	Experiment title: Local order in the model multi-component alloy Fe-Co-Ni-Cr	**Experiment number**: HC-2283
Beamline: ID01	**Date of experiment**: from: 12 May 2016 to: 16 May 2016	**Date of report**: 26 Aug. 2016
Shifts: 12+1	**Local contact(s)**: Peter Bösecke	*Received at ESRF:*

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Report:

The system Fe-Co-Ni-Cr is the basis of a series of multi-component alloys that exhibit promising mechanical properties (high strength, ductility, wear and corrosion resistance). To understand and deliberately modify these properties, detailed structural information is highly needed. Like for any non-binary system, x-ray scattering then requires to exploit anomalous scattering. No signs of long-range order were revealed from polycrystalline material [1]. Here, a single-crystalline solid solution with high local order was studied.

To explore reciprocal space like it is possible with a four-circle diffractometer, the standard set-up of ID01 had to be modified. Much time (> 2 days) was required to add a tilting table (chi motion) and a SmarAct stage (phi motion), hardware problems were faced with these two diffractometer components. An energy sensitive detector (Vortex-EM, energy resolution of about 300 eV FWHM below 10 keV) was used that allowed the simultaneous registration of the respective elastic line and all excited Kα and Kβ fluorescence lines, together with their escape peaks.

Together with the elastic scattering, resonant Raman scattering (RRS) is increasingly important close to an absorption edge. While RRS K-$L_{II}L_{III}$ shows up as a separate line and is easily separated from elastic scattering, RRS K-$M_{II}M_{III}$ was not discriminable. Here, RRS K-$M_{II}M_{III}$ was estimated from the intensity ratios Kβ/Kα of the fluorescence lines. We obtained Kβ/Kα ratios of about 0.15 for Fe, Co and Cr from the Ni-edge measurement, this value was also adopted for Ni.

By normalizing the scattering to the nearby RRS K-$L_{II}L_{III}$ line, changes in the incoming photon intensity, in the x-ray path due to a facility misalignment and to sample surface roughness were eliminated for any

diffractometer setting. Data were brought on an absolute scale (Laue units) by comparing them with the dominant thermal diffuse scattering (TDS) close to Bragg reflections. TDS was calculated up to third order using the elastic constants as determined by the pulse-echo-overlap method. Diffuse scattering at $h_1\ h_2\ 0$ positions was measured on a grid of 0.1 reciprocal lattice units at 8313 eV and 5969 eV, 20 eV below the K absorption edges of the Ni and Cr component (Fig. 1). Due to missing measuring time, scattering at the Co and Fe edges could not be taken.
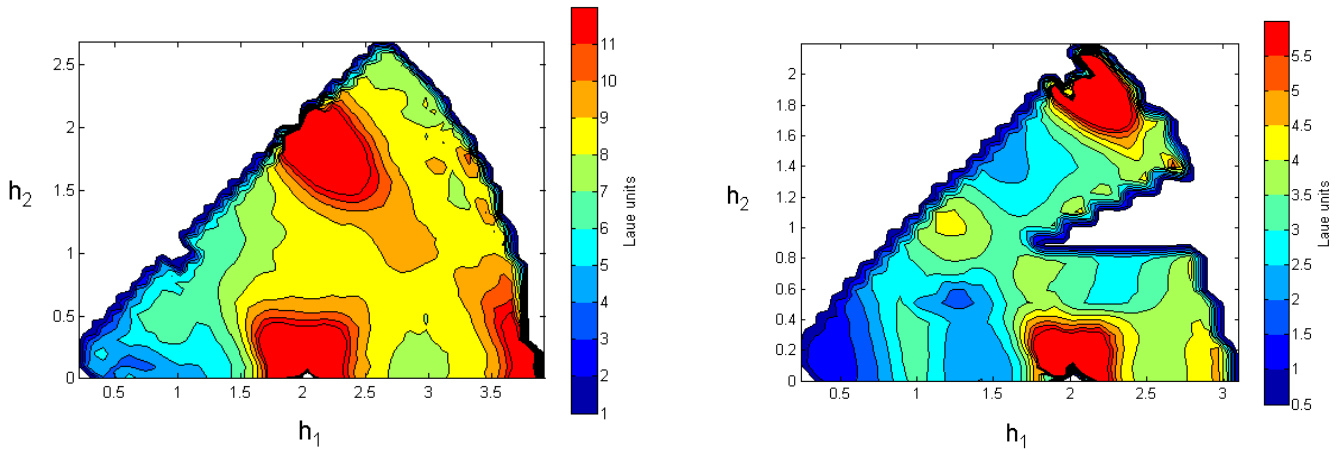

Fig. 1. Diffuse scattering (elastic and inelastic scattering) in Laue units from Fe-Co-Ni-Cr at $h_1\ h_2\ 0$ positions, at incoming photon energy of 8313 eV (left) and 5969 eV (right).

Fig. 1 commonly reveals a strong intensity increase towards the Bragg reflections. The diffuse scattering between them is flat at the Ni edge, but modulated at the Cr edge, thus indicating short-range order (SRO). The reason is understood in terms of the scattering contrast, the squared difference in the atomic scattering factors $|\Delta f(i\text{-}j)|^2$. The largest (but still low) value at 8313 eV is $|\Delta f(\text{Ni-Co})|^2 = 17.8$ e.u., but $|\Delta f(\text{Cr-Ni})|^2 = 70.5$ e.u. at 5969 eV. As $|\Delta f(\text{Ni-Cr})|^2 = 7.0$ e.u. at 5969 eV, the respective SRO modulations at 8313 eV will hardly be visible. Of course, there might still be modulations due to Cr-Fe SRO and/or Cr-Co SRO (with smaller scattering contrast than $|\Delta f(\text{Cr-Ni})|^2$).

In our alloy we observe 100 diffuse maxima with contour lines that resemble those of Cu-25 at.% Au. Such a pattern reflects a 3D atomic arrangement with diffuse antiphase boundaries [2]. Note that Ni-rich Ni-Cr gives SRO maxima at different positions, namely 1 ½ 0. Within a ternary Fe-rich Fe-Cr-Ni solid solution, however, Ni-Cr SRO shows maxima at 1 0 0 positions [3]. For Al-poor Al-Fe-Co-Ni-Cr, ab-initio electronic structure calculations give Ni-Cr SRO maxima at the 0 0 0 position [4]. To obtain a better insight into SRO (there are in total six pair SRO terms), at least scattering close to the Co and Fe edges still has to be done.

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