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In-situ Nanoscale Characterization of Polymer Melting and Crystallization via Multimodal Chemical Imaging

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Imaging mass spectrometry, including secondary ion mass spectrometry (SIMS) and matrix assisted laser desorption ionization (MALDI) are widely used for local chemical characterization of wide range of various materials and systems. Using of the focused ion beams or laser they enable imaging of the local chemical composition with micro- and nanometer resolutions [1-3]. Commonly those imaging techniques are applied for investigations of the static material state, with no or little attention to time evolution of the material properties. Here, we utilized time-of-flight secondary ion mass spectrometry (ToF-SIMS) and MALDI to study in-situ process of the Teflon coating melting and crystallization.

Studies were carried out using TOF.SIMS.5.NCS (ION-TOF GmbH, Germany) instrument combining ToF-SIMS with atomic force microscope in the same vacuum chamber and allowing temperature control of the sample. ToF-SIMS measurements were performed using Bi3++ liquid metal ion gun focused down to 100 nm spot size. Scanning was performed over grid of 256×256 px with 50×50 µm2 field of view with detection of secondary ions and secondary electrons. Secondary electrons signal was used to identification of the sample morphology as a function of temperature, while secondary ions were analyzed to study local changes in the chemistry. Autoflex TOF.MALDI (Bruker Gmb, Germany) instrument has been also used to identify large molecules of coating before and after crystallization.

We used ToF-SIMS to study in-situ process of the Teflon coating melting and crystallization on the steel substrates. In measurements samples were heated up above coating melting temperature and cooled down under control of the ToF-SIMS. At each temperature step secondary electron (SE) and secondary ion (SI) measurements have been carried out under irradiation of Bi3++ primary ion beam. SE measurements allowed to control change of the coating morphology and to identify melting and crystallization temperatures (Fig. 1a-c). Correspondent, chemical imaging via SI allowed to correlated chemical changes associated with phase transition during melting and crystallization (Fig. 1d-f). In particular, significant chemical changes were detected near the sample edge. To further understand those changes we performed MALDI imaging before and after sample heating. This allow to identify chemical phenomena near the sample edge.

The explored phenomena provide insight into the chemistry of Teflon coating melting and crystallization, while developed workflows enable in-situ studies of temperature depended processes in wider range of materials and systems.

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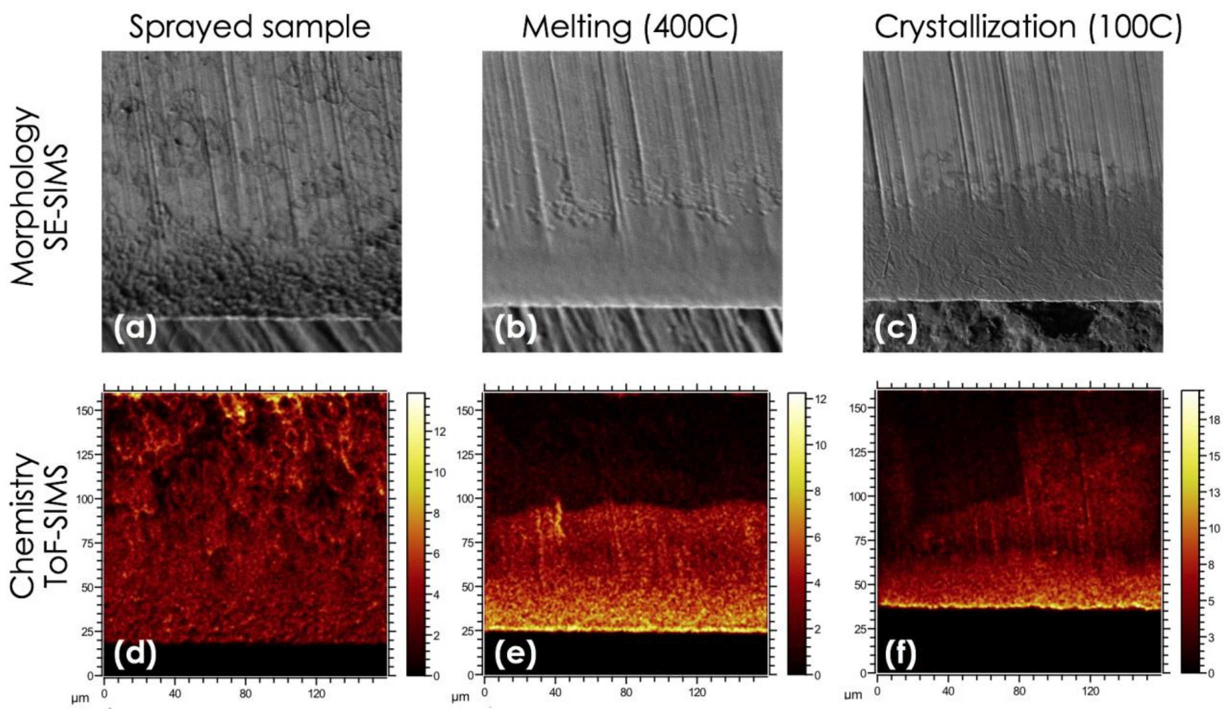

Figure 1. Multimodal in-situ study of polymer coating melting and crystallization. (a-c) Morphology of the sample surface measured via secondary electron signal; (d-f) Local chemical changes measured by ToF-SIMS. (a, d) Room temperature; (b, e) melted coating at 400ºC; (c, f) crystallization after cooling down to 100ºC.