To reveal H-induced cracking in duplex stainless steel by in-situ neutron imaging and x-ray diffraction

THE INDUSTRIAL CHALLENGE

Hydrogen embrittlement (HE) of metals is caused by absorption and accumulation of hydrogen (H) at "traps" in the metals, and leads to microstructure degradation and cracking. Being crucial for both safe use and for development of advanced materials, Alleima and the metal industry as such have long strived to understand the mechanism. However, due to the high mobility of H, the high complexity of HE, and the difficulty to detect H in metals in real time, little is known about the interplay between H distribution and strain localization in the microstructure.

WHY USING A LARGE SCALE FACILITY

Neutron and synchrotron radiation have a high penetration power, providing unique possibilities to map strain evolution and Hinduced damages in metals. High energy synchrotron X-ray diffraction (XRD) can map the strain evolution and microstructure degradation, and provides high temporal and spatial resolutions. Neutrons, however, have potential for direct visualization of H in metals. We demonstrate this possibility via situ neutron imaging for realistic in conditions that cause HE. By operando neutron and synchrotron measurements, we aim to elucidate the roles of H accumulation and strain in H-induced crack initiation.

HOW THE WORK WAS DONE

To reveal the local distribution of H, the local strain evolution, and the microstructure degradation in a super duplex stainless steel, a special sample environment was designed to enable *operando* Time-of-flight (ToF) neutron imaging measurement under combined electrochemical H-charging and tensile loading. An electrochemical cell of 200 ml was constructed for samples of 6x12x60 mm. The *operando* ToF neutron imaging of the sample was performed during electrolytical H-charging with linear increase of tensile loading at the IMAT beamline at ISIS in UK, with the help of beamline scientist Winfried Kockelmann. High energy

operando synchrotron XRD was carried out at the Swedish Material beamline P21 at PETRA III in DESY, Germany, with the help of beamline manager Ulrich Lienert.

THE RESULTS AND EXPECTED IMPACT

The neutron attenuation images of the tensile sample after 12 hours electrolytical H-charging revealed local H accumulation in the alloy. The 'diffraction contrast' and Bragg edges of the neutron attenuation spectra also enable phase, texture and strain analysis of the ferrite and the austenite phases. The synchrotron XRD results show higher H-induced strain evolution in the austenite phase than the ferrite phase, and heavily deformed lattice in the grains. The results thus provide consistent and complementary information. This also demonstrates the feasibility to utilize the neutron imaging for in-situ/operando studies of HE. We plan to further improve the setup to reduce noise and achieve higher spatial resolution for H mapping.



Figure. (1) The sample mounted on the EC cell; (2) the tensile setup for neutron measurement; (3) the force vs. deformation curve; and (4) an example of the first neutron images: starting (point A) and after 12 hours H charging (point B).

"It is exciting to see that it seems to be possible with neutron technique to study the location of hydrogen in the material." /Ulf Kivisäkk, Alleima



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