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Session III

SL12

THERMAL EVOLUTION AND MICROSTRUCTURE OF GAS AGGREGATION CLUSTER SOURCE PRODUCED METAL NANOPARTICLES

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Nanoparticles belong to modern, progressive materials with wide application potential. Its interesting properties come from the high surface to volume ratio and often differ from the properties of bulk or coarse-grained materials with the same chemical composition. Nanoparticles catalytic, magnetic, electric or optical properties are just examples. In this study we investigated niobium nanoparticles prepared by magnetron sputtering in combination with gas aggregation cluster source operated in DC mode in argon atmosphere.

Studied nanoparticles deposited on silicon substrates were characterized by combination of X-ray scattering and diffraction methods and transmission and scanning electron microscopies with energy-dispersive X-ray spectroscopy. These analytical methods provide the information about the nanoparticles sizes, shapes, their distributions, chemical composition and the real atomic structure. In order to determine the thermal evolution of nanoparticles microstructure, the in situ high temperature XRD measurement was done in ambient air atmosphere up to 800 C. Measured SAXS data for temperatures in range between 50 C – 800 C are shown in Figure 1. Two main transition are observed and described.

Thin oxygen layer is present on the surface of as prepared niobium nanoparticles, which acts as a protection barrier against further corrosion. The thickness of this amorphous oxide layer, as determined by fitting the SAXS patterns, is about 0.9 nm. The nanoparticle core is formed by pure niobium bcc phase and its mean radius is 11 nm. With increasing temperature, the growth of the oxide shell layer at the expense of the size of nanoparticle metallic core occurs. Slightly above 200 C the nanoparticles are fully

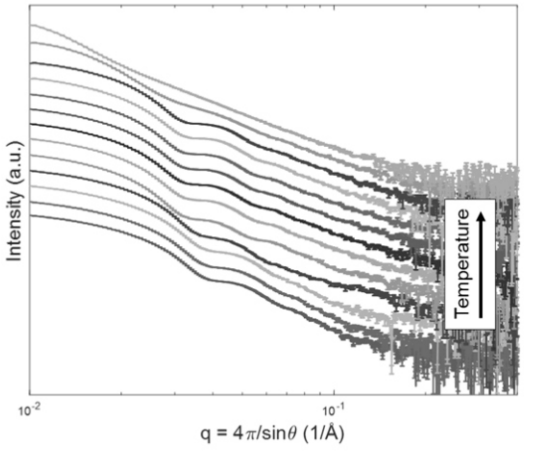

Figure 1. The SAXS patterns measured during annealing of nanoparticles from 50 °C to 750 °C, at ambient air atmosphere.

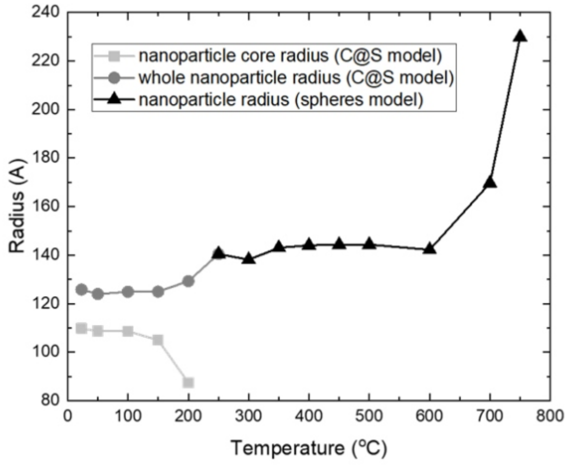

Figure 2. Temperature dependence of nanoparticle sizes, two different models were used to fit the measured data: core@shell model up to 200 °C, above 200 °C spheres model.

amorphized which is clear from XRD patterns. In this temperature range the mean nanoparticle size remains almost same. The results of SAXS measurement are plotted in Figure 2. Crystalline phase of niobium pentoxide is formed at temperature around 450 C. Additionally, we observe one more phase transformation at 625 C after which also pronounced increase of nanoparticles sizes starts. Based on the combination of SAXS and XRD measurement we conclude that the size of original niobium nanoparticles corresponds to the mean crystallite size, i.e. it means one nanoparticle consists of one coherently diffracting domain. At the end of the annealing process, after heating to 800 C and formation of the orthorhombic niobium pentoxide phase, there are more crystallites in one particle.

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SL13

STRUCTURAL ANALYSIS OF CLAY MINERALS INTERCALATED WITH DRUGS SOLVED BY CLASSICAL MOLECULAR DYNAMICS AND DFT METHODS

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We report comparison of results of computational approach to physico-chemical description of clay minerals-based drug nanocarriers. Two different types of layered clay structures were described. The first one was Mg_2Al layered double hydroxide (LDH) intercalated with three drugs – mefenamic acid, sulindac and pravastatin. These drugs have anti-inflammatory and ant nociceptive effects (anti conduction of painful stimuli) and clay drug nanocarrier promotes its stability and sustained release process. The series of models with different amount and initial arrangement of intercalated drug were built and analysed in order to compare the calculation results with experimental data published in [1-3]. The second clay mineral was Halloysite intercalated with irinotecan drug. This mineral posses spiral shape nanotube morphology with different charge distribution on inner and outer surfaces. The results for different pH conditions will be presented [4].

DFT calculations for small crystal cell were carried out in QuantumEspresso using PBE exchange-correlation functional and MD calculations in Materials Studio software package using combination of two compatible forcefields CVFF and ClayFF. Comparison of calculated XRD patterns, basal spacings, concentration profiles, free volumes and mean square displacements will be presented for LDH supercell with total surface area of 400 Å2. DFT calculation were used for studying of interactions between LDH surface and drug. The whole complex structure was analysed by MD calculation on ns time scale. Results for modified forcefields in comparison with DFT substructure calculation and experimental results will be presented.

The energetically preferred bilayer or pseudo bilayer arrangement of intercalated drugs will be discussed in order to describe differences in behaviour for individual drugs and its impact on sustained release process.

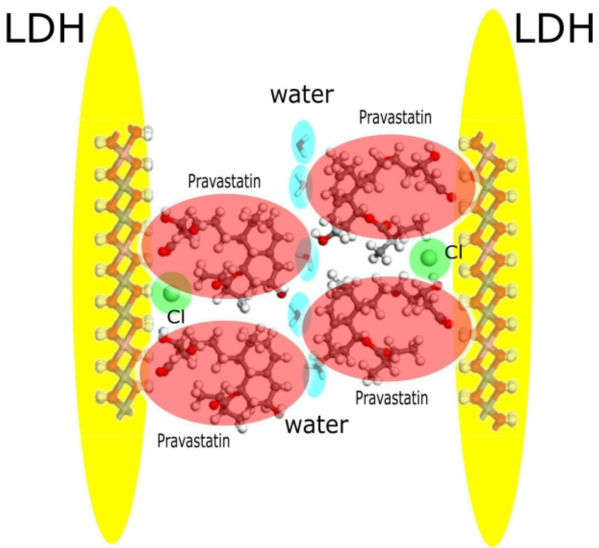

Figure 1. Preferred bilayer arrangement of Pravastatin anions in the interlayer space of LDH.

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SL14

STUDY OF ARRANGEMENT OF INTERCALATED ORGANIC SPECIES WITHIN LAYERED MATERIALS BY MOLECULAR SIMULATION METHODS

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We used classical molecular simulations to study the arrangements of various intercalated organic species (i) optically active 4-4’-dipyridylamine derivatives [1] within the layers of zirconium 4-sulfophenylphosphonates (**ZrSPhP**) and (ii) molecules of anti-inflammatory drugs within the layers of Mg_2Al layered double hydroxides (LDH). We present the arrangement of the intercalated molecules of 3-methoxo-N-(pyridin-4-yl)pyridin-4-amine (**moAPY2**, see Fig. 1) between the layers of **ZrSPhP** and mefenamic acid (see Fig. 2) intercalated within the layers of LDH. Their mutual positions and various orientations were calculated by the molecular simulation methods with the respect to the lowest value of the total potential energy.

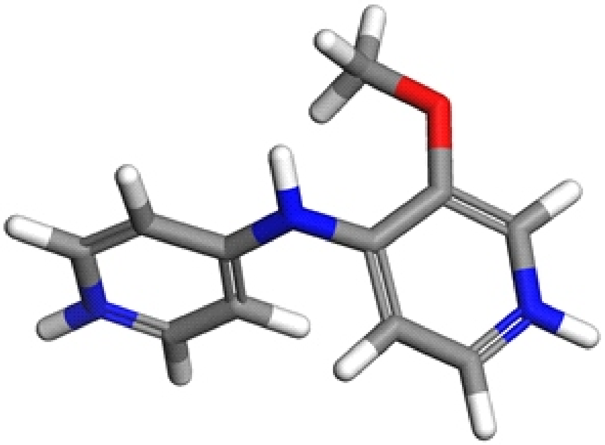

Figure 1. Molecule of 3 methoxo-N-(pyridin-4-yl)pyridin-4-amine (**moAPY2**).

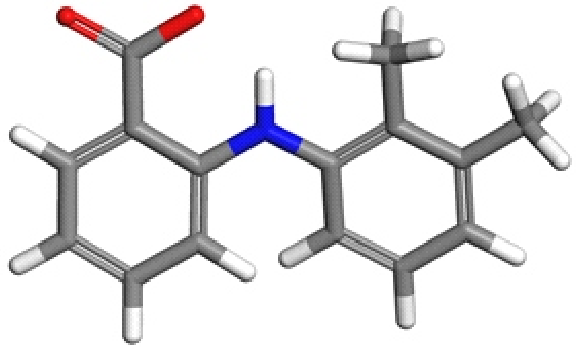

Figure 2. Molecule of mefenamic acid.

For the case of **ZrSPhP**, the simulations revealed that the alternating orientation of the intercalated **moAPY2** molecules is preferred. The best calculated model is shown in the Fig. 3. The intercalate is characterized by disordered arrangement where at several places the intercalated molecules even trench on the sulfo groups’ region. In all initial models, the intercalated molecules were arranged in rows in each interlayer, variously perpendicularly or parallel to the cell axis *a*. The calculation showed that one orientation of the rows is not favoured as the best model includes both types of those arrangements in two interlayers. The interca-

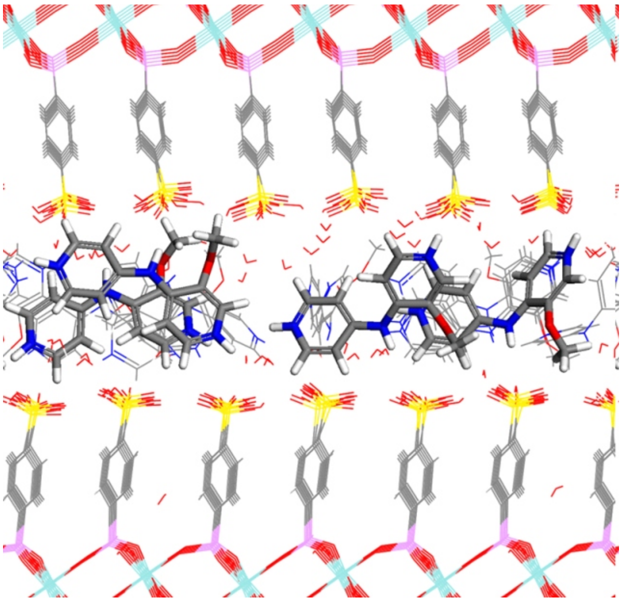

Figure 3. Optimized arrangement of the intercalated **ZrSPhP** with **moAPY2** molecules, side view.

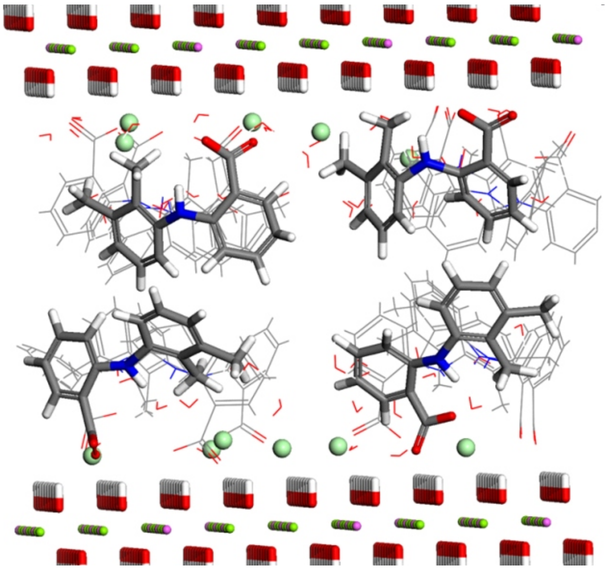

Figure 4. Optimized arrangement of the intercalated layered double hydroxides with molecules of mefenamic acid.

lated molecules are stacked together in the optimized arrangement by the interactions between the pyridine rings.

Regarding the intercalation of LDH, molecules of mefenamic acid as drug with anti-inflammatory effect were inserted between the layers of Mg_2Al LDH together with chlorine anions, in accordance to the chemical analysis [2]. From different types of arrangement of the intercalated molecules, the lowest value of potential energy and the best agreement with experimental XRD pattern [2] was reached for the bilayer arrangement, shown in Fig. 4. Due to the electrostatic interactions, the chlorine anions are closely attached to the layers of positively charged hydroxides and the COO^- functional groups of mefenamic acid molecules are turned to the LDH layers.

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SL15

BOUND STATE OF CRYSTAL ELECTRIC FIELD EXCITATION AND A PHONON IN $CePd_2Al_2$

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The existence of a bound state (vibron state) between 4f electrons and phonons is closely associated with $CeAl_2$ [1] compound. The magnetic excitations observed by neutron scattering were not possible to be described by standard crystal electric field theory using cubic symmetry for Ce surrounding. Also local static distortion on Ce site was excluded. The observed magnetic excitation was finally described by the Thalmeier-Fulde model of vibron state [2]. This model explains the observed magnetic excitations and also gives a prediction which phonon mode should be involved in the bound state (in case of $CeAl_2$ it is $_{15}$). This phonon mode is then influenced by the vibron state – the phonon mode softens and broadens [1].

For a long time $CeAl_2$ was a unique compound in which this enhanced interaction was observed. The increasing interest in recent time is motivated by new tetragonal materials which exhibit similar behaviour of magnetic excitations: $CePd_2Al_2$ [3], $CeCuAl_2$ [4] and $CeAuAl_3$ [5]. But the study of phonon dispersion curves and the influence of the vibron state is still missing.

Our interest is focused on $CePd_2Al_2$ and non-magnetic analogue $LaPd_2Al_2$ which crystalize in tetragonal $CeBe_2Ge_2$ structure type and undergo a structural phase transition decreasing their symmetry in the basal plane. The resulting structure can be then described within the orthorhombic *Cmme* space group. We succeeded with preparation of single crystalline $(Ce,La)Pd_2Al_2$ samples

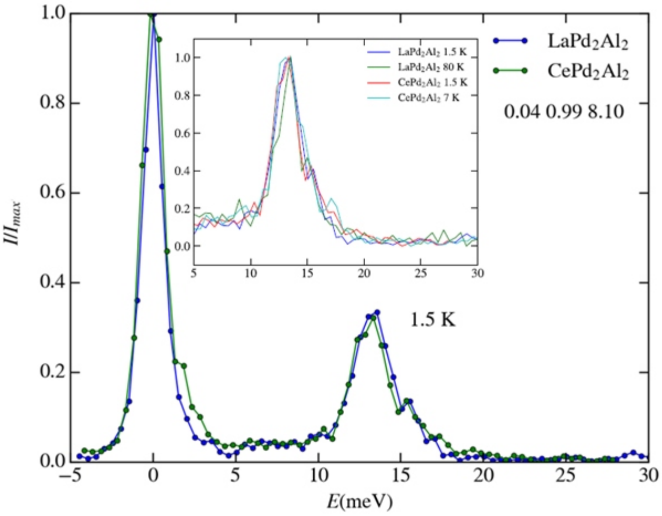

Figure 1. Comparison of A_{1g} phonon mode of $CePd_2Al_2$ and $LaPd_2Al_2$ at 1.5. K.

and investigated their phonon dispersion curves, mainly the temperature dependence of A_{1g} phonon mode, see Fig. 1. The results will be discussed in terms of enhanced magneto-elastic interaction which is responsible for formation of the vibron state.

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25TH GENERAL ASSEMBLY AND CONGRESS OF THE INTERNATIONAL UNION OF CRYSTALLOGRAPHY

Workshop Current Trends and Future of Crystallography Meeting of the International Programme Committee

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It is a tradition of the IUCr congresses that the congress programme is put together during three day meeting of International Programme Committee (IPC), usually close to the congress venue. Such a face-to-face meeting can be very efficient with respect to sometimes endless e-mail discussions.

During discussions on the form of the meeting, we took into account that this will be anniversary the 25th congress and therefore it would be worthwile to prepare some overviews what is going on in current crystallography and since the work of the IUCr is also based on activities of its commissions we asked them to send us texts representing commissions, their subject, main topics, recent highlights etc..

We started the IPC meeting with presentations of all the IUCr commissions (their representatives in IPC) in one-day event we called workshop *Current Trends and Future of Crystallography* that was open to public. We also put an effort to early and quite frequent e-mail communication to IUCr commissions and IPC members in order to keep their discussions on IUCr congress live and thus avoid just picking up the topics a few hours before the deadline. It appeared, that everything was useful and well accepted and led to very efficient work of the IPC. Therefore, we can recommend it also for future and some points are summarized below.

Early setup of IPC

During the 24th IUCr congress, the commission chairs were asked to discuss and select their representatives for the Prague congress. The request to the commissions for submitting more candidates to IPC was sent in December 2017. More candidates are useful and also necessary to have a chance to make gender and regional balance of IPC. We have selected IPC members and asked them to accept the role in e-mails containing also a list of tasks they are expected to do, in spring 2018.

The IUCr EC approved the IPC in May 2018. Then the place and time of the IPC meeting and CTFC workshop were determined. Actually, from offered three months March-May, only one week in May seemed to be suitable for all. Such early announcement is useful for all since they can plan it well. Moreover, one may get better prices in the hotel and possibly for travel.

IUCr commissions

Chairs of all the IUCr commissions received a request to prepare a text representing the commissions at the workshop (in proceedings) and they were also informed that their representatives in IPC should have about 15 mins presentations there. The announcement was sent in August 2018 and then reminded several times with a few extensions of deadline from March 1, 2019 to and of April 2019. In instructions for the texts it was written the following.

„Since the IUCr commissions are different we are not putting any strict instructions for the text length nor the contents scheme. In all cases, it can be introduced by a short description and meaning of the commission subject. Then possibly structured, if there are more streams in the subject with examples of advances achieved. Highlights from last years, problems to be solved etc. It should not be only about avtivities of the commissions. They can be mentioned there too, in particular in connection to the above items.“

Database driven software for online input, editing and list of everything.

True online database allows very flexible work and easy generation of different lists, categories etc. It was designed and prepared in autumn 2018 but scripts were modified actually all the time. All data have been available for all the IPC members for all the time and, of course, ost important during the IPC meeting. In addition to the lists ordered by commissions, priorities and for people also by regions, countries and gender the so-called classified lists were generated according to the fields of interests in soem way similar to the ECA focus areas. They were also used to make some balance between these areas in the programme. Unlike the ECA, mutiple assignments of the lectures and sessions was allowed. The used itmes were: Biological and macromolecular crystallography, Physcis and fundamental