

In situ XPS of Al₂O₃ break-up at flux-free brazing conditions

THE INDUSTRIAL CHALLENGE

Braze clad on aluminium sheets enables fast and convenient brazing assembly of complex heat exchangers. The dominant brazing technique today is controlled atmosphere brazing (CAB) which is performed in a furnace with nitrogen (N₂) gas and temperatures around 600 °C. Before brazing it is necessary to dissolve the inherently formed surface oxide. The CAB technique however requires the use of a potassium fluoroaluminate flux for the oxide break-up process. Beside a negative effect on health and environment, the flux residues in the ready-made heat exchanger can interact chemically with coolants and form a gel in the heat exchanger tubes. The tubes become clogged and suppresses the cooling capacity. Due to this, flux-free brazing where magnesium acts as an oxide break-up agent is an attractive alternative. The knowledge about this process is however very limited and it is a challenge to obtain a stable process – so far it has been a trial and error procedure.

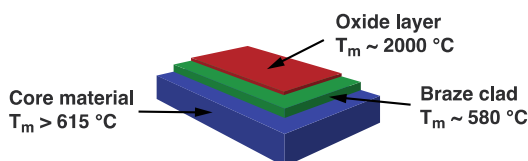


Figure 1. Braze clad aluminium sheet where the braze clad melts at about 580 °C. The surface oxide must be dissolved before the molten braze clad can flow to regions where to form joints.

WHY USING A LARGE SCALE FACILITY

X-ray photoelectron spectroscopy (XPS) is an element sensitive technique that can provide chemical-specific information about the probed elements in a sample. It is, thus, possible to follow the changes in the XPS spectra for oxygen (O), aluminium (Al), and magnesium (Mg) during a heat treatment. That means that the kinetics of the break-up process of Al₂O₃ initiated by Mg is tracked while it occurs. To avoid evaporation of Mg it is necessary to perform the experiment at 1 mbar N₂, which require the high X-ray intensity that can be obtained in a synchrotron radiation (SR) facility. In addition, the high X-ray intensity facilitate fast data acquisition necessary for *in*

operando measurements. By using SR it is also possible to tune in the provided photon energy for depth profiling through the oxide overlayer. Hence, this study would be impossible using conventional XPS.

HOW THE WORK WAS DONE

An initial investigation was performed at Linköping university using a conventional surface science XPS system. The samples were then heat-treated at Gränges R&I in a CAB furnace and the obtained spectra showed O 1s, Al 2p, and Mg 2p before and after the heat-treatment. The SR-based *in operando* study was performed at ambient pressure at the XPS beamlines HIPPIE and SPECIES at MAX IV in Lund, Sweden. Project participants were representatives from Gränges R&I, Uppsala Synchrotron AB, and Linköping University.

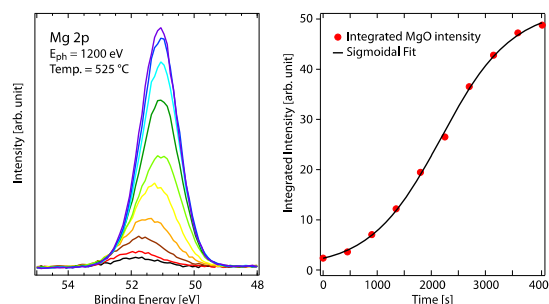


Figure 2. Mg 2p XPS of the oxide layer above the braze clad and how the intensity evolves as a function of time at a temperature of 525 °C.

THE RESULTS AND EXPECTED IMPACT

The study has successfully provided new information regarding changes in the oxide composition, as well as the Mg-kinetics. It also displayed the oxide break-up mechanism, when Mg diffuses from the aluminium sheet core material up to the oxide through the braze clad at temperatures close to brazing conditions. The project has provided new perspectives on the Al₂O₃ break-up process and valuable inputs for the development towards a stable process that facilitates flux-free brazing.

***“Nobody has done this study before us.
It has simply not been possible”
/ Lars-Åke Näslund, Gränges R&I***



GRÄNGES

Uppsala Synchrotron AB

Contacts: Linda Ahl – Gränges Sweden AB, linda.ahl@granges.com
Robert Moberg – Uppsala Synchrotron AB, robert@synchrotronix.se

Vinnova’s project No: 2018-04410 **Duration:** November 2018 -- February 2020

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.