Nano 2023 Innovation Conference & Exhibition — Rome, 18-22 September

ENEA — Agenzia nazionale per le nuove tecnologie, l'energia e lo sviluppo economico sostenibile

Evaluation of the effects of recovery processes on the phase transitions of shape memory filaments investigated through the comparative analysis of resistance-temperature measurements.

Miscioscia R., Borriello C., Mongibello L., Tammaro L., De Girolamo Del Mauro A., Galvagno, S., Citarella A., Verde F., Pandolfi G., Tassini P. and Portofino S.

ENEA - Italian National Agency for New Technologies, Energy and Sustainable Economic Development

Research Centre Portici – Piazza E. Fermi 1, 80055 Portici (NA) - ITALY

Introduction

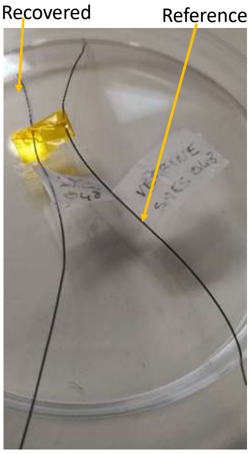
Nitinol is an electrically-conducting SMA widely commercialized thanks to its excellent mechanical properties and chemical resistance. Changes in shape of Nitinol samples follow from reversible thermal transformations of its Austenitic phase "A" into Martensite "M" and vice versa often involving a transition through an intermediate phase "R" dependent on composition and thermomechanical history of the sample. In Nitinol, phase transformations can be obtained through Joule heating then, electrical conductivity plays a key role in the assessment of materials' performances. Recovery and recycle of SMAs can be considered itself a relevant technological issue because European Union is even today totally dependent from international raw material market, but SMA characteristics can be heavily modified by recovery processes which should be carefully analyzed.

Goals

The purpose of this work is comparing the dependence of electrical resistance from temperature of Ni-Ti filaments recovered in different gaseous ambient conditions with factory references to assess the performance of the recovered material and helping in forecasting its possible second-life applications.

Materials and methods

SMA specimens

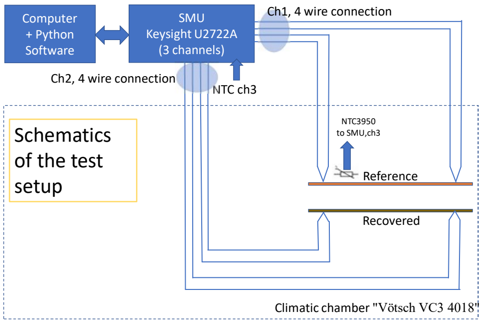


- Commercial Nitinol cold drawn wires manufactured by SAES® Smart Materials (SSM)
- Surface finishing: black oxide
- Diameter : 0.48mm
- Length approx: 150mm

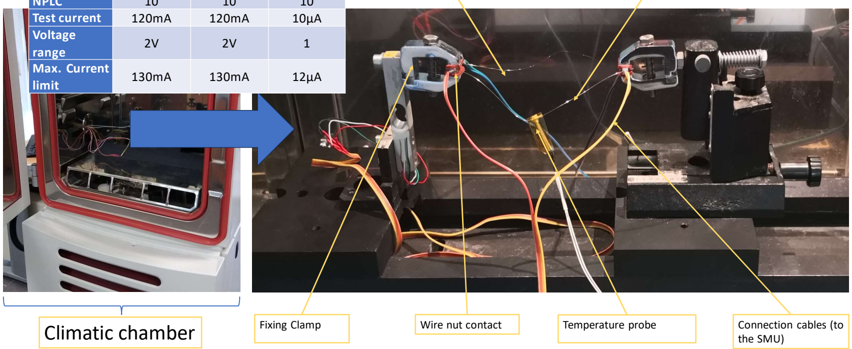
Recovery processes

- thermal treatment in a Lenton Tubular furnace LTF12/600/610
- Gas flow rate 1 l/min
  - Process «1» - gas flow is an **air flow**
  - Process «2» – gas flow is **Nitrogen flow**
- Duration: 60 minutes
- Recipe
  - Heating ramp rate: 3°C/min
  - Isotherm of 60 min at 600°C
  - Free descent to room temperature
- Purification in ethyl alcohol

Resistance-Temperature Test setup



	Channel #1	Channel #2	Channel #3
NPLC	10	10	10
Test current	120mA	120mA	10µA
Voltage range	2V	2V	1
Max. Current limit	130mA	130mA	12µA

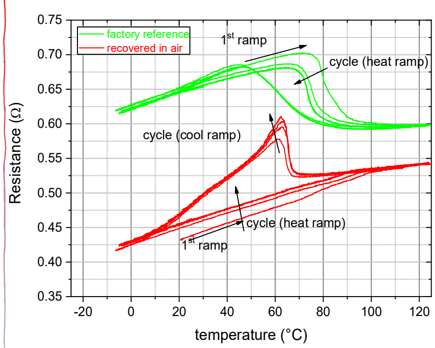


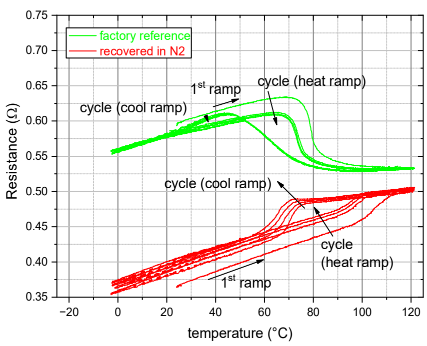
R-T test parameters

- Measurement type: constant current, voltage sensing, 4 wires per channel
- Timing:
  - Simultaneous sampling of reference wire current, recovered wire current, temperature of reference (while temperature ramping in thermal chamber)
  - Sampling period: 1.4s
- Thermal sensor parameters: R=100kΩ (R25 = 100kΩ ±1%, β25/50 = 3950±1%, assumed model : Steinhart-Hart)
- Stress condition during thermal cycles: 0N –> no mechanical loading
- Thermal ramps from -20°C and +125°C

Results

Resistance vs. Temperature





Comments

Samples in the pristine state

- After the first heating curve (M->A)
  - electrical resistance decreases
  - Activation temperatures decrease

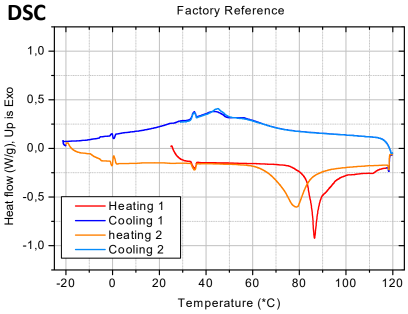
Samples recovered in Air

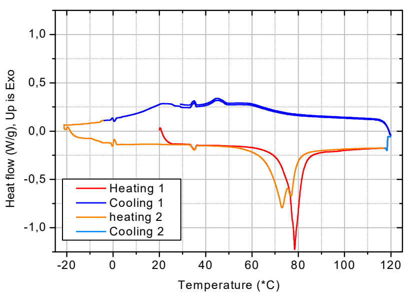
- After the first heating curve (M->A)
  - electrical resistance increases
  - Activation temperatures increase

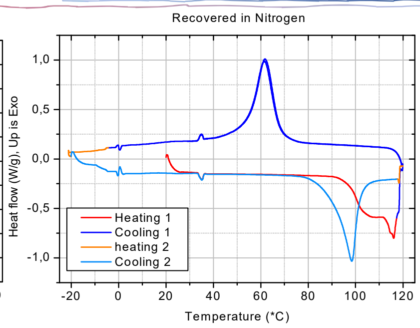
Samples recovered in Nitrogen

- Initially show no response from intermediate phase during cooldown ramps
- After the first heating curve (M->A)
  - electrical resistance increases
  - Activation temperatures decrease

DSC







Conclusions

- Thermal history and gaseous environment employed in recovery processes affect R phase and then, R-T characteristics
- "R" phase is clearly detectable from peaks in R-T curves in pristine samples and in samples recovered in air
- "R" phase electrical activity during thermal transitions seem to be suppressed after recovery in Nitrogen
- Stress effects in recovered samples increase their electrical resistance
- The effect of gaseous reaction ambient during thermal recovery processes needs to be carefully evaluated while assessing second life performance required to Nitinol-based SMAs

Acknowledgements

The support of Project ARIA "Active Responsive Intelligent Aerodynamics", CUP B86G18000610005 (PON "R&I" 2014-2020) is gratefully acknowledged