SIMULTANEOUS MEASUREMENT OF ELECTRICAL RESISTIVITY AND X-RAY DIFFRACTION DURING R-PHASE FORMATION ON Ni-Ti SMA



F.M. BRAZ FERNANDES Professor Associado CENIMAT/I3N, FCT-UNL Caparica – Portugal



K.K. MAHESH Investigador Auxiliar CENIMAT/I3N, FCT-UNL Caparica – Portugal



R.J.CORDEIRO SILVA Professor Auxiliar CENIMAT/I3N, FCT-UNL Caparica – Portugal



R.M. SANTOS MARTINS Bolseiro Post-Doc ITN Sacavém – Portugal

ABSTRACT

In situ XRD has been used by the authors to study the structural changes during (i) the crystallization of Ni-Ti thin films [1], (ii) the growth of Ni-Ti thin films by sputtering [2-4], and (ii) the transformation characteristics of bulk N-Ti subject to thermomechanical treatments [5-6]. The phase transformations of Ni-Ti SMA can also be investigated by measuring some physical properties such as electrical resistivity (ER) as a function of temperature. During cooling of Ni-Ti SMA from B2-phase, the resistivity value decreases linearly with the temperature down to Rs, where R-phase self-accommodated (by twinning) starts to be formed. Twinning in an alloy matrix results in electron scattering, which in turn leads to the increase of the ER [7]. Additional cooling below Rf promotes the continuous increase of rhombohedral distortion angle of the R-phase. It is assumed that this rhombohedral distortion is the reason for a further increase of ER (between Rf and Ms). Below Ms, this distortion is relaxed by the R-phase transforming to monoclinic B19' martensite, giving a gradual decrease of ER. These transformations can be thermally induced or stress induced and the electrical resistivity variations that are associated may be used to monitor different types of situations [7].

The simultaneous measurement of the electrical resistivity and XRD during thermal cycles allows establishing a direct correlation between features of electrical resistivity variation and structural changes. It is usually assumed in the published literature [8] that, during cooling, the B2 to R-phase transformation shows a significant electrical resistivity increase associated to (i) formation of R-phase from Rs to Rf, followed by (ii) increasing rhombohedral distortion (with no further R-phase formation).

Data obtained during beamtime at ESRF (2009 and 2010) confirmed that the rhombohedral distortion is notorious (variation of the peak position of (211) and (300) from R-phase) when cooling below the temperature usually identified as Rf. But data obtained also show that the net areas of R-phase peaks are still increasing during the last step of the electrical resisitivity increase. These results suggest that, besides a significant rhombohedral distortion below the temperature usually assigned as Rf, there is still new R-phase formation.



Figure 1: (a) Overall phase transformation sequence of a Ni-Ti thin film during cooling from 100°C to – 100°C, followed by heating to 100°C.. (b) Superposition of integrated intensity of R-phase peak (102) and electrical resistivity variation as a function of temperature

ACKNLOWLEDGEMENTS

Financial support from ESRF for the beamtimes (January 2009 and February 2010) and FCT/MCTS for the project PTDC/CTM/66380/2006 and the pluriannnual financial support of CENIMAT/I3N is gratefully acknowledged by the authors. KKM gratefully acknowledges the fellowship with the Ref. C2007-443-CENIMAT-6/Ciencia2007. J. van Borany, C. Baehtz and N. Jeutter are gratefully acknowledged for the preparation of the experiments at ROBL - ESRF.

REFERENCES

- R.M.S. Martins *et al.* "The influence of a poly-Si intermediate layer on the crystallization behaviour of Ni-Ti SMA magnetron sputtered thin films". *Applied Physics A - Materials Science & Processing* 83 (2006) 139-145.
- [2] N. Schell et al. "Real-time and in-situ structural design of functional NiTi SMA thin films". Applied Physics A - Materials Science & Processing, 81 (2005) 1441-1445.
- [3] R.M.S. Martins *et al.* "*In-situ* study of Ni-Ti thin film growth on a TiN intermediate layer by x-ray diffraction". *Sensors and Actuators B: Chemical* 126 (2007) 332-337.
- [4] F.M. Braz Fernandes et al. "Texture Development in Ni-Ti Thin Films". Advances in Science and Technology Vol. 59 (2008) 69-76.
- [5] A.S. Paula et al. "Study of the textural evolution in Ti-rich NiTi using synchrotron radiation". Nuclear Instruments & Methods Physics B 246 (2006) 206-210.
- [6] A.S. Paula et al. "Evolution of phase transformations after multiple steps of marforming in Ti-rich NiTi SMA". European Physical Journal 158 (2008) 45–51.
- [7] V. Novák *et al.* "Electric resistance variation of NiTi SMA wires in thermomechanical tests: Experiments and simulation". *Materials Science and Engineering A* 481–482 (2008) 127–133.
- [8] P. Šittner *et al.* "Deformation processes in functional materials studied by *in situ* neutron diffraction and ultrasonic techniques". *Materials Science and Engineering A* 462 (2007) 12–22.