ESRF	**Experiment title:** In-situ phase evolution of sputtered reactive multilayers: Effect of the substrate	**Experiment number**: MA 1985
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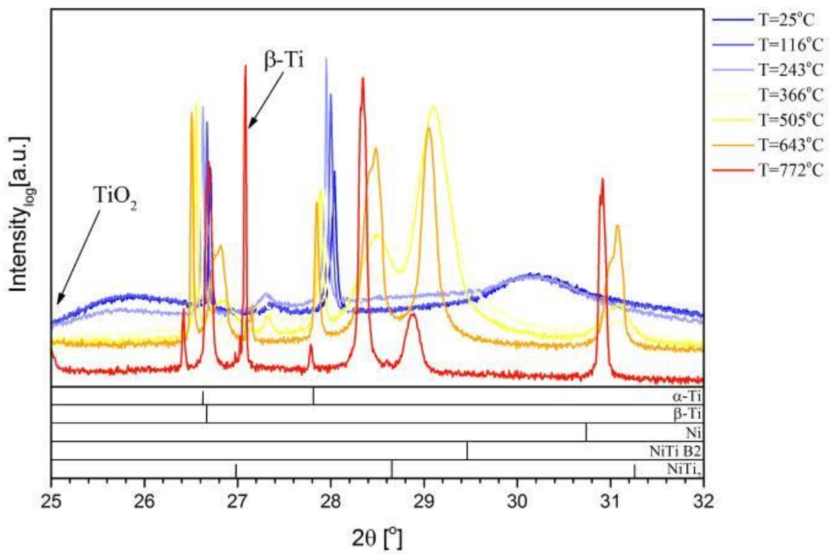
Report:

Ni/Ti (low energy) and Pd/Al (high energy) reactive multilayers (ML) with nanometric periods (Λ) were studied regarding structural evolution and thermal cycle/susbtrate influence. Moreover, a tungsten diffusion barrier was deposited onto the NiTi substrates (in the case of the Ni/Ti MLs) to assess the influence of Ni diffusion in the reaction.

Low energy Ni/Ti MLs diffusion study

2.5 µm thick Ni/Ti MLs were deposited onto a Ti6Al4V substrate with two modulation periods (12 and 25 nm) and subjected to isothermal XRD scans at different temperatures. Two different ML configurations were studied:

1. MLs with a Ni interlayer to ensure a high Ni concentration at the substrate/film interface – Figure 1 a);
2. MLs with a 15 nm W interlayer to work as diffusion barrier – Figure 1 b).



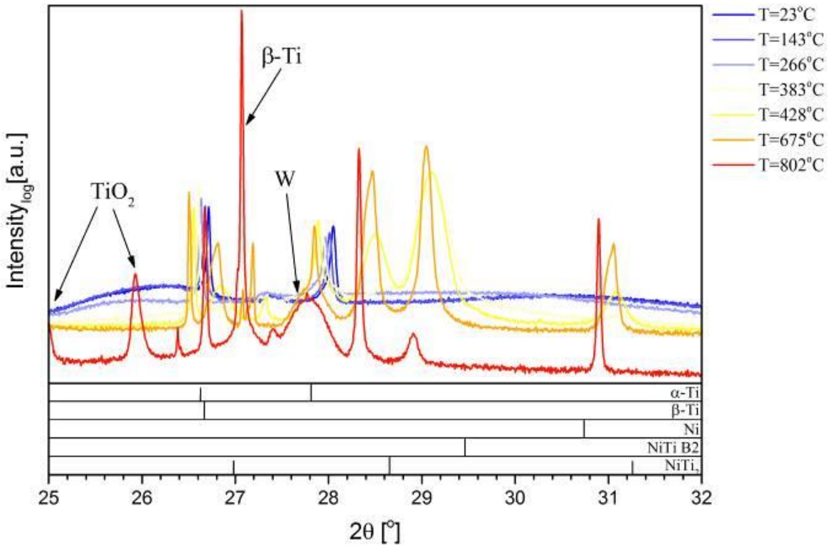


Figure 1. XRD diffractograms of a Ni/Ti ML (Λ= 25 nm) as function of temperature. a) Ni interlayer, b) W interlayer.

Some of the conclusions obtained can be summarized as follows:

- The W diffraction peak becomes detectable only after a certain reaction time and may be interpreted as resulting from a decreased fraction of Ni (higher Z than Ti) on the layers above W.
- The Ni diffusion across the W layer is corroborated by the increased intensity of Ti-β (Ni is a beta-stabilizer).
- Above 750°C it was not possible to avoid the presence of TiO_2.

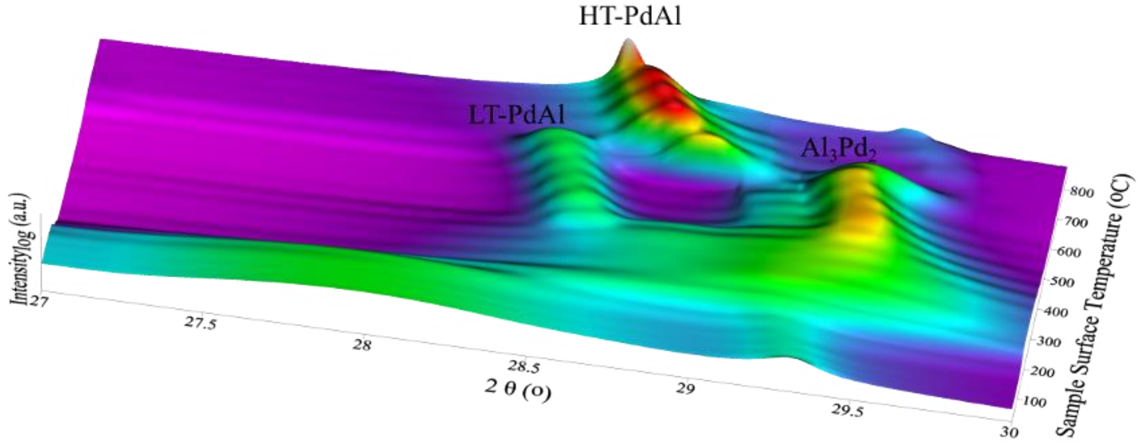
High Energy Pd/Al MLs thermal evolution

2.5 µm thick Pd/Al MLs were deposited onto a NiTi substrate with different modulation periods (5, 15 and 35 nm) and subjected to two different heating cycles:

1. Slow heating – 50°C steps from 100°C to 900°C with isothermal θ-2θ scans at each temperature step – Figure 2 a);
2. Moderate heating – single step heating with linear array of detector's pixels fixed enabling relatively fast (~1.3s) acquisitions centered at 2θ=28.4° – Figure 2 b);

The use of synchrotron radiation allowed the phase evolution sequence of Pd/Al MLs to be unequivocally resolved, while highlighting the influence of the heating rate. Some of the conclusions obtained can be summarized as follows:

- Pd- and Al-rich nanolayers are perfectly distinguishable in as-deposited films, although during sputtering intermixing and reaction occurred at the interfaces;
- Whatever the period, the films annealed at 900°C always evolve to the high temperature HT-AlPd phase
- During slow heating, the formation of intermediate phases was never avoided and the presence of Al_3Pd_2 and low temperature PdAl phases was detected, even for Λ=5 nm;
- At moderate heating rates it was possible to form HT-PdAl at lower temperature.



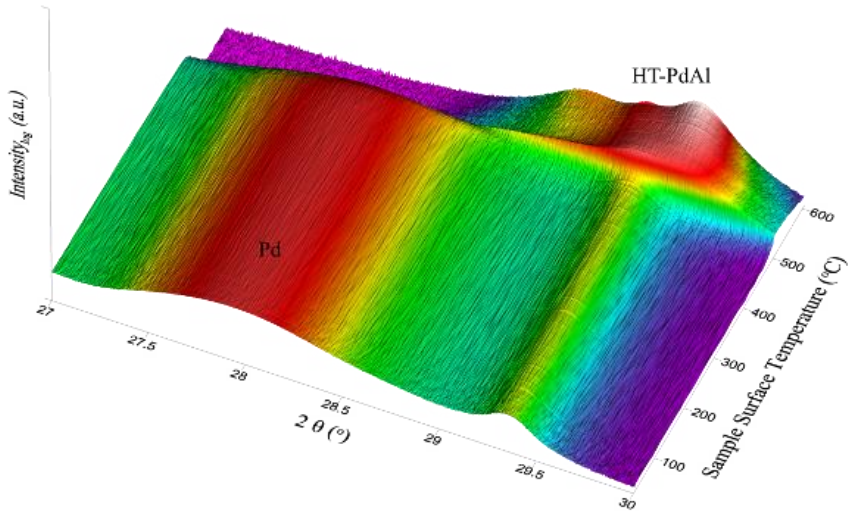


Figure 2. XRD diffractograms of a Pd/Al ML (Λ= 35 nm) as function of temperature. a) slow heating, b) moderate heating.

Publications:

[1] A.J. Cavaleiro, "Ni/Ti reactive multilayers for Joining", PhD Thesis, University of Coimbra, 2015.

[2] A.J. Cavaleiro, A.S. Ramos, F.M. Braz Fernandes, C. Baehtz, M.T. Vieira, paper in preparation regarding the Ni diffusion from the Ni/Ti multilayer towards the Ti6Al4V substrate .

[3] A.J. Cavaleiro, A.S. Ramos, F.M. Braz Fernandes, C. Baehtz, M.T. Vieira, "Thermal evolution of Pd/Al nanomultilayers using two distinct heating cycles", poster presented at the 3rd Portuguese National Meeting of Synchrtron Radiation Users – ENURS. Marinha Grande, Portugal, April 2014.