Article

{111}<110> Orientation Induced Anisotropy of Shape Memory Effect in NiTiNb Pipe Joints

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Abstract: This work aims to clarify the influence of texture type and intensity on the shape memory effect (SME) in NiTiNb shape memory alloy (SMA) pipe joints, especially revealing the causes for the anisotropy of SME via texture changes. Three NiTiNb rods with different intensities of the {111}<110> texture were fabricated, and their microstructures, crystalline orientation distribution functions and inverse pole figures were obtained by X-ray diffraction and electron backscatter diffraction measurements. Simultaneously, the SME was characterized by inner-diameter recoverability of the corresponding pipe joints. For a given intensity of the {111}<110> texture, the SME of the NiTiNb pipe joints strongly depended on the expansion direction due to {111}<110> orientation-induced anisotropy of SME. In addition, both the SME and anisotropy of NiTiNb pipe joints increased with the increased intensity of the {111}<110> texture. Therefore, a suitable expansion direction and strong texture intensity should be considered for high SME in NiTiNb pipe joints.

Keywords: NiTiNb; anisotropy; texture; SME; pipe joints

1. Introduction

NiTi-based shape memory alloys (SMAs), due to their unique shape memory effect (SME), have been widely used in many fields such as aerospace, biomedicine, mechanical electronics and automotive industries [1–4]. Among them, one of the most important and successful applications is the pipe joint [5–7]. NiTiNb alloys, especially those with a nominal composition of Ni₄₇Ti₄₄Nb₉ (at%), have attracted much attention as SMAs because they demonstrate wide transformation hysteresis after pre-deformation [8–12]. Thus, pipe joints constructed from NiTiNb alloys do not require storing and installing at low temperatures, which is quite useful for the engineering uses [13–15].

Generally, pipe joints are machined from NiTiNb rods and then expanded at low temperature to augment their inner diameter [16,17]. According to previous studies, in order to store and ship NiTiNb pipe joints at room temperature and simultaneously optimize the SME, researchers mainly pay attention to the key problem of finding appropriate expansion temperatures and critical expansion strains [18–22]. However, the expansion direction is rarely considered, despite the anisotropy of SME induced by texture in NiTiNb rods. Identifying the expansion direction that optimizes the SME is therefore crucial for fabricating high-performance NiTiNb pipe joints. The expansion direction can be controlled by preparing the pipe joints along the radial or axial direction of the rods.

As is well known, the NiTiNb rods used for pipe joints are typically prepared by vacuum melting, followed by thermomechanical deformation and finally by a suitable heat-treatment [23–26]. These processes inevitably generate crystallographic textures that profoundly affect the SME. It is

necessary to understand which type of orientation features a higher reversibility and the extent to which texture intensity should be improved to satisfy pipe joints' application. Currently, studies on texture of NiTiNb are mainly focused on the effect and control of deformation and heat-treatment on the final texture types [26–29]. For example, Yan et al. found that the main texture types in $\text{Ni}_{47}\text{Ti}_{44}\text{Nb}_9$ hot-rolled rods concentrate on γ fiber ($\langle 111 \rangle //$ axial direction), while heat-treatment only decreases its intensity [26]. In addition, Yin et al. obtained $\langle 113 \rangle$ fiber texture in $\text{Ni}_{47}\text{Ti}_{44}\text{Nb}_9$ hot-forged rods, which turned into a strong γ fiber texture after cold-drawing [27]. In contrast, the relationship between texture and SME has been rarely investigated. The few studies on this topic have concentrated on the variations of recovery strain between the rolling direction (RD) and transverse direction (TD) in thin sheets, whereas pipe joints are generally prepared from rods [26]. Moreover, the positive role of texture in the SME of NiTiNb pipe joints has not been clearly clarified, because the expansion stress states of these joints are far more complex than those of uniaxial tensile or compressed specimens.

In this study, using $\text{Ni}_{47}\text{Ti}_{44}\text{Nb}_9$ as an example, three rods with different intensities of the $\{111\}\langle 110 \rangle$ texture were investigated. The study focused on clarifying the influence of texture type and intensity on the SME of $\text{Ni}_{47}\text{Ti}_{44}\text{Nb}_9$ pipe joints, especially revealing the causes for the anisotropy of SME via texture changes. The aim is to provide referential data for engineering applications.

2. Experimental Procedures

As shown in Figure 1, first, vacuum induction melting was used to produce the master ingot of $\text{Ni}_{47}\text{Ti}_{44}\text{Nb}_9$ (at%) with a diameter of 150 mm and a height of 200 mm. The master ingot was multi-directionally forged at 900 °C into 35 mm diameter rods for the sake of improving and homogenizing the microstructure of the as-cast ingot. To account for the influence of texture intensity on the resulting SME, the rods were rotary hot-forged along the axial direction by different passes to diameters of 15 mm, 10 mm and 8 mm (forming Samples A, B and C, respectively). Then, all rods were heat-treated in an evacuated quartz tube at 900 °C for 2 h followed by air quenching (AC) from high temperature to room temperature without cryogenic treatment. Finally, all samples were electric-discharge machined into the sizes needed for relevant measurements.

Figure 1. Schematic of the fabrication process of hot-forged rods and the orientations of the samples cut from the rods for texture measurements.

Phase identification and texture measurements were carried out by $\text{Cu K}\alpha$ radiation using X-ray diffractometer (Empyrean, Panalytical, Almelo, Netherlands). The X-ray diffraction (XRD) patterns of the three samples are similar, as shown in Figure 2. The pole figures from the crystallographic planes $\{110\}$, $\{200\}$, $\{211\}$ of the B_2 phase were measured at $\alpha = 0\text{--}70^\circ$ and $\beta = 0\text{--}360^\circ$ with a step size of 5° and presented as orientation distribution function (ODF) charts. The texture-test Samples A,

B and C (with dimensions of 15 mm, 10 mm and 8 mm in diameter, respectively, and 5 mm in length) were machined in the excision direction, which is perpendicular to the axial direction of the rods, as seen in Figure 1. Thus, the measured texture component $\{hkl\}\langle uvw \rangle$ means that the $\{hkl\}$ planes perpendicular to the axial direction of the rods, the $\langle uvw \rangle$ directions aligned along the radial direction and the $\langle rst \rangle$ directions aligned along the circumferential direction.

Figure 2. X-ray diffraction (XRD) patterns of Samples A, B and C, with the β -Nb and NiTi (B_2) peaks identified.

The phase transformation behavior was determined by differential scanning calorimeter (DSC, DSC 214 Polyma, Netzsch, Selb, Germany) at a 10 °C/min heating and cooling rate. DSC specimens (with dimensions of $1 \times 1 \times 2 \text{ mm}^3$) were chemically washed in a mixed acid solution.

The microstructures were characterized by electron backscatter diffraction (EBSD) to reveal their crystallographic grain boundaries and grain orientations. Both cross sections and longitudinal sections of the rods were tested. Specimens for EBSD were mechanically polished and then vibratory polished in colloidal silica. Field emission scanning electron microscope (SEM, JSM-6700F, JEOL Ltd., Tokyo, Japan) equipped with an EBSD detector and data analysis software (OIMTM, TSL-EDAX, Mahwah, NJ, USA) was used for EBSD test. For a convenient analysis, the normal direction (ND) of the EBSD test zones was set perpendicular to the test plane of the rods and the RD for specimens with cross section and longitudinal section paralleled the radial direction and axial direction of the rods, respectively.

Tensile tests were carried out in an electronic universal testing system equipped with an environmental experiment box under a loading speed of 0.5 mm/min. Dog-bone-shaped tensile specimens were cut to a gauge length of 7 mm and a width of 1 mm, along the axial and radial direction of the rods, as shown in Figure 3a.

Figure 3. Schematic of the samples used for (a) the tensile test and (b) the shape memory effect (SME) test.

To ensure that the results closely matched those of actual engineering applications, the SME was characterized by inner-diameter recoverability of real pipe joints. The pipe joints were machined with

an initial inner diameter (D_0), a wall thickness of 1.5 mm and the expansion direction (ED) along the radial and axial direction of the rods, as shown in Figure 3b. Then, the pipe joints were heat-treated at 900 °C for 2 h in an evacuated quartz tube and then expanded at −60 °C by using a core bar. After the inner diameter (D_1) was measured, the pipe joints were heated to 200 °C for 10 min to complete the inverse martensitic transformation. Then the inner diameter was again measured and recorded as D_2 . The recovery rate (η) and recovery strain (ε_r) of the pipe joints were calculated according to the following formulas:

$$\eta = \frac{D_1 - D_2}{D_1 - D_0} \times 100\% \quad (1)$$

$$\varepsilon_r = \frac{D_1 - D_2}{D_0} \times 100\% \quad (2)$$

3. Results

3.1. Texture Comparison and EBSD Characterization

The ODF results of Samples A, B and C are shown in Figure 4. The preferred orientation of all samples concentrates on $\{111\}\langle 110 \rangle$, as shown in the $\varphi_2 = 45^\circ$ sections of Figure 4a–c. According to Yan's analysis, on rotary hot forging, the radial direction of rod is subjected to compressive stress, slip planes rotate toward the direction perpendicular to the external stress axis and slip directions toward the plastic flow direction or to the rod axis [23]. Thereby, the $\{111\}\langle 110 \rangle$ texture formed in the hot-forged rods are mainly related to the activation of $\{111\}\langle 110 \rangle$ slip systems in the B_2 phase. Meanwhile, the maximum orientation density of $\{111\}\langle 110 \rangle$, from low to high, is ordered as A, B and C, indicating that the texture intensity can be controlled by the deformation degree. The inverse pole figure (IPF) maps of above three samples are obtained by EBSD test to present grain orientation (Figure 5). A strong $\{111\}\langle 110 \rangle$ texture is also observed in all samples, with grains in blue ($\langle 111 \rangle$ // axial direction) crossing most of the cross section and grains in green ($\langle 110 \rangle$ // radial direction) crossing most of the longitudinal section of the rods. This is consistent with the result tested by XRD above. In addition, grains of different shape with equiaxed grains on cross section (Figure 5a–c) and elongated grains on longitudinal section (Figure 5d–f) are observed, which means that the grains are elongated along the axial direction. Meanwhile, with the increase of deformation degree, the average grain size decreases and the grain morphology is further elongated.

3.2. Tensile Tests and Corresponding Anisotropic SME in the Rods

In order to clearly investigate the $\{111\}\langle 110 \rangle$ orientation-induced anisotropic SME in $\text{Ni}_{47}\text{Ti}_{44}\text{Nb}_9$ pipe joints, the deformation mode of uniaxial tension in $\text{Ni}_{47}\text{Ti}_{44}\text{Nb}_9$ rods is considered firstly. Taking Sample A as an example, dog-bone shaped tensile specimens are loaded at temperature of −60 °C, along the axial and radial direction (that is $\langle 111 \rangle$ and $\langle 110 \rangle$ direction, respectively) of the rods. As shown in Figure 6, the anisotropic stress–strain curves exhibit different length of phase transformation plateaus and different plateau stresses along the $\langle 111 \rangle$ and $\langle 110 \rangle$ directions. Specifically, the phase transformation plateau is longer along the $\langle 111 \rangle$ direction than along $\langle 110 \rangle$ direction and the plateau stress is higher along the $\langle 111 \rangle$ direction than that along the $\langle 110 \rangle$ direction. These results suggest that the stress-induced martensitic critical stress σ_{sim} along the $\langle 111 \rangle$ direction is larger. Meanwhile, the strain of the martensite nominal yield point (the nominal starting point that the dislocations begin to slip in martensite) is obviously larger along the $\langle 111 \rangle$ direction than along the $\langle 110 \rangle$ direction. In addition, after tension to 16%, the specimens are heated at 200 °C to calculate their recovery property. The recovery strain along the $\langle 111 \rangle$ direction is 9.7%, versus 9.1% along the $\langle 110 \rangle$ direction.

Figure 4. Orientation distribution function (ODF) results of the $\text{Ni}_{47}\text{Ti}_{44}\text{Nb}_9$ rods. (a) Sample A, (b) Sample B and (c) Sample C. Point A represents the $\{111\}\langle 110 \rangle$ component.

3.3. Recoverability of the Pipe Joints

Six pipe joints in each sample were measured for SME. The average value of η and ε_r are shown in Figure 7a,b, respectively. For the same expanding direction of ED1 or ED2, the average value of η and ε_r from low to high, is ordered as A, B and C. Meanwhile, in each sample, both η and ε_r along ED1 are higher than along ED2. In addition, the difference of recoverability between ED1 and ED2 increases on the order of A, B and C. These results reflect that Sample C with strongest texture of $\{111\}\langle 110 \rangle$ has the highest recoverability and the strongest anisotropy of recoverability and the reasons are discussed in the following section.

Figure 5. Inverse pole figure (IPF) maps of the NiTi (B_2) phase in different states, representing the preferred crystalline orientation in the normal direction (ND) of the test planes. (a–c) cross sections of Samples A, B and C, respectively; (d–f) longitudinal sections of Samples A, B and C, respectively.

Figure 6. Tensile curves obtained during loading at $-60\text{ }^{\circ}\text{C}$ along the $\langle 111 \rangle$ and $\langle 110 \rangle$ direction. The martensite yield nominal point is got from the intersection of true stress–strain curve with the martensite elastic stage tangent after 0.2% horizontal movement.

Figure 7. Recoverability of Samples A, B and C along the expansion directions of ED1 and ED2: (a) recovery rate η and (b) recovery strain ϵ_r .

4. Discussions

4.1. Effects of $\{111\}<110>$ Orientation on SME in the Uniaxial Tensile Samples

As is well known, the SME of SMA is determined by the crystallographic reversibility of the reverse martensite transformation. Therefore, different lattice orientations lead to distinct recoverability. Previously, several researchers concentrating on NiTi single crystals have shown that the tensile recovery strains of $\langle 001 \rangle$, $\langle 110 \rangle$, $\langle 111 \rangle$ are 2.7%, 8.4%, 9.8%, respectively, while compressive recovery strains of $\langle 001 \rangle$, $\langle 110 \rangle$, $\langle 111 \rangle$ are 4.2%, 5.2%, 3.6%, respectively [30,31]. As for $\text{Ni}_{47}\text{Ti}_{44}\text{Nb}_9$ polycrystals, it is NiTi phase which plays the main role in recoverability, thus the recovery strain of the polycrystalline alloy can be computed as:

$$\bar{\epsilon}_M = \sum_{i=1}^n \epsilon_{ri} I_i \quad (3)$$

Here $\bar{\epsilon}_M$ is the average recovery strain, ϵ_{ri} is the recovery strain in each orientation, I_i is the proportion of each orientation among the total orientations, and n is the number of the orientations. Therefore, as for the uniaxial tensile samples, the recovery strain along the $\langle 111 \rangle$ direction is larger than that along the $\langle 110 \rangle$ direction, which is in agreement with above experimental results.

To deeply understand the causes for anisotropic SME between the $\langle 111 \rangle$ and $\langle 110 \rangle$ direction, the tensile curves are analyzed in detail. According to previous studies on NiTiNb, the phase transformation plateau in stress–strain curves is formed by stress-induced martensite transformation and reorientation, the reversibility of which contributes to the strain recovery [32,33]. Therefore, when subject to the same deformation strain, a long plateau generally indicates a large recovery strain. As shown in Figure 6, the plateau is longer along the $\langle 111 \rangle$ direction than along the $\langle 110 \rangle$ direction, so the most favorably oriented martensite variants originating from $\langle 111 \rangle$ can generate larger strain than that from $\langle 110 \rangle$, thus resulting in high SME. In addition, the strain of martensite nominal yield point along $\langle 111 \rangle$ direction is obviously larger than that along $\langle 110 \rangle$ direction. Thus, when loaded to the same strain of 16%, dislocations are easily generated along the $\langle 110 \rangle$ direction, which will partly impede the reverse transformation, according to the previous research that deformation-induced dislocations/vacancies are considered to be related to the martensite stabilization [34]. Therefore, from this point of view, the recovery strain along $\langle 111 \rangle$ direction is also larger than that along $\langle 110 \rangle$ direction.

For more details, Figure 8 shows the DSC curves before and after 16% tension along the $\langle 111 \rangle$ and $\langle 110 \rangle$ directions at -60°C . Shown in figure, the A_s and A_f are largely increased after tension. In addition, the reverse transformation temperature of A_s' along the $\langle 111 \rangle$ direction is 69°C , which is lower than 72°C along the $\langle 110 \rangle$ direction, indicating that the reverse transformation occurs more easily along the $\langle 111 \rangle$ direction. The reason for the difference in the transformation temperatures of both conditions presented is related to the martensite stabilization introduced by deform-induced dislocations/vacancies.

Samples with more dislocations tend to highly impede the reverse transformation and make the reverse transformation temperature higher. The result also provides evidence that $\langle 111 \rangle$ direction tends to generate fewer dislocations, thus achieve higher recoverability, as is mentioned above.

Figure 8. Differential scanning calorimetry (DSC) curves (a) before and (b) after tension to 16% at $-60\text{ }^{\circ}\text{C}$ along the $\langle 111 \rangle$ and $\langle 110 \rangle$ direction.

4.2. Effects of $\{111\}\langle 110 \rangle$ Orientation on SME in Pipe Joints

Different from uniaxial tensile sample, as for pipe joints, it should be analyzed in cylindrical coordinate rather than rectangular coordinate. With the inner diameter D_0 expands to D_1 in the cross section, the mechanical strain ε can be divided into a radial compressive strain ε_C and a circumferential tensile strain ε_L , as shown in Figure 9. During the heating process, the recovery of ε_C and ε_L make the inner diameter D_1 decreasing to D_2 , thus realizing the connection of the two pipes. According to previous studies, the SME of pipe joints is mainly determined by circumferential strain ε_L rather than radial strain ε_C [27]. Hence, the recovery strain of pipe joints can be calculated as

$$\bar{\varepsilon}_M = \sum_{i=1}^n \varepsilon_{Lri} I_i \quad (4)$$

Here ε_{Lri} is the recovery strain originating from the circumferential tensile strain ε_L in each orientation.

Figure 9. Schematic of the strain state in the cross section of a pipe joint.

Therefore, in samples of A, B, C with the same ED, the texture types are the same, while the texture intensity is different. In the order of A, B and C, the intensity of $\{111\}\langle 110 \rangle$ texture increases gradually, so the recoverability increases too, which is consistent with the results obtained in Figure 7.

In the same sample with different expansion directions of ED1 and ED2, the texture intensity is fixed, so the $\bar{\varepsilon}_M$ is mainly determined by the ε_{Lri} of the preferred orientation along the circumferential direction of the pipe joints. Taking sample C for an example, the IPF of ND, RD and TD is shown in Figure 10. There is high density preferred orientation in $\langle 111 \rangle$ district of ND IPF and $\langle 110 \rangle$ district

of RD IPF and TD IPF, indicating that the $\langle 111 \rangle$ direction is parallel to the axial direction while the $\langle 110 \rangle$ direction is parallel to the radial and circumferential directions of the rod. Hence, when the pipe joint is expanded along ED1, the preferred orientation is $\langle 110 \rangle$ and this preferred orientation causes higher recovery strain, as is performed in single crystal materials. However, when expanded along ED2, the preferred orientation of circumferential direction in pipe joint is quite complicated, maybe including many orientations such as $\langle 111 \rangle$, $\langle 110 \rangle$ and $\langle 001 \rangle$ with different recovery strains, thus the average recovery strain along ED2 is supposed to be smaller than that along ED1 due to the imbalance of recovery strain in many orientations.

Figure 10. Inverse pole figures (IPFs) of Sample C, representing the preferred crystalline orientations in the axial direction (ND), radial direction (RD) and circumferential direction (TD) of the rod.

In addition, the reason that the recoverability difference between ED1 and ED2 increases on the order of A, B and C can be related to the texture intensity obviously. The stronger texture intensity the rod has, the bigger the recoverability difference is. Hence, strong texture intensity can strengthen the anisotropy of the SME in $\text{Ni}_{47}\text{Ti}_{44}\text{Nb}_9$ rods.

It is worth noting that, along with texture, grain size and grain morphology may also contribute to anisotropic SME. Both slender and small grains are expected to increase the anisotropy of SME. This trend may be explained by grain-boundary strengthening theory. Samples with more grain boundaries tend to own higher martensite yield stress. Thus, when loaded to the same strain, samples with more grain boundaries tend to generate fewer dislocations and exhibit better recoverability. However, it is still hard to know which factor plays a dominant role, since grain size, grain morphology and texture usually change together under deformation, and it is very hard to study a single factor without changing others. Obviously, to further clarify the cause, more work needs to be done.

5. Conclusions

1. The uniaxial tensile recovery strain along the $\langle 111 \rangle$ direction was larger than that along the $\langle 110 \rangle$ direction in NiTiNb rods;
2. For the same texture type of $\{111\}\langle 110 \rangle$ with the same expansion direction, the ε_r and η of the NiTiNb pipe joints increase along with increasing texture intensity. Thus, a strong texture intensity is desired in engineering applications;
3. For the same texture type of $\{111\}\langle 110 \rangle$ with the same texture intensity, the ε_r and η of the pipe joints along ED1 were higher than along ED2, indicating that the recoverability of NiTiNb pipe joints strongly depends on the expansion direction. Thus, the suitable expansion direction should be selected to improve the SME in pipe joints;
4. The recoverability difference between ED1 and ED2 increased along with increasing texture intensity, suggesting that a strong texture intensity further strengthens the anisotropy of the SME in NiTiNb rods.

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