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An Apparatus to Measure the Shape Memory Properties of Nitinol Tubes for Medical Applications

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Abstract. A new apparatus has been developed to characterize the shape memory properties of Nitinol tubes and stents. In tests run on this apparatus, the samples are deformed in the radial direction in the martensitic state to a prescribed outer-fiber strain. Recovery strain and force are monitored continuously on heating. Values of properties as measured on this apparatus are compared to corresponding values from three-point and four-point bending tests, as well as DSC. We have found that this is a fast and reliable system to test transformation temperatures and recovery forces of tubes and stents.

1. INTRODUCTION

Nitinol tubing is being increasingly used in the medical community for a variety of purposes, including catheters, components for endoscopic procedures, and self-expanding stents [1-5]. It is necessary to control the shape memory properties of the tubes through thermomechanical processing so that these devices are activated by either stress-induced or thermally induced transformations. This requires not only precise process control during manufacturing, but also development of reliable testing methods. Recently, we have studied the mechanical properties of Nitinol wire and tubes in three-point, four-point, and pure bending modes of deformation [6,7]. In these papers, we argued that traditional methods, such as uniaxial tensile tests, do not adequately simulate the bending deformation behavior usually found in medical applications.

Many methods have been used to characterize the transformation properties of shape-memory alloys [8]. However, none of these methods were specifically designed to evaluate medical components manufactured from Nitinol tubing. Therefore, in this paper, we introduce a new apparatus to characterize the shape memory properties of tubes and stents.

2. EXPERIMENTAL

2.1 Equipment, materials and methods

Tubes and laser-machined and expanded tubes (stents) were analyzed with a new apparatus that included a thermocouple, a linear variable differential transformer (LVDT), and a load cell. A photograph of the apparatus and its associated computer-driven data acquisition system is shown in Figure 1.

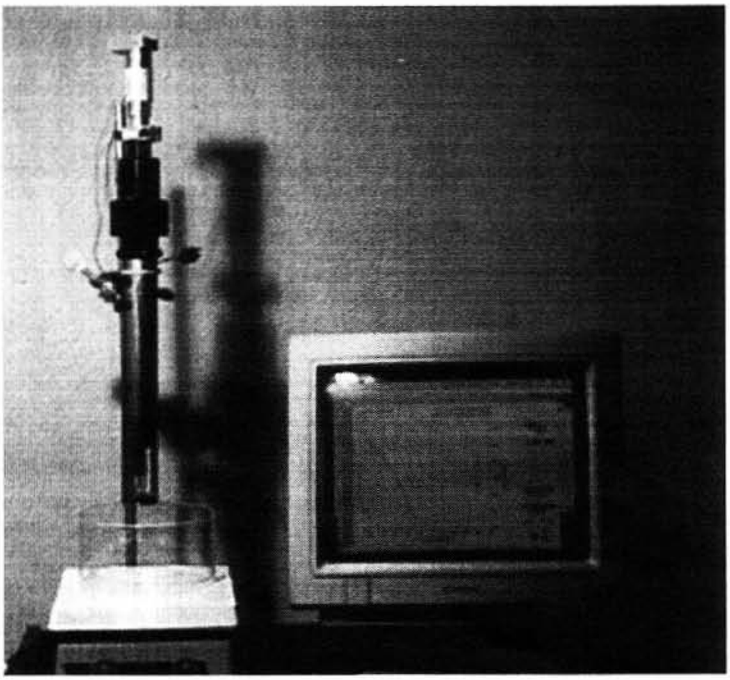


Figure 1: Apparatus for measuring austenite transition temperatures and forces exerted by stents and tubes. Samples are placed in the window near the bottom of the device.

During experimentation, both the sample and the part of the apparatus containing the sample were immersed in a bath of denatured alcohol. A laboratory heater-stirrer was used to stir the bath to provide even heating of the sample during experimentation. A thermocouple measured the temperature of this bath near the sample.

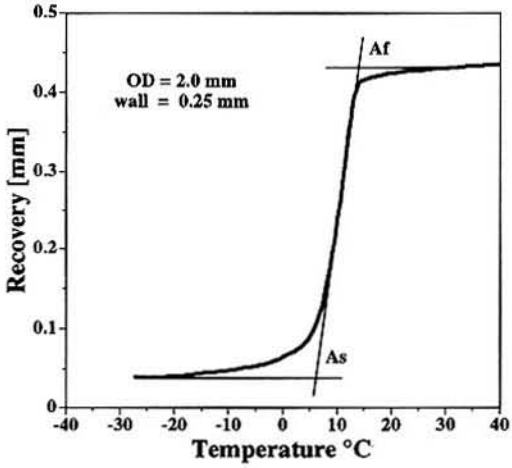
All tests involved the compression of a cylindrical sample on a level plate parallel to its axis. Compression and expansion of the sample was measured by an LVDT as follows: A gage pin of prescribed diameter is inserted through the sample. The sample is cooled to approximately -40^{o}C and compressed between a platen and the base of the testing device. The platen is connected on one end to an lightweight NiTi tube which does not buckle with loading. The small circular platen rests on the top of the compressed tube or stent and moves up as the sample expands. The opposite end of the tube feeds through the magnetic core of an LVDT which monitors the displacement of the sample. An example of the output of such an "unconstrained recovery" test is shown in Figure 2a.

In some cases, the stent is not allowed to expand fully as it is heated. The platen is constrained not to go beyond a prescribed height and prevents expansion of the sample beyond that point. The height is fixed with a pin gage standard before the sample is inserted. The force that the sample exerts upwards onto the platen when the tube or stent expands is measured by a load cell mounted to the top of the apparatus. Figure 2b shows a typical output for a constrained recovery test.

All data (temperature, platen height, and load) are collected by a computer-driven data acquisition system. The austenite start, A_s, and austenite finish, A_f, temperatures can be determined using these data. Repeatablility of data was tested by running seven samples on the equipment under identical conditions, and the standard deviation of A_f was found to be 1.2°C.

2.2 Output

Typical output for the determination of A_f of a NiTi tube (2.0 mm x 0.25 mm) is shown in Figure 2a. Note that the curve shape is almost identical to those of some traditional load-controlled test methods [8, 9]. Figure 2b shows a typical output for the measurement of load and A_S on a constrained stent. This stent was cooled to -45°C, compressed to 0.75 mm, and then allowed to recover until the OD had expanded to the preset height of 2.05 mm. As the temperature continued to increase, the force that the stent applied on the platen increased. The observed behavior was similar to that observed in previous, well-known constrained recovery tests [10].



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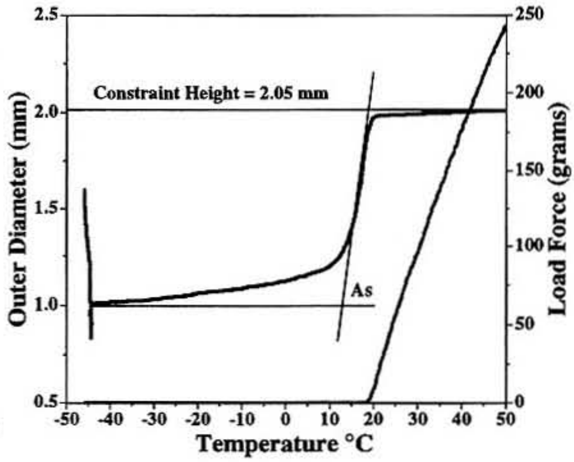

b

Figure 2a: Tube expansion as a function of temperature in an unconstrained recovery test. A_f is measured from this graph by the tangent method. **2b**: Stent expansion and load in a constrained recovery test.

The tubes used in the following experiments were NDC SE 506 with dimensions 2.0 mm OD x 0.25 mm wall and 2.18 mm OD x 0.09 mm wall. Stents of different dimensions were also tested. Approximately 0.3g of material was used for each test.

3. EXAMPLES

3.1 Measurement of A_f

The transformation behavior of a 2.0 mm x 0.25 mm tube was measured by four methods: 1) recovery from three-point bending, 2) recovery from four-point bending, 3) compression between two plates in the new apparatus, and 4) by differential scanning calorimetry. Samples for the first three tests were strained to 3.9% [6, 7]. These samples recovered with a small load due to the testing apparatus: The tube in the three-point bending test recovered with an applied load of 13 g due to the weight of the sensor resting on the sample; in the four-point bending test, the load was 22 g; testing with the new apparatus entailed a 5 g load (the combined weight of the platen and the NiTi tube). The A_f were 12°C, 7°C, 14°C, and 10°C in three-point bending, four-point bending, compression, and DSC, respectively, as shown in Figure 3. The similarity in the data is an indication that the tests are valid and comparable for measuring transformation temperatures. The small differences in the values for the transformation temperature are most likely due to the influence of load on the sample or dissimilarity in the true strain state between samples.

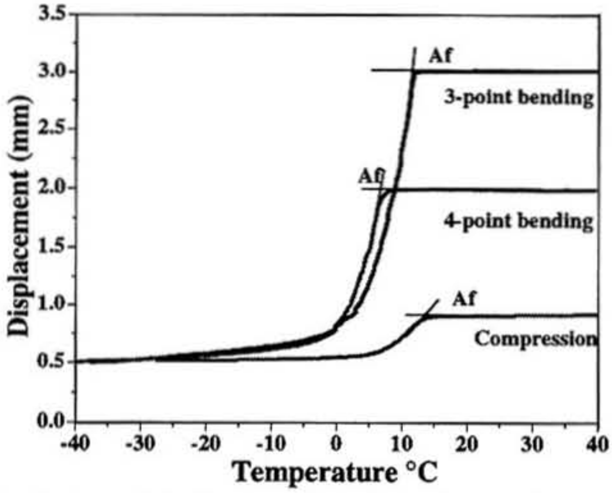

Figure 3: A_f as determined by three-point bending, four-point bending, and compression.

3.2 A_f versus tube strain

A NiTi tube of OD 2.18 mm and wall 0.09 mm was tested by compression to different strains. The displacement-temperature data were measured in order to make a correlation between tube strain and A_f.

The maximum strain in a tube under compression was calculated as:

$$\varepsilon = (1 + 2\,R_{after}/\text{wall})^{-1} - (1 + 2\,R_{before}/\text{wall})^{-1}$$

Table 1 shows the pin gages that were used and their corresponding strains. Results are shown in Figure 4.

Pin (mm)	**1.78**	**1.50**	**1.27**	**1.02**	**0.76**	**0.53**	**0.28**
Strain (%)	**0.50**	**1.00**	**2.00**	**3.50**	**5.60**	**8.70**	**15.7**

Table 1.

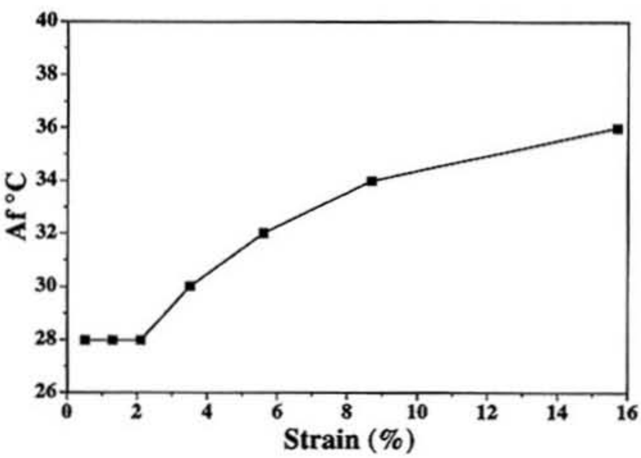

Figure 4: A_f as a function of strain. Tube dimensions: OD = 2.19 mm, wall = 0.09 mm. Transformation temperature is not a strong function of strain for strains of less than 2%.

3.3 Effect of load on Af

The effect of load on a stent as it recovers was examined. The stent tested (OD = 4.46 mm, wall = 0.52 mm) was loaded with masses ranging from 5.3 g to 325.3 g. The expected change in transition temperature goes as a modification of the classic Clausius-Clapeyron equation [11]:

$$\frac{d\sigma}{dT} = \frac{-\Delta H}{T\,\varepsilon_o}$$

where σ is the applied stress; T is the temperature; ΔH is the enthalpy change; and ε_o is the transformation strain in the direction of the applied stress. The compression apparatus used in these experiments measures force rather than stress. However, the slope of the load versus temperature curve is expected to follow a linear $d\sigma/dT$ behavior since the area over which the force is applied does not change dramatically. The results of the test are shown in Figure 5.

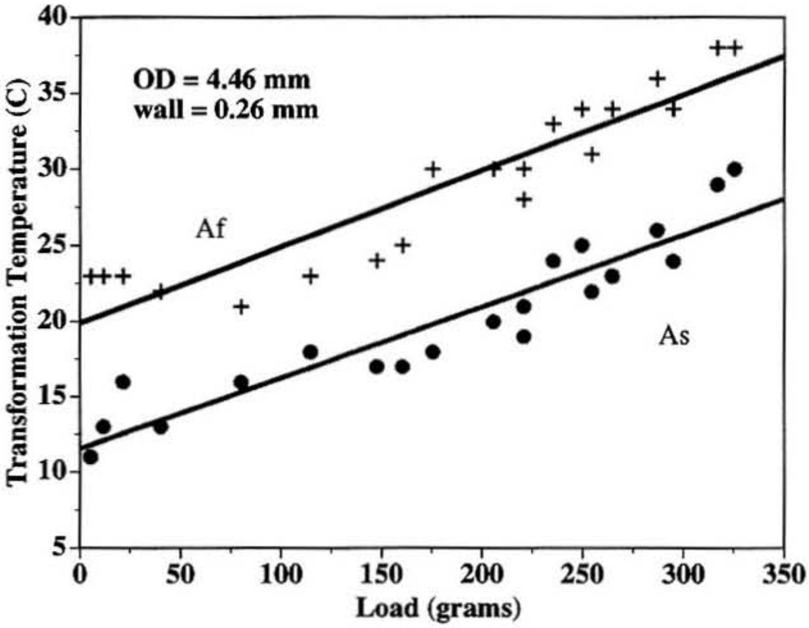

Figure 5. Transformation temperature as a function of applied load. Notice that both A_s and A_f increase linearly with load. In this test setup, small loads have a negligible effect on the transition temperature.

3.4 Constrained Recovery: Force Applied by a Recovering Stent as a Function of Constraint [3]

Stents (4.92mm x 0.14 mm) were compressed onto a pin diameter of 0.48 mm at -40°C. In heating to +40°C, the force applied by the stent on the platen (fixed at a prescribed height) was measured. Results are shown in Figure 6. As expected, the data shows the trend of increasing transition temperature with the amount of constraint. This behavior has also been noted in constrained recovery tests on shape-memory couplings [10].

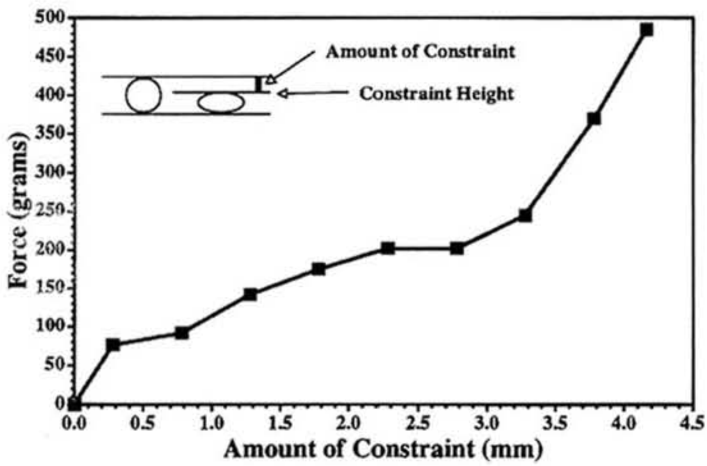

Figure 6: Stent force as a function of constraint. Note the increase in force with increase in amount of constraint. This expected because the force measured is nearly the same as the force required to compress the stent to the given constraint level.

4. SUMMARY

A new apparatus has been constructed for measuring the transformation recovery behavior of tubes and stents. Results on this machine are accurate and repeatable. Test runs are efficient and require a small volume of material. Data from tests run on this device show good corroboration with measurement of A_f from three-point and four-point bending tests and DSC. Data from other experiments on the new device show A_f as a function of strain, load, and constraint height, and these data fit well with theory. This indicates that measurements with the apparatus are sound and useful for NiTi characterization of tubes and tube derivatives (stents) in compression (bending).

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