ESRF	Experiment title: "Anomalous Wide Angle X-ray Scattering of a Reversible Short Range Order in the metallic alloy Zr2(CuxNi1-x) x=0., 0.5, 1."	Experiment number: 02-02-773
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Shifts: 15	**Local contact(s)**: Jean-Paul SIMON	*Received at ESRF:*

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By Differential Scanning Calorimetry (DSC) a reversible enthalpy change of melt spun metallic ribbons is observed upon cycling between two temperature. This effect is seen after the initial relaxation by thermal annealing in the vicinity of the glass transition temperature. Ex-situ annealed samples were prepared in both the low temperature and high temperature states. These ribbons, about 0.020 mm thick, were measured in transmission in a symmetric configuration from $q=0.3nm^{-1}$ to 7.4 nm^{-1}. A bent graphite analyzer (~30 eV resolution) and a PM were employed to suppress the fluorescent scattering and part of the Compton scattering at the Ni and Cu edges.
The drift detector, which were planning to use, was unfortunately not available as planned. The following energies were used together with the f' and f":

E (eV)	f'_{Zr}	f''_{Zr}	f'_{Ni}	f''_{Ni}	f'_{Cu}	f''_{Cu}
8133	-0.33	2.20	-3.46	0.500	-2.111	0.578
8322	-0.36	2.12	-6.46	0.48	-2.34	0.554
8769	-0.44	1.932	-2.27	3.55	-3.41	0.504
8955	-0.47	1.864	-1.85	3.45	-5.41	0.486

The measurement time was determined to yield a precision better than 0.5%. For the first samples, Zr_2Cu and Zr_2Ni, no reversible change in the scattering profile at a given energy was found (although we have problems with reproducibility when the 2θ axis reached the vertical).
The problem was identified: the analyzer mounting was not sufficiently tight; finally during the last night, it lost the Bragg reflection at the beginning of each scan.

We have tried to analyze the data nonetheless by utilizing the Zr_2Cu as a standard, expecting that the analyzer did not become de-tuned for a given energy. This is a possibility since only the sample holder was moved between between the two scans. However, this also failed to give reliable results as illustrated below for Zr_4CuNi (x=0.5). The intensities curves are shown as a function of the modulus of the scattering vector (red – 8322eV, black– 8133eV, red 8322eV, green – 8955eV , blue – 8955eV, ordered top to bottom). Here the signal has been divided by $<f^2>$, and the thus normalized signal should oscillate around one at large q values. Since the anomalous variations should not exceed in relative some per cent, these measurements are unreliable.