<table>
<tr><td rowspan="1">ESRF</td><td colspan="1">Experiment title: Grain boundary migration in bicrystals of an Fe – Si alloy observed by in situ X-ray topography</td><td>Experiment number:
HS-1477</td></tr>
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Report:

Boundaries between grains (GB) in polycrystalline materials can migrate, if the displacement reduces their area and hence the total energy. The most applied technique of studying this process is repeated annealing at high temperatures followed by inspecting the position of the GB on the specimen surface [1]. In the present experiment the GB migration was observed in situ by SR transmission topography. This technique eliminates the uncertain effect of repeated heating and cooling of the specimens and the number of observations at a given temperature is much larger. The interaction of different kinds of crystal defects visible in the topographs with moving GB can be recorded.

The specimens were cut of Σ5 bicrystals described by mutual rotation of grains 36.9° around [001] direction. The GB was symmetrical, parallel to {310} planes in both grains, or asymmetrical, parallel to (710)/(110) planes. The specimens were plates parallel to (001) plane, 1 mm thick and 4 mm wide. For the orientation of the symmetrical bicrystal see Fig. 1a. The specimens were annealed in a high temperature stage developed at Ecole des Mines, Nancy, at temperatures in the range of 915 to 1090°C, in vacuum. The transmission topographs were taken using monochromatic beam (λ = 0.0207 nm). For the symmetrical GB the reflection was 420, ϑ = 9.44° in both grains, for the asymmetrical one 400, ϑ = 8.32° or 330, ϑ = 8.83°. The topographs were recorded by the FReLoN camera placed at the distance of 25 cm from the specimen. The lower resolution of topographs taken at high temperatures is caused by lower intensity of diffraction peaks and higher diffusion scattering.

The results of the experiment are still under evaluation, but some interesting features have been already obtained. At temperatures above 900°C the GBs move as to reduce their area characterized by their length. At a constant temperature the length of the GB decreases exponentially with time as it was found also in previous experiments [1]. At higher temperatures the GBs move faster. An example of GB migration is shown in Fig. 1. In Fig.1a (500°C) a subgrain boundary (the white band) and 90° magnetic domain walls parallel to the plane $(110)_B$ are visible in the B grain. After fast increase (15mins) the temperature was kept constant at 965° ± 2°C. The GB moves fast at the beginning, later its speed decreases. After 76 min of annealing the shape of the GB is changed substantially (Fig.1b), the upper and lower segments being slightly different. The upper end is nearly straight, parallel to the $[110]_A$ direction. Similar effects were observed also in other specimens, especially on asymmetrical GBs. This finding is compatible with the measurements of impurities segregation at GBs: low amount of segregation was detected at GBs parallel to low-energy low-index crystallographic planes [2]. After 303 min at 965°C the annealing was stopped.

When the specimen was heated slowly, the GB did not move till a temperature higher than 1000°C was reached. This is probably due to the pinning the GB by segregated impurities. This can be also the reason of occasionaly observed not smooth movement of the GBs: after staying for some time at one position they jump to a new one.

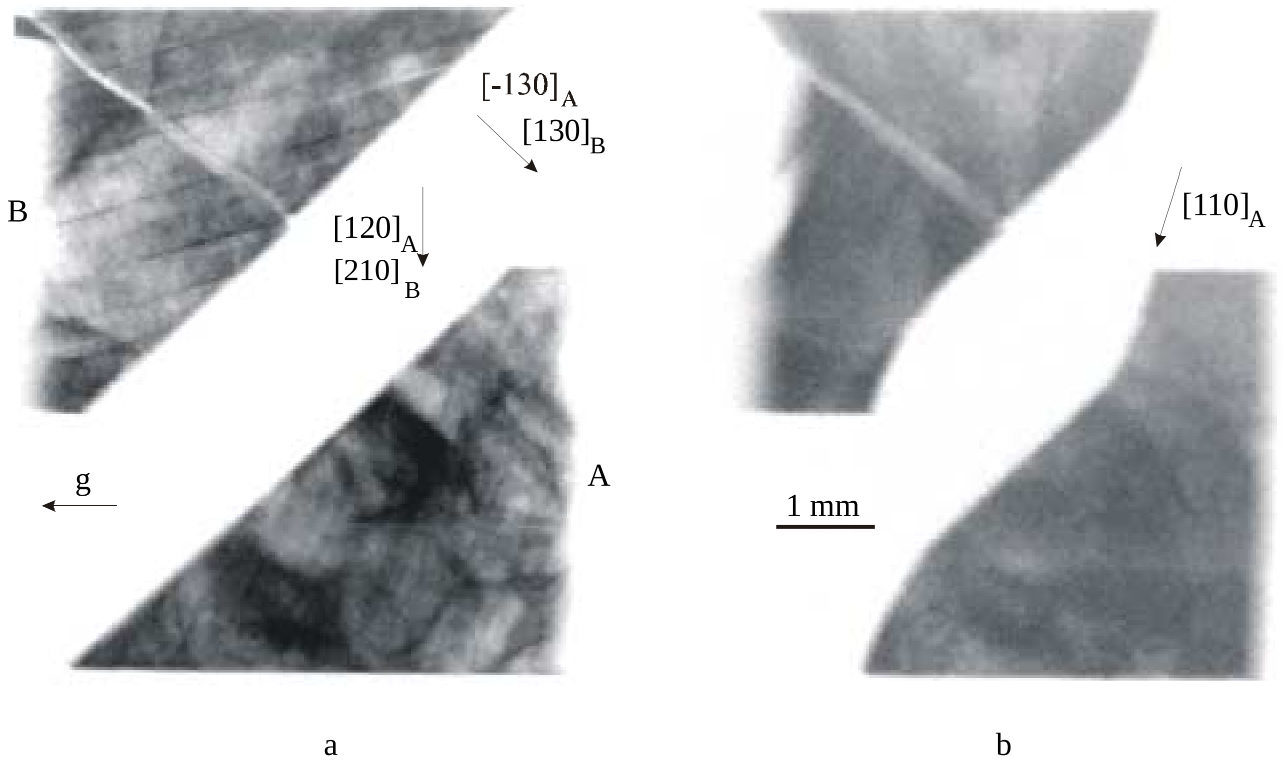

Fig.1 *In situ* topographs of a Σ5 bicrystal before annealing (a) and after 75 min at 965°C (b).

[1] P. Lejcek, V. Pajdar, J. Adamek and S. Kadeckova, Interface Science **1** (1993), 187.
[2] P. Lejcek, V. Paidar and S. Hofmann, Mater. Sci. Forum **294-296** (1999), 103.