Book of Abstracts

INTERM 2021

8th International Congress on Microscopy & Spectroscopy

INTERM

October 17-23
2021

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8th International Congress on Microscopy & Spectroscopy

(INTERM 2021)

October 17-23, 2021

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INVITED SPEAKERS

Id-471

X-ray Absorption Near Edge Spectroscopy Used to Determine Local Atomic Structure of Ions in Solution. The Case of as in Water

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Abstract: X-ray absorption Near Edge Structure (XANES) refers to the region of the x-ray absorption spectrum which is situated few electron volts above the absorption edge of a particular element. Usually this part of the x-ray absorption spectrum is used to infer qualitative information about the local electronic structure around the absorbing atom, e.g., formal valence. The region of the x-ray absorption spectrum above 30-50 eV from the edge referred as extended x-ray absorption fine structure (EXAFS) is used to obtain in a straightforward manner information about the local atomic structure around an absorbing atom, e.g., coordination numbers, bond-length distance, and mean squared fluctuations about the average bond length. However, in highly disordered systems or ions in solution EXAFS can only provide information about the nearest neighbor coordination shell. In those systems a detailed interpretation of the XANES spectra can yield local atomic structural information not available using other spectroscopical techniques, like neutron diffraction, x-ray diffraction or EXAFS. As an example, we discuss the local atomic structure around arsenic ions in solution. Such information is important in order to understand the toxicity of arsenic at the molecular level. XAFS measurements of acidic arsenic solutions indicate that arsenic appears as a cluster of As(OH)3 with As bonded to 3 OH radicals. The comparison between theoretically generated EXAFS and experiment, indicate that nearest neighbor structural configurations generated using ab-initio density functional theory calculations are not consistent with the experimental results, while configurations obtained from molecular dynamics simulations are. These simulations also yield configurations, which include neighboring water molecules. The use of those configurations in ab-initio XANES calculations reproduces experimental results. Such configurations have as a common element water molecules forming an irregular hexagonal array above the equatorial plane defined by AS-OH bonds, with three water molecules forming hydrogen bonds with the As(OH)3 cluster. This structural arrangement, known as clatharte structure has been proposed as a possible structure that would cross the cell membrane, explaining the high toxicity of arsenic (III) in solution.

Keywords: XANES, local structure in liquids.

INVITED SPEAKERS

Id-529

Research&Development of inorganic nanoparticles for Biomedical Applications on the Example of Metal Sulfides and Metal Oxides

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Abstract: In the previous and this year, some infectious diseases continue to spread, despite the creation of a vaccine for their prevention. Therefore, it is very important to develop the express methods for the viruses and bacteria detections. Moreover, we need new materials with enhanced properties to keep human health. In recent years, remarkable advances have been made in design of inorganics materials for medical and biological applications. Each nanoparticle system has a niche and that an 'one for all' approach cannot be applied. That is why various microscopy techniques are used for the studies. In ours studies two types of nanoparticles were studied, namely, sulfides: Ag_2S and CdS in water solution with varies stabilized agents for fluorescent imaging and detections and oxides: titanium monoxide (TiO_y) with varies stoichiometries to enhance the properties of hydroxyapatite (HAp) for tissue engineering applications. We have examined interaction of the nanoparticles with red cells of peripheral blood and the others biological cells. The results of the interaction have been shown to depend on the particle size and charge. The possibility of observing the structure of the cytoplasm and nucleus allows one to explore processes induced in the cell by various external influences, e.g., the breakdown of the cytoplasm and nucleus under the action of viruses. Conjugates are shown to retain their fluorescence properties for a long time. New method of synthesis of a stable colloidal solution of Ag_2S nanoparticles has been suggested, new concept of nanoparticle size regulation has been developed. The results of the investigation of the nanocomposite materials TiOy/HAp with different stoichiometry of additives by the XRD, HRTEM, IR and Raman spectroscopy techniques showed that the properties of the composite material depend on the stoichiometry and amount of TiOy additives. This findings allow~~s~~ to design a new properties of nanocomposites for bone tissue engineering like enhanced mechanical properties, adsorption capacity, porosity, photocatalytic activity etc. This work is

partially (since 2019) supported by the Russian Science Foundation (project No. 19-73-20012) at the Institute of Solid State Chemistry, UB RAS.

Keywords: Inorganic Nanoparticles, Biomedical Applications.

INVITED SPEAKERS

Id-530

In situ Disordering of Nonstoichiometric Monoxides of IV-V Groups by Means of Transmission Electron Microscope

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Abstract: Material design with specified properties is of pivotal interest at present time owing to their importance for synthesis of the materials with the required functional properties. One way to change structure and properties is to introduce structural defects. Structural vacancies are associated with different effects of nonstoichiometry, short-range order, and long-range order. These effects make it possible to synthesize a wide variety of phases and modifications with different properties by changing the number of vacancies in the sublattices, as well as the manner of their arrangement in the sites of the sublattices. In the disordered state, vacancies are arranged in the sites of the sublattices without a long-range order and do not lead to the breaking of the symmetry of the basic crystal structure. Upon the ordering, vacancies are redistributed over the sites of the sublattices, the long-range order is formed in the structure, and the symmetry of the structure is reduced. These two different states lead to order-disorder phase transformations in crystal under some conditions and change their properties. The aim of present work is disordering of nonstoichiometric monoxides of IV-V groups, namely titanium and niobium monoxides which contain huge amount of structural vacancies on metal and nonmetal sublattices simultaneously. In order to determine the energies required for the displacement of atoms, i.e. threshold displacement energies, a systematic study of the disappearance of superstructure reflections with increasing electron energy and electron bombardment dose has been performed *in situ* in a transmission electron microscope (TEM).The specimens have been irradiated at room temperature in a Tecnai G^2 30 Twin (FEI Company) at accelerating voltages of 100, 120, 140, 150, 200 and 300 kV. The electron diffraction patterns have been recorded with a Gatan slow scan digital camera. The phase transformation of the ordered monoclinic Ti_5O_5 (sp.gr. $C2/m$) to disordered cubic phase TiO_y (sp.gr. $Fm\text{-}3m$) under electron beam irradiation have been observed. A threshold has been found between 120 and 140 keV. This incident electron energy interval corresponds to threshold displacement energy between 6.0

and 7.5 eV per Ti atom and is attributed to the displacements of titanium atoms. From the determined displacement threshold energies, titanium-oxygen bond energy about 1.5 eV is deduced for this compound. The superlattice of ordered niobium monoxide Nb_3O_3 is destroyed by irradiation with a low-energy electron beam. This effect was observed in NbO_y for the first time and is explained by the redistribution of atoms and vacancies in oxygen and niobium sublattices. It was found that irradiation by electron beam from 140 to 300 kV, disordering occurs, the duration of irradiation for disordering was from 30 minutes (at 300 kV) to 4 hours (at 140 kV). The order-disorder phase transformations in NbO_y from ordered Nb_3O_3 (sp.gr. *Pm*-3*m*) to disordered Nb_4O_4 (sp.gr. *Fm*-3*m*) structure was observed experimentally for the first time. This work is supported by the Russian Science Foundation (project No. 19-73-20012) at the Institute of Solid State Chemistry, UB RAS.

Keywords: Transition Metal Oxide, Nanocrystal, Nonstoichiometry, Crystal Structure, Phase Transition.

INVITED SPEAKERS

Id-533

In situ Photoemission Microscopy of Catalytic Surface Reactions

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Abstract: Scanning photoelectron microscopy (SPEM) and photoemission electron microscopy (PEEM) allow local surface analysis and visualising ongoing reactions on a µm-scale. These two spatio-temporal imaging methods are applied to polycrystalline Rh, representing a library of well-defined high-Miller-index surface structures. The combination of these techniques enables revealing the anisotropy of surface oxidation, as well as its effect on catalytic hydrogen oxidation. In the present work we observe, using locally-resolved SPEM, structure-sensitive surface oxide formation, which is summarised in an oxidation map and quantitatively explained by the novel step density (SDP) and step edge (SEP) parameters. In situ PEEM imaging of ongoing H_2 oxidation allows a direct comparison of the local reactivity of metallic and oxidised Rh surfaces for the very same different stepped surface structures, demonstrating the effect of Rh surface oxides. Employing the velocity of propagating reaction fronts as indicator of surface reactivity, we observe a high transient activity of Rh surface oxide in H_2 oxidation. The corresponding velocity map reveals the structure-dependence of such activity, representing a direct imaging of a structure-activity relation for plenty of well-defined surface structures within one sample. Valuable contributions by P. Winkler, J. Zeininger, M. Raab, S. Buhr, M. Stöger-Pollach, J. Bernardi, P. Zeller, M. Amati, L. Gregoratti and H. Grönbeck are gratefully acknowledged. Work supported by the Austrian Science Fund (FWF: P 32772-N and SFB TACO F81-P08).

Keywords: Catalysis, Surface Science, In Situ Microscopy.

INVITED SPEAKERS

Id-535

Microstructural Investigation of SnAgCu-TiO_2 Composite Solder Alloys

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Abstract: Transition to the lead-free solder materials resulted in the widespread use of the Sn96.5Ag3Cu0.5 (SAC305) and Sn95.5Ag4Cu0.5 (SAC405) solder alloys. These alloys have good soldering properties; however, they have two significant drawbacks due to the relatively high silver content: their higher price and possible reliability problems. If the silver content is over ~3 wt% in the solder alloy, a not inappropriate cooling might form large Ag_3Sn islands in the solder bulk, which could lead to shrinkage defect. Therefore, in the past 10 years, solder paste researches tried to reduce the silver content in the SAC solder alloys. A good alternative is the Sn99Ag0.3Cu0.7 (SAC0307) low-silver content solder alloy, which usually contains some further micro-alloyed (<0.1 wt%) metals (like Bi, Sb, Ni, etc). These metals can improve the wetting and mechanical behaviour, which properties might be decreased due to the lower silver content. With the micro-alloying elements, the abbreviation of the solder alloy is SACX0307. The most novel solution to improve the low-silver content solder alloys is the use of submicron- and nano-sized ceramic reinforcement particles, and this results in „composite" solder joints. A wide range of ceramic particles was already tried in SAC305 and SAC405 alloys, like TiO_2, ZrO_2, Al_2O_3, Fe_2O_3, Si_3Ni_4, SiC, La_2O_3, etc. Usually, the reinforcements increase the solder joints' quality and/or reliability, but in some cases, they increase the liquidus point as well. In the present study, the microstructure of SACX0307 nano-composite solder alloys were studied. The SACX0307 solder alloys were reinforced by 1wt% TiO_2 nano-particles. Primary particle sizes <50nm and <200nm) were used. The nanoparticles were mixed into the solder paste homogeneously using the ball milling process, which was carried out for 10 min at 300 rpm using a planetary ball mill. Solder joints were performed from the composite solder alloys and from the reference SACX0307 as well by regular reflow soldering technology on solder pads covered by ENIG surface finishes. The solder pastes were stencil printed and reflowed in an IR reflow oven with a linear thermal profile. Metallographic cross-sections were prepared from solder joints. The microstructural properties of the solder samples were investigated with optical microscopy, Scanning Electron Microscopy (SEM) and Scanning Ion Microscopy (SIM). Preliminary results showed that the nanoparticles slightly increased the melting point of the composite solder alloys (with 1-2K), The microstructural investigations showed considerable grain refinement and the modified grain boundary/interfacial properties. The nano-particles located at the β-Sn and Cu_6Sn_5 grain boundaries and they supressed the grain formation in liquide state of the solder alloy. More detailed conclusions will be included in the final paper.

Keywords: Reflow Soldering, SAC0307, Ceramics, Composite Solder Alloy, SEM.

INVITED SPEAKERS

Id-536

Drug Delivery into Single Cancer Cells and 3D Multicellular Constructs: Dynamic Microscopy and Spectral Analysis

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Abstract: The cell monolayer has been the traditional model for *in vitro* experimentation in cancer biology and therapeutics. However, cell monolayers cannot replicate essential aspects of tumor tissue such as the extracellular matrix, 3D cell-cell communication, uptake and transport of oxygen and nutrients in the capillary microenvironment, and drug penetration from blood capillaries into the surrounding tissue. To address these issues 3D multicellular constructs have been developed. These constructs better mimic tumor tissue and its *in vivo* response to therapy than monolayers do. However, the observed phenomena are the result of superposition of cell-bound processes, and multicellular processes determined by interactions of individual cancer and other cells and the ECM in 3D tissue. Therefore, besides 3D constructs, experimenting on single isolated cells is also necessary to obtain relevant information on therapeutics. Drug resistance is an unwanted response of tissue to therapy. Multidrug resistance is the term used for drug resistance of individual cells to treatment, and it is due to their genetic makeup which require experimentation on single cells positioned far from other cells. Multicellular resistance is superimposed on multidrug resistance due to the cancer cells being in a 3D tissue-like environment. Elucidating both types of resistance and their interactions requires experimentation on 3D multicellular constructs in addition to single cell studies. Monolayer based experiments have little value despite that the majority of studies have been, and still being performed on monolayers. The current mainstay of *in vitro* experimentation on cancer and therapy is still the 2D monolayer but 3D constructs are increasingly being adopted. Very little work, if any, is being done on single isolated cells, however. In our research program single cells and 3D multicellular preparations are both studied using an array of novel approaches. The results are compared with similar experiments on monolayers. In this presentation drug delivery into single cancer cells and efflux of drug molecules from those cells will be discussed, using dynamic epifluorescence and confocal microscopy as well as micro-electrochemistry in tandem. Additionally, a new spectrophotometry technique that we call

temporal ratiometry will be introduced that we used to asses dynamic drug distribution in single cells during drug delivery. Further, oxygen metabolism and drug uptake in 3D multicellular spheroid preparations will be analyzed using dynamic microscopy as well as electrochemical sensing. Thus both cell-bound and multicellular processes are characterized using an array of novel techniques. The significance of this combined approach is that for the first time both levels of hierarchy – single cells and their 3D assembly – are addressed.

Keywords: Cancer Therapeutics, Single Cells, 3D Multicellular Spheroids, Dynamic Spectral Microscopy, Microsensors.

INVITED SPEAKERS

Id-539

Imaging of Electric and Magnetic Near Fields of Plasmonic Antennas by EELS

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Abstract: Localized surface plasmon resonances are self-sustained collective oscillations of free electrons in metallic nanostructures. Mapping of localized surface plasmon modes with high spatial and energy resolution is necessary to understand their nature and spatio-spectral characteristics. This can be done by scanning transmission electron microscopy (STEM) combined with electron energy loss spectroscopy (EELS) which measures the energy transferred from electrons to the LSP. Importantly, EELS is sensitive to the electric near field component of localized surface plasmon modes parallel with the electron beam and cannot be directly associated with the magnetic near field. In our contribution, we use the Babinet's principle to facilitate characterization of both the electric and the magnetic near field of plasmonic antennas. Babinet's principle relates the properties of a planar plasmonic antenna (particle) and a complementary plasmonic antenna (aperture) in a thin metal film of the same size and shape. In particular, the energies of LSPR in both types of antennas shall be identical and the magnetic near-field distribution of the particle antenna shall correspond to the electric near field distribution of the complementary aperture antenna. The spatially resolved EELS mapping of the aperture antennas can thus help to deduce the magnetic near fields associated with the localized surface plasmon resonances in particle antennas and vice versa [1]. We demonstrate such use of Babinet's principle in a combined experimental and theoretical study for disc-shaped antennas/apertures and plasmonic bow-tie and diabolo antennas/apertures with electric/magnetic hot spots. Moreover, we will also discuss optimal experimental conditions in STEM-EELS for achieving the best signal-to-background ratio as well as suitable techniques for fabrication of high-quality plasmonic nanostructures.

Keywords: Localized Surface Plasmons, EELS, Babinet's Principle.

INVITED SPEAKERS

Id-541

Localized X-Ray Photoelectron Impedance Spectroscopy for Liquid/Solid Interfaces

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Abstract: Supply and Storage of Energy is at the center of current and future scientific and technological activities for which the batteries are still on top of the list. But, batteries are slow, since they involve sluggish chemical reactions. On the other hand, Electrical-Double Layer Capacitors, or Super-Capacitors, have recently been proposed as an alternative, since they involve transport of ions at liquid/solid interfaces. Ion Transport Dynamics is the key issue for controlling performance of these devices and need to be investigated during their operation (In-Operando). XPS is one of the very informative surface analyses techniques, which can deliver chemical as well as electrical properties of systems. However, the technique requires Ultra-High-Vacuum environment, unsuitable for volatile liquids like water, but there are also several non-volatile liquids, like ionic liquids, which are also excellent electrolytes for battery and super-capacitor applications. Emergence of Ionic Liquids with several promising properties, including their low volatility, has rekindled the use of XPS. Our recent efforts had also concentrated on ionic-liquids and their interfaces under **dc** and **ac** bias, and extended to monitoring electrochemical reactions, as well. The common theme in our studies is the use of the bias dependent binding energy shifts of the atomic core-levels as reflection of the electrical potentials, recorded in a non-invasive and chemically resolved fashion. We use the magnitude and the frequency dependence of such potentials to extract pertinent information related to **static**, as well as **dynamic** chemical and/or electrochemical properties of the materials and their interfaces, configured as devices, with particular emphases on the ionic liquids' certain chemical/physical parameters, like steric effects, ion size, diffusivity, etc. Examples using ionic liquids, liquid poly-ethylene-glycol (PEG) and salts, as well as their mixtures,

as electrolytes, with special emphases on energy storage systems, will be presented and discussed.

Keywords: In-Situ and In-Operando XPS, Localized Potential Developments, Liquid/Solid Interfaces, Ionic Liquids.

INVITED SPEAKERS

Id-547

Mössbauer Spectroscopy in Studies of Thin Films and Multilayers

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Abstract: The phenomenon of recoil-free γ-ray resonant absorption and emission discovered by Rudolf Mössabuer in 1958 developed rapidly to a spectroscopic technique applied in many fields such as physics, chemistry, material science, mineralogy, metallurgy and so on. This powerful nuclear analytical method provides information about the magnetic, electronic and lattice properties of the solids and is used to study the local electronic, chemical and crystallographic nucleus surroundings, which are described by the hyperfine interactions between the nuclear charge distribution and the extranuclear electric and magnetic fields. These numerous applications of Mössbauer spectroscopy in various scientific disciplines result primarily from the fact that the most popular nuclide used in this method is the iron isotope ^{57}Fe. Iron is an element that is part of many biological and environmental samples as well as artificial samples such as multilayers and other nanostructures. The conversion electron Mössbauer spectroscopy (CEMS) technique is a surface-sensitive method that offers a unique possibility to analyze, at the atomic scale, the buried interfaces in multilayer systems without the sample destroying. The interface roughness affects the magnetotransport properties, especially the giant magnetoresistance (GMR) effect of multilayers which determines their applications. The observed literature discrepancies concerning the influence of interface roughness on GMR could be explained thanks to the CEMS technique, which gives a possibility to distinguish two kinds of interface roughness. The roughness caused by the interdiffusion of Fe atoms into the Cr layer leads to the atom intermixing at the interface. On the other hand, the interface corrugation is simply the roughness of the surface on which the deposition occurs. We found that these two kinds of interfaces influence GMR in opposite directions. The interface roughness caused by intermixing generate point defects which are known as spin-independent scattering centers of electrons and reduce the GMR effect, while the corrugation develops the interface areas, which are spin-dependent scattering centers in Fe/Cr systems and increase GMR. The introduction of the ^{57}Fe Mössbauer probe in selected areas of multilayers made it possible to study the chosen interfaces and indicated the asymmetry in the interface roughness between Fe-on-Cr and Cr-on-Fe interfaces. The CEMS measurements were also used to analyze the influence of the swift heavy ion irradiation on the Fe/Cr multilayers structure and revealed an increase of the interface roughness at high irradiation fluences. The transmission geometry measurements of environmental samples and nanostructures enabled the magnetic phase composition to be determined. Mössbauer spectroscopy is a versatile technique that can be used in many fields of science and industrial applications.

Keywords: Mössbauer Spectroscopy, İnterface Roughness, Multilayers.

INVITED SPEAKERS

Id-548

Features of Microstructure and Composition of 18th Century Dutch Tiles Revealed by SEM, XRF, XPS, and TOF-SIMS

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Abstract: The ceramics restoration workshop of the Department of Restoration and Examination of Cultural Heritage Objects of the St. Petersburg State Institute of Culture received 22 partially fragmented faience tiles from a private collection. The tiles are made of ceramic dough of light yellow color with a fine texture, covered with a white glaze (enamel) and painted in an underglaze technique. Five of them have an ornamental painting in brown pigment, eight ones have an ornamental painting in blue, the ornament in both cases is floral. Nine tiles painted with blue pigment are decorated with a plot painting in a circle with corner ornamental filling, three of them contain image of a ship, six ones have a landscape painting including images of buildings and people. Visually identifiable technical-technological and stylistic features and the presence of analogs are providing basis to identify these tiles as Dutch products of the 18th century. This study had two aims: to verify the visual attribution comparing composition of tiles with literature data obtained from analogous tiles with known provenance and to find new features in their composition and microstructure. Several fragments of the tiles with blue and brown paintings were selected for instrumental studies. The samples dimensions were less than 20x20 mm. The paper presents results on microstructure of the tiles obtained by SEM and composition measurements fulfilled by X-ray fluorescence (XRF) and X-ray photoelectron spectroscopy (XPS), time-of-flight mass-spectroscopy (TOF-SIMS) methods in comparison with literature data describing features of microstructure and elemental composition of attributed Dutch tiles fabricated in Amsterdam, Rotterdam, Utrecht, and Harlinger. There was found that investigated samples can be attributed as Dutch ones fabricated in the beginning of 18th century.

The information on microstructure and composition of glaze layers including pigments and ceramic base of the tiles obtained in this research will be used in further conservation efforts.

The research was supported by RSF grant No. 21-19-00412.

Keywords: Faience Tiles, the Netherlands, 18th century, XRF, SEM, XPS, TOF-SIMS.

INVITED SPEAKERS

Id-549

Characterisation of Crystalline Microstructure in Polycrystalline Materials by Analysing Powder X-ray Diffraction Patterns

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Abstract: X-ray diffraction is a principal phenomenon exploited to revealing microscopic properties of crystalline materials (and nanomaterials), particularly to characterising the crystalline microstructure. An adequate mathematical model of a material, an accurate description of a corresponding X-ray diffraction pattern and a stable method for solving an ill-posed inverse problem involved are necessary for determining microstructural parameters of a polycrystal reliably from experimental X-ray diffraction data. An effective procedure for determining microstructural characteristics of polycrystalline materials has been developed recently in subsequent works, based on ideas proposed earlier by founders of X-ray crystallography. The method is based on description (in the frame of kinematical theory of X-ray diffraction) of X-ray diffraction line profiles with accounting for both instrumental and microstructural contribution with material characteristics as parameters to be refined from experimental data. The crystallites are modelled as simple solids like prisms, cylinders or spheres with corresponding free shape parameters. The instrumental function for computing standard line profiles is determined from X-ray diffraction pattern from a standard reference material. The computational algorithm exploits simultaneously several line profiles extracted from powder X-ray diffraction pattern for a specimen interpreted as a set of randomly oriented crystallites. The method was verified to be useful for investigating materials with crystallite sizes spanned between several and thousands of nanometers. Quantitative characteristics like a prevalent crystallite shape, a volume-weighted crystallite size distribution and a second-order crystalline lattice strain distribution (together with derivative parameters as mean standardised crystallite size, mean-absolute second-order strain and standard deviations) were computed for several polycrystalline materials by using this method. These microstructural characteristics enabled unique insight into formation of polycrystalline phases in processes of crystallite nucleation and growth. Particularly, observations of bimodality of crystallite size distribution

evidenced traces of concurrent processes of crystallisation in resulting microstructure of obtained materials. The investigated specimens were examined also by other techniques, like electron scanning and transmission microscopy, calorimetry or measurements of specific surface, to complement and confirm results of X-ray diffraction analysis. In the lecture the procedure will be concisely described with short explanation of principal steps. An example of the microstructure of boehmite will be presented and discussed briefly, to illustrating both robustness of the method and characterisation of a real material. The boehmite nanoparticles were prepared in a procedure comprising the formation of a precipitate and subsequent hydrothermal aging with possible application of an additive. The obtained specimens were investigated by using X-ray diffraction and electron microscopy. The nanoparticles of boehmites were found to be monocrystalline grains with characteristic habits and sizes of order of ten nanometers and with bimodal logarithmic-normal size distributions. Based on the computed characteristics, three types of the microstructure correlated with the production procedures were observed. This last work is common with Pablo Pardo, José Miguel Calatayud, José María Amigó and Javier Alarcón who realised especially the experimental part.

Keywords: Powder X-ray Diffraction, Crystalline Microstructure.

INVITED SPEAKERS

Id-552

Study of the Polymorphism of an Organogel: Nanotube to Crystallites Transition

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Abstract: Low molecular weight gelators are molecules able to gel organic solvents at low concentration, by forming high aspect ratio aggregates.[1,2] Most of the research on these gelators has been underpinned by their potential applications in different fields such as biomedical field and organic electronics. Despite this active work, many fundamental aspects remain poorly understood, especially in the thermodynamics of gelation, the stability or metastability of the formed self-assemblies. Moreover, the structure of the gels may depend on supersaturation or cooling rate.[3–5] As shown very recently, some organogels can exhibit gel-to-gel transitions,[6] showing a polymorphism in organogels. Although in these examples the phase diagrams were not mapped, they must be impacted by these transitions and should appear more complex than commonly granted with only two domains: the sol and gel phase. In this paper we study the gels formed in alkanes by a simple monoamide, HUB-4.[7] The *c*-*T* phase diagram of HUB-4/*trans*-decalin was mapped by rheology, turbidimetry and microDSC experiments. The structures of both polymorphs were studied by different electron microscopic techniques. Freeze-fracture TEM in organic solvent[8] allowed the observation of the aggregates in their native form. CryoSEM after sublimation of the solvent was implemented to follow the transition between both polymorphs and to observe their spatial distribution and confirm phase separation. As shown by these microscopic studies, HUB-4 form nanotubes at low temperature and transits to crystalline fibers either by annealing or by ageing. The structure of both morphs was studied at lower scale by SAXS and WAXS and afforded a model of the packing in the nanotubes. The intermolecular interactions, especially the H-bonds, were followed by variable temperature FTIR to identify the changes accompanying the transitions.

Keywords: Organogels, Freeze Fracture TEM, CryoSEM, Phase Diagrams, SAXS.

REGULAR SESSIONS

Id-532

X-Ray Microscope with High Spatial Resolution (of ≥ 2 microns) and Time Resolution of 1 ns

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Abstract: X-rays allow us to obtain images of objects that are opaque to normal radiation. To achieve an image of high contrast the X-ray should be noticeably absorbed in an object. A projection method is generally used to achieve an enlarged image without lenses. In this method a source of radiation should be small enough to achieve a high spatial resolution. All this set of properties is offered by an X-ray source called X-pinch. This X-ray source of no more than 1 micron size was created in seventies of the last century [1]. X-ray emission duration of this source does not exceed 1 ns, and the range of emitted quanta lies within 1-20 keV. X-pinch was actively used for researches of fast processes in large physics installations [2]. Using an experience gained with large installations JSC "SRC RF TRINITI" has created a new automated X-ray apparatus (MIX-1), based upon X-pinch and purposed for researches in medical and biological field. Unlike previous developments this apparatus does not require x-ray protection and can be operated by non-engineer personnel. Its small size and wheeled trolley allow it to be placed even in a small room. This device provides an automated system of X-pinch replacement that allows us to make series of shots with intervals of several minutes without damaging an observed object. The object’s image immediately transfers to a computer that is a part of MIX-1. MIX-1 is fully controlled by the computer. Contrast images with the resolution of 1 micron are obtained for objects of less than 2 mm thick. The thickness is less for most metals and materials with high atomic number.

Keywords: X-Ray Microscope, X-Pinch, Live Objects, Intact Objects, Enthomology.

REGULAR SESSIONS

Id-553

EBSD Analysis Application to Study Evolution of Deformation Structures in Materials Science

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Abstract: The structural evolution under large plastic deformations has been the subject of many studies in recent decades. The main incentive is the creation of ultrafine-grained materials, the structure of which is formed as a result of fragmentation, that is the subdivision of initial crystallites into disoriented microvolumes, the misorientations of which increase with the deformation degree increasing. In the present report, some examples of the deformation structure evolution was considered, studied using EBSD technique after different types of deformation treatment of metals: in aluminum specimens after dynamic channel-angular pressing (DCAP), at different distances from the contact surface during explosive welding of aluminum plates, and also during necking of iron tensile specimen. The aluminum microstructure after high-speed deformation by the method of DCAP after one and four passes was investigated. Deformation twinning in coarse-grained aluminum was shown for the first time under conditions of high-rate deformation during DCAP. An analysis of the misorientation deviation from the ideal twin boundary showed that the deformation twins formation can occur both at the early stages of DCAP and at later stages. A detailed analysis of misorientations and deformation structures of plastic flow vortex-like instabilities in the contact zone of the aluminum-aluminum joint obtained by explosive welding has been carried out. At different points of the vortex, the rate of deformation is different: the lowest at the periphery and sharply increasing as it approaches the center of the vortex. In the vortex of the aluminum-aluminum welded joint, six areas of the evolutionary development of deformation structures were identified. The features of the microstructure fragmentation in the neck of iron tensile specimen was also studied. Special attention was paid to deformation-induced high angle boundaries, which are preferable sites of microcrack origination. It was shown that at true strains of the order of 1, the main contribution to the formation of such boundaries is made by shear microbands. The width of these bands is of the order of 1 μm or less, down to 0.1 μm, and the misorientations at their boundaries vary from ~ 10 to 45 degree.

Keywords: EBSD Analysis, Materials Science, Deformation Structures.

POSTER SESSIONS

Id-540

X-Ray Apparatus with Spatial Resolution of ≥ 2 Microns and Time Resolution of 1 ns.

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Abstract: In the report we present an apparatus for radiography of live and intact biological objects of millimeter size with X-ray emission with a spatial resolution of 1-2 microns. X-rays allow us to obtain images of objects that are opaque to normal radiation. To achieve an image of high contrast the X-ray should be noticeably absorbed in an object. A projection method is generally used to achieve an enlarged image without lenses. In this method a source of radiation should be small enough to achieve a high spatial resolution. All this set of properties is offered by an X-ray source called X-pinch. This X-ray source of no more than 1 micron size was created in seventies of the last century. X-ray emission duration of this source does not exceed 1 ns, and the X-ray energy range lies between 1 and 20 keV. X-pinch was actively used for researches of fast processes in large physics installations. Using an experience gained with large installations JSC "SRC RF TRINITI" has created a new automated X-ray apparatus (MIX-1), based upon X-pinch and purposed for researches in medical and biological field. Unlike previous developments this apparatus does not require x-ray protection and can be operated by non-engineer personnel. Its small size and wheeled trolley allow it to be placed even in a small room. This device provides an automated system of X-pinch replacement that allows us to make series of shots with intervals of several minutes without damaging an observed object. The object's image immediately transfers to a computer that is a part of MIX-1. MIX-1 is fully controlled by the computer. Contrast images with the resolution of 1 micron are obtained for objects of less than 2 mm thick. The thickness is less for most metals and materials with high atomic number.

Keywords: X-Ray Microscope, X-Pinch, Live Objects, Intact Objects, Enthomology.

POSTER SESSIONS

Id-550

The Study of pH and Aging Time Influence on Waste Derived-MCM-41 Mesoporous Silica Material Properties by Microscopic and Spectroscopic Analysis

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Abstract: In 1992, Mobil company for the first time synthesized the mesoporous silica material MCM-41 (Mobil Composition of Matter No. 41). Since then, due to its large specific surface, excellent adsorption properties and hexagonal ordered structure, it have become a very interesting material for many scientists. Initially, MCM-41 was obtained from pure chemical reagents consisting primarily of a silicon source (most often tetraethyl orthosilicate - TEOS) and a surfactant (hexadecyltrimethylammonium bromide - HDTMABr) used as a matrix. This method turned out to be not very popular in the context of high-scale production because of chemicals prices. Hence, there were more and more attempts to synthesize this material from waste, implementing the principle of sustainable development through the use of waste and reduction of process costs. Additionally, literature data confirms that the structure of this type of material can be preserved using higher temperatures or synthesis times. The aim of this study was to investigate the effect of not only the aging time, but also the pH value during the synthesis on the properties of the obtained MCM-41 using microscopic and spectroscopic methods. Mesoporous silica was synthesized from a waste solution (as a silicon source) obtained as a result of the production of zeolites from fly ash on a semi-technical technological line and HDTMABr (as a matrix). The MCM-41 materials after synthesis were subjected to the aging process for 24 and 48 hours (with control sample without aging). Additionally, each of the mesoporous silicas were obtained in different pH conditions - 9, 10 and 11. All materials were tested using the XRD, FT-IR, SEM/TEM methods and for texture parameters the nitrogen adsorption/desorption isotherms at liquid nitrogen temperature were prepared. The results showed that with the increase of the pH during the synthesis, the specific surface area of MCM-41 increased (from 1232 up to 1410 m^2/g) and the pore diameter decreased (from 3.7 up to 3.5

nm). Moreover, increasing the aging time had the opposite effect. This was also confirmed by microscopic analyses. Summing up, the tested parameters (aging time, synthesis pH) significantly influenced, in particular, the specific surface area and pore diameter. Such knowledge may have a strong influence on the application direction of this material, which will be the subject of further work. This research was financed within the National Centre for Research and Development project No. LIDER/19/0072/L-9/17/NCBR/2018.

Keywords: MCM-41, Waste Solution, Microscopy, Spectroscopy, Porosity.

POSTER SESSIONS

Id-551

Sorption Potential towards CO_2 and Microscopic Analysis of Na-X and Na-A Zeolites Obtained from Waste

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Abstract: The progressing technological progress and the fossil fuels combustion are still very dangerous to the environment, mainly due to the CO_2 emission and other greenhouse gases. The latest climate change IPCC report estimated that human activity caused an increase in global average temperature of around 1˚C compared to pre-industrial periods. Maintaining such a pace of changes will increase this value to 1.5˚C already between 2032 and 2050. The key is to reduce greenhouse gas emissions as soon as possible. In order to halt global warming, emissions of these gases must be reduced by 50% by 2030 and 100% by 2050. This means that many developed countries (including Poland) must quit burning coal by 2030 at the latest, and the rest - by 2040. Various types of sorbents, including zeolites, are excellent tools for the capture and adsorption of CO_2. They are natural or synthetic, microporous spatial aluminosilicates with a crystal structure containing free spaces (channels and chambers) with a size of several nm. Depending on the created form, they may differ in shape, number and size of channels. Zeolites can be divided into natural and synthetic. Nowadays, zeolites are often obtained from various types of waste - including fly ash which reduced their final price. The aim of this study was the physicochemical analysis of zeolites with different structures in terms of their sorption potential in relation to CO_2. For this purpose, Na-X (faujasite type) and Na-A (Linde A type) zeolites were synthesized from the waste solution obtained after the production of zeolites directly from fly ash. In order to approach the conditions used in the industrial use of this type of material, the zeolites were granulated by adding 15% bentonite as a binder. Granulated forms of zeolites were analysed by microscopic and spectroscopic methods. In addition, basic textural parameters were determined as a result of nitrogen adsorption/desorption isotherm at liquid nitrogen temperature analysis. In order to estimate the sorption potential towards CO_2, sorption tests were carried out at the 0°C. The results showed that zeolites are good CO_2 sorbents, which resulted, inter alia, from their interesting textural parameters and, as confirmed by microscopic analyzes, their ordered structure. Zeolite Na-X

has greater adsorption capacity comparing to Na-A both in powder (300 compare to 213 mg/g) and granulated form (184 compare to 169 mg/g). It is worth mentioning that granulation process caused a decrease of CO_2 sorption capacity of studied zeolites - 38.77% for Na-X and 20.50% for Na-A. Summing up, the studied zeolites can become an excellent material that allows to significantly reduce CO_2 emissions to the atmosphere without being an additional burden for the environment as they were obtained from waste. This is in line with the principle of sustainable development and the circular economy. This research was financed within the National Centre for Research and Development project No. LIDER/19/0072/L-9/17/NCBR/2018.

Keywords: Synthetic Zeolites, Waste Solution, Microscopy, CO_2 Sorption, Porosity.

ALL SUBMISSIONS & TOPICS

Topic	Submission
Applications of Microscopy &Spectroscopy in the Biological Sciences	536 - Drug Delivery into Single Cancer Cells and 3D Multicellular Constructs: Dynamic Microscopy and Spectral Analysis
	541 - Localized X-Ray Photoelectron Impedance Spectroscopy for Liquid/Solid Interfaces
Biomedical Applications	529 - Research&Development of Inorganic Nanoparticles for Biomedical Applications on the Example of Metal Sulfides and Metal Oxides
Applications of Microscopy & Spectroscopy in the Physical/Chemical Sciences, at All Dimensional Scales	539 - Imaging of Electric and Magnetic Near Fields of Plasmonic Antennas by EELS
	552 - Study of the Polymorphism of An Organogel: Nanotube to Crystallites Transition.
Analysis of Cultural Heritage/Ancient Materials	548 - Features of Microstructure and Compostiion of 18th Century Dutch Tiles Revealed by SEM, XRF, XPS, and TOF-SIMS
Composites	535 - Microstructural Investigation of Snagcu-TiO_2 Composite Solder Alloys
Metals	553 - EBSD Analysis Application to Study Evolution of Deformation Structures in Materials Science
Nanophases, Nano-Structures, Clusters and Catalysts	533 - In Situ Photoemission Microscopy of Catalytic Surface Reactions
Novel Functional Materials	550 - The Study of Ph and Aging Time Influence on Waste Derived-MCM-41 Mesoporous Silica Material Properties by Microscopic and Spectroscopic Analysis

	551 - Sorption Potential Towards CO_2 and Microscopic Analysis of Na-X and Na-A Zeolites Obtained from Waste
Phase Transformation in Metals, Alloys and Ceramics	530 - In Situ Disordering of Nonstoichiometric Monoxides of IV-V Groups by Means of Transmission Electron Microscope
Diffraction Techniques	549 - Characterisation of Crystalline Microstructure in Polycrystalline Materials by Analysing Powder X-Ray Diffraction Patterns
X-Ray Microscopy	532 - X-Ray Microscope with High Spatial Resolution (of ≥ 2 Microns) and Time Resolution of 1 Ns
	540 - X-Ray Apparatus with Spatial Resolution of ≥ 2 Microns and Time Resolution of 1 Ns.
Mössbauer Spectroscopy	547 - Mössbauer Spectroscopy in Studies of Thin Films and Multilayers
X-Ray Absorption Spectroscopy	471 - X-Ray Absorption Near Edge Spectroscopy Used to Determine Local Atomic Structure of Ions in Solution. The Case of as in Water