

ESRF	Experiment title: DISPLACEMENT FIELDS in an unmixed AuNi alloy by Anomalous Diffuse Scattering near Bragg peaks	Experiment number: 02 02 180 ESRF ref 2665
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Structural hardening occurs at low temperatures, far from equilibrium; it is triggered both by the thermodynamics of the system and by elastic effects. If the consequence of the first contribution has been an active domain of research and is already fairly well understood, the elastic effects, with their long range fields, are more tricky to handle although they can lead to a wide variety of patterns. The O.N.E.R.A. (Paris) is nowadays dealing this problem by a field theory and, experimentally, O Lyon has settled a new measurement procedure by recording the diffuse scattering in the vicinity of Bragg's peaks (called in the following to shorten as Huang scattering): Taking the antisymmetric part of this signal (i.e. I(q+dq)-I(q-dq)), cancels the chemical contribution of the composition fluctuations (which are periodic in the reciprocal space) and any fluorescence (isotropic). Moreover up to the third order, its limited development is the Fourier transform of the displacement field. The system Au Ni was chosen since the large difference in atomic volumes (35%) between species may be the reason of an unusual behaviour during phase separation, such the locking of growth for nanometric sizes. The aim of this experiments is to precise the involved mechanisms.

It concerns an Au 30at%Ni alloy which was grown by the Chokravski method for crystallography a single grain. If it was only obtained a basaltic polycristal, grains are large enough for the measurement and the mosaicity has been swept by a thermal treatment of one week 50K below the melting temperature. 0.3mm thick slices were cut by a diamond saw, polished down to 1micron roughness, then rehomogeneised in a salt bath (at 1073K, the lowest temperature in the single phase region of the phase diagramm in order to minimize the reorganisation during the quench in water). Since there is no Small Angle Scattering signal, the evolution of the structure has been

followed by measuring the scattered intensity near the 200 Bragg peak. This diffraction spot was selected because the diffuse scattering was foreseen to occur in {200} reciprocal planes, extrapolated from results in thin layers (scattering results as monolayered clusters of Ni separated by 12 planes from Abadias, DRF-CENGrenoble).

The scattering in the [001] plane near the 200* Bragg peak was measured in the asquench state, after 1h and 12 hours at 423K; as shown on figure 1 the scattering increases by almost two order of magnitude, indication that the quench was efficient and that aging temperature was adequate for the atomic diffusion. We choose to probe the neighborhood (four energies) of the Au L_{III} edge to increase the X ray penetration (12µm instead of 8µm near the Cu edge), to have more flux and less parasitic scattering, finally to increase the anomalous variations (f'Au between -12 and -18 electrons). Figure 2 shows a typical 2d reconstruction of a 002 slice of the reciprocal space, obtained from about 50 images, rocking omega near the 200 Bragg peak. The scattering on the upper part of the figure is much stronger than below and decreases as soon as 0.05 A⁻¹: this the inter-cluster distancefound by Abadias on thin layers. results disagrees with Moreover, there is no anomalous variation while its relative variation would have been of about 15% if the scattering has varied as $\langle f^2 \rangle$ and larger than 30% for $(f_{Au}-F_{Ni})^2$. Finally we recorded several cuts around 200 Bragg peak, changing the zone axis from 002* to 022*: 2d- images then present two wings on the upper part and none on the bottom, a pattern which is not vet explained; a complete 3d-cartography of the scattering near the 200 and probably the 220 Bragg peaks and extending in a larger q range seems necessary for the interpretation and is foreseen with a large net mesh during in-house periods.

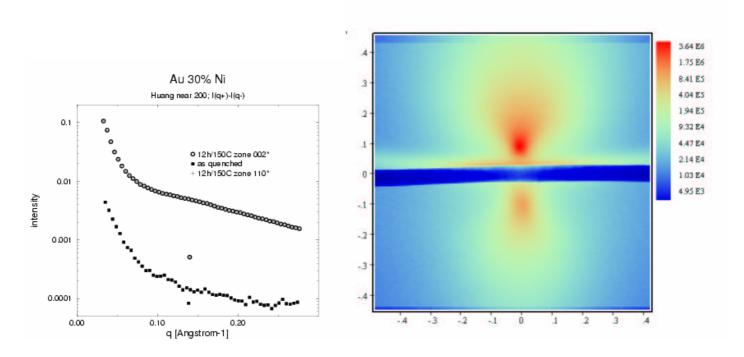


Fig. 1: Kinetics of the Huang scattering

Fig. 2: typical 2d reconstruction: in the {002} plane, the 200 direction is vertical and the Bragg spot is masked by the beam stop.