ESRF	**Experiment title:** ***In-situ* XRD study of sputtered Ni-Ti SMA (Shape Memory Alloy) thin films (cont.)**	**Experiment number:** **ME-814**
Beamline: BM 20	**Date of experiment:** from: 03.03.2004 to: 09.03.2004	**Date of report:** 12.08.2004
Shifts: 18	**Local contact(s):** Dr. Norbert Schell	*Received at ESRF:* *16.08.04*

Names and affiliations of applicants (* indicates experimentalists):

F.M. Braz Fernandes*, **Rui J.C. Silva*, Márcia Silva***: CENIMAT – Centro de Investigação de Materiais, Campus da FCT/UNL, 2829-516 Monte de Caparica, PORTUGAL

N. Schell*, Rui M.S. Martins*: FZR, ROBL-CRG at ESRF, B.P. 220, F – 38043, Grenoble, FRANCE

Report:

Previous experiments at ROBL (**20_02_608 and ME-584**) were carried out using separate NiTi and Ti targets, where the power applied to each magnetron was kept constant. This new series of experiments was intended to make the *in-situ* study of the structural evolution during co-sputtering using NiTi and Ti targets with the power applied to the Ti magnetron being changed during the deposition process in order to actively design the film composition.

EXPERIMENTAL

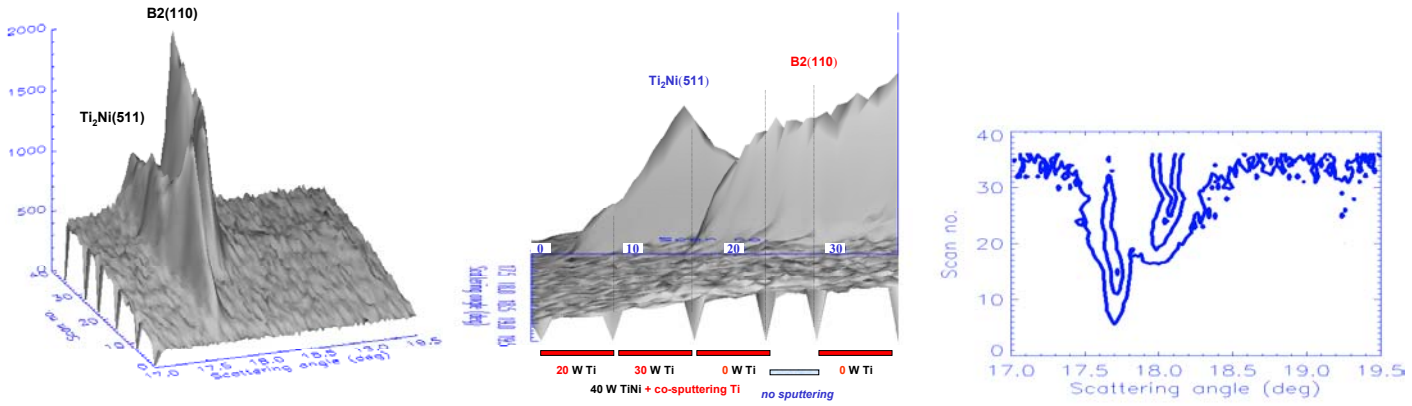
The experimental conditions used were the same as those of the previous series of experiments. All the depositions were made on Si(100) substrates.

sample	*Power (W)*		*Bias (V)*	*Annealing (min)*
	NiTi	*Ti*		
S18	40	30 min (Ti 8W) + 30 min (Ti 20W) + 34 min (Ti 30W) + 66 min (only NiTi)	0	16
S19	40	44 min (Ti 20W) + 40 min (Ti 30W) + 45 min (only NiTi) + 40 min (annealing) + 40 min (only NiTi, substrate at 600°C)	0	21.5
S20	40	42 min (Ti 8W) + 37 min (Ti 20W) + 43 min (only NiTi)	-45	29
S21	40	44 min (Ti 20W) + 45 min (Ti 30W) + 39 min (only NiTi)	0	96

Table 1 – Deposition conditions.

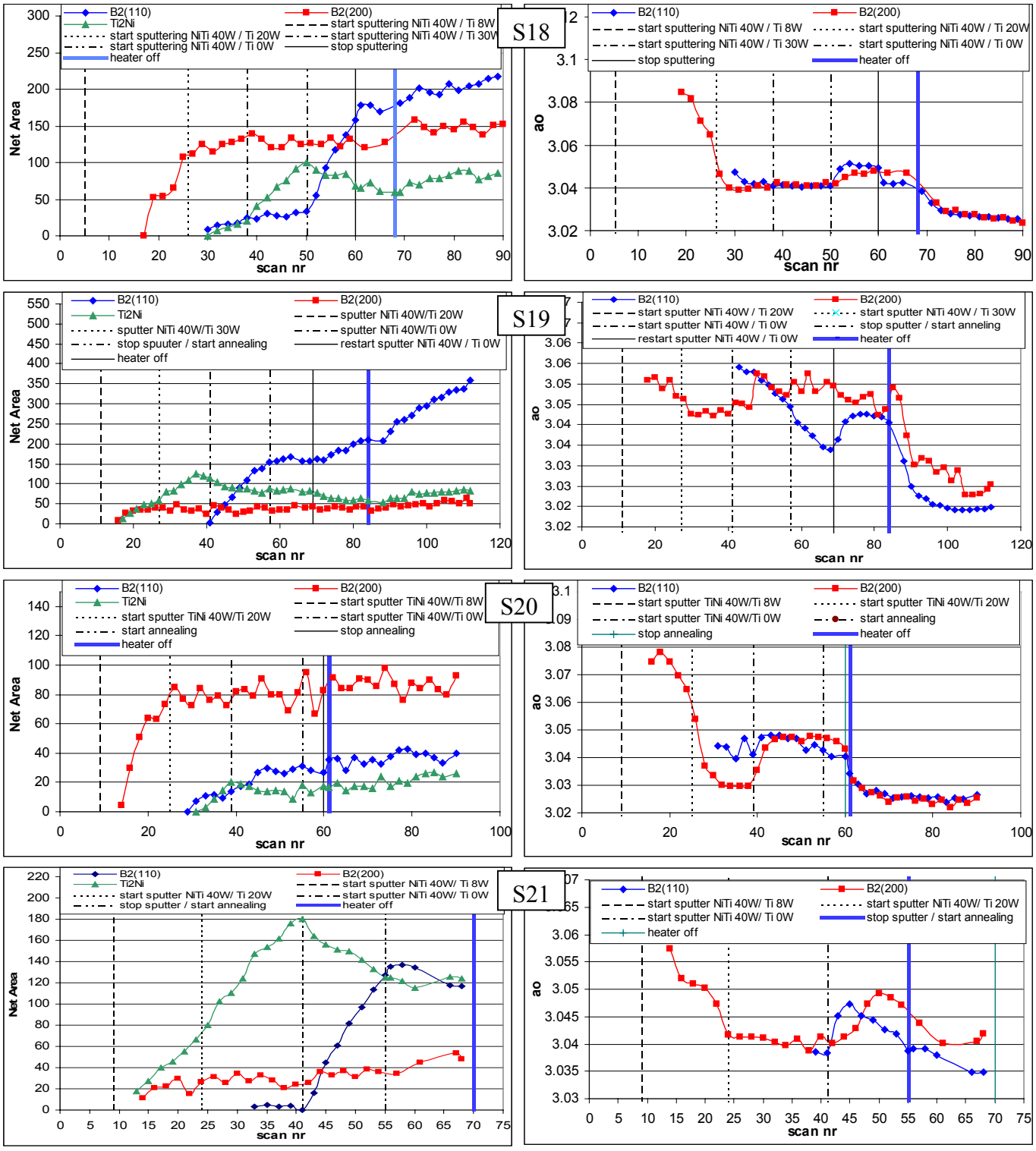
RESULTS

The 3D plot capability of the program XOP has been used to follow the structural evolution of the films during the deposition as the power of the Ti magnetron was being changed (Fig. 1).



(a) **(b)** **(c)**

Fig. 1: 3D plot of the peak intensity (a, b) and 2D intensity contour level (c) during deposition of sample S19.



(a) **(b)**

Fig. 2: (a) Net area of the B2(110) and B2(200) and Ti_2Ni(511)peaks. (b) B2 lattice parameter calculated from (110) and (200).

SUMMARY

The results (comp. Fig. 2) show that:

- there is clearly a ***different variation of the intensity peaks for different powers*** applied to the Ti magnetron,
- under these deposition conditions, the ***B2 phase always started by stacking onto (200) planes*** and, when the (110) stacking was started, the (200) peak intensity was stabilized,
- significant ***stress relaxation processes*** are taking place ***during deposition*** as seen by the significant changes of the lattice parameter as calculated from $d_{(110)}$ and $d_{(200)}$.

CONCLUSIONS

The sputter chamber installed on the 6-circle-goniometer of ROBL proves to be a very efficient instrument to follow the deposition process.