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Temperature Harmonic Analysis Of Austenitic Niti Under Dynamic Cyclic Loading

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Abstract – Due to their super-elastic behaviour, NiTi alloys operating above the phase transformation temperature are employed in several high-risk medical devices. Hence, the most advanced approaches shall be employed for their structural characterisation. This work investigates the thermo-mechanical response of NiTi alloys when analysed with infrared thermography. A nitinol strip was subjected to a dynamic sinusoidal loading under displacement control, with increasing average and amplitude ranges, within the austenite elastic state. Analysis of the harmonic content of the temperature signal in the frequency domain was carried out and compared with the predictions of the higher-order thermoelastic effect theory. Results indicate that, although the modulation of temperature signal is related to the sinusoidal load signal, the alloy response is different from traditional materials.

***Keywords*:** super elasticity; austenitic NiTi; temperature harmonic analysis; thermo-mechanical coupling; infrared thermography.

1. Introduction

Nitinol is a nearly equiatomic Nickel-Titanium alloy, which provides unique features essential in a number of high-risk biomedical applications, such as angioplasty stents, stent grafts, vascular occluders, self-expanding transcatheter heart valves and delivery systems. These exploit the material biocompatibility and its superelastic behaviour, i.e. the ability to reversibly undergo large deformations of the order of 8 %, thanks to the stress-induced phase transformation from austenite to martensite (Stress-induced Martensite Transformation, SIMT).

Some advanced characterisation techniques have been adopted for a more effective study of nitinol, and the use of infrared thermography (IR-T) has recently emerged as a powerful tool to understand the thermo-mechanical response of the material under quasi-static or cyclic loading conditions. IR-T has been used to evaluate the progression of superelastic nitinol phase transformations [1]–[4], monitoring the onset and evolution of phase transformation fronts. In fact, the latent heat of transformation introduces a marked signature on the temperature map, which can be well detected by infrared cameras. In particular, the material releases heat in the stress induced transformation from the austenitic crystal configuration (stable at the operating temperature) to the martensitic phase, and absorbs heat when returning from martensite to austenite.

The release/absorption of latent heat during SIMT (*elastocaloric effect*) produces a reversible temperature fluctuation [5]. This overlaps with another source of reversible temperature fluctuation, i.e. the thermoelastic effect associated with the elastic volume change in solid matter. Both these intrinsic heat sources are activated by loading and, therefore, a temperature modulation is expected when a modulated load is applied, such as in fatigue loading scenarios [6], [7].

In this work, the thermoelastic effect is investigated in isolation, by analysing the temperature modulation features of a nitinol strip subjected to traction-traction sinusoidal loading, but maintaining it in the austenitic state, i.e. before the onset of the austenite-to-martensite phase transformation stress. The temperature was sampled with a cooled-sensor IR-camera, and the harmonic content was retrieved with Discrete Fourier Transform (DFT) signal processing. The peculiar thermal behaviour of the material required the adoption of a model based on the higher-order thermoelastic effect theory [6].

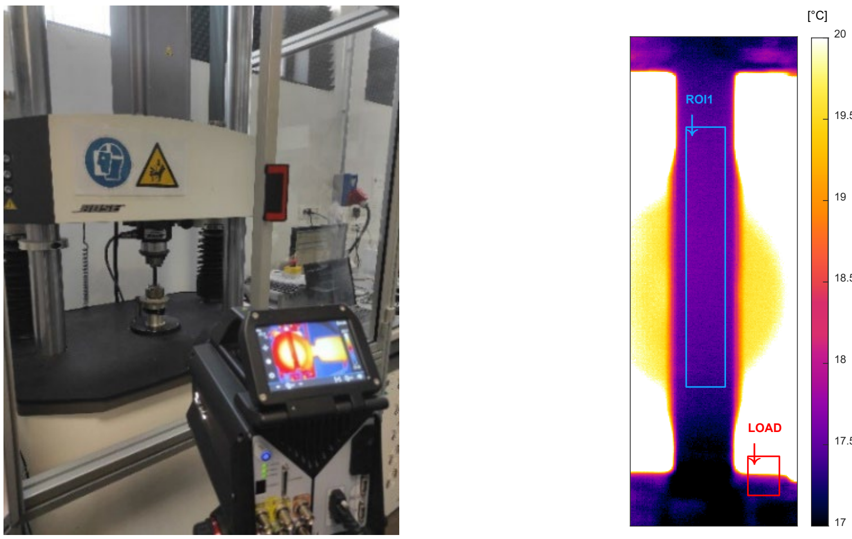
2. Material and method

A strip of 100 mm length and 10 mm width was cut from a 0.4 mm thick NiTi plate by means of electrical discharge machining, so as to minimise the onset of thermo-mechanically altered zones during machining. The flat specimen surface simplifies the implementation of full-field non-contact IR-T technique.

The material phase transformation temperatures were identified by differential scanning calorimetry (DSC) analysis, following ASTM F2004, in order to confirm that austenite was stable at room temperature. Moreover, the specimen was initially pretested applying a quasi-static load, at a rate of 0.5 mm/min, in order to identify the phase transformation stresses. To improve emissivity and reduce reflection during the thermographic analysis, one face of the sample was uniformly coated with matt black paint.

The sample was then dynamically tested on an electrodynamic testing machine BOSE traction-torsion 3550 (TA Instruments, UK), applying cyclic sinusoidal loading at 1 Hz, under displacement control, in different blocks each comprising 60 load cycles. The cyclic loading blocks were applied with increasing average and amplitude displacement, maintaining the maximum stress below the stress initiating the phase transformation (upper plateau stress). In particular, cycle 1 was set up between 0.1 and 0.15 mm, cycle 2 between 0.1 and 0.2 mm, cycle 3 between 0.1 and 0.25 mm and cycle 4 between 0.1 and 0.3 mm. Tests were carried out at room temperature (T_0=20 °C).

Full field thermograms were acquired during each block of cyclic loading, at a sampling frequency of 50 Hz, using a FLIR X6540SC thermal camera (Teledyne FLIR LLC, UK). The experimental setup is shown in Figure 1.a.



(a) (b)

Figure 1. a) experimental setup; b) example of an acquired thermogram and the regions of interest ROIs set to analyse average temperature and load signals.

The harmonic analysis of the discrete temperature signals was carried out on a specifically written Matlab script implementing the DFT, determining the amplitude-frequency spectra.

A signal representative of the loading wave was extrapolated by selecting a region of interest (ROI) between the moving grip of the testing machine and the background (ROI LOAD shown in Figure 1.b). The different temperature between the moving grip and the background was used to obtain a modulated average temperature signal representative of the applied loading wave. The temperature signal of the sample was averaged over the ROI1 shown in Figure 1.b.

Previous experimental studies [6], [7] have shown that, contrary to standard materials, the complexity of the thermo-mechanical behaviour of nitinol requires for its interpretation the so called higher-order thermoelastic law, which under a uniaxial loading can be expressed as in Eq. (1):

$$\Delta T = -T_0\sigma_a[k_0 - k_1\sigma_m]\sin(\omega t) - T_0{\sigma_a}^2[k_2]\cos(2\omega t) \tag{1}$$

where σ_m and σ_a are the mean and amplitude of the stress, respectively, $k_0 = \frac{\alpha}{\rho c_\varepsilon}$, $k_1 = \frac{1}{E^2}\frac{\partial E}{\partial T}\frac{1}{\rho c_\varepsilon}$ and $k_2 = \frac{1}{4E^2}\frac{\partial E}{\partial T}$, with E corresponding to the material Young's modulus, α to the thermal expansion coefficient, ρ to the material density and c_ε to the specific heat at constant volume. Eq. (1) comprises of the sum of two terms: the first one oscillates at the same frequency as the load (ω), and the second one oscillates faster, at a frequency equal to 2ω.

3. Results and Discussion

DSC and pre-test results indicated an austenite finish temperature equal to 5.1 °C and an austenite-martensite transformation starting at a stress of about 500 MPa, corresponding to a strain of about 0.7 %. These confirm that austenite is stable at the test temperature and under all adopted crosshead displacements. The average temperature, load waves and related amplitude spectra for each applied block of cyclic loadings are shown in Figure 2.

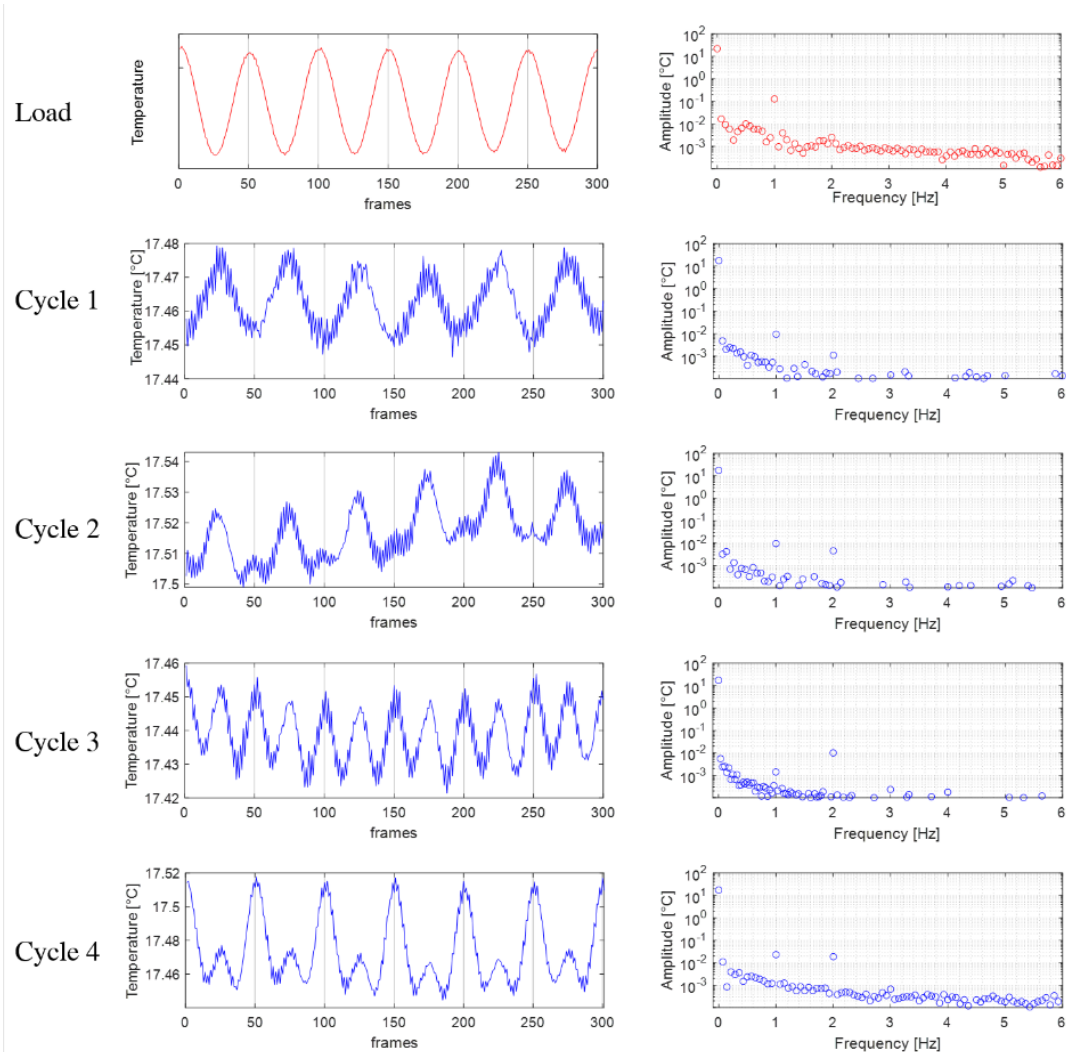


Figure 2. Normalised temperature signal of the LOAD and temperature signal of ROI1 on the left and relative amplitude spectra on the right.

In cycle 1 (second row in Figure 2), the temperature measured in ROI1 decreases when the load increases, showing an opposite phase with respect to the load signal. This is in agreement with the behaviour commonly observed with conventional materials. As the load amplitude increases, in cycle 2 (third row in Figure 2), the temperature signal presents a major peak in phase opposition with the load signal, but as the load increases, the temperature inverts its trend, departing from the expected sinusoidal wave, and forming a small local peak in phase with the load wave. The growth of this in-phase peak determines the rise of a strong second harmonic component at 2 Hz, well visible in the amplitude spectrum.

In cycle 3 (fourth row in Figure 2), the in-phase temperature peak grows, reaching similar amplitude as the out-of-phase peak. In this configuration, the second harmonic has higher amplitude than the first harmonic.

In cycle 4 the peak in phase with the load wave becomes the dominant peak, while the out-of-phase peak is significantly reduced. Therefore, the peak of the temperature signal in phase with the load signal becomes predominant over the peak in opposite-phase. Hence, it is clear that the temperature signal, in this stage, is mainly modulated in phase with the load signal. This effect could be interpreted due to different phenomena. A possible justification could be that, as the mean stress σ_m increases over a certain threshold, $\partial E/\partial T$ becomes larger than $E^2\alpha/\sigma_m$ [7].Therefore, the first term of Eq. (1) switches its sign from negative to positive. Another explanation could be that the latent heat of phase transformation starts to manifest prior than the local SIMT transformation, producing a uniform and significant temperature increase that is in phase with the load.

4. Conclusion

This work adopts a full-field, non-contact infrared thermography technique with the aim of studying the thermomechanical response of nitinol samples under cyclic loading. The approach is based on the harmonic evaluation of the temperature signal under sinusoidal loading at increasing amplitude and average strain values.

In particular, tests carried out in the elastic fully austenite zone show that, at a certain value of applied load, the material changes its thermal behaviour. In fact, the material initially behaves as conventional materials, with temperature modulated in opposite-phase to the load wave. Exceeding this loading value, the behaviour changes showing a temperature signal becoming gradually in-phase with the load. This response, under certain assumptions, can be described by the higher-order law of the thermoelastic effect.

The presented approach offers a new perspective to evaluate NiTi thermomechanical behaviour, that can have a major impact in fatigue characterisation studies, both at a bulk-material level or prosthetic device level. It is worth to observe that common nitinol medical devices, under typical operating conditions, are essentially in the austenitic phase with exception of small, confined regions. A more informed monitoring of the fatigue thermo-mechanical behaviour of NiTi-based collapsible biomedical devices can be of great relevance to improve the safety of these applications.

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