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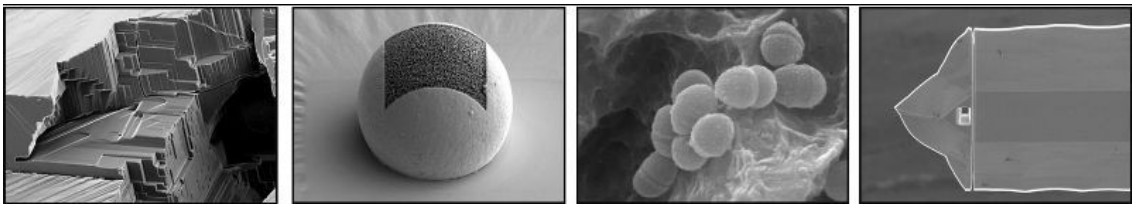
University of Basel
Swiss Nanoscience Institute

Swiss Nanoscience Institute
Exzellenzzentrum
der Universität Basel und
des Kantons Aargau

NANO IMAGING LAB

Newsletter

November 11, 2024



The NI Lab @ EMC in Copenhagen

At the end of August the NI Lab team traveled to beautiful Copenhagen and attended the European Microscopy Conference 2024. Besides learning a lot about new techniques, applications and technologies in this field and reunite with longtime aquaintances, the team was also able to build on new friendships. The team was invited to visit the Technical University of Denmark (DTU) and was able to gain interesting insight into the cleanrooms and the work of Dr. Katja Wurster. The NI Lab team would like to thank Katja for the hospitality.

In order to even more substantiate their new collaboration, Marcus gave a seminar talk at

the DTU entitled "Advanced Nanofabrication in the Nano Imaging Lab". His talk was very well attended and met a great response, which led to profound discussions and new orders for the Nano Imaging Lab.

The collaboration with Prof. Dr. Nini Pryds, head of the research section 'Functional Oxide Materials' at the Department of Energy Conversion and Storage at The Technical University of Denmark (DTU) comprises the beam induced deposition of precise gold structures on top of AFM-cantilevers. Two different studies are being supported. On the one hand the mechanical properties of STO membranes on shaped surfaces are being investigated and, on the other, new scanning probe microscopy measuring tips are being developed. The NI Lab team is looking forward to the collaboration.

Our contribution to research: latest publications 2024

New ceramic ternary coating for medical devices

CoCr based metal alloys are the most commonly used compounds for orthopedic implants nowadays. In order to avoid sensitization of patients and to increase wear resistance, ceramic coatings of implants play a very important role as a protective layer.
This paper presents the characterization of the microstructure, the crystallographic phases and the mechanical properties of a newly developed ternary Al_2O_3-ZrO_2-TiO_2 ceramic coating, which has potential in high wear applications, for example medical devices.
The ceramic coating was produced by atmospheric plasma spraying (APS) in a thickness of 400µm, using the newest generation of the Debye-Larmor cascaded plasma torch.

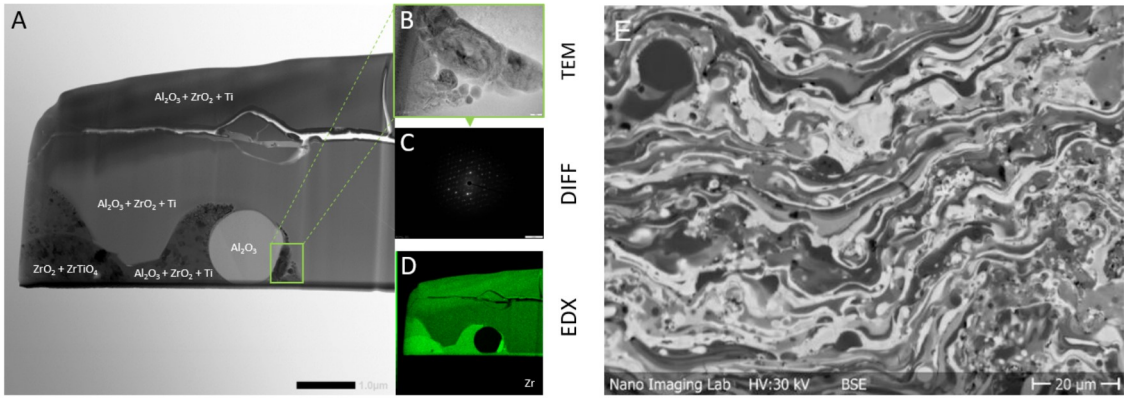


Figure1: (A) BF-STEM image of one of the lamellae investigated. (B) HRTEM image. (C) Selected Area Diffraction (SAD) of the region shown in (B). (D) EDX mapping of the same area shown in (A). (E) Backscattered SEM picture of the coating.

The microstructure of the powder and the cross-section of the coating were investigated by Scanning Electron Microscopy (SEM) in the Nano Imaging Lab. The coating appears very compact and consists of lamellar splats (Fig1.), which is a typical microstructure for APS coatings. No through-thickness cracks nor delamination from the substrate were observed.

The NI Lab could also help with examining the nano-crystalline phases in the coating by producing FIB-lamellas and performing HRTEM as well as scanning TEM (STEM) imaging in combination with EDX-mapping on the cross-sections. With the help of electron diffraction, it was possible to analyse the coating, which consists of single phase α-Al_2O_3, monoclinic m-ZrO2 and a nano-crystalline dual phase structure of α-Al_2O_3 and ZrO_2. Ti is either present as $ZrTiO_4$ or as solute in the dual phase (Fig1).

Cynthia Sin Ting Chang, **Marcus Wyss**, Michal Andrzejewski, Geoffrey Darut, Lukas Graf, Vladimir Novak, Margie Olbinado, **Susanne Erpel, Alexander Vogel,** Simon Bode, Michael de Wild, Armando Salito
Microstructures, phase and mechanical characterisation of Al2O3-ZrO2-TiO2 coating produced by atmospheric plasma spraying
Open Ceramics. 20, 100698 (2024)
doi.org/10.1016/j.oceram.2024.100698

Early detection of crystallinity in ternary solid dispersions

Amorphous solid dispersions are commonly used in pharmaceutical product development to improve bioavailability of poorly water-soluble molecules by enhancing the rate and extent of dissolution. These formulations comprise an amorphous active pharmaceutical ingredient stabilized by a polymer matrix. This paper presents a novel computational approach for the selection of co-formers in ternary solid dispersions prepared by hot-melt extrusion. The model drugs cinnarizine and bifonazole were selected based on their known unstable glass-forming ability to detect early signs of physical instability of ternary solid dispersions.

Conventional bulk stability testing, such as differential scanning calorimetry (DSC) and powder X-ray diffraction analysis (XRPD), were complemented with the surface sensitive 3D-confocal laser scanning microscopy (LSM) imaging, as well as atomic force microscopy (AFM) to detect possible recrystallisation during storage.

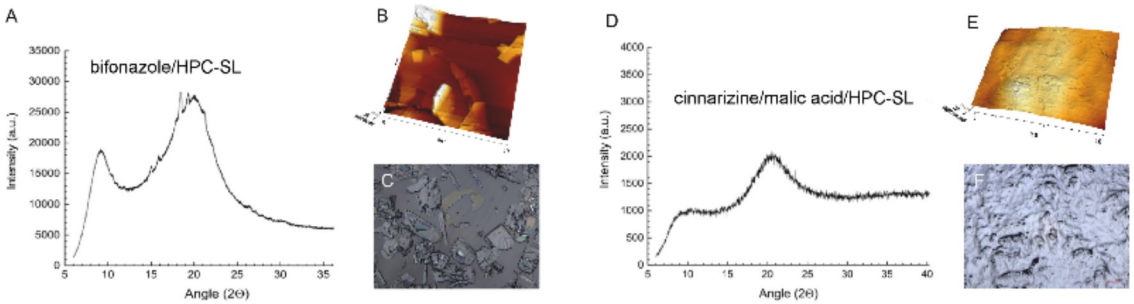


Figure 1: XRPD diffractogram of an unstable binary solid dispersion of bifonazole and HPC-SL grade (A) with corresponding AFM (B) and 3D-confocal LSM (C) images, in comparison to the XRPD of a stable ternary solid dispersion of cinnarizine, malic acid, and HPC-SL grade (D) with corresponding AFM (E) and 3D-confocal LSM (F) images. Both formulations had been stored for 3 months at room temperature, sealed.

In this collaboration with Prof. Dr. Martin Kuentz from the School of Life Sciences FHNW in Muttenz, the Nano Imaging Lab was able to lend a hand by measuring the surfaces of the extrudates at different times during storage using laser microscopy and AFM. The resulting images played an important role in the interpretation of the DSC and XRPD analyses and finally allowed a well-founded evaluation of the best drug-polymer mixtures in terms of stability.

Andreas Niederquell, Susanne Herzig, **Monica Schönenberger**, Edmont Stoyanov, Martin Kuentz
Computational Support to Explore Ternary Solid Dispersions of Challenging Drugs Using Coformer and Hydroxypropyl Cellulose
Molecular Pharmaceutics, XX (2024)
doi.org/10.1021/acs.molpharmaceut.4c00592

FIB-SEM Crossbeam 540 from ZEISS: Now open for users!

Fields of application are: • Imaging, analysing, milling and cross-cutting of samples in the nanometre range • TEM lamellae: ‘lift out’ and thinning • Nanofabrication and micromanipulation • Gas injection system for Pt deposition (FEBID and FIBID)

http://nanoimaging.unibas.ch