ESRF	**Experiment title:** **Combined in-situ XRD and Electrical Resistivity Study of the Phase Transformations in Ni-Ti SMA**	**Experiment number**: **MA- 895**
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REPORT:

The proposal for the experimental session was drafted with a view to approach a combination of characterization techniques, wherein, the acquisition of the X-ray diffraction (XRD) and the electrical resistivity (ER) measurement will be made during the thermal induced phase transformations in Ni-Ti shape memory alloys (SMAs).

Specimen were mounted on the 6-circle goniometer. ER measurement was carried out by employing HL5500 Hall system. Electrical probes are contacted with the specimen in Vander Pauw configuration as shown in Fig. 1. X-ray energy is set to 11.5keV.

Samples S32 (Ni-Ti thin film (~ 720 nm thick) on SiO_2/Si(100) from ME-1087, April, 2005) and S23 (Ni-Ti thin film (~ 800 nm thick) on SiO_2/Si(110) from ME-936, Feb, 2005) specimens were tested. During the periods of experiments with both the specimens, unexpected beam instabilities had come across together with alignment of the optics of the beamline. Hence, the experiments with both specimens remained inconclusive.

On the final day, when the beam was stable and optics was set, sample S40 (Ni-Ti thin film ~ 1450 nm thick, on MgO(110), from ME1255, Feb, 2006) was chosen for the study. The specimen was heated to 100ºC. XRD scanning were performed in the range $25º < 2\theta < 34º$ at selected temperatures to track the phase transformation until -99ºC. Again, while heating, diffractions were registered in the predetermined temperatures. Simultaneously, ER values were noted both while cooling and heating. In Fig. 2, XRD profiles obtained at selected temperatures while cooling and, ER profile both while cooling and heating are shown. From the XRD profiles, it can be observed that the diffractograms corresponding to the temperatures while cooling from 70 to 40 ºC possess a single peak at $2\theta = 29.2º$ attributed to B2(110). For the diffractogram obtained at 20 ºC, this peak splits into R(112), at $2\theta = 29.1º$, and R(300), at $2\theta = 29.4º$. Together with this, other peaks also appear corresponding to M-phase. As the cooling is continued below – 30 ºC, the diffractograms are found to possess the peaks corresponding to M-phase.

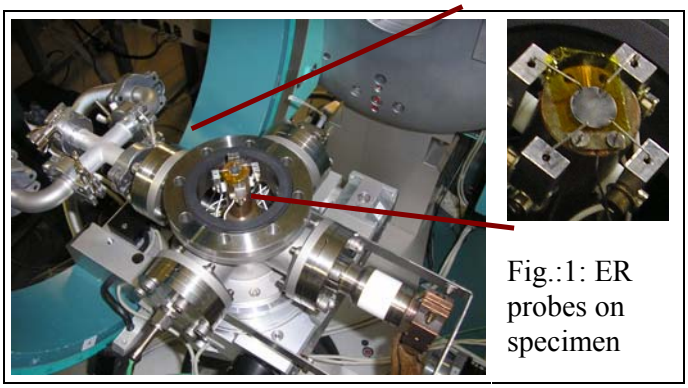

Fig.:1: ER probes on specimen

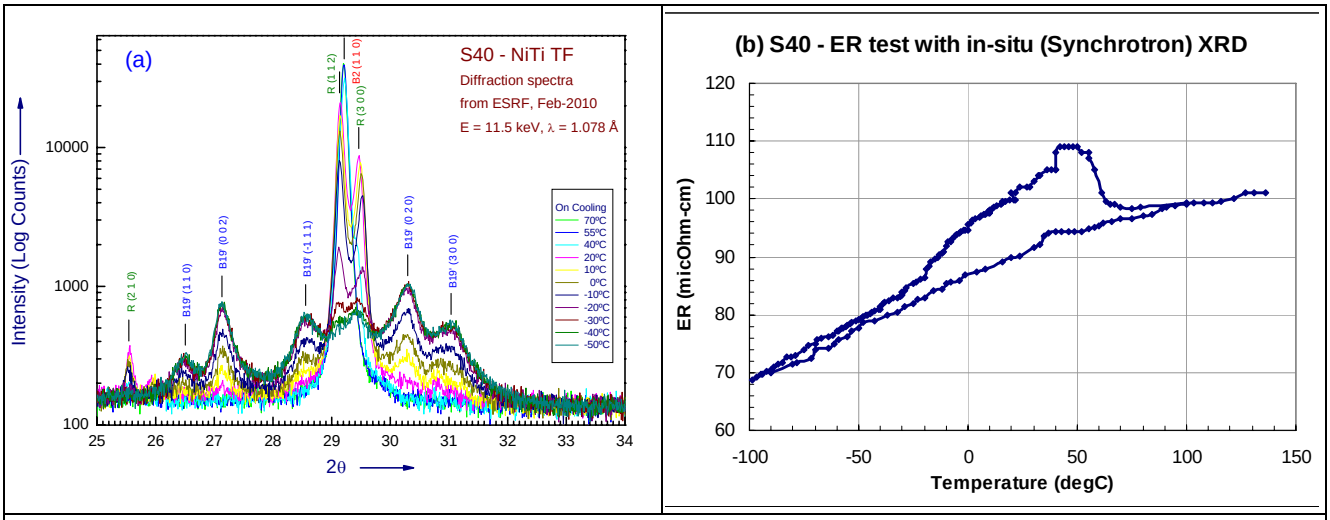

Fig. 2: (a) XRD profiles obtained at selected temperatures while cooling and, (b) ER profile both while cooling and heating

In the ER profile shown in Fig. 2(b), as the temperature is decreased, ER is observed to decrease followed by increase around 60 ºC associated with A→R phase transformation. On further cooling, the ER increases to have a plateau corresponding, followed by a decrease with cooling associated with R→M phase transformation. While heating, ER is increased with increase in temperature up to around 30 ºC followed by a sudden increase corresponding to M→A phase transformation.

In Fig. 3, main parameters, such as, (a) full width at half measure (FWHM) and (b) Integrated Area (IA), obtained from the diffractogram peaks corresponding to B2(110), R(112), R(300) and R(210) are presented. It is observed that the peaks corresponding to R-phase can be traced even at temperature of 40ºC. When the phase transformation nature in the ER profile is compared with the XRD profiles, it is clear that ER detects R-phase transformation well before, than by XRD, at around 60 ºC and R→M phase is observed around 40 ºC. Further, the presence of A-phase is not observed below 10ºC. From IA observation in Fig. 3(b), R-phase is found to be more prominent in the temperature range between 20 and – 10 ºC. In the ER curve, R-phase transformation is more prominent in the temperature range between 55 and 40 ºC.

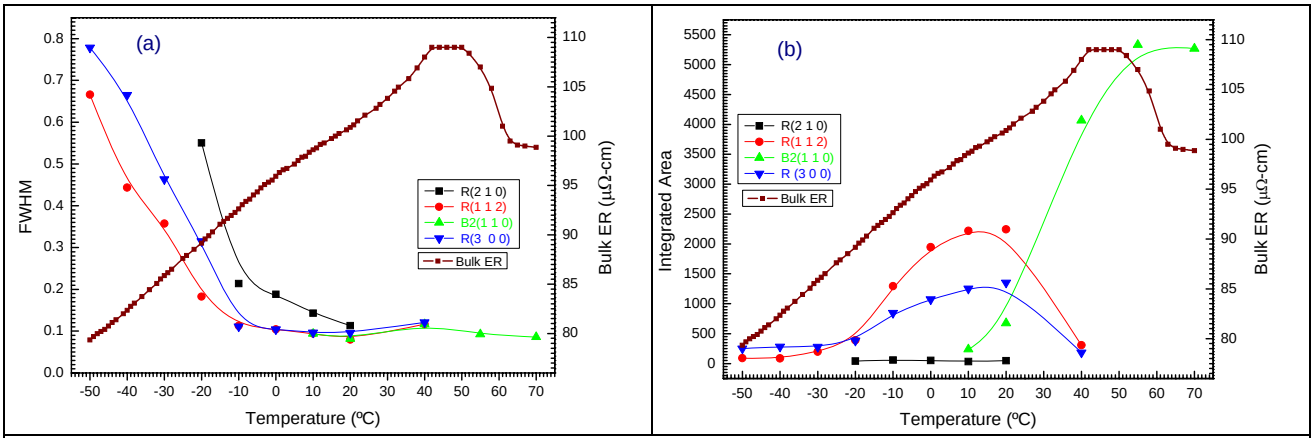

Fig. 3: (a) Full Width at Half Maximum (FWHM) and (b) Integrated Area values obtained from the diffractogram peaks corresponding to B2(110), R(112), R(300) and R(210) while cooling.

Conclusions

From the above observations, it can be concluded that ER technique is more sensitive to the early stages of the phase transformations. However, with the information from the XRD profiles, quantitative estimation of the presence of phases over a temperature range can be made. It is foreseen that the combined analyses of the data obtained would lead to quantitative input into the knowledge of the phase transformation behaviour in Ni-Ti shape memory alloys.