 ROBL-CRG	<b>Experiment title:</b> <b>Simultaneous XRD and Electrical Resistivity Measurements of the phase transitions in Co-Ni-Ga ferromagnetic shape memory alloy system</b>	<b>Experiment number:</b> <b>20-02-693</b>
<b>Beamline:</b> BM 20	<b>Date of experiment:</b> From: 14-06-2010                      to: 19-06-2010	<b>Date of report:</b> 24.01.2011
<b>Shifts:</b> 12	<b>Local contact(s):</b> <b>Carsten Baehtz, Nicole Jeuter</b>	<i>Received at ROBL:</i>
<b>Names and affiliations of applicants (* indicates experimentalists):</b> <b>C.M. Craciunescu*, F.M. Braz Fernandes*, K. K. Mahesh*:</b> CENIMAT-Centro de Investigação de Materiais, Campus da FCT/UNL, 2829-516 Monte de Caparica, PORTUGAL <b>Carsten Baehtz *,Nicole Jeuter*:</b> FZD, P.O. Box 510119, 01314 Dresden, GERMANY		

## Report:

The experiments have been performed on bulk  $\text{Co}_2\text{NiGa}$  samples in three states: quenched, deformed and quenched, and quenched and stabilized at  $350^\circ\text{C}$ . The in situ X ray scans were performed along with ER measurements in the temperature range where the phase transformations occur. The tests have been done in the beryllium dome, with the electric resistance vs temperature (ER) measurements performed using a Keythley 2010 multimeter. The heating and cooling was performed between  $-100$  and  $+200^\circ\text{C}$ . The shape memory alloys were insulated using kapton foil from the heating device. The beam size used for the experiments and focused on the sample had to be limited due the contacts for the ER measurement device. Thus, corroborated with larger grain sizes for the investigated alloys, it is possible that only particular crystallographic orientations have been observed.

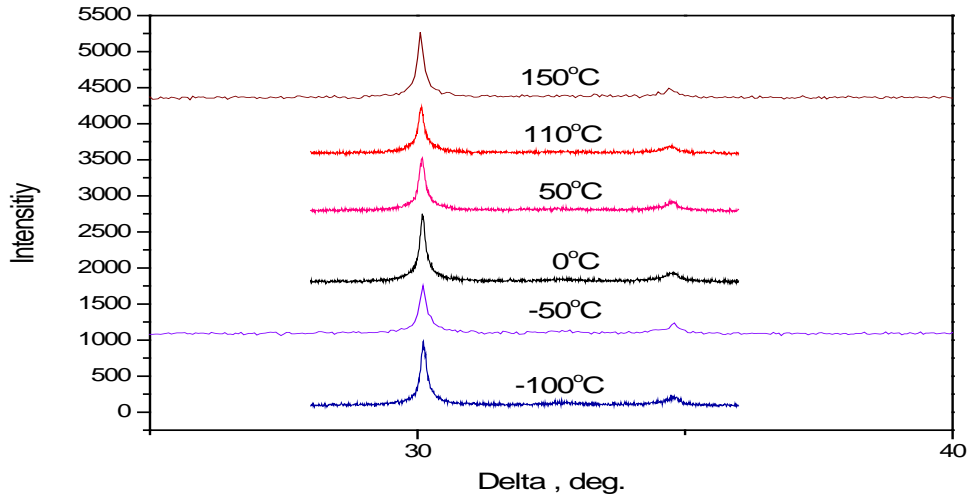
## EXPERIMENTAL

The results recorded for the quenched sample are shown in figure 1. Apparently, it is difficult to observe a phase transition from the spectra detailed in fig. 1a. This appears in contrast with the expected Martensite and Austenite peaks reflecting the martensitic phase transition on heating and on cooling. A further analysis of the (110)M peak intensity in figure 1b, based on ER measurements during heating and cooling indicates that the phase transition actually exists, but is difficult to distinguish since the (111) $\gamma$  and (110)M peak are coincident. Adjustments and tests of the ER equipment for thicker samples allowed the observation of the relative change of the electric resistance (as the average ER result for the stabilized temperature recorded during a XRD scan- fig.1 c) only on heating, but the result appears to be consistent with the one based on the analysis of the (111) $\gamma$  and (110)M peak height. There was no significant influence of deformation on the structure at RT and at  $200^\circ\text{C}$ , as it also results from fig. 2, but it was not possible to detect the phase transition in the materials, a result consistent with preliminary experiments.

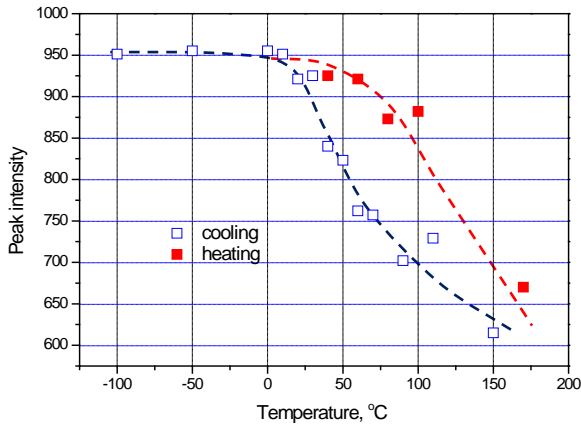
## Conclusions

The comparison with experimental results obtained using a Bruker XRD equipment and the results recorded at ESRF suggests that the large grains that resulted after quenching had orientations that did not allow us to observe the phase transition related to the A(110) peak. The restrictions related to the beam spot due to the presence of the contacts of the ER device only allowed us to focus on a limited

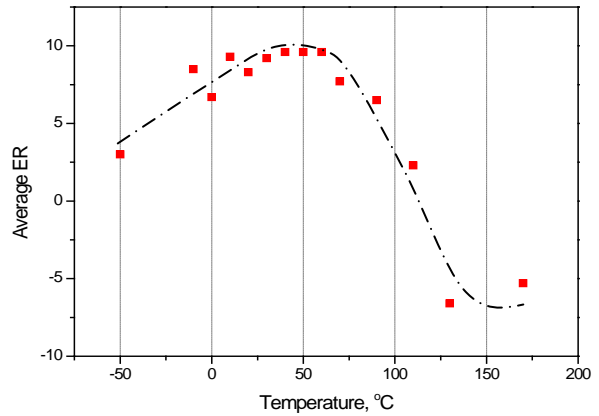
surface, most likely on grains with other orientation. For this reason it is possible that only the martensite peak M(110) superposed on the  $\gamma$  (111) could be used to observe the phase transition.



a. In situ XRD of the Co<sub>2</sub>NiGa in the quenched state

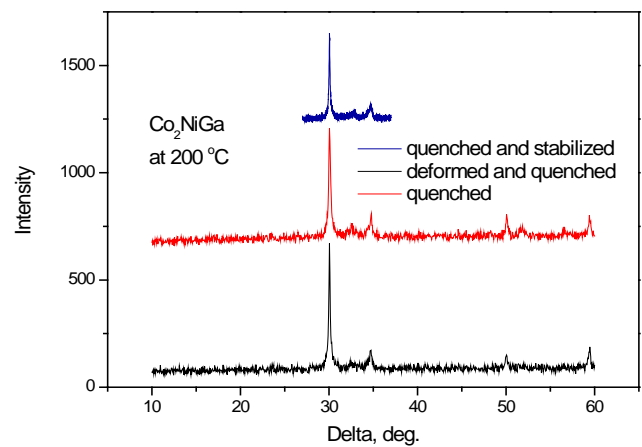
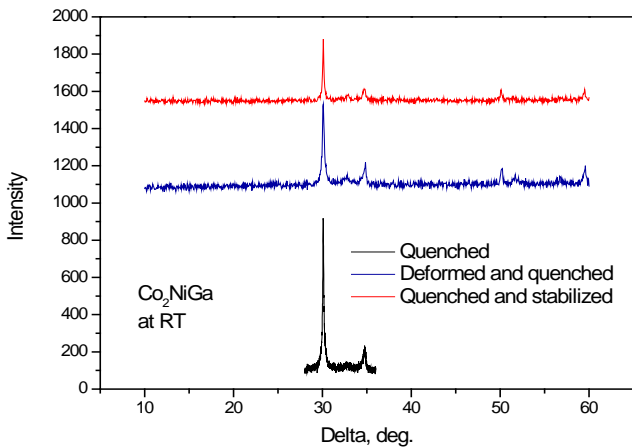


b. Max intensity of overlapping (110)M and (111)γ peak intensity



a. Average ER values vs temperature data

**Fig. 1** In situ XRD and ER results of CoNiGa samples in the quenched state



**Fig. 2** In situ XRD of Co<sub>2</sub>NiGa samples in the quenched, deformed and quenched and quenched and stabilized state