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Characteristics of the stress-induced formation of R-phase in ultrafine-grained NiTi shape memory wire

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ABSTRACT

The transformation between the cubic B2 and monoclinic B19′ phases in ultrafine-grained pseudoelastic NiTi can occur as a two-step process involving the intermediate rhombohedral R-phase. Experimental work using differential scanning calorimetry, electrical resistance measurements and transmission electron microscopy has demonstrated the formation of this intermediate phase during thermal cycling and during mechanical loading. In the present paper, complementary mechanical and thermographic results are presented which allow to further assess the character of the stress-induced R-phase formation. The transformation from B2 to R-phase is demonstrated to occur homogeneously within the gauge length rather than via advancing Lüders-type transition regions as it is the case in the localized transformation from B2 or R-phase to B19′.

1. Introduction

Near-equiatomic binary NiTi shape memory alloys are of great technological interest because of their good structural and functional properties [1,2] and their range of possible biomedical and engineering applications [3,4]. Commercial NiTi wires and tubes typically exhibit ultrafine-grained microstructures which provide high resistance to plastic deformation, thus favoring the reversible martensitic transformation necessary for pseudoelasticity. The transformation from the high-temperature B2 cubic "austenite" phase [2,5] to the low-temperature monoclinic B19′ "martensite" phase [6,7] may occur via an intermediate rhombohedral structure referred to as "R-phase" [8]. The lattice correspondences between these three phases have been established in [9–12].

Differential scanning calorimetry (DSC) and electrical resistance measurements reported in [13] using both partial and full thermal cycles support the view that the transformation between B2 and B19′ in commercial pseudoelastic NiTi wires (nominal composition 50.9 at.% Ni) on heating and cooling without an applied load does occur via an intermediate transformation to R-phase. Transmission electron microscopy (TEM) using in-situ cooling confirmed load-free thermally induced formation of R-phase at −20 °C by the appearance of diffraction reflections corresponding to its crystal structure [13].

The stress-induced formation of the R-phase at constant temperature has been investigated in a NiTiFe alloy [14] and in binary NiTi [15]. In both systems, two-stage yielding phenomena associated with the separate R-phase and B19′ martensite transitions were observed. Optical microscopic observations have demonstrated that the R-phase exhibits all features typical of a martensitic microstructure: It has four variants that accommodate one another, it shows the one way shape memory effect and on application of a mechanical stress, the favorably oriented variants grow at the expense of the others [16]. During TEM in-situ straining experiments performed in Ni rich 50.9 wt% NiTi pseudoelastic wires, the formation of B19′ was observed but some of the emerging diffraction reflections could only be accounted for by considering the additional presence of the R-phase [13]. This is direct evidence of stress-induced R-phase formation. Šittner et al. used in-situ neutron diffraction and ultrasonic experiments to investigate the transformation between B2 and R-phase and the reorientation of R-phase during loading [17]. They concluded that these methods and also variations in electrical resistance are more sensitive than mechanical methods for the characterization of transformations which involve the R-phase [18].

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The stress-induced transformation to B19′ in NiTi ribbons and wires is well known to occur in a localized (Lüders type) manner [19–27]. Typically, narrow transformation fronts emerge from the grips and move into the gauge length on loading. In displacement controlled tests, these fronts propagate at constant stress until the whole gauge section of the specimen is covered by the transformation, involving a maximum strain of up to 8%, and the situation reverts back on unloading [19–27]. The stress–strain evolution of such a test exhibits characteristic features that are related to the localized type of transformation [19]: (i) a plateau-like constant stress interval is observed in the stress–strain path, both during loading and unloading, for the time period in which the fronts move through the gauge length, and (ii) different sections of the gauge length start to transform (and thus elongate) at distinct points of time. The latter was demonstrated by applying several extensometers during the mechanical test and comparing their reactions on a time scale [19]. A similar analysis of the stress-induced formation of R-phase in a two-extensometer test setup showed that strains of this transformation step were simultaneously evolving in all regions of a pseudoelastic NiTi wire specimen [13], suggesting a homogeneous type of transformation.

Strain measurement methods often overlook detecting the R-phase transformation, as the transformation strains associated to the B2 to R-phase transition are much smaller than those of the B2 to B19′ transformation [15]. Therefore, infrared imaging was used as a complementary method in [13] to prove that non-linearity in the early stress–strain behavior results from transformation events. It was already mentioned there that the obtained temperature profiles suggested homogeneous transformation, and this conclusion was in line with the result of the two-extensometer strain measurements. However, the thermographic analysis in [13] did not cover the whole specimen length and additional experimental verification seemed necessary. The present work aims to further clarify the character of the R-phase transformation by combining mechanical findings with results of an actual thermal imaging analysis.

2. Experimental

Pseudoelastic NiTi wires with a nominal chemical composition of 50.9 at.% Ni were obtained from Memory Metalle, Weil am Rhein, Germany. In the factory, they were hot extruded at 650 °C and wire drawn with 45–55% cold work in the last step. Then, a straight annealing treatment (60 s at 520 °C under a small tensile load) was performed to induce an ultra-fine grained microstructure with optimum pseudoelastic properties. Surface oxides formed during processing were removed by chemical etching. The final wire diameter was 1.2 mm. Further information on the material can be found in [21].

An Instron 5567 electromechanical test machine equipped with a temperature chamber was used for the mechanical characterization of the global pseudoelastic behavior tests reported in this study. Wire specimens with a free gauge length of 40 mm were directly gripped. These tests were carried out at a crosshead displacement rate of 1.67×10^{-3} mm/s (0.1 mm/min) to minimize the effect of the latent heat of transformation. A MTS 632.13F-20 extensometer with 10 mm initial gauge length was attached to the central section of the wire gauge length.

In order to gain further insight into the localized or homogeneous nature of R-phase formation, the surface temperatures of the wire specimens were measured during loading and unloading in a second series of experiments. These experiments were performed in a MTS Bionix 858 servo hydraulic machine. A VarioTHERM infrared imaging system from Infratec, Dresden, Germany was used. The thermal and spatial resolutions of this equipment are better than 0.1 K and 0.2 mm, respectively. Details of the thermal imaging technique were published elsewhere [21,23]. A wire specimen of 35 mm free gauge length was pre-deformed to 0.2 mm. Next, it was elongated from 0.2 mm to 0.354 mm (nominal homogeneous strain: 0.006 to 0.010) at 0.12 mm/s (7 mm/min) crosshead displacement rate, then held for 150 s for thermal equilibrium to be reached. This was followed by unloading to allow contraction of the sample from 0.354 mm to 0.2 mm elongation at similar crosshead displacement rate. After that, the deformation was again kept constant for 150 s to equilibrate temperature with the environment. This cycle was repeated 10 times between the same strain levels of 0.006 and 0.010. Each individual loading or unloading step was completed within 1.32 s and infrared imaging was performed at a frame rate of 1 Hz.

3. Results and discussion

3.1. Mechanically-induced transformations

For the material considered here, stress–strain curves in the range of temperatures between −150 °C and 100 °C have been obtained in a previous study [13] for the assessment of the temperature dependence of stress–strain behavior. At intermediate temperatures, a significant deviation from the initial stress–strain linearity was observed. It was suggested that this effect could be attributed to the stress-induced formation of R-phase. This deviation from linearity is examined more thoroughly in the present work. Fig. 1 illustrates the non-linear behavior at a temperature of 28 °C. The bold line in the figure represents a first cycle in which the wire specimen was only deformed up to the onset of the B19′ transformation plateau and then unloaded. With a sufficiently high resolution (note the expanded strain axis at low strain values), the deviation from linearity and the hysteresis between the loading and unloading branches of the cycle can be clearly appreciated. On complete unloading, no permanent strain is detected, and a second loading (broken line) follows the original load path until the stress level for B19′ formation is reached. In this second cycle, the sample was taken to complete the transformation to B19′. The cycle now exhibits the wide plateaus and hysteresis characteristics of this transformation.

Fig. 2 illustrates how this data can be further analyzed. The first loading/unloading cycle from Fig. 1 is re-plotted here as a solid line and denoted as the original cycle (note the further expanded strain scale compared to Fig. 1). Now, a linear fit to the early deformation range, where the specimen exhibits a basically linear (elastic) behavior, is carried out. Anticipating that this fitted line represents the elastic behavior of the material that could be expected if no phase transformation would occur, the strain deviation of the loading/unloading cycle from this line towards higher stresses can now be determined. Plotting only the strain contribution attributed to R-phase without considering the elastic contribution, the cycle shown in the left part of the diagram is obtained. This stress–strain cycle is characterized by a defined onset of transformation close to 250 MPa and a maximum strain of nearly 0.0035.

An analysis of additional test results in [13] has demonstrated similar reversible non-linear behaviors in temperature range between 28 °C and 42 °C. The non-linearities exhibited a temperature

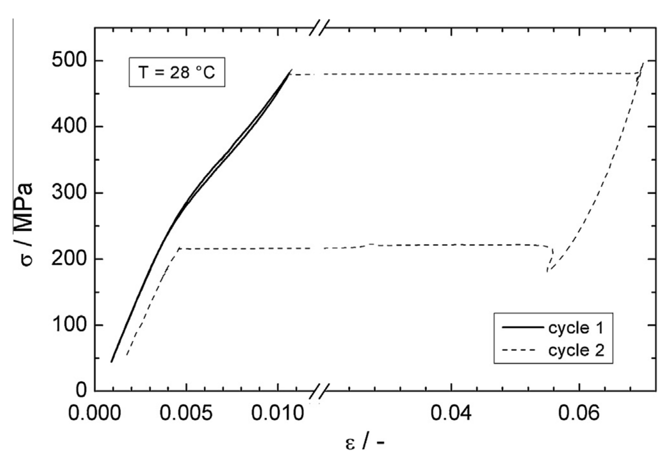

Fig. 1. Two consecutive loading/unloading cycles performed at 28 °C. In the first cycle, the sample is loaded up to the onset of the transformation plateau, and then unloaded. The deviation from linearity and hysteresis in the 275 MPa to 475 MPa stress region is attributed to the transformation to R-phase. The second cycle represents complete transformation to B19′. Note that the strain axis is expanded at low stresses for clarity.