INDUSTRIAL PILOT PROJECT

In-situ neutron scattering analysis of the early stages of phase separation in duplex stainless steels at elevated temperature

THE INDUSTRIAL CHALLENGE

There is a strong industrial interest in increasing the service temperature and/or prolonging the service life of duplex stainless steels to make use of their excellent strength and corrosion resistance. Low temperature embrittlement of duplex steels, often referred to as 475 °C embrittlement, however, restricts their application at low temperatures. The embrittlement is caused by the immiscibility of the key alloying elements Fe and Cr in the ferrite phase at temperatures below about 550 °C. If the alloy is exposed in the temperature range between ~250 - 550 °C for extended times, the Fe and Cr will demix and form a nanostructure constituting two ferrite phases. This leads to increasing hardness and decreasing toughness with time, and may cause catastrophic failure.

WHY USING A LARGE-SCALE FACILITY?

Most of the prior research has been devoted to long term isothermally aged materials and *ex-situ* metallography using techniques such as transmission electron microscopy and atom probe tomography. However, little effort has been paid to the early stages of demixing. Hence, *in-situ* measurements of the nanostructural evolution during the thermal cycle could provide new information on the early stages of demixing, which is not possible to capture accurately with post mortem analysis. *In-situ* measurements during thermal cycling are possible using small-angle neutron scattering (SANS).

HOW THE WORK WAS PERFORMED

The SANS experiments were carried out at the ISIS Neutron and Muon Source, UK, by representatives from Outokumpu Stainless and KTH in collaboration with Dr. Stephen King at ISIS. The SANS2D beamline were uesd at an incident neutron wavelength of 2-10 Å for analysis of 10x10x1.1 mm samples from two duplex stainless steels (2205 and 2507). The *in-situ* experiments during heat treatment were carried out at three different temperatures for up to six hours. To supplement the in-situ measurements, heat treatments of *ex-situ* specimens were conducted in advance using three temperatures and aging times. *Ex-situ* specimens were also prepared in advance by thermal simulations using a Gleeble instrument in order to simulate different cooling rates due to different steel plate thicknesses. Specimens heat treated according to the *ex-situ* SANS conditions were subjected to property testing using hardness and impact toughness testing.

THE RESULTS AND EXPECTED IMPACT

The early stages of thermal cycling was found to play an important role on the demixing kinetics. For example, the ex situ specimens slowly cooled in the Gleeble showed a pronounced demixing, whereas this was not found in the fast cooled specimen. The difference disappears after 20h of subsequent aging at 475°C, but the initial condition alters the kinetics in the important early stage when embrittlement occurs. The Figure shows an example from the *in-situ* analysis of the 2507 duplex steel at 450 °C up to six hours of aging. The periodic length scale of the concentration fluctuations can be directly evaluated from the peak position as $2\pi/Q$ ($Å^{-1}$), while the amplitude requires model-dependent fitting.

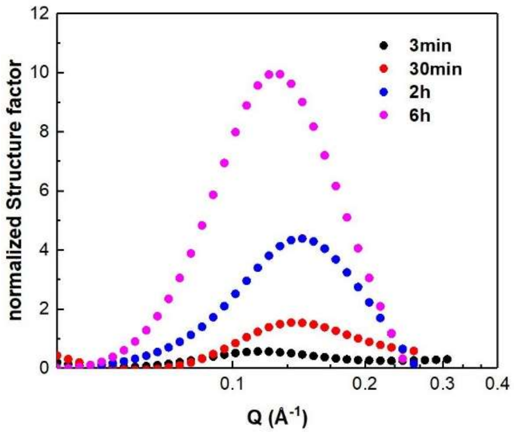

Figure. In-situ SANS data for 2507 during aging at 450 °C. Structure factor vs. Q [$Å^{-1}$].

The difference in demixing kinetics between alloy 2205 and 2507 were analysed. Finally, an unexpected parameter that can delay demixing was revealed in the in-situ experiments. This is currently explored for commercial protection.

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