ROBL-CRG	**Experiment title:** **In situ study of nanostructured NiTi shape memory alloys during annealing after severe plastic deformation**	**Experiment number:** **MA-419**
Beamline: BM 20	**Date of experiment:** from: 07.02.2008 to: 12.02.2008	**Date of report:** 22.11.2008
Shifts: 15	**Local contact(s):** **Carsten Baehtz**	*Received at ROBL:*

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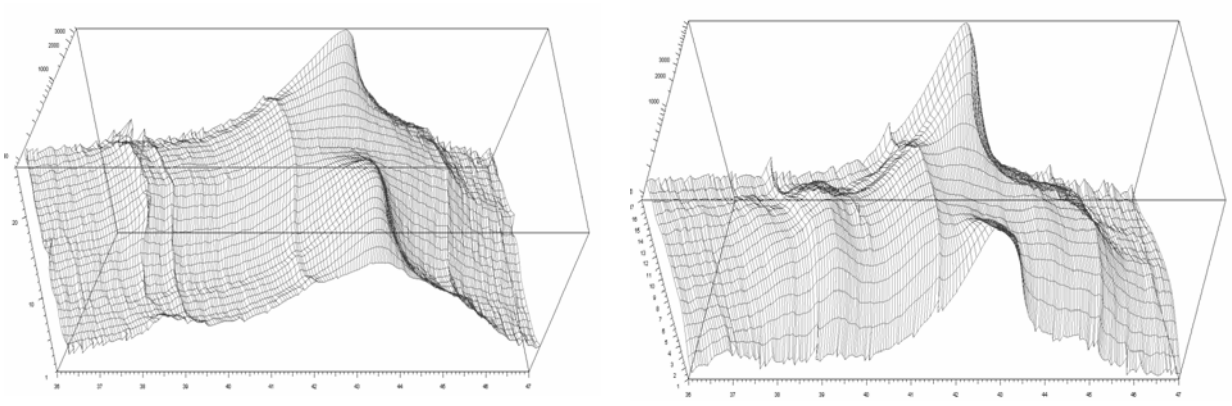
Report:

This campaign has suffered from a significant instability of the beam supplied (~40% beam time loss due to low intensity and no refilling during 6 shifts due to booster failure). As a consequence, only a small part of the scheduled experiments have been performed: the annealing at 200 and 300ºC for 2 Ni-Ti alloys: a Ni-rich (alloy "N") and a Ti-rich (alloy "H") that have been subject to severe plastic deformation by cold-rolling to 40% thickness reduction.

It was intended to complement these studies with the following information (not possible during MA-419):

- the effect of deformation heterogeneity (in depth) by analysing the surface of a sample extracted from mid-thickness of the rolled material;
- the effect of annealing temperature of 350ºC (only 200 and 300 have been studied previously)

EXPERIMENTAL

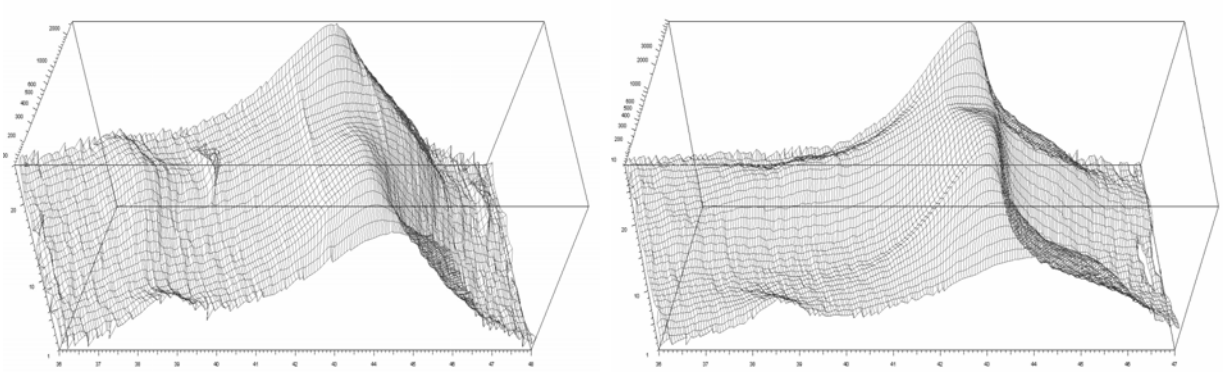


(a) (b)

Fig.1: In situ XRD of Ti-rich NiTi alloy initially cold-rolled (up to 38% thickness reduction):
(a) structural evolution during annealing up to 200ºC, cooling to -100ºC, heating to +80ºC;
(b) structural evolution during annealing up to 300ºC, cooling to -115ºC, heating to +80ºC.

The results obtained with Ti-rich alloy (H) are shown in Fig. 1. It may be noticed that:
- during the first heating cycle – annealing up to 200ºC, Fig. 1 (a); annealing up to 300ºC, Fig. 1 (b) – the transformation temperature (As) from martensite (B19') to austenite (B2) is shifted above 200ºC (compared with the As close to 100ºC of this alloy); this transformation seems to take place without intermediate phase (R-phase) formation;
- during the thermal cycle between room temperature and – 100ºC (Fig. 1 –b) for the determination of the transformation characteristics of the annealed material, the transformation from B2 to B19' (cooling) and B19´to B2 (heating) is taking place in one single step, without the formation of the R-phase.

The results obtained with Ni-rich alloy (N) are shown in Fig. 2. It may be noticed that:
- the as deformed material (at room temperature) contains stress induced martensite;
- during the first heating cycle, the transformation temperature (As) from martensite (B19') to austenite (B2) is shifted above 250ºC, so that after annealing at 200ºC still some martensite remains; only after annealing at 300ºC the martensite diffraction peaks fully disappear;
- during the thermal cycle between room temperature and – 100ºC (Fig. 1 –b) for the determination of the transformation characteristics of the annealed material, the transformation from B2 to B19' (cooling) and B19´to B2 (heating) seems to take place in one single step, without the formation of the R-phase.



(a) (b)

Fig. 2: In situ XRD of Ni-rich NiTi alloy initially cold-rolled (up to 38% thickness reduction):
(a) structural evolution during annealing up to 200ºC, cooling to -115ºC, heating to +100ºC;
(b) structural evolution during annealing up to 300ºC, cooling to -118ºC, heating to +100ºC.

Estimation of the crystallite size using Scherrer formula gives 150 nm and 90 nm for the austenite at 300ºC, respectively for the Ti-rich and the Ni-rich alloy.

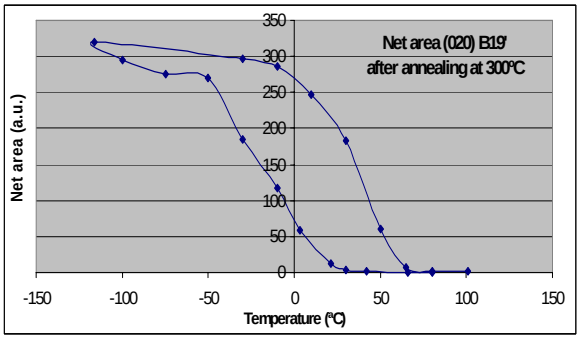


Fig. 3 – Transformation characteristics of cold-rolled H alloy (Ti-rich) after annealing at 300ºC.

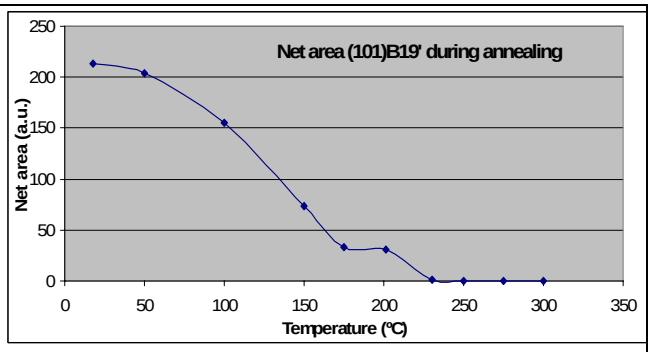


Fig. 4 – Stability of martensite during annealing of cold-rolled N alloy (Ni-rich).