<table>
<tr><td> </td><td>Experiment title:
In situ XRD study of sputtered Ni-Ti SMA (Shape Memory Alloy) thin films</td><td>Experiment number:
ME-584</td></tr>
<tr><td>Beamline:
BM 20</td><td>Date of experiment:
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18</td><td>Local contact(s):
Dr. Norbert Schell</td></tr>
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Report:

Deposition of useful Ni-Ti thin films has been problematic due to compositional sensitivity. According to Ho *et al.* [1], the chemical composition of Ni-Ti sputtered films may deviate by up to 2 at% (lower Ti) when compared to the target composition. A more robust procedure is clearly desirable, and sputtering using multiple targets is an obvious solution. Preliminary studies performed by other researchers [2], indicate that the excessive roughening of Ni targets during service affects the vapor flux at constant power and suggest that Ni-Ti is a better source than pure ones. In continuation of earlier work done at ROBL (Exp **20_02_608**), experimental ***in-situ*** **studies of the structural evolution during co-sputtering using NiTi and Ti targets** were carried out.

EXPERIMENTAL

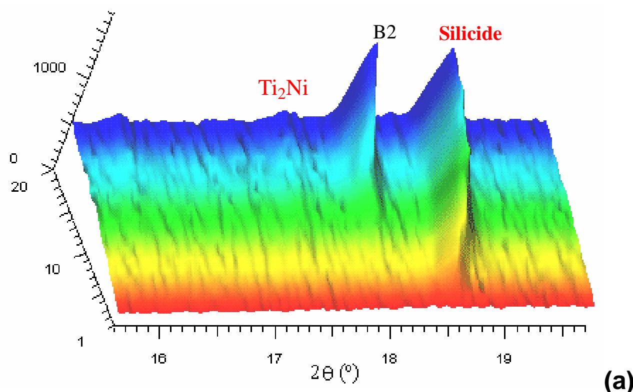
The deposition chamber is described in detail in ref. [3]. For the *in-situ* studies (incident x-rays monochromatized to 18.367 keV / $\lambda = 0.675$ Å), two different scattering geometries were used: (1) Bragg-Brentano large-angle scattering (XRD) and (2) low-angle specular reflectivity with information on film thickness and surface roughness.

Further experimental parameters were: base pressure < 3×10^{-6} mbar, target material Ni-Ti (49 at% Ni – 51 at%Ti) and pure Ti (99.999%), sputter gas Ar (99.9996%) at a pressure of 3.5×10^{-3} mbar. The substrate temperature varied from 460ºC to 480ºC during deposition.

RESULTS

Table 1: Parameters used for the *in-situ* sputtering deposition.

Sample	*Power (W)*		*Substrate*	*Bias (V)*	*Deposition procedure*	*Annealing (min)*
	NiTi	*Ti*				
8	40	40	Poly Si / Si(100)	0	Ti deposition (28 min) / NiTi deposition (2h 10 min)	80
9	40	-	Poly Si / Si(100)	-45	NiTi deposition (2h)	65
10	40	40	Si(100)	-45	4 × (6 min Ti deposition + 30 min NiTi deposition)	78
11	40	5	Si(100)	-45	4 x (35 min NiTi + Ti co-sputtering / 25 min annealing)	85
12	40	5	Si(100)	0		90
13	80	5	Si(100)	0	1h 30 min (NiTi + Ti co-sputtering)	46 at 470ºC 100 at 545ºC



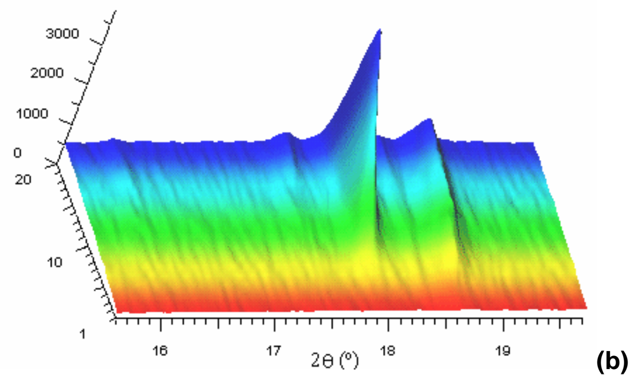
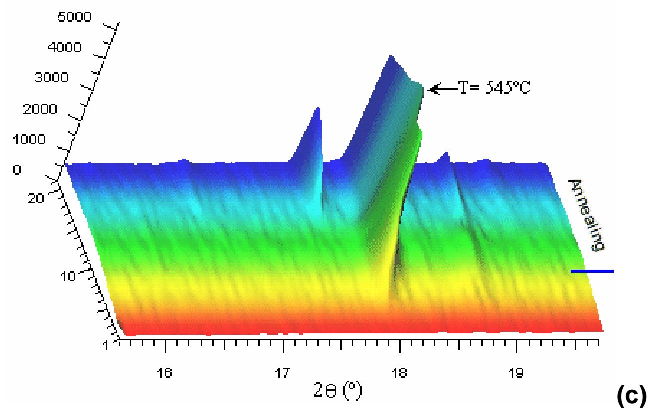
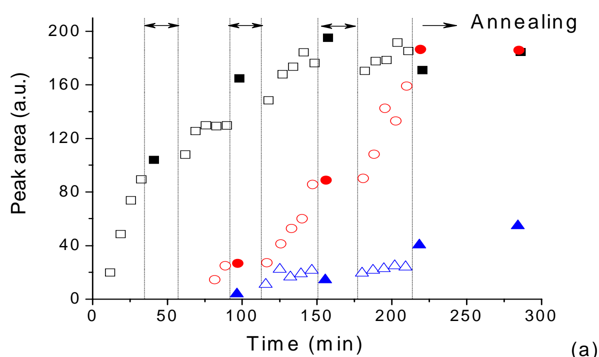


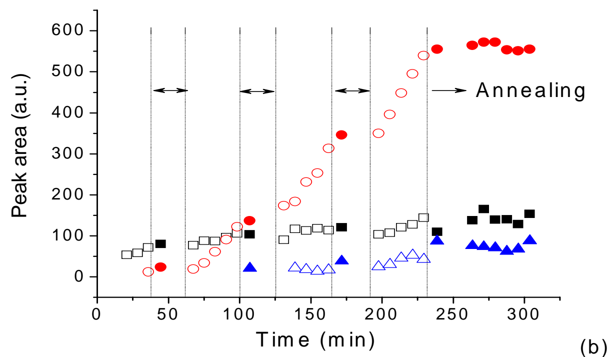
Fig. 1: *In-situ* XRD study during co-sputtering deposition and during annealing (steps of ≈ 7.5 min). *Samples:* (a) 11; (b) 12; (c) 13.

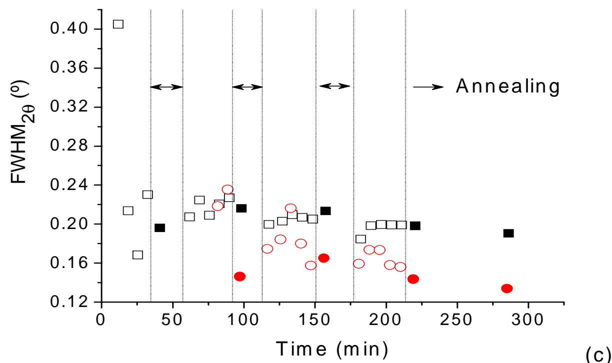


The experimental results show that:

- **a bias (-45 V) gives rise to a significant change of the peak area ratio B2(110) / B2(200)**; the **first layers of B2 stack preferentially on (h00) planes; when no bias is applied, (110) stacking prevails over (h00)**, (**Fig. 2** - a, b);
- the **FWHM changes** significantly during deposition and remains quite stable during annealing (at 470ºC); this trend is more noticeable when no bias is applied.







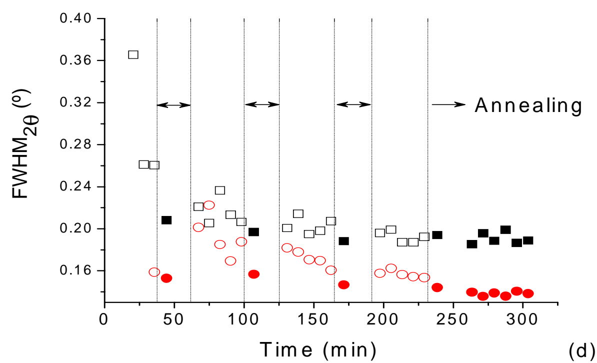


Fig. 2: Area and $FWHM_{2\theta}$ of *in-situ* XRD peaks recorded during deposition (co-sputtering NiTi + Ti) of *sample 11* (a, c) and *sample 12* (b, d), *versus* time. ( )/(■) **Ti_2Ni** (deposition/annealing); (○)/(●) **B2**; (△) (▲) **Silicide**.

This series of first experiments on the *in-situ* study of co-sputtering shows that it is possible to follow the structural evolution of sputtered thin films along the deposition process, using the facilities of the ROBL beamline.

REFERENCES

[1] K. Ho, K. Mohanchandra, and G. Carman, Thin Solid Films, **413** (2002), p. 1.
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[3] W. Matz, N. Schell, W. Neumann, J. Bottiger, and J. Chevallier, Rev. Sci. Instrum., **72** (2001), p. 3344.