



	Experiment title: Diffusion studies in crystalline materials with x-ray fluctuation spectroscopy	Experiment number: HS-1275
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Report:

With coherent x-rays of third generation synchrotron radiation sources it became possible to study the dynamics in metallic systems with a new technique: x-ray photon correlation spectroscopy (XPCS) [1].

In this experiment we investigated the fluctuation of intensity of the speckle pattern taken in the (100)-superstructure Bragg reflection of Co₆₀Ga₄₀ single crystalline B2-ordered sample. Time correlation of the intensity-fluctuations yielded relaxation times representative for the dynamics in the sample. The temperature dependence of the relaxation times obeyed the Arrhenius law. We explain the fluctuation of speckle intensities by the motion of antiphase boundaries in the sample. An investigation of the Laue-diffuse scattering from Co-Ga and from Fe-Al single crystalline samples failed due to much too low intensity of the scattered radiation.

A coherent beam has been obtained by the transmission of photons from a monochromator through an $8\mu\text{m}$ diameter pinhole close to the sample. A CCD camera (direct illumination chip 1242×1152 pixels; pixel size $22 \times 22 \mu\text{m}^2$) was mounted 2.3 m away from the probe, so that the speckle of about $30 \mu\text{m}$ size was slightly larger than one pixel. The sample was measured at different temperatures between RT and 1000K in a vacuum furnace with a Kapton window mounted on a goniometer head. Series of 1000 up to 3000 exposures/frames of 300×300 pixels were taken. The exposure time was usually 1s. An autocorrelation function was computed for each pixel in the peak: $\rho(k) = (N G^{(2)}(k) - M_0 M_k) / M_0^2$, where N is the number of frames (time steps), $G^{(2)}(k) = \sum_{i>k}^N n_i n_{i-k}$, $M_0 = \sum_{i=1}^N n_i$ and $M_k = \sum_{i>k}^N n_{i-k}$ with n_i the intensity at i -th time step. ρ is an estimator which gives proper results when the measurement time of the signal is only a few times longer than its characteristic relaxation time. Intensities n_i have been normalized by dividing the intensity of the specific pixel in the i -th frame by the integrated intensity of all pixels in this frame. Finally the mean $\rho(\tau)$ has been calculated averaging over all pixels in the frame, see Fig. 1. Relaxation times of the dynamical processes in the sample were fitted with an exponential decay function as shown in Fig. 2. Due to the exponential temperature dependence of relaxation times (Fig. 3) an activation energy Q of about $0.6(1) \text{ eV}$ has been estimated. This

value is rather small compared to the activation energy 2.5 eV for self-diffusion of Co- or Ga-atoms in Co-Ga alloys [2].

We explain this Q value as an activation energy for the motion of antiphase boundaries (APB) in thermodynamic equilibrium. The small Q value can be understood by the vacancy path being restricted to disordered regions. Consequently the vacancy is bound to the APB's favoring their motion. This result is in agreement with the results of Monte Carlo simulations [3]. This result is very promising and opens new possibilities for the studies of APB motion and of coarsening processes at low temperatures, investigations inaccessible to conventional methods (TEM, X-ray or neutron diffraction) [4].

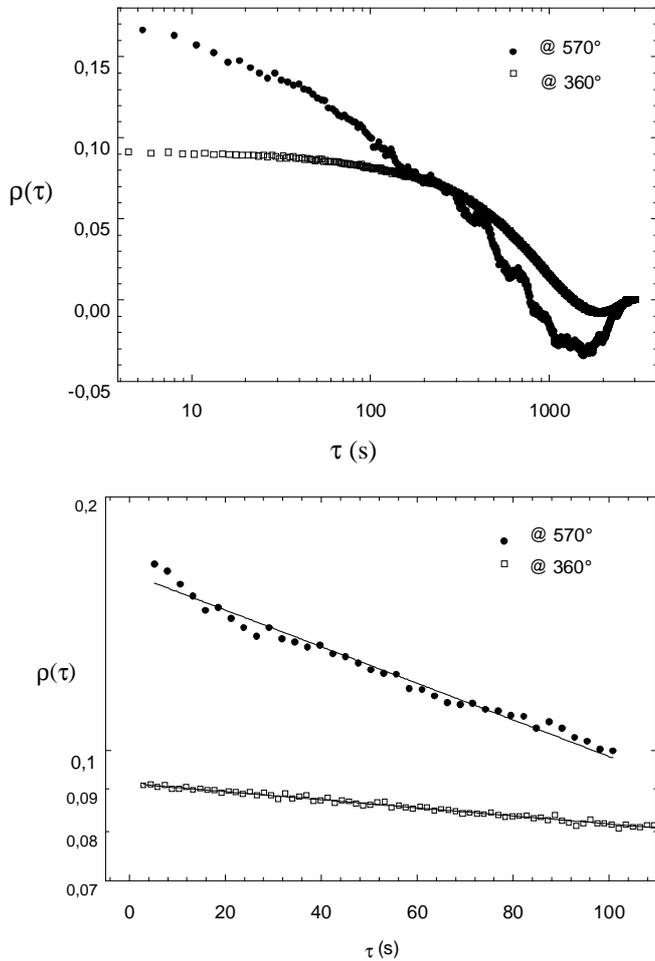


Fig. 2: Autocorrelation functions at the same temperatures as in Fig. 1, but with linear scale for time and logarithmic scale for the autocorrelation function. Fitting exponentials result in lines with characteristic decay times of 400s (circles) and 1800s (squares).

Fig. 1. Autocorrelation functions vs time at 570°C (circles) and at 360°C (squares). Time scale is logarithmic, autocorrelation function linear.

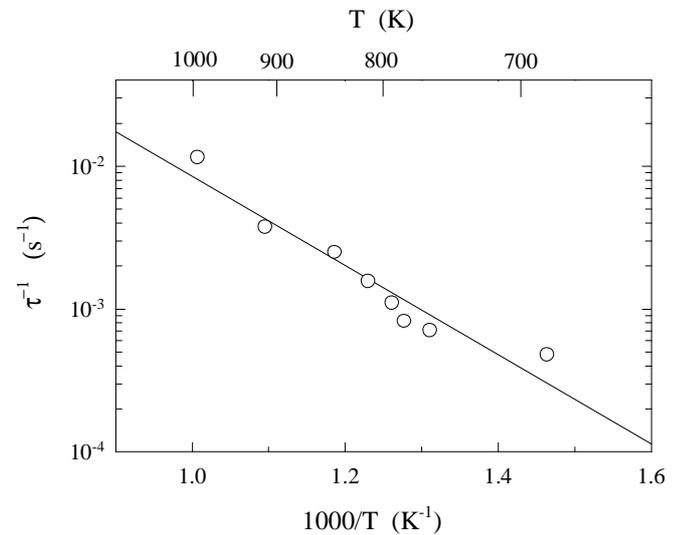


Fig. 3. Arrhenius plot of relaxation times vs reciprocal temperature.

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