

## A new type of intrinsic two-way shape-memory effect in hooks of NiTi-wires

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**Abstract.** Shape-memory hooks were fabricated in a single processing step ("one-time procedure") by a combination of tensile and bending deformation of a martensitic NiTi-wire. The hooks reversibly changed curvature on heating and cooling in a reproducible manner over 50 thermal cycles. This new type of intrinsic two-way shape memory effect was studied by subjecting the hooks to a beam of high-energy synchrotron x-rays at room temperature ( $<M_f$ ) and at 150°C ( $>A_f$ ). This technique demonstrated that the hooks were martensitic at room temperature but contained both austenite and martensite at 150°C. The presence of martensite above  $A_f$  is due to a high dislocation density, which stabilizes martensite and inhibits its transformation. The amount of stabilized martensite scales with the extent of plastic deformation and thus increases from the inner to the outer curvature of the hook. On heating, the inner part of the hook transforms to austenite, while the stabilized martensite in the outer part is deformed pseudo-elastically by twinning. On cooling, the back-transformation of the inner part is biased by the pseudo-elastic recovery of the outer part, thus forcing the hook back into its initial shape.

### 1. INTRODUCTION

The intrinsic two-way effect (TWE) is usually introduced into shape memory alloys (SMAs) either by cyclic thermo-mechanical training or by providing sufficient plastic deformation in one single loading step. The stress fields of dislocations introduced during the plastic deformation favor the formation of specific martensite variants on cooling, and this results in the macroscopic shape change associated with the TWE. Sufficient amounts of plastic deformation in a martensitic SMA can induce twinning pseudo-elasticity (or linear superelasticity), which is characterized by a near-linear stress-strain curve over large strains [1]. Deformation occurs by reversible twinning, which is made possible by the interaction of dislocations produced during prior plastic deformation with the martensitic variants. The presence of dislocations also shifts the martensite to austenite transformation temperature to higher values. We investigate here a new type of intrinsic TWE induced by plastic bending of a SMA wire into a hook, which leads to a gradient of dislocation density and thus a gradient from shape-memory to superelastic properties.

### 2. EXPERIMENTAL PROCEDURES

NiTi wires with near-equiatomic composition (Ti-49.7 at.% Ni) and a diameter of 0.8 mm were procured from Memory-Metalle (Germany) in a "straight-anneal" condition (600°C anneal). In the as-received condition, the wires are characterized by martensite start and finish temperatures of  $M_s \approx 67^\circ\text{C}$  and  $M_f \approx 54^\circ\text{C}$  on cooling, and austenite start and finish temperatures of  $A_s \approx 93^\circ\text{C}$  and  $A_f \approx 116^\circ\text{C}$  on heating. Hooks were produced from the wires in a tensile test apparatus at room temperature (Figure 1). The wire was fixed at one end and bent 180° around a 6.4mm diameter pin by applying a static tensile load of  $P=29, 49, 98$  or 147 N to the other extremity of the wire. As the pin was moved upwards by the cross-

head, the wire was bent into a hook. After cutting the wire and repeating the operation to produce multiple hooks (ca. 50 mm long), a short heat treatment (200°C for 1 min) was carried out to ensure that no “normal”, unstabilized martensite was present. The hooks were then fixed at one end and heated and cooled between room temperature and 150°C with an isothermal hold of 5 min., the TWE was characterized in terms of the evolution of the angle  $\varphi_{TWE}$  ( $\varphi_{heating}$ ,  $\varphi_{cooling}$ ), as shown in Figure 1.

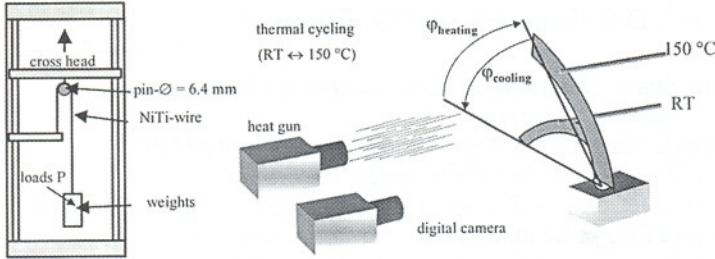


Figure 1. Experimental set-up for shaping the hooks (left) and for assessing the TWE (right).

Also, synchrotron X-ray diffraction was used to identify the phases present in the hooks at room temperature and at 150°C in the first thermal cycle. A monochromatic, parallel 65 keV beam was used with exposure times of 5 or 20 min. The experimental set-up is schematically illustrated in Figure 2 and has also been described in Ref. [2] for in-situ tensile experiments.

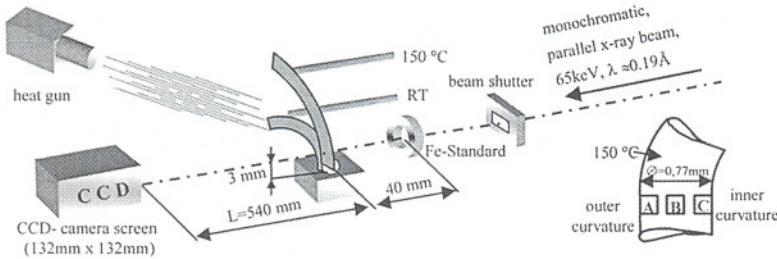


Figure 2. Schematic illustration of synchrotron diffraction experiments. The figure shows the geometry of the synchrotron experiment and also illustrates three local positions in the hook A, B and C.

Two types of diffraction experiments were performed. In a first coarse assessment, diffraction was performed over the whole cross-section of the hook with an exposure time of 5 min., using beam dimensions of  $2 \times 1 \text{ mm}^2$  encompassing the whole hook diameter (including locations A, B and C in Figure 2). Second, a shutter reduces the beam area to  $0.2 \times 0.2 \text{ mm}^2$ , thus exposing for 20 min. a smaller volume at positions A, B or C in the hook, as shown in Figure 2. Diffraction patterns were recorded on a plane normal to the incident beam using a CCD camera placed at a distance  $L = 540$  mm from the sample (accessible Bragg angle range:  $\theta \leq 3.48^\circ$ ), using reference iron powder for correction. From the Debye-Scherrer rings of diameter  $D$ , the Bragg angle  $\theta$  is obtained by geometry (Figure 2) as:

$$\theta = \frac{1}{2} \arctan\left(\frac{D}{2L}\right) \quad (1)$$

and the corresponding d-spacings are:

$$d_{hkl} = \frac{\lambda}{2 \sin\left(\frac{1}{2} \arctan\left(\frac{D}{2L}\right)\right)} \approx \frac{2\lambda L}{D} \quad (2)$$

Where  $\lambda$  represents the x-ray wavelength and the approximation in Equation 2 is valid for small Bragg angles ( $D/2L \ll 1$ ). The d-spacings of a monoclinic lattice are given by [3]:

$$\frac{1}{d_{hkl}^2} = \frac{1}{\sin^2 \beta} \left( \frac{h^2}{a^2} + \frac{k^2 \sin^2 \beta}{b^2} + \frac{l^2}{c^2} - \frac{2hl \cos \beta}{ac} \right) \quad (3)$$

With martensitic B19' NiTi lattice parameters as  $a=0.2898\text{nm}$ ,  $b=0.4108\text{nm}$ ,  $c=0.4646\text{nm}$  and  $\beta=97.78^\circ$  [4]. The d-spacings of the cubic lattice are given by [3]

$$\frac{1}{d_{hkl}^2} = \frac{h^2 + k^2 + l^2}{a^2} \quad (4)$$

with the austenitic B2 NiTi lattice parameter as  $a = 0.2998 \text{ nm}$  [5].

### 3. RESULTS AND DISCUSSION

Table 1 lists the angular TWE characteristics of hooks produced with different loads P.

**Table 1.** Effect of bending load P (Figure 1 – left) on angular TWE characteristics (Figure 1 - right).

| P [N] | $\Phi_{\text{heating}}$ for 1 <sup>st</sup> thermal cycle [°] | $\Phi_{\text{heating}}$ for 50 <sup>th</sup> thermal cycle [°] |
|-------|---|--|
| 29    | 46.0  | 36.5   |
| 49    | 35.2  | 31.4   |
| 98    | 12.2  | 17.0   |
| 147   | 0   | 0  |

It can be seen from the data shown in Table 1 that an increase in bending load results in a decrease in the TWEs. Table 1 also shows that, upon thermal cycling, the magnitude of the TWE decreases somewhat for the two smaller loads and increases for the higher load  $P=98\text{N}$ .

For all room temperature diffraction experiments, only B19' martensite was detected, as expected. However, for all experiments at  $150^\circ\text{C}$ , a mixture of martensite and austenite was detected; as shown in Figure 3, where the flux-normalized and volume-corrected intensity (for details see Ref. [6]) is plotted as a function of the d-spacing in the range of  $1.95\text{-}2.40\text{\AA}$  (highlighting the A(110) austenite peak). Figure 3 (left) shows the volume-averaged information, which is obtained for all hooks listed in Table 1. It can be seen that the amount of stabilized martensite increases as the bending load increases. Figure 3 (right) shows results obtained for a single hook deformed with  $P=49\text{N}$ ; the results show that phase compositions vary spatially: the amount of austenite increases (respectively the amount of martensite decrease) when moving from A (outer hook curvature – Fig. 2) to C (inner hook curvature – Fig. 2). Both the global results (Fig. 3 - left) and the spatially-resolved results (Fig. 3 - right) can be rationalized if dislocations (which are introduced by plastic deformation during the bending step) increasingly stabilize the martensitic phase. This is why the global diffraction results show decreasing amounts of austenite when P increases. And this is also the reason why more martensite is observed at position A, where the superposition of bending (forcing the wire around the pin – Fig. 1) and pulling (tensile load P – Fig. 1) results in higher total accumulated strains than at position C, where bending and tensile stress have opposite sign and partially cancel each other. The tension/compression asymmetry in NiTi mechanical behavior may also be important, but is not discussed here. The resulting gradient in plastic strain and dislocation density gives rise to a new type of intrinsic TWE: while most of the hook material in region C transforms to austenite on heating, a considerable amount of the material remains martensitic near the outer hook curvature (position A) where plastic strain is maximal. The transformation induces the changes in hook shape on heating, with the stabilized martensite in region A deforming pseudo-elastically by twinning. On cooling, this material exerts a pseudo-elastic recovery stress, thus forcing the hook back into its original shape, with a concomitant transformation to martensite in Region C.

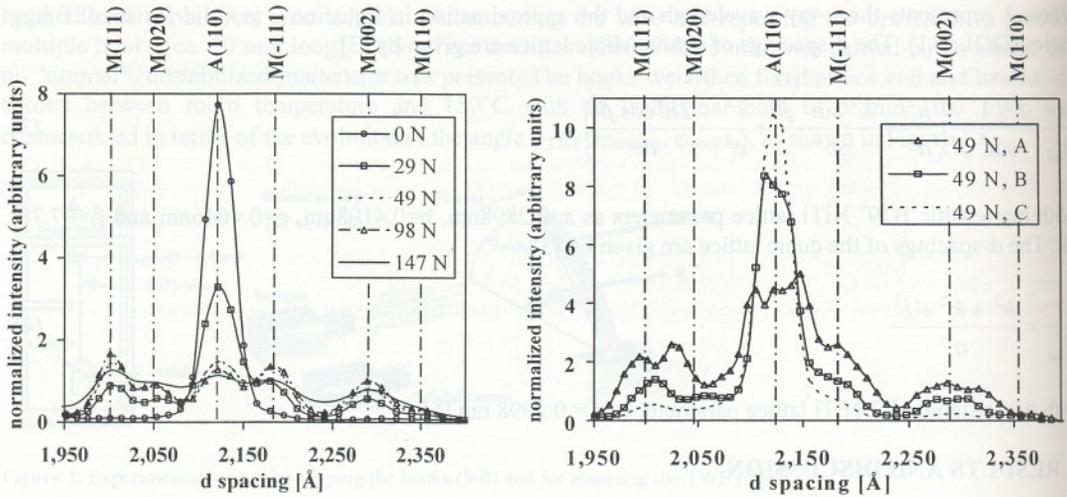


Figure 3. Diffraction results for global volume (left - all hooks from Table 1) and local volume (from one hook with  $P=49$  N at positions A, B and C).

#### 4. SUMMARY AND CONCLUSIONS

A plasticity gradient was introduced in a martensitic NiTi wire by superposition of tensile and bending stresses, leading to a curved hook exhibiting the two-way shape-memory effect (TWE). Martensite in the outer curvature of the hook is most strongly deformed and does not transform to austenite on heating. Martensite in the less strongly deformed regions of the inner curvature of the hook is less stabilized and transforms to austenite on heating, thus producing a shape change in the hook, which is accommodated pseudo-elastically by twinning in the outer curvature of the hook. On subsequent cooling, the energy stored in this region restores the original shape of the hook, thus biasing the transformation to martensite in the inner curvature of the hook. This new type of TWE was shown to be stable over 50 thermal cycles.

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