INDUSTRIAL PILOT PROJECT

In-situ analysis of ultrathin passive film of advanced tooling alloy by synchrotron X-ray photoelectron technique

THE INDUSTRIAL CHALLENGE

Uddeholms AB is specialized in developing and producing advanced tooling alloys for demanding applications. A great challenge is to achieve sufficient corrosion resistance and desirable mechanical properties of the alloys simultaneously, which requires a detailed understanding of the passive film formed on the alloy surface and the influence of heat treatment.

WHY USING A LARGE SCALE FACILITY

Tooling alloys are martensitic steels with magnetic property, which is problematic for surface analysis using lab-source X-ray photoelectron spectroscopy (XPS). Moreover, normal XPS is done in ultrahigh vacuum condition, so the information of the passive film is not really relevant for service conditions in air or aqueous environments. Synchrotron-based XPS, with high flux and tunable energy of the beam, and especially the newly-developed ambient pressure XPS (APXPS) measuring system, can tackle the challenge, allowing us to obtain reliable and relevant information of the passive film.

HOW THE WORK WAS DONE

Tooling alloy samples were prepared and heat treated by Uddeholms AB, and electrochemical measurements to define the experimental conditions for the synchrotron APXPS measurements were performed at KTH. By using a special designed sample environment (electrochemical cell) and the dip-and-pull method, we were able to do XPS measurement at a pressure up to 17 mbar, immediately after lift-up of the sample from the corrosive solution that was subjected to electrochemical polarization. The APXPS measurements were performed at the HIPPIE beamline of MAX IV, in collaboration with Prof. Edvin Lundgren's group at Lund university, and supported by the APXPS team leader, Andrey Shavorskiy, and beam scientist, Mattia Scardamaglia.

THE RESULTS AND EXPECTED IMPACT

With the help of the beam scientist, we solved the problem of magnetic effect on the XPS measurement. During the APXPS measurement (at 17 mbar), there is still an aqueous adlayer on the sample surface, so the information obtained represent the whole passive film including the hydroxide part. Moreover, we could analyse the passive film immediately after anodic polarization, yielding valuable information of the chemical composition and stability of the passive film.

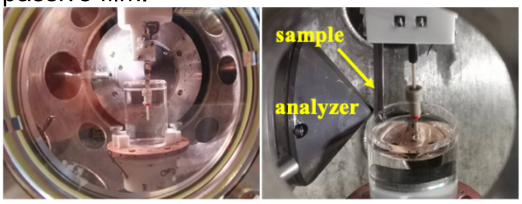

Figure. Left: the electrochemical cell in APXPS chamber. Right: sample in pull position for XPS measurement.

This experiment demonstrates the unique possibility to analyse, in-situ, the passive films formed on the surface of metallic materials including martensitic tooling alloys, and the information obtained are helpful for the optimization of the tooling alloy and heat treatment. We also extended the collaboration with other academic and industrial partners. The technique has already been included in other projects.

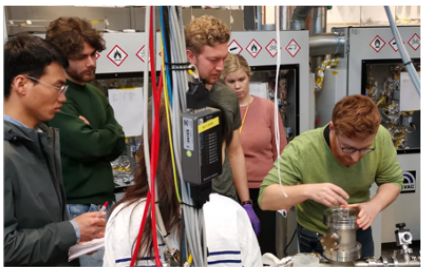

Figure. Beam scientist, Mattia Scardamaglia, shows how to remove dissolved gas from the corrosive solution.

"This project has demonstrated the unique possibility for us to do detailed and relevant surface analysis of our tooling alloys, which is of great importance in our R&D work" /Krishnan Anantha, Uddeholm.

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