Heat treatment of thin NiTi filaments by electric current

J.Pilch, L.Heller and P.Sittner
Institute of Physics of the ASCR, Prague, Czech Republic

Heat treatment as an important step of NiTi technology

The conventional processing route from nickel and titanium metals to final superelastic or shape memory components made of NiTi alloy – melting and ingot casting, extrusion, hot/cold working, and final cold drawing (rolling) – is completed with a final thermomechanical treatment of the NiTi wire (sheet) at temperatures 400 °C-800 °C. This final step is called either “heat treatment” or “shape setting” in cases when the shape of the element is mechanically constrained. From a microstructural point of view, two major changes occur in the alloy.

i) The heavily deformed, cold worked microstructure existing in the as-drawn component is rebuilt into an annealed microstructure through a sequence of lattice recovery processes.

ii) Diffusional phase transformation processes change the phase composition and/or chemical composition of the matrix.

Following heat treatment, the NiTi component displays functional properties (e.g. superelasticity), and the shape in which it existed at high temperature is memorized. The outcome (figure of merit) of the final thermomechanical treatment is thus two-fold – the functional properties of the component are set as required and its new parent austenite shape is established.

Since the NiTi component in most engineering applications experiences thermal and/or mechanical cycles, it is essential to realize that both the functional properties and shape must not only be set as required, but also must be stable during cycling. The shape and functional properties, including their stability and fatigue properties, are in large extent controlled by the final thermomechanical treatment. Hence, this treatment is a very important step in NiTi production technology and has always been given special attention (see articles dealing with conventional heat treatments of NiTi in an environmental furnace (Ref 1)).

This article presents a non-conventional final thermomechanical treatment of NiTi using a DC electric current FTMT-EC method developed through the European research program, Across high-added-VAlue sectors for knowledge-based product service creatiON (AVALON) (Ref 2). The AVALON program focuses on technologies for the production of NiTi hybrid fabrics and their industrial applications. Hybrid NiTi fabrics can be shape set to 3D shapes and/or partially inherit functional properties such as superelasticity or shape memory from the integrated NiTi filaments.

The idea to perform heat treatment of NiTi by electric current was motivated by two needs.

i) To heat treat continuous thin NiTi filaments on textile-compatible equipment (which excludes the use of long, tubular electric furnaces).

ii) To heat treat the NiTi filaments already integrated in fabrics together with natural or polymeric yarns capable of resisting temperatures only up to ~300 °C.
Heat treatment of NiTi wires by pulsed DC electric current

NiTi wire can be heat treated by a short DC electric pulse as an alternative method to the conventional heat treatment in a furnace. Heat treatment experiments (Ref 3) were carried out using a miniature deformation rig, MITTER, consisting of a stepping motor, 100 N load cell, electrically isolated grips, Peltier furnace, laser micrometer for strain measurement, and special electronics capable of sending a controlled electric power pulse that could heat the Ni-Ti wire up to the melting point while simultaneously performing electric resistance measurements.

Fort Wayne Metals #1 superelastic, as-drawn Ni-Ti wire (56.0 wt.% Ni) with diameter $d = 0.1$ mm was used. After mounting the wire on the rig, the initial length $l_0$ (~50 mm) and initial electrical resistance of the wire $\rho_i$ at room temperature were determined, and the wire was preloaded to the desired tensile stress (400 MPa in the present experiments), and its length was fixed. While the length is kept constant, the wire was exposed to a piecewise controlled DC power pulse, $P(t)$, characterized by maximum power, $P_{HIGH}$, pulse time, $t_{2}$, and an additional six parameters: $P_{LOW\_0}$, $P_{LOW\_1}$, $t_{01}$, $t_{11}$, $t_{02}$, and $t_{12}$ (Fig. 1a). Evolution of the wire temperature, $T(t)$, as a function of time (Fig. 1b) is calculated according to Eq. 1, taking into account the Joule heat supply, $P$, and the ambient temperature losses.

![Figure 1a](image1.png)
![Figure 1b](image2.png)

*Figure 1: a) Parameters of DC electric power pulse $P(t)$ and b) resulting temperature profiles $T(t)$ generated by various power pulses used in pulsed FTMT-EC treatments, the results of which are shown in Fig. 2 and Fig. 3.*
Evolution of the wire temperature, $T(t)$, as a function of time (Fig. 1b) is calculated according to Eq. 1, taking into account the Joule heat supply $P$ and the ambient temperature losses.

$$\frac{d}{dt} \left( T(t) \cdot C \right) = P - h \cdot \left( T(t) - T_{\text{ext}} \right) - \varepsilon \cdot \sigma \cdot A \cdot T^4$$  \hspace{1cm} \text{Eq. 1}

The heat capacity, $C$, is assumed to be temperature independent, $\sigma$ is a Stefan-Boltzmann constant and $A$ is the surface area of the wire. The specific heat transfer coefficient, $h$, describing the heat dissipation into air per unit time and the NiTi wire emissivity, $\varepsilon$, needed for the calculation of the radiation heat loss per unit time were indentified from series of calibration experiments. Other effects influencing the wire temperature such as heat conduction losses into grips and latent heats were neglected.

**In-situ stress and electrical resistivity measurement during treatment**

As the temperature of the wire increases due to the supplied heat (Fig. 1b), lattice recovery processes are triggered in the heavily cold worked microstructure of the as drawn wire and change it in a fraction of second to the nanosized microstructure (Ref 4-6) in which the wire exhibits functional properties. In response to these recovery processes, the electrical resistance of the wire and macroscopic tensile force change dramatically. The evolution of temperature, electrical resistivity, and tensile stress measured in-situ during the electric power pulse treatment ($P=5\text{W}/100\text{mm}$, $t_c=0.90\text{s}$) are shown in Fig. 2a. After this particular treatment, the wire shows an excellent superelastic plateau type stress-strain response (Fig. 2b) with B2-R-B19' stress induced transformation at room temperature.

Information contained in the in-situ recorded electrical resistivity and tensile stress responses (Fig. 2a, Fig. 3a,b) is discussed next.

The tensile stress of a prestrained metallic wire which is heated should normally decrease with increasing temperature due to thermal expansion and thermal dependence of elastic modulus. In the present case, however, the stress starts to increase with increasing temperature right after the onset of heating (Fig. 2a, Fig. 3a). This is assumed to be due to unlocking of elastic deformation resulting from internal stresses frozen in the heavily cold worked microstructure of the as-drawn NiTi wire, which keeps the wire longer at room temperature. Also, it is possible that a small fraction of the martensite phase (R-phase) transforms back to the austenite phase on heating and tends to shorten the wire. These two phenomena are interrelated. This is supported by the observation that an unrestrained, as-drawn wire heated by the same electric power pulse becomes 3.3% shorter after the treatment. The tensile stress increases (Fig. 2a) with increasing temperature only up to 450 °C, where it reaches a maximum at ~650 MPa and decreases down to zero with further heating. The stress decreases due to thermal expansion, decrease of the elastic modulus, and plastic deformation processes including dynamic recrystallization (Ref 4-6), which dominates at higher temperatures. On cooling, the stress linearly increases back to the starting value of 400 MPa due to thermal contraction and an increase of the elastic modulus.

The elastic moduli of the as-drawn and heat treated wires are not that different, suggesting that the lengths of the wire before and after the treatment are nearly equal. Nonetheless, it is assumed that the plastic deformation processes did proceed in the microstructure. They compensated the 3.3% elastic strains originally blocked in the as-drawn microstructure by the internal stresses which disappeared during the treatment. Even if the wire length did not change, the shape setting thus took place in
The electrical resistivity of a metallic wire normally should increase with increasing temperature. Indeed, if an already fully heat treated NiTi wire is subjected to an FTMT-EC pulse, its electrical resistivity increases with increasing temperature (Ref 4) right after the onset of heating and fully restores its original value after cooling back to room temperature. In the present experiments on as-drawn wires, however, electrical resistivity starts to decrease only when the temperature reaches ~200 °C (Fig. 3b),
Figure 3: Comparison of results of FTMT-EC treatments with $P=2.6\,\text{W/mm}/t_2=0.90\,\text{s}$ and $P=10\,\text{W/mm}/t_2=0.18\,\text{s}$ (see $T(t)$ in Fig. 1b) under a 400 MPa preload and constant wire length: a) in-situ stress-temperature traces, b) in-situ electrical resistivity-temperature traces, c) superelastic stress-strain curves after treatments.
and it decreases about 25% and saturates at ~650 °C. On heating beyond the knee point, the electrical resistivity remains nearly constant or even slightly increases. On cooling back, the electrical resistivity further decreases due to the intrinsic thermal dependence of the electrical resistivity of metals. Thus, the electrical resistivity is not restored after heat treatment since it reflects the drastic microstructure change from the as-drawn to the heat treated wire.

Comparison of in-situ stress (Fig. 3a) and electric resistance (Fig. 3b) responses evaluated in various FTMT-EC treatments (P=2,3,4,5,6W/100mm, t=0.90s and P=10W/100mm/t=1.8s (see corresponding T(t) in Fig. 1b)) suggest that, besides the maximum temperature reached, the temperature rate dT/dt also affects the stress and electrical resistance responses (curves in Fig. 3a,b do not follow the same path) and ultimately, the obtained functional properties. The superelastic response of the treated wires (Fig. 3c) thus depends not just on the maximum temperature but also on the temperature rate of T(t) and in fact, on the whole T(t) profile.

The activity of the lattice recovery processes having fast kinetics (ms) at high temperatures can be controlled partially by the fast heating rates used in the FTMT-EC treatment (Fig. 3), which is not the case in long time furnace heating. Different heating rates are the reason why individual responses in Figure 3a,b do not follow the same path upon heating (Ref 5). Since the diffusional processes having slow kinetics (min.) are essentially suppressed by the short time FTMT-EC treatment, it is possible to establish a heat treatment parameter – microstructure - functional property relationship for the FTMT-EC treated NiTi wires (Refs. 3, 6). This has always been problematic in the case of long time furnace heating where the effects of microstructure recovery and diffusion driven changes of chemical composition on functional properties overlap.

Since the treated wire is exposed not just to high temperature and stress, but also to large electric current, it cannot be ruled out that the passing electrons have their own direct effect on the lattice recovery processes (electron wind effect (Ref 7)). Nevertheless, since the microstructures (Ref 6) and superelastic functional properties (Ref 3) of the FTMT-EC treated wires are essentially very similar to those obtained by conventional continuous annealing of the same wire at constant stress for less than 60s (Ref 8), the possibility, and even likelihood, of direct electron effects on recovery processes is neglected.

**Technicalities of the pulsed FTMT-EC treatment**

The functional properties of the NiTi wire treated by the pulsed FTMT-EC method thus depend on

i) the existing microstructure in the as-drawn wire prior to the treatment (chemical composition, final cold work, texture, grain size, precipitates etc.),

ii) temperature profile, T(t),

iii) stress profile, σ(t).

While the temperature profile T(t) is controlled by the parameters of the supplied electric pulse and environmental conditions, the stress profile σ(t) is controlled (besides the electric pulse parameters) by the applied mechanical constraint. Temperature and stress profiles are used as process parameters for the FTMT-EC method. Care must be taken since σ(t) is affected by T(t) (Fig. 3a) and the treated filament may be easily broken or extensively deformed during the treatment. For
each particular wire (i) and desired functional property, optimal $T(t)$ and $\sigma(t)$ have to be found experimentally.

Electric power up to 200W and pulse times ranging from microseconds to seconds were used in FTMT-EC treatments of thin NiTi wires (d < 0.1mm) for textile applications. Regarding the stress profile, in addition to the fixed length constraint used here, there are other possibilities such as free wire, constant tensile force, variable tensile force, or complex stress constraint. This, although very important for the FTMT-EC treatment, is not further elaborated here due to a lack of space.

Reproducibility of the results of the FTMT-EC treatment relies on the precise control of $T(t)$ and $\sigma(t)$. Considering the large variation of the electrical resistance of the wire during treatment (a decrease of ~45%), it is essential that the electronics system used is capable of controlling precisely with time the supplied electric power $P(t)$ (Fig. 1a). It is also essential to carefully control the air environment of the treated wire (controlled $T_{ac}$ and controlled air flow), otherwise Eq. 1 does not yield reproducible $T(t)$ results. Finally, it is necessary to carefully control the mechanical constraint with time. If all this is accomplished, the $T(t)$ and $\sigma(t)$ history can be perfectly reproduced, and NiTi wires with required functional properties can be readily obtained by the FTMT-EC treatment. The primary advantages of the FTMT-EC method compared to conventional furnace treatment of NiTi, which relies on the temperature and time that the material spent in the furnace, are high process speed, less energy consump-

![Diagram of NiTi wire treatment process](image)

**Figure 4:** Schema of the NiTiTEC equipment for FTMT-EC treatment of continuous thin NiTi filaments (FWM #1 NiTi 0.1mm) for textile applications.
tion, and increased precision with which the activity of the lattice recovery processes can be controlled.

**Continuous FTMT-EC treatment of NiTi filaments for textile processing**

The first application the FTMT-EC treatment found is the textile-compatible conditioning of NiTi filaments which takes advantage of the high speed of the method. A prototype of dedicated equipment called NiTiTEC (Fig. 4) was designed and built. The NiTi filament is heat treated during re-spooling while passing over two electrodes (Ref 9) with a speed of several m/s. Electric power, wire speed, and wire tension are the key process parameters. The temperature between the electrodes and the electrical resistance of the treated wire when it cools down are monitored and used as feedback signals for process control. Compared to the FWM #1 NiTi wire straight-annealed by the provider, the characteristics of the continuously FTMT-EC treated wires (~6V) are comparable, except for the hysteresis width which is smaller.

**Low temperature heat treatment of NiTi filaments embedded in textiles**

The second application of the FTMT-EC method in the textile field is the so-called “low temperature heat treatment” (Ref 9) of NiTi filaments embedded in fabrics. This special heat treatment takes advantage of the fact that the heat comes from inside the wires as well as of the previously described precision of the FTMT-EC method. It relies on a combination of relatively large external stress, environmental temperature \( T_{eu} < 300 \, ^\circ C \), and electric pulses utilized to shape set NiTi filaments already embedded in textiles. The method can be applied to partially heat treated NiTi filaments.

**References**


2. www.avalon-eu.org


9. Czech patent application PV2009-279