JMEPEG (2009) 18:836–842
DOI: 10.1007/s11665-009-9406-x

1059-9495/$19.00

Tensile Testing of Nitinol Tubes and Wires with Higher Strain Rates

Rainer Johe

(Submitted September 26, 2008; in revised form January 16, 2009)

During tensile testing of superelastic Nitinol material, the specimen temperature increases as result of the exothermic Austenite-to-Martensite phase transformation. The increase in specimen temperature has great influence on the stress-strain response—in particular, upper and lower plateau values—and limits the strain rate of the tensile test, so that for larger specimen dimension, the strain rate has to be reduced. A special setup of the tensile testing equipment has been developed using a fan to improve the heat exchange between the specimen and the ambiance to allow much higher strain rates as well as even gradient of the upper and lower plateau. It could be shown that the strain rate of the first loading and unloading cycle could be two to four times higher as recommended in ASTM F 2516-07 without any negative impact on the determined values. The needed time for tensile testing of Nitinol products could be reduced considerably. The improved heat exchange gives a better comparability and reproducibility of the tensile test data.

Keywords advanced characterization, biomaterials, mechanical testing

1. Introduction

Tensile testing of Nitinol is a curious affair. Common and well-known tensile testing methods as described, e.g. in ASTM E8, DIN EN 10002-1, or ISO 6892 are not adequate and do not relate to the pseudo-elastic properties and shape memory behavior.

For this reason, a new testing standard has been established; the procedure is defined in the ASTM F 2516 (Ref 5), which is the "Standard Test Method for Tension Testing of Nickel-Titanium Superelastic Materials" such as wire, strip or tubing in the superelastic condition.

Because the thermal exchange in conjunction with the strain rate has a huge impact on the gradient of the tensile test curve and the results derived, a comparative study with various testing speeds and setup modes on tubing and wire was done.

2. Testing Procedure and Technical Background

Starting tensile testing (see Fig. 1), Nitinol shows normal linear elastic behavior; that means increasing extension of the tensile test specimen leads to an increasing tensile stress response.

This article is an invited paper selected from presentations at Shape Memory and Superelastic Technologies 2008, held September 21-25, 2008, in Stresa, Italy, and has been expanded from the original presentation.

Rainer Johe, EUROFLEX GmbH, Kaiser-Friedrich-Str. 7, 75172 Pforzheim, Germany. Contact e-mail: r.johe@rau-pforzheim.de.

At a certain stress level, the characteristic, stress-induced Austenite-to-Martensite phase transformation of Nitinol begins: typically in the range of about 400 to 600 MPa depending on the Af-temperature of the specimen.

Due to mechanical stress, single lattice areas of Austenite begin to shear and change their shape to Martensite. The effect is that gradually the whole specimen starts elongating at a constant load. The stress-strain curve shows typically the so-called upper (loading) plateau. The Austenite-to-Martensite transformation is completed (end of plateau) at a total elongation of about 7%.

To see the reversible phase transformation back from Martensite-to-Austenite—which occurs due to the superelastic effect of Nitinol—the test specimen gets unloaded within the plateau after a total elongation of 6%. After passing through an unloading hysteresis of about 300 MPa, the backward Martensite-to-Austenite phase transformation starts forming the unloading or lower plateau because of the reversible lattice movement of Nitinol.

Ideally, the superelastic sample returns to the starting point without any residual strain.

There are two main effects which have remarkable influence on the gradient of the tensile test curves of Nitinol as follows:

1. The level of the upper and lower plateau depends on the test temperature that means increasing test temperature leads to increasing plateau levels (typically 5-6 MPa/K) (see Fig. 2) and
2. The stress-induced Austenite-to-Martensite transformation is an exothermic phase transformation (Martensite-to-Austenite is endothermic).

The exothermic Austenite-to-Martensite transformation leads to an increase in the specimen temperature during the loading cycle, and in consequence, to an ongoing increase of the plateau stresses with increasing elongation. On the other hand, during unloading, the specimen temperature decreases because of the endothermic Martensite-to-Austenite transformation which leads to a permanent decrease of the lower plateau stresses.

Consequently, it is essential to control the temperature of the tensile test specimen within close limits.

One option is to get an adequate thermal exchange between the specimen and the static ambient air by reducing the tensile test speed (mainly during the first cycle). The limited strain rate allows the heat to transfer out of the specimen.

A second option is to modify the tensile test equipment to improve the thermal exchange between the specimen and the circumfluent medium, e.g. water or stirred air.

The maximum crosshead speed (strain rates) according to ASTM F 2516-07 depending on the specimen dimensions are listed in the following table:

***d*, diameter or thickness (mm) for round tubing: *d* = OD − ID = 2× wall**	**Maximum crosshead speed in mm/min per mm of initial length**	
	First cycle (load to 6% strain and unload)	**Second cycle (load to failure)**
$d \le 0.2$	0.08 (8%/min)	0.8
$0.2 < d \le 0.5$	0.04 (4%/min)	0.4
$0.5 < d \le 2.5$	0.02 (2%/min)	0.2
$d > 2.5$	0.01 (1%/min)	0.1

The strain rate for the second cycle to failure is ten times higher than for the first cycle

3. Equipment and Samples

The tensile testing equipment has been modified, which means a ventilator has been integrated providing a constant forced flow of ambient air (no cooling) against the test specimen to transfer the exothermic heat out of the specimen during the loading cycle and to improve the thermal exchange so that the specimen can pick up heat during the unloading cycle.

So the dimensional range of 0.5-2.5 mm is typical size for the most common Nitinol products, hence sample material of superelastic Nitinol wires in that range have been chosen for investigation.

Samples of Nitinol tubing were taken with the range of $d = 0.5$ that means tubings right below and above this are chosen to see the influence of change in testing speed.

The tensile tests were performed with different sizes and various strain rates during the first cycle. For the second cycle of load-to-failure, the recommended strain rate as per ASTM F 2516-07 was used (Ref 5).

4. Results

4.1 Nitinol Wire with 0.6 and 2.4 mm

Lower and upper end of the range ($0.5 < d \le 2.5$; strain rate, first cycle 2%/min)

Left side: stress-strain-curve in static ambient air (see Fig. 3 and 5)
Right side: stress-strain-curve in forced air flow (see Fig. 4 and 6)

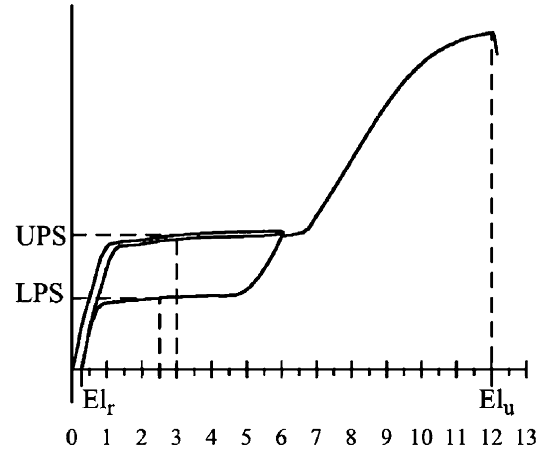

Fig. 1 Schematic stress-strain curve of superelastic Nitinol; picture taken from ASTM F 2516-07. Testing procedure: to see the superelastic behavior of Nitinol, a tensile test specimen gets pulled to the upper reversal point of 6% total elongation, unloaded to 7 MPa, and reloaded until failure

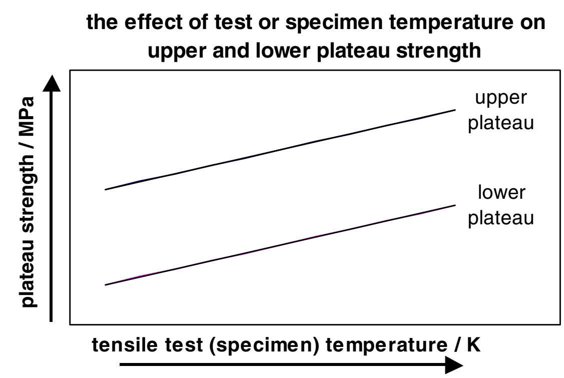

Fig. 2 Principal schematic dependence of upper and lower plateau stresses on the tensile test or specimen temperature. Upper plateau stresses (UPS) as well as lower plateau stresses (LPS) is increasing with increasing test or specimen temperature

4.2 Nitinol Tubing with 0.34 and 0.50 mm Wall

Mid of the range ($d = 0.68$ and 1.0; strain rate, first cycle 2%/min)

Left side: stress-strain curve in static ambient air (see Fig. 7 and 9)
Right side: stress-strain curve in forced air flow (see Fig. 8 and 10)

In Fig. 3, 5, 7, and 9, it can be seen that depending on the specimen dimension the stress-strain curve shows more or less a slope during the first loading cycle.

In case a fan is used—see Fig. 4, 6, 8, and 10—the slope of the stress-strain curve is much smaller than in static ambient air. That means, the forced air flow improves the heat exchange between specimen and ambient air.

UPS is about 30 MPa lower and LPS is about 30 MPa higher when measured using a fan than the respective values when measured without fan (Diagram 1 and 2). Therefore, the hysteresis between upper and lower plateau gets more pressed together in case of forced air flow.

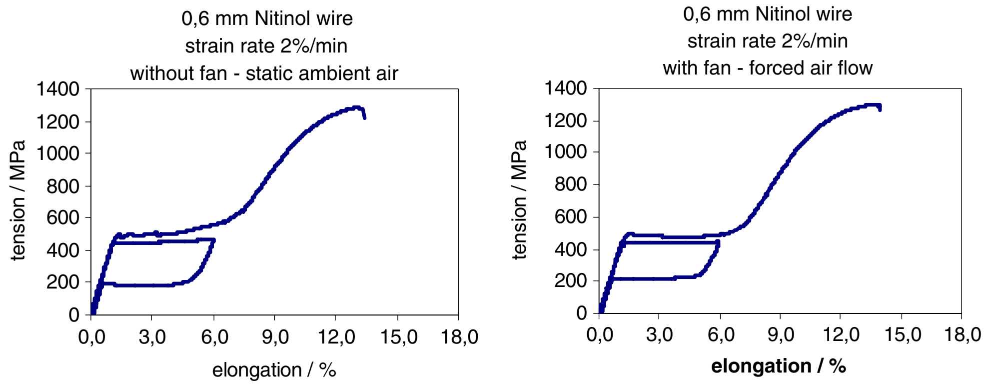

Fig. 3-4 Tensile test curves on a 0.6-mm Nitinol wire with the ASTM F 2516 recommended strain rate of 2%/min; *left*: without/*right*: with fan

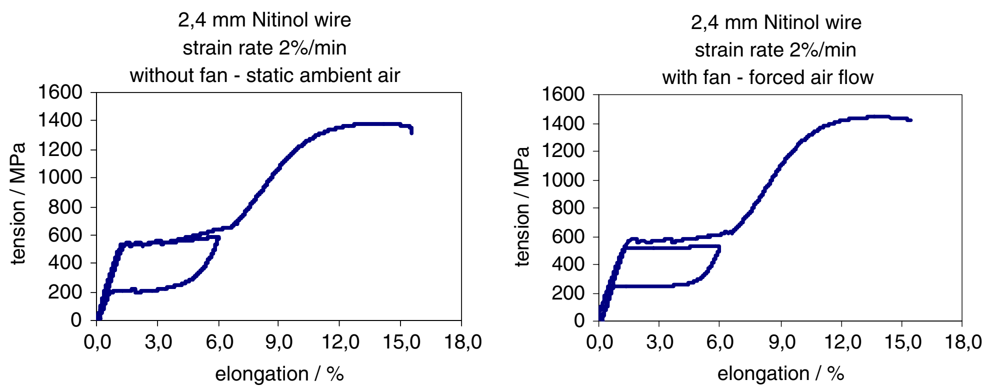

Fig. 5-6 Tensile test curves on a 2.4-mm Nitinol wire with the ASTM F 2516 recommended strain rate of 2%/min; *left*: without/*right*: with fan

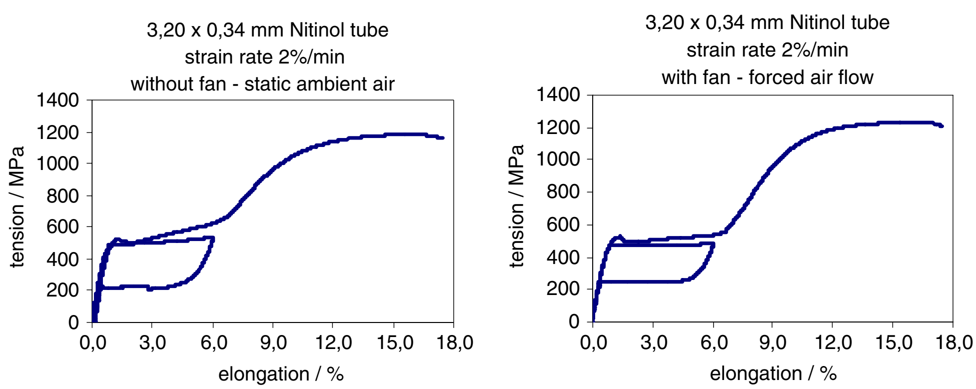

Fig. 7-8 Tensile test curves done on a 3.20×0.34 mm^2 Nitinol tube with the ASTM F 2516 recommended strain rate of 2%/min ($2\times$ wall = 0.68); *left*: without/*right*: with fan

Residual elongation (Permanent Set), UTS, and elongation are not influenced.

4.3 Nitinol Wire and Tubing—Tensile Test with Higher Strain Rates

In the following, only the first cycle is shown to see better the influence of various strain rates and the thermal exchange between specimen and ambient air to the slope of the stress-strain curve.

Left side: stress-strain curve in static ambient air (see Fig. 11, 13 and 15)
Right side: stress-strain curve in forced air flow (see Fig. 12, 14 and 16)

In Fig. 11, 13, and 15, it can be seen that—in case of static ambient air—the gradient of the upper plateau increases with increasing testing speed. The unloading plateau becomes more and more imprecise and the hysteresis between upper and lower plateau get wider with higher strain rates.

Whereas using a fan to force the air flow—see Fig. 12, 14, and 16—the upper plateau is still even and linear due to the improved thermal exchange between specimen and ambient air (Diagram 3 and 4).

Figures 17, 18, 19, and 20 show the heightened effect on 2.4-mm wire, which means—in case of static ambient air—the gradient of the upper plateau increases with increasing testing speed. On the right side, the gradient of the upper plateau is much more linear and even more so than on the left side because of the improved thermal exchange between specimen and ambient air.

Furthermore, there is a positive effect to the unloading plateau and the hysteresis between upper and lower plateau.

Figures 21 and 23 show that the increase in strain rate because of the thinner wall leads to a higher slope in the stress-strain curve of the 3.00 × 0.235 mm tubing.

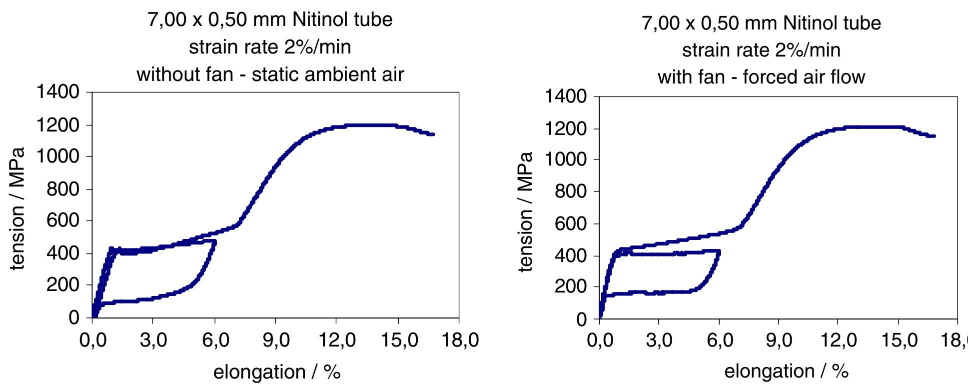

Fig. 9-10 Tensile test curves done on a 7.00×0.50 mm^2 Nitinol tube with the ASTM F 2516 recommended strain rate of 2%/min (2× wall = 1.0); *left*: without/*right*: with fan

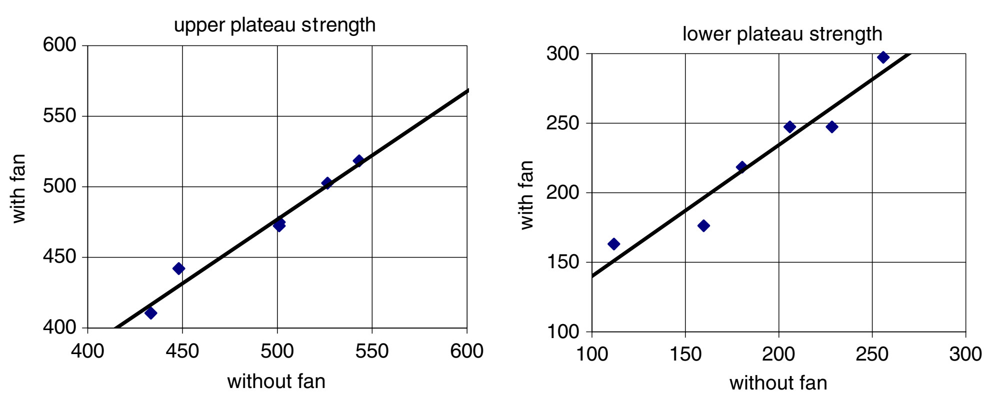

Diagram 1-2 Influence of forced air flow on UPS and LPS

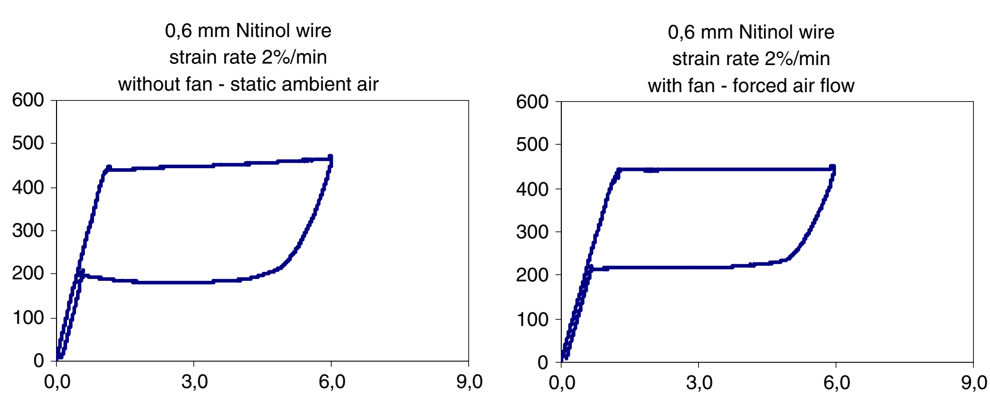

Fig. 11-12 Tensile test curves of a 0.6-mm Nitinol wire with the ASTM F 2516 recommended strain rate of 2%/min; *left*: without/*right*: with fan

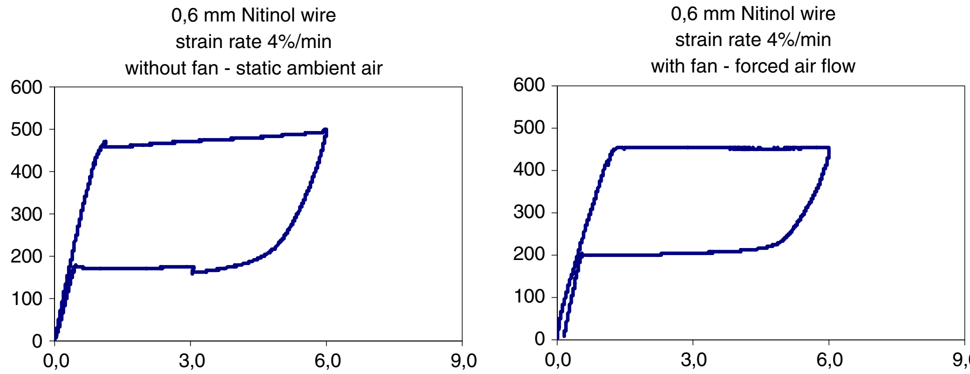

Fig. 13-14 Tensile test curves of a 0.6-mm Nitinol wire with two times higher strain rate, i.e. of 4%/min; *left*: without/*right*: with fan

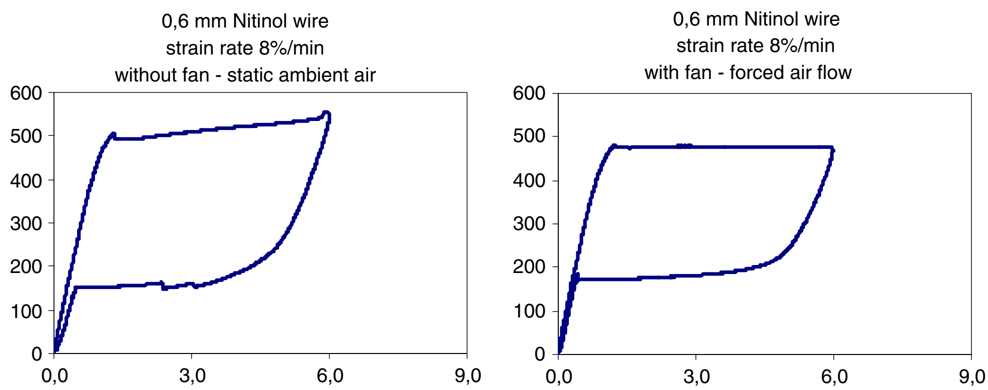

Fig. 15-16 Tensile test curves of a 0.6-mm Nitinol wire with four times higher strain rate, i.e. of 4%/min; *left*: without/*right*: with fan

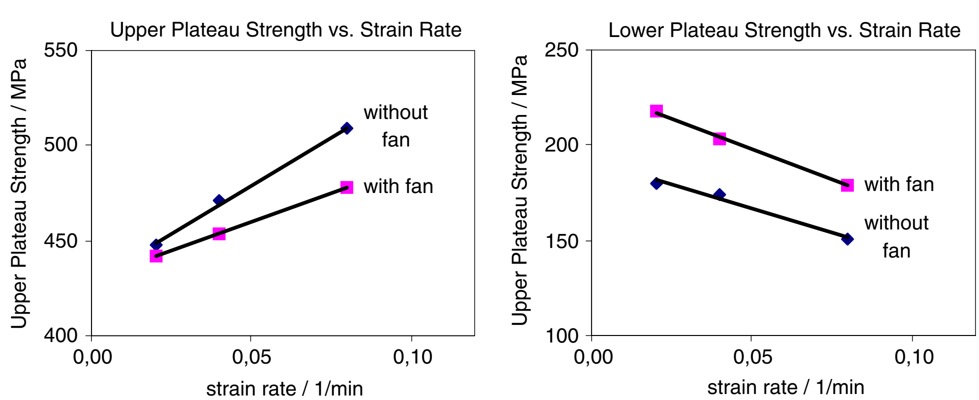

Diagram 3-4 Increase of UPS and decrease in LPS due to higher strain rates; effect of better heat exchange between specimen and ambient air using a fan

With the use of a fan—Fig. 22 and 24—the same material could be tested four times faster without increasing slope of the stress-strain curve. The upper plateau is linear and even because of the improved thermal exchange between specimen and ambient air.

Furthermore, there is a positive effect to the unloading plateau and the hysteresis between upper and lower plateau.

5. Conclusion

The exothermic Austenite-to-Martensite phase transformation during the loading cycle leads to an increase of the tensile test specimen temperature which has a huge influence on the stress-strain response. The exothermic heat depends on the

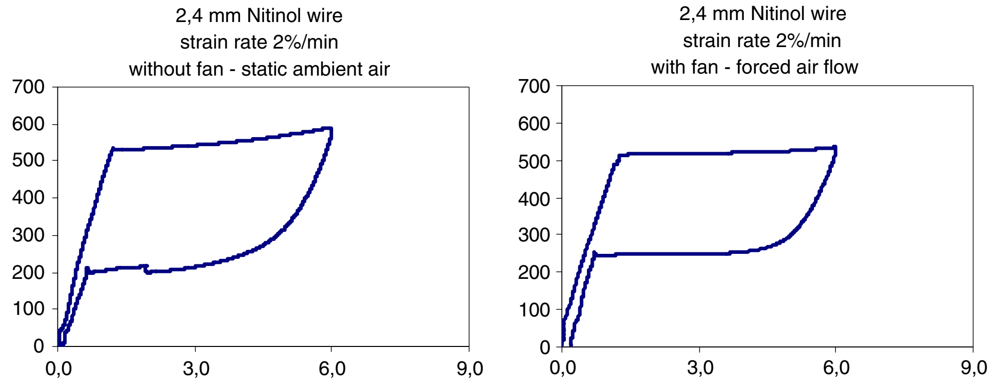

Fig. 17-18 Tensile test curves of a 2.4-mm Nitinol wire with the ASTM F 2516 recommended strain rate of 2%/min; *left*: without/*right*: with fan

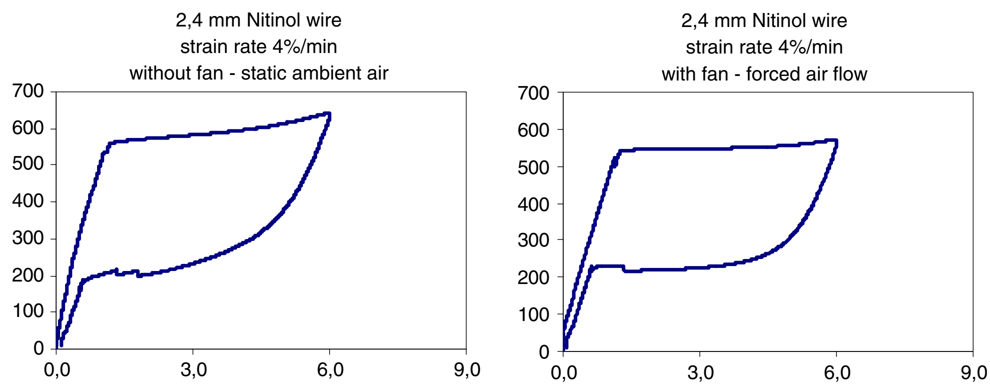

Fig. 19-20 Tensile test curves of a 0.60-mm Nitinol wire with two times higher strain rate of 4%/min; *left*: without/*right*: with fan

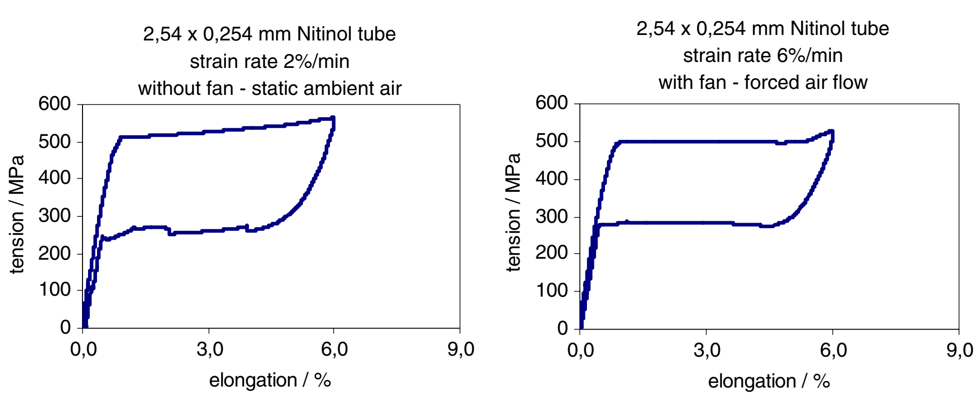

Fig. 21-22 Tensile test curves of a 2.54 × 0.254 mm^2 Nitinol tubing ($d > 0.5$). *Left side*: with the recommended strain rate of 2%/min without fan. *Right side*: Three times higher strain rate of 6%/min with fan

geometry of the tested specimen. In ASTM F 2516, the strain rate is limited depending on the wire diameter or the tube-wall thickness to allow the heat to transfer out of the specimen. For tubing, the defined steps are relatively large that means small differences in wall thickness could lead to different slopes in the stress-strain curves.

The use of a fan or any other equipment to improve the heat exchange between the specimen and the ambiance will reduce the slope effect on upper and lower plateau strength and will lead in consequence to more reproducibility of the data derived.

Improved heat exchange leads to lower UPS and higher LPS values because of the reduced gradient in the loading and unloading plateau.

Improved heat exchange allows higher strain rates during the first cycle without negative impact on UPS, LPS, Residual Elongation (Permanent Set), UTS, and elongation.

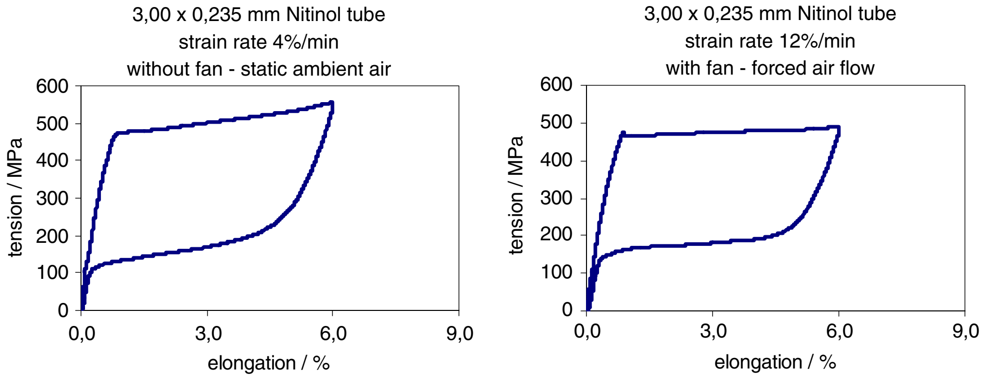

Fig. 23-24 Tensile test curves of a 3.00 × 0.235 mm^2 Nitinol tubing ($d < 0.5$). *Left side*: with the recommended strain rate of 4%/min without fan. *Right side*: Three times higher strain rate of 12%/min with fan

In the case of tubing, the impact of improved heat exchange is higher than that for wire because of the higher surface-to-volume ratio of tubing, which could allow three to four times higher strain rates during the first cycle.

The time needed for tensile testing could be reduced by about one-half.

The limitation of strain rates depending on rough dimensional groupings seems to be insufficient for all types of samplings. This could be a point for discussion in the next review of ASTM F 2516.

References

1. ASTM F 2004 Test Method for Transformation Temperature of Nickel-Titanium Alloys by Thermal Analysis
2. ASTM F 2005 Terminology for Nickel-Titanium Shape Memory Alloys
3. ASTM F 2063 Specification for Wrought Nickel-Titanium Shape Memory Alloys for Medical Devices and Surgical Implants
4. ASTM F 2082 Test Method for Determination of Transformation Temperature of Nickel-Titanium Shape Memory Alloys by Bend and Free Recovery
5. ASTM F 2516 Test Method for Tension Testing of Nickel-Titanium Superelastic Materials