Effects of Grain Size Gradient on Deformation Behavior of Superelastic NiTi Plate

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Abstract. The superelastic NiTi shape memory alloy with grain size distributed from 20 nm to 175 nm along the gauge length is fabricated by cold rolling and gradient heat treatment. This study investigated the effects of grain size gradient on deformation behavior of the NiTi plate under strain controlled quasi-static loading-unloading. Experimental results show that when the grain size decreased from 175 nm to 35 nm, the phase transformation stress increased from 380 MPa to 509 MPa, and the stress slope increased from 0.6GPa to 14.15GPa. With a simple phenomenological model, we have simulated the stress-strain responses and demonstrated the mechanism of the deformation behaviors of the grain size gradient superelastic NiTi plate. The study indicates that the particular behavior of the plate is due to grain size effects of coexisting grain size gradient.

Introduction

For NiTi polycrystalline shape memory alloys (SMAs) with known novel physical, mechanical and biocompatible properties [1, 2], there are different thermomechanical responses between the coarse-grain (CG) and the nano-grain (NG) specimens under the same external loading condition [3, 4]. For example, during tensile loading-unloading, the CG SMAs have significant temperature variation and typical pseudoelasticity with large recoverable strain which caused by matensitic phase transition [5, 6]. It shows great potential to applications for smart recovery, energy-absorbing system, solid refrigeration, etc. [7-9]. However, there are still some problems unsolved due to its poor fatigue life and large hysteresis [10, 11].

Meanwhile researches demonstrate that when the grain size (GS) decreased to nano-scale, series of changes such as better cyclic stability, higher strength and low energy dissipation have taken place compared to CG NiTi [12, 13]. This new phenomenon not only provides opportunities to improve SMAs performance, but also brings unknowns and limitations owing to a few drawbacks, including weakened fracture toughness and smaller recoverable strain [14-16].

Therefore how to develop SMAs to adopt advantages of both CG and NG and avoid their shortcomings has profound significance for practical application. A.S. Mahmud and B.S. Shariat [17-19] provide some experimental ideas that the microstructurally graded NiTi alloy can be created by gradient temperature annealing and aging. Although the gradient structure were not characterized in previous studies, thanks to the effect of gradient annealing, we can build grain size gradient (GSG) SMA material, realizing the graded function in a single specimen, to investigate the relationship of microstructure characterization and GSG effects on deformation behavior.

This paper investigated the GSG effects on the mechanical response of superelastic NiTi plates under quasi-static loading-unloading. The material preparation and experimental methods are described in Section 2. The experimental, modeling and analytical results are described in Section 3. Conclusions are given in Section 4.

Material Preparation and Experimental Setup

The superelastic NiTi SMA used in this study are commercial polycrystalline plates with slightly Ni-rich composition (50.9 at.%Ni-49.1 at.%Ti) purchased from Nitinol Devices Company, USA.
as-received plates with original thickness 1.85 mm and average GS about 100 nm were annealed at 800°C for 1 h and quenched in water. Then a number of sequential cold-rolling passes reduce the thickness gradually to 1.00mm (46% reduction). The dog-bone shaped specimens with gauge length of 27.34 mm, width of 2.00 mm, and thickness of 1.00 mm were wire-cut along the rolling direction from the same plate and then mechanically polished to mirror like surface. To realize GSG structure, gradient annealing was implemented via the heat treatment system as shown in Fig. 1(a). The surface temperature of the heating platform and annealing time were set as 550°C for 5 min. The temperature gradient (550°C~120°C) along gauge length were measured by thermocouples.

![Gradient Annealing Setup](image)

Figure 1. (a) The gradient annealing setup, (b) The TEM bright-field images of the two ends of the GSG specimen.

After heat treatment, the typical microstructures of the three treated specimens were acquired by high resolution transmission electron microscopy (HRTEM-JEOL 2100), as Fig. 1(b) shown. With average GS increased from 20nm to 175nm along the gauge length, the grains and grain boundaries become clearer with deceasing amorphous.

The mechanical responses of the GSG specimens were tested via Instron 5969 quasi-static testing machine at temperature $T_0 = 22\,^\circ C$. Strain control tensile tests were adopted at a nominal strain rate of $4\times10^{-5}\,/s$ to ensure isothermal condition.

**Results and Analysis**

**Experimental Results**

Fig. 2 shows the isothermal $\sigma$-$\varepsilon$ curves of the GSG specimens under quasi-static tensile loading-unloading. It is seen that the phase transformation (PT) still occurred for the GSG specimen as evidenced by the hysteresis loop (H= 6.48 MPa) and large recoverable strain (4.0%). In the initial stage of PT propagation (‘a’ to ‘b’), typical stress plateaus are occurred in CG region. When the nominal strain goes to 2.8% (marked point ‘b’), the stress began to rise as the PT band propagated to the transition region. The PT stress ($\sigma_{PT}$) increased with the decreasing GS, and $\sigma_{PT}$ will follow the principle of GS effect [4]. In the stage from ‘b’ to ‘c’ with slope of 4.82 GPa. In the stage from ‘c’ to ‘d’, the slope increased to 11.83 GPa with the stress increased from 438 MPa to 509 MPa. During the unloading stage from ‘e’ to ‘f’, the GSG specimen also shows difference from CG specimen. It does not have significant PT plateau, but showing a gradual decline with the decreasing strain, even under isothermal condition.
Modeling and Analytical Results

In this subsection, a simple phenomenological model for simulating and interpreting the $\sigma$-$\varepsilon$ curves of GSG NiTi is proposed. As shown in Fig. 3(a), the GSG NiTi plate can be divided into three parts for simplicity: 1) CG part with typical stress plateau and hysteresis; 2) NG part without stress plateau and hysteresis; 3) transitional part between CG and NG parts. We assume that CG part (GS > 110 nm) and NG part (GS < 30 nm) exhibit CG and NG $\sigma$-$\varepsilon$ curves, respectively. While the $\sigma$-$\varepsilon$ curve of the transitional part (TG, 30 nm ≤ GS ≤ 110 nm) is simply represented by the $\sigma$-$\varepsilon$ curve of 50 nm GS NiTi. Therefore, the mechanical response of GSG NiTi plate can be seen as the combination of all the three parts.

In order to explain the GSG effects, the $\sigma$-$\varepsilon$ curves of the three parts of the plate are simplified and presented in Fig. 3(a). As the experimental results indicate, $\sigma_{PT} = 390$ MPa, and PT strain $\varepsilon_{PT} = 0.056$; $E_\varepsilon = 56$ GPa and $E_{PT}^{TG} = 7$ GPa [4] are elastic modulus and phase transformation stiffness of

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TG part, respectively, and the phase transformation stiffness of CG part is simplified as zero; $\sigma_{H}^{TG} = 130$ MPa and $\sigma_{H}^{CG} = 222$ MPa are stress hysteresis of TG and CG parts, respectively.

Under an external loading stress, the current stiffness/modulus of the GSG plate can be calculated by

$$E^{GSG} = \frac{1}{\eta_{NG} + \eta_{CG} + \eta_{TG}}$$

where $\eta_{NG}$, $\eta_{CG}$ and $\eta_{TG}$ are the volume fraction of the three parts. The linear gradient is assumed and the maximum and minimum grain sizes are 175 nm and 20 nm, respectively. Then we can obtain $\eta_{NG} = 6.5\%$, $\eta_{CG} = 41.9\%$ and $\eta_{TG} = 51.6\%$.

By using the simplified $\sigma$–$\varepsilon$ curves of the three parts in Fig. 3(a), the variation of stiffness/modulus and strain under quasi-static tensile loading-unloading can be calculated by

\begin{align*}
E &= \begin{cases} 
  \sigma_{max} - \sigma_{H}^{TG} < \varepsilon \leq \sigma_{max} \\
  \eta_{NG} + \eta_{CG} + \eta_{TG} \\
  0 \leq \varepsilon < \sigma_{max} - \sigma_{H}^{TG} \\
  \varepsilon = \frac{\sigma - \varepsilon}{\sigma_{max} - \sigma_{H}^{TG}} \\
\end{cases} \\
\sigma = \sigma_{max} - \sigma_{H}^{TG} < \varepsilon \leq \sigma_{max} \\
\varepsilon = \frac{\sigma - \varepsilon}{\sigma_{max} - \sigma_{H}^{TG}} \\
\sigma_{H}^{CG} = \eta_{CG} \varepsilon \\
\sigma_{H}^{CG} = \eta_{CG} \varepsilon \\
\sigma_{max} = 500$ MPa. Fig. 3(b) shows the comparison of the $\sigma$–$\varepsilon$ curves between the experimental and theoretical results (Eqs. 2-3). It is shown that the theoretical result agrees with the experimental data.

The theoretical result indicates that the phase transformation takes place in CG part once the tensile loading stress reaches $\sigma_{pt} = 390$ MPa ($\varepsilon = \varepsilon_{A}^{end}$), resulting to typical stress plateau of CG NiTi. When the phase transformation is completed in CG part ($\varepsilon = \varepsilon_{B}^{end}$), the phase transformation starts in TG part as the stress increases. Since the phase transformation stiffness of TG part is nonzero, the phase transformation in TG part leads to overall rise of the stress and stress slope ($\varepsilon = \varepsilon_{B}^{end} \sim \varepsilon_{C}^{end}$). During the stress unloading ($\varepsilon = \varepsilon_{C}^{end} \sim \varepsilon_{H}^{end}$) from $\sigma_{max} = 500$ MPa, the $\sigma$–$\varepsilon$ curve also exhibits combined behavior of the three parts of the GSG NiTi plate.
Conclusion

The superelastic NiTi with grain size distributed from 20 nm to 175 nm along the gauge length is fabricated by cold rolling and gradient annealing. We have investigated the effects of grain size gradient on the mechanical responses ($\sigma-\varepsilon$ relation) under quasi-static loading-unloading. Based on the phenomenological model, we have shown the internal mechanism of the phase transformation during loading, and drawn the following conclusions: The GS distribution does affect the deformation behavior of GSG specimen, resulting in some unique characteristics during the quasi-static loading. The rise of the stress (380 MPa-509 MPa) and the stress slope (0.6 GPa-14.15 GPa) in loading are due to the grain size dependent phase transformation stress. The theoretical analysis indicates that the particular behavior of GSG SMA plate is due to grain size effects of coexisting grain size gradient.

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