INDUSTRIAL PILOT PROJECT

In-situ photoelectron microscopy study of σ-phase growth in DSS

THE INDUSTRIAL CHALLENGE

Outokumpu Stainless and Sandvik Materials Technology are the global producers of advanced steels, including duplex stainless steels (DSS). It is of great importance to control and predict intermetallic phase formation and homogenisation for producing DSSs with long lifetime and better properties. Formation of σ-phase in DSSs at these elevated temperatures causes segregation of alloying elements which can lead to deterioration of steel properties even at low fractions of σ-phase. Numerical models have been developed to describe the phenomenon, but lack of experimental verification hinders their application.

WHY USING A LARGE SCALE FACILITY

Laboratory-based electron microscopy is often used for ex-situ studies, e.g., to illustrate σ-phase after performed annealing. Studying of chemical and structural changes accompanying σ-phase formation, however, requires a very high spatial and good temporal resolution as well as capability to mimic the process of annealing while simultaneously measuring. Such performance and equipment for in-situ heating requires synchrotron light sources.

HOW THE WORK WAS DONE

Samples of 2507 DSS were polished down to 0.25 um and studied in Spectroscopic PhotoElectron and Low Energy Electron Microscope (SPELEEM) at the MAXPEEM beamline of MAX IV. The in-situ annealing and characterisation was performed in the T range 600 – 1000°C.

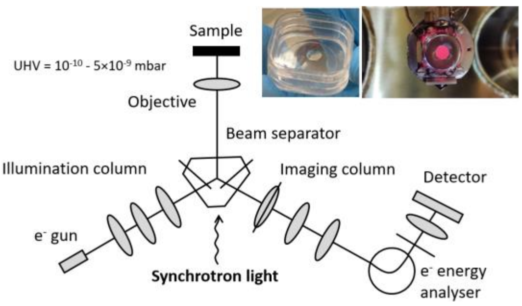

Figure 1. Schematic diagram for SPELEEM. The sample can be illuminated with low energy electrons or photons. Sample in the sample box and mounted in the cartridge during heating is shown. Dr. A. Zakharov and Dr. Lin Zhu from MAX IV operated the instrument.

THE RESULTS AND EXPECTED IMPACT

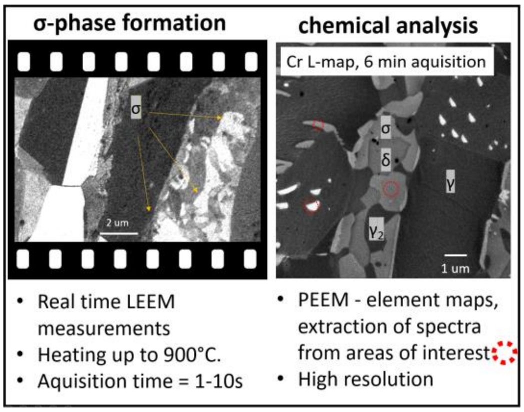

Figure. 2 Left: LEEM image recorded at 786°C. Right: Cr PEEM map.

Growth of σ-phase was observed in-situ in real time with low-energy electron microscopy (LEEM) (Fig 2, Left). Chemical analysis with photoemission electron microscopy (PEEM) allowed to record high-quality element maps at the Cr L-, Fe L-, Ni L-, Mn L-, Mo M2-, N K-, and O K- absorption edges, to identify chemical state of species, and to obtain element profiles – all in one dataset (Fig. 2, Right). The high surface sensitivity of PEEM and LEEM also allowed to observe surface segregation of active elements during annealing. The fact that the instrument can operate only at high vacuum conditions, however resulted in formation of larger number of precipitates than expected at true process conditions in air. This complicated the homogenisation of σ-phase and comparison with computational models. Information on σ-phase nucleation, surface chemistry, and precipitate growth during in-situ annealing was, however, never reported before and additional in-depth analysis of data is planned.

"Results indicate that precipitation on a surface can be examined. It may become a useful tool to study precipitation at grain boundaries which could be very beneficial."
/J. Y. Jonsson, Outokumpu

Contacts: J. Y Jonsson – Outokumpu Stainless, jan.y.jonsson@outokumpu.com
U. Borggren – Sandvik Materials Technology, ulrika.borggren@sandvik.com
K. Simonov – Swerim AB, konstantin.simonov@swerim.se

Vinnova's project No: 2019-05278 **Duration:** March 2020 -- December 2021

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.