

Mechanical Properties of Sputtered Nitinol Thin Films

Sputtered Nitinol films can exhibit either superelastic or shape memory properties at room temperature, depending on the Ni:Ti ratio of the sputtered film. They can exhibit fracture strains of more than 40%, or high fracture stresses in the range of 1600 MPa¹. They can be as thick as 80 μm , and structured with high feature resolution on 150 mm diameter substrates or larger, and are hence a feasible alternative for many medical and industrial applications². Superelasticity and most other properties of sputtered Nitinol are quite similar to standard Nitinol (e.g. drawn tubes, wires and rolled sheets), but some differences exist. Differences related to mechanical properties are discussed in this paper.

In the “as-deposited” state, without heat treatment, sputtered Nitinol is amorphous. Amorphous, sputtered Nitinol is interesting in particular for spring applications, since it exhibits a tremendous tensile strength, in the range of 2000 MPa, and a perfectly linear stress-strain behavior, see Fig. 1. After heat treatment at high temperatures, crystallized, sputtered Nitinol recovers its shape, with the end of the plateau being at 4 to 5%. This value is smaller compared to standard Nitinol due to the lack of a preferred grain orientation in drawing or rolling direction. Upper and lower plateau stress (UPS, LPS) can be varied by changing the Ni:Ti ratio, or – within a smaller range – by adjusting the heat treatment parameters. Superelastic Nitinol films are slightly Ni rich, and the UPS ranges typically between 450 and 500 MPa at 37°C.

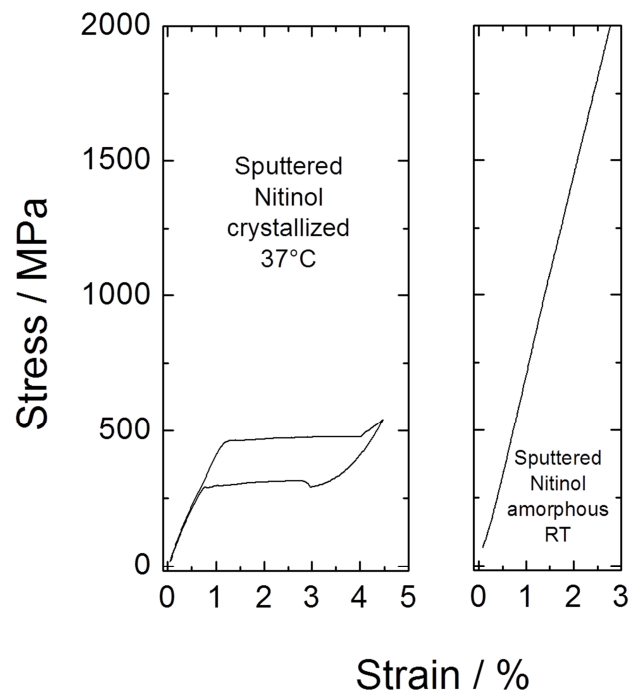


Figure 1: Left: Heat treated, sputtered Nitinol thin films exhibits typical superelastic properties with hysteresis. Right: The amorphous, “as-deposited” structure reveals extremely high tensile strength, up to 2000 MPa and a linear elastic behavior.

Mechanical stability during superelastic cycling

Despite the lack of cold work and the connected lower dislocation density, sputtered films show good mechanical stability during mechanical cycling. Fig. 2 shows a tensile curve with 200 full superelastic cycles, with a final remanent deformation of $\sim 0.35\%$. The functional properties degrade slightly with cycle number, but a flat plateau with lower UPS remains even after 200 cycles. This behavior is similar or superior to standard Nitinol^{3,4,5}.

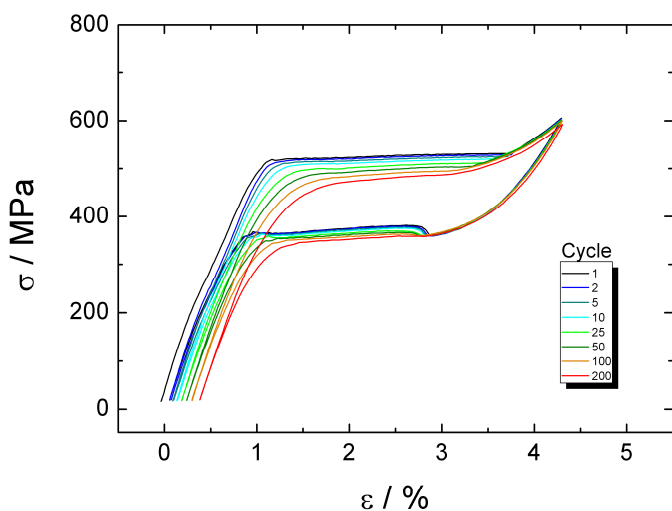


Figure 2: 200 full superelastic cycles at 37°C of a sputtered Nitinol film: the UPS decreases slightly with cycle number, but a rather flat UPS and LPS remains, with a final remanent deformation of 0.35% only.

Significant Increase in fatigue safety limit

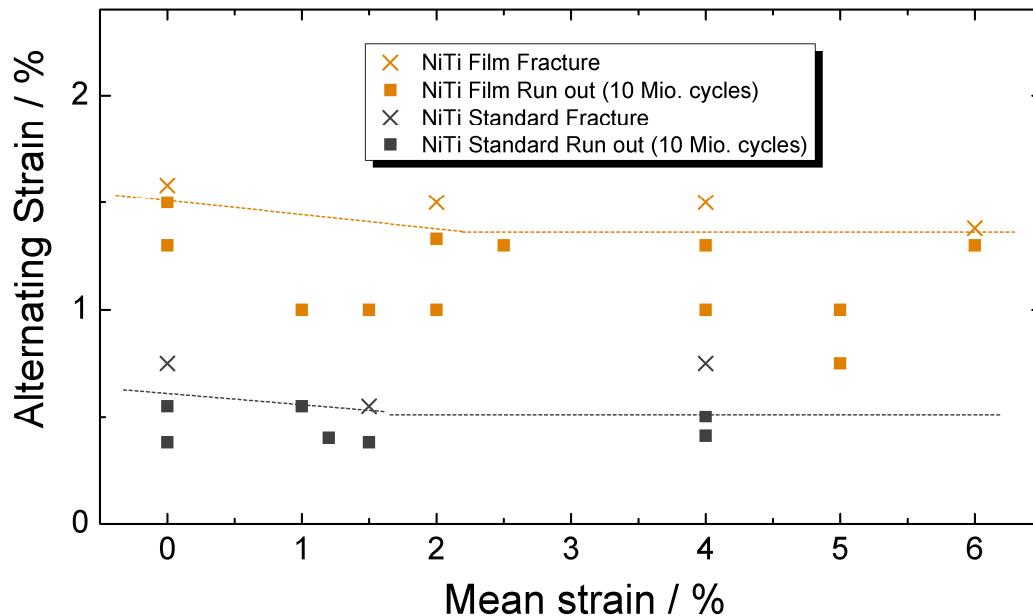


Figure 2: The fatigue safety limit of sputtered Nitinol films amounts to approximately $\pm 1.5\%$ alternating strain, surpassing the fatigue safety limit of standard Nitinol⁶ by a factor of three. Diamond shaped structures of structured thin film and of laser cut sheet metal were compared, and material strain was calculated by finite element methods. More details in ⁷.

A key feature of sputtered Nitinol films is their material purity, which has significant impact on pivotal properties. Going through the gas phase during the deposition process, oxide and carbide inclusions are not present in sputtered Nitinol material. Oxide and carbide inclusions, which are present in standard Nitinol, are often connected with material voids. These particle-void-assemblies (PVAs) have been identified as a major source for crack initiation^{8,9}. The lack of these PVAs eliminates therefore this source for crack initiation. Furthermore, FEM simulations of stents revealed that the TiC inclusion itself greatly changes the stress distribution. The maximum stress level of a stent model with TiC defect is about ten times as large as the structure without defect. It can be concluded that cracks/fractures are likely to occur near the defect¹⁰. In smaller stents, the relative size of inclusions increases with decreasing strut diameter. To fabricate endovascular devices for smaller vessels the application of thin film techniques is therefore considered to be an attractive alternative approach¹¹. Due to this fabrication method the formation of TiC inclusions is avoided.

The lack of oxide and carbide inclusions has also a positive impact on corrosion properties (due to a highly homogeneous native oxide without impurities at the surface), as well as on mechanical fatigue properties: testing of sputtered and patterned Nitinol diamond shape specimen and laser cut and electro-polished specimen from standard sheet metal revealed an excellent fatigue safety limit of the sputtered films of $\pm 1.5\%$ alternating strain⁷. Below $\pm 1.5\%$ alternating strain, all sputtered Nitinol samples reached test run out at 10 million cycles, see Fig. 3.

About ACQUANDAS GmbH: ACQUANDAS GmbH is a technology company that supplies thin film components to the healthcare industry – in particular to medical device OEMs – and other industrial markets, such as the automotive and consumer electronics industries. ACQUANDAS is located in Kiel, Germany.

Based on state-of-the-art microsystem technology processes, we fabricate an entirely new generation of metallic components for applications in medical devices and many other products. The combination of properties that our devices have is unique: miniaturized structures with high geometrical complexity, integrated micro-electrode arrays, increased radiopacity, high feature resolution, excellent biocompatibility and improved mechanical properties!

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- ¹ *Martensitic transformation and shape memory behavior in sputter-deposited TiNi-base thin films*, S. Miyazaki, A. Ishida, Mater. Sci. Eng. A, **273-275** (1999) 106-133
- ² *Capability of Sputtered Micro-patterned NiTi Thick Films*, C. Bechtold, R. Lima de Miranda, E. Quandt, Shape Memory and Superelasticity **1** (2015) 3, 286
- ³ *Effect of cyclic deformation on the Pseudoelasticity Characteristics of Ti-Ni Alloys*, S. Miyazaki, T. Imai, Y. Igo, K. Otsuka, Metall Trans. A **17** (1986) 115
- ⁴ *Cyclic deformation mechanisms in precipitated NiTi shape memory alloys*, Gall, Maier, Acta Mater. **50** (2002) 18, 4643-4657
- ⁵ *Thermomechanical properties due to martensitic and R-phase transformations of TiNi shape memory alloy subjected to cyclic loadings*, H. Tobushi, S. Yamada, T. Hachisuka, A. Ikai, K. Tanaka, Smart Mater. Struct. **5** (1996) 6, 788-795
- ⁶ *Mechanical fatigue and fracture of Nitinol*, S.W. Robertson, A.R. Pelton, R.O. Ritchi, Int. Mater. Rev. **57** (2012) 1, 1
- ⁷ *Comparison of the Fatigue Performance of Commercially Produced Nitinol Samples versus Sputter-Deposited Nitinol*, G. Siekmeyer, A. Schübler, R. Lima de Miranda, E. Quandt, JMEPEG **23** (2014) 2437
- ⁸ *Carbon and Oxygen Levels in Nitinol Alloys and the Implications for Medical Device Manufacture and Durability*, N. Morgan, A. Wick, J. DiCello, R. Graham, in: SMST Proceedings 2008, pp. 821-828.
- ⁹ *Impurity levels and fatigue lives of pseudoelastic NiTi shape memory alloys*, M. Rahim, J. Frenzel, M. Frotscher, J. Pftzing-Micklich, R. Steegmüller, M. Wohlschlägl, H. Mughrabi, G. Eggeler, Acta Mater. **61** (2013) 3667
- ¹⁰ *The FEM simulation of mechanical properties characterization of stents under quasi-static loading/unloading conditions*, Y.H. Zhi, X.M. Wang, M. Frotscher, G. Eggeler, Z.F. Yue, Materialwiss. Werkst. **38** (2007) 862-867.
- ¹¹ *Recent developments in SMA thin film based microactuators for biomedical and fiber optics applications*, V. Gupta, V. Martynov, A.D. Johnson, Actuator (2002) 355-358.