Understanding Hydrogen Embrittlement in 110XF Steels

Sabrina King, John Liu, Xin Yi Ng, Daniel Whelan
Faculty Advisors: Dr. David Johnson
Industrial Sponsors: Dr. Hongbin Yin

School of Materials Engineering, Purdue University

Our group was tasked with looking for a correlation between the microstructure and Charpy impact energy of two steel alloys. We were charged with introducing hydrogen into the Charpy samples and observing any changes in impact energy. We were able to determine the transition temperature needed to perform the Charpy test and determine which alloy is more brittle. The microstructures of each alloy were analyzed, and TDA was done to analyze existing hydrogen trapping sites.

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Project Background

This goal of this project was to determine a correlation between the microstructures and the likelihood of hydrogen embrittlement for two High Strength Low Alloy (HSLA) steels. The analysis of this project can be broken down into five sections: hydrogen charging, Thermal Desorption Analysis (TDA), Charpy impact energy, microstructure analysis, and quantitative metallographic fractography.

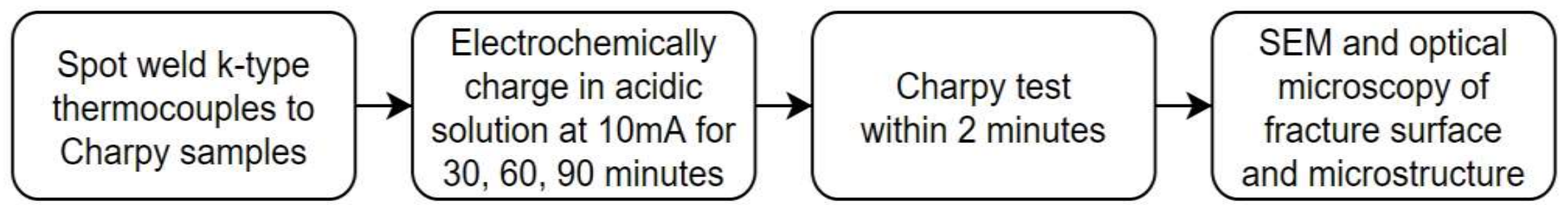
10" thick slabs of two grades of steel at ArcelorMittal, 302 and 304, had been stress cracking across the thickness in the cooling yard after being continuously cast and cut. It is worth noting that these slabs are cracking prior to any further processing and the final desired microstructure is finer grained than what is being observed in this project. Continuous casting is when metal is heated to a liquid and allowed to solidify into a slab by going through a mold. This process must be constant and run uninterrupted to prevent solidification within the mold itself. ArcelorMittal often runs several ladles in shifts to keep the caster full. Usually, several slabs of the same grade of steel are made on the same shift to minimize changeover. ArcelorMittal has five continuous casters at the Indiana Harbor facility.

It was determined by ArcelorMittal that the cause of the cracks was a high concentration of hydrogen in the liquid steel. After degassing to below a specified threshold of hydrogen, cracking no longer occurred. Hydrogen tends to segregate towards larger grains, and carbides in the austenitic phase specifically. [1]

Stacked steel slabs after being cast at ArcelorMittal. [2]

[1] Bhadeshia, H. K. D. H. (2016) "Prevention of Hydrogen Embrittlement in Steels" ISIJ International, Vol. 56 (1), pp. 24-36. DOI: 10.2355/isijinternational.ISIJINT-2015-430
[2] (2016) "Controlled slab cooling ensures product quality" ArcelorMittal, online: https://usa.arcelormittal.com/news-and-media/our-stories/2016/nov/11-18-2016

Experimental Procedure

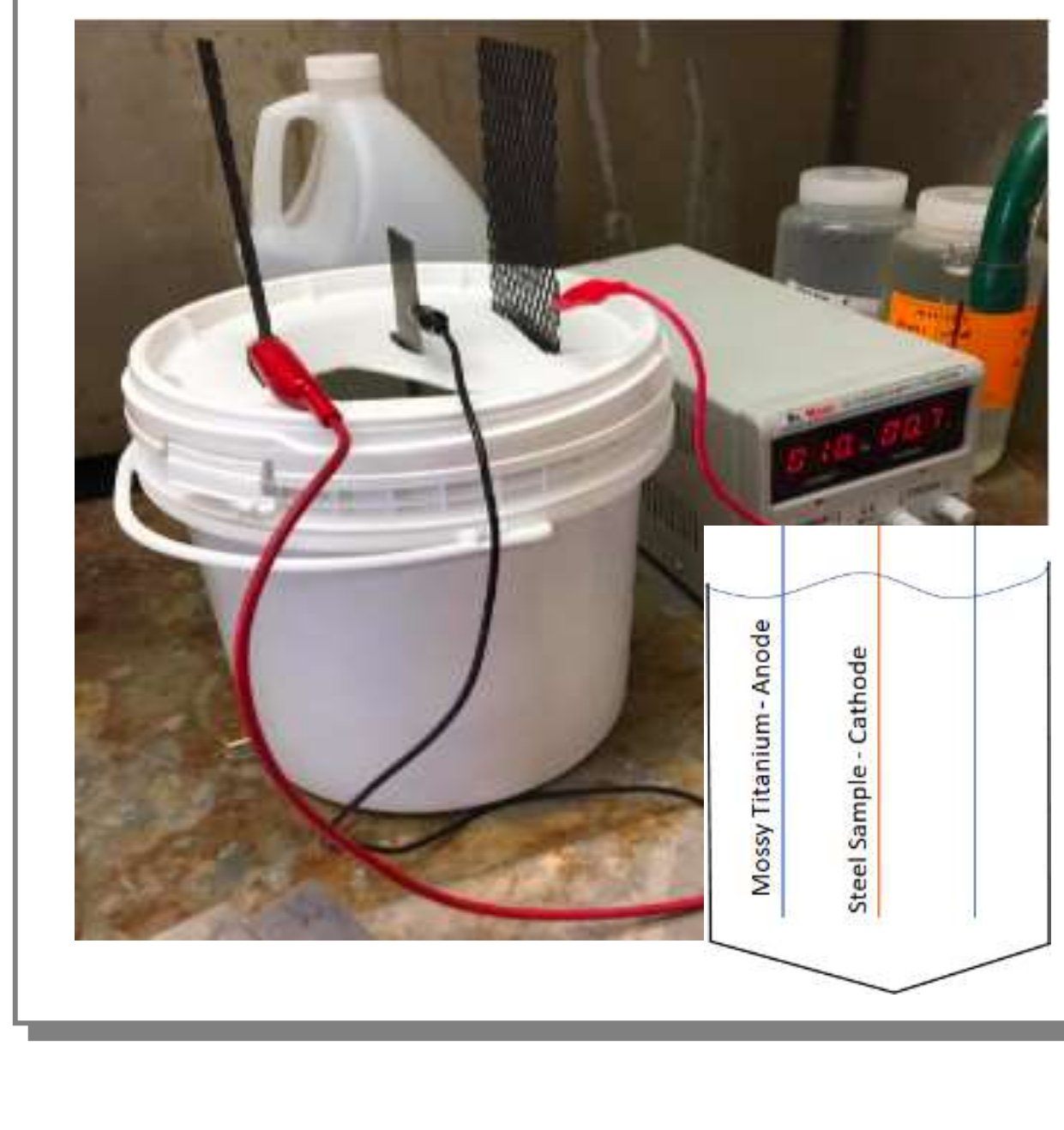


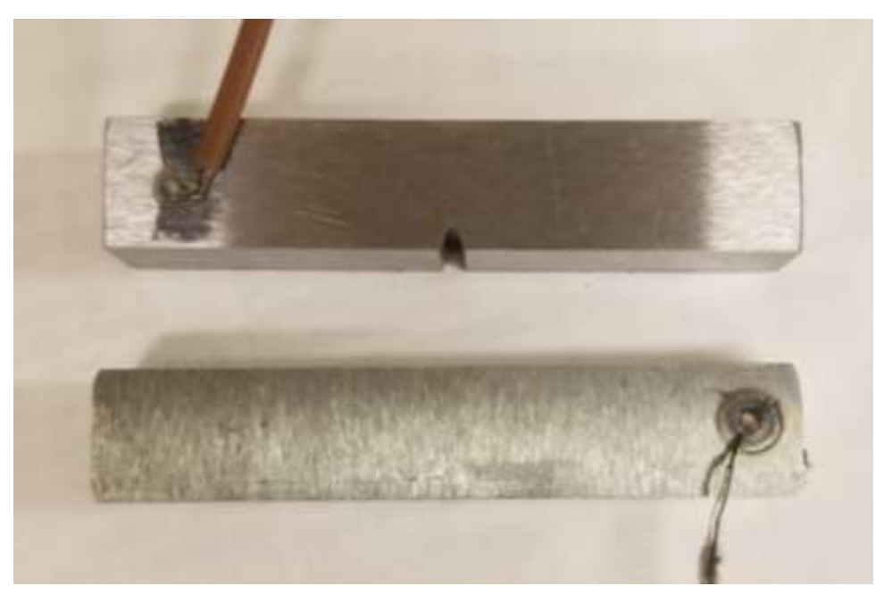
Spot weld k-type thermocouples to Charpy samples → Electrochemically charge in acidic solution at 10mA for 30, 60, 90 minutes → Charpy test within 2 minutes → SEM and optical microscopy of fracture surface and microstructure

Charpy test samples were prepared according to the process above. The figure below shows a detailed setup of the charging station. ArcelorMittal requested that after charging is complete, the samples be Charpy tested within two minutes after being removed from the solution to minimize the diffusion of hydrogen from the samples.

Prior to Charpy testing charged samples, a curve near the Ductile-Brittle-Transition-Temperature (DBTT) had to be determined. We determined that using boiling water did not get the samples to a high enough temperature, so we decided to use a furnace to heat the samples until the DBTT curve was found. Charpy tests were then performed on the DBTT curve.

After Charpy testing, the fracture surfaces and microstructures were analyzed using optical microscopy and Scanning Electron Microscopy (SEM). TDA is the use of a quadrupole mass spectrometer to measure gas desorption rates at increasing temperatures and was performed on charged samples at ArcelorMittal to determine hydrogen concentrations in the alloys.



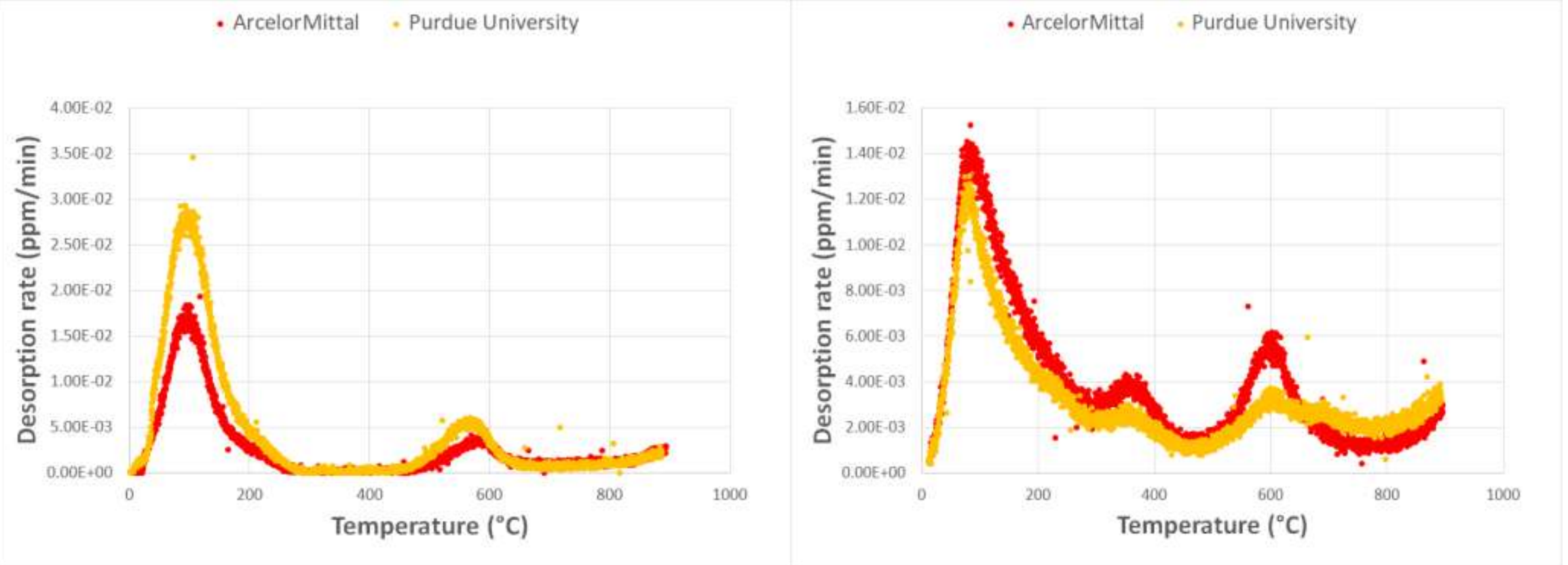


Left: The set-up for charging hydrogen into the samples. **Above**: Spot welded thermocouples on two samples.

Results

Thermal Desorption Analysis (TDA)

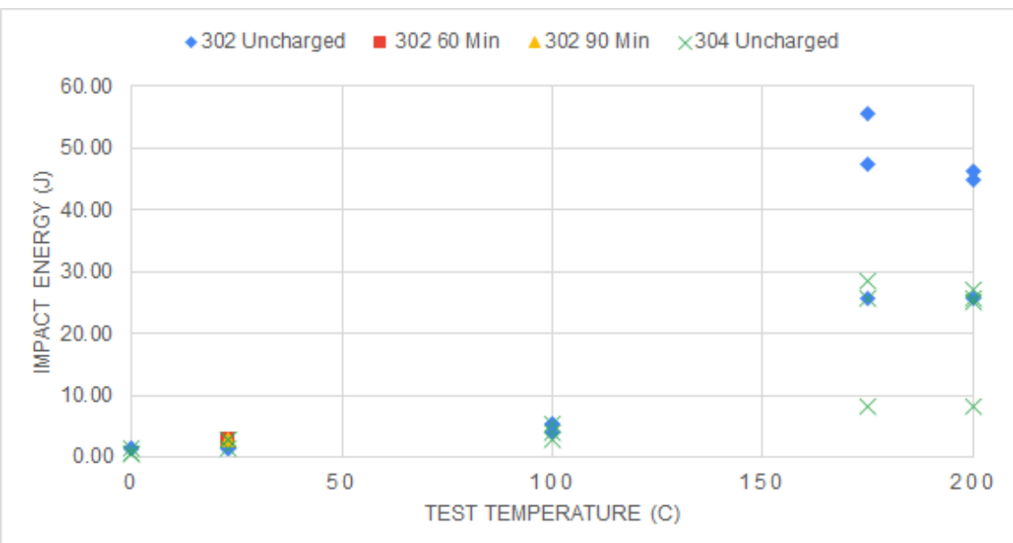
Looking at the TDA results, which represent hydrogen charging, we can see that the hydrogen adsorption at a given temperature is similar between the ArcelorMittal and Purdue University setups; therefore, charging and testing can be done at Purdue because the results are similar. The differences can be attributed to limited TDA tests, since ArcelorMittal normally averages three samples per charging duration for precision. The low temperature peaks correlate with reversible hydrogen trapping sites. The greater desorption rate of the 302 TDA results show that more hydrogen is released after charging than 304.

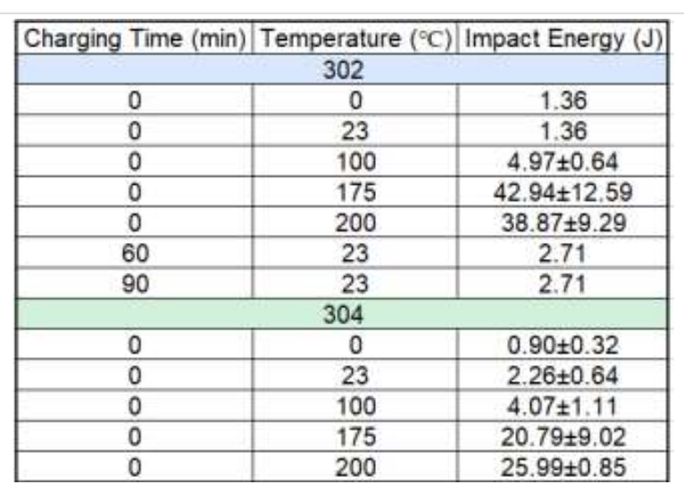


302 (left) and 304 (right) TDA results from ArcelorMittal using the setups from ArcelorMittal (red) and Purdue University (gold). Samples were charged for 60 minutes. Data was obtained once per setup at 60 minutes for each grade of steel.

Charpy Impact

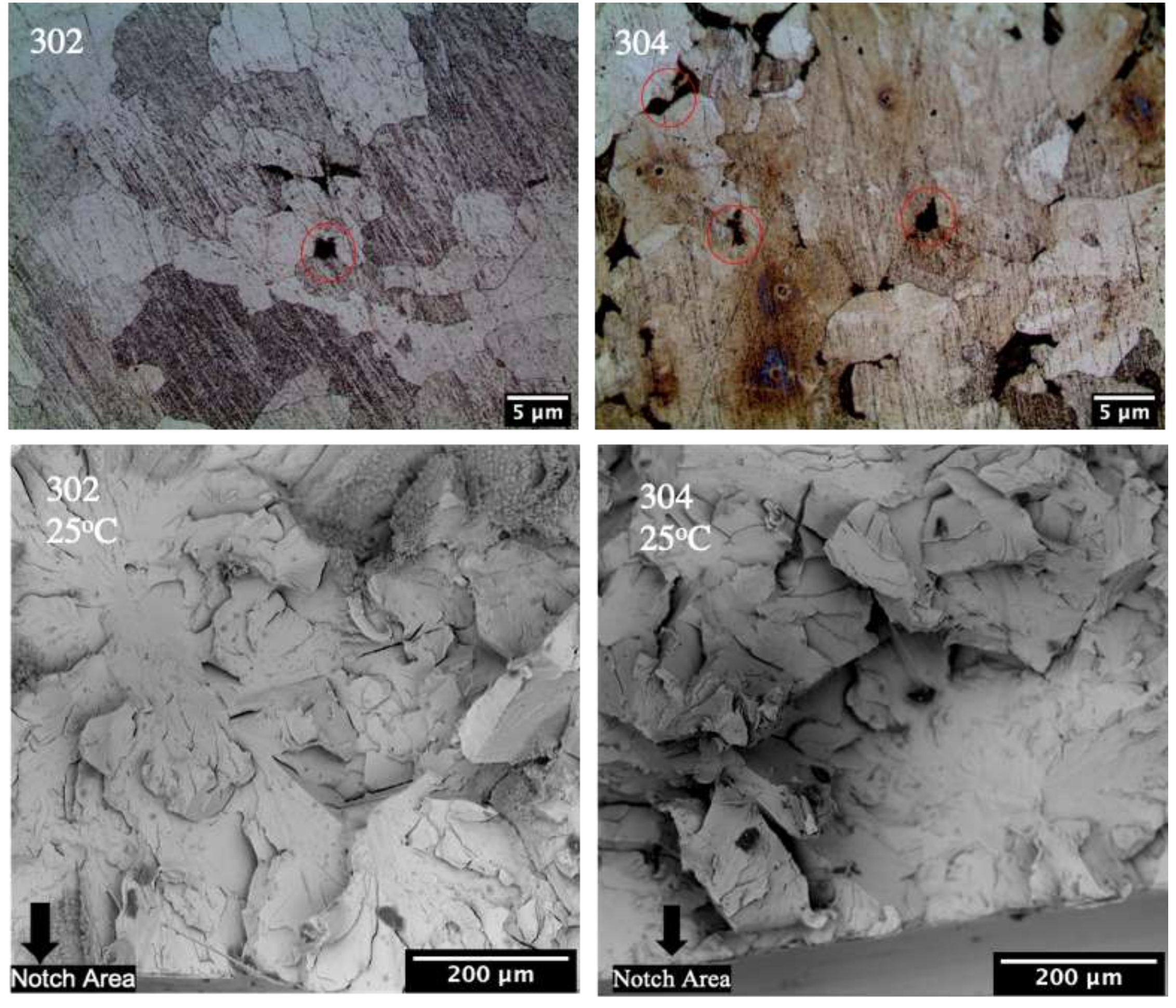
The figure to the right shows the procedure of establishing an effective testing temperature. 25 C and 100 C were determined to be on the lower shelf leading to the usage of a furnace to test around 200 C, where discernible differences between charged and uncharged samples as well as between 302 and 304 were found.





Charging Time (min)	Temperature (°C)	Impact Energy (J)
302		
0	0	1.36
0	23	1.36
0	100	4.97±0.64
0	175	42.94±12.59
0	200	38.87±9.29
60	23	2.71
90	23	2.71
304		
0	0	0.90±0.32
0	23	2.26±0.64
0	100	4.07±1.11
0	175	20.79±9.02
0	200	25.99±0.85

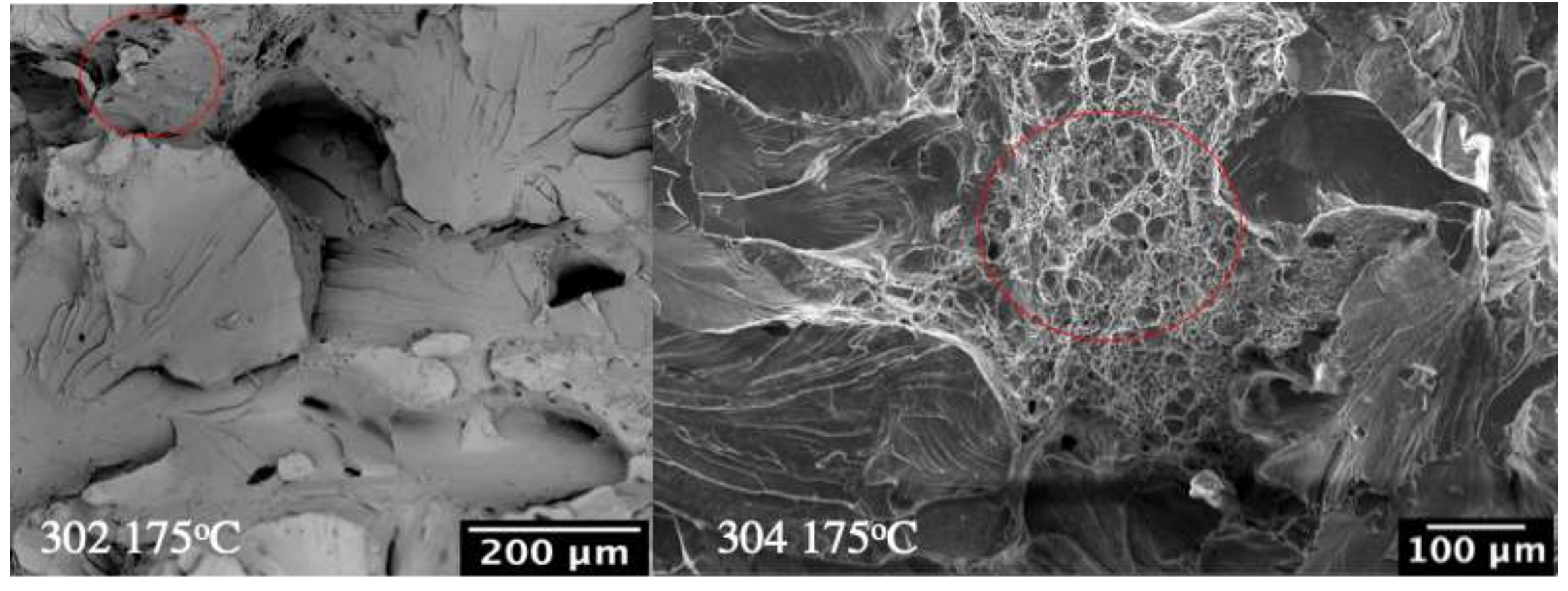
Microstructures and Fractography



Microstructure of the steel samples on top with the respective fracture surface at 25ºC uncharged. Circled region indicates pearlite within the sample.

Sample Name	Average Grain Size (µm)	Standard Deviation
302	6.232	1.432
304	3.199	0.700

As observed in the microstructure and its fractography, there are more cleavage facets in the 304 samples where the pearlite appears to be at.



Fracture surface of both steel sample tested at the DBTT range with microvoid coalescence shown in circle. Note that the 304 samples are observed under secondary electron and the 302 samples in backscattered electron.

Discussion

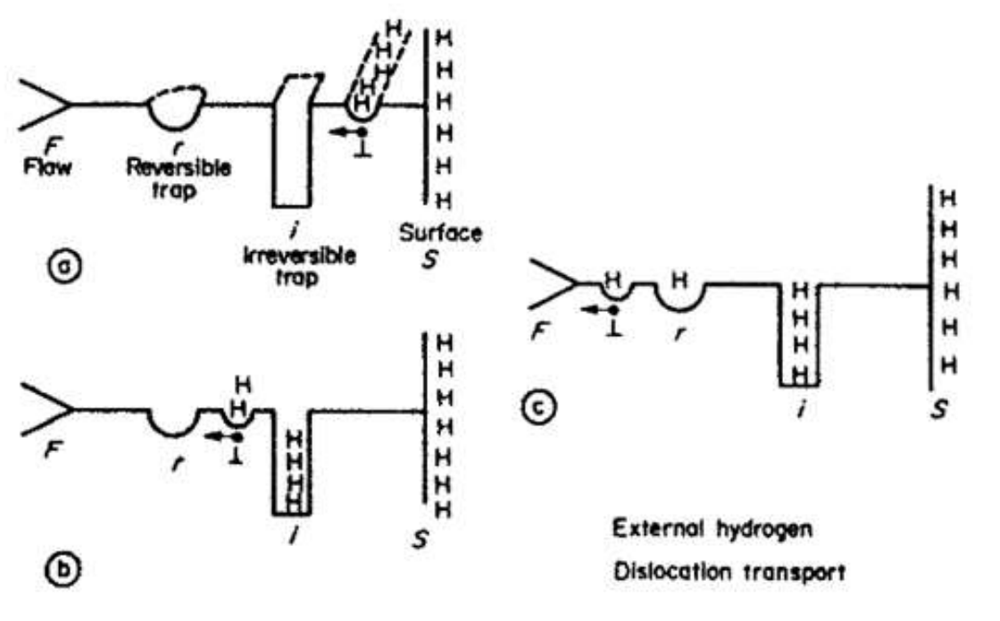
TDA and Charpy Impact

Analysis of the TDA data allowed us to determine the presence of different kinds of trapping sites in both steels. Low temperature peaks are indicative of reversible trapping sites and are more prominent in both samples than high temperature peaks, which indicate irreversible trapping sites. Reversible trapping sites require a lower energy to desorb hydrogen. This means that reversible trapping sites have a higher presence in the samples. [3]

After determining the DBTT range, the Charpy test results showed that the 304 samples are more brittle than the 302. Through comparison of the samples tested at room temperature and in the DBTT range, presence of microvoid coalescence was observed. The alloying elements in 304 facilitate carbide formations near grain boundaries, as shown by the presence of pearlite near the boundaries to decrease grain size. The pearlite near grain boundaries decreases the toughness of 304. This is supported by the lower Charpy energy, as observed in the results. [3]

Hydrogen Embrittlement

Hydrogen embrittlement occurs when hydrogen is trapped in the microstructure, usually introduced by an external environment. Large-grained structures are more prone to hydrogen embrittlement than fine-grained structures. [1] There are two types of trapping sites: irreversible, and reversible. Irreversible traps act as sinks, and if they become saturated, can become reversible and serve as sources of hydrogen. [1,4] Having more irreversible trapping sites decreases the amount of diffusible hydrogen in the microstructure. While Mo does not show resistance to hydrogen, Mo carbides can serve as weak hydrogen traps. [1]



Hydrogen entrapment and transportation by dislocations. (a) The dislocation just nucleated at the surface has picked up hydrogen and moves toward the flaw. (b) On passing over an irreversible trap, some hydrogen is lost by the dislocation. [4]

[3] Krauss, G. (2015) "Steels: Processing, Structure and Performance", ASM International, pp.475-476.
[4] Pressouyre, G. M. (1980) "Trap Theory of Hydrogen Embrittlement" Acta Metallurgica, Vol. 28 (7), pp. 895-911. DOI: https://doi.org/10.1016/0001-6160(80)90106-6

Conclusions & Recommendations

Based on the TDA results, reversible trapping sites serve as the primary cause of hydrogen embrittlement. Additionally, the Charpy results and microscopic analysis indicated that 200 C is the optimal temperature for conducting these tests.

Further research should focus on trapping site behavior between charged and uncharged samples to determine hydrogen embrittlement sites in both samples. More Charpy testing on charged samples of 302 and 304 should be conducted to establish the magnitude of the effects of hydrogen embrittlement. The next team should charge samples of 302 and 304 for 60 minutes and Charpy test at 200 C to finish testing. We recommend to ArcelorMittal that they continue degassing to minimize hydrogen cracking in 302 and 304.

Acknowledgements

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