

The magnitudes of the strains decrease outside the width of the laser beam footprint. The first laser pass induces through-thickness strains close to yield, whereafter their magnitudes decrease with increased number of laser beam passes. A comprehensive mapping of the longitudinal stresses as function of the number of laser passes is given.

Keywords: strain scanning, laser bending, residual stress

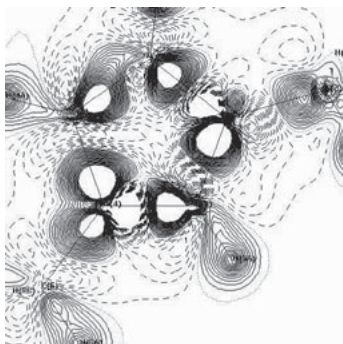
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Charge density and *in-situ* studies of ionic and ionothermally prepared materials

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X-ray diffraction is an important technique in structure determination. However zeolites and many of the porous materials we look at are notoriously difficult to grow as large single crystals. With the use of synchrotrons such as the SRS we are able to probe these fascinating structures to obtain not only initial structural models but also high resolution X-ray data sets (figure1). Our group uses stations 9.8 and 16.2 SMX to investigate these materials. We are also using the high energy dispersive beam line of 16.4 to investigate how they crystallise. We are able to produce porous materials with the use of ionic liquids which act as both a template and as a solvent reducing the number of interactions in the vessel making it easier to try and understand the mechanism.



Keywords: ionothermal prepared materials, high Resolution Charge Density, porous materials

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Hydrothermal synthesis and structural features of metal phosphates in the presence of amines

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Since the first crystalline compound, α -Zr(HPO₄)₂·H₂O, was obtained, these compounds have been extensively studied due to the possibility of relating their properties and crystalline structure, and the many potential applications. Initially, γ -Zr(H₂PO₄)(PO₄)·2H₂O, was erroneously believed to be a modification of the α -phase. Advances in the knowledge of the γ -layered structure were fundamental for the interpretation of the topotactic mechanism of formation and development of the chemistry of the γ -derivatives. Although only two crystalline forms of layered titanium phosphates are known, γ -

-Ti(HPO₄)₂·H₂O and γ -Ti(H₂PO₄)(PO₄)·2H₂O, many derivatives have been reported. Surprisingly, the γ -layered titanium phosphate is less studied than the α -phase, even though the γ -layers are more rigid, thicker, acidic and amenable to intercalation processes. More than a decade ago, we have reported the mechanism of intercalation of n-alkylamines in γ -titanium phosphate, showing that only 66% of the P-OH groups interact with the amine groups of the guest molecules. Geometrical constraints dictate that compounds with maximum intercalation usually have the empirical formula of γ -Ti(H₂PO₄)(PO₄)·1.3C_nH_{2n+1}NH₂·H₂O (with n = 1 to 6), and crystalline phases with lower amine content can also be isolated. The hydrothermal synthesis and the structural characterization of γ -titanium phosphate intercalation compounds have been only recently reported.[1,2]

[1] Mafra, L.; Almeida-Paz, F.A.; Rocha, J.; Espina, A.; Khainakov, S.A.; Garcia, J.R.; Fernandez, C., Chem. Mater. 2005, 17, 6287.

[2] Mafra, Luis; Rocha, Joao; Fernandez, Christian; Castro, German; Garcia-Granda, Santiago; Espina, Aranzazu; Khainakov, Sergei; Garcia, Jose R., Chem. Mater. 2008, 00, 0000.

Keywords: phosphates, hydrothermal synthesis, amines

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Iron incorporated in cubic mesoporous silica: Synthesis, composition and structure

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Ordered mesoporous silica (OMS) grafted with different atoms are interesting new structures with high specific surface, large pore size and uniform pore distribution for potential applications in, for example, catalysis, green chemistry and drug delivery systems. In this work, we report the direct incorporation of iron in OMS, with cubic FDU-1 structure. The structural properties of samples prepared under different conditions were investigated by Small Angle X-ray Scattering (SAXS) and large angle X-ray Diffraction (XRD). The local structure around the iron atoms was analyzed by X-ray absorption spectroscopy (XAS). The composite material is mostly formed by amorphous silica and hematite, with some amount of Fe²⁺ in the structure. The morphology, structural and textural properties were analyzed by N₂ adsorption isotherms. The variation of the synthesis pH from 1 to 3 decreased the total surface area. Rutherford Backscattering Spectrometry (RBS) was used to determine the amount of Si, O, Cl and Fe in the samples. The amount of iron inside the silica matrix depended on the preparation method, and an optimized synthesis process has been developed aiming to obtain high iron content (up to 4 wt.%) and low chlorine incorporation.

Keywords: porous materials, SAXS, EXAFS and XANES

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Cation exchange mechanism for natural apatite with a simulated Cd-polluted solution TEM-AEM and XPS

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