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Crystallisation kinetics in sol-gel PbTiO$_3$ films by real-time XRD analysis. <u>Ann-Christin Dippel</u>[a], Theodor Schneller[b], Rainer Waser[b], [a]*HASYLAB, DESY Hamburg, Germany.* [b]*Institute for Materials in Electrical Engineering, RWTH Aachen University, Germany*
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Electroceramic thin films have become an essential part in various fields of modern technology. In this regard, lead titanate based perovskite compounds such as $Pb(Zr_{1-x}Ti_x)O_3$ (PZT) are especially relevant owing to their piezoelectric, ferroelectric, and pyroelectric properties. A flexible, technologically straightforward, and cost-effective method to derive these films is the chemical solution deposition (CSD) method. In this work, we report on the mechanisms that were identified to govern the grain growth and morphological characteristics of CSD films of PbTiO$_3$. The results for the pure binary oxide provide a basis to study the respective processes in more complex lead titanate derivatives like PZT.

The initial step of the CSD routine consisted in the synthesis of a precursor solution from organometallic lead and titanium components. Subsequently, the as-prepared solution was deposited on a substrate and heat-treated in order to form the oxide layer. Crystallisation set in after the organic constituents were decomposed due to the thermal impact. By use of a temperature chamber mounted to a laboratory x-ray diffraction instrument, the evolution of the perovskite reflections were observed *in situ* during the heating of the films. The time-resolved data were evaluated using the Johnson-Mehl-Avrami approach that yielded information about germ formation and grain growth. As the numeric results indicated, the perovskite crystallites nucleated instantaneously and grew one-dimensionally in a diffusion controlled process. These findings suggested that laminar nuclei formed at the substrate film interface in the very early stages of the crystallisation. When the whole interface was covered after impingement of the nuclei, the latter proceeded to grow in the remaining direction towards the layer surface. This model applied independent of the particular crystallographic orientation of the grains. Furthermore, it effectively accounted for the observed columnar morphology of the layers. In further experiments, the influence of substituents in lead titanate on the described mechanism is sought to be determined.

Keywords: *in situ* time-resolved powder diffraction, nucleation and crystal growth, lead titanate thin films

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Conditions for reception of x-ray monochromatic bunches of the maximum intensity in geometry Laue. <u>V.V. Margaryan,</u> K.T. Hayrapetyan, S.N.Noreyan, *X-Ray Pungs Laboratory. Yerevan, Republic of Armenia*
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In the presented work dependence of absolute value of intensity of monochromatic x-ray radiation, diffracted from a direction of a incidence primary bunch in a reflection direction in geometry Laue in the presence of external influences (a condition of a full pumping), from a thickness of a single-crystal is investigated. As is known, the angular width of a full pumping x-ray bunch, in the presence of external influences, depends as on a thickness of a single-crystal (t) [1], and from distance a radiation source - a sample [2]. At the fixed distance a radiation source - a single-crystal in process of increase in a thickness of a single-crystal the angular width of a full pumping x-ray bunch linearly increases.

From the previously mentioned follows, that with increase in a thickness of a single-crystal the sizes of angular area of a full pumping from a direction of a primary bunch in a reflection direction increase, integrated intensity of the reflected bunch therefore increases.

On the other hand, the increase in a thickness of a single-crystal leads to increase in the integrated intensity absorbed by a single-crystal. It is natural to assume, that in the described conditions, changing a thickness of a disseminating single-crystal, it is possible to find that optimum thickness at which the absolute value of a full pumping x-ray bunch in a direction of reflection will be maximum.

As a result of the detailed analysis of results of an experimental research it is shown, that the absolute size of integrated intensity full pumping from a primary direction in a direction of reflection of a x-ray bunch reaches the maximum at condition observance: $\mu t=1$, where μ - factor of linear absorption, t - a thickness of a single-crystal.

The validity of the given condition is confirmed also by theoretical calculations.

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Application of a frame-store pnCCD in protein crystallography. <u>Sebastian Send</u>[a], Ali Abboud[a], Ullrich Pietsch[a], Wolfram Leitenberger[b], Robert Hartmann[c], Lothar Strüder[a,d,e], [a]*University of Siegen, Germany* [b]*University of Potsdam, Germany,* [c]*PNSensor GmbH, Munich, Germany,* [d]*MPI Halbleiterlabor, Munich, Germany,* [e]*Max-Planck-Institut für extraterrestrische Physik, Garching, Germany*
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The fast and precise determination of crystal structures using the Laue diffraction method requires a simultaneously position- and energy-resolved detection of photons. By means of a back side illuminated frame-store pnCCD [1] invented at the Halbleiterlabor of the Max-Planck Institut (MPI-HLL) for applications in X-ray astronomy the spatial distribution of Laue spots as well as their energies can be measured at the same time. The sensitive volume consists of a fully sideward depleted n-Si layer of 450μm thickness subdivided into 256x256 pixels of 75x75μm^2 size with a typical readout frequency of about 120 Hz. The system has found applications for soft and hard X-rays [2].

Using white synchrotron radiation the frame-store pnCCD delivers a three-dimensional intensity distribution spanned by two pixel directions and an energy direction covering a three-dimensional data volume in reciprocal space which is expanded to 4D (time) in case of dynamic measurements. In this sense the system provides a good possibility to investigate unknown crystal structures in arbitrary orientations in space as well as structural phase transitions at fixed scattering geometry. Previously it was shown that the crystallographic unit cell of a γ-LiAlO$_2$ crystal can be determined from the three-dimensional pnCCD data sets without any a priori information about the sample [3]. Consequently the energy-dispersive Laue diffraction is a powerful tool for single shot structure analyses in macromolecular crystallography. In the subsequent application the potential of the frame-store pnCCD is exploited to analyze small organic crystals exhibiting complex structures and weak scattering signals. As a test example the energy-resolved Laue pattern of a lysozyme single crystal from hen egg white (HEW lysozyme) is considered in transmission geometry and used for structure determination. The results will be compared to measurements obtained from HEW lysozyme polycrystals.

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Keywords: pnCCD, energy-dispersive Laue diffraction, protein crystallography