



Experiment title:
Crystallization of Ni-Ti thin film Shape Memory Alloy (SMA)

Experiment number:
ME - 474

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1. Thin films production

NiTi thin films were deposited on Si(100) wafers using sputtering dc technique. The chemical composition of the target material was 44 wt% Ni – 56 wt% Ti, i.e. 49 at% Ni – 51 at% Ti (60 x 232 x 2 mm). The substrates used for the deposition of the Ni-Ti thin films were (i) Si(100) oxidized wafers and (ii) polysilicon thin films deposited on Si(100) oxidized wafers. The thickness of the sputtered Ni-Ti films ranged from 1.4 μm to 1.6 μm (D3: 70 mm, D4: 40 mm; 5.75 W/cm², P_{Ar} = 1 mTorr). The temperature range covered for the crystallization studies was from room temperature to 430°C.

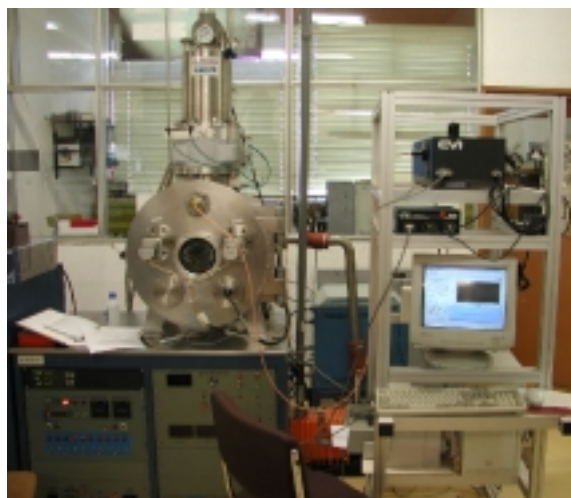


Fig. 1 – Dc sputtering equipment.

Designation	Position on the sample holder	Substrate/Target distance (mm)
D3M93	a)	70
D3M47	b)	70
D3064	a)	70
D35104	a)	70
D4M64	a)	40
D4M68	b)	40
D4044	a)	40

- a) sample located inside the vertical projection of the target on the substrate
- b) sample located outside the vertical projection of the target on the substrate (~ 25 mm from the edge)

2. GIXRD *in situ* annealing

Samples of 10x10 mm² were cut to be analysed by GIXRD at ROBL. The annealing of the films took place under vacuum (pressures ranging from 4x10⁻⁸ to 2x10⁻⁶ mbar). The grazing incidence angle was 1°.

3. Results¹

Fig. 2 shows the diffraction patterns of the NiTi thin films during and after crystallization (RT).

¹ presented at “Materiais 2003” and submitted for publ. in Mat. Sci. For. (*In-situ* GIXRD characterization of the crystallization 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).

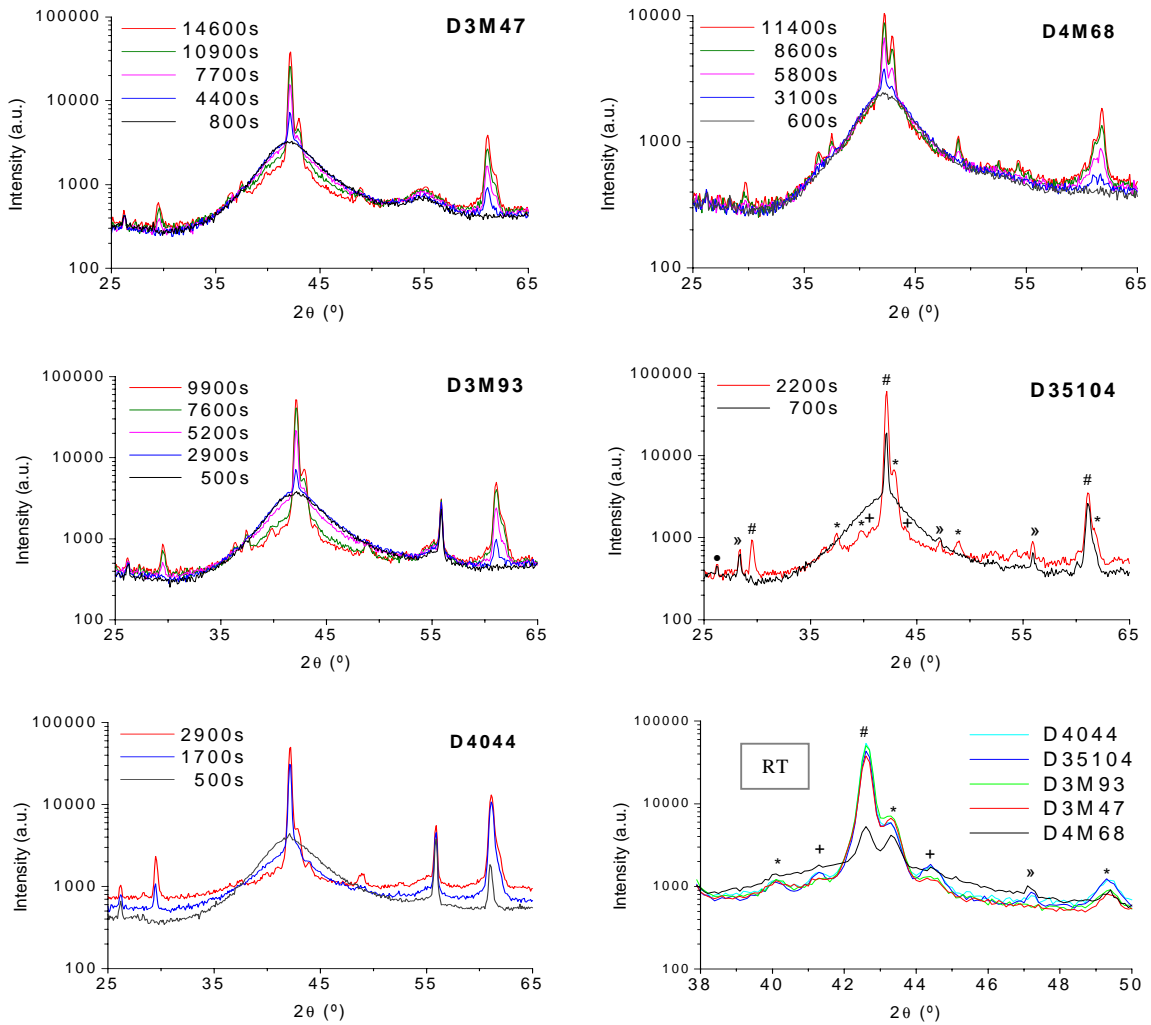


Fig. 2 – GIXRD patterns of dc and rf sputtered films at crystallization temperature and at RT after crystallization (# B2, * Ni_4Ti_3 , + Ni_3Ti_2 , • SiO_2 , » Si).

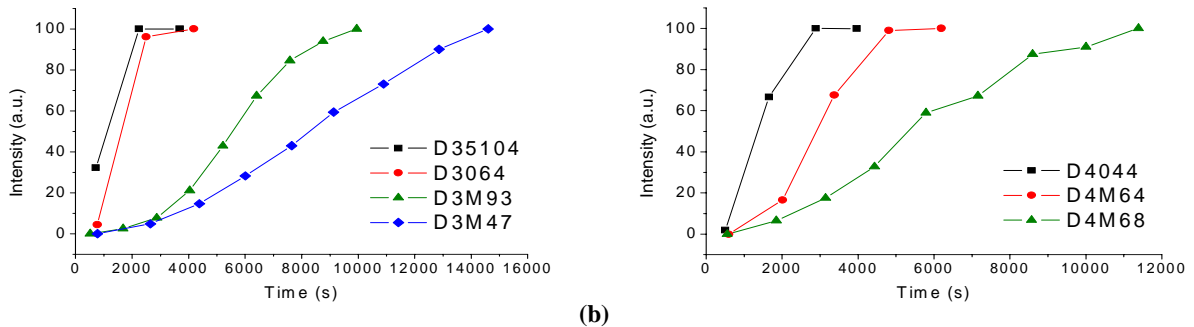


Fig. 3 – Crystallization evolution represented by the integrated intensity of the (110) austenite peak versus time during annealing at 430°C for the distance target/substrate of (a) 70 mm, (b) 40 mm.

4. Discussion

From the kinetic results, we observe that the **crystallization is significantly enhanced by:**

- **the presence of an intermediate film of polysilicon**
- **the fact that the sample lies within the limits of the vertical projection of the target on the substrate.**

During crystallization, *Ni_3Ti_2 and Ni_4Ti_3 precipitate* (as it is also reported by other authors). Using the distance target/substrate of 40 mm instead of 70 mm gives:

- no significant change of the kinetics for the depositions on polysilicon
- a significant change for the deposition on the Si(100) wafer (no intermediate polysilicon).