Technical White Paper:

Microstructure of Sputtered Nitinol Thin Films

During the deposition of Nitinol thin films by magnetron sputtering – a physical vapor deposition (PVD) method – material atoms are ejected from a solid target, go through the gas phase and deposit on a substrate[1,2,3] (usually a silicon wafer). This fabrication route is consequently significantly different to conventional Nitinol fabrication methods, where the material is processed from the melt. The resulting material reveals therefore some differences compared to standard Nitinol, and differences related to microstructure are discussed in this paper.

One particularity of sputtered Nitinol thin films is that, in the "as-deposited" state, the material is amorphous, and requires a heat treatment to become crystalline. After heat treatment, crystallized, sputtered Nitinol shows either superelastic or shape memory properties, depending on the Ni:Ti ratio. Grain size (to a certain extent), as well as Ni_4Ti_3 precipitate size, and distribution, can be adjusted with the proper heat treatment, similar as in hot- and cold-rolled wires[4]. While standard Nitinol requires cold work in order to tease out the superelastic properties, sputtered Nitinol does not. Hence, standard Nitinol has a high density of dislocations[5], and a pronounced preferred crystal orientation of the grains in drawing or rolling direction. Sputtered Nitinol has no such anisotropy but an isotropic crystal orientation and grain sizes typically in the range of 1 to 5 µm[6,7], see Fig. 1.

Isotropic grain orientation

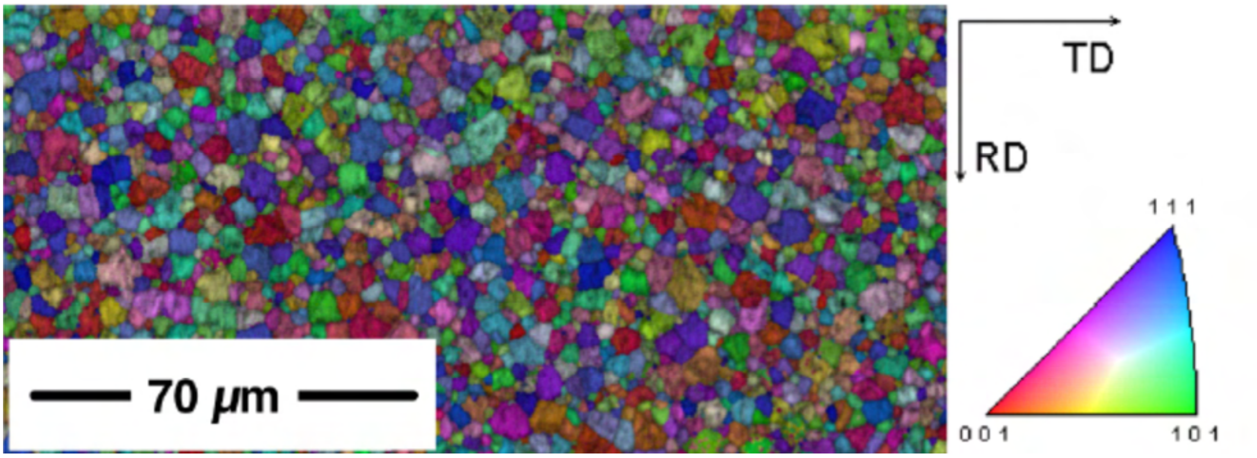

Figure 1: Isotropic distribution of grain orientiations: in contrast to drawn wires or rolled sheets, sputtered and heat treated thin films have no preferred crystal orientation (EBSD: University of Chemnitz).

The transformation temperature of the alloy can be varied between -10 and +100°C, mainly by choosing a specific Ni:Ti ratio, but also – within in a smaller range – by the application of different heat treatment parameters. Depending on composition and heat treatment, sputtered Nitinol can exhibit a two step transformation in DSC measurements, or a three step transformation involving the R-phase, just like standard Nitinol. The difference in transformation behavior is caused due to the competition between preferential grain boundary precipitation of Ni_4Ti_3 precipitates and a homogeneous precipitation within the grains for higher Ni contents[8].

Ni4Ti3 precipitates: orientation, size and distribution

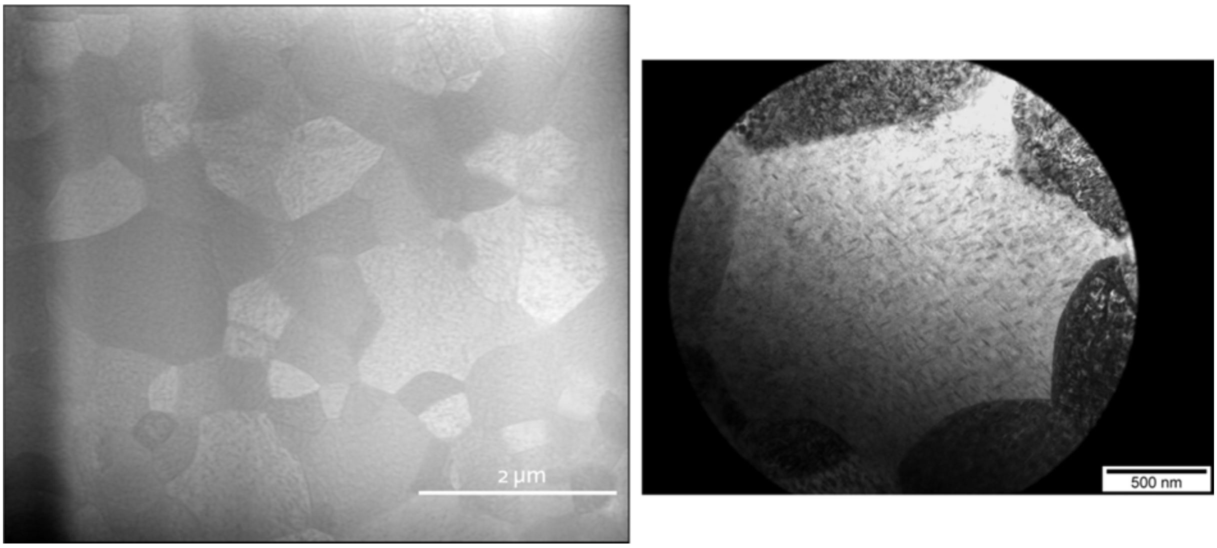

Figure 2: Typical microstructure of sputtered Nitinol: grain size in the range of 1 to 5 µm, with a fine distribution of precipitates with tens to hundreds of nm in size, finely distributed within the grains and at grain boundaries.

A key feature of sputtered Nitinol films is however their lack of oxide and carbide inclusions: going through the gas phase during the deposition process, sputtered Nitinol completely lacks oxide and carbide inclusions. In standard Nitinol, these inclusions are a source of crack initiation and propagation[9], and a source for corrosion breakdown. It was found that the smaller the inclusion size, the smaller the fraction of samples showing corrosion breakdown[10]. Sputtered Nitinol, lacking oxide and carbide inclusions, has therefore great mechanical, fatigue and corrosion properties[11].

Lack of oxide and carbide inclusions:

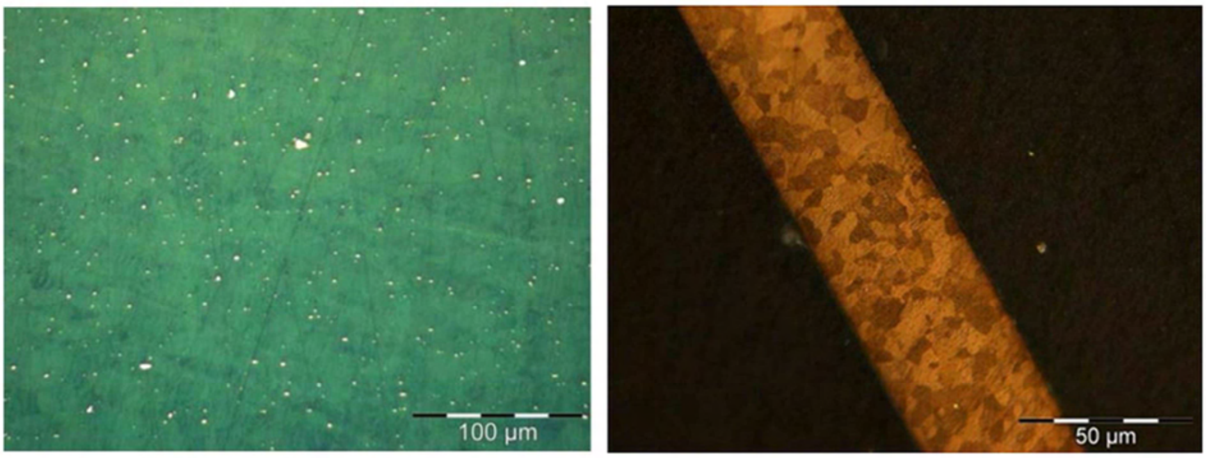

Figure 3: Left: Standard tube material drawn from standard VIM/VAR material. A high number of inclusions smaller 3µm are present, but also, however fewer, inclusions up to 5 to 10 µm. Right: Sputtered Nitinol shows a microstructure without carbide or oxide inclusions. Images in collaboration with Admedes GmbH, courtesy of Admedes GmbH, see also [11] for further details.

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