ESRF	**Experiment title:** ***In situ stress state analysis during thermal cycling of Ni-Ti Shape Memory Alloy***	**Experiment number:** **ME-705**
Beamline: BM 20	**Date of experiment:** from: 30.10.2003 to: 04.11.2003	**Date of report:** 29.02.2004
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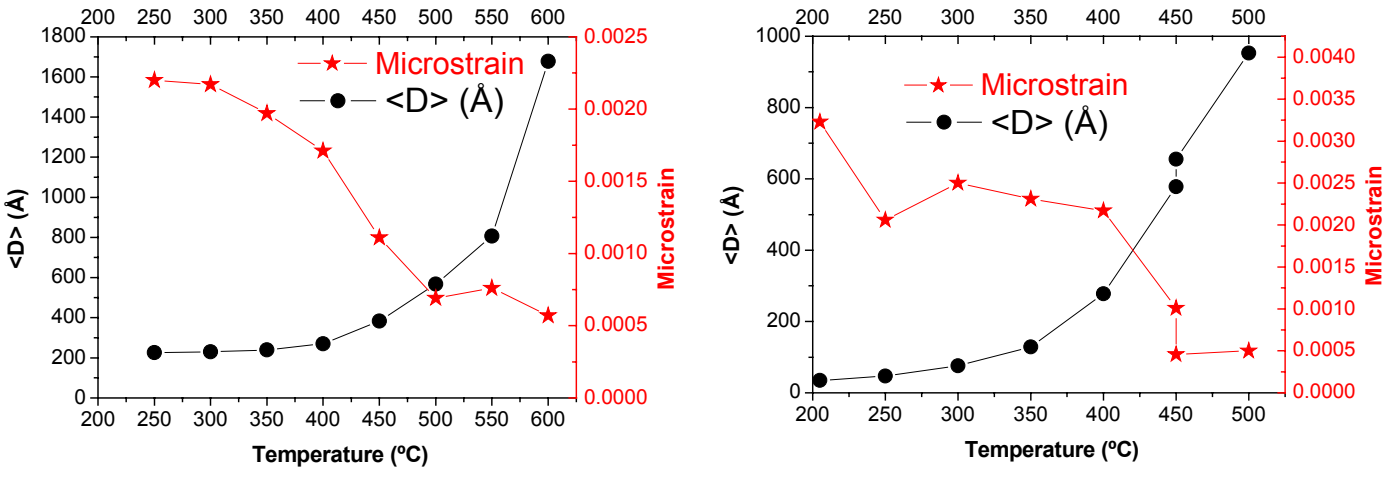
Report:

The aim of the experiment was to follow the microstrain/microstress evolution during annealing of Ni-Ti shape memory alloy subject to different thermomechanical treatments. As this type of structural evolution is accompanied by changes in the preferential orientation, the conditions for the in situ determination of pole figures were also tested. This type of study is relevant to the envisaged applications, because of the anisotropic response of the shape memory effect.

Experiment

A series of samples in different thermomechanical conditions have been studied using the Be-dome furnace installed on the 6-circle goniometer of the BM-20 beamline. The samples were extracted from a 2 mm thick plate supplied by Memory-Metalle GmbH, the chemical composition being 49 at% Ni –51 at% Ti; in situ annealing has been carried out up to 700 ºC, under a vacuum better than 10^{-6} mbar. The temperature control of the sample was done by a thermocouple in contact with the sample's upper surface. The wavelength of the incident beam was set at 1.54 Å. The pole figures have been determined for $-45º<\phi<+45º$ and $-60º<\chi<0º$, the rolling direction (RD) being aligned with $\phi = -45º$.

Results



(a) (b)

Figure 1 - Coherency domain length <D> and microstrain as a function of annealing temperature for the (a) as-received sample and (b) quenched from 500ºC, followed by 40% deformation (rolling).

Between 300 and 500ºC there is a significant change of the microstrains and the coherency domain length (Figure 1), as well as the type of preferential orientation of the samples that have been studied.

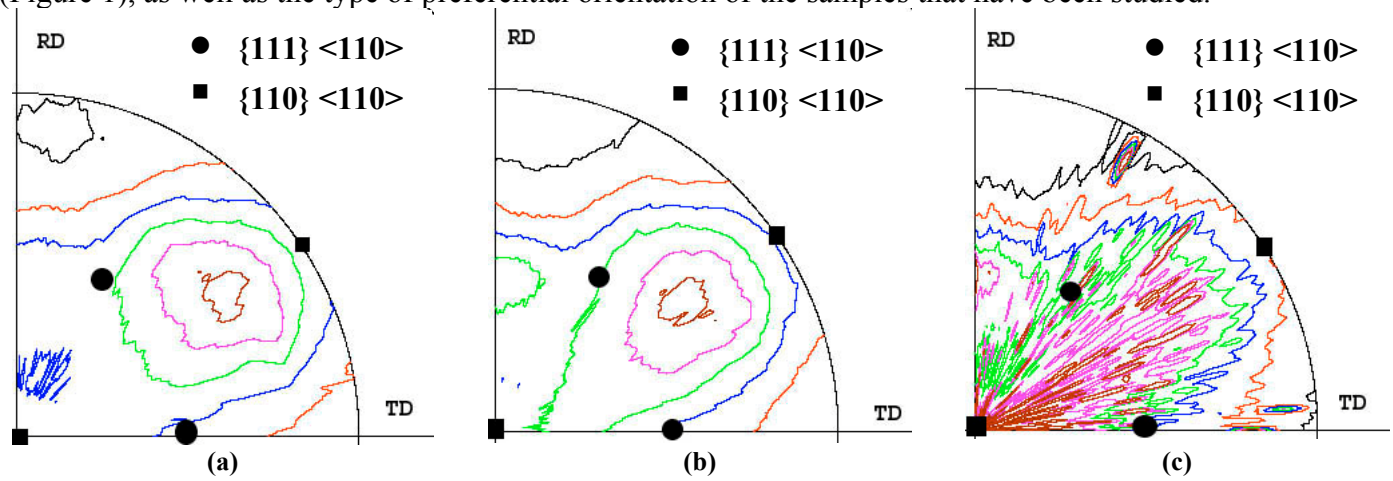


Figure 2 – {110} pole figures of B2 phase for the as received material during annealing at (a) 150ºC, (b) 400ºC, (c) 500ºC.

It has been identified the presence of Ti_2Ni already in the as received material before any further treatment (Figure 3). The corresponding peaks are not detected at room temperature, where the martensite phase (B19') is stable, because of the superposition of the peaks Ti_2Ni / B19'. Only after heating the sample into the austenitic (B2) field the Ti2Ni peaks can be detected. But, at the end of the annealing at 700ºC, the Ti_2Ni peaks have significantly increased, denoting a precipitation during annealing (Figure 4).

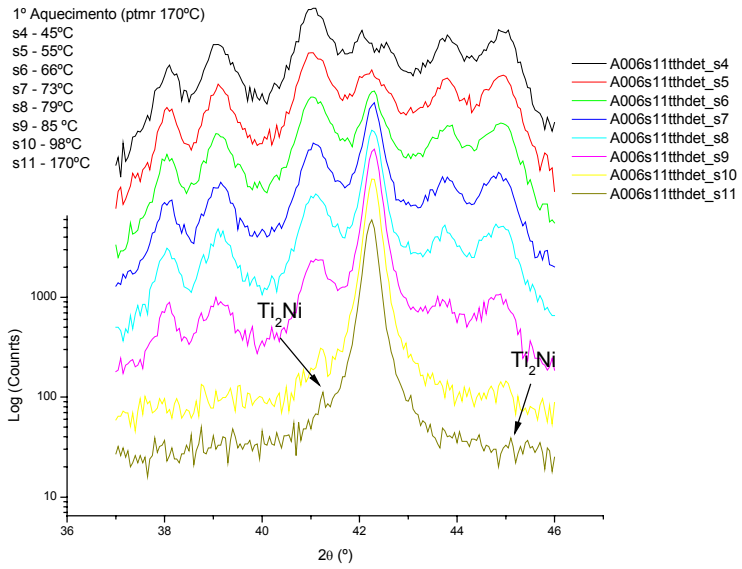


Figure 3 – Spectra sequence showing the gradual transformation of martensite (B19') into austenite (B2) during heating (from top to bottom) before annealing at 700ºC.

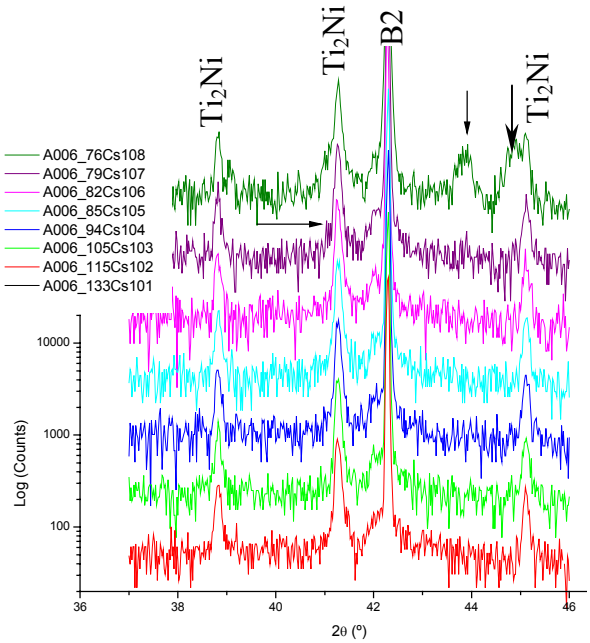


Figure 4 – Spectra sequence illustrating the start of formation of B19' (marked by arrows) during cooling after the material has been annealed at 700ºC.

Discussion

The only high temperature texture determination for polycrystalline Ni-Ti that is reported in the literature [1] has been made at 120ºC. In this study we have tested the feasibility of high temperature pole figures determination in BM-20 and we have showed that there is a relation between the preferential orientation changing and the structural evolution taking place during annealing. It is thus very interesting to continue this study by evaluating the evolution of the preferential orientation during annealing and correlating it with the other structural changes that are taking place.

REFERENCES

[1] Texture Development and Anisotropic Behaviour in a Ti-45Ni-5Cu (at.%) Shape Memory Alloy; Lie Zhao; PhD Thesis, University of Twente, Oct 1997.