

Development of the Operando Atom Probe: The Influence of the electric field on Fe oxidation

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Heterogeneous catalysis is one of the pillars of the chemical industry and the “*green chemistry*” concept. However, the sparse understanding of surface mechanisms at molecular scale impedes development of high-performance catalysts and requires development of new tools and methodologies. Heterogeneous catalysis is a surface phenomenon and in order to maximize the active surface available for a minimum of bulk material used, catalyst are presented as supported nanoparticles. Nanoparticle surfaces are the site of reaction processes and their size/shape drastically influence the catalyst’s features and its performances towards the reaction of interest. Oxidation and corrosion of metals is another research field with very similar research questions. Metals used in variety of structural applications from nuclear reactor internals to automobiles degrade due to oxidation and corrosion. Both oxidation and corrosion are also fundamentally surface reaction mechanisms, with clear parallels to the heterogenous catalysis reaction mechanisms.

Starting from very sharp needles, whose needle apexes are similar to model nanoparticles by their sizes and shapes, we are able to explore these surface mechanisms at the nanoscale with the use of Atom Probe Microscopy (APM) techniques. From Field Emission Microscopy to Atom Probe Tomography, APM techniques were extensively used from the 1980s to track and identify reactions mechanisms. Our most recent development led us to adopt the approach introduced by Block et al. with their Pulsed Field Desorption Mass Spectrometry (PFDMS), using the electric field pulses frequencies applied in atom probe techniques to explore the surface dynamics.

In this work we dedicated our efforts to understand the electric field driven oxidation of Fe and Fe-alloys. A clean Fe needle sample is exposed at 2.5×10^{-9} mbar of pure oxygen while held under an applied electric field inside the APT analysis chamber and the change in surface composition was measured as a function of time by running the atom probe at 302.9K specimen temperature. The voltage pulse intensity is kept constant while the DC standing voltage is modified progressively to observe the impact of the latter on the oxide formation. The figure 1 shows the schematic of the operando APT set up. From the experiments we directly observed that the V_{stat} drastically changed the oxide composition measured in APT. For higher V_{stat} , a relatively pure Fe surface was observed. on changing the V_{stat} a direct change in the oxide stoichiometry was measured, establishing a powerful method to quantitatively study the electric field assisted oxidation of Fe. This approach can now be used for Fe based metallic alloys to reveal the oxidation mechanisms under the applied electric field conditions [1].

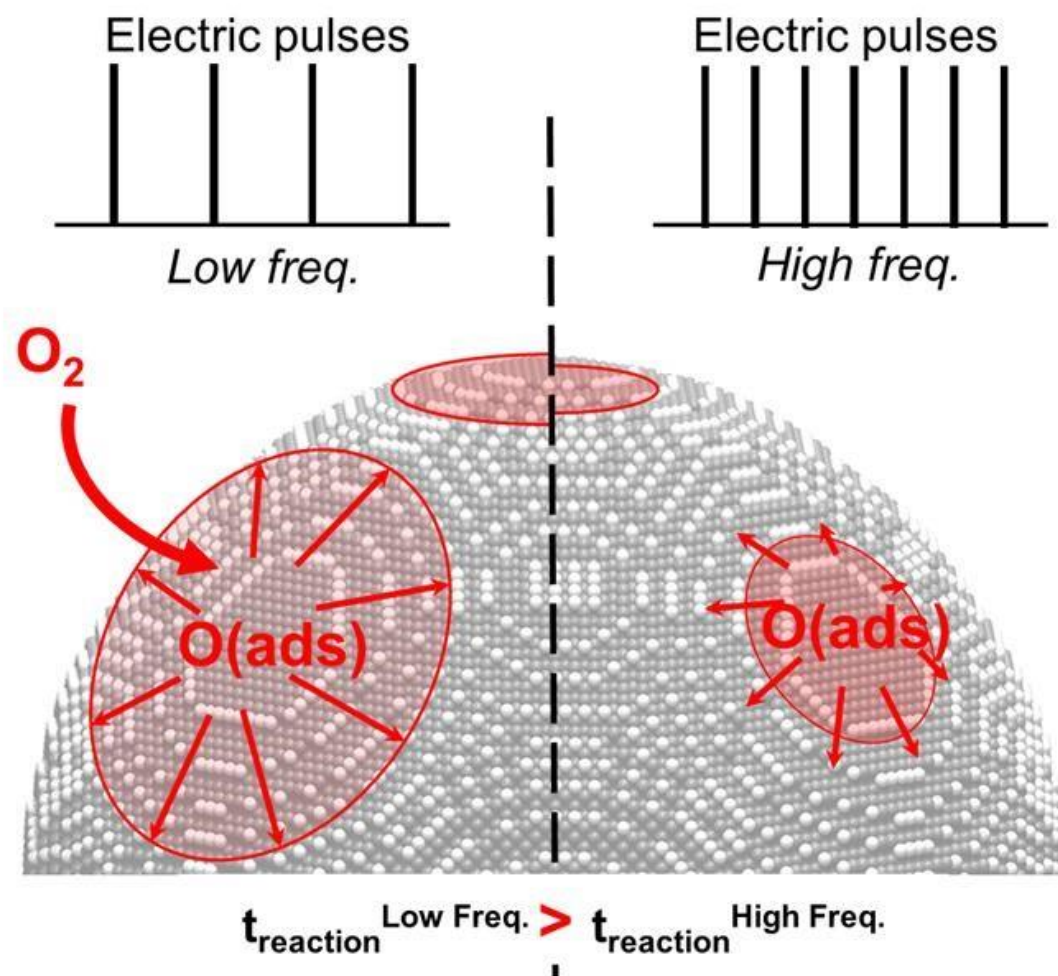


Figure 1. Schematic explanation of electric field assisted oxidation of Fe during operando APT

References

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