Contract No:

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Kelvin Probe Force Microscopy for High-Resolution Imaging of Hydrogen in Steel Alloys

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Abstract

Kelvin probe force microscopy (**KPFM**) was used to image and co-locate the presence of hydrogen in stainless steel (SS) with microstructure. Various SS samples were investigated, including forged, welded, LENS® fabricated, and pinch welded materials.

KPFM images the local work function of a surface as a function of spatial position using ***atomic force microscopy***. KPFM is capable of delivering high spatial (~10nm) and potential (~5mV) resolution maps characterizing the microstructure and the locally varying work function of surfaces. Hydrogen segregated at the surface of stainless steels (SS) increases the local work function by either reducing tensile strain or by reducing the chemical potential of surface species. In a process known as hydrogen embrittlement, hydrogen segregates to and becomes trapped at extended defects (e.g. dislocations, boundaries, pores), which leads to the initiation of cracking and can result in structural failure. Hydrogen storage necessitates a proper understanding of the mechanisms of hydrogen embrittlement in SS. Developing advanced characterization techniques for hydrogen embrittlement is critical to identify long term solutions for materials exposed to hydrogen. Current techniques of observing hydrogen embrittlement involve mechanical testing, and optical microscopy with low resolution. Advanced microstructural imaging techniques capable of resolving features down to the nanometer scale are needed. KPFM provides high resolution imaging for the characterization and development of stainless steel components for hydrogen storage.

W. Melitz et al. / Surface Science Reports 66 (2011) 1–27

Use advanced imaging techniques to better understand the effect of hydrogen in stainless steel

Overview & Relevance

Project start date: 10.01.2017
Project end date: 09.30.2019
Percent complete: ***50%***

Budget

- Total project funding: $395,000
- Funding received in FY18: $175,000
- Total funding planned for FY19: $220,000

Overall Objectives:

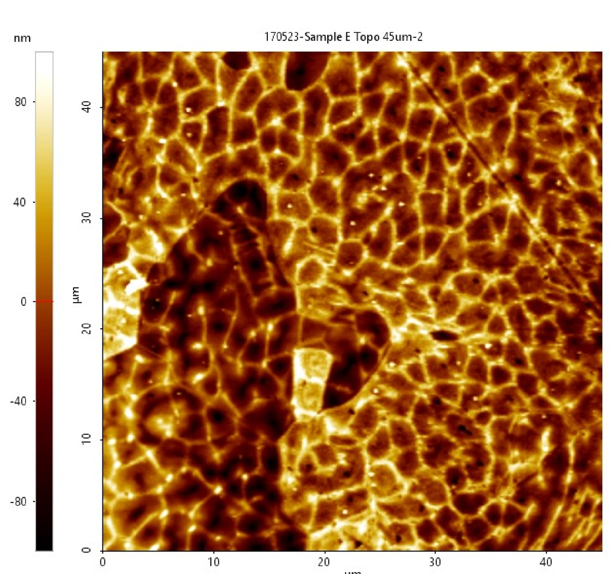
- Understand the behavior of hydrogen in materials used for hydrogen storage.
- Correlate hydrogen embrittlement with microstructural features.
- Image the presence of hydrogen at the surface as a function of microstructural defects.
- Attribute hydrogen segregation to sample growth, fabrication, treatment methods, and specific defects.

Barriers addressed:

- Advanced imaging technique visually identifies the presence of hydrogen.
- High resolution imaging technique.
- Configured dry nitrogen environmental chamber to reduce screening potential from humid air.

Objectives in this reporting period:

- Characterized microstructural differences between forged, welded, LENS®, and pinch welded SS specimens.
- Developed sample prep methodology, and nitrogen environment for AFM.
- Imaged hydrogen charged and uncharged samples, and confirmed hydrogen segregation to grain and cell boundaries.



Use Kelvin Probe Force Microscopy to investigate H in defects

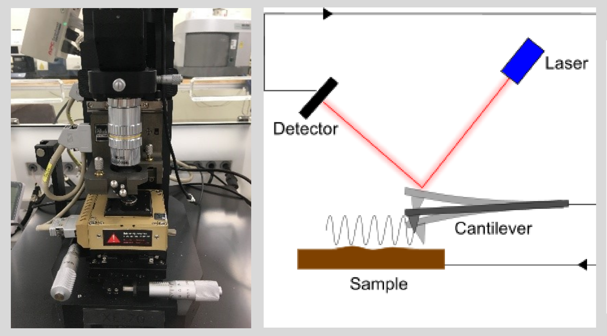


Figure 2: Atomic force microscope in HTRL lab 138, and schematic of SKPM setup (http://www.dme-spm.com).

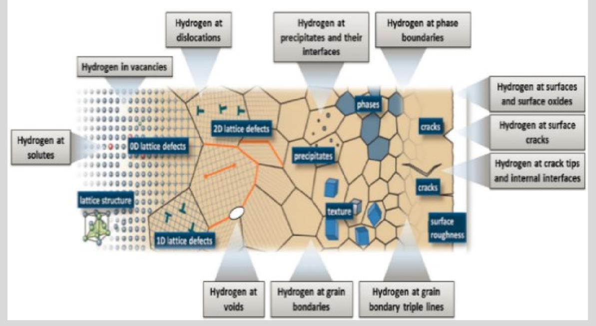
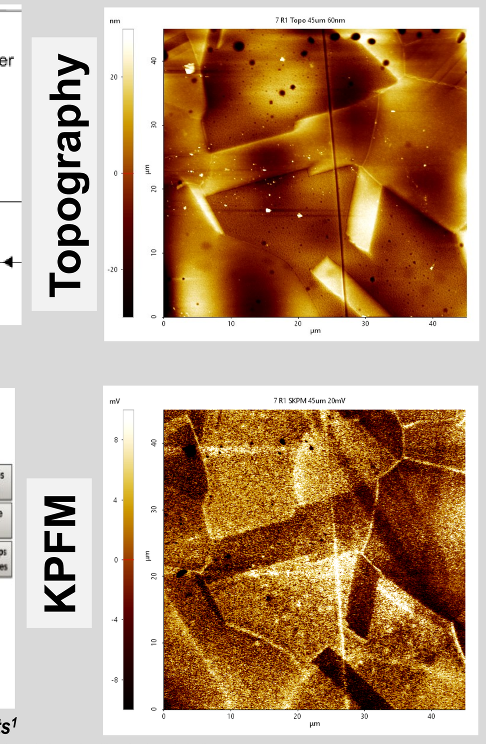


Figure 1: AFM of SS microstructure and hydrogen in extended defects[1]

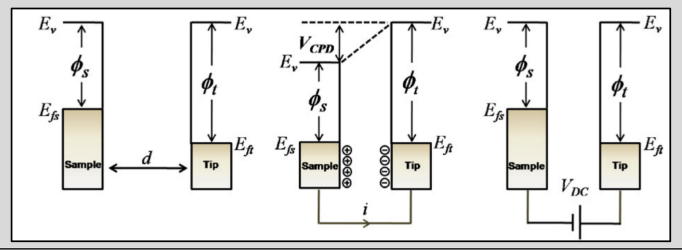
[1]M. Koyoma et al., Mater. Sci. Tech. Recent progress in microstructural hydrogen mapping in steels: quantification, kinetic analysis, and multi-scale characterization



Approach

- Atomic force microscope uses superposition of AC and DC voltage signals to modulate electrostatic force between AFM tip and sample.
- Contact potential difference (V_{CPD}) between sample and AFM tip gives ***local work function (ϕ)***.
- High resolution of AFM image allows for **correlation of microstructure with H segregation**.

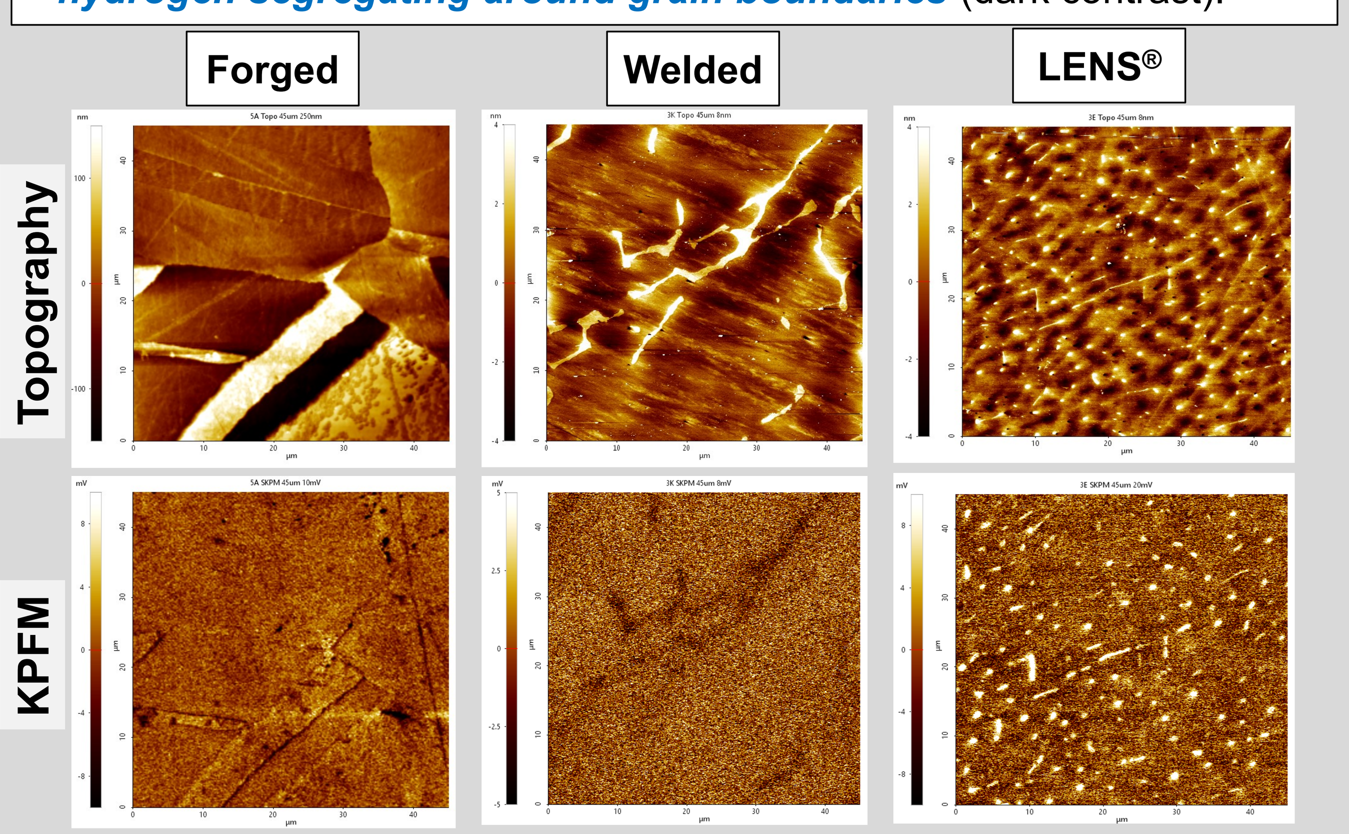
$$V_{CPD} = \phi_{tip} - \phi_{sample}$$



Milestones	Progress Notes	% Complete
Milestone 1 (FY18): • KPFM development at SRNL and sample preparation optimization • Develop inert nitrogen atmosphere for AFM enclosure	• Established KPFM functionality. • Engineered N_2 atmosphere and applicable safety documentation	100%
Milestone 2 (FY18): • Comparing AM and forged SS • Studying effects of passivation on SS surface • Studying microstructure of pinch welds	• Characterized microstructures of forged, welded, LENS, and pinch weld samples.	75%
Milestone 3 (FY19): • Electrochemical charging • Magnetic force microscopy • Deuterium charging • Temperature and time-resolved thermal desorption studies	• Setting up electrochemical charging. • Acquired equipment for magnetic force microscopy.	10%
Milestone 4 (FY19): • Mid-year report and final report		50%

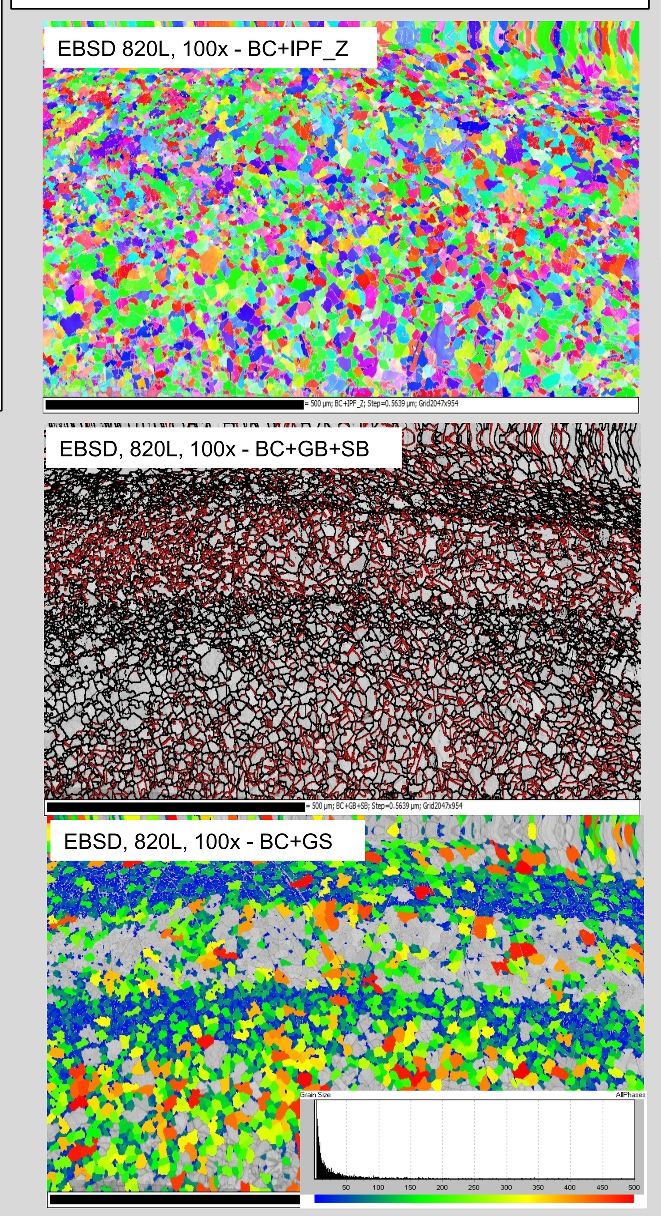
Technical Progress

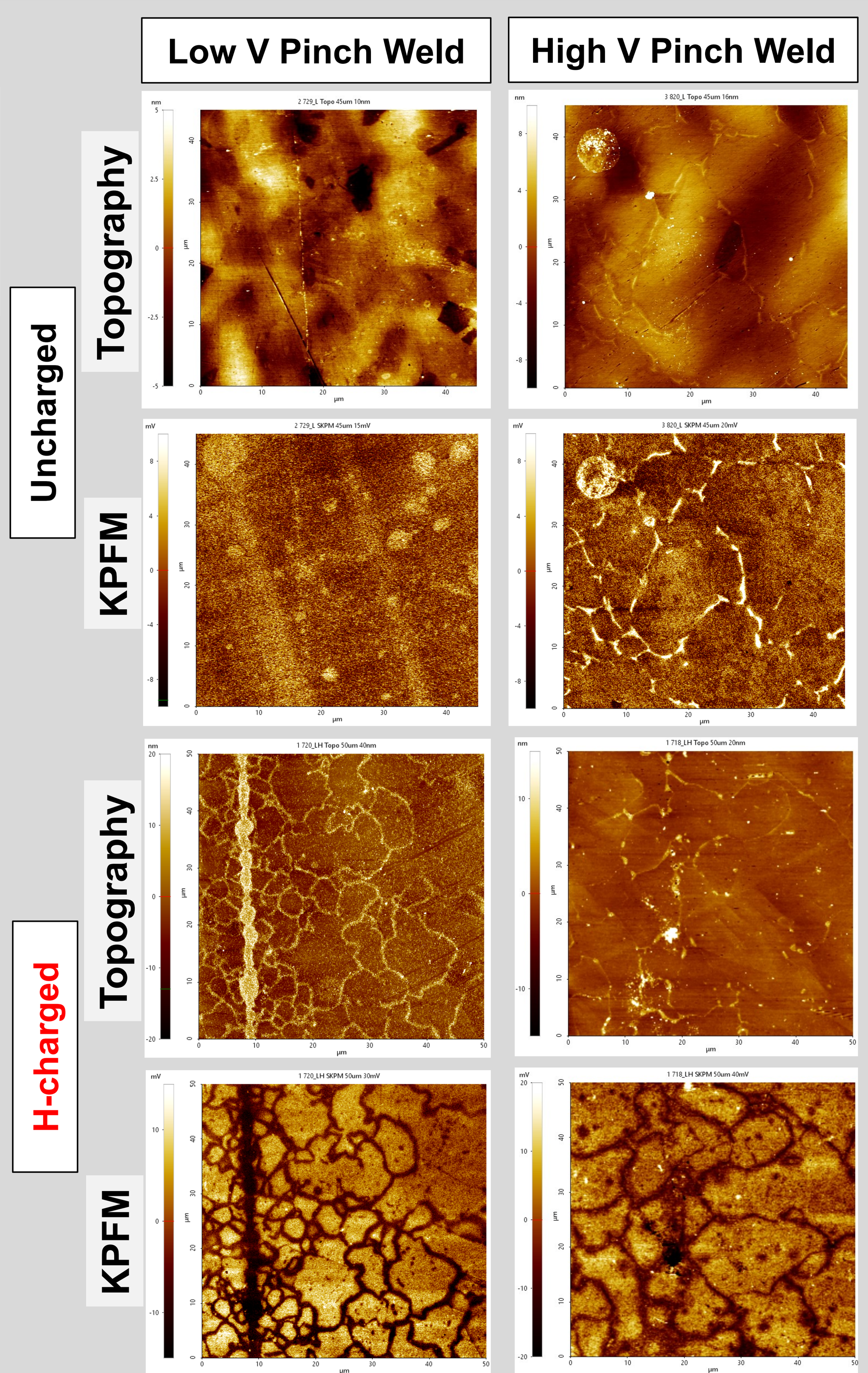
- Characterized microstructure of various types of specimens.
- ***Hydrogen is expected to increase the work function***, which is evidenced by a decrease in the CPD signal.
  - ***Increase in H concentration = dark contrast in KPFM signal.***
- Forged, welded, LENS®, and pinch weld materials were H-charged at 350°C with 17 Mpa of hydrogen gas for about 2 weeks.
- KPFM data for forged and welded show ferritic regions.
- KPFM data for LENS® show elemental variation.
- KPFM data for ***uncharged*** high and low voltage pinch welds show microstructural variation similar to EBSD maps.
- KPFM data for charged high and low voltage pinch welds ***shows hydrogen segregating around grain boundaries*** (dark contrast).



Electron backscatter diffraction (EBSD) on high V pinch weld:

- Weld is in the center of the image.
- Bands of large and small grain size distribution run parallel to weld boundary.





Collaborations

- Savannah River National Laboratory: Materials Science and Technology (Science and Technology), and Non-proliferation Technologies (National Security)
- Sandia National Laboratory
- Los Alamos National Laboratory
- Kansas City National Security Campus

Remaining Challenges and Barriers

- Understand isotopic effects of deuterium/ tritium.
- Correlate direct imaging KPFM with indirect quantitative techniques for hydrogen studies.
- Increase resolution of AFM and KPFM.
- Determine the role kinetics play in H-charging and redistribution after charging.
- Image specifically engineered defects and microstructures.

Proposed Future Work

- Develop system for deuterium/ tritium work.
- Parallel KPFM, TDS (thermal desorption spectroscopy), and magnetic force microscopy (MFM) studies.
- Incorporate Isolation table to increase resolution.
- Incorporated electrochemical H-charging and time-resolved studies for H decay studies.
- Improve sample preparation methods.

Project Summary

- Demonstrated KPFM at SRNL as a technique to image hydrogen in metals and alloys.
- Characterized microstructures of forged, welded, LENS®, and pinch weld materials intended for use in hydrogen storage.
- Paired KPFM with AFM, SEM, EDS and EBSD to characterize microstructures.
- Investigated the role of welding parameters in hydrogen segregation in pinch welds.

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