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Fabrication Process and Characterization of NiTi Wires for Actuators

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Abstract

A production route of NiTi wire with shape memory effect has been experimented on laboratory equipment and transferred to pilot scale. Technological aspects of vacuum induction melting, hot and cold working operations have been reported. The material has been characterized throughout the production steps by DSC, stress-strain, fatigue and thermo-mechanical measurements.

The process has been optimized for long life applications with particular focus on stabilization of functional properties over lifetime. Preliminary characterization of actuators using shape memory alloy wires deriving from the experimented production route have been carried out.

Introduction

NiTi is an enabling material in an increasing number of applications especially in the biomedical field where in the last years NiTi is successfully used in engineered devices such as stents, orthopaedic implants and surgical tools [1]. Additional mass volume applications such as automotive and appliance can benefit from using SMA based devices. As an example over 60 electromechanical actuators can be installed in a modern vehicle, some of which could be simplified or replaced by using SMA [2]. The relatively low transformation temperatures and the critical availability of material with tailored characteristics have been the limiting factors for implementation in large scale applications. In addition, very often performances of commercially available SMA material can be detrimentally modified during shaping or training of the prototype SMA devices. With this in mind integrated cooperation of alloy producers and SMA device designers is key to guide the fabrication procedures of tailored semifinished products.

This study investigated the technological aspects of NiTi wire manufacturing from the melting operations of a Ti rich NiTi alloy to the stabilization of the functional properties of the semi-finished material. The resulting wire maintained its functional characteristics over 100.000 cycles showing at least comparable performances to the commercially available high fatigue life SMA wires. Wires of 0.4 and 0.5 mm in diameter were then successfully applied in SMA actuators.

Experimental Methods

Melting Process

Vacuum Induction Melting (VIM) is a typical melting technique for production of NiTi based alloys. The magnetic stirring effect of the molten pool guarantees an excellent compositional homogeneity degree of the VIM processed materials. This is particularly appreciated for NiTi alloying because of the strong influence of the chemical composition on the alloy phase transformation temperatures. The major disadvantage of VIM is the contamination coming from the crucible, which is usually made of graphite.

$Ni_{49}Ti_{51}$ (at.%) alloy with high martensitic transformation temperatures was melted by using VIM plants starting from electrolytic nickel and titanium sponge (grade 1). Preliminary small ingots (5 Kg each) were prepared by using a laboratory VIM furnace (Balzers VSG 10) equipped with graphite crucible and a cylindrical metallic casting

mould. After preliminary melts, 40 bigger NiTi ingots (20 Kg) were produced with industrial VIM equipment (Balzers VSG 50) at Saes Getters (fig. 1). Melting and casting conditions were optimized to prevent solidification cracks and reduce shrinkage pipe into the cast alloy.

Figure 1- Industrial VIM plant	Figure 2 - 5 kg ingot

Hot and Cold Working

NiTi ingots were hot extruded using both direct and indirect horizontal extrusion presses. Direct extrusions were performed without protective sleeve; processing temperature (950°-1050°) and extrusion ratios (11:1 and 6:1) have been varied to optimise the procedure.

For indirect extrusion NiTi billets were canned into a protective Cu alloy sleeve and processed at temperature of about 900°C with extrusion ratios from 27:1 to 18:1. [3]

After extrusion the bars were machined to remove oxides and the protective Cu alloy layer before hot rolling (900°C) down to a cross section of 50 mm^2. The rods were hot and cold rolled and finally drawn to wire with diameters of 0.4 and 0.5 mm with intermediate fully annealing heat treatments. Several process conditions were experimented in order to optimize the hot and cold procedures using pilot scale equipments with a production capability up to 150 Kg/year.

Training

The cold worked NiTi wire does not exhibit the desired shape memory performances, which can be achieved through a series of cold work and heat treatments [4-6]. Our target was to obtain a wire showing a shape memory effect with a minimum recovered strain κ_{re} of 4 % at constant applied (bias) stress of 200MPa for at least 50.000 cycles life. Continuous strand thermal treatments were performed in a temperature range of 350-600°C under applied stress of 50-300MPa. The control of tensile load, and annealing temperature and time are crucial aspects to guarantee stabilization of the SMA functional properties [7]. The process was first experimented on laboratory scale Reel-To-Reel equipment and then transferred to an automatic pilot plant. A schematic view of the annealing apparatus is depicted in figure 3.

Among the various training procedures, SMA literature teaches that the constant stress thermal cycling seems to be the most effective to promote two way shape memory

effect[4-6]: we experimented constant stress thermal cycling as well as martensite deformation routes. As for the hot and cold working production steps, a non continuous training equipment was used for experimentations at laboratory scale before transferring the process to a continuous pilot training plant specifically designed and manufactured for this purpose.
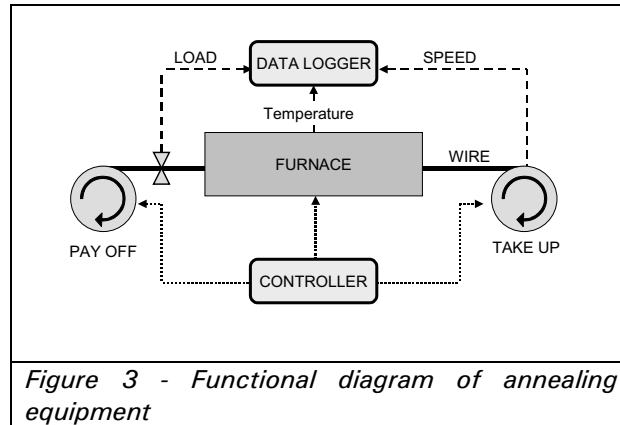

Figure 3 - Functional diagram of annealing equipment

Characterization
Microstructure of as cast and extruded materials were analysed by optical microscopy and SEM. The latter was also employed to characterize fracture surface of the cycled specimens.

The impurities content (O and C) as well as the transformation temperatures were systematically measured in specimens cut from different longitudinal and radial positions from the ingots to verify homogeneous distribution of impurities and composition. Analytical determination of oxygen and nitrogen has been carried out by gas extraction using a LECO TC 436 instrument, while C was analysed using a LECO CS 444. Average O and C ingots contamination were 780 ppm and 650 ppm respectively. Similar mean values were also measured on trained wire showing that wire preparation and training process do not significantly add contamination to the material.

The transformation behaviour was examined at different material production steps by Differential Scanning Calorimetry (DSC) using a Seiko DSC220C. All the DSC scans were carried out upon cooling/heating at rate of 10 °C/min, according to the F 2004-00 ASTM standard.

Thermomechanical fatigue tests were performed by using cycling stations where 150 mm long specimens can be vertically hanged and tensioned by a constant applied load (suspended weights). The wire is heated by Joule effect and the displacement is measured during heating/cooling (air) cycles between room temperature and nearly 150°C under at constant tensile stress of 200 MPa.

For hysteresis measurement a dedicated station similar to that reported above was enclosed in an environmental cell, and the wire was thermally cycled at heating/cooling rate of 1 °C/min.

Results and Discussion

The material was melted and cast under high vacuum (P < 10^{-4} mbar); typical shrinkage pipes due to solidification in a cylindrical mould were observed. Microstructures of ingots cross sections are reported in figure 5.
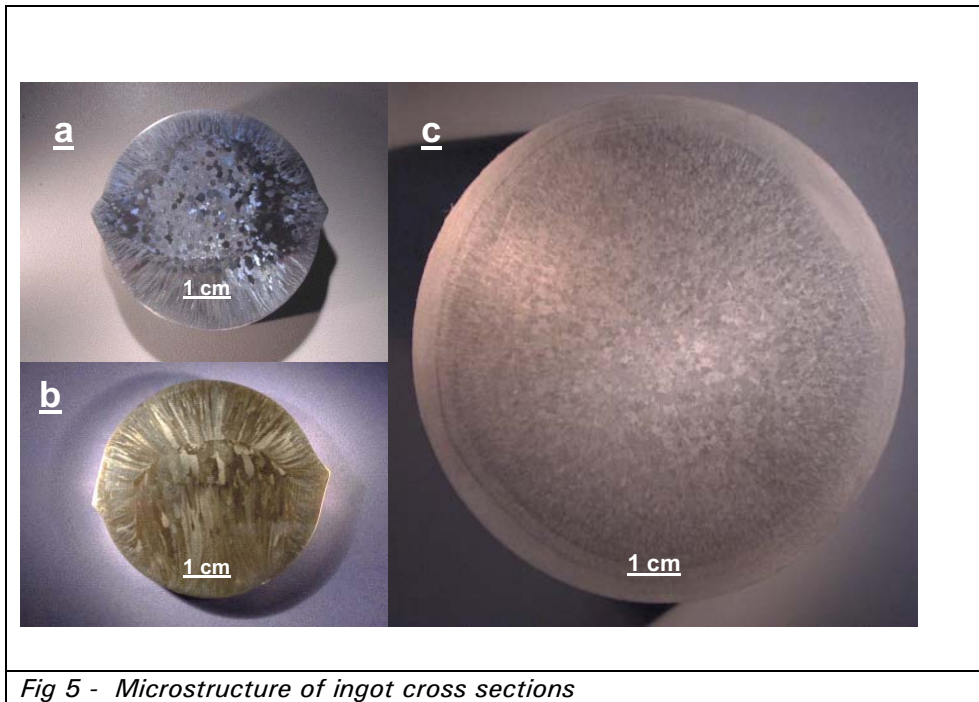

Fig 5 - Microstructure of ingot cross sections

At the bottom of small ingots (fig. 5a) typical solidification structures can be observed: i) small grains at the outer part due to the rapid solidification; ii) a columnar grains region obtained by an high radial temperature gradient during the solidification; iii) equiaxial grains in the central part. The central small ingot area of fig.5b, instead, shows long columnar grains due to a different solidification condition. The difference in grain morphology is likely due to the variation in feeding rate resulting from a manual cast operations, and it could causes workability properties variation during the subsequent ingot hot working.

For all the lengths of bigger ingots (20 Kg each) the solidification structure shows a more regular grain growth and a typical cross section is reported in figure 5c.

The big ingots (20 Kg) were hot extruded and, as expected, the microstructure of the extruded rods shows a grain size adjustment due to the deformation and primary recrystallization phenomena relative to the hot extrusion process. Micrograph of the cross section and longitudinal sections of a hot extruded rod is depicted in fig.6: uniform equiaxial grains with mean size of about 30 σm were observed in all the. Indirect and direct extrusion methods resulted in similar rod microstructures.
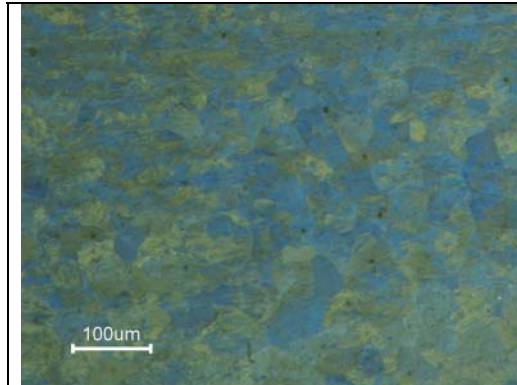
Fig. 6 - Optical micrograph e of hot extruded rod

Calorimetric analyses were performed on both as cast and heat treated (900°C for 1 h + water quench) ingot samples. The DSC scans upon cooling/heating at rate of 10 °C/min show typical martensitic transformation behaviours B2 B19' with transformation heat of -33.17 J/g for the forward and 31-46 J/g for reverse transformation (see fig.8). Moreover for both as cast and solution treated specimens taken from different zone of ingot showed Ms and A_f mean values of 72°C and 107°C respectively with a very low dispersion.

Values of Austenite and Martensite temperatures from six different positions and four different ingots are summarized in the following table:

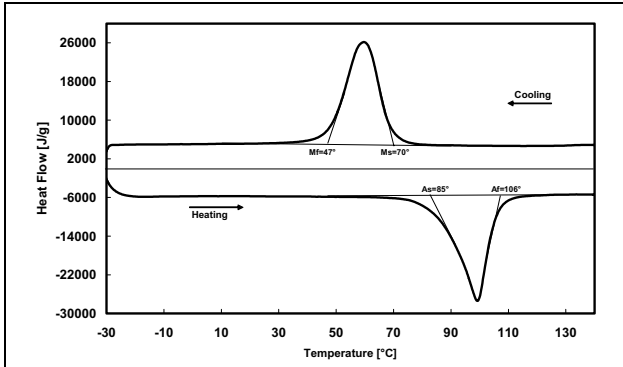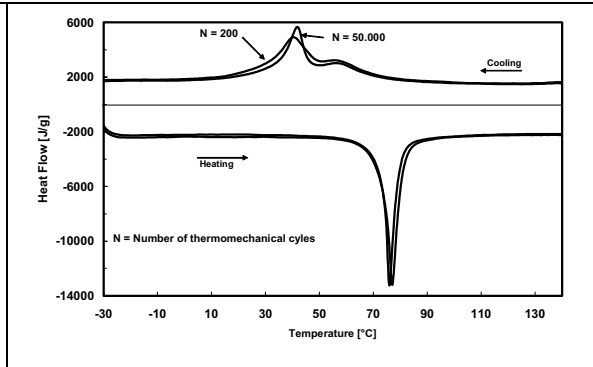| Parameter	Mean Value (°C)	Standard deviation (°C)
As	82.9	5.65
A_p	101.4	3.90
Af	107.8	5.42
Ms	72.2	5.20
M_p	59.0	2.90
Mf	47.8	3.56
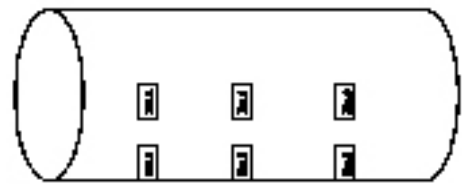
Figure 7 - DSC scan of as cast material

Figure 8 - DSC of trained wire after N thermo-mechanical cycles

DSC curves after N = 200 and N = 50.000 thermo-mechanical cycles of the trained wire reported in figure 9 show the B2 R and R B19' transformation peaks upon cooling. After 50.000 the R B19' peak is sharper than 200 cycles one and the T_p shifted only of 2° at higher temperatures. A single transformation signal B19' B2 is detected upon heating with a very stable behaviour as a function of thermo-mechanical cycling.
These data confirm the very high stability of the wire properties over cycling.
It has been observed that the R B19' transformation is strongly affected by the training thermo-mechanical treatments and its parameters (behaviour, transformation heat, transformation temperatures), could give important indications for the industrial training processes.
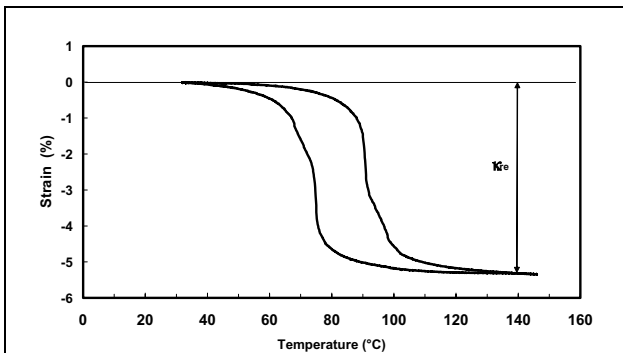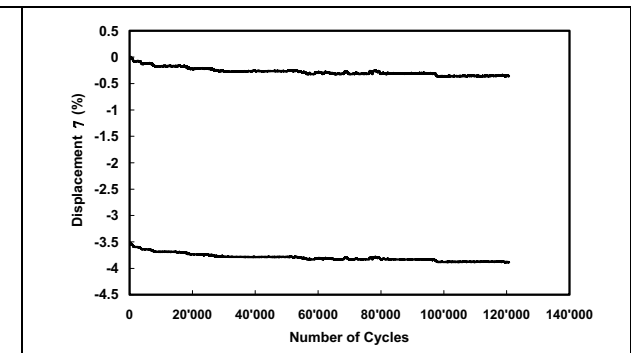

Figure 9 - Hysteresis measurement

Figure 10 - Fatigue life test

Thermal cycle of 0.4 diam. mm trained wire carried out with an applied constant stress of 200 MPa is reported in figure 9. A sharp martensitic transformation occurs at temperature above 70°C with a thermal hysteretic cooling/heating behaviour of 20°C. A recovered strain κ_{re} of 5.3 % is detected.

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Tensile fatigue tests were also performed at constant stress of 200MPa. In some tests the applied heating current was controlled to limit the strain level at 3.5%. In figure 10, the minimum and maximum position versus fatigue life shows stable behaviour with an accumulated permanent plastic deformation of 0.3 % after 120.000 cycles. The data are at least comparable to the ones of the best commercial stabilised products

The fracture surfaces of the cycled wires were analyzed by scanning electron microscopy to clarify rupture mechanisms. The initiation point of failure is often associated with the presence of defects, such as scratch, surface cracks localized at the wire surface. In our case, as expected, EDXS microanalysis carried out at the fracture surfaces of cycled (>100.000) wires showed the presence of oxides and carbon enriched segregations at the initiation points (see fig.11) or TiC inclusions (see fig.12).
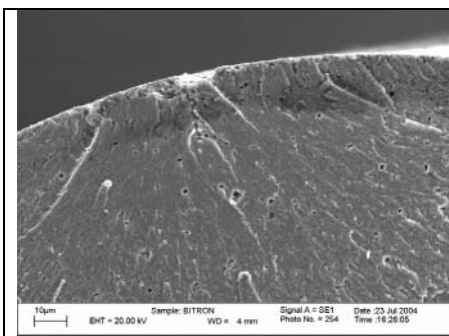
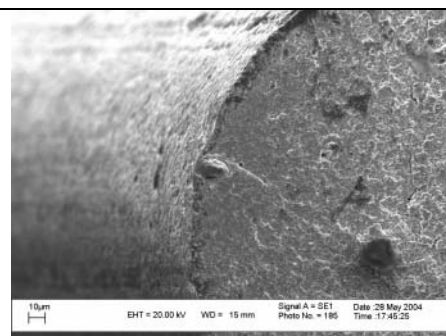|	
Fig 11- Microfractograph of cycled >100000 wire.	Fig 12 - Microfractograph of cycled wire

Wire Testing in Automotive SMA Device

The stabilised wire was tested in a linear actuator, developed at Centro Ricerche FIAT, shown in the picture below:
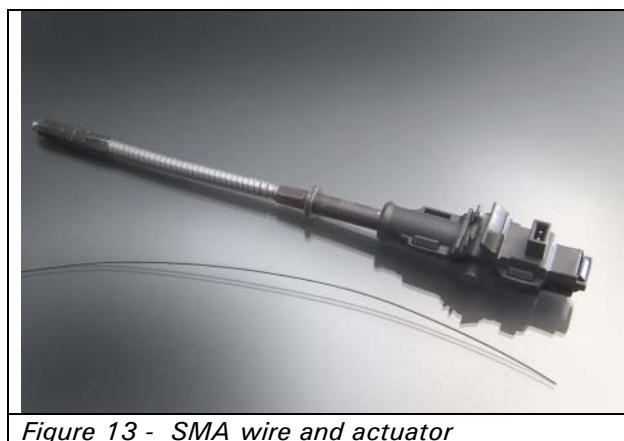
Figure 13 - SMA wire and actuator

The SMA actuator performs both electrical and mechanical operations [8]. In normal condition the SMA wire is activated by Joule effect and it moves retracting the terminal end of 6 mm, in misuse. By pulling the actuator, a manual operation is also available.
The actuator was tested according to the standards for electrical actuators in automotive field.
Thanks to the SMA wire performances all tests were passed, especially a good performance in terms of life cycle and repeatability was achieved. In fact the new actuator can perform over 100.000 cycles in the specified temperature range (from −30 °C to + 80°C) without accidental breaks.
Several vehicle components can be equipped with this actuator, for instance it can substitute some of the mechanical bowden cable used to actuate latches, seats, vents etc…, adding the electrical functionality without any change in components volume and mechanical structure.

Conclusions

An integrated SMA wire production process, from melting to wire training has been set-up, both on lab and pilot scale. An industrial production line is currently under construction.
Ingots and wires have been characterised on the functional and microstructural point of view.
The production process lead to the preparation of wires with state of the art performances and stability, which make them especially suitable for the application in actuators.
A special actuator, with combined electrical and mechanical functionality, has been designed and manufactured, based on the wire developed in this work.
This actuator is currently being evaluated by a major car manufacturer.

Acknowledgements

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