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STATIC AND DYNAMIC LOAD RESPONSES OF 304 STAINLESS STEEL IN CHLORIDE SOLUTION AT LOW TEMPERATURES

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Abstract

The cryogenic properties of metal alloys have received great attention with new developments in petrochemical processes that require operation at low temperature. This paper presents the evaluation of 304 stainless steel under static and dynamic loading at extreme temperatures after being immersed in chloride solution. An environmental chamber was used to test the influence of sub-zero process temperatures on the tensile and corrosion properties of the specimens. Each specimen requiring pre-immersing in chloride solution was subjected to low temperature thermal cycling. The results show that with the decrease in temperature the mechanical properties of the immersed 304 stainless steel specimens under both static and dynamic loadings decreases as compared to dry specimens. However, the mechanical properties of the immersed specimens still acceptable since the decreases in mechanical properties were with 25% change for one low temperature, and they are close to the properties of dry specimens at most of the examined temperature. Microstructure examination of the immersed specimens reveals low ductility at low temperatures.

***Keywords**: 304 stainless steel; low temperature, dynamic load, static load, tensile test; microstructure analysis.*

1. Introduction

Austenitic Stainless Steel (ASS) 304L is Fe-Cr alloys with sufficient nickel and manganese (and sometimes nitrogen) to stabilize austenite, a face-centered cubic (FCC) phase. The most typical austenitic stainless steels are variations of the 18Cr-9Ni alloy. Although none of the commercial alloys were specifically developed for low-temperature service, several of them have been widely used in a variety of cryogenic applications [1]. Cryogenic engineering has a particuar need for the evaluation of the performance of materials at very low temperatures. Materials tend to become stiff and brittle at low temperatures; therefore, reasonable concerns are raised about their safety and reliability in such harsh environment. One the other hand, advanced and lighter materials and products for engineering applications, specially the structures, and systems are speedly replacing the typical and heavier materials and parts. There is an expanding need to assess these materials at extremely low temperatures to utilize them in engineering applications such oil production and other energy production [2].

This steel is being extensively used in the field of defense, nuclear science and cryogenic applications due to its excellent corrosion. This property of ASS 304L is due to the presence of chromium, which prevents chloride corrosion. Austenitic stainless steels are subjected to stress corrosion cracking (SCC) in solutions thoase contain chlorides [3–9]. In magnisium chloride solution, the scenarios of climate-induced corrosion cracking of austenitic stainless steels can be either hyrdogen embrillment(HE) or SCC, determind by the environment temperature [4-5]. A reasionable amount of cold work may reduce the SCC tendency of austenitic stainless steels, whereas too much cold work invert that trend [10-11]. The worked 304L SS showed an obvious increase in SCC tendency in compasion with the unmachined, cold worked and solution-annealed, unmachined samples [12-13].

The alloy steels that are used at cryogenic temperatures are tailored to combine high structural strength with good fracture toughness. For particular applications, they may also be required to meet other criteria, such as good fatigue resistance, weldability, low thermal expansion or low magnetic permeability. The steels that have been most often proposed for use in cryogenic structures can be conveniently divided into three categories: (1) Ferritic steels that contain 5-9Ni and are heat treated to have good combinations of strength and toughness at 77K. These are attractive because of their relatively low cost. (2) 300-series austenitic stainless steels, including particularly the low-carbon, high—nitrogen modifications, 304LN and 316LN, which combine high strength and toughness at 4K with good weldability. These alloys are metastable austenitic steels that transform under strain at 4K. (3) High-strength, stable austenitic alloys that have specifically developed for structural use at 4K. These include Fe-Ni-Cr N alloys (e.g., JNl: 15Ni-25Cr-4Mn-0.4N-O.3Si—0.01C) and Fe·Mn·Cr—N alloys (e.g., OCR Output JK1: 22Mn-13Cr-5Ni-0.2N-0.02C-0.5Si)[14].

It is generaly known that chloride stress corrosion cracking (CLSCC) starts from sites of potential pitting or crack corrosion [15] and as a sequence, cracks are estimated to grow in the concentrated chloride, strongly acidic, semi saturated solution that develops at active sites of localized corrosion. A theory [16] assumed that CLSCC only possible when a crevice grows more rapidly than the speed of material removal by pitting corrosion from the base of a crack or pit; in other words, it is a matter of competition between the speed of CLSCC growth and the speed of localized corrosion. Crack growth is also limited to the electrochemical potential that is determined by an upper limit where removal of materils exceeds crack growing and a lower limit determined by re-passivation. This proposition has been considered by Tsujikawa to demonstrate a temperature dependency of CLSCC since crevice growth increases more quickly with temperature than the speed of pitting corrosion. Stress corrosion study carried out by Tsujikawa and another workd carried at HE [17] have proven that it is very tricky to initiate CLSCC on smooth and bare specimens under test conditions when pitting corrosion of the surface under stress is missing. The tendency of austenitic stainless steels to CLSCC determined by a range of environmental parameters that include temperrature, pH, and chloride concentration. Temperature is the most effective environmental variable based on numerous studies. Cold regions material problems are many, and it is basically known that several alloys which show ductile behavior at room temperature face to become brittle under extreme conditions of low temperature or high rate of deformation [2]. The temperature range of practical interest for most of the cryogenic application is -60 and 0°C (Exploration and production of oil and gas have begun to take place in climates down to -60°C). At present, the knowledge necessary to prevent brittle fracture, which is fatal in engineering aplliation, is far from complete. There is an obvious lack of data on the mechanical behavior of austenitic stainless steels in chloride solutions at the extreme cold environment. Most of the available information regarding the corrosivity induced by chloride solutions are at high temperatures [18]. The aim of the work is the study of the rate of growth of stress corrosion cracking of traditional 304L austenitic stainless steels exposed to extremely low temperatures in chloride solutions.

2. Material

The steel specimens were prepared, using 30 mm thick plates of 304 L austenitic stainless steel. Table 1 shows the chemical composition properties of the specimens

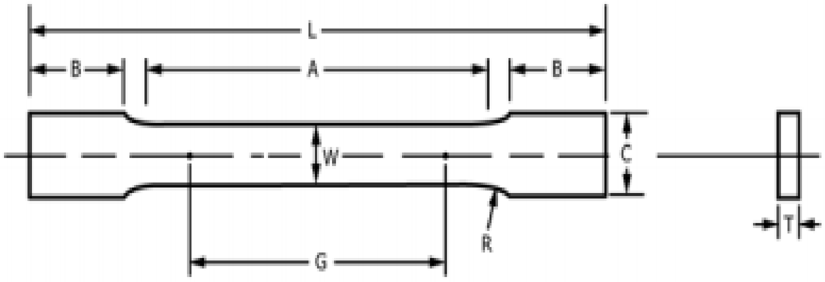
Table 1. Chemical composition steel specimen

	C	Mn	A P	S	Si	Cr	Ni	Cu	Mo	Pb	Ca	Sn
%wt.	0.052	0.084	0.021	0.01	0.49	18.25	9.51	0.21	0.18	<0.001	0.11	0.006

3. Method

Many experimental techniques can be used to determine, thermal, static and dynamic properties of materials under very low temperatures, like drop weight, tension, thermal conductivity, etc. in this study the impact of exposure to very low temperatures on mechanical properties of 304 stainless steel are studied under a tensile load. To maintain the desired temperature, all tests were conducted in an environmental chamber supplied with a temperature controller used to adjust the flow rate of liquid nitrogen, and the data were acquired and analyzed at each temperature.

The cryogenically treated specimens were immersed in deaerated 1 M NaCl solution in an open circuit potential cell for 1 hour. The specimens were dried kept in a desiccator until used for testing. Fig.1 shows the stranded shape of tension specimen.



G—Gauge length: 50 mm; W—Width 12.5 ± 0.2 mm; T—Thickness of material 0.5 mm; R—Radius of fillet, 12.5 mm; L—Overall length, 200 mm; A—Length of reduced parallel section, 57 mm; Length of grip section, 50 mm; C—Width of grip section, 20 mm

3.1. Tensile test

Universal testing machines (100 Series Universal Test Machines, Testresources, US, loads up to 10 kN (2,250 lbf)). This series includes five load frames, four actuators, two controllers, three software levels and load cells to the test requirements. Fig. 2 shows the experimental equipment used for tensile stress tests.

Fig. 2. Tension-compression equipment

Displacement control was applied to the tensile test at all temperatures, and a quasi-static strain rate of 2 × 10^{-3} /s was applied. In this process, liquid nitrogen is used for extreme cooling in an environmental chamber. In this chamber, liquid nitrogen is passed over the shaped specimens for 90 minutes to reduce the temperature from room temperature to 0, -15, -30, -45 and -60 °C. The chamber will be maintained at the desired temperature for 10 hours and leave for getting back to ambient temperature naturally. The specimens of 16 cm^2 area immersed in the solution, were used to obtain the data of electrochemical noise. The second group of specimens was used to collect the electrochemical noise data during localized corrosion; those were with 22 cm^2 surface area immersed in the chloride solution under tension conditions. The data of signals created by electrochemical noise were acquired at 4 H frequency. The reference electrode was used in this test represented by calomel electrode.

4. Results and discussion

4.1. Dynamic load-displacement behavior

Figure 3 shows the dynamic load –displacement results of the immersed specimens at different low temperatures to determine the effect of high loading rates on 304 SS performance. It is observed that the behavior at low temperatures is completely different from that at ambient and high temperatures that have been studied earlier [19-22]. The effect of temperature on dynamic load–displacement curves of 304 SS obtained during instrumented impact experiments is oscillatory. Fig. 3 showed overlap fluctation of the load signal due to the typical dynamic effects of tension loading [23]. It also shows that the highest displacement occurs is 0.17 mm at 0^{o}C with the loading of 40,000 N. The displacement becomes less at lower temperatures because of brittleness behavior [24]. At higher temperature, it becomes more ductile and displacement increases due to the expansion of 304 SS.

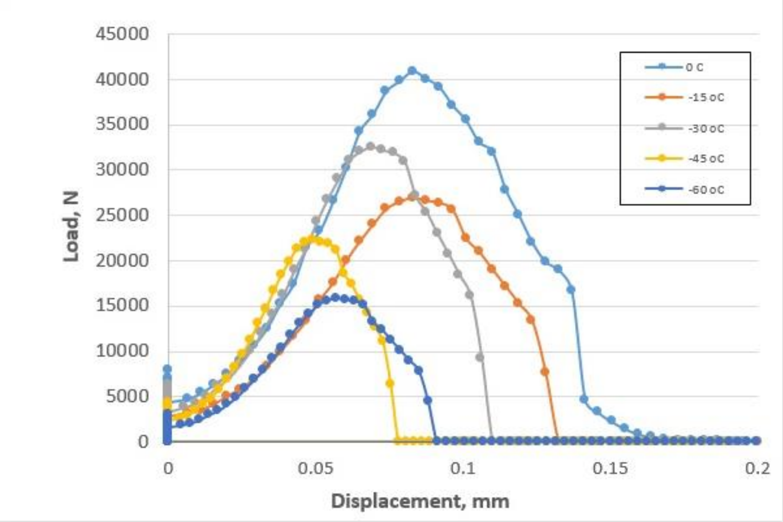


Fig. 3. Dynamic load vs. displacement curves for immersed 304 SS at different low temperature

Fig. 4 shows the effect of temperature on dynamic load-displacement curves for the dry 304 SS specimens.

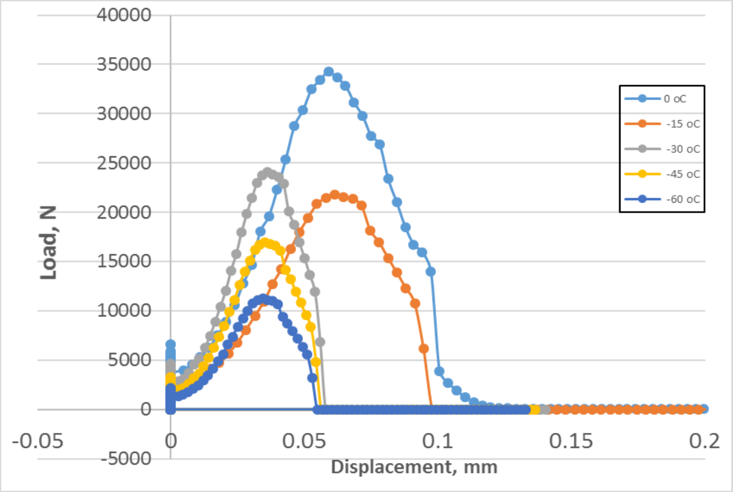


Fig. 4 Dynamic load vs. displacement curves for dry 304 SS at different low temperature

The metal strength and modulus can degrade with increasing temperature [25]. However, testing at low temperature revealed high strength and modulus. Figure 4 shows less oscillation in load-displacement curves and the lowest displacement, 0.05 mm, at -60°C. Although the 304 SS creeps under a sustained load, it can be designed to perform satisfactorily. The fiber itself is regarded as an isotropic material and has a lower thermal expansion coefficient than that of steel [26].

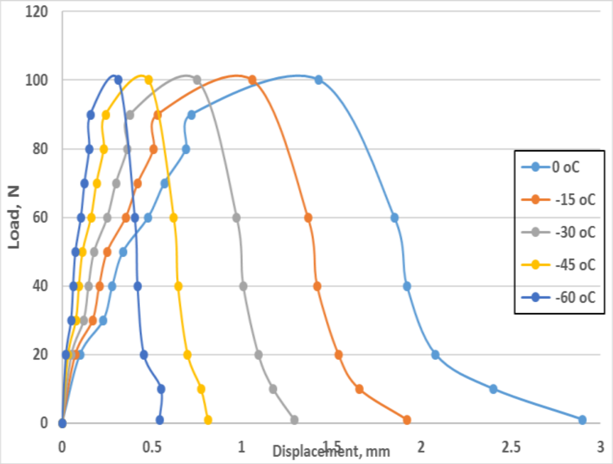


Fig. 5. Static load vs. displacement curves for immersed 304 SS at different low temperature

4.2. Static load-displacement behavior

Response to the static load applied on immersed 304 SS is shown in Figure 5, which represents load-displacement curves obtained by the impact tester at different low temperatures. It is evident that response to static load is more stable than the response to dynamic load. All curves have similar trends and exhibit the highest displacement, 2.9 mm, at 0°C.

The behavior follow fourth order polynomial as follow:

$$D= a+ bl+ cl^2+ dl^3+ el^4 \qquad (1)$$

where D: Displacement, mm and l is static load, N.

The numeric values of polynomial coefficients are listed in Table 2:

Table 2. Polynomial coefficients of static load-displacement curves for immersed 304 SS at different low temperatures

Temperature	a	b	c	d	e	R^2
0°C	2.216	129.28	30.401	80.865	18.973	0.9675
-15°C	5.334	118.84	233.85	366.57	110.33	0.9831
-30°C	5.4175	164.59	479.42	1050.4	447.37	0.9825
-45°C	5.1102	269.51	1084.3	3834.4	2565.7	0.9811
-60°C	5.8474	367.1	3102.8	-15755	16181	0.979

Fig.6 shows the static load-displacement behavior at different low temperatures for the dry 304 SS. As for the dynamic response, the displacement due to applying the static load is much less than the immersed metal. This is due to shrinkage of the structure and transformation from ductile to brittle metal. Table 3 lists the polynomial coefficients of equation 2 for dry 304 SS.

Table 3. Polynomial coefficients of static load-displacement curves for the dry 304 SS at different low temperatures

Temperature	a	b	c	d	E	R^2
0°C	18.089	230.05	96.935	47.677	21.122	0.9746
-15°C	1.4285	336.73	346.27	20.15	64.81	0.9134
-30°C	1.3708	387.59	227.25	-787.65	588.19	0.9542
-45°C	4.7264	896.95	2959.8	3001.1	846.97	0.9219
-60°C	-4.781	1383.2	-7040.5	11038	4849.9	0.921

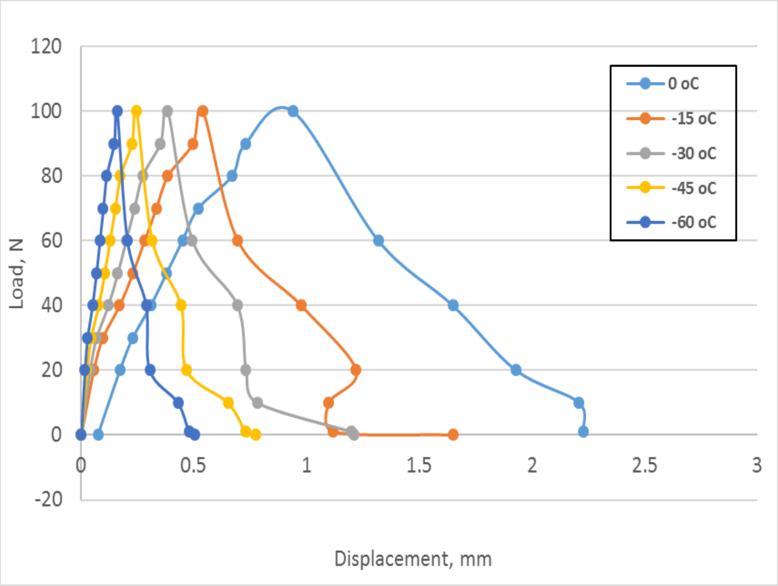


Fig. 6. Static load vs. displacement curves for dry 304 SS at different low temperature

4.3. Stress corrosion cracking

A microstructure study was conducted on the failed immersed specimens loaded with 10 kN. Several small cracks have been observed that resulted in specimen failure. Most of the fracture is brittle as shown in Figure 7 that is account for 70% of the fracture, whereas the rest of the surface fracture was ductile as shown in Figure 8 due to applying the load at 10 kN.

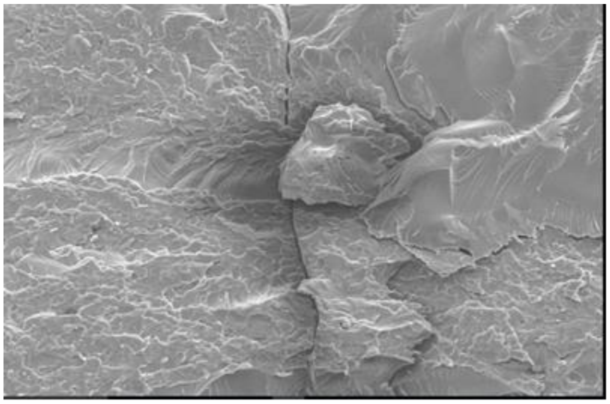


Fig. 7. Brittle part of fracture for the immersed 304 SS at -60ºC

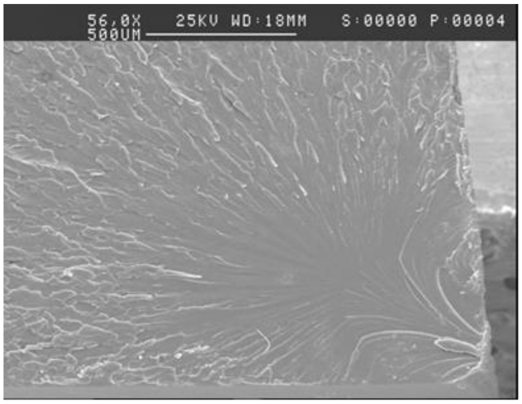


Fig. 8. Ductile part of fracture for the immersed 304 SS at -60ºC

5. Conclusions

The mechanical characteristics of 304 stainless steel immersed in chloride solutions under static and dynamic loading have been investigated through tensile tests by various sub-zero temperatures and SEM characterizations in the present study. Immersion of the tested specimens in chloride solution reduces the tensile strength by approximately quarter at zero temperature under static loading. Also the ductile properties were reduced however, the tensile elongation was maintained at extreme low temperatures. Under dynamic loading, tensile elongation was enhanced by immersion in chloride solution for all sub-zero temperatures examined. It is likely to be more effective at sub-zero. However, the results of microstructure showed that immersion in chloride solutions has a significant impact of immersion on ductility. It showed major reduction in ductility that resulted in brittle failure.

Declarations

Authors' contributions

Saad Ahmed made substantial contributions to the present study specifically in the conception, experimental works, and data analysis. He also administrates the SEM test of the failed specimens and prepared them for testing. Since he is the principal author of the manuscript, he was involved in the drafting of the manuscript, reading and approved the final manuscript.

Competing interests

The author declare that they have no competing interests.

Finding

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Availability of supporting data

Data will not be shared during the current study because of an agreement with the funder.

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