

ESRF	Experiment title: Dislocation transmission through grain boundaries in Fe 4% Si observed by in situ X-Ray Topography	Experiment number: HS-565
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Shifts: 1	Local contact(s): J. Baruchel, E. Pernot	<i>Received at</i> <i>ESRF:</i> 28 AOUT 1998

, Names and affiliations of applicants (* indicates experimentalists):

M. POLCAROVA	Institute of Physics, Czech Academy of Sciences,
J. BRADLER	Na Slovance 2, 18040 PRAHA 8
A. GEORGE	Laboratoire de Physique des Materiaux
A. JACQUES	(CNRS URA n°155)
F. VALLINO	Ecole des Mines
J.P. FEIEREISEN	Parc de Saurupt
O.FERRY	F-54042 Nancy Cedex (France)
M. LEGROS	

Report:

The experiment was designed to investigate the transfer of deformation by slip across coincidence grain boundaries in Fe-Si bicrystals. In Fe-4 % Si, plastic deformation is achieved by slip bands (groups of like dislocations moving in a sheet of parallel slip planes) to which grain boundaries are obstacles of various strengths, depending on crystallographic characteristics.

Slip bands are conveniently observed by X-ray topography in reflection setting.

Bicrystalline specimens, more precisely samples containing a $\Sigma = 3$ GB (70.54° rotation around a [110] axis, $(\bar{1}1\bar{2})_A$ grain boundary plane)

Samples with a $\Sigma = 3$ GB (38.9° rotation, [110] axis, $(1\bar{1}4)_A$ grain boundary plane)

Samples with a $\Sigma = 15$ GB (48.19° rotation, [201] axis, $(11\bar{2})_A$ grain boundary plane) were tested. They were strained in compression at room temperature using the mechanical testing stage built in Nancy.

In situ observations were done using symmetric reflections on the specimen surface ($20\bar{1}_A/201_B$ for $\Sigma = 3$, 110 for $\Sigma = 9$, 201 for $\Sigma = 15$), with a monochromatic beam. Due to the mosaic structure of our samples (subgrain, ~1 mm in size, misoriented by a few tenths of a degree), a 1.5 degrees scan was necessary to record the full image of the samples, which considerably increased the times of exposure (up to 2 mn in most cases). Monochromatic beam topographs, however, have a much better contrast than white beam topographs.

The effect of the film position (fixed or rotating with the specimen during the scan) on the contrast of the images was investigated. In the first case, the contrast depends only on the surface deformation of the specimen. In the second one, it depends on the deformation and the rotation of the surface. (Images taken in white beam conditions with a fixed specimen are only sensitive to surface rotation). The three methods can then give complementary insights of the strain state of the specimen.

The specimen were loaded at a constant rate to the stress at which the formation of the first slip bands was expected. The loading rods were then kept immobile, to allow the recording of an image. If necessary, the load was then increased by steps, until slip bands were observed and further to maintain them in motion. Proper loading conditions were difficult to adjust since slip bands are usually hardly detectable on the monitor screen and the critical stress was seen to vary from one specimen other.

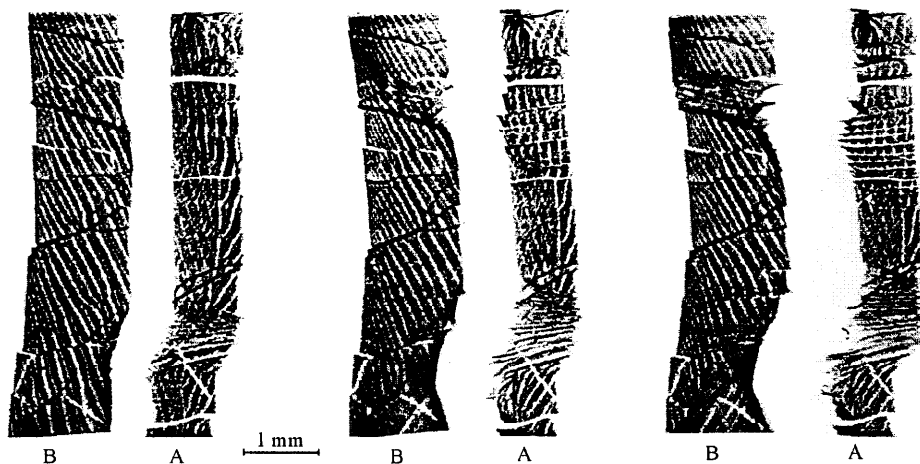
The results of the experiments are still under evaluation, but some interesting features could be obtained :

- probably thanks to better control of stress conditions, we were able to obtain well-defined isolated slip bands, whilst earlier experiments at ESRF had shown a systematic grouping of slip bands which made observation difficult and strongly affected the condition for slip transfer

- it is confirmed that GBs act as strong obstacles to slip bands, even in the so-called “easy case” of $\Sigma = 3$ bicrystal. In such bicrystals, however, slip transfer over the GB was observed frequently. In situ observation sequences showed that slip bands first stopped at the GB, creating pile-up stresses which are revealed by steps or cusps on the topographs. Large steps give evidence of a great number of accumulated dislocations and such steps were seen where the slip bands ended without continuation of slip behind the GB. At the spots where the slip was transferred over the GB, step sizes were seen to diminish, which is taken as a good evidence that part of the dislocations constituting the meaning slip bands has crossed the GB. Figure 1 gives an example of deformed $\Sigma = 3$ bicrystal, with large steps at the GB at the head of blocked slip bands and small steps at crossing slip bands.

- In $\Sigma = 15$ bicrystals for which dislocation transfer over the GB is very unlikely since available slip planes do no meet at the GB and expected Burgers vectors are different, it was possible to see transfer of slip, made evident by the continuity of slip bands across the GB. However, unlike the $\Sigma = 3$ bicrystals, the dimensions of the steps did not diminish but even grew up after the slip transfer. This is explained by the activation of dislocation sources in the opposite grain by the pile-up stress of the incoming slip band, without true dislocation transfer.

- The difficulty of transmission, even in the “easy” case was confirmed by in situ TEM experiments done on tensile bicrystalline specimen ($\Sigma = 3$ GB). Further experiments are planned, in coordination with the ESRF project.



Slip bands development during compression deformation. *In situ* white beam SR topography. Specimen: bicrystal Sig 15, grain boundary parallel to $\{112\}$ plane, deformation axis $\langle 125 \rangle$.