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Functional fatigue recovery of superelastic cycled NiTi wires based on near 100 ºC aging treatments

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Abstract. Functional fatigue affecting superelastic behaviour of NiTi wires includes an accumulation of residual strain and an uneven decrement of transformation stress on cycling. Although this evolution is observed to diminish asymptotically, it represents an important loss in the maximum recoverable strain level and in the hysteretic dissipative capacity of the material. In this work, the effect of moderate temperature aging treatment on the functionally degraded material properties was studied with two experimental setups. NiTi pseudoelastic wire samples of 0.5 and 2.46 mm diameter were subjected to different cycling programs intercalated by aging treatments of different durations up to 48 h at 100ºC. Results show that important levels of recovery on the residual strains and the transformation stresses were attained after the aging treatments. The analysis indicates that the characteristics of the recovered cycles are rather independent from the treatment duration and from the reached condition before each treatment.

1 Introduction

The martensitic transformation (MT) is the origin of the unique properties of pseudoelastic shape memory alloys (SMA), as superelasticity, shape memory effect, and high damping, in which rely many applications [1, 2]. The properties of Shape Memory Alloys in pseudoelastic state give high mechanical damping, and NiTi wires have been proposed as dampers in civil engineering [3-6]. For this kind of SMA applications, high reliability is needed [7].

In NiTi alloy, several effects induced by temperature aging have been described, usually for temperatures greater than 473 K, which induce measurable structural effects [8, 9]. Then, the time at maximum temperatures should be limited.

For applications that mean many cycles of work, such as damping, fatigue is very relevant [7]. Fatigue can be defined as the change of the properties of materials subjected to fluctuating stresses, which can lead to fracture. Then, the appearance of fatigue depends on the kind of stress cycles applied to the material, i.e. axial (tension-compression), bending, torsion, or others, as combined actions. For classical materials such as steel, usually fatigue refers to structural fatigue, which means incapacity to support further loads and might end in fracture. In fact, fatigue of NiTi alloys has been extensively studied [10-17]. During the test or during the working life, the SMAs accumulate micro-structural defects and nano-scale precipitates which might induce significant modifications in functional properties, this can happen much before the structural failure. We should then address also the problem of functional fatigue in SMA, as failure to perform the expected work or describe the expected stress-strain-temperature designed trajectories after a number of cycles [16]. The functional fatigue is very important in applications of SMA such as dampers with re-centring properties [3]. Functional fatigue can be also important when working with high stresses [18-19].

Evolution with working is observed, for instance, when mechanically cycling NiTi, a "ratcheting" is developed, consisting on increased strains when working, that should be controlled [10-11]. Alternatively, at constant maximum strain, a decrease of stress to transform is observed [6]. From the pioneering work of [20] it has been reported the evolution of transformation curves with pseudoelastic cycling, and also with thermal cycles in [21], the evolution was associated to an increase of dislocation density [22-23]. The generation of dislocations and deffects has been also linked with the increase on line widths of diffraction peaks. [24-25].

It has been also found that some heating of the sample at zero external stress might reduce the residual deformation, this has been interpreted as due to thermal retransformation of retained martensite [26]. A detailed physical model can help to understand the observed characteristics [27]. The importance of these effects on practical applications is increased when large strains and repeated actuation are needed [28]

We have checked the response of SMA wires to moderate thermal treatments (temperatures near 100ºC) after cycling. Results show the partial recovery of

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functional fatigue and the possibility to extend working life of some mechanical components. The reasons of this recovery or "healing" property are also discussed

2 Experimental

For our experimental study, focused on engineering applications, we used a NiTi alloy in the pseudo-elastic state, furnished by Memry (CT, USA), a division of SAES Getters (Italy), and previously, by Special Metals Corp. (New Hartford, New York, USA). The straight-annealed wires had Ni-rich composition (55.95 wt % Ni, balance Ti), the surface of the samples was finished in a light (gray) oxide surface for the wires with diameter of 2.46 mm, and darker oxide surface for the wire of 0.5 mm diameter. The austenite start temperature, As, measured by DSC was 237 K.

Mechanical testing machines Instron 5567 and MTS 810, equipped with climatic chambers, have been used. Also, some home-made computer-controlled devices were used to measure the thinner wires and for thermal treatments at moderate temperature. These devices had isolated grips, so a small (less than 10 mA) electrical current was sent through the wires to measure electrical resistance, and eventually, to heat the samples (electrical current used, near 2 A). Temperature was measured by K-type or E-type thin Omega thermocouples (0.076 mm thermocouple wire diameter) attached to the NiTi wires. The electrical resistance of a 1 m long NiTi wire of 0.5 mm diameter, at room temperature (295 K), was near 4.7 Ohm in austenite and near 6.5 Ohm in martensite (under 8 % strain and stress near 700 MPa) phases. Reoriented and deformed martensite showed a higher electrical resistance than corresponding austenite phase at the same temperature.

The NiTi wires used had a Clausius-Clapeyron coefficient, or dependence of traction stress to transform with temperature, that was 6.5 MPa/K [29]. The used speed of cycling was 850 s per cycle for the 2.46 mm diameter wire, at constant cross-head displacement mode, and 4000 s per cycle for the 0.5 mm diameter wire (in a home-made equipment).

3 Results

The NiTi pseudoelastic wires (2.46 mm diameter) show considerable evolution of the stress-strain hysteretic behaviour during the first mechanical cycles at room temperature. To restrict or avoid any room temperature evolution, as that detected in [30] for Ni-rich alloy quenched from high temperature, we used wires that had been supplied by the furnisher more than one year before the experiments, and kept in the laboratory at 295 K. Appropriate "conditioning" or "training" by initial cycling, 20 to 100 cycles to 8% strain, allows "S-shaped", stabilized cycles, with lower hysteresis (hysteretic energy of around one third of that at the first cycle) and some accumulated "permanent" deformation (to about 2%) [15]. These wires have reasonable damping ability even at low strains (see the first stress-strain cycles in figure 1).

The 0.5 mm diameter wire performed with some differences respect the 2.46 mm diameter wire (figure 2): A functional fatigue exists, the main traits were not very different from the 2.46 mm diameter wire, as residual strain accumulates and stress to transform decreases, but the shape of the hysteresis cycle is less affected at the speeds of cycling used.

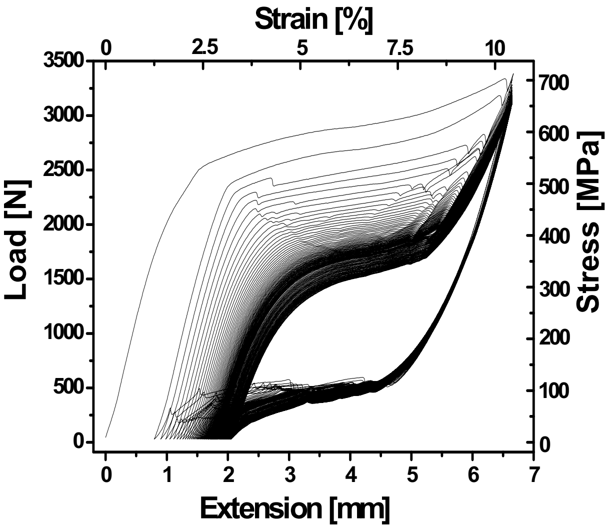

Figure 1. Cycling effect on 2.46 mm diameter NiTi wire. Cycles 1 to 100. The first cycle includes some gripping effects from the mechanical testing machine.

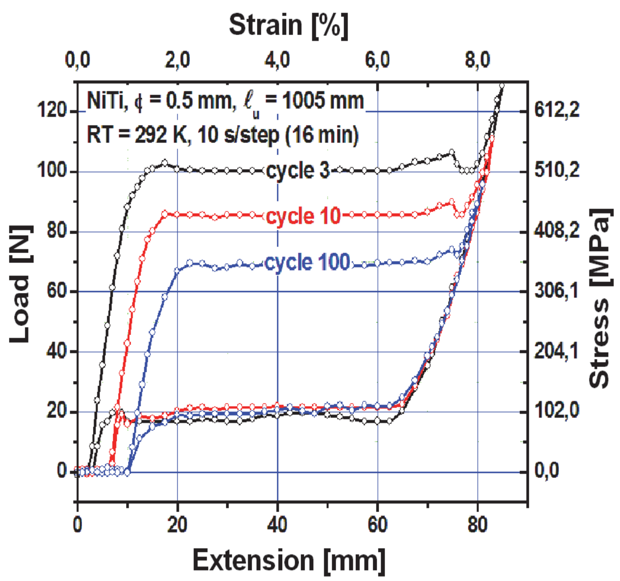

Figure 2. Cycling effect on 0.5 mm diameter wire: cycles 3, 10 and 100.

The electrical resistance of NiTi wires increased slightly with mechanical cycling, during the first cycles. The increase of electrical resistance follows the increase of permanent deformation (creep).

Thermal treatments at low temperatures (around 100ºC), during 5 min., were shown to produce a decrease of the electrical resistance of NiTi thin wire which had been previously cycled (figure 3). The measures showed an increase of electrical resistance recovery when the temperature was increased from 70ºC to 100ºC, but the recovery was not larger when the thermal treatment was done at 130ºC. The residual strains followed a similar tendency. Then, we choose the main thermal treatment temperature to recover properties as 100ºC.

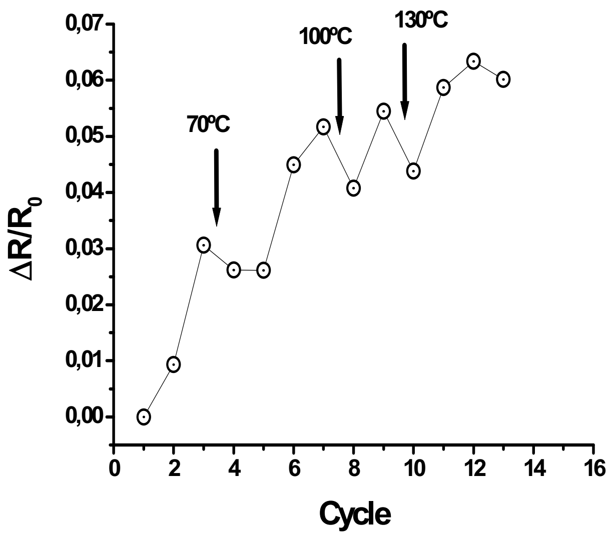

Figure 3. Cycling effect to 8% strain on 0.5 mm diameter NiTi wire. Relative change of electrical resistance on cycling (lines are only visual guides). Cycles 1, 2, 3, 4, 5 to 8% strain. Cycles 6, 7, 8, 9, 10, 11, 12, to 9.5% strain. Cycle 14, to 8% strain (lines are only visual guides). A thermal treatment at 70ºC (during 5 min) produces a small electrical resistance recovery. A thermal treatment to 100ºC (5 min) produces a recovery very near that of a 130ºC thermal treatment.

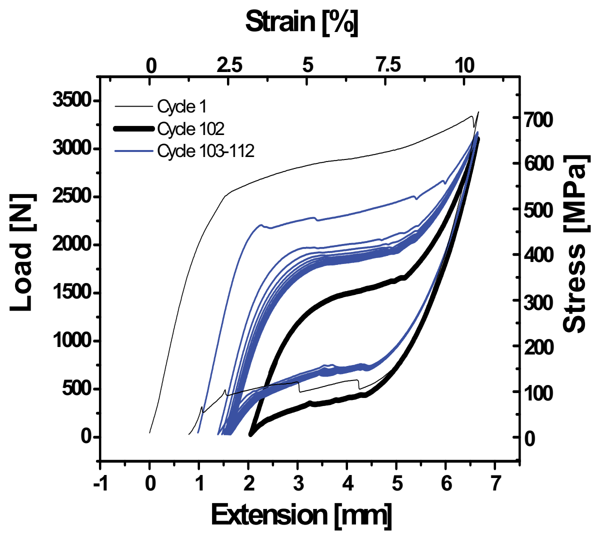

Figure 4. Recovery effect on 2.46 mm diameter NiTi wire. Cycles 1, 102 done after 5 h at room temperature once finished the first 100 cycles, and cycles 103-112 after heating to 100ºC for 3.5 h.

The wire intended to be used for damping (2.46 mm diameter) was subjected to testing. The residual deformation after cycling recovered partially with a thermal treatment at 100ºC. After 100 cycles from the as furnished wire, it had acquired a residual strain near 2%, and the transformation stress had lowered some 300 MPa from the first cycle (figure 4). Waiting 5 h at room temperature produced very small changes in the mechanical cycle. After applying a thermal treatment at 100ºC for 3.5 h, the strain recovered near 1%, and the stress to transform increased near 200 MPa at the middle of the transformation (extension 4 mm in figure 4).

Further cycling and further treatments to 100ºC were done to follow the recovery processes (figures 5 and 6). First, after cycle 112, a thermal treatment at 100ºC during 7 h, then 10 cycles more (to cycle 122), then a thermal treatment at 100ºC during 14 h, then 10 cycles more (to cycle 132), and a thermal treatment at 100ºC during 28 h. Later, 100 mechanical cycles were done, and afterwards aging at 100ºC, during 24 h. The results show a remarkable recovery of properties.

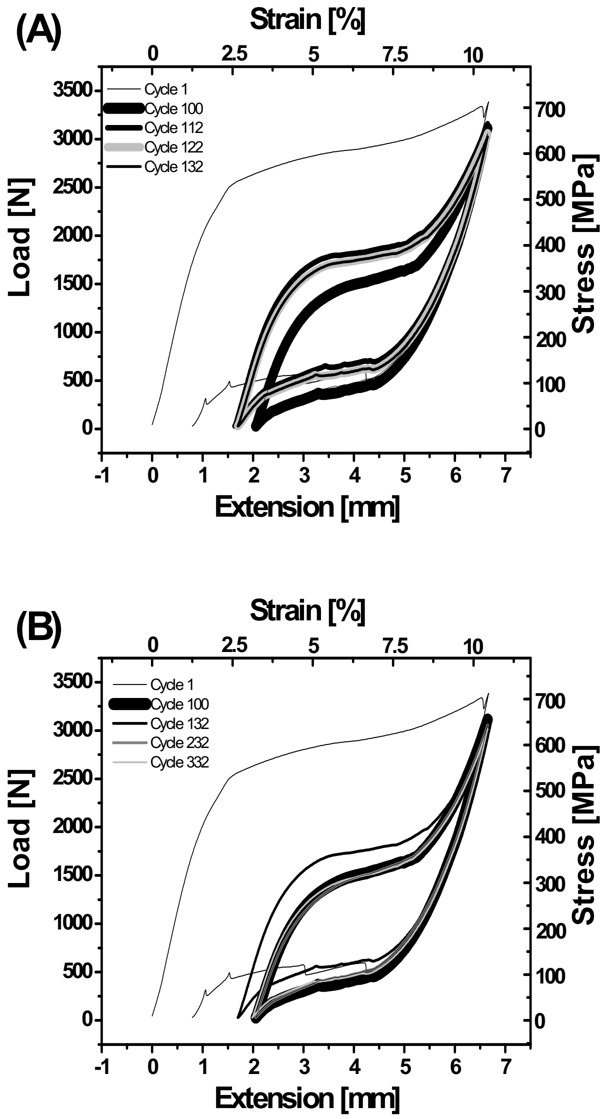

Figure 5. Cycling effect on 2.46 mm diameter NiTi wire. (A): Cycles 1, 100 (at the end of continuous cycling), 112 (10 cycles more after first heat treatment), 122 (10 cycles more after second heat treatment), 132 (100 cycles more after third heat treatment). (B): Cycles 1, 100, 132, 232 (100 cycles more after fourth heat treatment), 332 (100 cycles more after fifth heat treatment).

It was also checked the dependence of the recovery with the time at 100ºC. Registering the extension at constant load of 10 MPa, it is seen that the important part of the extension change occurs in the first near 2 h at 100ºC, and practically no changes are seen up to 28 h (figure 7). The final extension was very similar in all cases.

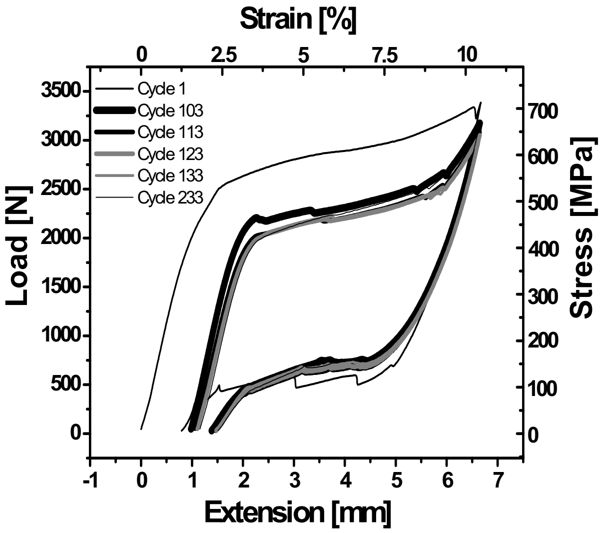

Figure 6. Recovery of 2.46 mm diameter NiTi wire by heating to 100ºC. Cycle 1 compared with the cycles after thermal treatments to 100ºC: cycles 103 (first recovery), 113 (second recovery), 123 (third recovery), 133 (fourth recovery), 233 (fifth recovery), just after the cycles in figure 4 and 5. The cycles after heat treatment to 100ºC result very similar among them.

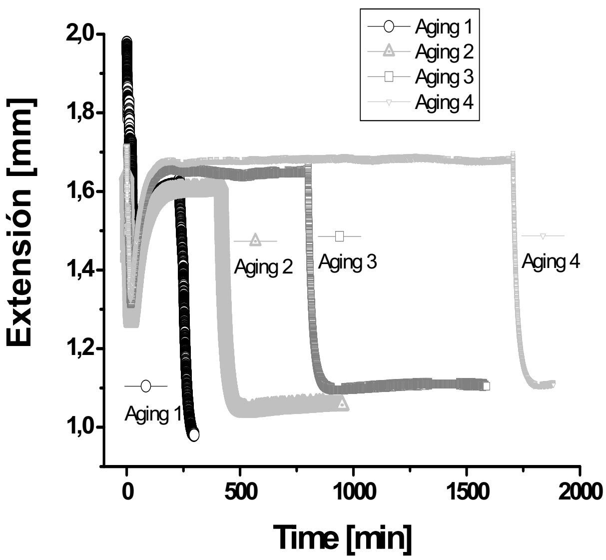

Figure 7. Extension as a function of time during the thermal treatment of NiTi wire 2.46 mm diameter, at 100ºC, for different recoveries. Aging 1: 210 min (3.5 h) at 100ºC; Aging 2: 400 min at 100ºC; Aging 3: 800 min at 100ºC. Aging 4: 1700 min at 100ºC

Some tests were done at different starting room temperature at which the wires of NiTi were initially cycled (295 K). Testing was done at 310 K, 15ºC above the previous room temperature, with the same cycling speed. The recovery with thermal treatment at 100ºC existed, but was smaller than it was when cycling at the previous room temperature. The residual strain accumulation was larger in cycling, indicating a larger plastic deformation, coherent with the reduced recovery.

For a sample tested at 310 K, 15ºC higher than the one in figure 3, the mechanical results indicate a higher residual strain accumulation (up to near 2.35% in 100 cycles to 8% maximum strain), and a lower recovery with thermal treatment to 100ºC for 2 h. The strain recovery was 0.25% compared with near 1% for sample tested at room temperature of 22ºC, and the stress recovery for the sample tested 15ºC higher was near 60 MPa at the middle of the cycle, compared with near 200 MPa for the sample of figure 4.

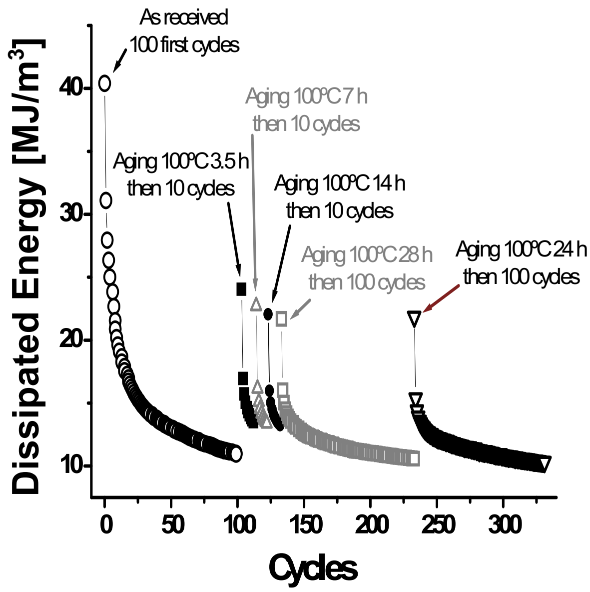

Figure 8. Specific dissipated energy per cycle. Recovery with thermal treatment to 100ºC on 2.46 mm diameter NiTi wire.

The calculated specific energy dissipation follows also a recovery with each of the thermal treatments at 100ºC, for the sample cycled at the standard room temperature (295 K), see the figure 8.

Some tests were also done on overstrained samples. The results in figure 9 are from a 1 m long, 0.5 mm diameter NiTi sample, which had followed 100 cycles to 8% and then was overstrained 2 cycles to 11% and one cycle to 9%. The continuous line represents a further cycle to 8.75%, and successive cycles after thermal treatment at 130ºC for 2 min, 6 min, 2 h and 24 h. The residual strain recovers some 0.5% and does not evolve appreciably on successive treatments, but the stress to transform increases successively with longer thermal treatments, see the figure 9.

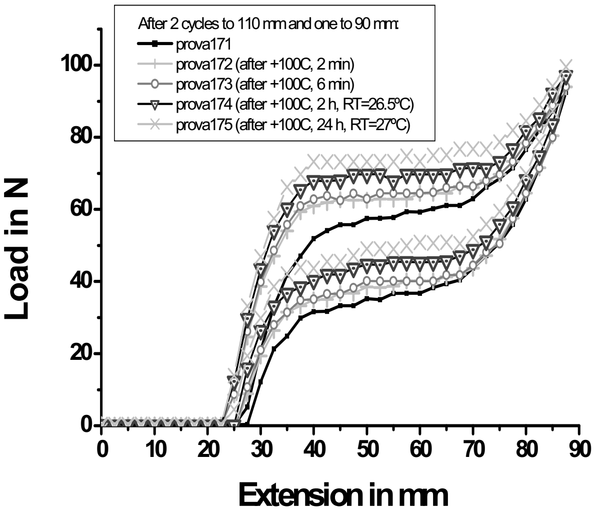

Figure 9. Overstrained 1m long, 0.5 mm diameter NiTi wire. An as furnished wire was subjected to 2 cycles to 11% and one to 9%, and then followed the represented cycles: one cycle (prova171) to 8.75%, 2 min at 130ºC, one cycle to 8.75%, 6 min at 130ºC, one cycle to 8.75%, 2 hour at 130ºC, one cycle to 8.75%, 24 h at 130ºC and one cycle to 8.75% (prova175).

4 Discussion and Conclusions

The properties of SMA tend to degrade with mechanical cycling, this is called functional fatigue when mechanical failure (fracture) does not occur, but the working point of the material can hinder its applications. For the NiTi wires tested, the transformation stress decreased, and the residual permanent deformation increased with number of cycles in an asymptotic, nearly exponential way, if maximum strain on cycling was kept constant. The dissipated energy per cycle also decreased.

By moderate thermal treatment of the wires after cycling, part of the residual permanent deformation was recovered, as well as part of the specific energy dissipated per cycle, and the stress to transform did also recover. The recovery at 100ºC was larger than the recovery at 70ºC, but the recovery at 130ºC was similar to the one at 100ºC. It is suggested that part of the degradation of properties was due to retained martensite in the samples, producing residual permanent deformation. The retained martensite coexists with an internal stress distribution change (respect the material without martensite) and different density of defects. These internal stress distribution and density of defects are related to the decreased stress to transform in the cycled samples.

Both changes in properties, residual strain and reduced stress to transform, would produce the reduction in dissipated mechanical energy per cycle. The moderate heating to 100ºC is able to retransform a large part of the retained martensite, producing a change in residual strain. In our as-furnished wires, the residual strain remaining after heating was reduced to 60% of the residual strain after a hundred mechanical cycles to 8% strain. However, the residual strain evolved fastly with further cycling to the previous values, so the use of this technique should be limited to few cycles actions. Overstraining (overstressing) of the wires produced strongly reduced recovery possibilities. A large part of the remaining residual strain should be due to plastic deformation. The retransformation of martensite would give a change in the distribution of internal stresses that recovers partially the transformation stress, and, as a consequence of extended strain span useful and higher transformation stress, the dissipated energy per cycle recovers.

The electrical resistance increase produced by cycling can be interpreted as due to two terms: the appearance of retained martensite, and the defect accumulation (related to plasticity) [22]. The applied thermal treatment relieves retained martensite that retransforms to beta, this quantity increases when the thermal treatment temperature is increased respect to room temperature. In our case, at 100ºC a large part of the retained martensite is relieved. The temperature of the thermal treatment is able to give a partial recovery of the electrical resistance, in a parallel way to the residual deformation reduction.

The very low dependence of the recovery with the time at 100ºC is coherent with a very slow change of defect density with time at 100ºC. In fact, NiTi alloy has been shown to present a very slow change of transformation temperature with time at 100ºC, with representative times of the order of a year [31]. At 130ºC, however, the dependence of recovery with time becomes more effective. The observed near constancy of residual strain on the time, and the increase of the stress to transform on the time at this temperature, are coherent with the strain being determined by retained martensite in the sample, and a slow evolution of defect density with time would relate to the increase of transformation stress with time at 130ºC.

In conclusion, part of the functional fatigue produced by mechanical cycling on NiTi 2.46 diameter wires can be recovered by moderate thermal treatment (to 100ºC, during some hour). However, the degradation of properties with cycling continues after the thermal treatment (see figure 8). The thermal treatment at 100ºC would ease the use of NiTi wires as dampers for extreme situations as earthquake mitigation in civil engineering, because after an event it is easy to recover partly the properties of the implied material.

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