



Acta Materialia 58 (2010) 3357-3366



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EBSD studies of the stress-induced B2–B19' martensitic transformation in NiTi tubes under uniaxial tension and compression

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Received 26 August 2009; received in revised form 8 February 2010; accepted 8 February 2010 Available online 10 March 2010

Abstract

In situ electron backscattering diffraction (EBSD) investigations were conducted on polycrystalline NiTi tube specimens during tensile and compressive deformation. The long-range cooperative and catalytic martensitic transformation under tension induces the transformation to proceed in the form of helical Lüders band. Propagation of the band is closely related to the spatial distribution of the orientations of individual grains. In uniaxial compression, the larger variation in Schmid factors, and consequently the larger variation in the critical transformation stresses among grains, leads to a homogeneous martensitic transformation, and therefore the absence of the Lüders band. To interpret the observed tension–compression asymmetry, a crystallographic model of the critical transformation stress and transformation strain for polycrystalline NiTi under tension and compression is proposed. The model defines three crystallographic regions: tension-favorable, compression-favorable and neutral zones. The orientation population in which tensile strains are larger than compressive strains is much higher than that of orientations with higher compressive strains. For resolved shear stress, orientation populations favoring tension and compression do not show any great difference.

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Keywords: Shape memory alloys (SMA); Martensitic phase transformation; Electron backscattering diffraction (EBSD); In situ

1. Introduction

Near-equiatomic NiTi shape memory alloys (SMAs) are well known for their superelasticity, shape memory effect (SME) and good biocompatibility. The diffusionless and reversible transformation from a high-temperature austenitic phase (B2 structure) to a low-temperature martensitic phase (B19' structure) gives rise to the superelasticity and SME. Studies of the tensile and compressive mechanical tests on single crystal and polycrystalline NiTi alloys revealed asymmetric transformation behaviors [1]. For most polycrystalline NiTi alloys and NiTi single crystals, tensile testing demonstrates lower transformation stresses

and higher transformation strains compared to compression. These observations indicate that it is easier to induce martensite in tension than in compression. This has also been verified in bending, where more martensite is formed on the tensile side than on the compressive side [2]. However, questions remain of whether tension is always a favored stress state to induce martensitic transformation in polycrystalline NiTi alloys, and, if so, what the reasons are. In the meantime, investigations into the formation of a macroscopic Lüders deformation band (LDB) in tensile tests on NiTi sheet specimens have stimulated some debate about the origin of these bands. Shaw and Kyriakides attributed the mechanical instability and the upper-lower yielding phenomenon of the LDB behavior to the difference in thermodynamic driving force between martensite nucleation and variant growth, in analogy to solidification [3–5]. Liu disputed Shaw and Kyriakides' hypothesis and

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argued, based on experimental observations of the occurrence of the LDB during deformation via martensite reorientation, where no transformation is involved, and the absence of the LDB during the stress-induced martensitic (SIM) transformation in compression, where nucleation does occur, that the behavior is more of a mechanical nature [6]. More detailed studies further demonstrated that the shear angle of the LDB can vary from 48° to 61° [7,8] in NiTi strip specimens, implying that the formation of LDB is not purely governed by the mechanical principle of maximum shear stress, but is possibly a result of the interaction between the mechanics and the martensite crystallography. Based on the minimum energy requirement, Sun determined the shear angle to be a constant value of 55.7°, which cannot describe the experimentally observed variations of the band angles [9]. Electron backscattering diffraction (EBSD) has proven to be a useful technique for studying the crystallographic structure of polycrystalline NiTi alloys [10]. Recent studies on NiTi alloys [11–13] by means of in situ EBSD have allowed the localized and global crystallographic martensitic transformation characteristics to be determined in bulk NiTi specimens with high spatial resolution. It was revealed that, in NiTi sheet specimens, the selection of SIM variants and propagation of the transformation front with respect to the external load axis were controlled by the orientation and distribution of individual grain. In other words, LDBs in NiTi SMAs can be described as a crystallographically correlated phenomenon [11–14]. With the above questions and uncertainties in mind, this study was conducted with three objectives: (i) to establish an interpretation of the observed helical LDBs in NiTi tube specimens induced by tensile deformation by means of in situ EBSD analysis; (ii) to establish an explanation for the absence of LDBs in compression; and (iii) to establish a correlation between grain orientation and stress state in crystallographic models. This investigation thus provides, for NiTi tubes, which are widely used for cardiovascular stents, a basic picture of the macroscopic mechanical responses associated with nucleation and propagation of SIM transformation in tension and compression. Our study also provides a compreexplanation of the tension-compression asymmetric mechanical behavior of NiTi polycrystalline materials in addition to the previously published literature [15–17]. The findings are helpful for the design and fabrication of NiTi tubes and stents with optimum microstructures and mechanics.

2. Materials and methods

A Ti-50.8 at.% Ni tube of 2.5 mm inner diameter and 3 mm outer diameter was supplied by Memry Co., USA. Tensile specimens 80 mm in length and compressive specimens 9 mm in length were cut using the spark erosion method. To allow effective EBSD analysis, specimens were annealed at 800 °C for 30 min to encourage grain growth and then quenched in water. Specimens for in situ tensile

EBSD investigation were cut from the tube by means of spark erosion into plates 3 mm wide and 10 mm long. Specimens for in situ compressive EBSD analysis were the same as for the compression testing. The surface of the specimens was mechanically polished to remove the oxide layer and then electropolished in a solution of 20% H₂SO₄ and 80% methanol at room temperature at 30 V. EBSD studies were conducted using a JEOL 6500 field emission scanning electron microscope with an accelerating voltage of 30 kV. Crystallographic orientations were obtained by indexing of the Kikuchi patterns from each grain with commercial TSL-EDAX software. In situ deformation in tension and compression was achieved using a self-designed system, which is able to hold the specimen at a constant strain for EBSD analysis. All mechanical deformations were conducted at room temperature and at strain rates below 10⁻³ s⁻¹. The transformation behavior of the heat-treated material was characterized by means of differential scanning calorimetry (DSC), and the critical temperatures of the B2-B19' martensitic transformation were determined to be $M_s = -22$ °C, $M_f = -30$ °C, $A_s = -10$ °C and A_f =-1 °C. Thus at the room temperature, the tube specimen is in the B2 structure.

3. Results and discussion

3.1. In situ EBSD investigation of stress-induced martensite transformation in NiTi tube specimens: tensile tests

Fig. 1 shows stress–strain curves of three tube specimens deformed along the axial direction. Samples (I) and (II) were tested in tension and sample (III) was deformed in compression. It can be seen that the material exhibited strong tension–compression asymmetry. The critical normal stresses for inducing the martensitic transformation are measured to be $\sigma_t = 297$ MPa in tension (sample I) and $\sigma_c = 417$ MPa in compression (sample III), with $\sigma_c/\sigma_t = 1.4$. Sample I exhibited a typical Lüders-type deformation over a stress plateau, with a stress window of $\Delta \sigma_t = 32$ MPa, whereas sample III exhibited a typical uni-

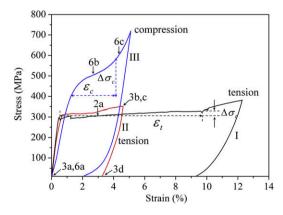


Fig. 1. Tensile and compressive stress–strain curves of specimens annealed at $800 \,^{\circ}\text{C}$ for $30 \, \text{min}$. The tests were carried out at room temperature (22 $^{\circ}\text{C}$) at a strain rate of $10^{-3} \, \text{s}^{-1}$.

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