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Characterization of Transformation Temperatures with the Bend and Free Recovery Technique: Parameters and Effects

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This study was conducted to investigate factors such as the deformation strain, straining temperature, and the number of testing cycles on the measurement of transformation temperatures using the bend and free recovery (BFR) technique. Ti-56.0%Ni wire with approximately 40% cold work and a 2 mm diameter was heat treated in an air furnace for 10 min at 490 °C to obtain an A_f of approximately 21 °C. Wire specimens were deformed with one of two mandrels to apply an outer fiber strain of 2.4 or 5.8%. Deformation was performed at one of four straining temperatures: 0, −30, −50, or −65 °C. Specimens were tested ten times to investigate the effect of repeated testing. The resulting BFR curves were analyzed to determine the trends in the R-phase start (R'_s) and austenite finish (A_f) temperatures. For specimens strained at both 2.4 and 5.8% there was no detectable change in A_f resulting from changes in the deformation temperature. Increasing the deformation strain from 2.4 to 5.8% tended to increase the measured A_f by approximately 1 °C independent of deformation temperature. Repeat testing seemed to result in a slight increase in A_f but the intrinsic scatter of the BFR data made it impossible to conclusively identify a trend.

Keywords intermetallics, mechanical testing, nondestructive testing

1. Introduction

When working with Nitinol, the ability to control and monitor transformation temperatures is necessary. Heat treatments can be used to alter transformation temperatures through the activation of Ni-rich precipitation and dissolution processes allowing the tailoring of the thermo-mechanical properties (Ref 1). These same heat treatments can provide enough energy to activate recovery and recrystallization processes, which will further alter the material properties (Ref 2).

The bend and free recovery (BFR) technique, as described in ASTM F2082-06 *Standard Test Method for Determination of Transformation Temperature of Nickel-Titanium Shape Memory Alloys by Bend and Free Recovery*, is widely used in the medical device industry to determine the transformation temperatures of incoming material, partially processed devices, and finished devices (Ref 3). The standard outlines a procedure for determining the austenite start (A_s) and austenite finish (A_f) temperatures for one-stage transformations and the rhombohedral, or R-phase, start (R'_s) and R-phase finish (R'_f) temperatures in the case of two-stage transformations. The current version of F2082-06 leaves some questions regarding testing parameters unanswered. The deformation temperature suggested for all room-temperature superelastic materials is −55 °C or lower. At these temperatures the standard refers to the material as "nominally fully martensitic". Studies have found that cold-worked and heat-treated material can have martensite start (M_s) and martensite finish (M_f) temperatures below −60 and −100 °C, respectively, suggesting that some samples cooled to −55 °C will not be in the fully martensitic state and most, if not all, of the martensite that is present after deformation will have been stress-induced (Ref 4). The F2082-06 standard also does not address the possibility of repeatedly testing the same part and any changes in the measured transformation temperatures that may or may not result. And finally, the standard asks for a deformation strain of between 2 and 2.5%. A previous study by Chen et al. has suggested that higher deformation strains will result in an A_f increase (Ref 5). Device manufacturers are often limited in their ability to control strain levels when testing devices with complicated geometries. This study was conducted to help understand the effects of deformation temperature, repeated testing, and deformation strain on the resulting transformation temperatures measured.

2. Experimental Methodology

As-drawn Ti-56.0at.%Ni wire of 2 mm diameter with approximately 40% cold work was heat-treated at 490 °C for 10 min. Heat treatment was performed in an air furnace followed by a water quench. Transformational properties were characterized by the BFR technique based on ASTM F2082-06. Figure 1 shows a schematic illustration of a typical BFR tester.

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The F2129-06 test protocol outlines a procedure for cooling a room-temperature superelastic specimen in a liquid bath (a 90% isopropyl alcohol bath was used in this case) to a temperature of less than −55 °C, deforming the specimen to an outer fiber strain between 2 and 2.5%, then heating the bath at a rate of no more than 4 °C/min. Temperature and displacement are monitored as the specimen recovers its original, undeformed, shape due to the shape memory effect (SME). A typical two-stage BFR curve from this study fit for determination of R'_s and A_f is shown in Fig. 2.

To investigate the effects of various parameters on the resulting transformation temperature measurements, deviations from F2082-06 were taken:

- Instead of cooling to below −55 °C, deformations were performed at −65, −50, −30, and 0 °C.
- Instead of the prescribed 2 to 2.5% outer fiber strain, strains of either 2.4 or 5.8% were applied to the wire specimens before testing.

Additionally, there were three repeat specimens for each test condition and each specimen was tested 10 times to investigate the effects of repeat testing on the measured transformation temperatures. The six specimens deformed at −65 °C were only tested twice each because the alcohol bath was viscous and opaque at this temperature, making testing difficult and time consuming. Table 1 outlines the experimental matrix.

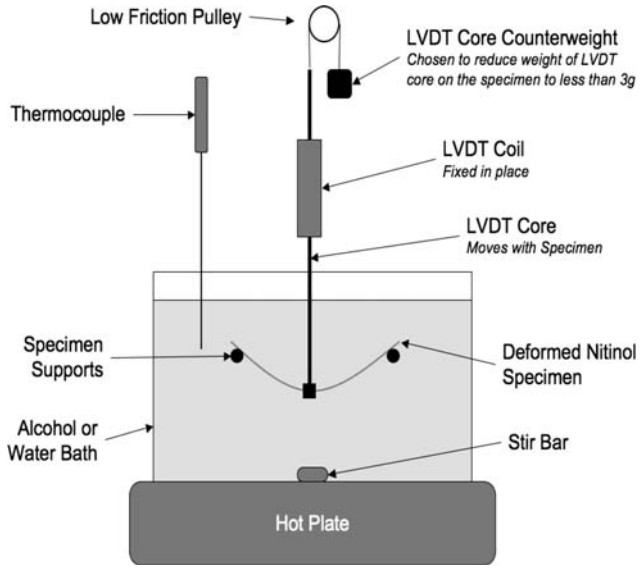

Fig. 1 BFR test apparatus

3. Results

The thermal energy provided by a 10-min heat treatment at 490 °C has been shown to enable the SME through the activation of annealing processes, maintaining a significant amount of residual cold work, and increasing the A_f to around

Table 1 Experimental matrix

Deformation temperature, °C	Deformation strain	Specimens	Repeat testing cycles	Number of tests
0	2.4	3	10	30
	5.8	3	10	30
−30	2.4	3	10	30
	5.8	3	10	30
−50	2.4	3	10	30
	5.8	3	10	30
−65	2.4	3	2	6
	5.8	3	2	6
Total		24		192

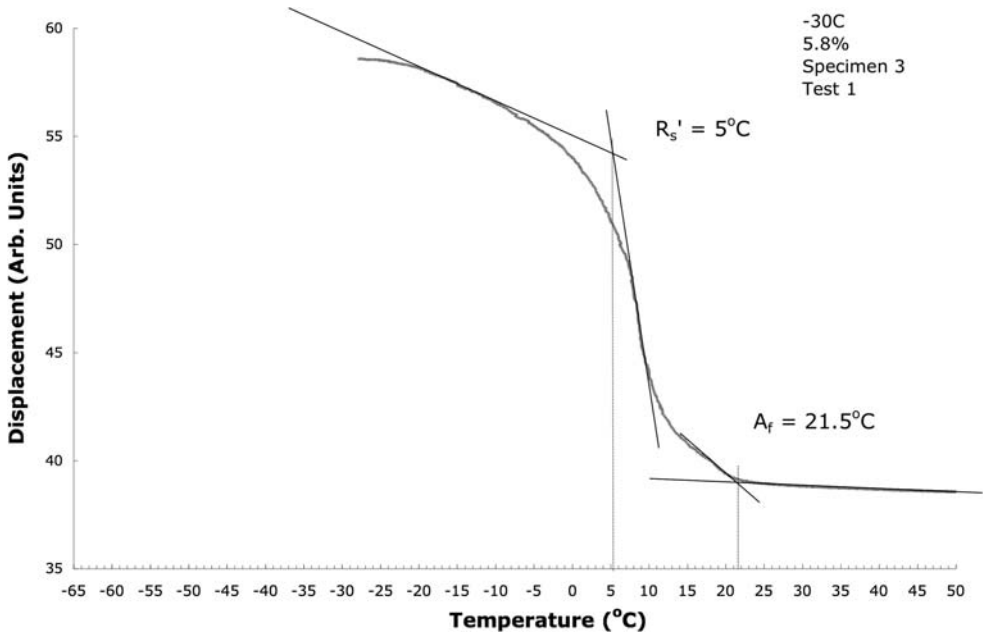

Fig. 2 Typical two-stage transformation with tangent lines fit for determination of R'_s and A_f

Table 2 A_f **data summary**

Strain, %	Def. Temp., °C	Average A_f of three specimens for each of 10 repeat tests (standard deviation of three specimens)										Average of all tests	Standard deviation of all tests
		1	2	3	4	5	6	7	8	9	10		
2.4	0	20.5 (0.5)	20.5 (0.5)	20.8 (1.0)	20.5 (0.9)	21.0 (1.0)	20.2 (0.3)	20.7 (0.3)	20.7 (0.8)	20.5 (1.3)	20.7 (0.6)	20.6	0.7
5.8	0	21.5 (0.5)	22.2 (0.3)	22.0 (0.0)	21.8 (0.3)	22.0 (0.0)	22.0 (0.0)	21.5 (0.5)	22.0 (0.5)	21.7 (0.6)	22.0 (0.5)	21.9	0.4
2.4	−30	20.2 (0.3)	21.2 (0.3)	21.5 (0.5)	21.2 (0.8)	21.5 (0.5)	20.7 (0.6)	20.8 (0.3)	21.3 (0.6)	20.8 (0.8)	21.0 (0.0)	21.0	0.6
5.8	−30	21.8 (1.0)	22.3 (0.8)	22.3 (1.0)	22.0 (0.5)	22.0 (1.0)	22.2 (0.3)	21.3 (0.6)	21.7 (0.8)	21.7 (0.8)	22.0 (0.5)	22.2	0.7
2.4	−50	20.2 (0.3)	20.7 (0.6)	20.5 (0.5)	20.0 (0.5)	20.2 (0.3)	20.2 (0.6)	20.3 (0.3)	20.0 (0.5)	20.0 (0.0)	20.0 (1.0)	20.4	0.5
5.8	−50	21.0 (1.0)	21.7 (0.6)	22.0 (1.0)	22.0 (1.0)	22.0 (1.0)	22.5 (0.5)	21.3 (0.3)	22.0 (0.5)	21.7 (0.6)	22.0 (1.0)	21.7	0.8
2.4	−65	20.3 (0.6)	20.5 (0.9)	…	…	…	…	…	…	…	…	20.4	0.7
5.8	−65	21.0 (1.0)	21.0 (1.3)	…	…	…	…	…	…	…	…	21.0	1.0

Table 3 R'_s **data summary**

Strain, %	Def. Temp., °C	Average R'_s of three specimens for each of 10 repeat tests (standard deviation of three specimens)										Average of all tests	Standard deviation of all tests
		1	2	3	4	5	6	7	8	9	10		
2.4	−30	1.7 (2.3)	2.3 (3.1)	3.0 (2.6)	3.0 (3.0)	3.0 (2.6)	2.3 (4.7)	2.0 (3.5)	2.0 (3.6)	1.7 (3.2)	2.3 (3.8)	1.7	2.8
5.8	−30	7.3 (3.2)	7.7 (3.1)	7.7 (3.5)	8.0 (3.0)	7.7 (3.1)	6.7 (2.9)	4.3 (4.5)	5.0 (3.6)	5.3 (3.2)	5.7 (3.1)	7.5	3.1
2.4	−50	−0.3 (1.2)	−0.7 (0.6)	−0.3 (1.2)	−0.7 (0.6)	0.0 (1.0)	0.3 (1.5)	0.3 (1.2)	0.3 (0.6)	−0.3 (0.6)	0.0 (1.7)	0.2	1.0
5.8	−50	5.7 (4.0)	6.7 (4.0)	6.7 (4.2)	6.7 (3.2)	6.3 (3.8)	7.0 (2.6)	6.7 (2.3)	7.0 (3.5)	6.3 (4.6)	6.3 (3.8)	5.7	3.1
2.4	−65	−3.0 (3.0)	−2.7 (3.2)	…	…	…	…	…	…	…	…	−2.8	2.8
5.8	−65	1.7 (3.8)	2.7 (4.6)	…	…	…	…	…	…	…	…	2.2	3.8

21 °C through the precipitation of Ni-rich phases (Ref 1, 2). These heat treatment parameters were chosen because they are similar to those often used by medical device manufactures who want a balance of strength, mechanical stability, and superelastic recovery. Heat treatments of as-drawn wire providing approximately this amount of thermal input often generate a microstructure that stabilizes the R-phase (Ref 2, 6, 7). R-phase stabilization can result in a two-stage transformation during heating. The F2082-06 standard identifies the starting temperature of a two-stage transformation, i.e. the start of the martensite-to-R-phase transformation, as R'_s, and the temperature corresponding to the end the transformation, i.e. the end of the R-phase-to-austenite transformation, as A_f.

The average R'_s and A_f values for each test condition and the corresponding standard deviations are presented in Table 2 and 3. Also provided in Table 2 and 3 are the average and standard deviations for each deformation strain and deformation temperature, neglecting the effects of repeat testing. R'_s values could not be determined for specimens deformed at 0 °C because there was no onset to these transformation curves to allow fitting of an upper tangent line. The M_s temperature of this material was determined to be −63 °C with differential scanning calorimetry (DSC), indicating that nearly all the martensite phase in these specimens was stress-induced and not thermally induced.

Determining trends with the BFR technique is limited by the intrinsic resolution of the test. For this data set the standard deviation values for the samples ranged from 0 to 1.3 °C with an average of 0.6 °C for A_f data and from 0.6 to 4.7 °C with an average of 2.8 °C for R'_s data (with a sample size of three).

Although there was a slight increase in the average A_f and R'_s values after ten repeat tests (0.6 and 0.1 °C, respectively), these increases were too small to conclusively identify as a trend. A larger sample size and increased number of tests may confirm the trend.

Comparing the average A_f from the first test of each deformation temperature and strain condition illustrates an increase in A_f with increased deformation strain, shown in Fig. 3. Although the error bars overlap for several of the strain and deformation temperature conditions, there is a consistent increase in A_f with increased deformation strain at all deformation temperatures. These A_f increases range from 0.7 °C at a deformation temperature of −65 to 1.6 °C at a deformation temperature of −30 °C. The average increase in A_f with increased deformation strain, from 2.4 to 5.8%, for the first test at all deformation temperatures was 1.0 °C.

Also shown in Fig. 3, there was no statistically significant change in A_f with increasing deformation temperature for either level of deformation strain. The A_f differences that can be seen in Fig. 3 are less than the noise level of the BFR technique. In an effort to increase the sample size, a similar plot was constructed using all the data from each specimen, i.e. ignoring any effects from repeat testing, and the same results were observed.

The measured R'_s values increased with both increasing strain and increasing deformation temperature, shown in Fig. 4. Again these increases have overlapping error bars but the consistency and underlying reasoning, as will be described in the next section, are considered sufficient to verify them. The average increase in R'_s as a result of increased strain, from 2.4 to 5.8%, was 5.6 °C. The increase in R'_s as a result of increasing the deformation temperature from −65 to −30 °C was about 5 °C for both deformation strains.

4. Discussion

The small standard deviation of the A_f values measured in this study is evidence that BFR testing is a reliable and repeatable means of characterizing the transformation temperatures of Nitinol. The higher amount of deviation in the R'_s data is largely a result of the difficulty in fitting a straight tangent line in a consistent manner to the curved onset of the transformation.

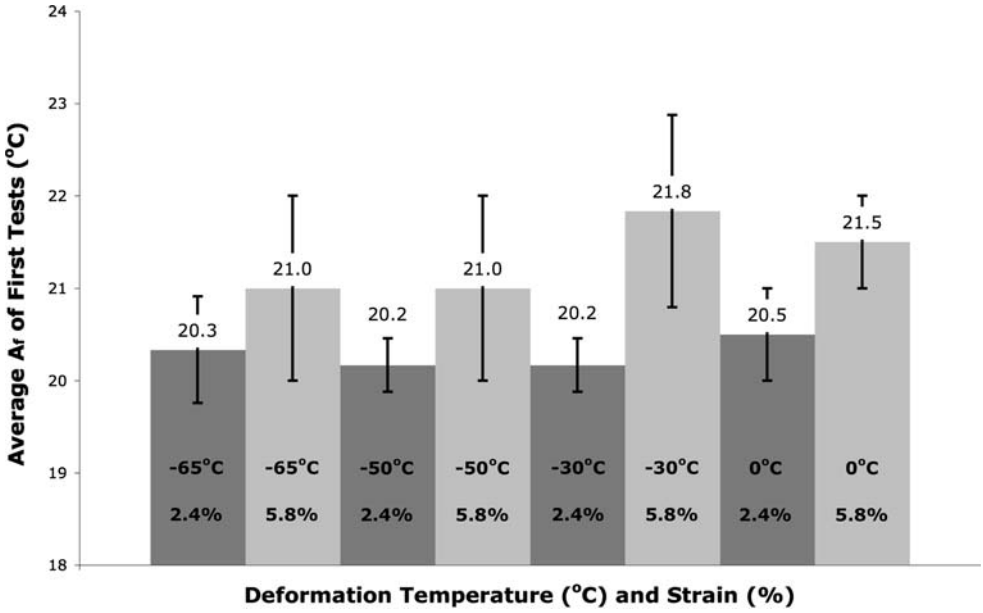

Fig. 3 Average A_f of the first test for each strain and deformation temperature condition. Two standard deviation error bars are shown

The increases in R'_s and A_f can be explained by considering a change in the character of the BFR curves. This change in character resulted from the larger amount of stress-induced martensite in specimens with 5.8% deformation strain. Figure 5 shows the BFR curves from specimens deformed 2.4 and 5.8% at −65 °C. As expected, the more heavily strained specimen has a larger amount of displacement, about twice as large in this case. This increase amount of stress-induced martensite in the 5.8% deformed specimens resulted in a steeper recovery curve in the martensite-to-R-phase region, which resulted in consistently fitting a steeper tangent line to the curve—consistently increasing the measured R'_s values. Although the A_f temperatures of the two BFR curves shown in Fig. 5 seem to be the same this is because the shift in A_f is subtle. When these BFR curves are fit in practice, they are usually scaled to fill the entire chart area, as shown in Fig. 6. When the 2.4% displacement curve is scaled to fill the entire displacement axis, the R-phase-to-austenite transformation is distorted, increasing its slope, as a result of the relatively small displacement from the martensite-to-R-phase transformation when compared with the 5.8% displacement curve. This change in scaling also resulted in an increase in the slope of onset and end of the transformations.

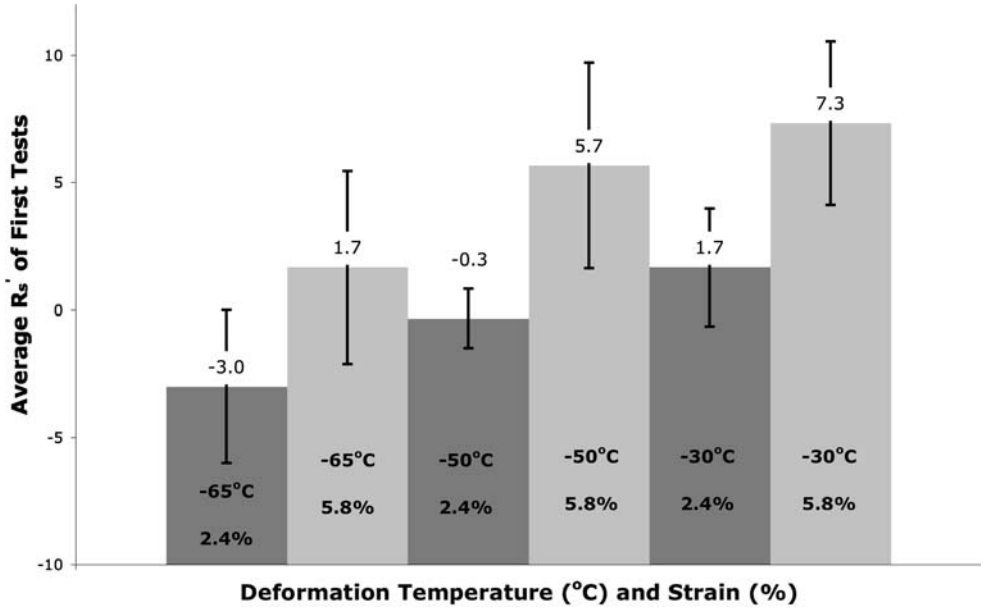

Fig. 4 Average R'_s of the first test for each strain and deformation temperature condition. Two standard deviation error bars are shown

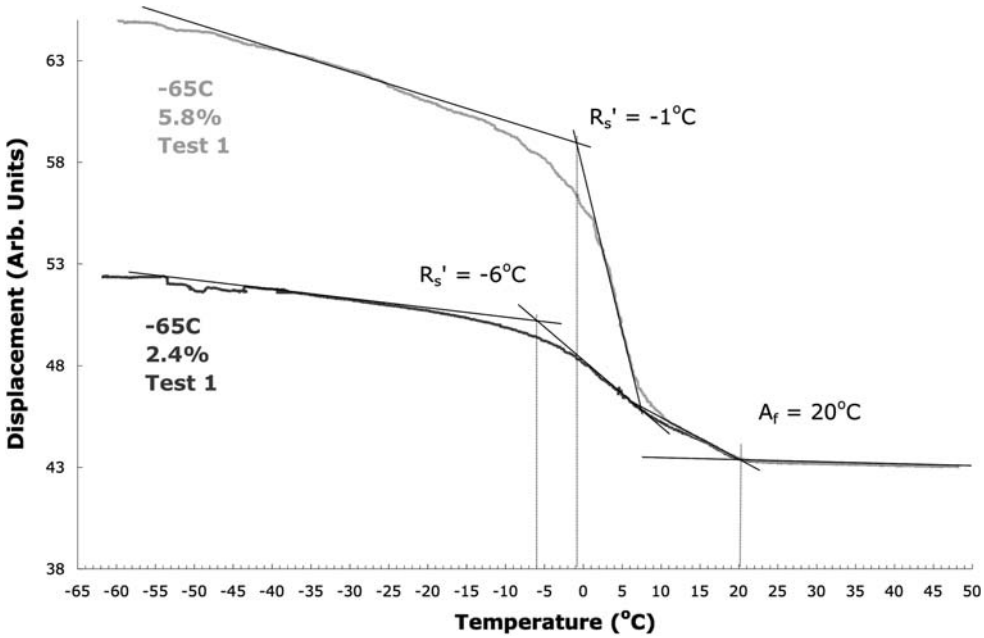

Fig. 5 BFR curves from specimens with 2.4 and 5.8% deformation strain

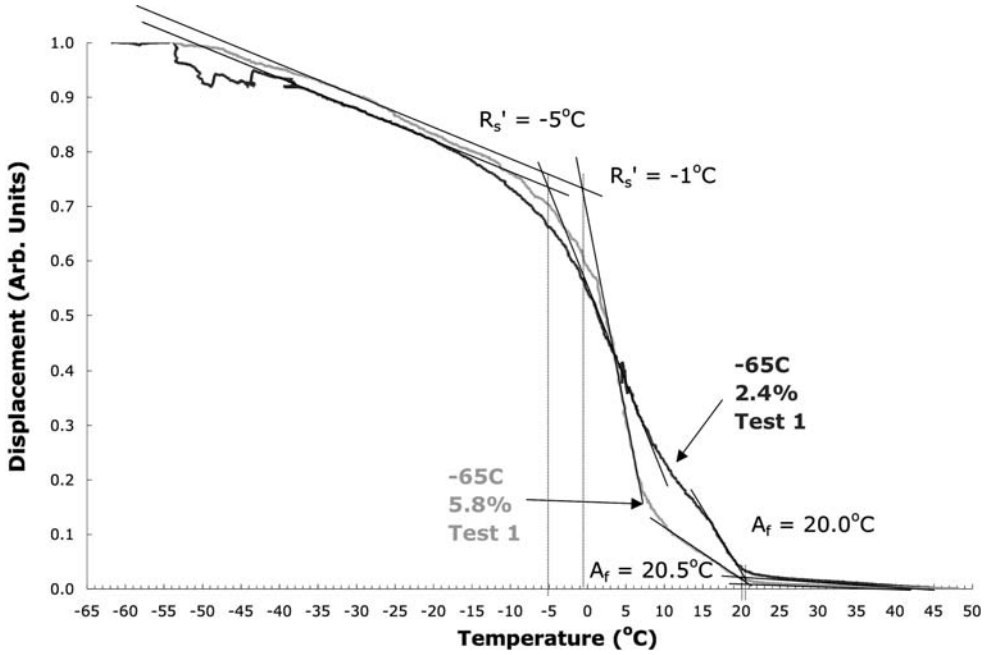

Fig. 6 Rescaled BFR curves from specimens with 2.4 and 5.8% deformation strain

These differences, resulting from scaling of the curves and a change in the character of the curves, seem to be more a result of the test than a material difference. To minimize this test-induced variability, it is recommended that consistent scaling and straining conditions be used.

The increase in R_s' with increasing deformation temperature originated from the length of the BFR curve onset. When more of the curved transformation onset is outlined, it is natural to fit the upper trend line at a lower temperature point along this curve, a point approximately halfway between the test starting temperature and the R_s' temperature is common. Therefore, one should again take care to be consistent in their test parameters and fitting technique when measuring and comparing R_s' values.

5. Conclusions

The conclusions from this study can be summarizes as follows:

- Increasing the outer fiber strain from 2.4 to 5.8% changed the character of the BFR curves causing a shift in the measured A_f and R_s' values by about 1 and 5 °C, respectively.
- Deformation temperature had no significant effect on the measurement of A_f and the effects on R_s' were mainly due to differences in trend line fitting.
- A slight increase in the transformation temperatures occurred after repeated testing but the intrinsic variability of the test did not allow a confident verification of any trend.
- The BFR technique is indeed a useful technique for measuring the transformation temperatures of Nitinol. If care is taken to minimize the effects of factors such at those outlined in this paper, i.e. deformation temperature, strain, repeated testing, and plot scaling, standard deviations of below 1 °C can be obtained.

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References

1. A. Pelton, J. DiCello, and S. Miyazaki, Optimization of Processing and Properties of Medical-Grade Nitinol Wire, *SMST-2000 Conference Proceedings*, S. Russell and A. Pelton, Eds., 30 April to 4 May, 2000 (Asilomar), SMST, 2001, p 361–374
2. M. Drexel, G. Selvaduray, and A. Pelton, The Effects of Cold Work and Heat Treatment on the Properties of Nitinol Wire, *SMST-2006 Conference Proceedings*, B. Berg, M. Mitchell, and J. Proft, Eds., May 7–11, 2006 (Asilomar), ASM International, 2008, p 447–454
3. "Determination of Transformation Temperature of Nickel-Titanium Shape Memory Alloys by Bend and Free Recovery," F 2082-06, *Annual Book of ASTM Standards*
4. S. Gupta and F. Sczerzeni, Effect of Cold Work on Mechanical Properties and on TTR's of a Nickel Titanium Shape Memory Alloy, *SMST-97 Conference Proceedings*, A. Pelton, D. Hodgson, S. Russell, and T. Duerig, Eds., March 2–6, 1997 (Asilomar), SMST, 1997, p 41–46
5. J. Chen, T. Duerig, A. Pelton, and D. Stoeckel, An Apparatus to Measure the Shape Properties of Nitinol Tubes for Medical Applications, *J. Phys. IV,* 1995, **5**(2), p 1247–1252
6. K. Otsuka, Introduction to the R-phase Transition, *Engineering Aspects of Shape Memory Alloys*, T. Duerig, K. Melton, D. Stockel, and C. Wayman, Eds., Buttersworth-Heinemann, 1990, p 36–45
7. S. Miyazaki, Thermal and Stress Cycling Effects and Fatigue Properties of Ni-Ti Alloys, *Engineering Aspects of Shape Memory Alloys*, T. Duerig, K. Melton, D. Stockel, and C. Wayman, Eds., Buttersworth-Heinemann, 1990, p 369–413