INDUSTRIAL DEVLOPMENT PROJECTS

Improvement of the conditions for in situ studies of heat treatment processes by photoelectron microscopy

THE INDUSTRIAL CHALLENGE

Alfa Laval, Gränges, and Seco Tools, specialize in manufacturing steel heat exchangers, rolled aluminum, and metal cutting tools, respectively. The companies have a common need to optimize their heat treatment manufacturing processes for achieving the required surface properties and share interest to secure analytical methods allowing in-situ studies of these processes.

WHY USING A LARGE SCALE FACILITY

Many synchrotron techniques for chemical analysis of surfaces are outperforming their lab counterparts and offer low acquisition times as well as higher resolution combined with versatility of the experimental setup. In particular, the in-situ heating experiments are often available. The Spectroscopic PhotoElectron and Low Energy Electron Microscopy (SPELEEM) is an imaging technique providing chemical information with nanometre-range surface sensitivity. The technique has proven to be an asset for surface analysis of industrial samples in previous Vinnova pilot project (2019-05278).

HOW THE WORK WAS DONE

In situ SPLEEM is available at the MAXPEEM beamline of MAX IV. The possibility of in-situ heating is also offered by other spectroscopy beamlines at MAX IV, but to successfully mimic the industrial heat treatments there is a common need to improve the temperature control. Therefore, a heating control system upgrade was implemented, comprising a package of hardware and software. This includes digital pyrometers with small focus of ~1mm^2, as well as the units for programmable heating. In-situ heating tests were conducted with SPELEEM on three materials: steels, clad aluminum, and TiAlN-coated WC. The pyrometers were calibrated in the temperature range of interest, which is 600-1000ºC for steels, 400-600 ºC for Al and 800-900 ºC for coated WC. The samples were heated with pre-set heating rate to the setpoint temperature as temperature data were saved for further evaluation. The project included two academic beamtimes where heat-induced chemical transformations of the sample surface were followed with SPELEEM.

THE RESULTS AND EXPECTED IMPACT

The installation and commissioning of the hardware for more precise temperature control was carried out successfully. The system was primarily optimized for MAXPEEM beamline, but the possibility to use it was also demonstrated at including FlexPES spectroscopy beamline. With careful calibration and use of PID feedback loop, the upgraded temperature control system **reproducibly** recorded the temperature history of samples with estimated precision of **± 3-5ºC**. The small focus of pyrometers allows local temperature measurements on inhomogeneous samples. New sample preparation protocols were successfully tested on TiAlN-coated WC and clad Al samples, both with coatings just a few µm thick, to achieve the required surface roughness of 1-0.25 µm. Figure 1 shows example of clad Al sample polished at an angle for studying of clad and core Al simultaneously.

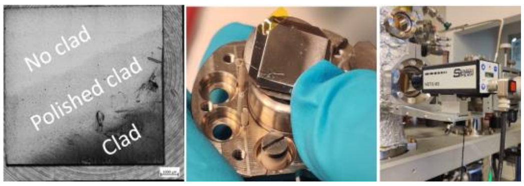

Figure 1. 10*10 mm^2 clad Al coupon polished at an angle. Sample mounting with thermocouple attached to the surface for pyrometer calibration. The pyrometer mounted at MAXPEEM beamline.

The results and hardware installation procedures have been already summarized in a detailed report, which is available and MAXPEEM beamline.

"The new instrumentation permit experiments close to the solidus and allows new insights to the oxide break-up of vacuum brazing alloys."
/T. Stenqvist, Gränges

Contacts: K. Simonov – Swerim, konstantin.simonov@swerim.se,
F. Lenrik – Lund University, A. Zakharov – MAX IV laboratory
T. Stenqvist – Gränges, M. Fredriksson – Alfa Laval, R. M'saoubi – Seco Tools

Vinnova's project No: 2021-03828 **Duration:** Nov. 2021 -- June 2024

Funded by Sweden's Innovation Agency, Vinnova, in order to build competence and capacity regarding industrial utilisation of large-scale research infrastructures such as MAX IV and ESS.