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Effect of Temperature and Strain Rate on the Embrittlement Behavior of Heat-Treated Carbon Steel (UNS) G10180

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Abstract: The microstructures of plastically deformed steel was studied to potentially promote ingress of atomic hydrogen raising its susceptibility to embrittlement. The as- received UNS G10180 was heat-treated to about 500oC. The apparent embrittlement of the specimen came after series of controlled pre-stressing by quasi-static loading (fatigue cycling) followed closely by pre-charging of the specimen with hydrogen in an ambient electrochemical environment of hydrochloric acid. Standard tensile test was hydrogenated from 2 to 6 hours, and deformed by cold working to 40%, 50%, 60% and 80%. The mechanical and laboratory tests on the dissolution and dissociation mechanism of the ions were also carried. The crack length and time of failure at low strain were also evaluated under the simulated conditions. The results showed that by calculation, pre-charging with hydrochloric acid raised the susceptibility of embrittlement of hydrogen specie by 25%. It was found that the heat-treated UNS G10180 experienced ductile to brittle transition effect leading to the resultant drop in mechanical duty. Increment in temperature of the electrolytic bath by every 25oC for the number of experimental hours further raised the susceptibility of the failure of the metal by 15%. This led to lower tensile strength or low bearing capacity by almost 10% compared to the as-received samples. The strain also decreased due to pre-stress and pre charging at the temperatures used. It can therefore be concluded that material like UNS steel can fail without apparent deformation in the field if subjected under high temperature and high loading, complemented by presence of acid attack.

Keywords: Hydrogen embrittlement, temperature and strain rate, mechanical properties, Nucleation, crack propagation

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1. Introduction

Across the wide spectrum of constructional industries, interest in the choice and use of high strength low alloy steel is being renewed hugely because of its inexpensive appeal and load bearing capacity [1]. Regardless of these mechanical indicators, high strength steels can be practically difficult to reconcile with SCC resistance owing to its inherent affinity to adsorb nascent species dissociated from mostly hydrogen rich environment and other industrial processes that are corrosion prone [8]. Promoting further attack would include sharp design details, micro structural defects, residual stress, temperature and environmental conditions which intensify stress concentrations, raising susceptibility to steel crack, embrittlement or blisters under intense local plastic deformation [1]. Generically, hydrogen tends to be attracted to regions of high tri-axial tensile stress where the metal structure is dilated [2] with possibilities of degrading crack size, operating stress level and this damages the material tolerant life [3]. The phase transformation associated with hydrogen induced blistering [3] is the precipitation of gaseous hydrogen at the inclusion-matrix interface [1]. As a rough guide, hydrogen embrittlement is unlikely to affect modern steels with yield strengths below 600 MPa, and is likely to become a major problem above 1000 MPa [4]. Atomic Hydrogen dissolution proceeds transgranularly to impede on moving dislocations at elevated or room temperature and the ability to undergo plastic strain is greatly reduced as a result. The atomic hydrogen upon diffusion is said to collect in the microstructural interstices of the lattice and voids in crystalline defects as second phase particles especially when strain rate is high. Repeated cold working or induced load is a variable which increases the dislocation density and vacancy concentrations. The same is true of strong steels with faulty thermal history sensitized to the threshold of inter-granular inconsistencies. With a slow travelling

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strain, there is minimal or no diffusion gradient leading to the chemical combination of nascent hydrogen to form diatomic molecule of hydrogen at micro cracks, voids or grain boundaries in the crystallographic planes nucleating a crack or blister by pressure effect. In this work, the fracture mode proceeded by cleavage, fish eye marks along the grain boundaries and shear mode cracking based on the controlled mechanical loading and geometry of the test specimen. The universal electrochemical method approved by ASTM is employed to study the embrittlement phenomena simulated in a closely monitored environment. The effects of hydrogen introduced into components prior to service may be reduced by baking for a few hours at around 200°C. This allows some of the hydrogen to diffuse out of the steel while another fraction becomes bound to relatively harmless sites in the microstructure [4]. Other route ways for embrittlement into steel include cathodic protection, electroplating, acid corrosion, phosphate and pickling etc.

2. Materials and Methods

i. A long cylindrical heat treated carbon UNS G10168 steel of 12mm diameter was cut into standard gauge lengths permissible for tensile experimentation on MTS 20/MH electromechanical tensile testing machine. UNS G10168 is a medium carbon industrial steel, and is commonly used for masonry and constructional works due to its excellent property of mechanical strength and tenacity. Stress raisers on the specimen were adequately chipped off by application of varying rough-to-smooth grades of emery paper until mirror finish. 240, 320 and 600 grits were successfully applied. The samples were ultrasonically degreased with acetone and finally treated with a micro etching 0.3% Nital solution.

ii. The test specimens were designed out of the UNS grade of carbon steel by carefully controlled lathe machining process. Thirty out of the fifty-four samples machined out for this work were dented heavily to a concentrated stress, forming the fracture stress specimen. The final geometry of the samples was such that will promote embrittlement upon pre-stressing and the cathodic hydrogen charging by the acid in accordance to American Society of Testing and Materials E8 standard [5]. The machined notches were carefully investigated microscopically for fracture phenomenon after thirty minutes' interval.

iii. AR grade Hydrochloric acid was prepared to a high concentration of 2.0mol/dm^3 as the electrolytic media to aid the charging process by means of strong dissociation of its species.

iv. The experimental setup is comprised of an integrated arrangement of a Jig-clamp system connected firmly to the specimen submerged into an electrochemical bath of hydrochloric acid of the stated concentration. It serves the dual purpose of hydrogen charging (ASTM G148-97) and pre-stressing simultaneously. The Jig-clamp system used comprises 1600mm by 45mm by 20mm grade 904 stainless steel with four high tensile bolts. The pre-stressing of the mild carbon steel is delivered by the gripping effect of the chuck of the clamp with the Jig bolt actuated carefully to apply stepwise progressive pressure at about 35% of the ultimate yield strength of the steel which is below the threshold of cracking or blistering. The extended sections of the jig- clamp system likely to be exposed to the Acid are epoxy coated to discourage local galvanic action. The electrochemical part of the experimental circuit fitted with a sensitive current measuring ammeter picks the flow of hydrogen electrons (current) into the steel at 25, 30, 35, 40, 45, 50 and 60^{0}c as they are allowed to age for 0 hours, 50, 100, 150, 200, 250, 300, and 350 hours consecutively in the acid under plastic loading. To further concentrate the hydrogen, a barnacle cell setup based on ASTM recommendations embedded into the arrangement isolates part of the metal as the hydrogen anode, the remainder as the cathodic half. The system measures the oxidation current when the proton reacts with the hydroxyl ions. The strain rate was obtained by a clip-on extensometer with a 25mm gauge length or a 45^{0}C strain gauge rosette. The hydrogen concentration value adsorbed in the matrix of the steel is given by equation 1.

$$I_P = FC_o\sqrt{\frac{D}{\pi T}} \quad (1)$$

Where, I_p is the current density, F is Faradays' constant 96485.2 C/mol, Co is Hydrogen concentration, T is Time of charging in hours or minutes, D is Diffusion Coefficient 2.5 × 10^{-8} Cm/S^2.

Table 1: Composition of the high strength low alloy Steel

Elements	Fe	Si	Mn	P	C	S	Cr	Ti	Al	Mo	Cu	Ni
% Composition	94.93	0.53	1.70	0.04	0.44	0.05	0.25	0.05	0.2	0.35	0.4	0.5

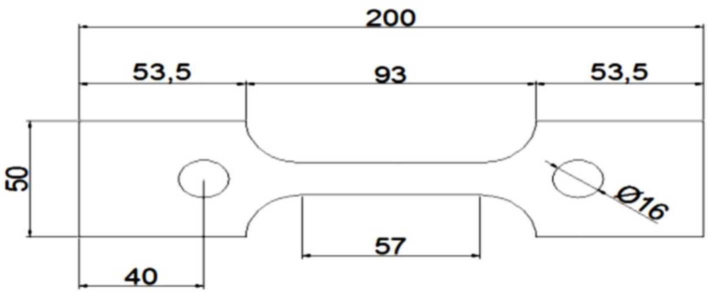

Figure 1: ASTM E8 based fracture pre-stress sample design

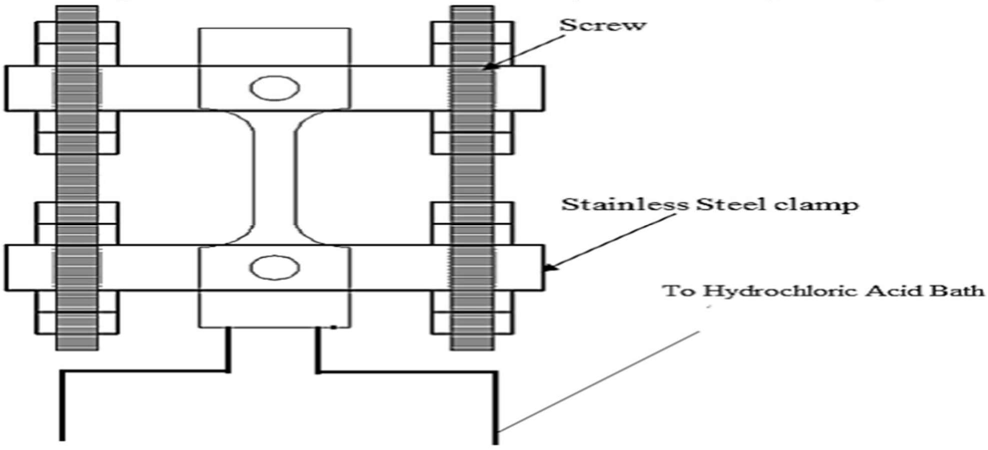

Figure 2: Jig-clamp system linked to hydrochloric acid bath for the experiment

3. Results and Discussion

Table 2: Time of Sample Charging and Current Density of Metal under Plastic Loading

Time (hours)	µA/Cm^2 at 25°C	µA/Cm^2 at 50°C	µA/Cm^2 at 75°C	µA/Cm^2 at 100°C	strain rate(e)/s × 10^{-3}
0	15	10	18	25	1.00
50	20	25	58	100	1.51
100	39	53	77	115	1.73
150	43	80	136	210	1.23
200	60	89	160	200	0.33
250	100	100	183	210	0.12
300	148	150	200	230	0.09

Table 3: Mechanical Tensile testing Results

Time (hours)	Tensile strength (MPa) at 25°C	Tensile strength (MPa) at 50°C	Tensile strength(MPa) at 75°C	Tensile strength(MPa) at 100°C
0	830	701	559	523
50	782	658	455	439
100	732	610	410	387
150	671	582	398	350
200	547	430	365	335
250	528	400	329	310
300	478	378	319	298

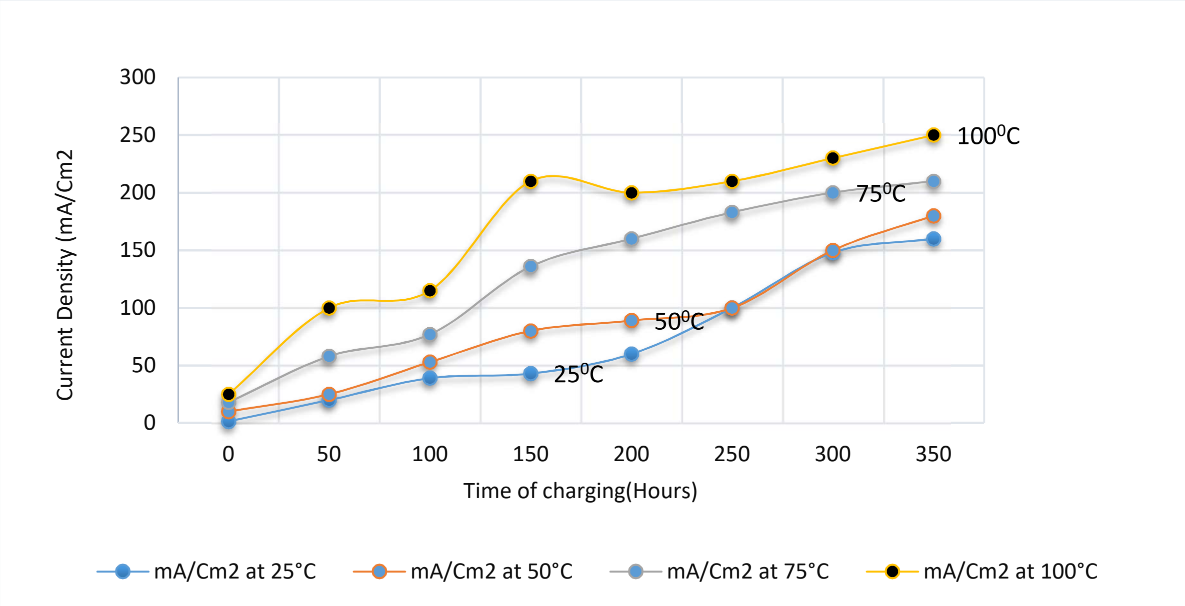

Figure 3: A graph of current against Times of charging

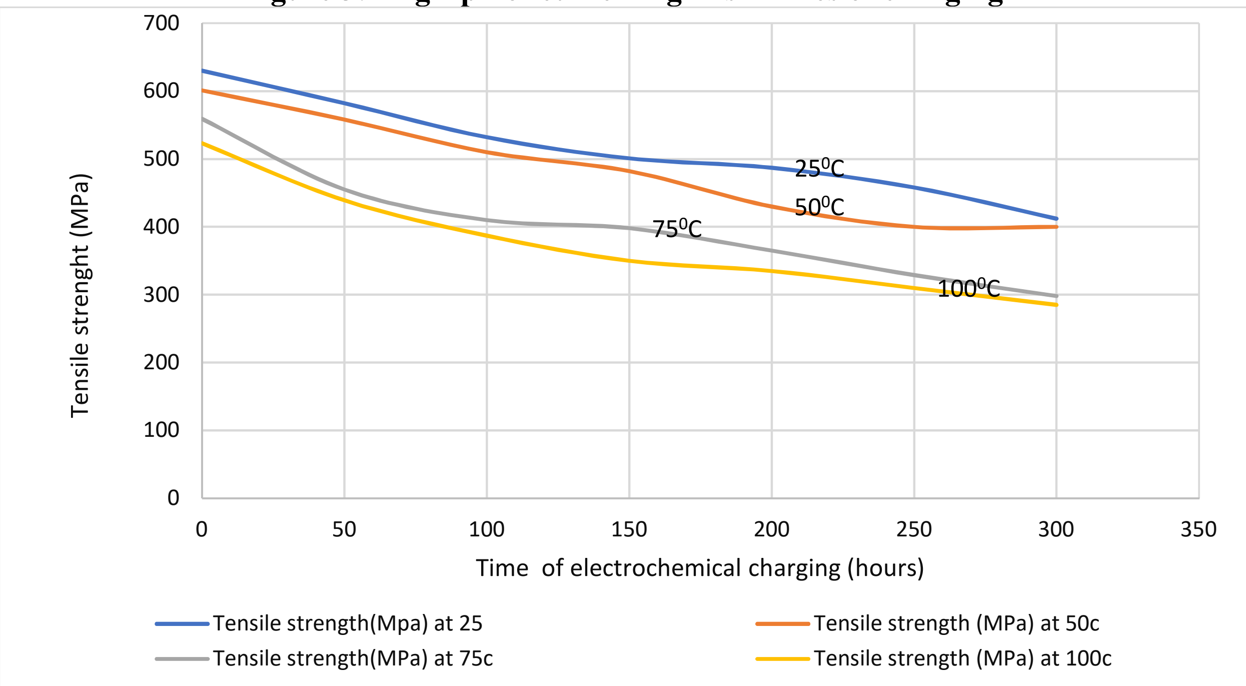

Figure 4: A graph of Tensile versus Time of Charging

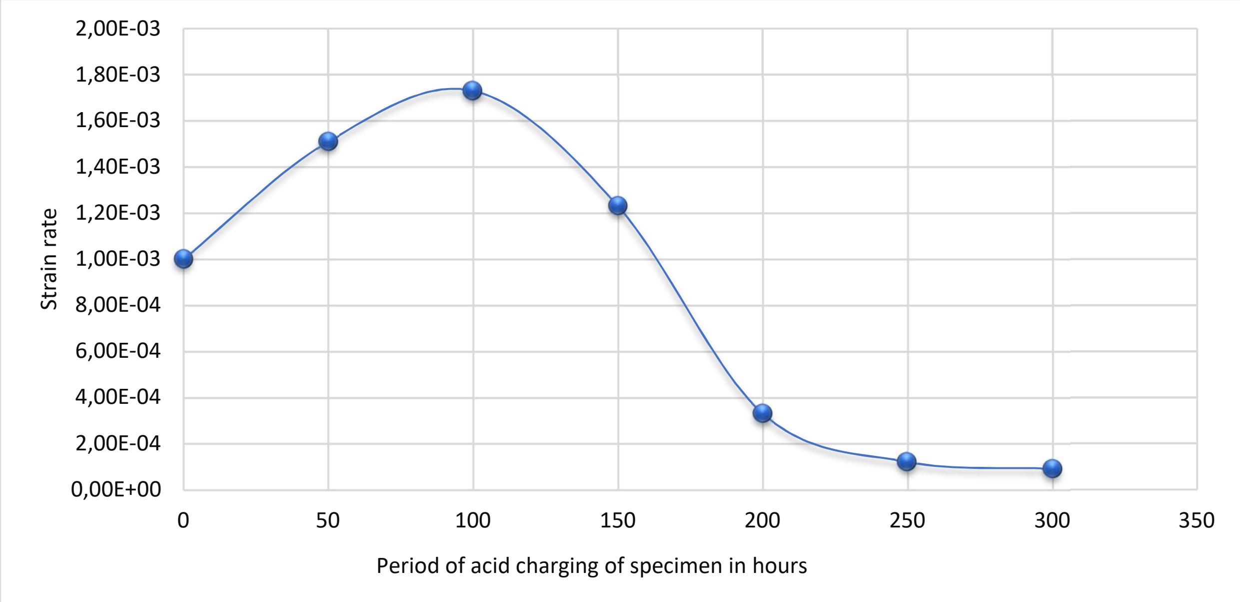

Figure 5: A graph of Strain rate versus period of charging of specimen in hours

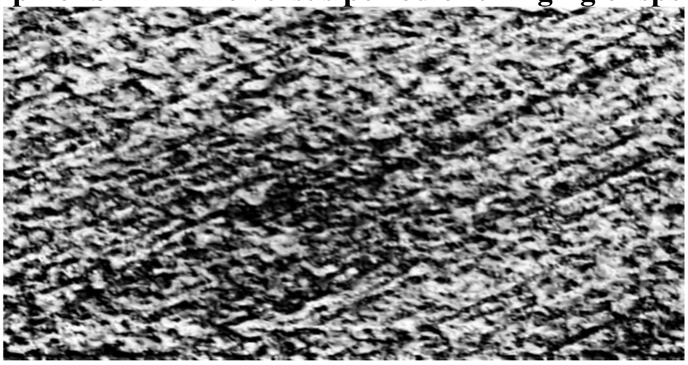

Plate 1: Fracture Surface of Control Sample

As shown in Plate 1, it is observed that fractured surface of the control specimen contains a large population of small and shallow dimples which is indicative of its relatively high tensile strength and ductility [6].

Plate 2: Fracture surface of the Sample at 25^{o}C

Fracture surface observed showed that hydrogen triggered the fracture along the grain boundaries as shown in Plate 2 [7].

Plate 3: Fracture surface of the Sample at 50°C

Plate 3 showed Transgranular quasi-cleavage type of fracture appeared, which indicates brittle failure.

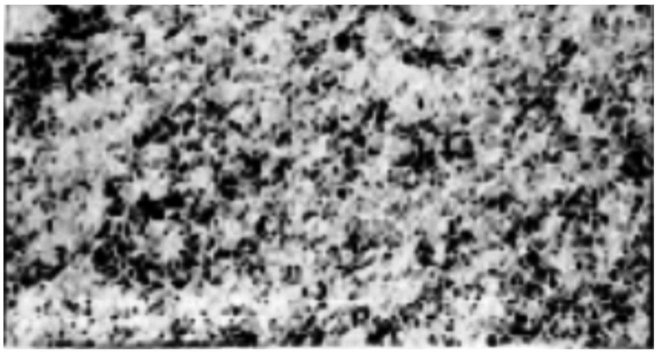

Plate 4: Fracture surface of the Sample at 75°C

The result in Plate 4 indicates that with increased hydrogen gas pressure, i.e. hydrogen content inside the material, enhances the propensity for intergranular cracking [7].

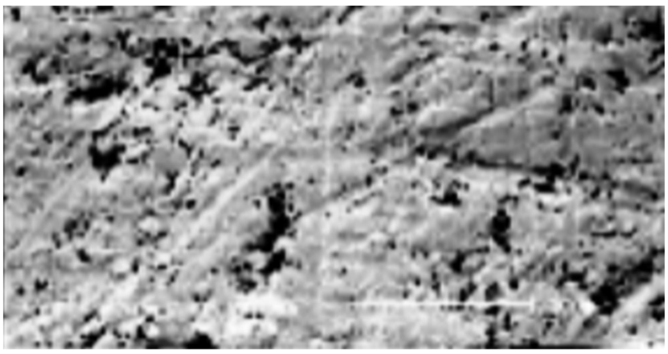

Plate 5: Fracture surface of the Sample at 100°C

Plate 5 shows microvoiding along the grain boundaries near the propagation tips, indicating hydrogen modified dislocation behavior and its interaction with grain boundaries and plays a crucial role in triggered the intergranular cracking [7].

The UNS steel specimen used during the course of the plastic loading in the media maintained at 25°C showed an increase in the electrons leaving the surface as captured by the increasing value of the current density. When the aging process was kept at 350 hours with samples immersed as shown by the jig arrangement. It follows that increasing the experimental temperature alongside time of charging led to increase in the current density. The samples failed in almost an embrittling manner with the increase in pre stressing coupled with the pre charging. As shown in Figure 4, observation of the graphical trend showed decreasing tensile strength as the recharging period increases. However, in Figure 5, the strain varied sinusoidally with time. The strain rate was critically reduced because of plastic loading was affected by the acid pre-charging leading to a compromise on the mechanical strength. It can be deduced that:

i. At entrance surface there is Hydrogen molecule adsorption, Hydrogen dissociation and dissolution.
ii. Within the metal there is Hydrogen diffusion and Hydrogen trappings.
iii. On exit surface, Hydrogen recombination and Hydrogen desorption depends on the strain rate and temperature of the material.

4. Conclusion

The (UNS) G10180steel grade ordinarily can withstand tensile stress above 830 (Mpa) when not subjected to a degrading or deteriorating media like hydrochloric acid used for the pre-charging process. It was discovered by calculation that the pre-charging by hydrochloric acid raised the susceptibility of embrittlement by hydrogen specie by 25%. Increment in temperature of the electrolytic bath by every 25°C for the number of experimental hours further raised the susceptibility of the metal failure by 15%. This led to the lowered tensile strength or low bearing capacity by almost 10% compared to the as-received samples. The strain also decreases due to pre-stress and pre charging at the temperatures used. It can therefore be said material like UNS steel can fail without apparent deformation in the field assuming they were placed under high temperature and under high loading complemented by acid attack.

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