ROBL-CRG	**Experiment title:** ***In-situ* study of the preferential orientation of sputtered Ni-Ti thin films as a function of bias and substrate type**	**Experiment number**: **20_02_637**
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REPORT:

The preferential orientation of Ni-Ti thin films is a crucial factor in determining the shape memory behavior. The texture has a strong influence on the extent of the strain recovery. The relationship between structure and deposition parameters is of extreme importance for future device applications. Our approach is *in-situ* XRD *during* deposition carried out in a process chamber installed on the 6-circle-goniometer of ROBL [1]. Near-equiatomic films were co-sputtered from Ni-Ti and Ti targets. Substrate type and bias voltage play an important role for the preferred orientation.

EXPERIMENTAL

For the deposition of the Ni-Ti films a 1-in Ni-Ti target (49 at% Ni – 51 at% Ti) and a 1-in Ti target (purity 99.99%) were inserted on the unbalanced magnetrons. The base pressure was about 2×10^{-5} Pa, and the films were deposited under an Ar pressure of 0.42 Pa. The Ni-Ti and Ti magnetrons were driven at a power of 40 and 20 W, respectively, resulting in deposition rates of roughly 1 Å/s. For the deposition of the TiN buffer layer, the Ti target was run at a constant power of 80 W with an Ar/N_2 gas flow of 2/0.5 sccm. The deposition and annealing temperature was ≈ 470°C. The processing conditions of the samples studied are presented in Tab. 1. The scans were run in Bragg-Brentano geometry, using 0.675 Å radiation. Specular reflectivity (XRR) has been used for growth rate calculation of the TiN buffer layer.

Sample	*Substrate*	*Buffer layer*	*Substrate bias (V)*		*Deposition (min.)*		*Annealing (min.)*	
			TiN deposition	*Ni-Ti deposition*	*TiN*	*Ni-Ti*	*TiN*	*Ni-Ti*
S36	SiO_2^*/Si(100)	–	–	0	–	122	–	146
S37	Si(111)	–	–	0	–	123	–	146
S38	SiO_2^*/Si(100)	TiN	-30	0	–	120	16	139
S39	Si(100)	–	–	-25	3	121	–	141
S40	MgO(110)	–	–	0	–	122	–	148
S41	SiO_2^*/Si(100)	TiN	-30	-45	3	122	94	138

* *1400 Å amorphous SiO_2 capping layer*

Tab. 1 – Deposition parameters for the various samples investigated.

RESULTS AND DISCUSSION

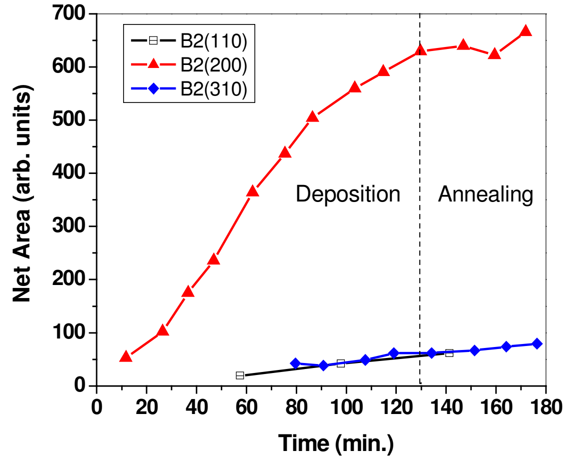


Fig. 1. – *Sample S36:* the integrated intensities of the Bragg-Brentano B2(110), B2(200) and B2(310) diffraction peaks as obtained from the positions of the respective peaks, recorded as a function of time after start of film growth.

For the Ni-Ti film **on thermally oxidized Si(100) without a substrate bias voltage** (*sample S36*), there is a strong preferential stacking on (h00) planes of B2 phase leading to a (100) fiber texture *(*Fig. 1*)* [2].

Figure 2 shows that the use of a **substrate bias voltage on naturally oxidized Si(100)** (*sample S39*) leads to a film growth where, at the beginning, the {200} planes are stacking parallel to the substrate and, after approximately 40 min, the B2(110) is finally detected; then, the B2(200) intensity stays constant but the B2(110) intensity also does not increase [3]. An XRD scan in a larger range has shown the existence of grains with the (310) orientation parallel to the substrate (Fig. 3). Work is in progress to investigate the complete texture by the realization of pole figure measurements.

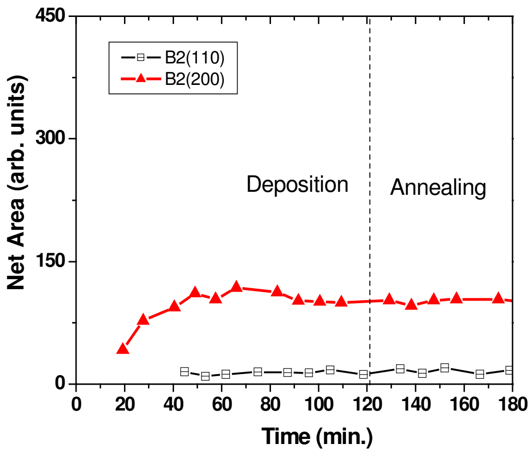


Fig. 2. – *Sample S39:* the integrated intensities of the Bragg-Brentano B2(110) and B2(200) diffraction peaks as obtained from the positions of the respective peaks, recorded as a function of time after start of film growth.

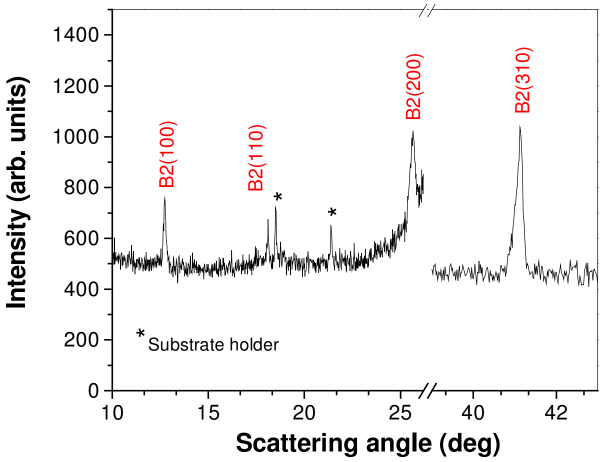


Fig. 3. – XRD diffractogram from the Ni-Ti *sample S39* during annealing after deposition stop at the same (deposition) temperature.

The behavior observed for *sample S40* by low angle time resolved XRR for the growing mode of the **first few monolayers on MgO(110)**, shows a growth mode of the type layer by layer of the Ni-Ti film on the substrate (Fig. 4). The intensity of the B2(110) peak is very high. However, contrary to the deposition on MgO(100) substrates (*sample S24*, [report ME-936]) where only the (100) orientation paralell to the substrate was detected [4], here not only the (110) but the (211) orientation of the B2 phase is also observed (Fig. 5), although not comparable in intensity.

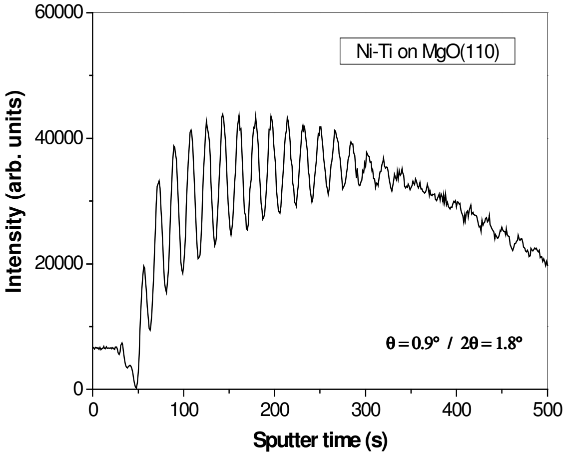


Fig. 4. – Time-dependent *in situ* XRR for the first minutes of deposition of the Ni-Ti film on MgO(110) substrate (*sample S40*).

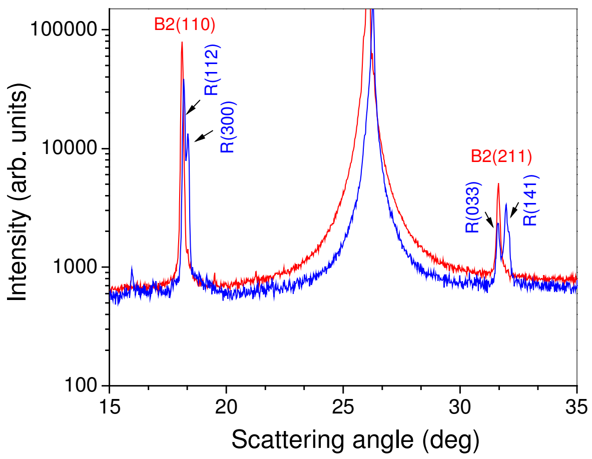


Fig. 5. – XRD diffractograms of *sample S40* during annealing after deposition stop at the deposition temperature (red line) and at room temperature (blue line).

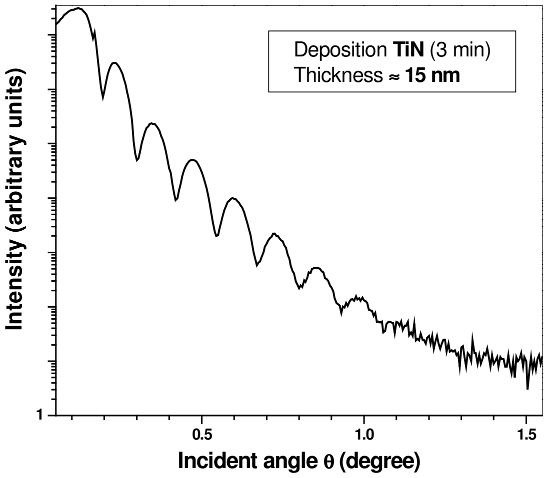
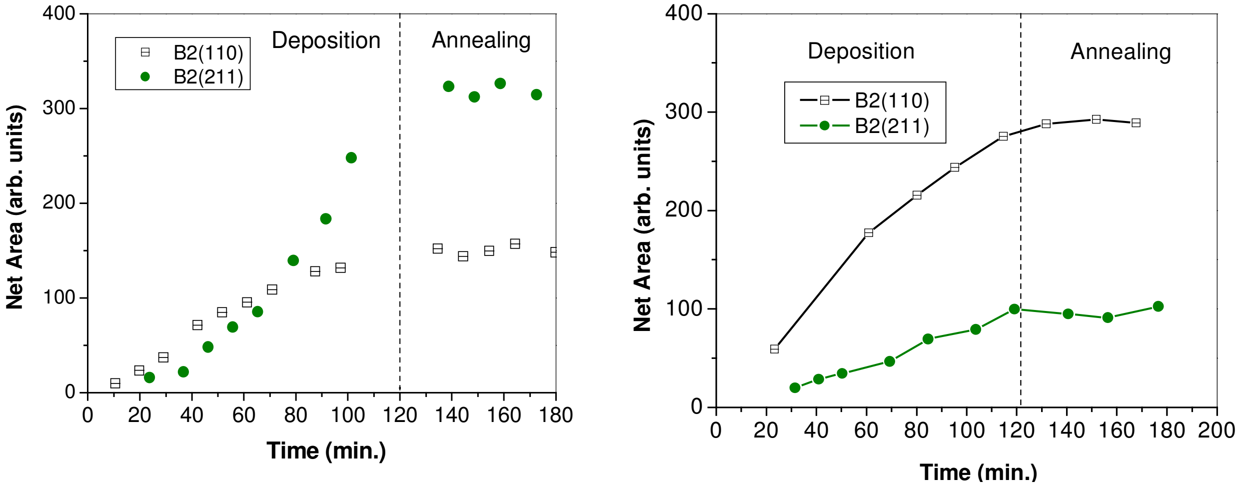


Fig. 6. – XRR spectra for a 3 min deposition of TiN (buffer layer of *sample S38*).

We have also been studying the effect of a **TiN layer deposited on top of the SiO_2/Si(100) substrate** prior to the deposition of the Ni-Ti films [5]. During this beamtime we could take profit of a new sputtering chamber with enlarged Be windows. This setup allowed to follow *in situ* also the B2(211) peak of the B2 phase. So, it was possible to observe that a TiN buffer can be used to develop the (211) fiber texture of B2.

The effect of a TiN buffer layer with a thickness of about 15 nm (Fig. 6) was tested. For the *sample S38* <110> oriented grains of the Ni-Ti B2 phase dominate at small thicknesses while <211> oriented grains take over at larger thicknesses (Fig. 7a). Apparently, the TiN buffer layer has a considerable role in the appearance of the <211> oriented grains of the Ni-Ti B2 phase. Most likely, already since the beginning of the deposition, <211> oriented grains are present. An initial competitive growth between the <211> and <110> crystal orientations occurs. The <211> orientation gradually overgrows the <110> orientation.



(a) **(b)**

Fig. 7. – The integrated intensities of the Bragg-Brentano B2(110) and B2(211) diffraction peaks, recorded as a function of time after start of Ni-Ti deposition. The Ni-Ti film was deposited on a TiN layer (≈ 15 nm) previously deposited on top of a SiO$_2$/Si(100) substrate: *a) sample S38* (without bias), *b) sample S41* (with bias).

The Ni-Ti deposition with a substrate bias of -45 V (*sample S41* – Fig. 7b) has shown that the development of the (211) fiber texture is visibly reduced when compared with the one deposited without bias (*sample S38*) and the (110) fiber texture dominates since the beginning. The more energetic ion bombardment of the growing film – due to the substrate bias – favours the stacking of (110) planes of the B2 phase parallel to the substrate.

CONCLUSIONS

- An amorphous SiO$_2$ buffer layer induces the development of the (100) orientation of the B2 phase during deposition on heated substrates (≈ 470ºC).
- In previous studies it has been observed that on naturally oxidized Si(100) substrates the Ni-Ti B2 phase starts by stacking onto (h00) planes and then, very early in the deposition process, it changes to a (110) fiber texture [6-8]. During this beamtime, using a substrate bias voltage the same initial behavior was observed, but a (110) fiber texture was not detected. Instead, grains with the (310) orientation parallel to the substrate dominate.
- Using a MgO(110) substrate, a strong stacking of (110) planes of the B2 phase parallel to the substrate could be induced.
- Ni-Ti films were deposited on top of a TiN layer where a dominating orientation of TiN could not be identified (primarily <001> and <111> oriented grains nucleate and grow). Without applying a substrate bias voltage, <110> oriented grains of the Ni-Ti B2 phase dominate at small thicknesses while <211> oriented grains take over at larger thicknesses. A substrate bias (-45 V) reduces the development of the (211) fiber texture leading to a dominating (110) fiber texture.

References

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