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<tr><td rowspan="1">
ESRF</td><td>Experiment title:
In situ sputtering of Shape Memory Alloy (SMA) Ni-Ti thin films</td><td>Experiment number:
20-02-608</td></tr>
<tr><td>Beamline:
BM 20</td><td>Date of experiment:
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<tr><td>Shifts:
12</td><td>Local contact(s):
Dr. Norbert Schell</td></tr>
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Report:

Ni-Ti thin films present great advantages for the fabrication of microactuactuators. Current intensive research demonstrates that unique fine microstructures are responsible for the superior shape-memory characteristics observed in thin films as compared with bulk materials [1]. The phase transformation and precipitation, which are responsible for the shape memory effect, have been widely studied in bulk and thin films. However, in situ studies have been limited to the aging/annealing treatments after deposition. In situ study of the sputter deposition of Ni-Ti thin films has not yet been reported. At ROBL, in situ studies of the crystallization kinetics [2] have been followed by in situ analysis of the sputter deposition of NiTi thin films.

EXPERIMENTAL

A sputter deposition chamber for the *in-situ* study of film growth by synchrotron x-ray diffraction and reflectivity was used [3]. The two magnetrons are placed at a distance of 100 mm from the substrates and tilted 30 degrees away from the substrate normal. To avoid cross contamination of the targets, chimneys (1 inch long) are mounted on the magnetrons. Air–pressure-controlled shutters are placed in front of the chimneys. Further experimental parameters were: base pressure appr. 2×10^{-6} mbar, target material Ni-Ti (49 at% Ni – 51 at%Ti), sputter gas Ar (99.9996%) at a pressure of 3.5×10^{-3} mbar. The magnetron (only one at the time) was run at a dc power of 40 W, resulting in a deposition rate of approximately 1.2 Å/s.

The deposition chamber was mounted on the six-circle goniometer in MRH. The incident x-rays were monochromatized to 18.367 keV (λ = 0.675 Å). To study the growth of NiTi films in-situ, two different scattering geometries were used:

1. Vertical Bragg-Brentano large scattering (XRD).
2. Low-angle specular reflectivity with information on film thickness and surface roughness.

Sample	*Substrate*	*Bias (V)*
1	SiO_2/Si (100)	0
2	SiO_2/Si (100)	-45
3	Si (100)	0
4	Si (100)	-45
5	SiO_2/Si (100)	0
6	Poly Si (CVD 675ºC)/Si (100)	0

Table 1: Parameters used for sputtering deposition.

RESULTS

Fig. 1: In-situ study during sputter deposition at 470ºC (steps of ≈ 110 nm) and during annealing (steps of ≈ 15 min).

The experimental results show that:

- when a bias is applied, a much more significant change of the peak intensities of the precipitates Ni_3Ti and Ni_4Ti_3 is present for the deposition on SiO_2/Si(100), compared to the one on Si(100) (Fig 1);
- there is a significant reduction of the B2 (110) peak area when the a bias (-45 V) is applied to the substrate (Fig. 2);
- after deposition, during the annealing (at 470ºC) that follows, the intensity of the B2(110) peak does not increase (Fig. 2); for sample 2 this stabilization is reached during deposition.

Further exploitation of these results is in course.

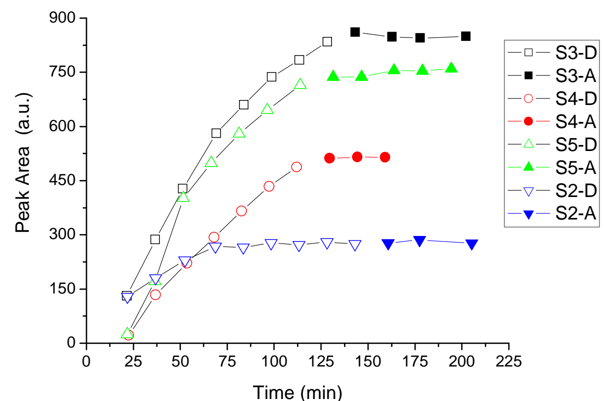

Fig. 2: Area of B2(110) in-situ Bragg-Brentano diffraction peaks, recorded during (*D*) and after (*A*) deposition at 470ºC *versus* time.

REFERENCES

[1] A. Ishida and V. Martynov, MRS Bulletin, February 2002, p. 111.

[2] "*In-situ* GIXRD characterization of the crystalli-zation of Ni-Ti sputtered thin films"; R.M.S. Martins, R.J.C. Silva, F.M.Braz Fernandes, L. Pereira, P.R. Gordo, M.J.P. Maneira, N. Schell; presented at "Materiais 2003" (submitted for publication in Materials Science Forum).

[3] W. Matz, N. Schell, W. Neumann, J. Bottiger, and J. Chevallier, Rev. Sci. Instrum. 72 (2001), p. 3344.