ESRF	**Experiment title:** Order and static displacements in Pt-Rh solid solutions	**Experiment number**: HS-935
Beamline: ID01	**Date of experiment**: from: 7 June 2000 to: 14 June 2000	**Date of report**: 4 Mai 2001
Shifts: 18+3	**Local contact(s)**: A. Mazuelas, H. Metzger	*Received at ESRF:*

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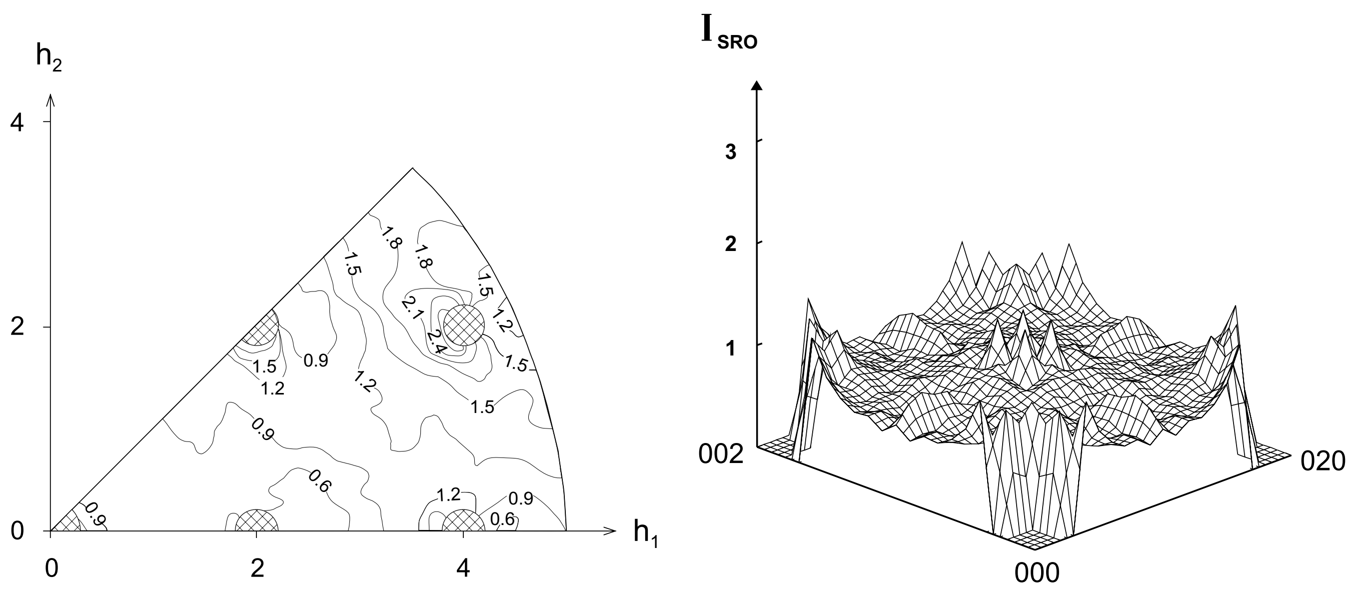
Report:

A central question on bulk property of Pt-Rh, whether the system orders or decomposes, is not yet definitely solved as kinetics are slow and interactions are weak. Theory predicts (i) disordering in following global phenomenological trends of alloys where both elements are late transition metals [1] or (ii) ordered structures on the basis of electronic structure calculations (superstructures $D1_a$, $D0_{22}$, X2 and '40') [2]. This unresolved problem prompted this investigation.

A single crystal was grown by zone melting from Pt-50 at.% Rh, and a slice with a ⟨421⟩ surface normal was cut. Its composition as determined by X-ray fluorescence was Pt-47.0(3) at.% Rh. The sample was aged for 36 d at 650°C and quenched into brine.

The diffuse scattering was measured with X-rays of an energy of 23.201 keV, 18 eV below the K absorption edge of Rh. Thus, the scattering contrast $2|f_{Rh}-f_{Pt}|/|f_{Rh}+f_{Pt}|$ (its value squared weights the short-range order scattering contribution) is increased by 22% over a laboratory facility employing Mo K_α radiation. The resonant Raman scattering $K\text{-}M_{II,III}$, shifted by 461.4eV and 521.3eV, was resolved using a Röntec detector (FWHM=300 eV at 23 keV). Its detective quantum efficiency was very low, amounting to only 2% with respect to a Cyberstar detector (FWHM=6.9 keV at 23 keV) that typically yielded 5000 to 10000 events per 20 s. For this reason, data had to be taken by the Cyberstar detector and the resonant Raman scattering contribution had to be subtracted numerically.

About 4600 positions were registered on a grid of 0.1 reciprocal lattice units (r.l.u.) for scattering vectors from 0.3 to 5 r.l.u. Data were corrected for background and put on an absolute scale by comparison with the scattering from C_8H_8 at $\sin\theta/\lambda=0.5$. Thermal diffuse scattering was calculated up to third order on the basis of the elastic constants obtained from ultrasonic measurements. The diffuse scattering of Fig. 1a shows two clear features. First, no maximum between the Bragg reflections is seen but a slight increase towards the direct beam. This might indicate short-range decomposition although data get more uncertain as background increases at small angles. Second, there is a large asymmetry around the Bragg reflections because of size effect scattering. This is surprising as Pt and Rh have a lattice parameter misfit of only 3%; also, no atomic relaxation was explicitly considered in the electronic-structure calculations [2]. As the larger atom (Pt) has the larger atomic scattering factor, larger scattering at smaller scattering angles around a Bragg reflection is expected, as seen in Fig. 1a.



(a) **(b)**

Fig 1. Pt-47 at.% Rh aged at 650°C. (a) Elastic and inelastic diffuse scattering $\mathbf{I}_{dif}$ in e.u. within the h_1h_20 plane. (b) Separated short-range order scattering $\mathbf{I}_{SRO}$ in Laue units within the 100 plane.

Short-range order scattering separated according to the Georgopoulos-Cohen method [3] is shown in Fig. 1b. No clear evidence for decomposition is seen as data close to Bragg reflections have to be discarded because of increased thermal diffuse scattering. Small-angle scattering data from a 25 µm thick single-crystalline foil taken in addition, were too uncertain (because of problems with the 2D detector) to combine them with the wide-angle scattering data. The slight ridge along 1h0 (Fig. 1b) is not considered significant for a firm conclusion either. Laboratory measurements are now under way to provide more diffuse scattering data as the number of data points within this experiment was at the lower limit (typically measurements at about 10000 positions are required). A combination of both data sets will stabilize the separation of short-range order scattering.

[1] A. Bieber and F. Gautier, Acta Metall. **34**, 2291 (1986).
[2] Z.W. Lu, B.M. Klein and A. Zunger, J. Phase Equilibria **16**, 36 (1995).
[3] P. Georgopoulos and J.B. Cohen, J. Physique **38**, C7-191 (1977).