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Correlation between Differential Scanning Calorimetry and Bend Free Recovery Test Methods when Evaluating Nitinol Wire Austenite Finish Transformation Temperature

Emily Nagel, Roger Dickenson, Marie Moloney, Lee Roach, and Sahit Bollam
Integer Holdings Corporation, Chaska, Minnesota, USA

Introduction

There are two primary test methods used in industry to evaluate nitinol phase transformations, bend free recovery (BFR) per ASTM F2082 [1] and differential scanning calorimetry (DSC) per ASTM F2004 [2]. Both methods are widely used, but the correlation between the method outputs for Austenite Finish (Af) temperature is not fully characterized. This can be challenging when different methods are specified or preferred by suppliers, customers, and manufacturers. DSC is a method that measures the change in energy as a function of temperature; it is a strictly thermal method. Whereas BFR monitors the displacement of a sample as temperature increases. There is a lack of published data discussing how different wire processing inputs impact the correlation between these two outputs.

This study will analyze the correlation between BFR and DSC test data for nitinol wires that have undergone various heat treatments in temperature ranges of 300°C-500°C and durations of 0.5 min – 240 min. How different factors contribute to the correlation of the methods will also be discussed.

Methods

Raw Material
The raw material for this study is 0.016” diameter nitinol #1 wire purchased in the straight annealed condition. The wire is specified to have certain mechanical, thermal, and cold work levels (40-50%).
Processing
Samples were heat treated in a sand bath across a temperature range of 300°C-500°C for durations of 0.5 min – 240 min followed by a 30 second quench in water.
Testing
One heat treated sample from each thermal condition, along with an untreated as-received wire were then tested per BFR method (ASTM F2082) and DSC method (ASTM F2004). Note on BFR test limitation: The BFR test method used does not operate above 35°C due to safety concerns related to the alcohol-based medium. This has caused a gap in data for some samples heat treated at 300°C and 400°C because of an incomplete transformation. The method was developed to align with ASTM F2082 for guidewires with an expected active Af value below room temperature.

Experimental Results

Results
The Af results are summarized in Fig. 1.

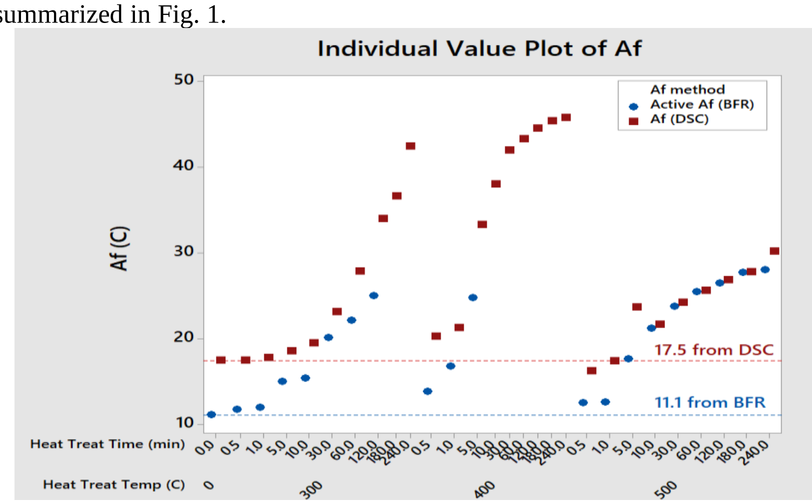

Figure 1: Summary of Af Results

Discussion

There are likely multiple factors that contribute to how closely the DSC and BFR data correlate. These factors are discussed further below.

Retained Cold Work: There is a trend where decreasing amounts of retained cold work aligns with a stronger correlation between test methods. Some mechanisms that may explain this trend include (1) that higher dislocation densities impede phase transformations, resulting in a transformation that occurs over a longer temperature region [3], (2) higher levels of retained cold work results in a more strained microstructure; stabilizing r-phase and demonstrating more resistance to applied stresses [3], and (3) higher heat treat time/temperature combinations causes significant release of stored energy within wire and removal of dislocations.

Transforming stress induced martensite vs thermal martensite to austenite: During BFR testing, the sample is exposed to bending strains which forms stress induced martensite (detwinned) in addition to thermal cold martensite (twinned). BFR testing results in the presence of tension/compression asymmetry within the deformed area [4]. Areas in tension will exhibit strain localization, while areas in compression will not [5].

Presence of r-phase: Previous work identified a correlation between the DSC values for Rf with active Af BFR values when r-phase was present in shape memory alloys [6]. Data from the present study suggests that r-phase can be present during both BFR and DSC testing while Af outputs have a range of correlations. Additionally, with a higher lower bound cooling temperature, it is possible that BFR testing does not fully transform all samples to thermal martensite, so the martensite that transforms will be stress-induced [4].

Conclusions

It is reasonable to expect that the BFR and DSC tests will result in different outputs as they are different methods. The presence of higher amounts of retained cold work in samples correlates with a larger difference between method outputs. Higher amounts of retained cold work (dislocations) cause the transformation to austenite (during heating) to take place over a larger temperature range for both methods. The presence of higher amounts of retained cold work creates a "baseline" microstructure that will then be more impacted by deformation in BFR testing and formation of stress induced martensite.

Acknowledgments

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