ROBL-CRG	**Experiment title:** **Crystallisation of Ni-Ti thin film Shape Memory Alloy (SMA)**	**Experiment number:** **20_02_061** 20_02_602 EU #31
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1. Thin films production

NiTi thin films were deposited on Si(100) wafers using sputtering rf technique. The chemical composition of the target material was 44 wt% Ni – 56 wt% Ti, i.e. 49 at% Ni – 51 at%Ti. The sputter deposition performed, using the conditions described, gave rise to films with thickness ranges from 0.3 µm to 1 µm.

2. GIXRD *in situ* annealing

Samples of 10x10 mm^2 were cut to be analysed by GIXRD at ROBL. The annealing of the films took place under vacuum (pressures ranging from 5.7×10^{-6} to 1.4×10^{-5} mbar). Samples of NiTi thin films (0.3 µm to 1 µm thick) deposited on Si(100) wafers, with different sputtering conditions, were studied. The temperature range covered for the crystallisation studies was from room temperature to 600ºC.

3. Results

The samples S8 and S11 (distance target-substrate during sputtering: 70 and 90 mm, respectively) showed, after annealing, the presence of the austenite phase (B2) of the Ni-Ti system and the intermetallic Ni_3Ti, as well as the presence of silicides. The sample S12 (distance target-substrate: 186 mm) only shows the presence of the Ni-rich solid solution (of Ti) and TiO_2.

Both S8 and S11 are not crystallised at 400ºC, but they show a significant degree of crystallisation at 450ºC. Increasing the temperature up to 550 or 600ºC, improves the degree of crystallisation.

Stress state determination by GIXRD was performed on the sample S8 at room temperature, after annealing at 550ºC. The results show that the residual stress state is very low.

4. Discussion

- the greater distance target-substrate (186 mm) used for the sample S12 led to a significant Ti depletion on the sputtered thin film; during annealing a further depletion of Ti by the oxide formation led to the appearance of the Ni solid solution (Ti solute);

- the films S8 and S11 showed the formation of the B2 phase, the crystallisation temperature being comprised between 400ºC and 450ºC; the presence of Ni_3Ti intermetallic shows that the sputtered thin film is slightly enriched in Ni;
- the residual stress state of the S8 film after annealing appears to be very low.