ESRF	**Experiment title:** Crystallography-driven long-range pore ordering in anodic alumina films	**Experiment number**: 26-02-693
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Anodic aluminium oxide (AAO) films prepared by anodic oxidation of aluminium under certain conditions possess hexagonally-ordered system of long cylindrical channels with a well-defined diameter. The purpose of the current experiment was the characterization of the positional, orientational and longitudinal order of the channel network as a function of the crystallographic orientation of the substrate. High-purity aluminium single-crystals with different crystallographic orientations ({100}, {110}, {111}, Mescrel, 99,9999 %) were used as the starting material.

Microradian diffraction patterns shown in Figure 1 demonstrate drastic difference in pore ordering for AAO membranes grown on aluminium single-crystal substrates with different crystallographic orientations. For {100} single-crystal several rings with uniform distribution of intensity along the rings indicate that within the irradiated area there are many domains that are completely disoriented with respect to each other in the plane of the oxide film. On the contrary, the aluminium substrate with {111} orientation exhibits a spot-like diffraction pattern that corresponds to the long-range orientational order over macroscopic distances larger than the beam size. In the case of aluminium single-crystal with {110} orientation (Fig. 1B) an intermediate situation is observed. Six-fold intensity modulation is visible but the diffraction peaks are significantly broadened in the azimuthal direction, which is induced by fluctuations of the in-plane orientation order, compared with {111} substrate.

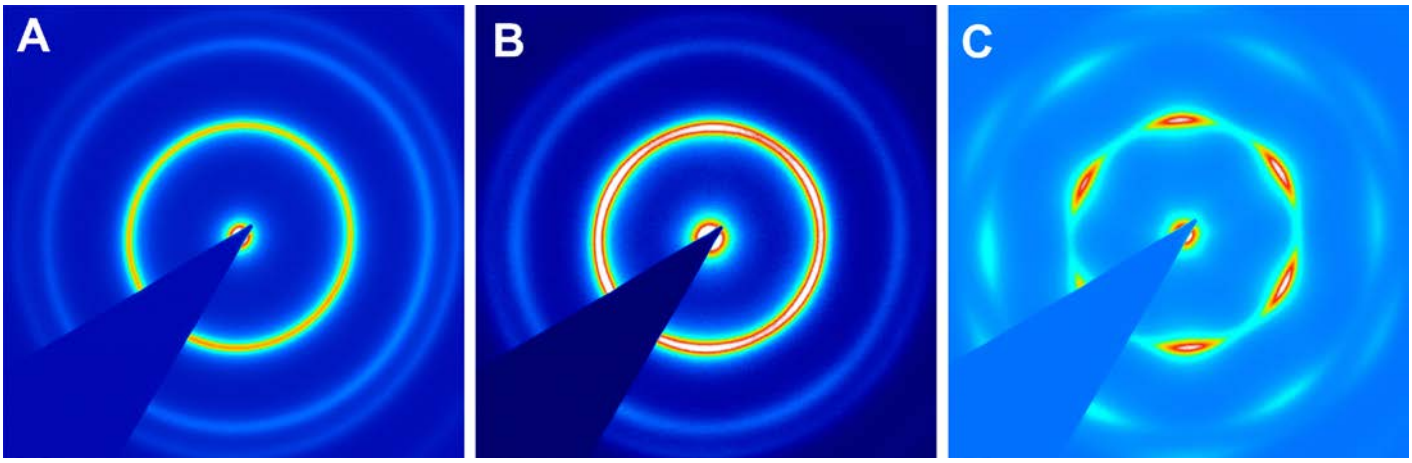

Fig. 1. Small-angle diffraction patterns recorded from AAO films, obtained in 0.3 M sulfuric acid at 25 V (the interpore distance is about 65 nm). Aluminium single-crystalls with {100} (A), {110} (B) and {111} (C) orientation were used as the substrates.

In order to observe the influence of the crystallographic orientation of the substrate on the longitudinal pore arrangement, a specially designed aluminium {100} single crystal (see red zone in Figure 2) with the vicinal edges having surface normal tilted from the {100} direction by 5° (see green, blue and violet zones) was used. Diffraction patterns recorded during rotation the sample around the vertical and horizontal axes normal to the beam are significantly different for each titled zones. In case of {100} plane diffraction patterns upon rotation change from a uniform ring to two symmetrical points, located on the rotation axis – vertical or horizontal, respectively (see the set of diffraction patterns in the red frame in Figure 2). Green and blue zones exhibit the same behaviour only in case of rotation around the direction of tilting (vertical for green zone and horizontal for blue one). On the contrary, in the perpendicular direction significant fluctuations of pore growth direction are observed, that leads to broadening of corresponding diffraction maxima. These microradian X-ray diffraction data demonstrate high influence of crystallographic orientation of Al substrate on orientational and longitudinal order that is in good agreements with our recent results [1,2]. More detailed analysis of the observed results will be published soon.

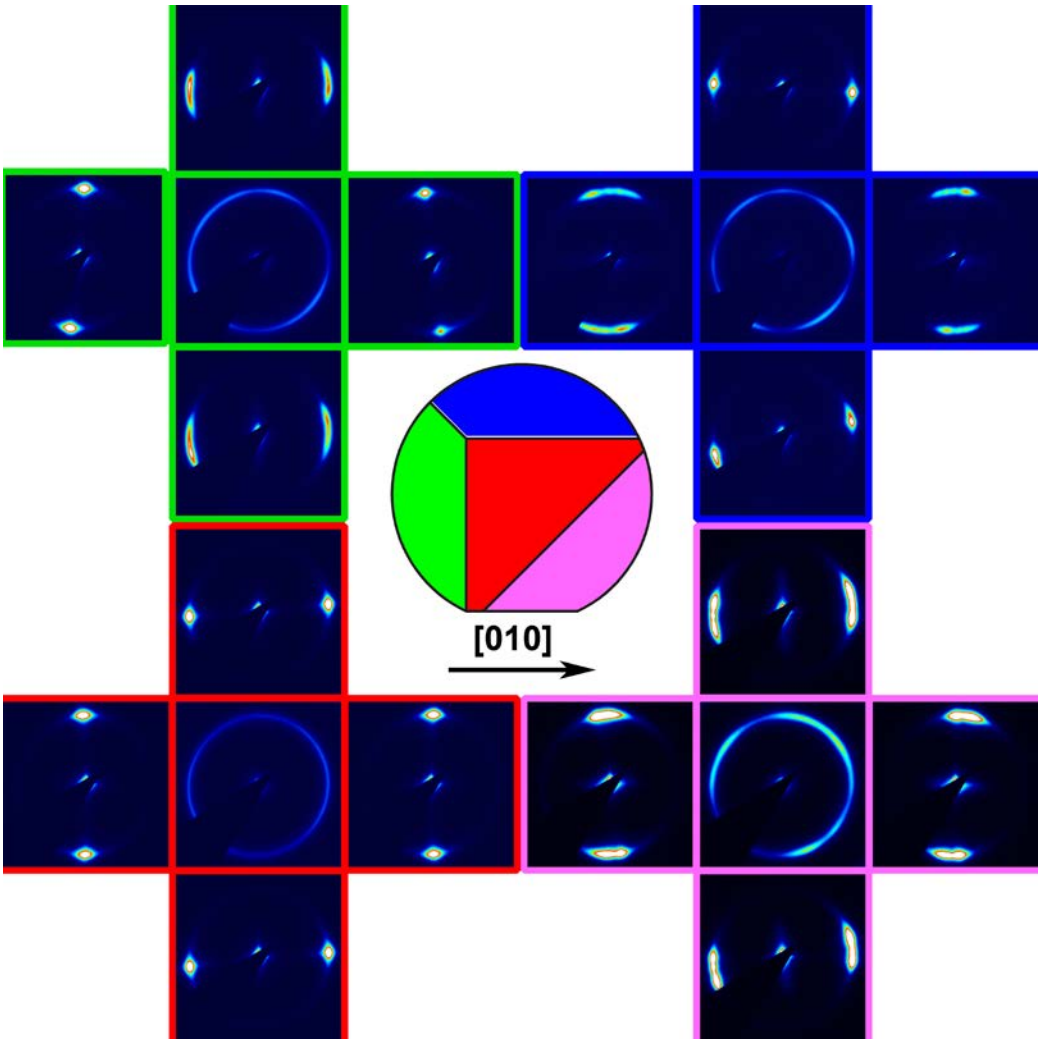

Fig. 2. A sketch of aluminium {100} single-crystal substrate with three vicinal zones tilted from the {100} direction by 5° (green, blue and violet) is shown in the centre. Small-angle diffraction patterns recorded at normal incidence of the X-ray beam to the AAO film surface (in the centre of each sets of diffraction patterns) and for rotation angles equal to ± 1.0^{o} around the vertical (left and right patterns) and horizontal (top and bottom patterns) axes normal to the beam. AAO porous film was obtained in 0.3 M oxalic acid at 40 V (the interpore distance is about 105 nm).

Finally we would like to thank Daniel Hermida Merino for his excellent support during the experiment.

References:

[1] K.S. Napolskii et al., J. Mater. Chem., 22, 11922-11926 (2012).
[2] I.V. Roslyakov et al., *J. Appl. Cryst.*, 46, 1705-1710 (2013).