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MS-13-P-1420 Identification of twin types in twinned natural quartz crystal by FEGSEM and EBSD analysis

Samardžija Z.[1], Lenart A.[1]

[1]Jožef Stefan Institute, Ljubljana, Slovenia

Email of the presenting author: zoran.samardzija@ijs.si

Representative sample of the ‘‘v”-shaped twinned quartz crystals from geological location Madan ore, Bulgaria was investigated using a field-emission gun scanning electron microscope (FEGSEM) JEOL JSM-7600F and an electron backscatter diffraction (EBSD) analytical system Oxford Instruments HKL Channel 5 with Nordlys detector. For optimum surface quality quartz sample was finally polished with 0.05-µm colloidal silica on diamond lapping films and coated with a 1.5-nm amorphous carbon layer using precise ion-beam sputtering technique in a Gatan PECS 682 apparatus. This very thin carbon layer was sufficient to prevent charging in the SEM and was adequate also for the acquisition of unblurred, good-quality EBSD (Kikuchi) patterns. SEM operating conditions for the EBSD analyses were set to a voltage of 20 kV, a 10 nA beam current, a working distance of 20 mm and with a specimen tilted to 70°. Quartz crystals are very sensitive to radiation damage which causes rapid amorphization under the intense point-focused electron beam dose. Therefore short pattern acquisition time of 40 ms was applied to avoid structure degradation of quartz consequently allowing us to collect sharp patterns and perform reliable pattern indexing.
The location of the twin boundary was clearly revealed on the SEM micrograph that was recorded using forward scattered electrons (FSE) and on the superimposed EBSD crystal orientation map (COM) where identical crystallographic orientations are displayed in particular colours: green, blue, pink and orange, as shown in Fig. 1. The misorientation angles along the line-profile across the twin boundary were determined relative to starting point in the left part of the crystal (green, A), as shown in Fig. 2. Obtained experimental value for misorientation angle between the inclined c-axes of the unit cells in two twin individuals (A and C) was ≈ 84° and fully agree with theoretical value 84.33° that is exactly attributed to the Japan-law twinned crystal. In the blue region (B) the unit cell is rotated about the c-axis for 60° as compared to the orientation in region A, so confirming the presence of Dauphiné twins which emanate in the vicinity of the Japanese twin boundary. Dauphiné twins in COM are displayed in blue and orange and were also recognized by the distinct change of the FSE contrast in the SEM micrograph.
Using an appropriate experimental FEGSEM-EBSD analytical set-up and optimized sample preparation we have successfully identified the types of twins in the “v”-shaped natural quartz crystal. The evaluation of the EBSD patterns and corresponding crystal orientations directly confirmed Japanese twins type with some Dauphiné twins located near the twin boundary.

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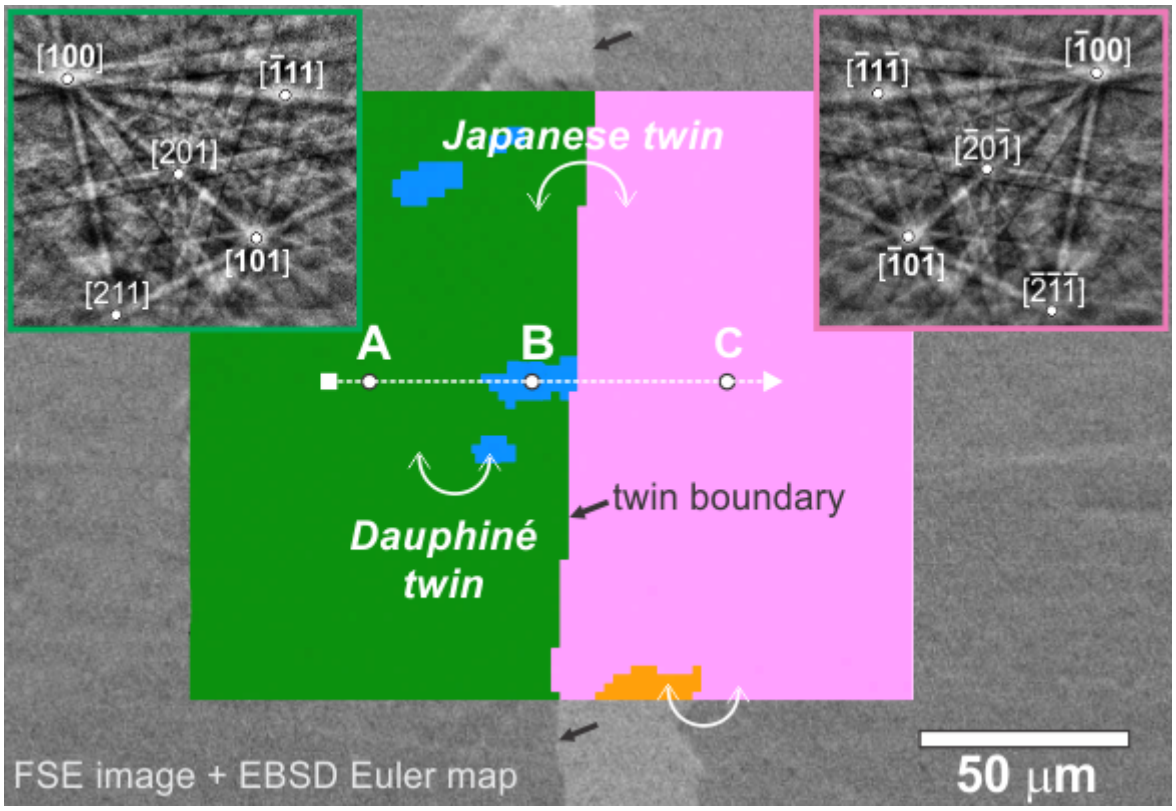

Fig. 1: The FSE micrograph with superimposed EBSD crystal orientation map of twinned quartz crystal showing the twin boundary location and the Kikuchi patterns acquired from the left (green) and the right (pink) region of the crystal. EBSD analysis confirmed Japanese twin type with some Dauphiné twins (blue, orange) near the twin boundary.

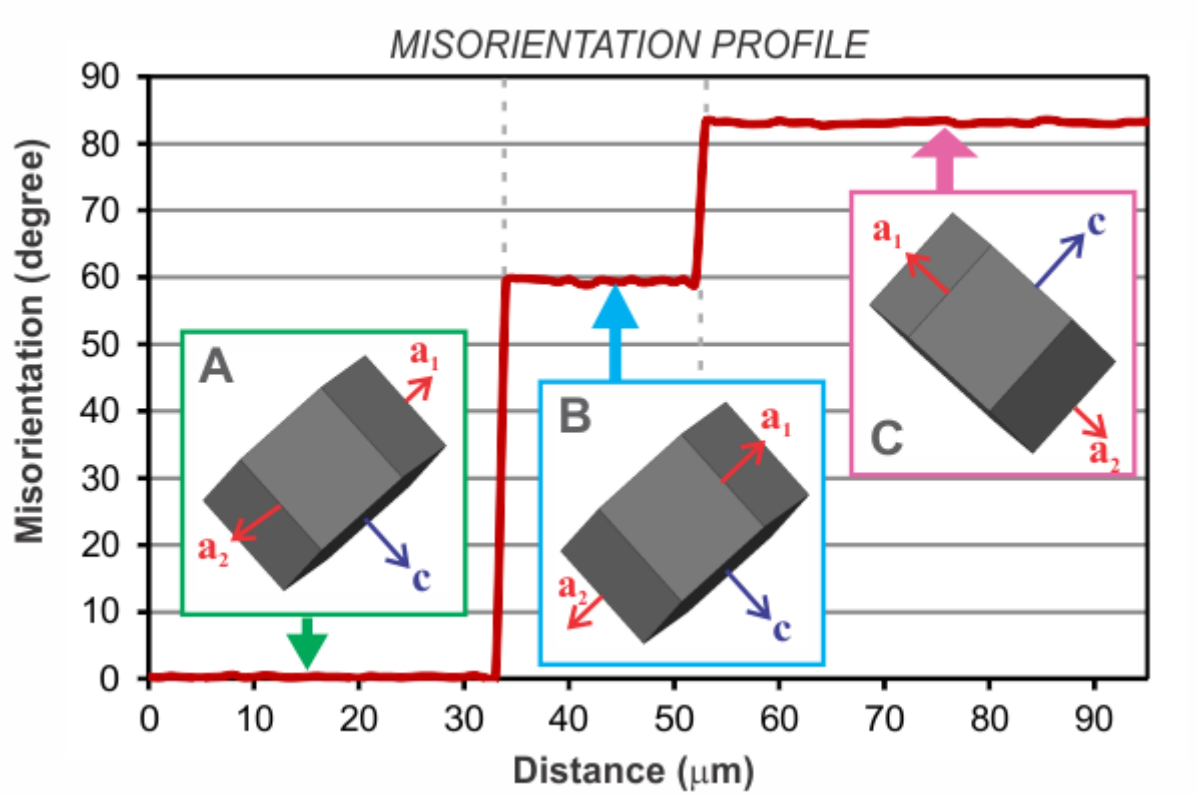

Fig. 2: Misorientation profile along the line shown in Fig. 1 with illustrated unit cell orientations in regions A, B and C. Relative to A region B has no change in c-axis but displays 60° rotation about c-axis which is specific for Dauphiné twinning. Region C clearly showed the rotation of the c-axis by ≈ 84° which corresponds to Japanese twins.