questions concerning the functioning of the binding domain and the whole endolysin, further work is needed, especially the structure of the whole molecule and the complexes with possible ligands should be solved.

1. G. Bukovska, L. Klucar, C. Vlcek, J. Adamovic, J. Turna, J. Timko, *Virology*, **348**, (2006), 57-71.
2. M. Gerova, N. Halgasova, J. Ugorcakova, G. Bukovska, FEMS *Microbiol Lett.*, **321**, (2011), 83-91.
3. D. Rodríguez, M. Sammito, K. Meindl, I. M. de Ilarduya, M. Potratz, G. M. Sheldrick and I. Usón, *Acta Crystallogr. D*, **D68**, (2012), 336-343.

Session IX - Thursday, June 23

L30

XRPD AS A POWERFUL TOOL FOR STUDY OF PAINTED ARTWORKS

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Laboratory X-ray powder diffraction is a very effective and non-destructive method for direct phase analysis of paint layers usually consisting of complicated mixtures of pigments, binders, dyes, fillers and/or degradation products. While a conventional Bragg-Brentano set-up allows direct non-invasive analysis of smaller painted objects, e.g. miniature portraits, a micro-diffraction mode plays a substantial role in the analysis of samples (usually smaller than 1 mm) taken from paintings. The application of mineralogical analysis for study of provenance and technology of late Gothic/early Renaissance painting materials as well as examples of uncovered degradation products will be presented. The methodological pros and cons will be also discussed.

L31

DETERMINATION OF STRUCTURE OF SMALL PARTICLES

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We are producing and studying Magnetic Nanoparticles (MNPs) due its applications in biomedicine. The suitable size have to be comparable to biological entities (cells, proteins, and genes), controllable transport of MNPs in human body (drug delivery). The purpose of our study is produce particles for their ability to generate heat when an AC magnetic field is applied (magnetic hyperthermia). In particular, magnetic hyperthermia therapy is based on the fact that some types of cancer cells are more sensitive at temperature 41-45 °C than the healthy cells and that the required heat can be produced by MNPs. Nowadays majority in-field investigations are based on *in vitro* or *in vivo* animal model, but also, in the case of iron oxide based MNPs, this approach is used at the clinical level [1]. The heating ability of MNPs is dependent on morphology, microstructural and magnetic properties of MNPs, but also related to the amplitude and frequency of an applied magnetic field. In that sense, during the last years the synthesis methods have been intensively developed in order to control particle size distribution, surface effects and the degree of interparticle interactions, so that magnetic properties favourable for particular application could be successfully tailored. Although the tons of studies was published, there is huge confusion and misunderstanding in terms such as

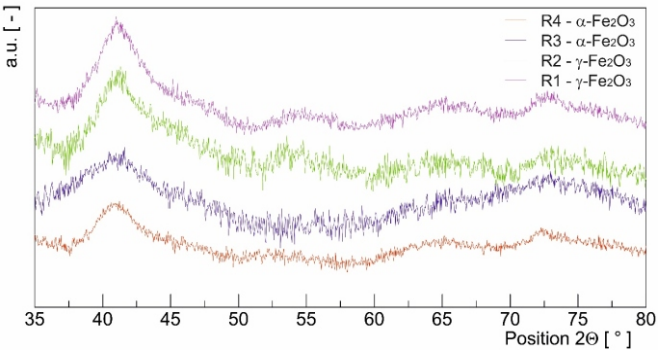

Figure 1. X-ray pattern of tiny particles.

particle, crystalline and grain size, which influenced the main characteristic – the magnetic properties. The confusion is originated by very wrong understanding of analytical method which are applied. For our purpose of magnetic hyperthermia, we are looking for magnetite Fe_3O_4 and maghemite $-Fe_2O_3$.

The standard method of determination of phase composition by X-ray powder diffraction (XRD) is not very helpful (see Fig. 1) in our case it produced results on the range

in between nano/amorphous which could be interpreted in any way or it could not be interpreted at all.

As well as the chemical composition obtain by EDS do not lead us to the correct results because the presence of S was overlooked in the samples R1 a R2. Eventually, the real structure and the chemical bound was determined by X-ray photoemission and Mössbauer spectroscopy which show as the presence of S and omit the existence Fe^{2+} type of bound for all samples. The measurement of magnetic properties by PPMS was much more helpful. It distinguish Neél temperatures and Morine transition of hematite (R3 and R4) at 940K and at 250K and the same temperatures when maghemite transform to hematite (R1 and R2) [2,3].

This more advanced technique helps us to determine the phase composition as maghemite and hematite instead of magnetite and maghemite, we are expecting.

1. M.R. Horsman, J. Overgaard, *Clinical Oncology,* **19**, (2007) 418.
2. Cornell, R. M., Schwertmann, *The Iron Oxides*, Weinheim, Germany, WileyVCH, 2003, 664.
3. MORIN, F., *Physical Review*, **78** , (1950) 819.

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L32

RTG TOMOGRAFIE – PRVNÍ ZKUšENOSTI

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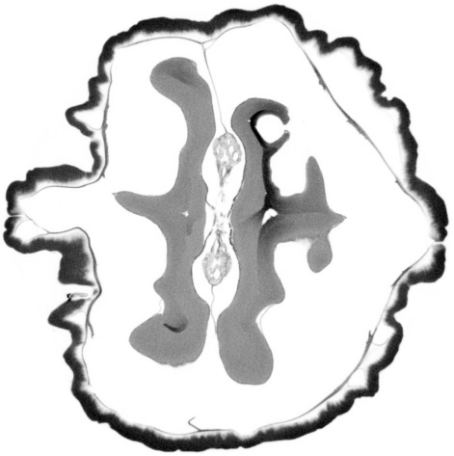
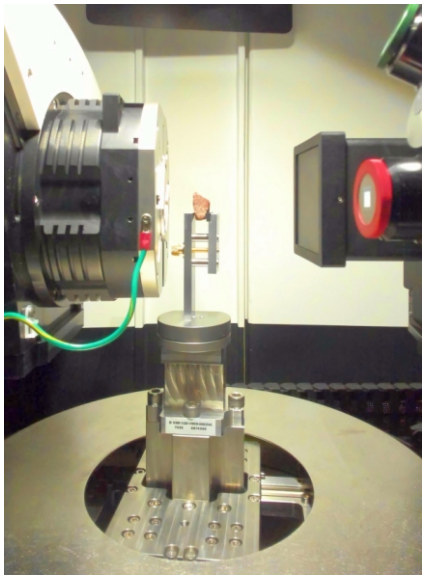
Začátkem letošního roku byl ve Fyzikálním ústavu uveden do provozu rentgenový tomograf ZEISS Xradia 610 Versa, viz obr. 1. V příspěvku bych se rád věnoval mým prvním zkušenostem nejenom s tímto tomografem, ale i z tomografií obecně.

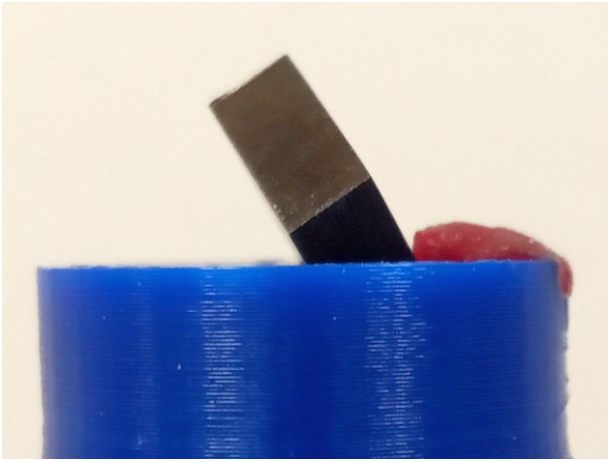
Princip měření je na tomto tomografu založen na kombinaci několika stovek až tisíců snímků s různou orientací vzorku kolem vertikální osy. Jako zdroj záření je zde použita wolframová anoda s volbou urychlovacího napětí od 30 do 160 kV. Čím je větší napětí tím má spektrum vlnových délek větší část v tvrdší části spektra, a tedy dochází obecně k menší absorpci záření ve vzorku. Pro modifikaci spektra je možné použít i sadu absorpčních filtrů které se umís ují na zdroj záření. Pro představu lze říci, že např. hořčíkové vzorky lze měřit až o tlouš ce několika málo centimetrů, zatímco u vysokoentropických slitin obsahující těžší kovy narážíme na problémy u vzorků o tlouš ce několika málo milimetrů. Náš tomograf je vybaven objektivy se zvětšením 4x a 20x a v ideálním případě dosahuje rozlišení až 200 nm na voxel. Dosažitelné rozlišení je dáno také velkostí a tvarem vzorku



Obrázek 2. Řez vnitřní strukturou vlašského ořechu.



Obrázek. 1. Vnitřek tomografu Xradia 610 Versa.

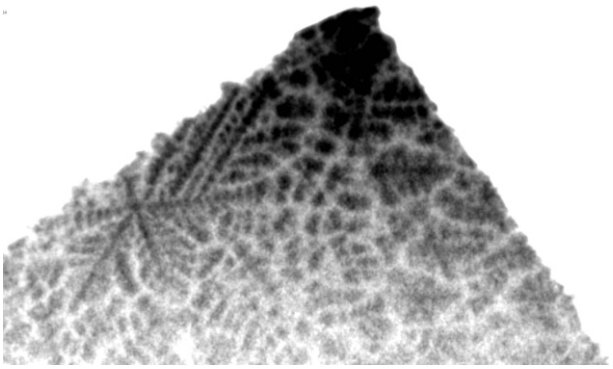


Obrázek 3. Držáček vzorku a vzorek vysokoentropické slitiny o rozměrech 5x5x3 mm.

(vzdálenosti mezi studovanou oblastí na vzorku a zdrojem záření) a jeho absorpčními vlastnostmi, protože s větším zvětšením objektivu se na detektoru používá tenčí detekční vrstva a tvrdší složky záření se hůře detekují.

Z principu metody je zřejmé že velmi dobře pozorovatelné jsou póry, či kontrast mezi vzduchem a materiálem. Jako ilustrativní příklad zde uvádím řez strukturou vlašského ořechu, obr.2. Zajímavější je již pohled na mikrostrukturu vzorku obsahujícím fáze s menším absorpčním kontrastem, viz. např. dendritická mikrostruktura v lité vysokoentropické slitině na obr 4.

Praktické zkušenosti ukazují, že pokud umístíme vzorek velkými plochami rovnoběžně či kolmo na osu otáčení dochází k nežádoucím artefaktům ve výsledném 3D obraze. Je proto vhodné umístit vzorek pod úhlem. Jako dobrý nástroj se ukázala 3D tiskárna, na které si celkem snadno můžete vytvořit držáček na míru, viz obr 3. Další výhodou umístění běžného vzorku ve tvaru kvádru tak aby byl špičkou nahoru a v ose otáčení je ten, že můžete studovat silně absorbují vzorky bez nutnosti jejich řezání. Což je v duchu rtg tomografie jako nedestruktivní metody,



Obrázek 4. Řez špičkou vzorku z obr. 3. Velikost pixelu cca 1.5 m.

která nám umožňuje nahlédnout do vnitřní struktury materiálu.

L33

PHASE TRANSFORMATIONS IN ZIRCONIUM AND TITANIUM ALLOYS

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The application potential of zirconium and titanium alloys can be seen in automobile, aircraft industry and in medicine as well. Thus, they are intensively studied mainly for their mechanical properties such as corrosion resistance, biocompatibility and specific strength. The mechanical properties are strongly influenced by the microstructure and phase composition of these alloys. Both pure zirconium and titanium can exist in two distinct crystal structures - alpha and beta phase. The beta phase is stable only at high temperatures above 700 °C (so-called beta-transus temperature), however the stability and coexistence of phases are strongly influenced by adding beta-stabilizing elements (stabilizes the beta phase) to the alloy, such as Mo or Nb. A higher content of the beta-stabilizers results in stable beta phase even at room temperature. The beta phase has a cubic body-centred unit cell. The alpha phase has a slightly distorted hexagonal close packed (hcp) structure where the most densely packed planes {110} of beta become the basal planes {0001} of alpha. In alloys with higher amount of the beta-stabilizing elements (~ 10-15 wt.%) a so-called omega phase may form. The omega phase has a hexagonal structure which is a result of a collapse of two-thirds of the {111} beta layers into one layer. The omega phase causes a brittleness of the material – its presence is undesirable. However, it forms in the alloys via martensitic transformation upon quenching from temperatures above beta-transus temperature and disappears upon heating to higher temperatures ~ 500 °C. Therefore, a complete understanding of phase transformations occurring upon heating in the alloys is necessary in order to choose an appropriate thermomechanical treatment to achieve the desired mechanical properties of the materials.

The presented study is focused on the investigation of phase content and stability in Zr12Nb, Zr15Nb and Ti15Mo alloys in temperature interval 30 – 800 °C. The high energy X-ray diffraction measurements were performed on polycrystalline bulk samples. In all three alloys the formation of the omega phase is observable which completely disappears above 500 °C and is followed by the formation of alpha phase. Although the sequence of the formation of phases is the same in both type of alloys, the mechanism and way of the formation of the omega phase is different.

L34

METASTABLE ALUMINAS IN PLASMA SPRAYED COATINGS

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Plasma sprayed coatings are materials with history of extreme thermal conditions. Very fine ceramic powder particles are carried by gas into high enthalpy plasma stream generated by plasma torch and molten droplets are projected onto cooled substrate. Solidified splats of added material are being layered and become a heat-sink for next incoming overheated liquid droplets. Therefore, interesting chemical compounds may be formed on the interface between splats despite high cooling rate. Moreover, recent advances in plasma technology enable injection of secondary material into the plasma torch by feeding of liquid suspension or solution. Using ethanol or water carriers influences already harsh conditions and new parameters of freedom for tailoring of coatings are available. Recently, we utilized hybrid plasma spraying process by simultaneously feeding the plasma stream with both powder and liquid feedstocks [1]. Very fine splats formed from liquid precursors may act as a cohesion improving agent for better durability of coatings.

Figure 1 shows an example of hybrid coating sprayed from Al_2O_3 powder and a water based TiO_2 suspension. At the interface of splats between alumina and titania, the newly formed Al_2TiO_5 phase was identified. However, only on the upper interfaces where the large alumina droplet interacted with already deposited miniature TiO_2 and the interdiffusion happened. Figure 2 shows the X-ray diffractogram of such coating. Major part of peaks intensity is in metastable phases and -Al_2O_3 whose defected spinel structure is still not well describable by Rietveld refinement fitting. Therefore, combination of standards of powder deposited coatings with XRF method is necessary to achieve accurate phase ratios

1. R. Musalek, T. Tesar, J. Dudik, J. Medricky, J. Cech, F. Lukac, *J Therm Spray Tech* (2022).

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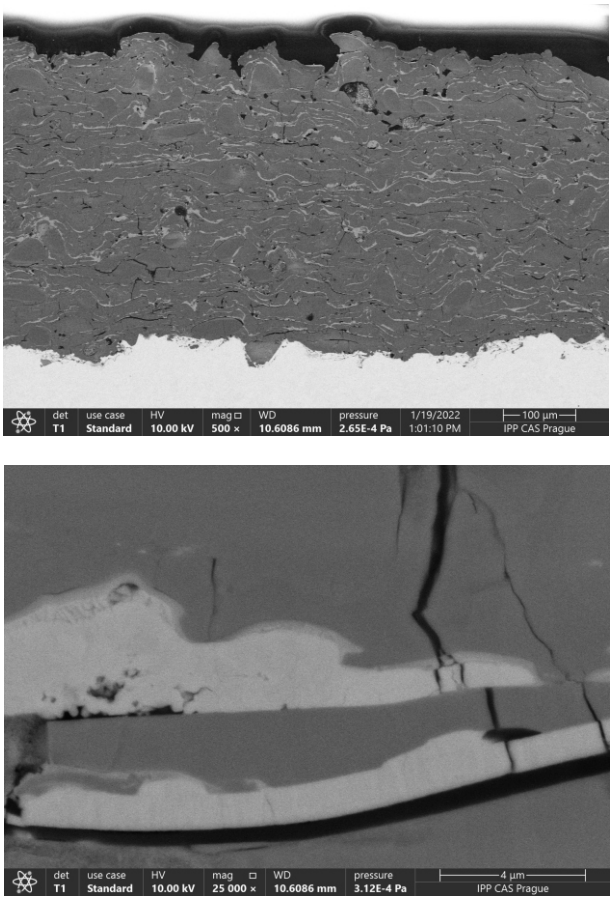

Figure 1. Micrographs of plasma sprayed coating of Al_2O_3 fed as a powder (dark) and TiO_2 fed as a liquid suspension (white). Interface between splats shows formed Al_2TiO_5 phase formed during cooling process.

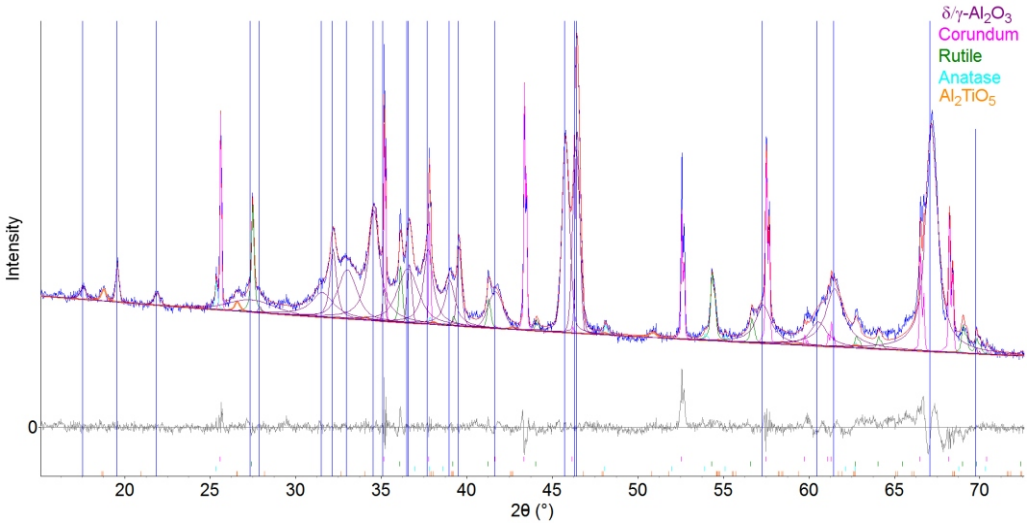

Figure 2. Micrographs of plasma sprayed coating of Al_2O_3 fed as a powder (dark) and TiO_2 fed as a liquid suspension (wihte). Interface between splats shows formed Al_2TiO_5 phase formed during cooling process.

L35

IN-SITU X-RAY DIFFRACTION DURING TENSION ON OFF-STOICHIOMETRIC Ni_2MnGa SINGLE CRYSTAL

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Ni_2MnGa is a broadly studied system because of its properties related to the magnetic shape memory. The compounds based on Ni-Mn-Ga system have also an interesting application potential as the micropumps or the sensors [1, 2]. Their shape memory properties are connected to the martensitic transformation, during which the high-temperature cubic phase (austenite) undergoes a transformation to the low-temperature phase with a lower symmetry (martensite) [3]. Because of a large magnetic anisotropy and a high mobility of the internal regions (so called twin variants/twinned domains)
induced reorientation could be achieved - it is more energetically preferable to reorient the whole unit cell than to rotate magnetic moments. A similar structural reorientation could be achieved by the application of an external mechanical force in tension or compression.

The high-resolution reciprocal space mapping with X-ray diffraction proved itself as a good tool to study the structure in Ni_2MnGa specimens [3, 4], which could contain several twin variants due to the shape memory effects. The reciprocal space mapping helps to distinguish between the Bragg reflections corresponding to individual twins. Moreover, reciprocal space mapping allows the precise study of the lattice parameters and a possible modulation in the structure. Our goal was to study the structure during the reorientation by X-ray diffraction in-situ in the applied tension.

For this purpose, we mounted the tensile stage (possible load up to 4 kN) inside the diffractometer. The studied specimen was $Ni_{50}Mn_{28}Ga_{22}$ with martensitic structure at the room temperature. The diffraction measurements revealed the strain in the direction of applied tension about approximately 3 % at 20 MPa leading to an exceptionally small Young modulus below 1 GPa. The structural modulation propagated along [1 1 0] is affected depending on the direction of applied tension. The modulation amplitude decreases when the applied tension is parallel to the basal plane (0 0 1). When the tension is applied perpendicularly to (0 0 1), the amplitude remains almost constant. The length of modulation vector remains the same within the range of errorbars regardless on the tension direction. The results also differ in dependence on the way how the sample is hold inside the stage. Holding directly with clamps allows almost full structural reorientation at approximately 10 MPa, but the sample cracks when the twin boundary reached the place on the sample hold by the clamps. Holding by a glue prevented the reorientation and the full reorientation did not occur up to 20 MPa.

1. A. R. Smith, et al., Microfluidic. Nanofluidics, 18 (2005), p. 1255, doi: 10.1007/s10404-014-1524-6.
2. A. Hobza, et al., Sensor. Actuator. A, 269 (2018), p. 137, doi: 10.1016/j.sna.2017.11.002.
3. O. Heczko, et al., Acta Mater., 115 (2016), pp. 250-258, doi: 10.1016/j.actamat.2016.05.047.
4. P. Cejpek, et al., J. Alloys Compd., 855 (2021) 157327, doi: 10.1016/j.jallcom.2020.157327.

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