Electrical Pulse Shape Setting of Thin $Ni_{49}Ti_{51}$ Wires for Shape Memory Actuators

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1. Introduction

Shape memory alloy (SMA) wires are employed as actuators in small devices for consumer electronics, valves, and automotive applications (Ref 1). NiTi intermetallic, thanks to its good ductility, cycling stability, and large recoverable strain capability, is the most successfully and widely employed shape memory material. SMAs have to undergo a shape setting heat treatment aimed at imparting the selected shape and to optimize the shape memory effect. This treatment can be designed in order to induce in the material specific microstructures which confer to the SMA appropriate functional properties in view of the specific application. Wire shape setting is commonly carried out by straight annealing with an applied axial tension (Ref 2, 3). In the current study, a non-conventional heating method, already proposed for superelastic wires (Ref 4-7) called FTMT-EC, is now first investigated on Ti-rich NiTi alloys for shape memory actuators. Small-size $Ni_{49}Ti_{51}$ wires, 25 μm diameter, were thermally treated by high-power electrical pulses of short duration under constant strain. In particular, after preliminary electrical shape setting we observed that two current (225 mA) pulses of 10 ms were suitable to impart good shape memory properties to the wire. The functional properties were characterized by thermo-mechanical cycling under constant load and correlated to shape setting parameters. TEM investigations were carried out to analyze the effect of the above pulses on the resulting microstructure of the NiTi wires.

2. Materials and Experimental Methods

A $Ni_{49}Ti_{51}$ (at.%) ingot was produced using a vacuum induction melting furnace (Balzers VSG10), in a dense graphite crucible and under controlled argon atmosphere. The ingot was hot forged, hot rolled, and finally cold drawn down to 25 μm in diameter (about 50% final area reduction). Full details on the thermo-mechanical process adopted for the NiTi wires are reported in Ref 8. The unconventional thermal treatments were performed by applying to the final cold-drawn wires (φ = 25 μm) high-power electrical pulses of short duration. Specimens of the thin wire (100 mm in length) were vertically positioned and constrained in the apparatus by fixing clamps. A precision current/voltage source unit (Agilent mod. 9201) forced current pulses through NiTi element, as two-wire method electrical connection, and the voltage through the same leads was measured by the same unit. In particular, the wire was subjected to two 225 mA electrical pulses of 10 ms under constant strain (Fig. 1). The elapse time between the two pulses was 10 s. After this fast current shape setting, to evaluate the shape memory performances of pulsed wires, thermal cycles under constant load (0.1 N, corresponding to about 200 MPa) were carried out by a thermostatic chamber (Angelantoni mod. Sunrise 250) over a temperature range between −50 and 150 °C, with a heating/cooling rate of 0.038 °C/s. For this purpose, the wire was axially loaded by a weight clamped on its lower edge. The displacement was measured by a linear voltage differential transducer (LVDT Macro Sensors HSTA 750-125). TEM images and selected area electron diffraction (SAED) patterns of the selected samples were acquired by an analytical transmission electron microscope operated at an accelerating voltage of 120 kV. The instrument was equipped with an energy-dispersive x-ray spectroscopy (EDXS) system to investigate the local composition of the imaged regions. To avoid any unwanted microstructural modification during TEM sample

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preparation, pieces of NiTi wires were sandwiched between a TEM copper disk—3 mm diameter, with a central hole of 0.1 mm diameter, and a copper grid. After room-temperature curing of the epoxy resin used in the preparation of the sandwich, this was ion milled at room temperature using a 5 keV argon ions until electron transparency was achieved in a sufficiently wide region of the wires. TEM observations have been conducted before and after having kept the specimen for 1 h at −30 °C to recover possible transformations induced by the ion bombardment heating.

3. Results and Discussion

10 ms electrical pulse was applied twice to the material for the wire shape setting. Parameters of the proposed pulse were chosen after preliminary tries for inducing an appropriate recovery strain in SMA wire. The recovery strain values were similar after applying either one or multiple pulses. In Fig. 2, the signal of the second pulse triggered after 10 s from the end of the first is depicted. The serial electrical circuit (current source unit, cables, clamps, and SMA wire as resistance load) is obviously characterized by transient phenomena. The transient behavior lasts for about 5 ms before and after the almost-constant current regime. However, the energy of about 0.09 J supplied to the wire induced a strong modification of the grain structure as shown by the TEM micrographs in Fig. 3 and 4. Before the heat treatment (reference condition), the strongly deformed microstructure of the wire (Fig. 3a) featured an ultrafine grain size, with preferred orientation along wire drawing direction. The blurry contrast is determined by the presence of cold drawing structural defects, like dislocations, visible in the elongated grains of the alloy (Ref 9, 10). The cold-drawn condition and relevant defects are compatible with the stabilization of the austenitic cubic phase (PDF card no.: 18-0899). This is the main phase detectable in the SAED in the inset of Fig. 3(a), acquired with a diffraction aperture selecting the whole field of view. The principal austenite diffraction lines are indicated. In the micrograph twinned grains are even visible, whose attribution to the martensitic monoclinic polymorph of NiTi (PDF card no.: 35-1281) would even agree with the presence of very weak diffraction spots of this phase. After the cooling treatment (1 h at −30 °C) of the TEM specimen, the situation remains substantially unchanged as it concerns both the microstructure and crystallography of the alloy sample (see Fig. 3b and relevant inset with SAED). The shape setting treatment induces the recrystallization of the cold-worked microstructure. The resulting equiaxed grains have an average size of about 100 nm (Fig. 4a) and the SAED still displays the clear presence of austenite NiTi (inset of Fig. 4a). Dislocation structures have fully annealed out and in this case the cooling treatment carried out on the TEM specimens introduces visible changes. The after-treatment microstructure, displayed in Fig. 4(b), features twin structures, extending to whole recrystallized grains, suggesting that monoclinic martensite NiTi has formed. This is confirmed by the relevant SAED pattern (inset Fig. 4b).

For the sake of completeness it is worth noticing that a secondary phase was occasionally observed in both samples (cold-drawn and pulse-treated) and tentatively identified as the intermetallic Ti_2Ni phase (PDF card no.: 72-0442) (Ref 11). It

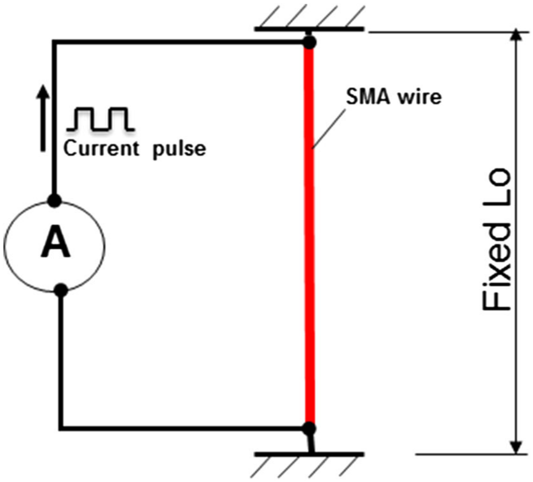

Fig. 1 Scheme of the electrical circuit. The wire was subjected to two 225 mA electrical pulses of 10 ms under constant strain

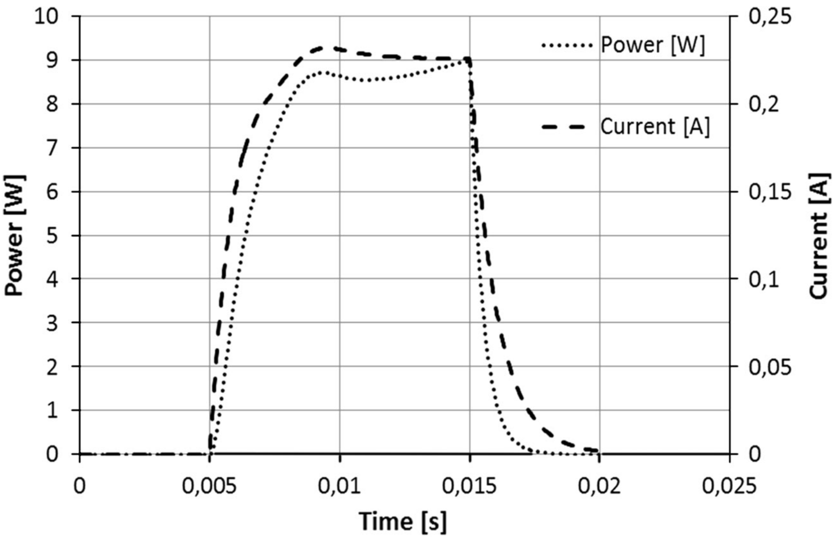

Fig. 2 Second electrical pulse used for wire shape setting

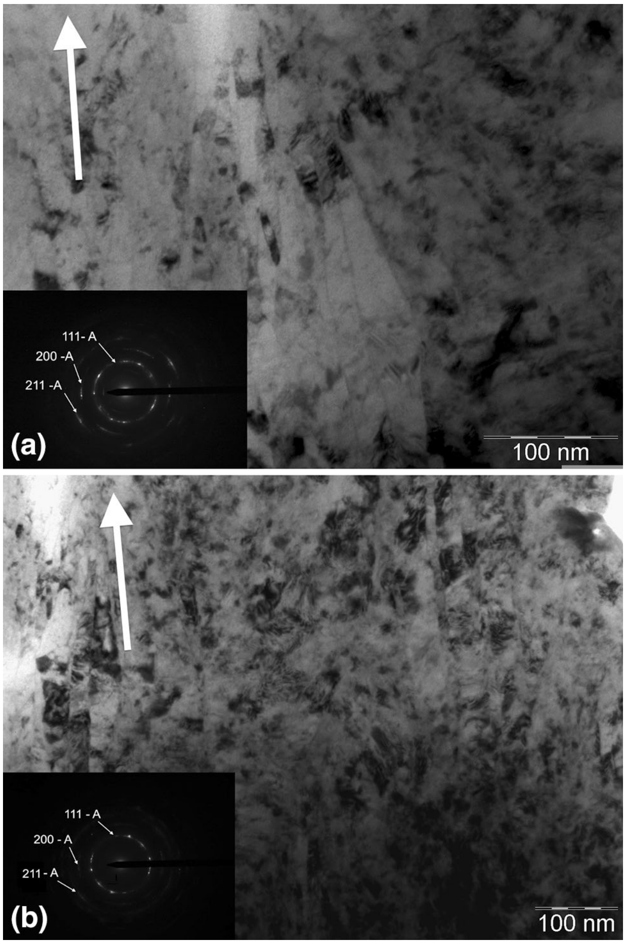

Fig. 3 TEM micrographs showing the microstructure of NiTi wire in the originally cold-drawn condition. TEM sample observed: (a) in the as-prepared condition and (b) after cooling at −30 °C for 1 h. The arrow indicates the wire drawing direction. Insets: SAED patterns of the imaged regions with indexed main diffraction lines. A: austenite NiTi

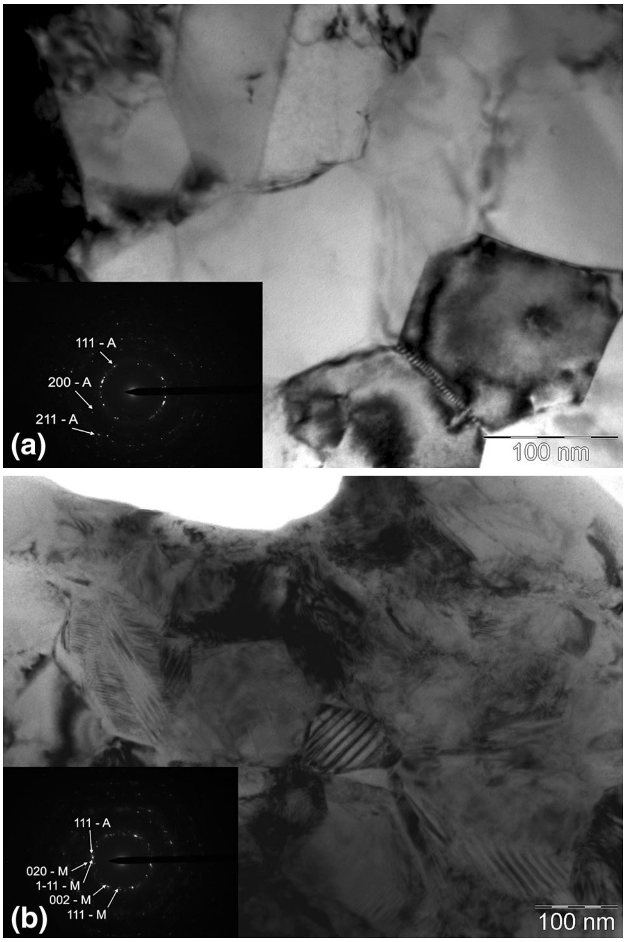

Fig. 4 TEM micrographs showing the microstructure of NiTi wire after shape setting heat treatment. TEM sample observed: (a) in the as-prepared condition and (b) after cooling at −30 °C for 1 h. Insets: SAED patterns of the imaged regions with indexed main diffraction lines. A: austenite NiTi and M: martensite NiTi

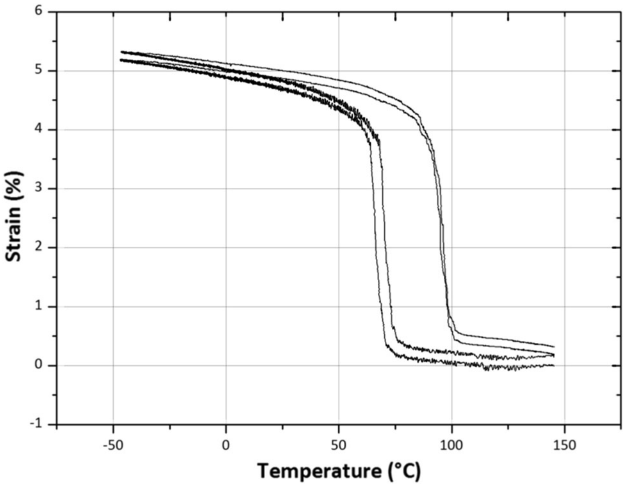

Fig. 5 Thermal cycles under constant load (0.1 N)

turns out that this secondary phase is very stable and unaffected also by the recrystallization phenomena induced by the electrical pulse shape setting procedure. It is also to be remembered that this highly stable intermetallic phase, reported to be resistant to aging or to solution heat treatments (Ref 12), is actually isomorphous to the spinel oxide phase $Ti_4Ni_2O_x$ ($0 \leq x \leq 1$) (PDF card no.: 72-0443) (Ref 13-15). Further investigations are, therefore, under way to make clear which one is the phase actually present, if not both, in the NiTi micrometric wires: This is an aspect to be definitely considered in view of the influence that precipitate phases may have on mechanical properties, like fracture toughness, fatigue behavior (Ref 16, 17), and characteristic transformation temperature (Ref 1, 18) of the NiTi material.

The ε-T curves obtained from thermal cycles under constant stress (200 MPa) carried out after a double pulse are shown in Fig. 5. Electrical pulse shape setting confers to the material good shape memory properties, analogous to those achievable with optimized conventional treatment (Ref 19, 20) and potentially interesting for the use of this sort of wires in actuators. In fact, the material shows a very narrow thermal hysteresis (about 25 °C) and the capability to recover high strain values (about 5%). These curves also show a comparatively low residual strain after the first cycle. Again, both the direct and reverse transformations take place over a temperature range of 5-7 °C with practical benefits in actuation and reset times.

4. Conclusions

Electrical pulse shape setting turns out to be a suitable heat treatment for SMA cold-drawn wires (25 µm). Two 9 W (225 mA) pulses of 10 ms induce a good combination of shape memory characteristics to the material. The material showed 25 °C wide thermal hysteresis and the capability to recover about 5% strain under a constant tensile stress of 200 MPa. TEM investigations displayed that electrical pulses involve recrystallization of the starting plastically deformed microstructure. The average equiaxed grain size of the alloy after the two electrical pulses is 100 nm. Incidentally, grains of a secondary phase, tentatively identified as Ti_2Ni, were observed before and after these unconventional heat treatments which were clearly unable to solution them. Further investigations are being conducted to make the actual type of these precipitates clear; as it cannot be ruled out that they might actually be the isomorphous spinel oxide phase $Ti_4Ni_2O_x$.

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