Surface Chemistry and Diffusion of Trace and Alloying Elements during in Vacuum Thermal Deoxidation of Stainless Steel

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Introduction

Removal of the native surface oxide is an important initial step during vacuum brazing [1, 2]. The synchrotron based X-ray photoemission electron microscopy (XPEEM) has been extended its usual field to study the surface of steel [3, 4]. The detailed surface chemical composition and grain morphology of the common stainless steel grade 316L is imaged and spectroscopically analysed at several stages of in-vacuum annealing from room temperature up to 850°C. The findings can potentially be used to optimize the vacuum furnace programs, generating shorter lead times and more efficient manufacturing.

Methods

Spectroscopic photoemission and low energy electron microscopy (SPELEEM) experiments were performed at MAXPEEM beamline, a dedicated beamline at MAX IV Laboratory. An important question that can be answered using XPEEM is the distribution of elements and chemical species laterally across the surface or not. We have developed a polishing method that allows us to prepare flat and optimal samples for the SPELEEM system from realistic industrial materials.

Results

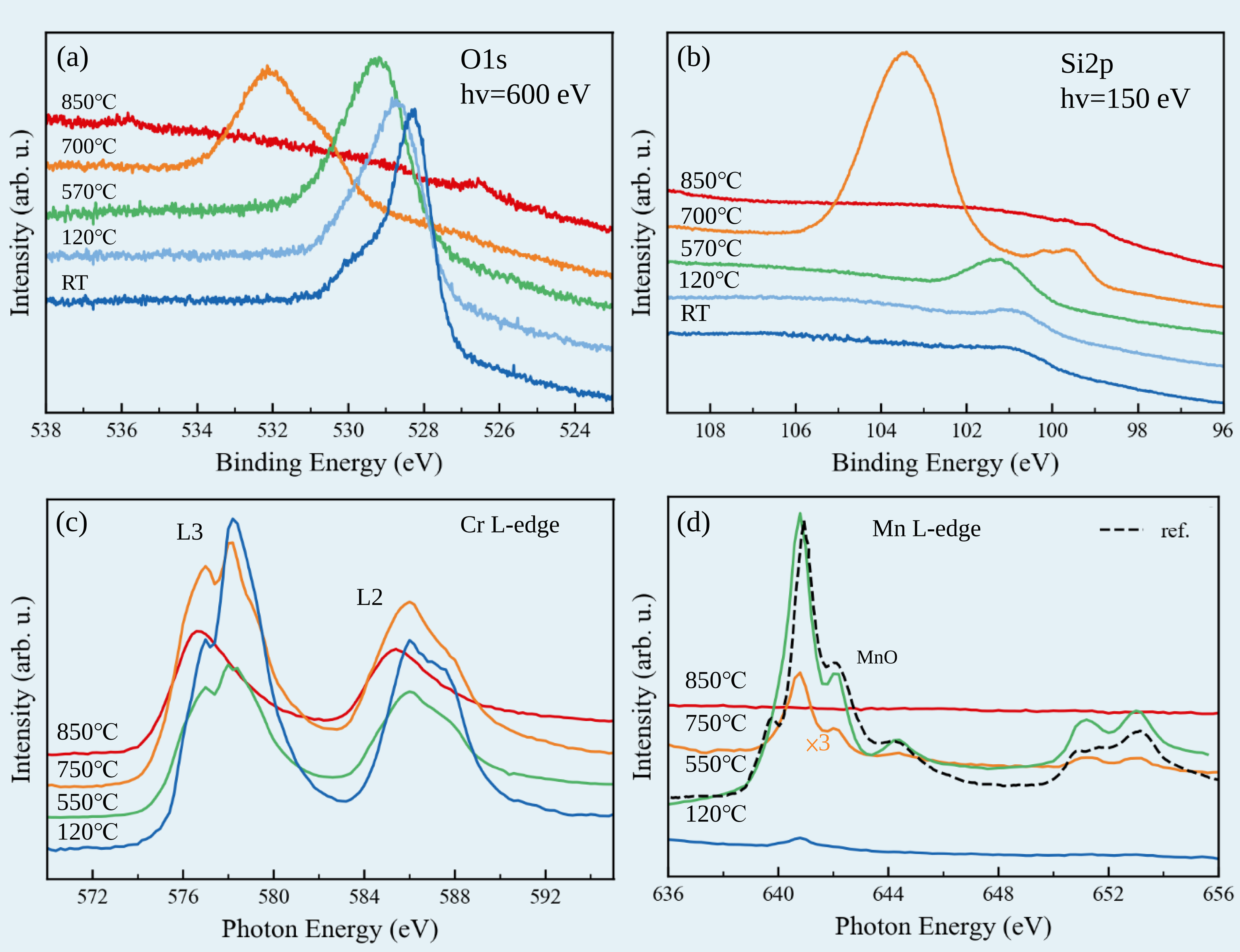

Figure 1. Core-level X ray Photoemission Spectra (XPS) for O1s (a) and Si2p (b) acquired after annealing at different temperatures. X ray Absorption Spectra (XAS) for Cr L-edge (c) and Mn L-edge (d) Cr L-edge spectra (c) upon annealing display a smooth transition from Cr oxide (the blue curve, 120°C) to a metallic Cr (the red curve, 850°C). Mn L-edge XAS show manganese segregation at the surface in the form of manganese oxide. The signal drops markedly and no traces of manganese can be detected at 850°C.

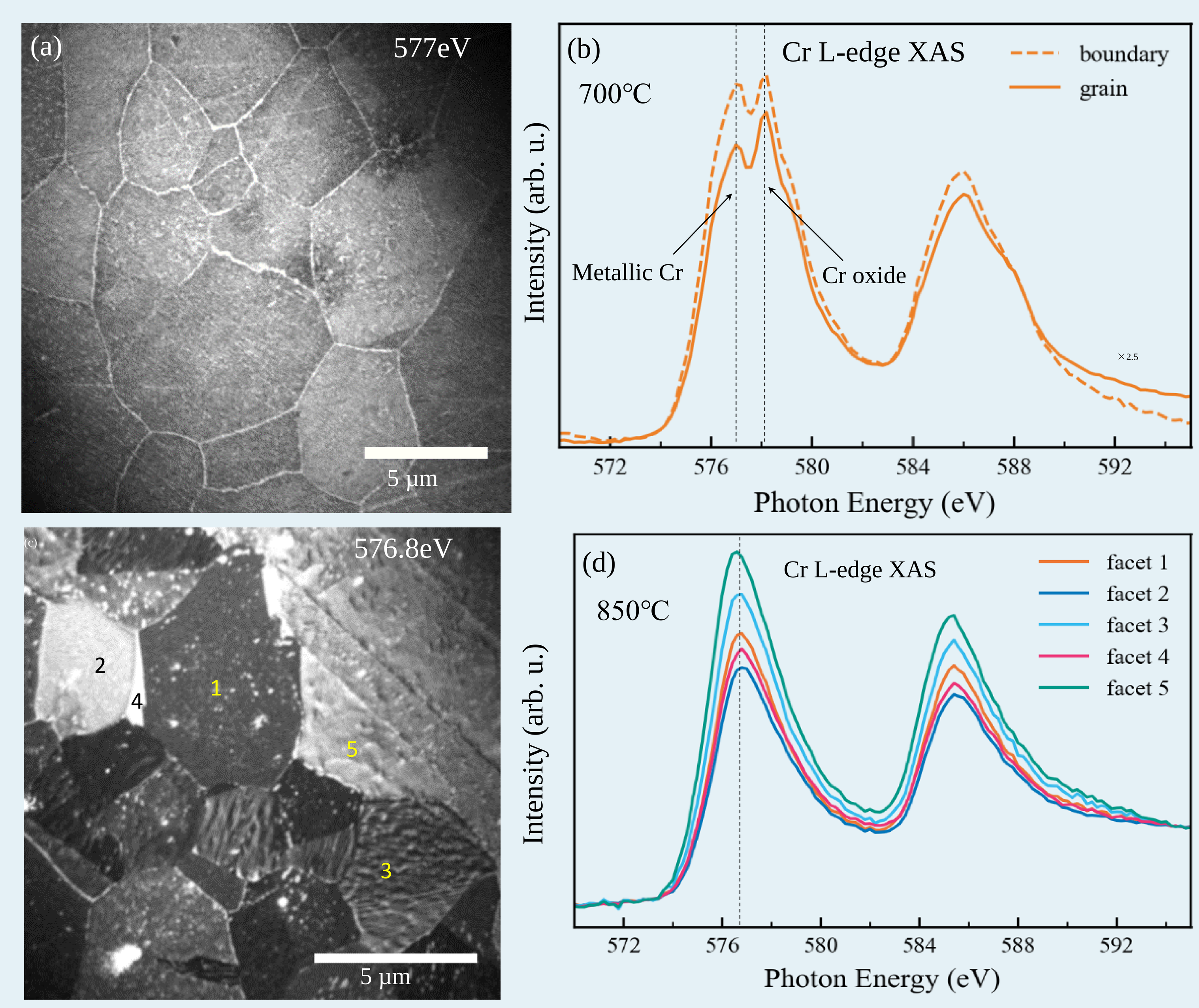

Figure 2. Chemical mapping of the grain boundaries. (a) XPEEM image at photon energy 577 eV after annealing at 700°C. (b) Cr L-edge XAS from grain boundaries and grains regions. (c) XPEEM image acquired at the edge (photon energy 576.8 eV) of the Cr absorption spectra after annealing at 850°C. (d) XAS spectra from five different grains labelled in (c). The intensities of the absorption spectra show the different Cr content in different grains.

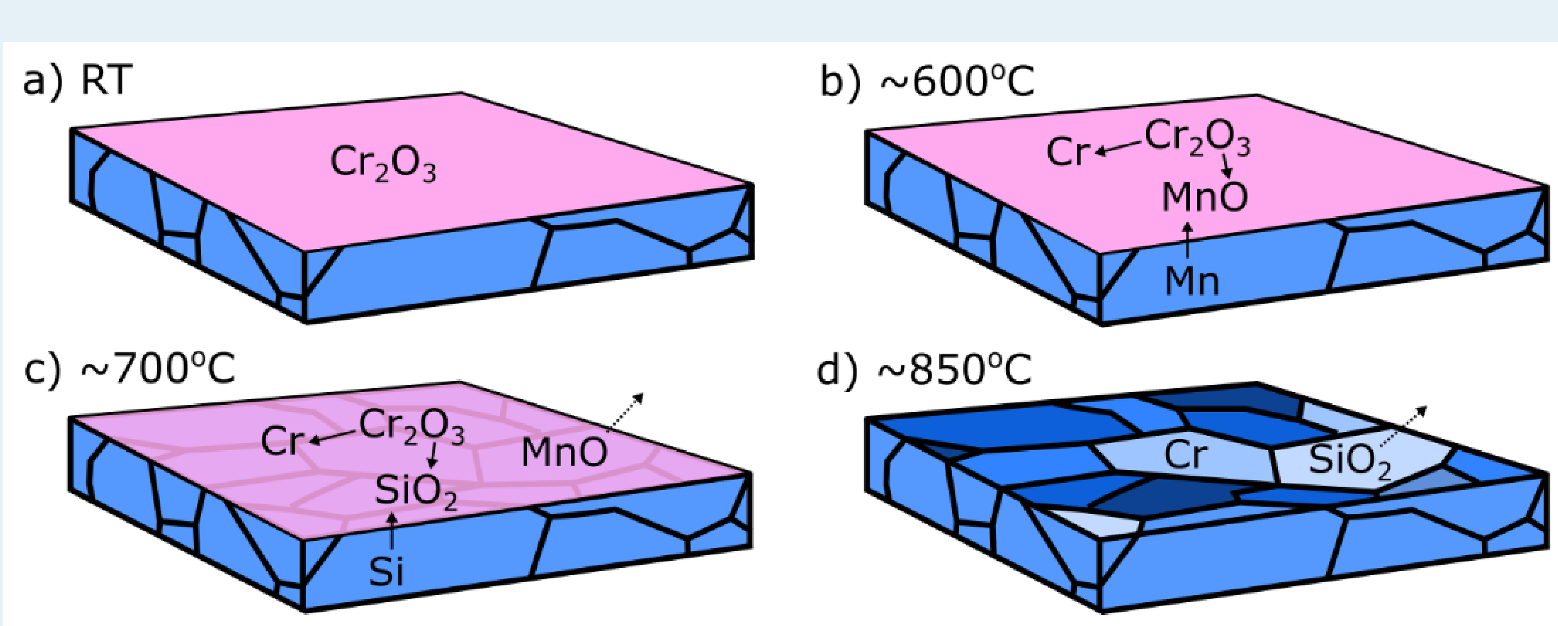

Figure 3. Schematic representation of a deoxidation model which is compatible with the findings: a) at RT the surface is covered by an amorphous and homogeneous Cr based oxide. b) at ~600°C the Cr oxide signal weakens, metallic Cr and MnO appear due to Mn diffusion from the bulk. c) at ~700°C the Cr oxide signal continues to weaken, metallic Cr signal grows, MnO signal weaken as it evaporates and SiO_2 signal appear due to Si diffusion from the bulk and O capture from the Cr oxides. d) at approximately 850°C SiO_2 signal disappear and the metallic Cr signal indicate slightly different Cr content on different grains and at boundaries.

Conclusions

- Trace and alloying elements can diffuse to the surface and influence surface deoxidation.
- Both Mn and Si appear as oxides taking up O from Cr oxide, while Cr oxide become metallic Cr.
- Surface concentration of first Mn and then Si increase significantly when annealing to 500°C and 700°C, respectively.
- Deoxidation of Cr occurs faster at the grain boundaries and the final Cr metal surface content vary between the grains.

References

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