Effects of temperature and deformation mechanism on the fatigue life of R-phase NiTi wires

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ABSTRACT

In this work, the effect of temperature on the fatigue life in a medical grade R-phase NiTi wire was assessed at room- and human-body temperatures. For that, temperature-controlled bending-rotating fatigue tests with a maximum strain of 4% were performed at 25 and 37 °C. A higher mean number of cycles to failure (4495 ± 238) was achieved at 25 °C in comparison to the one observed at 37°C (2791 ± 135). XRD analysis of the cycled samples detected the presence of martensite in the wires tested at 25°C. When evaluating the mechanical properties in tensile tests performed at both temperatures, the shape memory effect was observed at 25°C whereas superelasticity occurred at 37°C. Based on these results, the difference on the fatigue life in the two testing temperatures was related to the change in the functional behavior in the sense that different deformation mechanisms are expected. At 25°C, the martensite induced by tension is stable during unloading and should support the cycling deformation, while at 37°C direct and reverse martensitic transformations shall take place.

Keywords: *NiTi, fatigue, R-phase, shape memory effect, superelasticity,*

INTRODUCTION

Near-equiatomic NiTi alloys are functional materials that present the shape memory effect and superelasticity. These properties are due to the occurrence of a reversible martensitic transformation from a high-temperature B2 parent phase (austenite) to a low-temperature B19' one (martensite). If deformation takes place in a martensitic stability temperature, the shape recovery is achieved by heating the alloy to a temperature at which the structure will be transformed into austenite. When stresses are applied in the austenite, martensite is induced by tension but may be transformed back to austenite with the load removal, which characterizes the superelastic behavior [(1,2)].
Under cooling, the transformation of B2 into B19' may be accompanied with the appearance of an intermediate trigonal phase named R-phase, which is typical of NiTi alloys with excesses of Ni after proper heat-treatment. The presence of this phase has been associated with an improved low-cycle fatigue resistance when compared to the austenitic (B2 structure) alloys [(3–9)]. In fact, in dental applications, heat-treated endodontic NiTi files containing the R-phase have been vastly used due to their superior low-cycle fatigue properties when compared to conventional austenitic files [(10–12)].
In previous works, the fatigue resistance of different heat-treated endodontic files has been evaluated at room and human-body temperature. This comparison is relevant when testing files

that are used intracanal at room temperature. As results, some tested files have shown decreased fatigue lives at human-body, while this property was maintained in others (13–16). In this study, we aim to use medical grade heat-treated NiTi wires to evaluate the R-phase's low-cycle fatigue resistance in relation to their mechanical behavior at room temperature (25°C) and human-body temperature (37°C).

MATERIALS AND METHODS

Heat-treated NiTi wires with 1 mm diameter and a nominal composition 51at%Ni – 49at%Ti were used in this study. From previous investigations (5), the heat treatment was performed in the as-received austenitic wire at 400°C for 30 min in an argon-controlled atmosphere followed by water cooling to obtain the R-phase. To verify the presence of the R-phase, X-ray diffractometry (XRD) tests were performed with Cu-Kα radiation, 2θ ranging from 30° to 90°, and a scan speed of 0.02°/s in a Panalytical Empyrean diffractometer. Differential Scanning Calorimetry (DSC) analysis was performed to assess the direct and reverse martensitic transformation temperatures. The sample was submitted to a heating step from -100°C to 100°C and subsequent cooling back to -100°C at ±10°C/min in a Shimadzu – DSC60. Transformation temperatures were calculated by the tangent method.

Tensile tests were performed under controlled temperature, at 25°C and 37°C, in an Instron 5582 universal testing machine. One sample was subjected to loading to 6% strain followed by unloading to 7 MPa at 3×10^{-4} s^{-1} strain rate to assess the sample’s functional behavior. Another sample was then loaded until rupture at 3×10^{-3} s^{-1} strain rate. These testing conditions were based on the ASTM F2516 – 14 standard (17).

Bending-rotating fatigue tests with a maximum strain of 4% were performed under temperature control as detailed in Ref. (5) at 25°C and 37°C. The same tests were performed in the as-received austenitic wires, which was used as a control group, and one-way analysis of variance (ANOVA) was performed for data analysis at the confidence level $P > 0.05$. XRD analysis was also performed on the samples after the fatigue tests to detect eventual changes in the structure of the alloys because of the cyclic work.

RESULTS AND DISCUSSION

The x-ray diffraction pattern (XRD) and the differential scanning calorimetry (DSC) chart results are presented in Figure 1. The presence of the R-phase (ICSD #155028) was confirmed in the XRD spectra of the as-heat-treated sample, Figure 1-a. In the DSC chart, Figure 1-b, direct and reverse transformation temperatures and the testing temperatures were highlighted. During heating, R’s and R’f are the starting and finishing temperatures of the R phase transformation from B19’ martensite while As and Af are, respectively, the austentic start and finishing transformation temperatures. During cooling, Rs and Rf are the start and finishing R-phase transformation temperatures from B2 austenite. Ms and Mf were not determined because of detection difficulties, as discussed elsewhere (5).

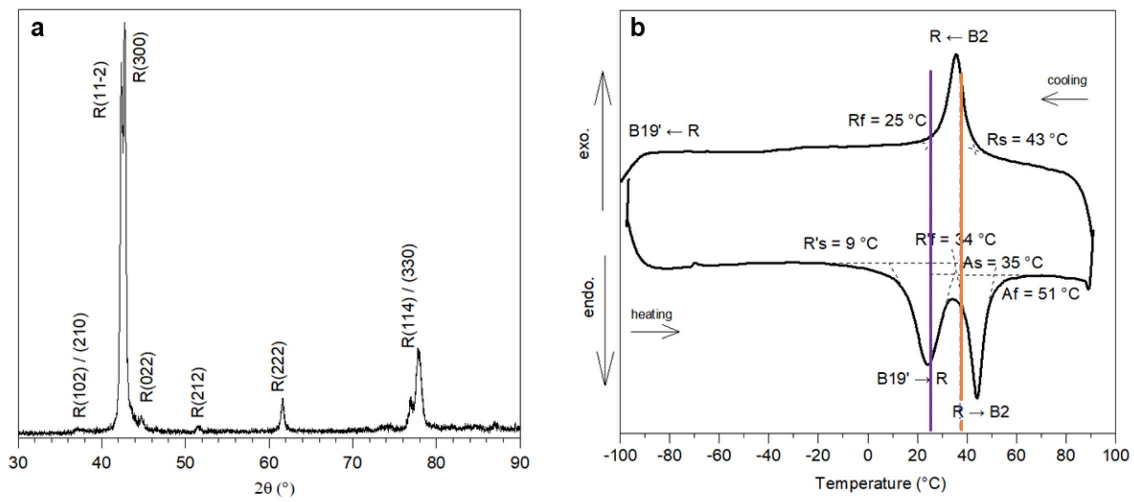

Figure 1 – a) XRD spectra and b) DSC chart of the as-heat-treated sample

Representative stress vs strain curves obtained by the tensile tests are shown in Figure 2. The sample exhibited superelasticity at 37°C whereas only a small amount of recovery was achieved at 25°C. In the later, the shape memory effect was confirmed after heating the sample. Comparing the martensitic transformation stress (σ^{Ms}) measured in the stress plateau of the loading/unloading curve, an increase from 25°C (315 MPa) to 37°C (390 MPa) was observed. On the other hand, no effect on the rupture stress and ductility were observed in the rupture curve.

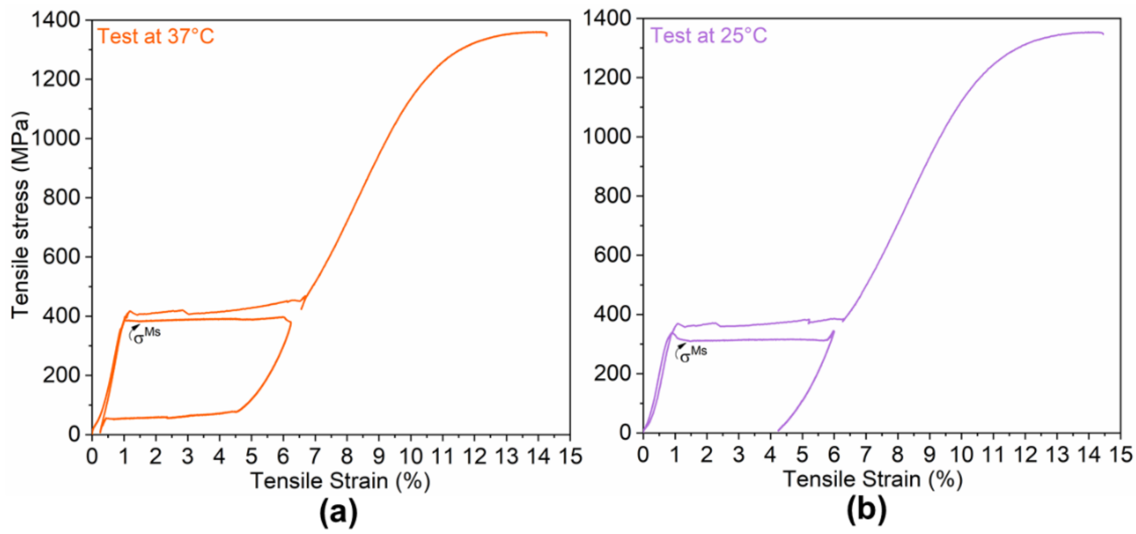

Figure 2 – Tensile test curves obtained at room temperature and human-body temperature

The mean numbers of cycles to failure (Nf) were 4495 ± 238 at 25°C and 2791 ± 135 at 37°C. In the austenitic as-received control group (Af = 19°C (5)), the mean number of cycles to failure were 1596 ± 93 at 25°C and 1257 ± 270 at 37°C. At the 0.05 level, all the fatigue lives are significantly different. The XRD spectra of the R-phase samples fractured during the fatigue tests are shown in Figure 3. B19’ martensite was detected, as indicated by arrows in Figure 3. By comparing the relative intensities of the R-phase and martensitic peaks, higher amounts of martensite are expected in the sample tested at 25°C. This qualitative analysis is made since the wires were tested under the same conditions.

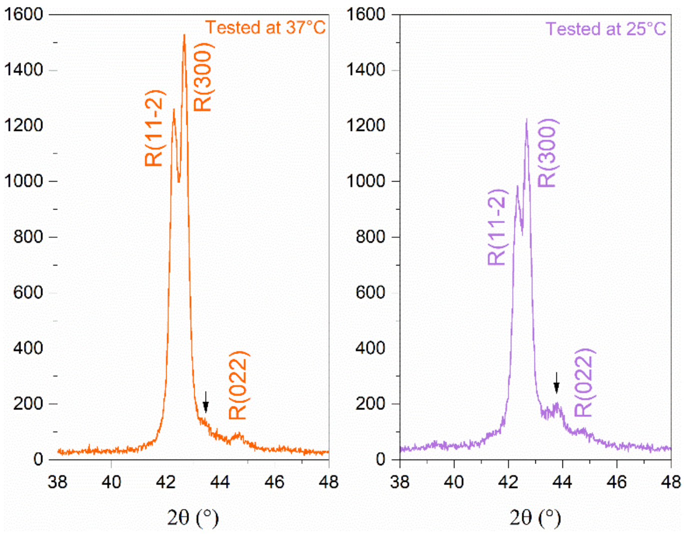

Figure 3 – XRD spectra of wires fractured in the fatigue test at (a) 37°C and (b) 25°C

Variations on the number of cycles to failure in strain-controlled fatigue with temperature may be related to changes in the stress required for starting the induction of martensite by tension. Since it increases with increasing temperature, the sample is subjected to higher stresses when subjected to the same strain [1,18–20]. The increase in the transformation stress is accompanied by virtually no variations on the rupture stress, which favors the occurrence of localized plastic deformation. However, the obtained dependence of the transformation start stress with temperature in the R-phase ($d\sigma^{Ms}/dT$ = 4.4 MPa/°C) is lower than the typical $d\sigma^{Ms}/dT$ for conventional austenitic superelastic NiTi of around 6 MPa/°C [2,20,21]. This is not coherent to the much higher dependence of temperature in the R-phase's fatigue life (dN_f/dT = -145 °C^{-1}) when compared to the as-received austenite (dN_f/dT = -28°C^{-1}) within the two testing temperatures.

On the other hand, a change in the functional behavior of the heat-treated R-phase wires took place. This difference can be explained by the transformation temperatures and stability regions observed in the DSC chart. Room-temperature lies within the B19' → R transformation peak in the heating curve. Thereby, even though martensite was not achieved during cooling after the performed heat-treatment, this phase can remain stable at room temperature when stabilized by tension since 25°C is in a B19' + R stability region. For that reason, the deformation is not recovered during unloading and the shape memory effect is achieved. On the other hand, 37°C is inside of the B2 ↔R transformation peaks regions at cooling and heating. Hence, the martensite induced during loading at 37°C is not stable, only the R-phase and austenite are, and superelasticity is observed.

A change in the functional behavior means that different deformation mechanisms will occur during cycling at the two tested temperatures. At 25°C, from the mechanical characterization, it is expected that the martensite becomes stable in the first cycles because no reverse transformation occurs during unloading, as discussed in Ref. (5). Thus, this phase may support the fatigue cycles by reorienting and deforming elastically. In turn, at 37°C, where superelasticity was obtained, the deformation may be due to the occurrence of direct and reverse martensitic transformation at least in a fraction of the fatigue life. Considering that the flexed

sample is submitted to different maximum strains along its length, this hypothesis is supported by the XRD of the samples fractured in fatigue, where martensite peaks were clear in the sample tested at 25°C.
The superelastic cycle in austenitic NiTi has been associated to a high accumulation of defects [22,23]. Considering the transformation hysteresis observed in the sample tested at 37°C, Figure 2-a, the R-phase wire may undergo a higher defect accumulation on this type of deformation when compared to the variant reorientation and elastic deformation mechanisms proposed at 25°C. This difference can be responsible for the significant decrease in the fatigue life at 37°C and this effect of temperature is not expected if a change in the deformation mechanism does not occur. It is worth pointing out that the number of cycles to failure are still higher than those achieved in austenitic wires even when superelasticity takes place in the R-phase.

CONCLUSIONS

The fatigue life of the R-phase wire in this study was significantly affected by a change in temperature when testing was performed at room-temperature and human-body temperature. This variation was associated to a change in the functional behavior and, hence, deformation mechanism. At 25°C, where the reverse martensitic transformation does not significantly occur during unloading, martensite will likely be the predominant phase in the deformed regions. At 37°C, on the other hand, the superelastic cycle is expected to occur in the R-phase, leading to lower fatigue resistance than the one achieved at room temperature. Variations this large are not expected to come about if a change in the functional behavior and deformation behavior do not occur.

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