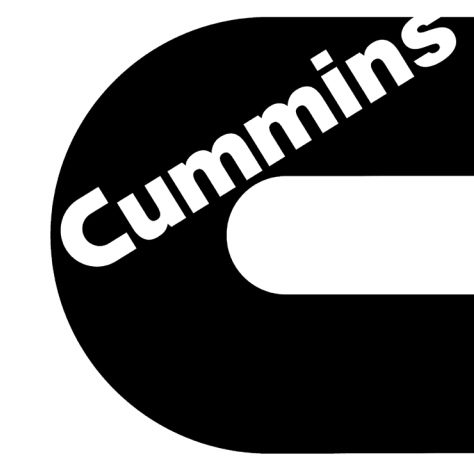


Triboconditioning® is a mechanochemical surface treatment that can reduce friction and wear, offering a lower-cost alternative to other wear-reducing surface treatments. However, the effect that this surface treatment has on susceptibility to hydrogen embrittlement, a common failure mode, remains to be characterized. In this work, both treated and untreated specimens are deliberately charged with hydrogen, and their mechanical properties, hydrogen trapping behavior, and surface roughness are evaluated to assess the impact of Triboconditioning® on hydrogen embrittlement susceptibility.

This work is sponsored by Cummins Inc.,
 Columbus, IN



Background

Hydrogen embrittlement is the process by which atomic hydrogen is absorbed by a metal material leading to decreased ductility and in some cases, material failure far below the nominal tensile strength [1]. Hydrogen can readily be introduced into a material via manufacturing processes or in service leading to premature material failure. Triboconditioning® is a mechanochemical surface treatment patented by Tribonex. The treatment combines mechanical burnishing with the deposition of a solid-state lubricant to reduce friction and wear. The purpose of this work is to investigate if this treatment has an impact on susceptibility to hydrogen embrittlement. To do this, we will deliberately charge treated and untreated specimens via an electrochemical process and investigate the impact this has on mechanical properties, surface roughness, and hydrogen absorption.

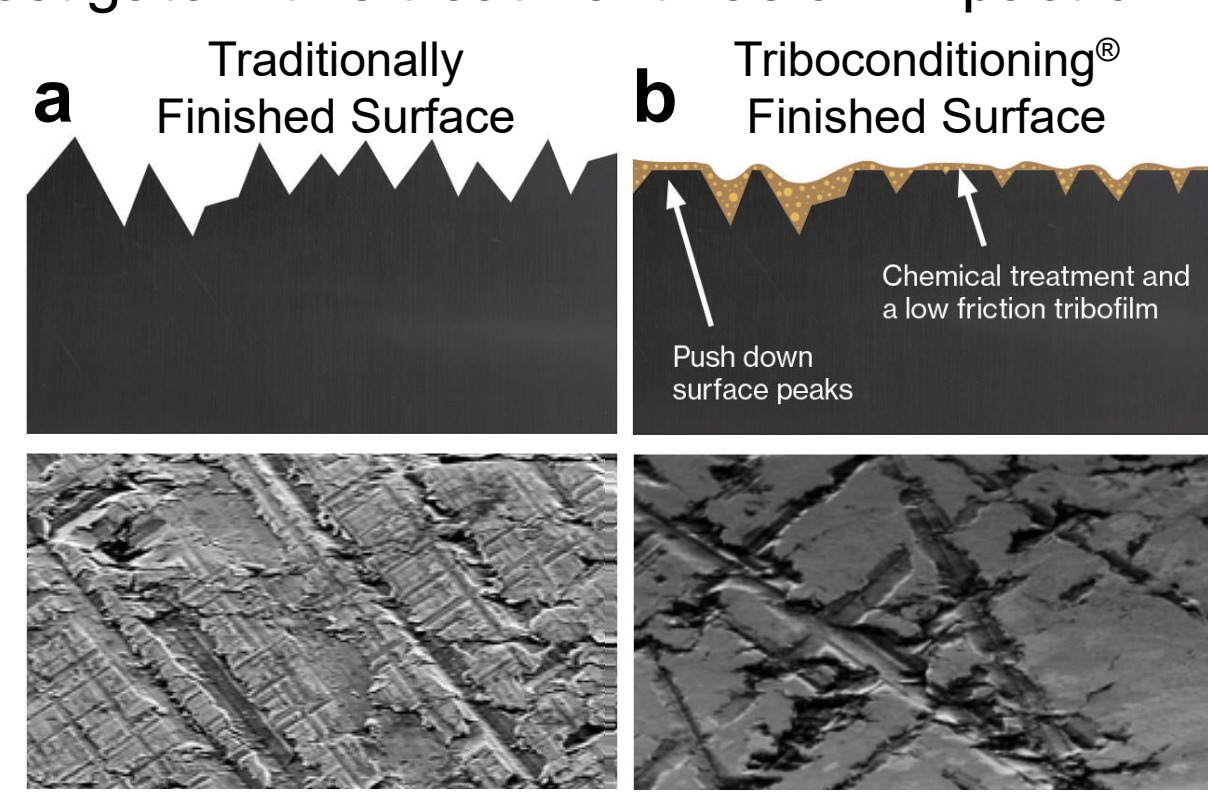


Figure 1: Diagrams and micrographs of a) a traditionally finished surface and b) a Triboconditioning® finished surface [2]

Methods

Specimens

Test specimens were designed by the senior design team and machined from 4130 quench and tempered steel by the industry partner and treated by Tribonex. Flat bar specimens (Fig. 2a) were used for three-point bend testing, optical profilometry, thermal desorption spectroscopy, and energy dispersive spectroscopy. Round notched specimens (Fig. 2b) were used for notched tensile testing with in-situ charging and fractography.

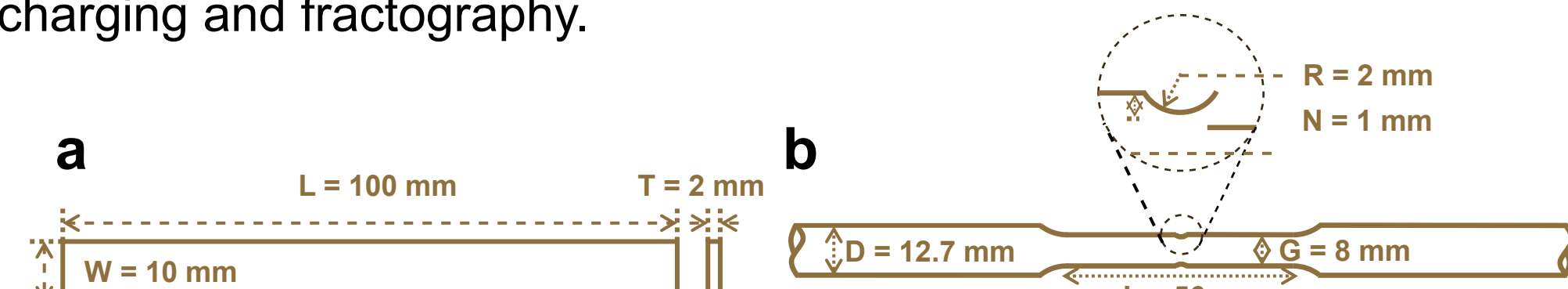


Figure 2: Diagrams of a) flat bar and b) notched round specimens.

Electrolytic Hydrogen Charging

Flat bar specimens were cathodically charged with hydrogen for varying times in a 0.5 M sulfuric acid solution with 10 g/L of thiourea at a current density of 10 mA/cm² at room temperature. Notched round specimens were pre-charged in the same solution but at 50 mA/cm² for 24 hours prior to tensile testing with additional in-situ charging. A diagram of the in-situ charging apparatus is shown in Figure 3.

Mechanical Testing

Three-point bend tests were conducted using an ADMET screw-driven load frame to quantify the rapid reduction in ductility at short charging times and fast strain rates (10⁻³ /s). Slow strain rate (10⁻⁵ /s) notched tensile tests which are more standard for evaluation of hydrogen embrittlement were conducted with a Syntec 30D load frame and in-situ hydrogen charging to evaluate the effect of sustained hydrogen exposure.

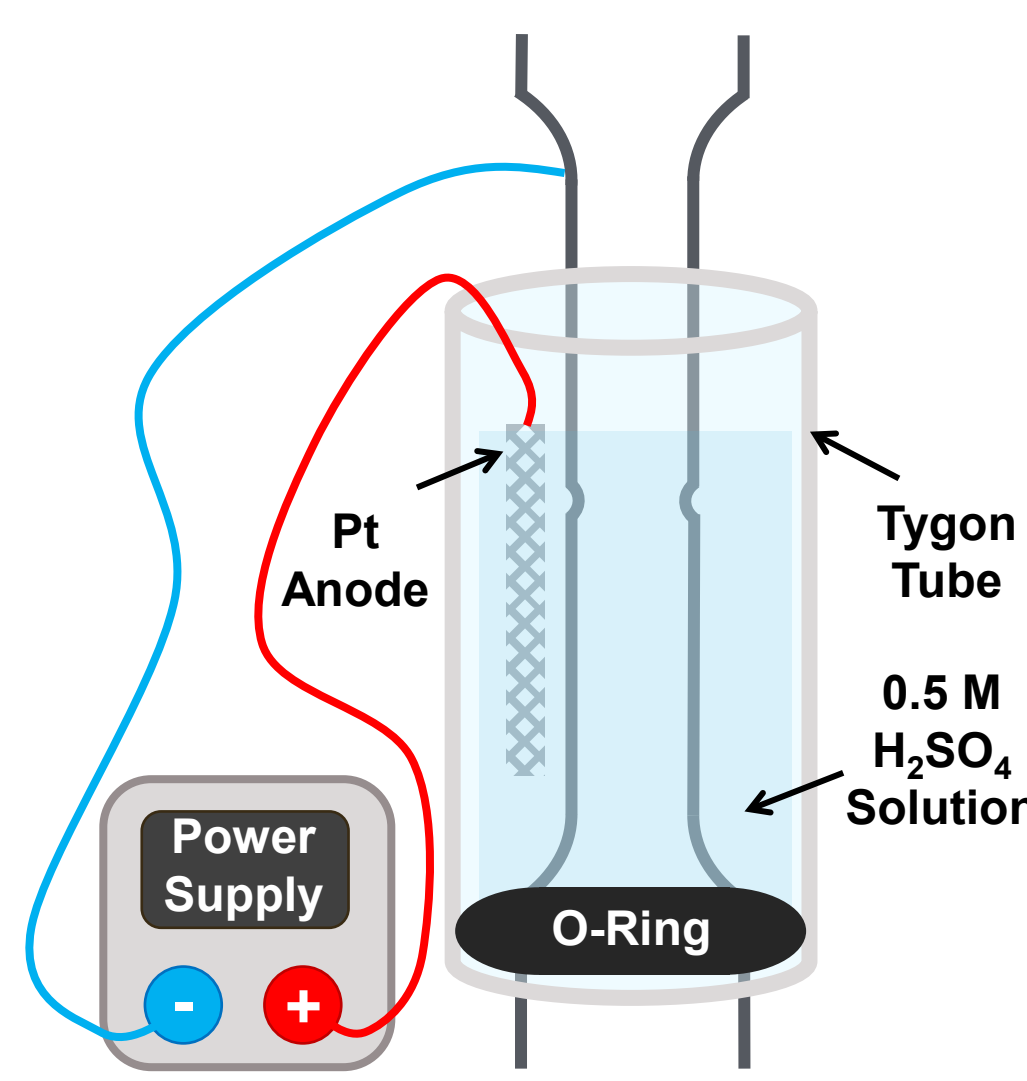


Figure 3: Diagram of in-situ hydrogen charging apparatus for tensile testing

Thermal Desorption Spectroscopy (TDS)

Thermal desorption spectroscopy was conducted using an Eltra H-500 hydrogen analyzer. Spectra are analyzed to compare hydrogen trapping behavior between treated and untreated specimens.

Optical Profilometry

Optical profilometry was conducted using a Zygo ZeScope optical profilometer to characterize the surface roughness of specimens, which may impact hydrogen charging and mechanical properties.

Energy Dispersive Spectroscopy (EDS)

EDS was conducted using an SNE Alpha SEM with a Bruker Xflash Detector to quantify the possible degradation of the solid-state lubricant with charging.

Fractography

Fracture surfaces of notched round specimens were imaged using an SNE Alpha SEM in secondary electron mode to compare the relative brittle and ductile character of the fracture surfaces.

Results & Discussion

Three-Point Bend Testing and Thermal Desorption Spectroscopy

- Significant reduction in elongation at break indicative of embrittlement is observed for all surface treatments at all charging times.
- Hydrogen content evolves according to a logarithmic curve and embrittlement is achieved with as little as ~ 2.5 ppm of hydrogen.

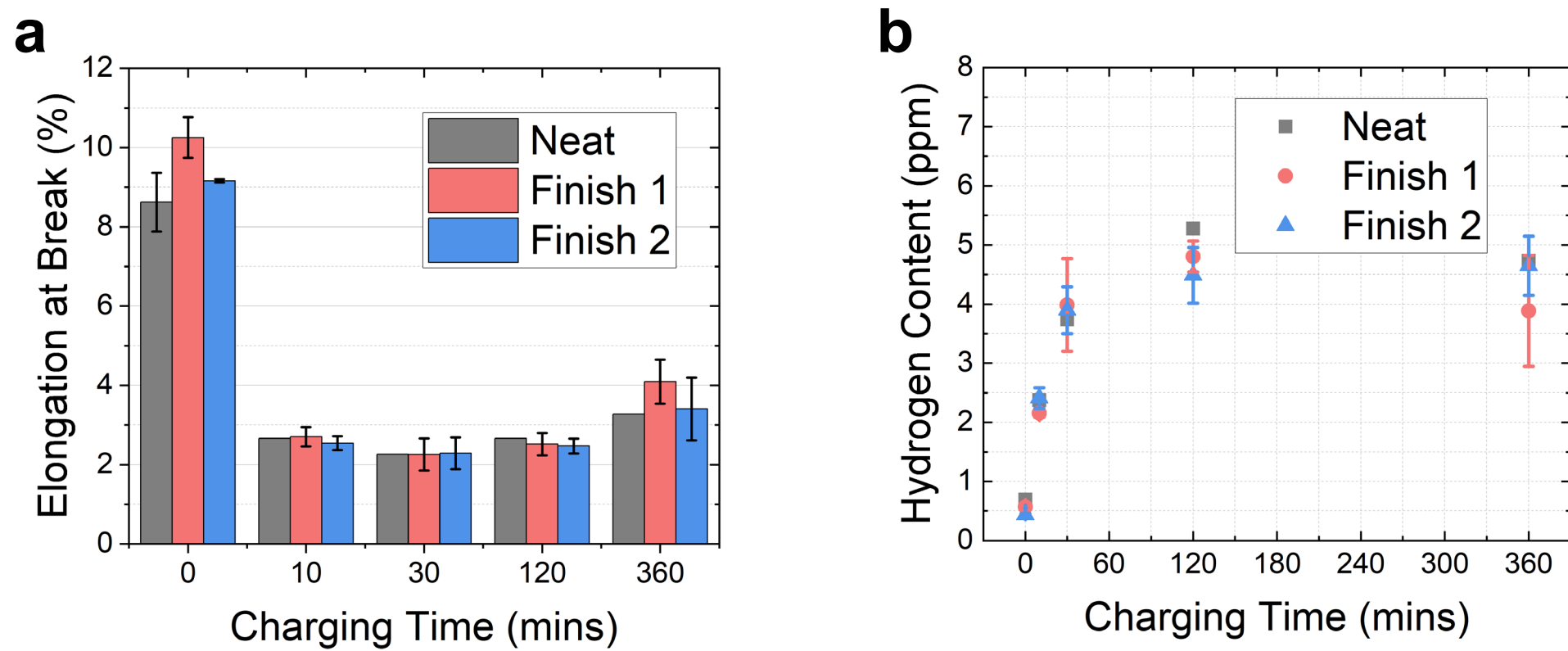


Figure 4: Plots of a) elongation at break and b) hydrogen content as a function of charging time for neat, finish 1, and finish 2 specimens. Error bars indicate the standard deviation

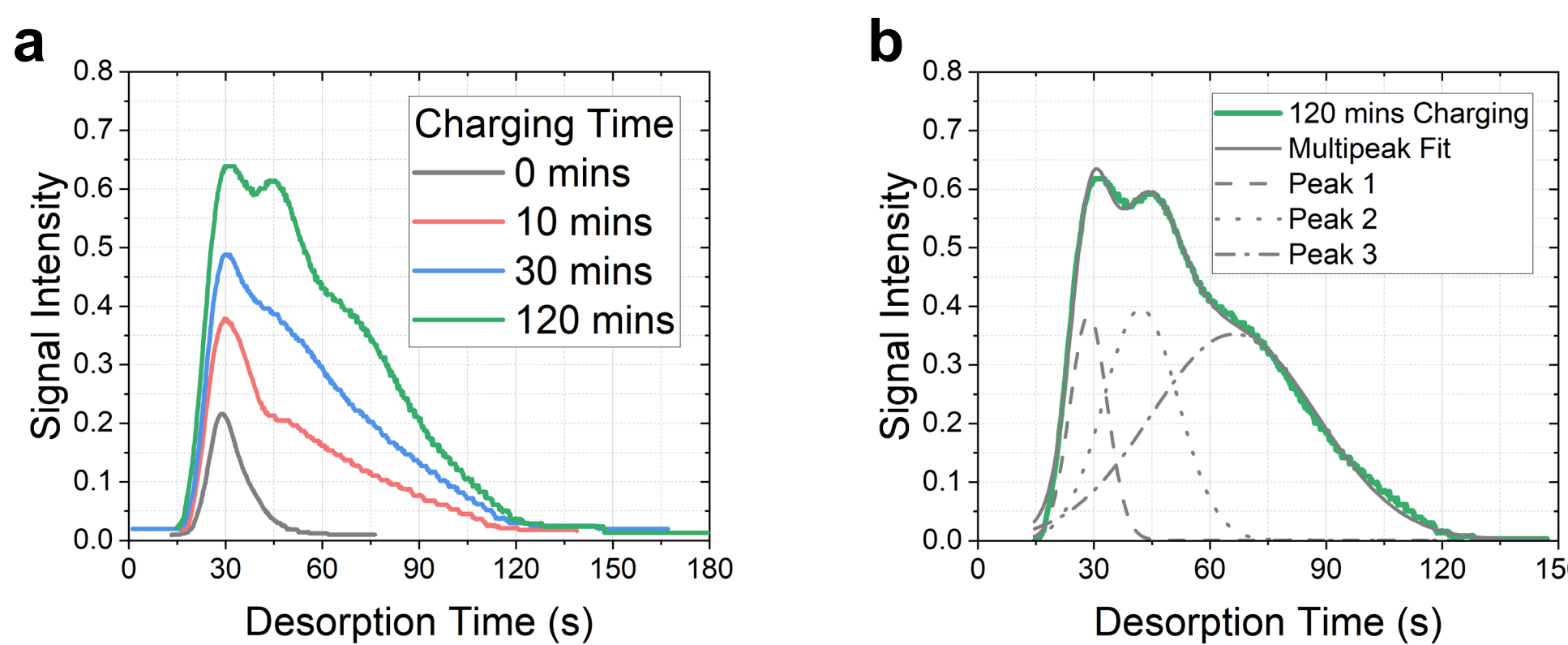


Figure 5: Plots of a) representative desorption spectra for finish 2 specimens at varying charging times and b) multiplex fitting used to determine the relative amount of hydrogen desorbed from each peak.

- As charging proceeds, multiple peaks appear at approximately the same desorption times for each treatment with peaks corresponding to hydrogen trapping sites in the material.
- Variance in the proportion of hydrogen at peaks 2 and 3 for finish 1 specimens is observed, indicating a possible difference in trapping behavior.

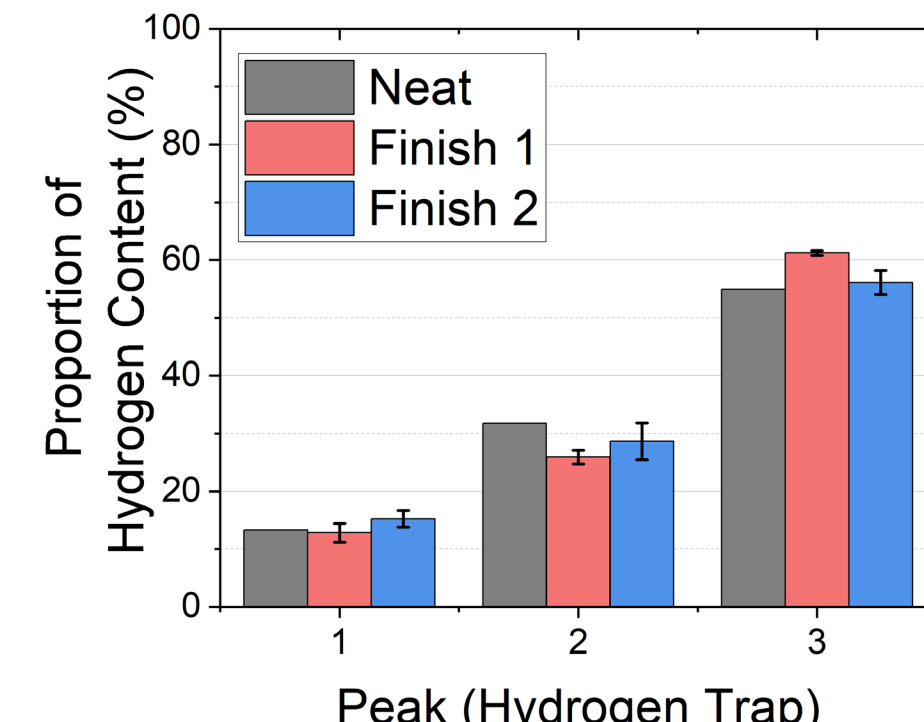


Figure 6: Bar graph of the proportion of hydrogen content desorbed from each peak for each treatment after 120 minutes of charging.

Notched Tensile Testing

- Both elongation at break and tensile strength are significantly reduced with hydrogen charging.
- There is no observable difference between the behavior of treated and untreated specimens.
- Uncharged specimens exhibit ductile necking while charged specimens fail prior to necking indicating more brittle behavior.

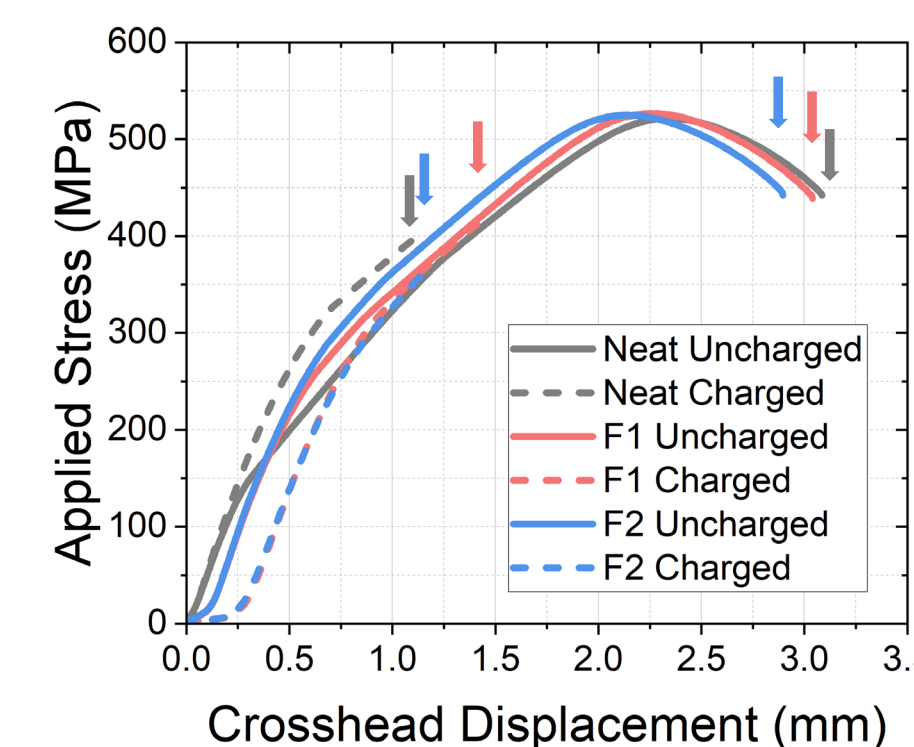


Figure 7: Plot of applied stress versus crosshead displacement for charged and uncharged specimens of each surface treatment.

Fractography

- The uncharged specimen fracture surface (Fig. 8a) displays mostly dimples with only some evidence of transgranular and intergranular fracture indicating an overall ductile failure [3].
- The charged specimen fracture surface (Fig. 8b) displays mostly transgranular fracture with some evidence of intergranular fracture indicating an overall brittle failure [3].

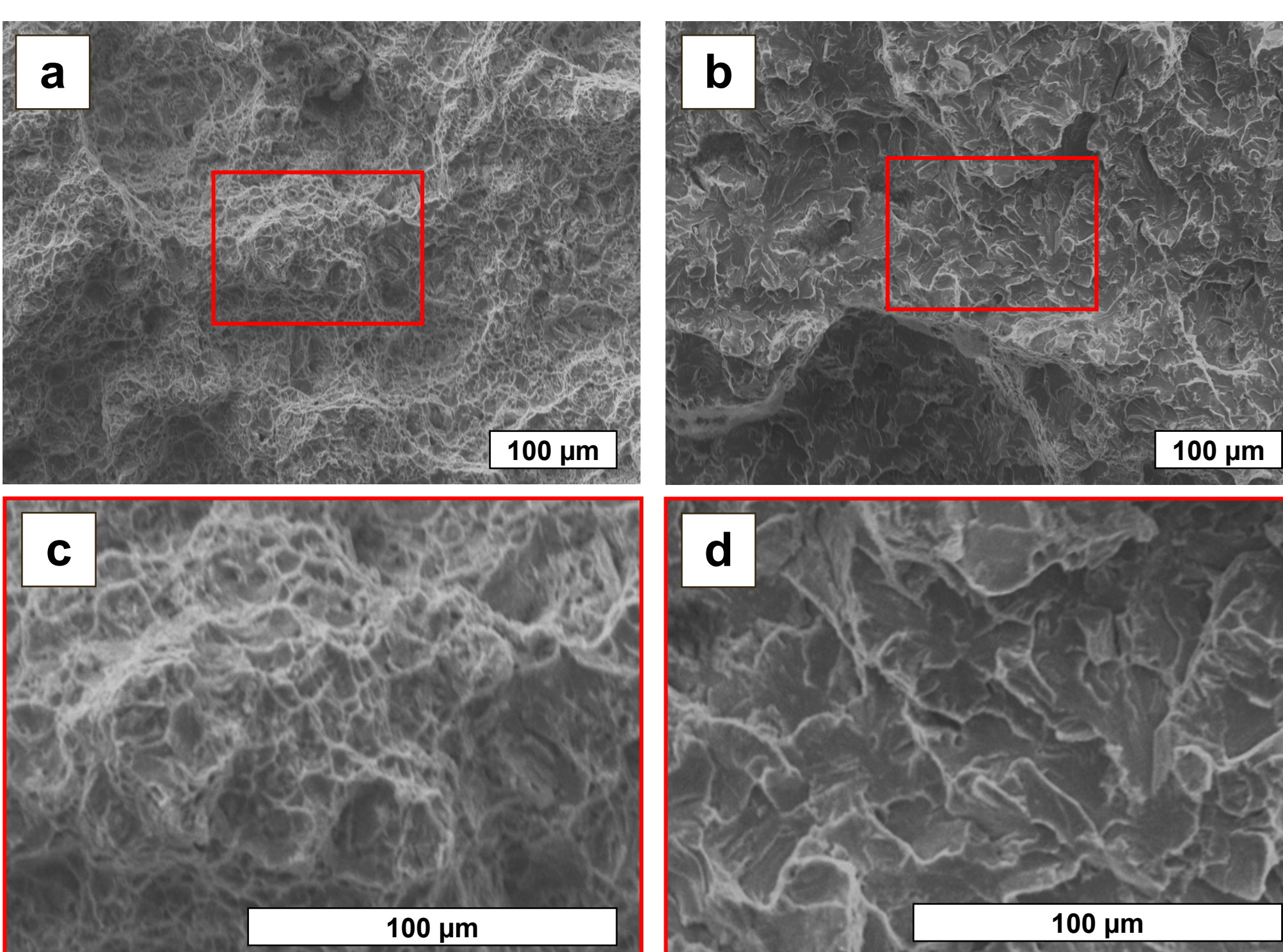


Figure 8: Micrographs of the fracture surfaces of a) uncharged and b) charged notched round specimens with finish 1. Higher magnification micrographs of the c) uncharged and d) charged fracture surfaces displaying dimpling and transgranular fracture respectively

Results & Discussion Cont.

Energy Dispersive Spectroscopy (EDS)

- The concentration of "element X", one component of the solid-state lubricant, is reduced with charging.
- After 30 minutes of charging, full embrittlement is achieved yet the concentration of element X is only slightly reduced indicating that degradation is not the cause of susceptibility to hydrogen embrittlement.

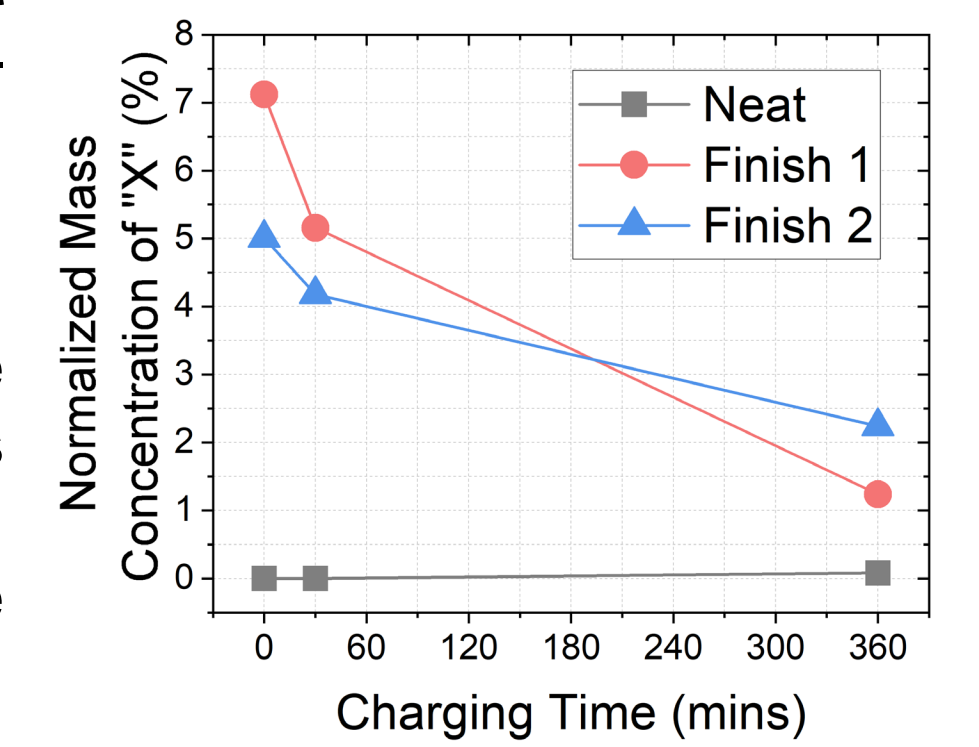


Figure 9: Plot of the normalized mass concentration of element X as a function of charging time for specimens of each treatment.

Optical Profilometry

Table 1: Areal average roughness of neat, finish 1 and finish 2 treatments before charging.

Neat	1.7092
Finish 1	
Finish 2	

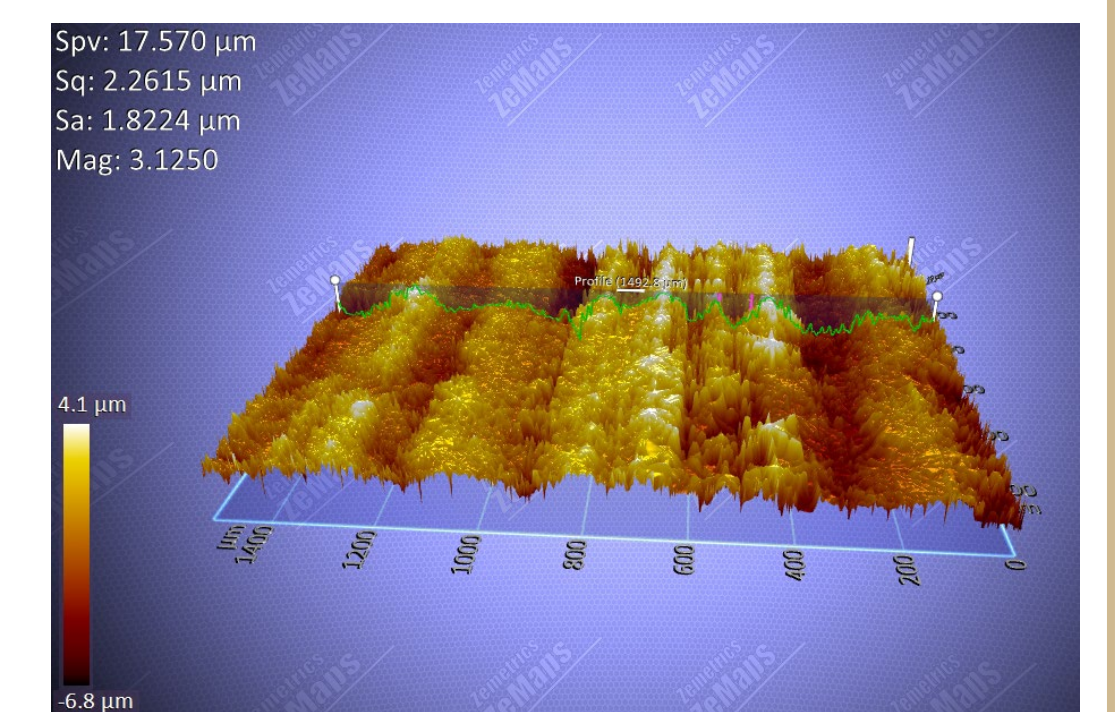


Figure 10: Representative optical profilometer map of neat, uncharged sample at 10x magnification.

- Optical profilometry was performed on uncharged samples for neat, F1 and F2 specimens. The areal average roughness was measured for each to determine variability in peak heights. As shown in Table 1, treated surfaces were more uniform than untreated surfaces.

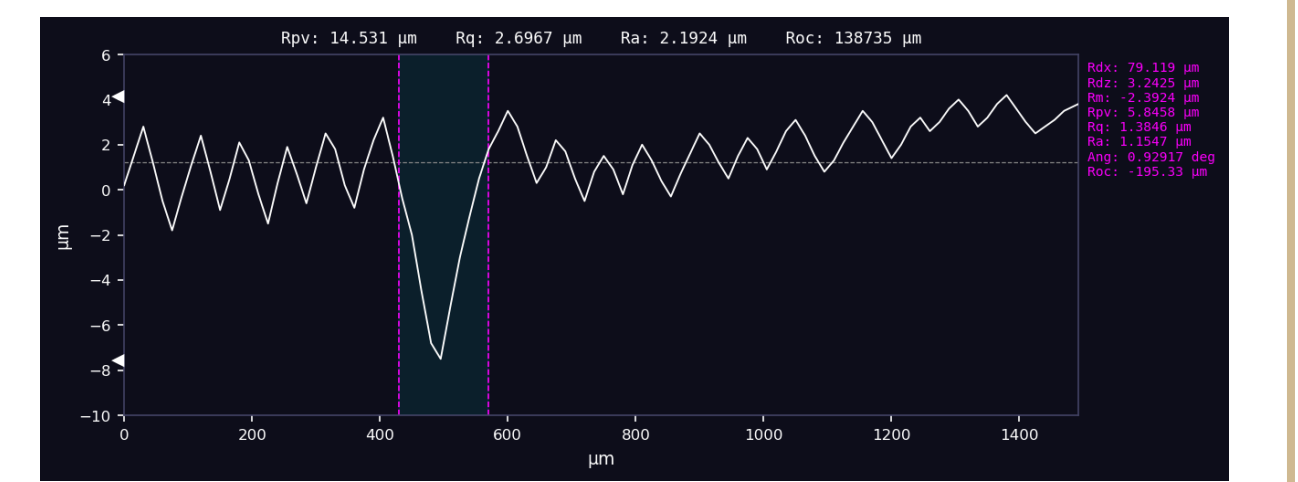


Figure 11: Representative optical profilometer profile plot of neat, uncharged sample at 10x magnification across peaks.

Conclusions

Conclusions

- Mechanical testing and fractography indicate that there is no significant difference in mechanical behavior after hydrogen charging for treated and untreated specimens.
- Thermal desorption spectra indicate that hydrogen diffuses into treated and untreated specimens at approximately the same rate and that once entered, hydrogen is trapped at the same sites in approximately the same proportions.
- Energy dispersive spectroscopy confirms that for short charging times, the integrity of the surface treatment remains well intact, yet hydrogen is still able to enter and induce embrittlement.

Overall, there is no indication that Triboconditioning® has an impact on the hydrogen embrittlement susceptibility of 4130 steel in the quench and tempered state.

Acknowledgements & References

Acknowledgements

Our team would like to thank the following individuals for their help with the project:

- Dr. Farshid Sadeghi and Ramakrishnan Jayasekaran for use of their hydrogen analyzer.
- Jia-Huei Tien, Dr. David Johnson, and Dr. David Bahr for helpful discussion and guidance.
- Ian Buck and the Cummins and Tribonex teams for the manufacturing of specimens and guidance.

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- [1] Bruce Craig, Hydrogen Damage, *Corrosion: Fundamentals, Testing, and Protection*, Vol 13A, ASM Handbook, Edited By Stephen D. Cramer, Bernard S. Covino, Jr., ASM International, 2003, p 367-380, <https://doi.org/10.31399/asm.hb.v13a.a0003634>
- [2] Tribonex. Triboconditioning®. <https://tribonex.com/triboconditioning/> (accessed April 9, 2026)
- [3] Dan Grice, Scanning Electron Microscopy in Fractography, *Fractography*, Vol 12, ASM Handbook, Edited By Craig J. Schroeder, Ronald J. Parrington, Joseph O. Maciejewski, James F. Lane, ASM International, 2024, p 241-253, <https://doi.org/10.31399/asm.hb.v12.a0006876>