on the type of electrolyte and the voltage for oxidation applied. For both electrolytes, the film thickness increased with the voltage applied.

The concentration of the electrolyte produced minor shifts in color, negligible enough to establish a relation between color and thickness of the film for each electrolyte as a way to have a quick method to determine thickness.

The films produced at low voltages were homogeneous, with low roughness and amorphous. At higher voltages, sparks discharges were observed and porous films, with a degree of crystallinity, were produced. The voltage for spark discharge depended on the electrolyte.

Using H_2SO_4 1M as electrolyte, the films produced were compact, homogeneous and no crystalline phases of TiO_2 were detected up to 60V. From 70V to 80V, the films were porous and crystalline with the anatase phase formed, and above 80V, the rutile phase was formed. As the concentration of electrolyte increased, the conductivity increased and the homogeneous/amorphous to porous/crystalline transition was produced at lower voltages.

In the case of the H_3PO_4 1M electrolyte, there were no spark discharges or crystalline phases up to a voltage of 100V, which was the highest voltage used.

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Keywords: electrochemical oxidation, coating, acid electrolyte

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Evaluation of polychromators for angle-wavelength dispersive X-ray reflectometry

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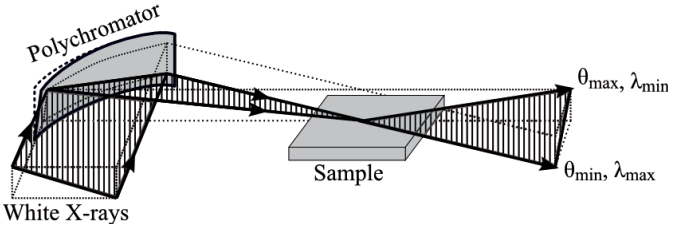
We are developing a method for time-resolved measurements of the specular X-ray reflectivity [1], [2], [3]. In this method, a convergent X-ray beam with a one-to-one correspondence between angle and X-ray energy impinges onto the sample (Figure). The incident beam has a range of specular angles θ and wavelengths λ. The reflected X-rays are detected with a two-dimensional detector. The reflectivity curve in the range $q_{min}=4\pi\sin\theta_{min}/\lambda_{max}$ to $q_{max}=4\pi\sin\theta_{max}/\lambda_{min}$ can be measured in a single detector exposure, with no need to scan the sample, incident beam, or detector.

A key element of the reflectometer is the polychromator that produces the convergent X-ray beam. It consists of a Si crystal curved in two dimensions so that the Bragg angle and the angle to the horizontal plane change continuously with the position on the crystal. The curvature is adjusted to focus the incident white X-rays onto the sample.

In this report, different methods for bending the Si crystal to the desired shape are compared. They were evaluated using visible laser light. Size and shape of the reflected laser beam were recorded with a CCD camera at different distances from the focus. Finite-element simulations of the bending were done as well.

In the first method for bending the Si crystal, the crystal is fixed at two opposite edges. The crystal is bent by moving the edges independently. This method has the advantage that the curvature of the crystal can be adjusted during the experiment, but it is difficult to produce small focus sizes. In the second method, a Si crystal is pressed to a copper support that was machined to the desired shape. Alignment is comparatively simple and small focus sizes are easier to achieve.



Setup for the X-ray reflectometry measurements.

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The five twin laws of gypsum. A theoretical analysis on interfaces of the growth contact twin

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All the twin laws of gypsum belong to the [010] zone, which is not an important one from the morphological point of view, as it ensues from the fact that the [010] direction does not correspond to a periodic bond chain (PBC), in the sense of Hartman-Perdok.

Our research starts from the experimental observation that the five twin laws of gypsum can be divided in two groups: to the first one belong the 100 and $\bar{1}$ 01 laws, both characterized by a high occurrence frequency, while to the second and less important group belong the 20 $\bar{1}$, 001 and the 101 twin laws. Concerning both the natural and the solution grown crystals, the $\bar{1}$01 penetration twins have by far the highest occurrence frequency, while the $\bar{1}$01 contact twins were never observed; the 100 penetration twins are more frequently observed that the 100 contact twins, for every supersaturation value.

We considered the contact twins only and attempted at calculating the twin energy for each twin law, starting from suitable unrelaxed profiles of each twin interface and evaluating the convergence of the twin energy values once the interface was allowed to relax. Hence, we were able to model both the amount and the extent of the perturbations occurring at atomic level, for each twinned interface, and compare with the relaxed and untwinned one.

The most probable contact twin law results to be 100, the corresponding twin energy being 13 erg cm^{-2}. The twin energy of $\bar{1}$01 and 001 laws is higher by an order of magnitude, being 145 and 255 erg cm^{-2}, respectively. For the remaining laws the twin energy value is even more higher and the convergence is hard to obtain.

This should change our mind on the interpretation on the genetic mechanism of gypsum twins:

- the only "true" contact twin is 100, all other twins being related to penetration mechanism;
- the morphological confusion between 100 and $\bar{1}$01 laws can be experimentally avoided by the control of the optical extinction, according to the Cody's hypothesis;

- the evaluation of the probability of twinned 2D nucleation on the (010) faces will be discriminating to understand the complex genetic mechanism of penetration twins.

Keywords: gypsum, twin

MS22.P01

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Structure of the whole plakin domain of plectin

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Plectin is a member of the plakin family of proteins that cross-links components of the cytoskeleton and link them to membrane-associated structures, such as desmosomes and hemidesmosomes. Plectin (500kDa) exhibits a multi-domain structure. The N-terminal region contains a ~ 1000-residue long sequence conserved between the members of this protein family, termed the plakin domain. The plakin domain of plectin is formed by an array of nine Spectrin Repeats (SR1 to SR9) arranged in tandem and a Src-homology 3 (SH3) domain inserted into the central SR5 [1].

We have combined X-ray crystallography and small angle X-ray scattering (SAXS) to elucidate the structure of the plakin domain. Here, we present the crystal structure of four fragments that correspond to the regions: SR3-SR4, SR4-SR5-SH3, SR7-SR8 and SR7-SR8-SR9. The SR-fold consists on three α-helices (A,B and C) connected by short loops and packed in a helical bundle with a up-down-up topology. In adjacent SRs, the helix-C of the N-terminal repeat and the helix-A of the C-terminal repeat are fused in a single helix that spans both SR, yet there is no conservation in the relative orientation of adjacent SRs. The SH3 domain of plectin shows the canonical SH3 fold, but exhibits alterations in its putative Pro-rich binding-site suggesting that this domain does not bind to Pro-rich motifs as the canonical SH3 domains [2]. Moreover, the SH3 binding-site is occluded by the SR4, making extensive contacts with it. Residues that participate in the SR4-SH3 interaction as the residues of the SH3 pseudo-binding site are conserved in other members of the plakin family. The structure of the plakin domain of plectin presented herein, serves as a structural model for other plakins.

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Keywords: plectin, spectrin repeat, SH3

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Conformational Plasticity of Histidine Kinases is Key for Signal Transduction

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DesK is a membrane-bound histidine kinase (HK) from *Bacillus subtilis*, able to sense the order of membrane lipids when cells are subjected to cold shock, ultimately behaving as a molecular thermometer. Although the relevance of sensor HKs in signal transduction is well established, we still do not understand at the molecular level how HKs transduce input signal information to regulate their output catalytic activities. We address this issue by using a combination of structural and biochemical approaches. We have determined eight crystal structures of the intracytoplasmic catalytic core of DesK, including the wild-type, the phosphorylated form, and point mutants that retain particular functional traits.

Structural analyses show that DesK has been trapped in three conformational states that correspond to alternate functions of the protein along the signaling pathway [1]. By comparing the 3D structures of a single HK in different functional configurations, we observe a remarkable plasticity in the central helical domain. Incoming signals induce helix rotations and asymmetric helical bends that modify the accessible surface of the phosphorylation site and the mobility of the ATP-binding domains, ultimately modulating the protein's catalytic activities. The transition between conformational states through helical rotational shifts, was analyzed using Targeted Molecular Dynamics simulations, further supporting their role as a functional signal transduction mechanism.

The central four-helix bundle domain includes coiled-coil structures that reach the histidine phosphorylation site. The trans-membrane sensor region seems to drive the helical rearrangements. Heptad-repeat sequence features allow for the extension or disruption of the coiled-coil towards the N-terminus of the catalytic core, ultimately serving as a signal transmission gear. In correlation with these movements, the flanking ATP-binding domains, remain either rigidly fixed to the 4-helix bundle, or otherwise free to move. We have explored the transient intradimeric autophosphorylation state by semiflexible docking algorithms, leading to a proposed mechanism working in *trans*, one monomer phosphorylating the other. Structure-based cysteine engineering lends support to the working hypotheses, allowing us to trap an intermediate state with disulfide bridges between the two domains [2]. Negative cooperativity leads to phosphorylation of only one monomer within the dimer.

Structure-based mutagenesis and protein engineering experiments *in vitro* and *in vivo*, confirm the importance of the 'coiled-coil'-mediated plasticity in the conserved central phosphotransfer domain. Similar switching mechanisms could operate in a wide range of sensor HKs. Structural studies of the interaction of DesK with its cognate response regulator DesR are currently underway.

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Keywords: coiled-coil, allostery, phosphorylation

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PLZF oncoprotein; An extensive SAXS analysis

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In Acute Promyelocytic Leukemia (APL) the balance between stem cell differentiation and proliferation is disrupted. The promyelocytic zinc finger protein (PLZF) is a transcriptional repressor, and is one of six