

Differential Scanning Calorimetry of Superelastic Nitinol for Tuneable Devices

A. Feeney^a and M. Lucas

School of Engineering, University of Glasgow, Glasgow, G12 8QQ, U.K.

^aandrew.feeney@glasgow.ac.uk

Abstract. Nitinol has been used to fabricate tuneable-frequency cymbal transducers by exploiting its solid-state phase transformation capability. The temperatures at which Nitinol transforms are commonly measured using differential scanning calorimetry (DSC). However, these measurements are shown to be inaccurate for superelastic Nitinol, reportedly attributed to residual stresses in the material resulting from the fabrication process. This study of DSC accuracy is conducted for untreated and heat-treated superelastic Nitinol. Cymbal transducers are fabricated from both materials and it is demonstrated, through comparison of results from DSC estimations of transformation temperatures with the vibration response measured through a heating cycle, that heat-treated superelastic Nitinol can be accurately characterised by DSC.

Introduction

Nitinol is a binary alloy of nickel and titanium which transforms in response to either heating stress or temperature [1]. The higher-temperature phase is a relatively stiff cubic austenite, and the lower-temperature phase is a more compliant monoclinic martensite. In certain cases, an intermediate rhombohedral R-phase can appear [2]. These phases are each characterised by start and finish (s and f) transformation temperatures (A , M , and R) which can be measured from endothermic (austenite) and exothermic (martensitic) events in DSC thermograms. Two principal classes of Nitinol exist, one being superelastic and the other shape memory. The A_f of shape memory Nitinol is generally far above room temperature, whereas the A_f of superelastic Nitinol is either below or near room temperature. The DSC method can be inaccurate for superelastic Nitinol, although the reasons for this are not completely clear [3]. Therefore, this research aims to improve the effectiveness of DSC for superelastic Nitinol, by conducting an annealing heat treatment for two hours at 450°C [4]. The intention is to relieve a portion of the residual stresses which have been attributed to the inaccuracy of DSC. Subsequently, two cymbal transducers are fabricated, one with end-caps of untreated superelastic Nitinol and the other with end-caps of annealed superelastic Nitinol. Vibration analysis of the two transducers is carried out through a heating cycle, to identify the transformation temperatures from the temperatures at which the modal frequencies change, and compare these with DSC estimations. Electrical impedance measurements (Agilent 4294A) were used to monitor the frequency response.

Differential Scanning Calorimetry

The transformation temperatures of the treated and untreated Nitinol measured from DSC (Perkin Elmer Diamond) at two heating rates are shown in Fig. 1. The upper curve for each data set indicates an endothermic transformation event, signifying a transition to an austenitic microstructure. The exothermic transformation event is characterised by the lower curve, representing a transition to martensite.

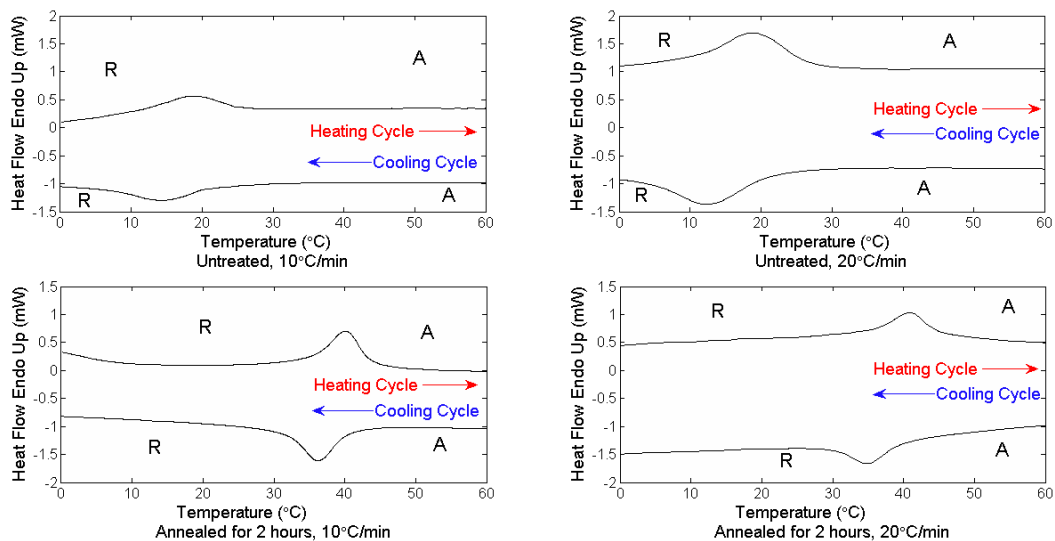


Fig. 1: Transformation temperatures of superelastic Nitinol from DSC.

The temperature gap between the endothermic and exothermic events is too small to be a transition between austenite and martensite [2] and therefore the lower temperature phase is the R-phase. The A_f is the

temperature at which the transformation event finishes on the heating cycle, having completely transformed from R-phase to austenite. The DSC thermogram results therefore show that the A_F of the annealed Nitinol is higher than for the untreated Nitinol.

Comparison of Cymbal Transducer Vibration Responses

Each transducer comprised cymbal-shaped Nitinol end-caps bonded to a piezoceramic disc using an insulating epoxy resin. A Nitinol cymbal transducer is shown in the inset of Fig. 2. The diameter of each transducer was approximately 12.7mm. The radial motion of the piezoceramic disc induces an axial displacement in each end-cap. There are two modes of vibration of interest, one being symmetric, characterised by out-of-phase end-cap oscillation, the second being asymmetric, where the motion is in-phase. The resonant frequencies measured for both transducers measured from electrical impedance analysis exhibited a rapid increase in the heating cycle, stabilising at approximately 40-45°C, suggesting the A_F for both untreated and annealed superelastic Nitinol is in this temperature range. The changes in resonant frequency for the modes of vibration for the transducers are shown in Fig. 2.

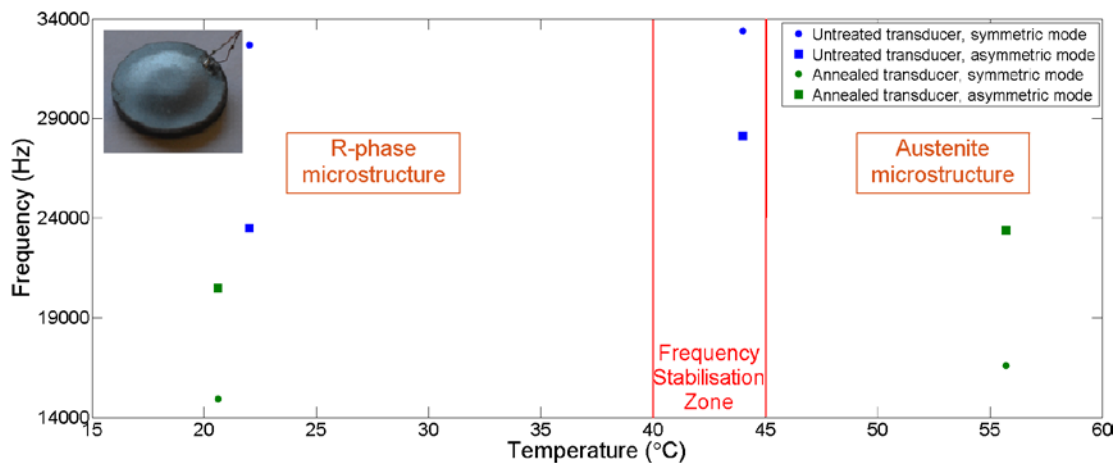


Fig. 2: The changes in resonant frequency for the modes of vibration of both transducers, and (inset) a superelastic Nitinol cymbal transducer.

For both modes of vibration for each transducer, a significant increase in the resonant frequencies has been measured, corresponding to the phase transformation of the Nitinol end-caps. The data shown in Fig. 2 and the DSC results presented in the thermograms in Fig. 1 together demonstrate that the DSC measurement of A_F is inaccurate for the untreated material, where the resonant frequencies continue to rise after the temperature measured to be A_F from DSC has been passed. However, it has been found that the A_F for the annealed Nitinol is very close to the temperature required to transform the resonant frequencies of the transducer with annealed Nitinol end-caps to austenite, based on the change in resonant frequencies of the annealed Nitinol transducer. There is hence a high level of correlation between the DSC and vibration response characterisation methods for the transducer fabricated using annealed superelastic Nitinol, but not for the untreated superelastic Nitinol. This suggests that the condition of the superelastic Nitinol is critical for accurate transformation temperature measurement, and DSC should be used with caution in the analysis of this material.

Conclusion

It has been demonstrated that the DSC method is unreliable for measurement of the transformation temperatures of superelastic Nitinol, but that the accuracy can be improved by annealing. There was a high level of correlation between the data obtained from the vibration analysis techniques used on the annealed transducer, and the resonant frequencies were shown to shift at a temperature close to the A_F transformation temperature measured using DSC. This is contrary to the observations of the transducer fabricated from untreated superelastic Nitinol. It is therefore vital to couple vibration characterisation techniques with DSC to enable the effective future design of tuneable Nitinol devices.

References

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