Material-oriented project example

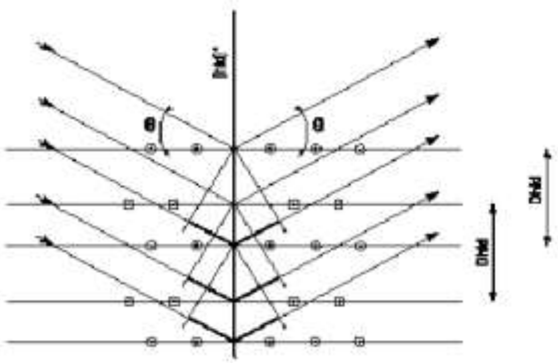
Crystal defects identification by X-ray topography

Purpose: some material, either for optical WGM sources (BaF_2, CaF_2, MgF_2), or acoustic (Si, ..) or piezoelectric material (SiO_2, $LiNbO_3$,…) for BAW or SAW devices, may exhibit perturbed properties (wave propagation velocity, acoustic/optical Q,…) impacting final device behavior or manufacturing yields. Thanks to nanotechnology, devices are becoming smaller and smaller, and that means that material properties under the active part of the device become more and more critical, requiring more and more attention.

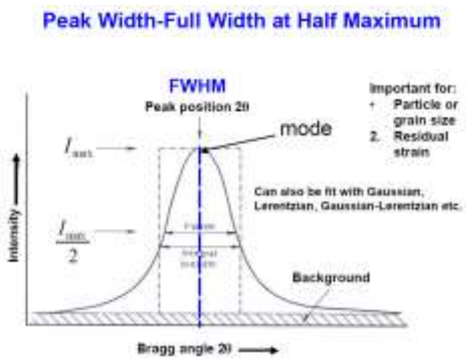
In crystalline media, the main defects are dislocations (edge, screw, mixed), inclusions, impurities (interstitial, substitutional), vacancies, etch channels, stacking fault … Any wafer sliced within a crystal bar containing defects such as inclusions (defined as number of inclusions of given dimension per cubic centimeter) will have a direct density of defects emerging on surface. All these defects may have an impact on devices manufactured from such a surface (Si, AsGa, $LiNbO_3$, SiO_2,…).

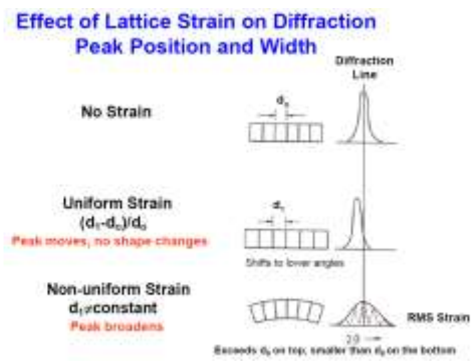
X-ray topography is mostly based on analysis of diffraction.

Basic is the Bragg law: X-ray is diffraction by a family of planes, distant by d_{hkl} when the geometrical configuration between the incident beam, the position of these planes and the position of the X-ray detector and the X-ray wave length fulfill: 2. d_{hkl} .sin $\Theta_{hkl} = \lambda$, where d_{hkl} is the distance between planes of the hkl family. $d_{hkl}=a.(h^2+k^2+l^2)^{-1/2}$ for a cubic crystal, and might be more complex for less symmetric materials.



Diffraction peaks are characterized by the "peak position" (average), the FWHM, the Full Width Half Maximum) and the integral intensity.



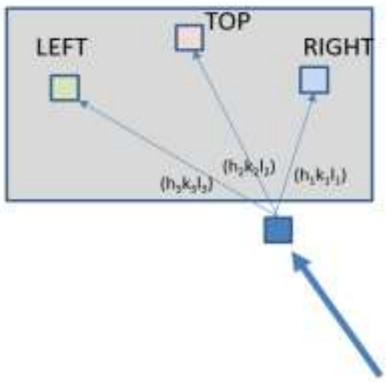


Diffraction peaks might be perturbed in peak position, intensity and width, by any local defects.

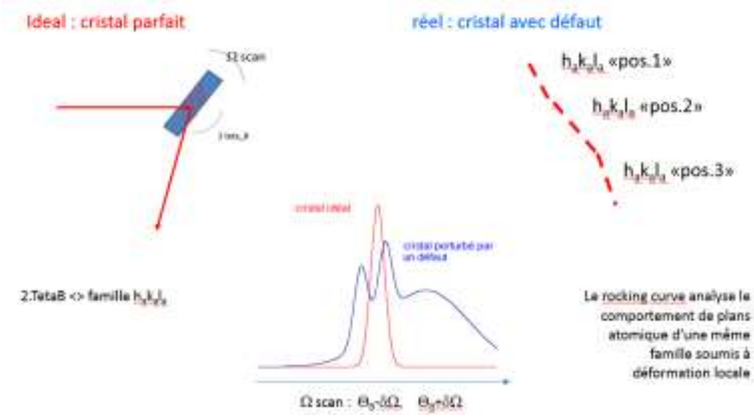
The FWHM might be defined as FWHM = $(FWHM_{instrument}^2 + FWHM_{intrinsic}^2 + FWHM_{defect}^2)^{1/2}$, which allows, by comparing best and worst case, to quantify defects impacts. All diffracted peaks are impacted by local defects. Comparison of FWHM, Integral intensity, peak position, each reveal some aspect of the defect, and allow to get a better understanding of nature, origin, size, orientation, depth, … of defects. Furthermore, the HIRSCH law, allows to define the defect disorientation impact on interatomic plane distance by $\Delta d_L = FWHM_{defect}^2 / 9b^2$, where b is the Burgers factor of the dislocation

Then, analysis of diffraction peaks, such as peak position, integral intensity, FWHM interpretation may provide a lot of informations about the defects , their origins and how to avoid or cure, such defects in crystalline material .

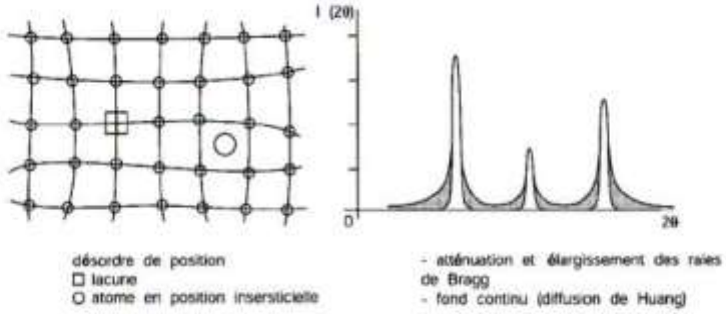
Laue diffraction: A high energy white beam illuminates a crystal sample. As there were many wavelengths simultaneously in the beam (white beam), some atomic planes ($h_ik_il_i$) fall in diffraction position, and the diffraction is observed on various position in the projection screen. Comparison of projected images through the different atomic planes, will provide informations on local defects (size, orientation)



Rocking curve, monochromatic beam, Bragg diffraction: In this monochromatic bean experiment, the Bragg diffractometer targets one family of plane, and the observation is the exact $\Theta(\Omega)$ local diffraction position. In short we observe the local "misorientation" of the diffracted beam via a small Ω scan around the average Θ_B diffraction position.



X-ray diffraction , such as HR-XRD, High Resolution X-Ray diffraction is a powerful tool to identify atomic place position disorder, which might be created by local defects (inclusions, dislocations, interstitial impurities, substitutional impurities, vacancies, stacking defects, etch channels,…)



Application: case one – Project BaF2 [111]

BaF_2 is a crystalline material widely used for its interesting optical properties, mainly its high transparency in a wide wavelength range, from near UV/visible to IR, from 200 nm to 10 μm. In WGM resonators (optical Whispering Gallery Mode resonators used in Brillouin scattering). RF-optical sources, optical and acoustic material Q are critical, and are highly impacted by crystal quality.

Experimental X-ray analysis shown below (*courtesy of CSEM Neuchâtel*) were used to characterize defects and allow to select curing solutions to devices issues.

Sample: BaF2_111_VUV

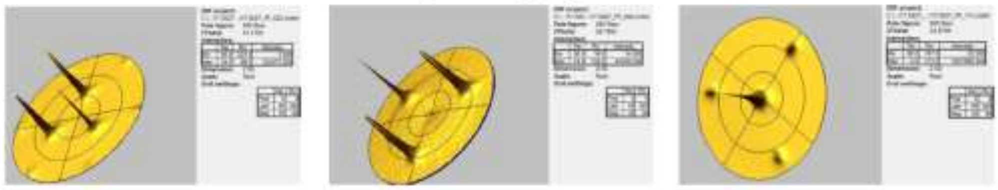


Fig 12. Pole Figure for (022) orientation of BaF2_111_VUV

Fig 13. Pole Figure for (002) orientation of BaF2_111_VUV

Fig 14. Pole Figure for (111) orientation of BaF2_111_VUV

- The observation of a 3-fold multiplicity (Figs. 12 and 13) for (022) and (004) planes confirms the (111) orientation of the sample. The additional bumps observed in Figs. 12 and 14 correspond to the other orientations in the same family of planes.

Crystalline Quality Assessment

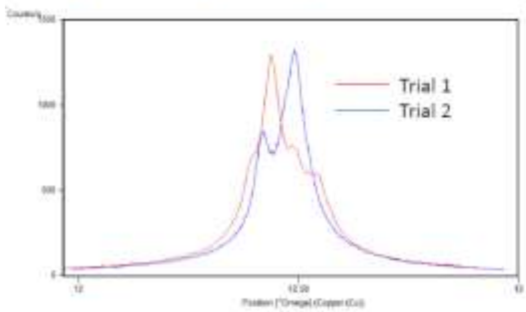
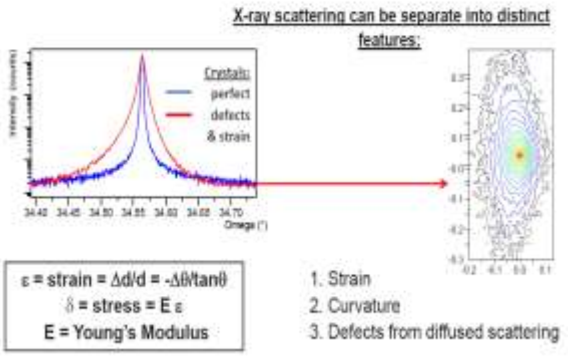
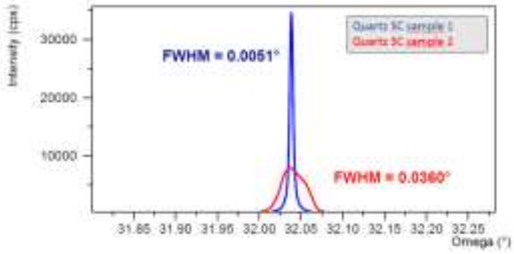


Fig. 16:Rocking Curve for the (111) peak for BaF2_111_IR

- The rocking curve measurement for BaF2_111_IR (Fig. 16) shows the splitting of peaks, observed consistently at different regions of the sample. The average FWHM of the sample is 0.121°.
- The peak splitting could be explained either by:
  - the slight difference in the scattering power in the depth of the monocrystal, linked to the lattice strain gradient. Large value of FWHM confirms the presence of the strain within the lattice
  - the system of the pseudo-planes (twinning)

Application: case two – Project thickness shear resonator quartz material



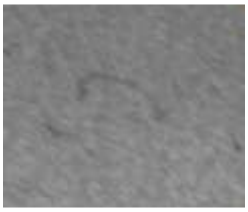


The presence of an asymmetry in the reflections could be related to lattice strain and / or tilt. Larger FWHM is related to imperfections within the quartz single crystal lattice.

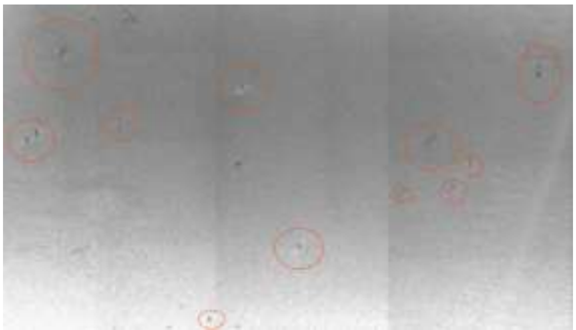
Application case three : crystal wafer inspection:

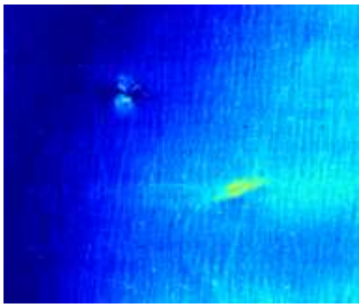
Purpose was to identify origin of defects affecting the manufacturing of devices on crystal wafer. Density and size of defects were highly impacting device yield and performances.

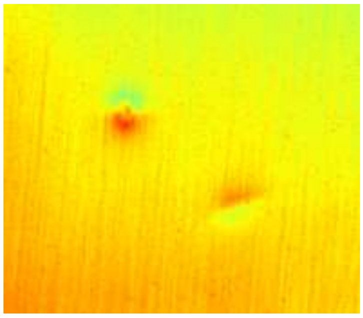
Dislocation loop (White Beam):



Typical observation of dislocation and inclusions







FWHM and integral intensity - Monochromatic Rocking curve. *Courtesy of ESRF*.

These topographies reveal crystal defects such as dislocations, inclusions, through specific “X-ray signatures”, or stacking defects, and even change in interplane distance d_{hkl} due, for example, to high concentration of interstitial or substitutional impurities.

Once properly done and analyzed by experts, this shows to be a very powerful tool, in regards of the technological evolution and miniaturization of devices.