An Examination of Effects of Corrosion on the Hardness of 1018 Carbon Steel and 316 Stainless Steel

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Group 13

Lab 2 Report

Introduction and Objective

Corrosion is the destructive attack by its reaction to the environment [1]. The interest in studying corrosion is based on safety hazards caused by corrosion and lowering the material cost of corrosion [1]. The corrosion of metals for infrastructure such as bridges poses a threat to the public's safety. One such example was the collapse of a pedestrian bridge in 2000 that resulted in 107 injured people and 13 in critical condition [2]. An investigation found that the steel cables that held the structure up were corroded and unable to hold the weight of the pedestrians. As a result, the steel and concrete bridge snapped in half and fell with pedestrians to the highway below. The cause of the corrosion was elevated levels of calcium chloride mixed into the grout that cemented the steel cables to the bridge.

This report aims to measure if there is a change in the hardness of a metal surface that has corroded. The two metals under observation are 1018 carbon steel and 316 stainless steel. Both metals are subjected to two environments that promote the corrosion of a metal.

Background and Theory

Corrosion can be separated into two categories, uniform, and localized corrosion. For the scope of this experiment, corrosion can be imagined as an electrochemical cell composed of a cathode, anode, electrolyte, and an electric potential difference. At the cathodic site, a reduction reaction occurs, and oxidation occurs at the anodic site. In other words, the anodic site is where the material is corroded or destroyed. In uniform corrosion, a metal is corroded evenly across its surface, and no portions of the metal surface are corroded preferentially. In localized corrosion, a region of material is preferentially corroded. The preferential corrosion is attributed to the heterogeneity of a material, a region of material functions as an anodic site while the rest of the material functions as a cathodic site.

The corrosion process can also be classified by whether its corrosion is active or passive. Active corrosion refers to when a metal freely corrodes in its environment. Passive corrosion or passivity refers to when a metal forms an oxide layer that further prevents corrosion or limits the rate at which it occurs—the passive film functions as a barrier between the metal and electrolyte. The passivity of a metal depends on the stability of the oxide layer in its environment [3]. An example of passivity would be the oxide layer that forms on stainless steel in the natural atmosphere, attributed to the amount of chromium alloyed with the steel [1]. Another example of passivity is iron (iii) oxide that forms on iron, commonly known as rust.
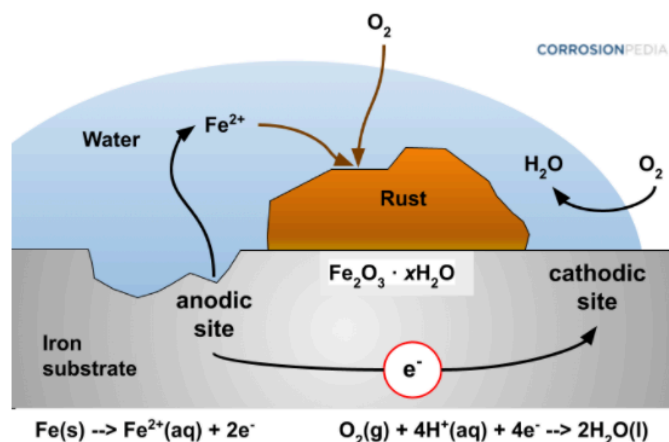
Figure 1. Graphic representation of the reduction-oxidation reaction that occurs in the corrosion process of iron [4].

Two conditions that promote the corrosion of a metal in its environment are the presence of chlorides and a pH that is not neutral. Chlorides refer to the negatively charged chlorine ion (Cl^-) and can also be defined as any compound that has chlorine in its composition [5]. The presence of chloride ions is detrimental to materials with natural passive films such as aluminum alloys and stainless steels. For stainless steel, a chloride ion is able to penetrate the chromium oxide layer. The result is the localized corrosion phenomena of pitting, the newly formed pit functions as an anodic site and will preferentially corrode. An acidic or low pH environment contains higher hydrogen ion concentration, which contributes to the corrosion process. For a majority of metals, their valence electrons are loosely bound and it is energetically favorable for the electron to transfer from the metal to the hydrogen ion.

Experimental Apparatus

The two metal alloys being tested are 1018 steel and 316 stainless steel. For 1018 steel, the hardness is 71 HRB [8]. The hardness of 316 stainless steel is 90 HRB [9]. In addition, 316 stainless steel is corrosion-resistant because of its chromium content (16-18.5%) [10].

The project concludes in two sections. The samples will be corroded by immersing them in the 3.5 wt% saltwater solution and the 5% sulfuric acid solution in the first section. The hardness of the samples will then be measured using a Rockwell hardness machine. The specific machine used was a Wilson Rockwell series B200 hardness tester. To find the uncertainty for this machine, hardness measurements must be made on a reference test block compared to the certified hardness of the reference block [11].

Experimental Procedures

Preparation of solution and samples
1. Measure 500 mL of 3.5 wt% NaCl into two different labeled Pyrex glassware.
2. Repeat step 1 to measure 500 mL of 5% H2SO4 into two labeled Pyrex glassware.

3. Measure and record the mass of each sample. To keep track of samples, label a sheet of paper with the sample name, and after weighing, place the sample onto the corresponding sample name.
4. Clean the samples with isopropyl alcohol and then air dry using the air hose in the vent hood. When cleaning the samples, use forceps and gloves to handle their sample.
5. Be sure to label and record the sample's position in the container.
6. Place the samples in the beakers according to their labeled positions.
7. Store the beakers in a safe place where they will be undisturbed.

Note: In total, there should be four samples in each container. There should be four 1018 samples in acid, four 1018 samples in a salt solution, four 316 samples in acid, and four 316 samples in a salt solution. See Table 1 for the experimental conditions for all samples.

Table 1. Metal sample and its corresponding conditions used in the experiment.

Sample Name (ID)	Material	Solution	Days in Solution
316 Control	316 stainless steel	n/a	n/a
1018 Control	1018 carbon steel	n/a	n/a
A1	316 stainless steel	3.5 wt.% NaCl	14
A2	316 stainless steel	3.5 wt.% NaCl	14
A3	316 stainless steel	3.5 wt.% NaCl	19
A4	316 stainless steel	3.5 wt.% NaCl	19
B1	316 stainless steel	5% H_2SO_4	14
B2	316 stainless steel	5% H_2SO_4	14
B3	316 stainless steel	5% H_2SO_4	19
B4	316 stainless steel	5% H_2SO_4	19
C1	1018 carbon steel	3.5 wt.% NaCl	14
C2	1018 carbon steel	3.5 wt.% NaCl	14
C3	1018 carbon steel	3.5 wt.% NaCl	19
C4	1018 carbon steel	3.5 wt.% NaCl	19
D1	1018 carbon steel	5% H_2SO_4	14
D2	1018 carbon steel	5% H_2SO_4	14

D3	1018 carbon steel	5% H_2SO_4	19
D4	1018 carbon steel	5% H_2SO_4	19

Transferring samples out of solutions
1. Take samples out of the solution at the specified times.
2. Use forceps or a similar tool to take the appropriate sample out of the solution.
3. Place the sample onto a paper towel. It is essential to know which sample is which once it is out of its solution. Do this by either having a system to organize the samples or label the paper towel and place the samples on the appropriate labels.
4. Dry the samples and proceed to measure the mass of the samples.

Hardness testing
1. Hardness testing requires the sample to be a flat surface so that it does not move or rock when the indentation occurs. During this process, remove any material that may have formed on the surface. If the sample is not stable, the hardness tester will take an improper measurement. To flatten the surface of the samples, either use a hand file or a grinder.
2. Verify Rockwell hardness tester machine is on.
3. Once on, verify that the machine is set to take measurements on Rockwell B (HRB).
4. Install the appropriate indenter, reference Table 2.
5. Place the sample on the stage.
6. Adjust the height of the indenter so that it is closer to the sample's surface. Make sure it does not touch the surface of the sample. There should be enough room to adjust the sample between indentations.
7. Adjust the sample position for the desired location to measure hardness. The indentation location should not be too close to the edge of the sample or other indentations. If pores or holes are on the surface, try to avoid them.
8. If the hardness value is negative or invalid, change the indenter and scale. Otherwise, repeat step 7 until the desired number of hardness values are taken.
9. Adjust the height of the indenter to take the sample off the stage.
10. Repeat steps 5-9 until hardness measurements have been taken for all samples.

Table 2. Rockwell hardness parameters for the used Rockwell hardness scales.

Hardness Scale	Identer	Load (kgf)	Typical Application
Rockwell B (HRB)	1/16" Ball	100	Copper alloys, soft steel aluminum alloys, malleable iron, etc.
Rockwell R (HRR)	1/2" Ball	60	Bearing materials and other very soft or thin materials including plastics.

Results

A critical note is that all D samples (1018 carbon steel in H2SO4) exhibited a decrease in hardness that required measuring the hardness on a different scale, HRR. All other sample species: A, B, and C, were measured in HRB. The parameters for the hardness scales are detailed in Table 2. Essentially, the hardness of all D samples decreased to the point that they required using a different indenter and a lighter load to conduct a proper hardness test.

Figures 2 and 3 are the hardness testing results for the metal samples. Figure 2 consists of the hardness measurements taken as an average of all points on a sample, and Figure 3 consists of the hardness measurement taken at the center of a sample. Along the ordinate are the 316 and 1018 control samples representing no time spent in solution, serving as a frame of reference. Both stainless steel sample groups (A and B) exhibited a slightly lower average hardness than the control group. The average hardness decreased by at most 3.2 HRB. For hardness taken at the center of the sample, the average hardness decreased by 4.2 HRB. In the group C samples, the average hardness increased for C1, C3, and C4, while C2 saw a minor change. The reason why the C1, C3, and C4 saw an increase in hardness could be attributed to variation in the manufacturing process of each sample.

Compared to the change in hardness observed in group D, the changes in groups A, B, and C were small or practically insignificant. A comparison of means is all that was possible because of the limitation of supplied samples. A proper ANOVA could not be completed because of the lack of a sufficient sample size.
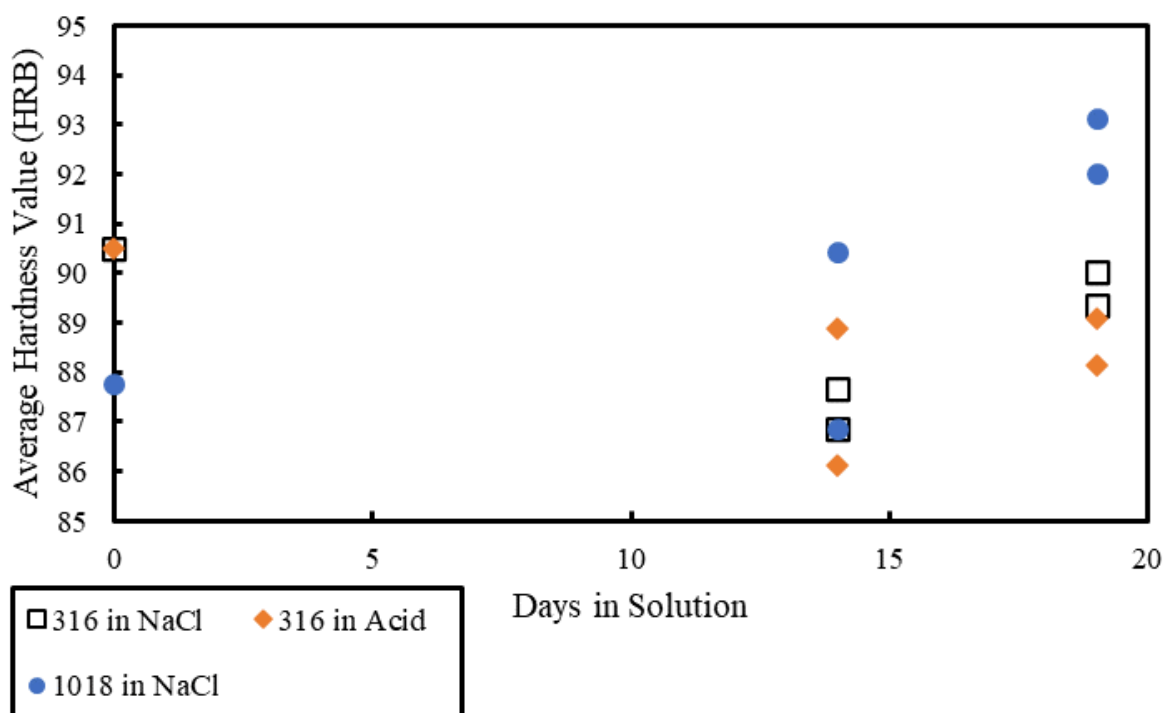

Figure 2. The average hardness measurements for all samples. Hardness for 1018 in acid is not included because it was measured using HRR.
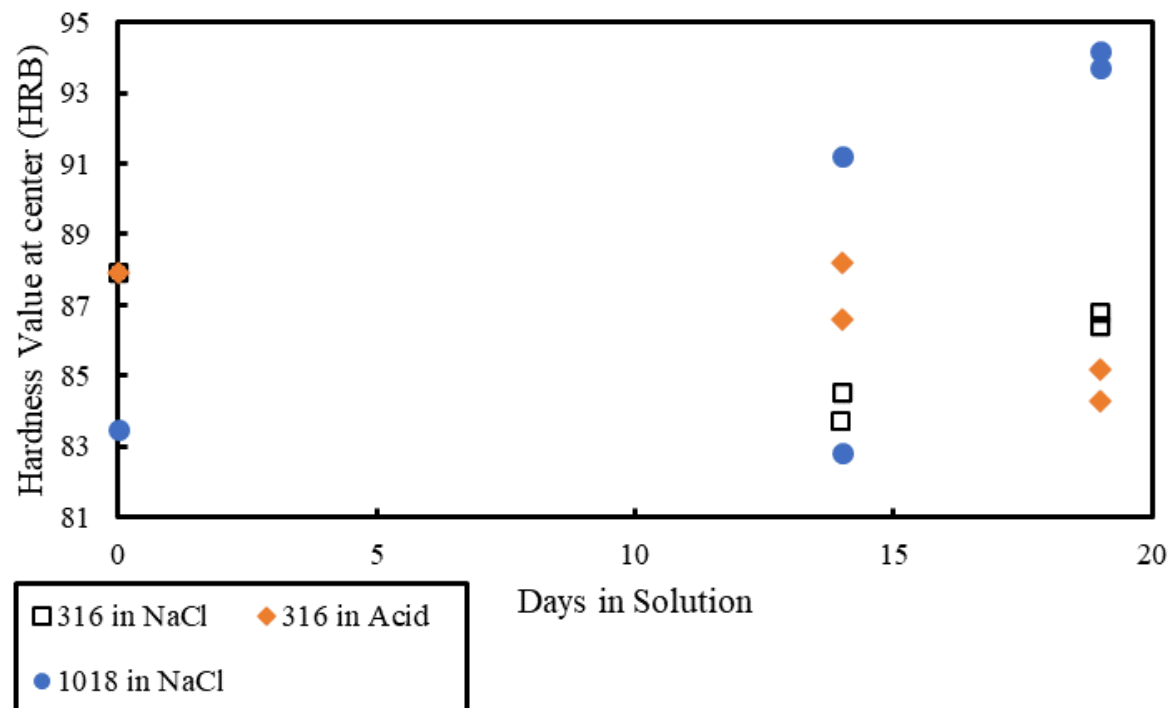
Figure 3. Hardness measurement taken at the center of the sample for all samples. Hardness for 1018 in acid is not included because it was measured using HRR.
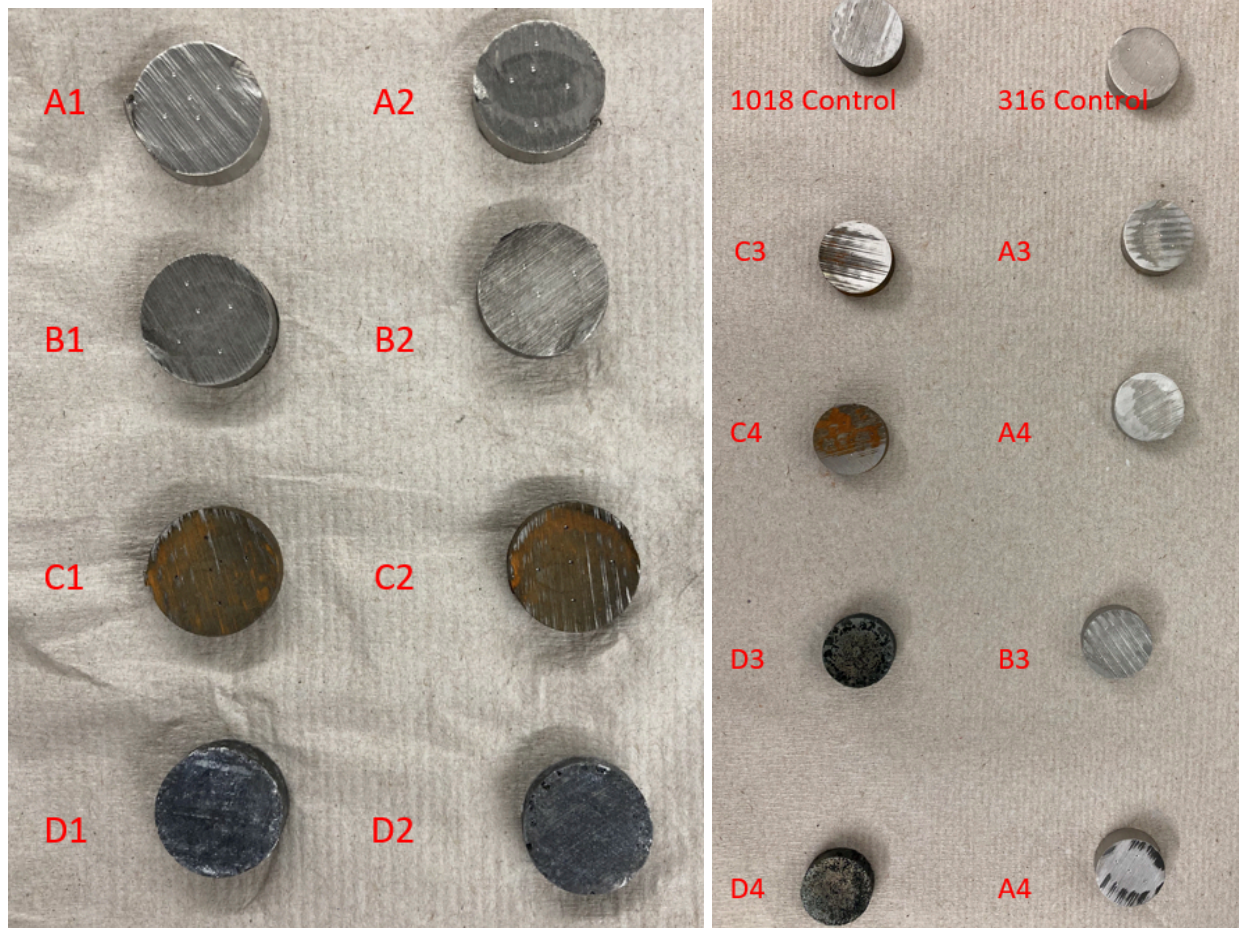
Figure 4. (left) Samples immersed for 14 day time period after hardness testing. (right) Control samples and samples immersed for 19 day time period after hardness testing.

Discussion

Three methods in this experiment can resolve whether or not a metal sample has corroded. These methods are visual observations, mass changes, and hardness changes. With the 316 stainless steel, there was no observed change when exposed to NaCl or H_2SO_4 solutions. The masses of the samples were also identical before and after. The hardness of the 316 samples were identical to the control sample, therefore the conclusion is the 316 was not corroded. This observation displays the resilience of 316 steel to the corrosion conditions because of no significant observable change or mass loss. The 1018 steel displayed remarkable effects from long-term exposure to NaCl and H_2SO_4 solutions. The 1018 steel samples exposed to the NaCl solution had orange-colored rust from corrosion and a slight mass loss. The other 1018 samples were in sulfuric acid solution and formed a blue salt precipitate on the outside of the samples. Not only was this salt formed, but large pitting occured as well because of the corrosion. For both 1018 samples, the surface had to be sanded down before hardness testing. For the 1018 sample in the sulfuric acid, the hardness varied a lot because of the pitting. The pits were difficult to avoid because of the large amount on the surface. The hardness was also varied for 1018 samples in the sulfuric acid solution. Because of the pitting, the metal was more easily formable

and indentable and thus this is why these samples had a softer hardness and required HRR to be used rather than HRB.

Conclusion

This lab was developed to test corrosion ability for 316 stainless steel and 1018 steel in sulfuric acid and NaCl solutions at various times. The impact of corrosion can be specified based on mass loss, visual rusting, and hardness testing and compared to control samples. The 316 stainless steel was highly resistant to corrosion in all scenarios for time and solution type. The 1018 steel was more prone to corrosion in both solutions. The most effective solution was the sulfuric acid, as it changed the mass and created a salt precipitate in the solution. The hardness for the 316 stainless steel samples stayed constant when compared to the control. The hardness values for the 1018 steel samples increased after corrosion, especially in the sulfuric acid solution. Time also affected 1018 steel samples, as the remaining samples had a higher mass loss. This lab has demonstrated the impact of corrosion on sample properties and reinstates the importance of selecting the suitable material for applications where corrosion is evaluated.

It should also be noted that this corrosion experiment occurred in a controlled lab environment. In a real-world situation, the corrosive environment is constantly changing. The results that were obtained in this experiment may not be indicative of corrosion in a real-world environment.

Recommendations

The hardness samples used in this experiment were small, and only five indents could be made on each sample. A sample with a larger surface area would allow for more indents and more certainty in the results. Having more samples would also increase the certainty of the results.

In the experiment, the hardness samples were cut from a metal rod. This resulted in the samples not being flat. Hardness tests cannot be done on a sample that is not flat, as it will cause wrong measurements. The samples for this experiment were either ground or filed down until they were flat after being removed from the solutions. Filing the samples flat took a long time and created large grooves in the samples that resulted in a few bad hardness measurements. Grinding took less time and resulted in flatter samples and fewer grooves. It is recommended that the samples are ground flat before they are put into solution. After removing the samples from the solution, any oxide or salt layer should be ground off until the sample is balanced enough for hardness testing.

If possible, a consistent hardness scale should be used. When testing the hardness of 1018 steel in the sulfuric acid solution, HRB values were negative, so the HRR scale had to be used. The different scales made it more difficult to compare the hardness of all the samples directly.

It can take years to see the results of corrosion. Ideally, the samples would be left in the solution for at least a month. This problem would likely result in a more significant mass change, and the corrosion rate could be measured more accurately. Besides that, it would also likely result in more prominent effects on the hardness of the samples.

It is also recommended that more samples are used in running the experiment. Two samples for each combination of immersion time, solution, and metal was not enough to run a

good statistical analysis. Needing to use two hardness scales also made it difficult to run the statistical analysis.

Contributions

Myron: Sample preparation, Hardness testing, Introduction, Objective, Background, Theory, Results, References

Michael: Sample preparation, Hardness testing, Discussion, Conclusion

Sheer: Experimental apparatus, Recommendations, Sample preparation, Hardness testing, References

Nhu: References, Procedure, Revision

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Appendices

Table 3. Initial and final mass of 316 stainless steel samples.

Sample ID	Solution	Initial Mass (g)	Final Mass (g)
A1	NaCl	19.321	19.322
A2	NaCl	21.472	21.474
A3	NaCl	21.627	21.627
A4	NaCl	21.285	21.284
B1	H_2SO_4	22.132	22.100
B2	H_2SO_4	20.448	20.424
B3	H_2SO_4	22.061	22.018
B4	H_2SO_4	22.014	21.972

Table 4. The initial and final mass of 1018 carbon steel samples. C samples shifted around while in solution, so the initial and final masses might not correspond to the same sample, which is why they are marked with an asterisk.

Sample ID	Initial Mass (g)	Final Mass (g)
C1*	20.808	20.819
C2*	21.525	21.517

C3*	19.250	19.236
C4*	19.882	19.896
D1	21.054	20.086
D2	19.949	19.493
D3	21.662	20.097
D4	21.482	19.519

Table 5. Hardness values of metal samples immersed in 3.5 wt.% NaCl.

Sample ID	Hardness Scale	HV_1	HV_2 (center)	HV_3	HV_4	HV_5
A1	HRB	88.6	84.5	83.1	91.1	86.8
A2	HRB	88.1	83.7	86.8	88.9	91.1
A3	HRB	88.7	86.8	91.5	93.9	89.2
A4	HRB	89.8	86.4	88.7	91.7	90.2
C1	HRB	92.2	91.2	88.8	86.6	93.4
C2	HRB	82.8	82.8	89.6	86.3	92.8
C3	HRB	92.4	94.2	86.9	94.6	92.0
C4	HRB	93.3	93.7	92.9	89.5	96.2

Table 6. Hardness values of metal samples immersed in 5% H_2SO_4.

Sample ID	Hardness Scale	HV_1	HV_2 (center)	HV_3	HV_4	HV_5
B1	HRB	92.6	88.2	85.5	89.6	88.6
B2	HRB	76.8	86,6	90.3	88.3	88.7
B3	HRB	90.4	84.3	89.6	90.4	90.7
B4	HRB	88.4	85.2	84.2	90.7	92.3

D1	HRR	109.4	110.3	109.5	113.4	112.4
D2	HRR	109.8	107.3	108.0	111.1	105.1
D3	HRR	101.0	117.2	91.9	94.5	98.4
D4	HRR	102.7	85.1	106.2	111.9	90.6