ESRF	**Experiment title: In situ topography of Grain Boundary migration in bicrystals of Fe-Si alloys – study of the dependence on GB parameters.**	**Experiment number**: HS-2250
Beamline: ID19	**Date of experiment**: from: 19-nov-03 to: 24-nov-03	**Date of report**: 23-aug-04
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Report:

Grain Boundary (GB) migration was studied using Synchrotron Radiation (SR) transmission toporaphy *in situ*. This technique eliminates the uncertain effect of repeated heating and cooling of the mostly used experimental method. High intensity of SR enables short exposure times and thus to follow the dynamical behaviour of various crystal defects during *in situ* experiments. Interaction of moving GB with different kinds of crystal defects visible in the topographs can be recorded.

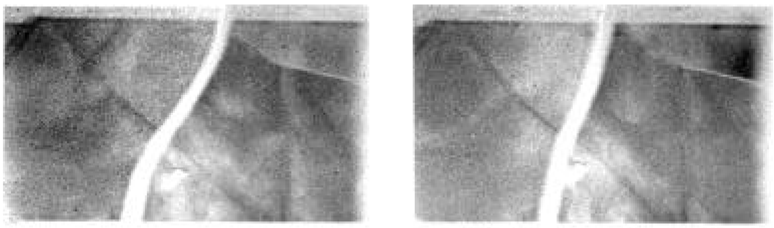
Two bicrystals of an Fe–2.8 wt %Si alloy having the diameter of 13 mm and the length of 50 mm were grown in the Institute of Physics, Prague, by the floating zone technique. The mutual rotation of the two grains was 45° around the [001] bicrystal axis in both bicrystals, but they differed in the GB orientation. It was either asymmetrical, parallel to the (100) crystallographic plane in one grain and to the (110) in the other one; or it was symmetrical {*hk*0}parallel to the plane bisecting the angle between {100} and {110} in both grains.

Specimens were plates of dimensions 12×4×1 mm^3. The GB was perpendicular to the largest surface, the angle between the GB and side surface being 45° before start of the experiment. A special evacuated stage (pressure of the order of 10^{-3} Pa) developed at Ecole des Mines, Nancy, that enabled us to heat the specimen to a high temperature, was installed at the beam line ID19. The transmission topographs were taken using a monochromatic beam (λ = 0.02 nm) and recorded by the FReLoN camera. The displacement *a* of the end of the GB from the starting position and length *L* of the GB were measured separately for the upper and lower half of the specimen (Figure 3). For more details concerning the experimental arrangement see [1]. Three specimens of each bicrystal were annealed at temperatures between 1223 K and 1323 K. Examples of the topographs taken during annealing at 1293 K are shown in Figures 1 and 2.

Topographs of the *asymmetrical* GB give evidence of interaction between the moving GB and Small Angle Grain Boundaries (SGBs). The SGB in the upper part of the right grain (figure 1(*a*)) does not affect the migration. It elongates and later grows out of the specimen. On the contrary the SGB visible in both grains and meeting the GB near the centre of the specimen (figure 1(b)) keeps locally the GB in its position. The upper and lower branch of the

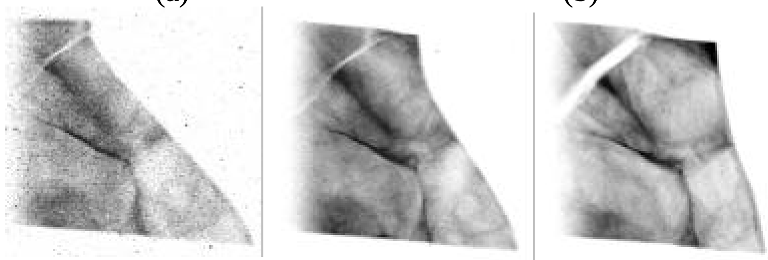
GB moves, changes its shape and orientation, but the junction does not move. End segments of the GB keep a constant orientation, which (supposing that the GB is perpendicular to the specimen surface) is parallel to the plane $(5\bar{1}0)$ in grain A and $(2\bar{3}0)$ in grain B. Such planar facets were observed also in other specimens.
At the beginning of the migration of the *symmetrical* GB, the motion of the upper branch is affected by a SGB visible near that edge of the specimen (figure 2(*a*)). Only a short segment of the GB migrates, the shape of the GB differs from that near the lower edge. Later the SGB grows out from the specimen, the shape of the GB changes (figure 2(*b*)), but the upper displacement remains larger than the lower one. After some time the migration is affected by a SGB meeting the GB near the centre of the specimen (figure 2(*c*)). The junction remains immobile, while both ends of the GB continue to migrate. A slight difference between the two ends can be seen even after the experiment was stopped. The upper branch of the GB is nearly straight, the lower one is curved. The straight sections of the GB are parallel to the planes $(5\bar{1}0)_A$ and $(2\bar{3}0)_B$ (with accuracy ±2°).

As it is apparent from figure3, corresponding values measured at the upper and lower branches of the *asymmetrical* GB are close each other in the limits of the measurement accuracy. Migration behaviour of the *symmetrical* GB is different. The displacement is larger and the migration faster in the upper part of the specimen than in the lower one (figures 4(*b*) and 5(*b*)). A similar effect is observed in all specimens of this bicrystal. This finding might seem surprising because an opposite behaviour could be expected at first sight: One could expect migration of both branches of the symmetrical GB of the same character while different for an asymmetrical interface. Our results indicate that orientation differences do not play important role in GB migration. This is probably the result of continuously changed orientation of the GB in its curved part that disables us to study fine orientation details. Further experiments as well as model calculations are necessary to explain fully the effect of orientation on GB migration.



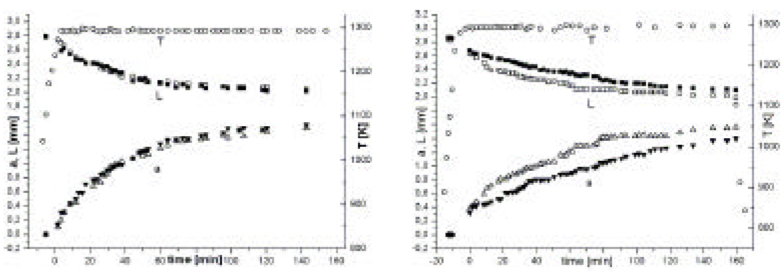
(a) (b)

Figure 1. Transmission topographs of the asymmetrical bicrystal, T = 1293 K. Time t is measured from the start of migration. (a) t = 44 min (left grain), t = 48 min (right grain), (b) t = 96 min (right grain), t = 98 min (left grain).



(a) (b) (c)

Figure 2. Transmission topographs of the symmetrical bicrystal, T =1293K. Time t is measured from the start of migration. (*a*) t = 11 min, (*b*) t = 56 min, (c) t = 168 min, T = 1103K.



(a) (b)

Figure 3. Dependence of the GB displacement a, the GB length L and the temperature T on the annealing time. ▼… displacement of the lower end of the GB, △…displacement of the upper end of the GB, ■…length of the lower half of the GB, □…length of the upper half of the GB, ○…temperature. (a) asymmetrical GB, (*b*) symmetrical GB

[1] Polcarová M, Brádler J, Jacques A, Lejcek P and Ferry O 2003 *J. Phys. D: Appl. Phys.* **36** A98