expect to widen the application area of SMA, which is now within that of simple one-dimensional loading behavior. However,

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there are few experimental results to support the above-mentioned idea⁽⁵⁾⁻⁽⁹⁾. With this basic motivation, the authors have performed systematical multi-axial loading experiments, by applying combined loads of axial force and torque to the thin-walled tube specimen of Cu-based shape memory alloy, and several interesting behaviors were observed. In this paper, the obtained results are reported and discussed from the viewpoint of the microscopic fundamental mechanism of phase transformation.

2. Experimental Methods

2.1 Material and geometry of specimen

The material used in the experiment is Cu-based shape memory alloy whose chemical constituents are 10 wt%Al, 5 wt%Mn, 5 wt%Zn and Cu. The characteristic temperatures of this material (heat treatment: rapid cooling by iced water following 2 hours-heating at 650°C) are as follows.

$$M_s = -30^\circ\text{C}, M_f = -55^\circ\text{C},$$

$$A_s = -35^\circ\text{C}, A_f = -15^\circ\text{C},$$

where M_s and M_f are the martensite phase transformation starting and finishing temperatures, respectively, and A_s and A_f are the austenite (reverse) phase transformation starting and finishing temperatures, respectively. Figure 1 shows the result of DSC measurement of the temperature.

Figure 2 shows the geometry of the specimen. As revealed by the figure, the specimen is a tube of a

Fig. 1 Characteristic temperatures of SMA measured by DSC

Fig. 2 Geometry of specimen

gauge length, outer diameter, and inner diameter of 40 mm, 8 mm, and 5 mm, respectively.

Figure 3 shows the uniaxial stress (σ)-strain (ϵ) curves at $T=293\text{ K}$ (20°C) and $T=233\text{ K}$ (-40°C). As revealed by the figure, the material shows a pseudo-elastic property at $T=293\text{ K}$ (20°C) and a shape memory property at $T=233\text{ K}$ (-40°C).

2.2 Experimental apparatus

The complex loading experiments on SMA are conducted using several two-dimensional stress paths obtained by applying the combined loads of axial force and torque to the thin-walled tube specimen shown in Fig. 2. The experimental equipment used here is the Shimadzu autograph (full-automatic complex loading testing machine) whose maximum axial load is 10 tonf (98.1 kN) and maximum torsion load (torque) is 2.0 kgf·m⁽¹⁰⁾. The temperature can be controlled by an electric furnace whose maximum temperature is 1100°C, and a low-temperature experiment is possible by introducing liquid nitrogen gas to the furnace.

3. Experimental Results

3.1 Proportional loading with temperature change

Figure 4 shows an example of experimental results for proportional loading with temperature change. Figure 4(a) shows the given thermo-mechanical loading path, where path 1→2 is the proportional loading of axial stress σ and torsion

Fig. 3 Uniaxial tension curve

Fig. 4 Proportional loading with temperature change

stress τ at room temperature, path 2 $\rightarrow$ 3 is the increase in temperature up to 370 K while the stress state is kept constant at $(\sigma, C_1\tau)=(280 \text{ MPa}, 240 \text{ MPa})$, path 3 $\rightarrow$ 4 is the decrease in temperature, and path 4 $\rightarrow$ 5 is the proportional unloading at room temperature. The obtained strain response is shown in Fig. 4(b), using the two-dimensional strain space $(\epsilon, C_2\gamma)$, where ϵ is the axial strain and γ is the shear strain of the tube specimen. C_1 and C_2 are the material constants, which are experimentally determined to obtain the coincidence of stress-strain relationships of tension and torsion. For example, $C_1=\sqrt{3}$ and $C_2=1/\sqrt{3}$ if the material is of von-Mises type. In the material used, $C_1=1.2$ and $C_2=0.5$. When the equivalent stress and strain are defined using these coefficients C_1 and C_2 , as follows,

$$\sigma_{EQ}=[\sigma^2+(C_1\tau)^2]^{1/2},$$

$$\epsilon_{EQ}=[\epsilon^2+(C_2\gamma)^2]^{1/2}$$

Relationships of $\sigma_{EQ}-\epsilon_{EQ}$ for the proportional deformations coincide very well with each other⁽¹¹⁾. As revealed by the fact that the values of C_1 and C_2 are different from those of von-Mises type material, the shape memory material used in this experiment may be strongly affected by not only the second invariant of stress deviator but also the first and third invariants of stress tensor. This observation may be quite important for the formulation of reliable constitutive equations using the phenomenological/mathematical theory of plasticity.

As revealed by Fig. 4(b), the deformation of SMA under proportional loading is also in proportional manner (see the strain response 1 $\rightarrow$ 2). Also, the deformation behavior obtained by the temperature cycle under the constant combined stress state is cyclic in a proportional manner (see the strain path 2 $\rightarrow$ 3 $\rightarrow$ 4). That is, we can obtain the two-dimensional actuating movement of SMA by applying the combined bias stress composed of axial force and torque. This type of two-dimensional actuating movement is quite interesting from the view point of SMA application.

Fig. 5 Non-proportional loading with temperature change (example 1)

3.2 Non-proportional loading with temperature change

Figure 5 shows an example of the experimental result obtained by using the non-proportional loading with temperature change. Figure 5(a) shows the thermo-mechanical loading path. In this figure, path 1 $\rightarrow$ 2 $\rightarrow$ 3 $\rightarrow$ 4 is the same one used in section 3.1, path 4 $\rightarrow$ 5 is the unloading of shear stress τ while keeping the axial stress σ constant, path 5 $\rightarrow$ 6 $\rightarrow$ 7 is the temperature cycle (293 K $\rightarrow$ 363 K $\rightarrow$ 293 K) while keeping the stress state constant, and the final path, 7 $\rightarrow$ 8 is the unloading of axial stress σ . Figure 5(b) shows the strain response to the thermo-mechanical loading 1 $\rightarrow$ 8. There are several interesting aspects observed here, for example,

(1) The shear strain γ still remains though the shear stress τ becomes zero at point 5.

(2) The strain state is not recovered after the temperature cycle 5 $\rightarrow$ 6 $\rightarrow$ 7 (the obtained strain states 5 and 7 are different from each other). That is, the shape memory alloy forgets the strain state 5 after the temperature change. This phenomenon is quite different from that observed in the case of proportional loading with temperature change shown in Fig. 4, where strain points 2 and 4 coincide with each other. This kind of path dependency observed in the shape memory alloy should be considered in the design of a two- or three-dimensional movement actuator of a shape memory alloy.

(3) Zero-stress corresponds to zero-strain after thermo-mechanical loading 1-8. That is, the shape memory alloy never forgets the initial state. Namely, the reverse transformation is completed.

Figure 6 shows another example of the experimental results obtained for the thermo/non-proportional loading path. Figure 6(a) shows the loading path. In this experiment, the specimen is first cooled to -40°C , (path 0 $\rightarrow$ 1). Path 1 $\rightarrow$ 2 is the torsion loading at -40°C , path 2 $\rightarrow$ 3 is the tensile loading (σ) while keeping the shear stress τ constant, path 3 $\rightarrow$ 4 is the change of shear stress τ while keeping the axial

Fig. 6 Non-proportional loading with temperature change (example 2)

Fig. 7 Torsion curve

stress σ constant, path $4 \rightarrow 5$ is the unloading of axial stress σ while keeping the shear stress τ constant, path $5 \rightarrow 6$ is heating while keeping the shear stress τ constant, path $6 \rightarrow 7$ is the unloading of shear stress τ while the temperature constant. Figure 6(b) shows the strain response. As found from the strain response $2 \rightarrow 3$, the shear strain γ increases with an increase of axial stress σ though the change of shear stress τ is not given. This phenomenon is similar to the plastic deformation caused by the slip mechanism. On the other hand, the axial strain ϵ increases along stress path $3 \rightarrow 4$ whose shear stress τ decreases (and whose axial stress σ is kept constant). This phenomenon is quite different from that of plastic deformation. Strain response $5 \rightarrow 6$ is much more unique than those mentioned above. First, the shear strain γ at point 5 is almost negligible. As found from the strain response $5 \rightarrow 6$, the shear strain γ appears while the axial strain ϵ decreases, and the shear strain γ turns to zero again after it attains a maximum value. This kind of complicated non-linear deformation behavior manifests the strong path dependency of SMA deformation behavior related to the micro-structural state change induced by the non-proportional loading.

3.3 Torsion test

In this section, the unique behaviors described in sections 3.1 and 3.2 are discussed using the results of torsion tests of SMA tube. The specially arranged torsion test is explained using Fig. 7. Figure 7 shows the shear stress τ -shear strain γ relationship obtained by simple torsion loading at -40°C , up to 150 MPa ($0 \rightarrow 1$), the unloading and following reverse loading up to -250 MPa ($1 \rightarrow 9$), and the unloading ($9 \rightarrow 13$). The residual (martensitic) strain was erased by the heating process ($13 \rightarrow 0$). During this one cycle torsion loading process, the temperature increased at each point 1–13 while keeping the shear stress constant, and the changes of shear strain were measured during this heating process.

Figure 8(a) shows the reverse phase transformation response obtained by heating from points 1–4,

(a)

(b)

Fig. 8 Torsion deformation by heating at constant stress state (I)

which are present during unloading process, and from points 10–13 which are present during the unloading of the reverse (backward) loading, while keeping the shear stress τ constant. As revealed by this figure, the mechanical states arrive at the elastic states related to each constant stress. That is, the mechanical states arrive at the elastic line $a-b$, because the martensite variants disappear due to heating process. Figure 8(b) shows the relationships between the amount of shear strain change ($C_2\Delta\gamma$) and the temperature (T) obtained by the heating process. As revealed by the figure, the changes of shear strain γ are monotonic to the temperature change.

Figure 9(a) shows the reverse phase transformation responses obtained by heating from points 5–9 which are present during the reverse loading process. In this figure, the response related to point 5 is the same as those in the cases of points 1–4 and points 10–13, as shown in Fig. 8. That is, the mechanical state approaches monotonically the elastic state. On the other hand, the response related to point 6 is quite different from the behaviors to the points 1–5 and 10–13. That is, the mechanical point approaches the elastic line ab , at first. Note that the state does not stop at point 6^{**} on the elastic line ab and arrives at point 6^* . That is, the mechanical state shows a kind of overshooting phenomenon, and the mechanical

Fig. 9 Torsion deformation by heating at constant stress state (II)

state finally arrives at the point 6** (on the elastic line ab) by the subsequent heating process. The result related to point 7 is more prominent. The mechanical state 7 is apparently on the elastic line ab. The mechanical state moves from point 7 to point 7* first and the state revert to the original point 7 on the elastic line ab by the subsequent heating process. Mechanical states 8 and 9 show the same kind of phenomenon under heating. That is, the mechanical states first move leftward and in the next stage they move in rightward and finally arrive at the points on the elastic line. Figure 9(b) shows the relationships between the amount of shear strain change ($C_2 \Delta \gamma$) and the temperature (T) obtained by the heating process. As revealed by the figure, the change of shear strain (γ) are not monotonic to the temperature change (T).

The mechanism of unique behavior, observed in the non-proportional loading described in sections 3.1 and 3.2, and observed in the special torsion experiments described in this section is quite interesting from the microscopic point of view. This mechanism may be related to the existence of several numbers of induced martensite variants and their interactions. In the case of the unique non-linear behavior observed in the simple torsion test, the mechanism may be explained on the basis of the idea that the variant in-

Fig. 10 Mechanism of non-monotonic change of phase transformation strain obtained by thermal loading

duced by the forward loading is different from that induced by the reverse loading after the forward loading. Figure 10 shows schematically the imagined appearance and disappearance of martensite variants in a selected grain component during the above-mentioned torsion deformation, related to point 7 in Figs. 7 and 9. Figures 10(a), (b) show the stress-strain relationship and the relationship between the change in shear strain ($\Delta \gamma$) and temperature (T), respectively. Illustrations A - F in Fig. 10 correspond to points A - F on the stress-strain relationship curve shown in Figs. 10(a), (b), and show the micro-structures with the behaviors of induced martensite variant at points A - F.

First, the temperature-induced martensite variant appears by cooling up to -40°C , as shown in illustration A. The variants have random orientations. The martensite phase transformation finishing temperature A_f is -55°C , thus the martensite variants do not cover the whole area of the crystal component, and the orientations of variants are randomly distributed; thus any significant macroscopic deformation is not observed at point A. In the next stage, the specimen is subjected to torsion loading while keeping the temperature constant (-40°C). The stress-induced martensite variants appear as shown in illustration B, by this torsion loading. The orientations of

these stress-induced variants are rather uniform (so-called preferable orientation) and thus significant inelastic (phase transformation) deformation is observed in the specimen. The subsequent reverse torsion loading up to -150 MPa (point C) produces martensite variants different from those obtained by the first forward loading. In this reverse loading process, the martensite variants produced by the first loading process may gradually decrease. But some of them remain at point C, and the amounts of two kinds of variants become the same at point C. That is, the strain related to the variant induced by the first process and the strain related to the variant induced by the second process cancel with each other out. As a result, the macroscopic inelastic strain is not observed, and thus the mechanical state C is on the elastic line a - b, though the microscopic state is not elastic. In this loading state (C), the martensite variant induced by the reverse loading is rather stable, and the martensite variant induced by the first forward loading is rather unstable, because the reverse torsion stress exists. Thus, the martensite variants induced by the forward loading decrease first by the heating process and thus macroscopic inelastic strain appears in the reverse direction. That is, the macroscopic deformation moves from point C to point D [Fig. 10(a)]. In the subsequent heating process, the martensite variant induced by the reverse loading decreases gradually and the deformation state moves from D to C(E), and stops at point C(E), where the mechanical state is elastic macroscopically as well as microscopically.

As explained above, the mechanism of the deformation process related to point 7 is the balance of two kinds of variants induced by different loading paths. In this case, the two kinds of variants are induced by the two different loading directions, and they co-exist; one of them is more stable than the other depending on the loading state. In the result, the unique path dependency appears in the deformation of the shape memory alloy. We recall that there are 24 potential variants in the crystal component, and it is possible for more variants than two to appear simultaneously in the complicated multi-loading stress path. The deformation behavior of shape memory alloy will be very complicated when the material is subjected the general thermo-mechanical loading conditions. Thus, it is quite important to formulate the reliable constitutive equations to predict the complex behavior.

4. Conclusion

The strong and interesting path dependency of shape memory alloys was observed experimentally by applying the combined loads of axial force and torque to the thin-walled tube specimen made of Cu-based shape memory alloy. The main mechanism of the path dependency is the decreasing and increasing of several numbers of martensite variants along the thermo-mechanical loading path. In this paper, two-dimensional examples were reported and discussed. In the general case that 6 stress-components change simultaneously in some loading courses, a larger number of variants appear and very complicated deformation can be given to the shape memory alloy. Finally, judging from these experimental results, we conclude that it may be very difficult to derive reliable constitutive equations by some traditional macroscopic theory, and the proposed constitutive equations constructed on the semi-micro approach may be useful. The third paper will describe the reliability of the proposed constitutive equations by comparing the experimental results and the simulated results.

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