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Thermal processing of polycrystalline NiTi shape memory alloys

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Abstract

The objective of this study is to examine the effect of heat treatment on polycrystalline Ti–50.9 at.% Ni in hot-rolled and cold-drawn states. In particular, we examine microstructure, transformation temperatures as well as mechanical behavior in terms of both uniaxial monotonic testing and instrumented Vickers micro-indentation. The results constitute a fundamental understanding of the effect of heat treatment on thermal/stress-induced martensite and resistance to plastic flow in NiTi, all of which are critical for optimizing the mechanical properties. The high temperature of the hot-rolling process caused recrystallization, recovery, and hindered precipitate formation, essentially solutionizing the NiTi. The subsequent cold-drawing-induced a high density of dislocations and martensite. Heat treatments were carried out on hot-rolled, as well as, hot-rolled then cold-drawn materials at various temperatures for 1.5 h. Transmission Electron Microscopy observations revealed that Ti_3Ni_4 precipitates progressively increased in size and changed their interface with the matrix from being coherent to incoherent with increasing heat treatment temperature. Accompanying the changes in precipitate size and interface coherency, transformation temperatures were observed to systematically shift, leading to the occurrence of the R-phase and multiple-stage transformations. Room temperature stress–strain tests illustrated a variety of mechanical responses for the various heat treatments, from pseudoelasticity to shape memory. The changes in stress–strain behavior are interpreted in terms of shifts in the primary martensite transformation temperatures, rather then the occurrence of the R-phase transformation. The results confirm that Ti_3Ni_4 precipitates can be used to elicit a desired isothermal stress–strain behavior in polycrystalline NiTi. Instrumented micro-indention tests revealed that Martens (Universal) Hardness values are more dependent on the resistance to dislocation motion than measured uniaxial pseudoelastic or shape memory response. Based on comparison of hardness and the stress required to induce martensite, it is shown that the resistance to dislocation motion and the ease of the stress-induced martensite transformation cannot be simultaneously maximized, although an optimal combination should exist. Measuring indentation depth before and after heating more distinctly confirmed shape memory or pseudoelastic behavior.

Keywords: NiTi; Shape memory alloy; Martensite; Transformation temperatures; Vickers indentation; Deformation processing

1. Introduction

Shape memory alloys, such as nickel–titanium (NiTi), demonstrate a unique ability to recover their initial shape after deformation through a reversible thermo-elastic phase transformation. Specifically, NiTi can restructure itself from a B2 austenite phase (A), to a B19′ martensite phase (M) through a decrease in temperature or an increase in applied stress. It is this solid-state phase transformation that allows NiTi to recover large strains, either spontaneously (pseudoelasticity) or through an increase in temperature (shape memory effect). In addition to strain recovery, NiTi is attractive for several medical applications [1–7] due to its biocompatibility [2,4], corrosion resistance [8,9], and fatigue behavior [2,10–17]. Additionally, NiTi is presently being considered for applications in the area of seismic resistance design and retrofit [18–22]. The enhanced damping of NiTi acts to absorb seismic energy, a significant improvement over current construction materials [18–22].

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Thermal processing of NiTi is frequently used to optimize the mechanical properties of NiTi applications. However, thermal processing is often performed without full understanding of the changes in microstructure or impact on all relevant mechanical properties. Much of the prior work has concentrated exclusively on the link between microstructure and the thermally induced phase transformation [23–31], without examining the effect of different heat treatments on mechanical behavior, even though the microstructure and resulting thermo-mechanical properties are quite sensitive to heat treatment. The present study aims to develop a more comprehensive and fundamental link between structural and various behavioral changes in NiTi in response to heat treatment.

For slightly nickel-rich NiTi, such as the Ti–50.9 at.% Ni used in this study, aging treatments cause the formation of Ti_3Ni_4 precipitates, known to influence transformation temperatures [23,25–28,32–35]. Affected by the coherency and size of the precipitates, internal stresses and depletion of Ni content in the matrix shift the transformation temperatures [24,26,33]. Furthermore, the internal stresses caused by aging typically facilitate the occurrence of the rhombohedral R-phase (R) [32]. Essentially, precipitates and dislocations create an energy barrier to martensite formation, making the formation of the R-phase a low energy alternative [32]. Interestingly, the R-phase transformation has been shown via in situ transmission electron microscopy (TEM) to nucleate near Ti_3Ni_4 precipitates [23,25]. It has also been shown in the literature that more complex, multiple-stage transformation behavior may develop after aging [23,25,28,32–35]. Several explanations as to the driving mechanism for the multiple-stage transformation have been proposed, however, the most widely accepted view is that the precipitates are distributed inhomogeneously [25–27], causing a localized phase transformation separate from the remainder of the matrix [25,27]. Because the transformation does not propagate throughout the entire material, but rather occurs at distinct spatial locations, multiple transformation peaks are measured [25,27].

The internal stress caused by Ti_3Ni_4 precipitates also has an effect on stress–strain behavior [26,36–38]. It has been qualitatively shown that coherent Ti_3Ni_4 precipitates increase the stress needed for plastic flow as well as decrease the martensite transformation stress [36]. Essentially the local stress fields caused by coherent precipitates act in addition to an applied stress, and become the location of the phase transformation [36]. While this effect of Ti_3Ni_4 precipitates on the stress–strain behavior has been demonstrated for single crystal NiTi, the effect in deformation processed polycrystalline NiTi has not been examined in detail.

In addition to the effects of precipitates, crystallographic orientation has been shown to have a profound effect on stress–strain behavior of NiTi [11–13,36,39–45]. Because the stress-induced martensitic transformation is a directional shear-like phenomenon, it is often described in a manner analogous to Schmid's law [13,36,45]. Therefore, the resolved shear stress necessary to induce formation of a martensite plate is dependent on both the crystallographic orientation and the direction of the applied stress. For example, a compressive load applied to a NiTi [1 1 1] single crystal will have a higher transformation stress than in a [1 0 0] single crystal [36]. Also, because the martensite plate formation is less favorably oriented in the [1 1 1] direction, it will have a smaller transformation strain [36].

Crystallographic texture is common in deformation processed NiTi [46–50]. This is of particular importance because deformation processing is carried out during fabrication for most polycrystalline NiTi materials used in civil and biomedical applications. Both hot working and cold working have been shown to produce a strong $\langle 1\,1\,1\rangle$ and/or $\langle 1\,1\,0\rangle$ texture [46–50]. Along with inducing texture, deformation processing typically increases the dislocation density. Previous studies have shown that dislocations created during these processes act to inhibit martensite transformation by hindering the reorientation process [51–54]. As a consequence, austenite and martensite transformation temperatures decrease [55], and thus the critical stress needed to induce martensite transformation increases [56]. Conversely, processing performed at above 600 °C causes recrystallization [30,31,53], as well as, dislocation annihilation.

Although it is important to understand how precipitation, texture, and deformation processing affect the transformation behavior of NiTi, it is ultimately necessary to quantify mechanical behavior in order to facilitate the design process. In addition to conventional stress–strain testing, instrumented micro-/nano-indentation has been used to determine mechanical properties due to its simplicity and a relatively non-destructive nature. For NiTi, recent emphasis has been placed on identifying shape memory and/or pseudoelastic behavior through indentation [58–62]. Using spherical indentation, both shape memory [59–61] and pseudoelastic [58–62] NiTi have shown full transformation strain recovery. In fact, it has been possible to illustrate the transition from pseudoelastic to plastic deformation by increasing indentation load [58–62]. Given that Vickers indenters induce approximately 30% strain at the tip of indention [63], the phase transformation strain is overshadowed by plastic deformation, and only partial recovery is possible [59–62,64,65]. While texture, deformation processing and aging have pronounced effects on stress–strain properties of NiTi, they have only a minor influence on sharp indentation force–displacement curves for single crystal NiTi [11,66]. Indentation tests alone may be adequate for discerning between pseudoelasticty or shape memory behavior; however, no study has correlated specific stress–strain values (i.e. transformation stress, transformation strain or stress hysteresis) to indentation results. In particular, a definitive relationship between indentation response and material heat treatment in NiTi does not exist. While recent indentation studies clearly demonstrate its importance as a new technique, in order to gain meaningful information about NiTi from indentation, the results must be presented in light of different microstructures and macroscopic stress–strain responses.