Investigating the distribution of 2D materials dispersed in organic conductive coatings via synchrotron-based microscopy techniques

INDUSTRIAL PILOT PROJECT

THE INDUSTRIAL CHALLENGE

Provexa Technology is a surface treatment company that develops advanced functional coatings based on polymer matrices with additions of functional 2D materials such as graphene and MXenes. MXenes are a class of 2D materials that consists of a single layer of transition metal (M) carbides or nitrides (X). The addition of relatively few amounts of functional 2D materials to traditional coatings is known to greatly affect their macroscopic properties, such as improving electric conductivity. However, the exact relationship between the preparation method, the resulting micro-/nano- structure, and the final properties is still poorly understood, and there is also a need to identify suitable techniques to investigate it.

WHY USING A LARGE SCALE FACILITY

Unlike traditional lab methods, synchrotron-based methods can simultaneously achieve sufficiently high spatial resolution, sensitivity and chemical specificity, for example for the identification and localization of specific chemical elements, and in some cases even their chemical states. For instance, soft X-ray photoemission electron microscopy (XPEEM) and microscale X-ray photoelectron spectroscopy (µXPS) can provide morphological and chemical information, respectively, and are known to be able to distinguish graphene flakes from the surface (≤10 nm deep) of a polymer matrix. On the other hand, MXene-based coatings contain heavier elements M such as titanium (Ti) buried in the bulk of the polymer matrix, so hard X-ray fluorescence (XRF) microscopy is more suitable to map their distribution.

HOW THE WORK WAS DONE

All experiments were carried out at the MAX IV Laboratory in Lund, on organic coatings containing either graphene or MXenes and with thicknesses ranging up to a few tens of microns. The XPEEM and µXPS analysis were carried out at the MAXPEEM beamline (Fig. 1) to investigate graphene distribution on the coating surface. XPEEM generated images several tens of microns wide with an estimated spatial resolution of 20 nm. µXPS generated spectra based on carbon from 1.5 µm diameter spots.

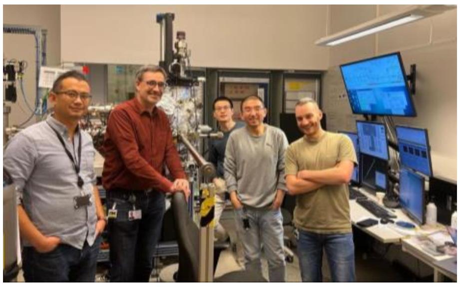

Figure 1. From left: Shun Yu (RISE), Christian Werdinius (Provexa), Lin Zhu and Yuran Niu (MAX IV), Simone Sala (RISE) at the MAXPEEM beamline

XRF at the NanoMAX beamline was used to map Ti distribution in the MXene-containing coatings. XRF generated several maps up to a few tens of microns wide with an estimated spatial resolution down to 108 nm.

THE RESULTS AND EXPECTED IMPACT

While soft X-ray XPEEM and µXPS at MAXPEEM remain valid tools to investigate carbon speciation, they turned out to be incompatible with the low concentrations of graphene relevant for Provexa's products. On the other hand, significant Ti inhomogeneities were successfully detected with hard X-ray XRF, indicating that MXenes form small clusters at the micron- to submicron scale (Fig. 2). The size and distribution were found to depend on both MXene formulation and concentration as well as on the coating preparation process. Such insight will help Provexa to optimize its functional coatings manufacturing process to achieve the desired macroscopic properties.

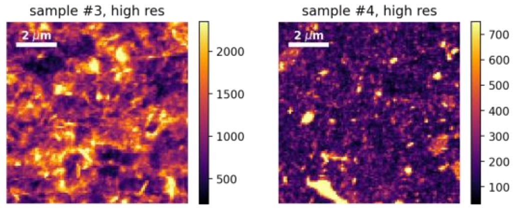

Figure2. Ti distribution maps from XRF data collected with hard X-rays at NanoMAX: colour bars indicate (projected) Ti concentration in ng/mm^2

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