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Environmentally assisted cracking of 18%Ni maraging steel

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ABSTRACT

Slow strain rate testing of notched cylindrical specimens of 18Ni2400 maraging steel has been carried out in air with 30% relative humidity and synthetic seawater environments. Peak-aged condition has been chosen, considering the relevance to engineering applications. Studies have also been carried out with different notch geometries to understand the effect of stress concentration factor. It is concluded from the study that (i) degree of stress concentration at the notch influences the notched tensile strength (ii) mild hydrogen embrittlement seems to occur in air environment, (iii) synthetic seawater environment drastically brings down the notched tensile strength and time to fracture (iv) environmentally assisted cracking occurs in air tests in quasicleavage and microvoid coalescence modes and in seawater tests in intercrystalline mode.

1. Introduction

Maraging steels, possessing the unique combination of performance characteristics – high strength, high toughness and good formability – have been widely used in aerospace, military and other critical applications. Different grades of maraging steel are commercially available covering a strength range of 1400–2400 MPa. With increasing strength level, the fracture toughness and formability decrease. Accordingly, the lower strength variants are used where high fracture toughness/ductility are important for design. The higher strength variants are used for applications where one can manage with moderate levels of fracture toughness but strength is more important. Titanium is used as the primary strengthening element in these steels and precipitation of a fine dispersion of titanium bearing intermetallic particles in martensite as a result of aging leads to a very high strength condition. Hence, the titanium level in the steel increases as one moves from low strength to high strength variants. Aging has to be optimally carried out to realize the maximum strengthening effect of the precipitates. Important developments and applications have been reviewed in 1988 Symposium [1] and recently by Rao [2].

It is known that environmentally assisted cracking (EAC) can lead to severe degradation of high strength steels. The higher the strength level, the more is the expected susceptibility to stress corrosion cracking (SCC) and hydrogen embrittlement (HE). The threshold stress intensity for SCC for high strength steels decreases with increasing yield strength. Increasing the yield strength of martensitic and precipitation hardening stainless steels, for example, increases the probability of cracking by SCC [3]. The susceptibility of steels to HE also generally increases with increasing tensile strength. Even small amounts of hydrogen picked up from the environment can have a deleterious effect, if the tensile strength of the steel is 1240 MPa or more [3]. Most high strength steels are susceptible to HE when they are stressed even under atmospheric exposure conditions. In addition, the environment can become highly aggressive if locations with stress concentration, e.g., notches are present. Notches with higher degree of stress concentration can be more damaging to the life of the structure. For example, Hardie and Liu [4] studied the effect of K_t on NTS of a high strength steel (ultimate tensile strength of 1720 MPa) in a hydrogen environment and found that NTS decreased with increasing K_t.

Maraging steels stand out for their high strength level and as such it is important to know as to what extent the phenomenon of EAC comes in the way of their usage for high load bearing structural applications. There is some published literature on EAC studies specific to maraging steels. It has been reported that the threshold stress intensity needed for SCC of maraging steels in aqueous environments decreases as yield strength increases [3]. Bradhurst and Heuer carried out slow strain rate tests in air and aqueous NaCl solution on three different grades of 18%Ni maraging steel using smooth specimens and reported that susceptibility to environment sensitive cracking increased with increasing strength of the steel [5].There have been a number of investigations in recent years on EAC of 18%Ni cobalt-free maraging steel grades 18Ni1400 and 18Ni1700 grades involving H_2S saturated solution, gaseous hydrogen and humid air as the test media [6–10]. The investigations also covered the effect of microstructure in the aged condition on the susceptibility to EAC. It was demonstrated that severity of EAC depends, among others, on degree of aging. It has been reported [11] that the ambient

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air environment, i.e. with no corrosive media surrounding the material, can by itself cause HE of cobalt-bearing 18%Ni maraging steel with a yield strength of ~1700 MPa under certain circumstances. For the cobalt-free 18%Ni maraging steel T-250 (yield strength ~ 1700 MPa), Zhang et al. concluded [9] that hydrogen assisted cracking (HAC) takes place in constant displacement tests in air with RH $\geqslant$ 30%. It has been reported that as with high strength steels, the more severe the notch, the greater is the susceptibility to SCC of 18%Ni maraging steels [3]. INCO published results of a study carried out on 18Ni1700, a 1700 MPa yield strength variant of 18%Ni maraging steels, bringing out that NTS decreases with increasing severity of the notch [12].

The purpose of the present investigation is to study the susceptibility to EAC of 18Ni2400 grade maraging steel in synthetic and 30% relative humidity air environments using notched specimens and slow strain rate testing. The effect of varying notch severity on the cracking process in the two environments was also studied. Material was tested in the peak-aged condition with the corresponding strength level being the highest obtainable with the commercially available 18%Ni maraging steels. Previous researchers [4,6,8,10,13] have adopted, among others, drop in notched tensile strength (NTS) in a corrosive medium compared to air environment as measures of susceptibility. Drop in time to fracture (t_f) in a corrosive medium compared to laboratory air environment has also been adopted as a measure of susceptibility [14]. These two criteria have been used in the present study for characterizing the behavior of the steel in synthetic sea water environment.

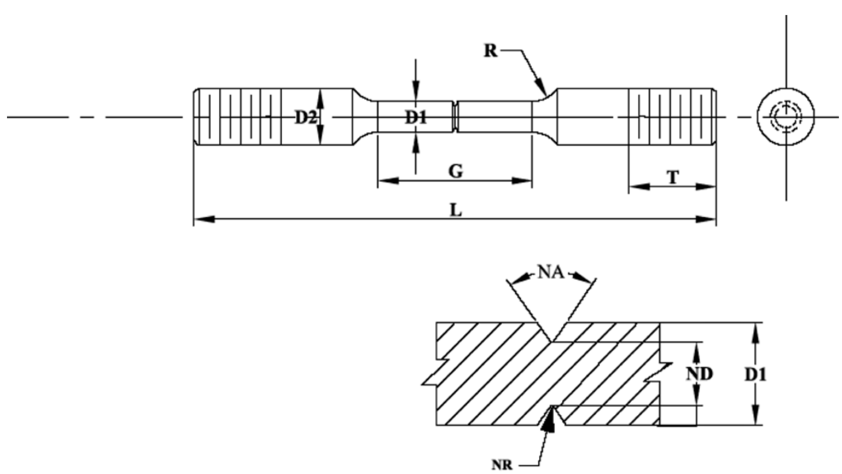
2. Experimental

The 18Ni2400 grade maraging steel used in this investigation was processed through vacuum induction melting followed by vacuum arc remelting. Hot working of the resulting ingot was carried out to produce the material in the form of rods 15 mm in diameter. The chemical composition of the alloy in weight percent was 18.22 Ni, 3.94 Mo, 1.57 Ti, 0.1 Al, 0.003 C, 0.1 Si, 0.1 Mn, 0.001 S, 0.006 P and balance Fe. The material was in the solution annealed condition with a hardness level of 331 BHN (35.5 HR_C).

The drawing of the notched tensile specimen adopted in this study is shown in Fig. 1. Specimens with different values of stress concentration factor (K_t) in the range 2.82–4.80 were prepared from the rod material in the solution annealed condition. The K_t factor was derived from Fig. 4 and Table 2 of the paper "Stress concentration factors for round and flat test specimens with notches" published by Noda et al. [15] giving calculation of K_t values for notched round bars with 60° V-shaped circumferential notches. Based on the value of K_t the specimens can be categorized into different groups as shown in Table 1. After the specimens were machined, peak aging treatment was given to them. The treatment comprised of 3.5 soaking hours at 510 °C. Viswanathan et al. carried out detailed Transmission Electron Microscopy of this grade of steel and concluded that two types of precipitates exist in the peak-aged condition – rod shaped Ni_3 (Ti, Mo) and spherical Fe_2Mo intermetallic phases [16]. The hardness after aging was 59.2 HR_C. Oxide layer formed during aging was removed by polishing with a fine emery paper. The notch was polished with a cotton thread with application of diamond paste. The specimens were subjected to slow strain rate testing (SSRT); the three strain rates used in this study were 4.17×10^{-6}, 8.33×10^{-7} and 2.10×10^{-7} s^{-1}.

The tests were carried out in two environments: (i) air having a relative humidity of ~30% (henceforth referred to as 30% RH air) (ii) synthetic seawater. The seawater has been prepared by the method devised by Kester et al. [17].

The susceptibility to cracking in synthetic seawater has been calculated as the fractional loss of NTS in seawater environment compared to air environment as shown below:



Nominal dimensions in mm

G-Gage Length	= 30.0
D1-Diameter of Gage section	= 6.0
D2-Diameter of Shoulder	= 11.1
R- Radius of Fillet, min	= 6
L-Total Length	= 101.6
T-Thread Length	= 19.0
NA- Notch angle	= 60°
ND-Notch Diameter	
NR-Notch Root Radius	

Fig. 1. Drawing of the notched tensile specimen adopted for this study.

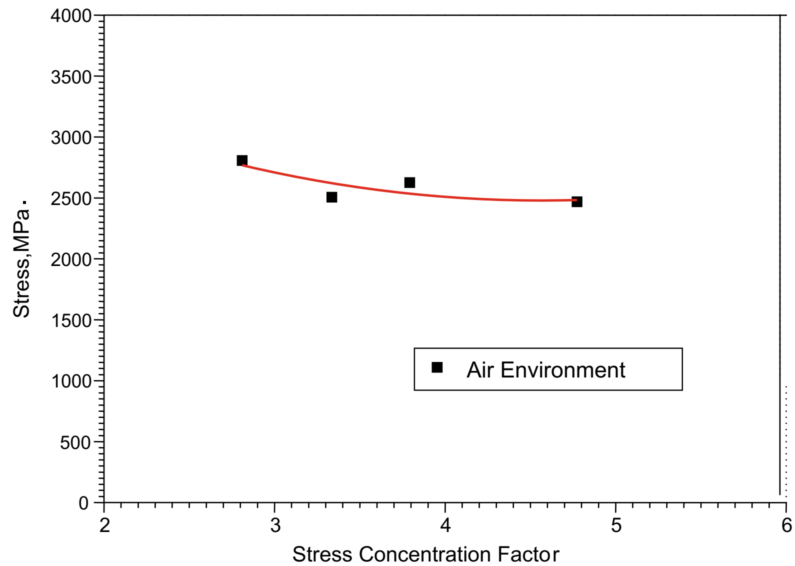


Fig. 2. Effect of stress concentration factor on notch tensile strength for tests carried out in 30% RH air at the strain rate of $4.17 \times 10^{-6}\,s^{-1}$.

$$\text{NTS loss} = \frac{[\text{NTS(in air)} - \text{NTS(in synthetic seawater)}]}{\text{NTS(in air)}}$$

The susceptibility to cracking in synthetic seawater has also been calculated as the fractional drop (t_f) in seawater compared to air environment:

$$t_f = \frac{t_f(\text{in air}) - t_f(\text{in synthetic water})]}{t_f(\text{in air})}$$

Tensile fractured specimens were examined in a scanning electron microscope (SEM) to get information about crack initiation sites and mode of fracture.

3. Results and discussion

3.1. Slow strain rate testing in 30% RH air environment

For the tests carried out at the strain rate of $4.17 \times 10^{-6}\,s^{-1}$, the NTS has been plotted in Fig. 2 as a function of the $K_{t.}$ There appears to be a decreasing trend of NTS with increasing $K_{t.}$ INCO has published results of a similar study carried out on 18Ni1700, a lower strength variant of 18%Ni maraging steel [12]. INCO's result brings out that NTS of 18Ni1700 decreases with increasing severity of the notch.

Fig. 3 shows the three data points experimentally obtained in the phase field of strain rate vs. NTS for samples with K_t of 3.35 tested in 30% RH air. There appears to be a decreasing tendency of NTS with decreasing strain rate. More data points would enable to reach a definitive conclusion. It is seen that the test carried at the lowest strain rate has given the lowest NTS of all the tests carried out in 30% RH air in this study.

Fractographic examination of fracture surfaces revealed that crack initiation occurred at the root of the notch. Fig. 4 shows a low magnification view of the fracture surface of specimen No. 9 pulled at the strain rate of $2.1 \times 10^{-7}s^{-1}$, indicating the location where crack has started. When viewed at high magnification, the modes of material separation in the elliptical flat fracture region were found to be quasicleavage and microvoid coalescence. Fig. 5

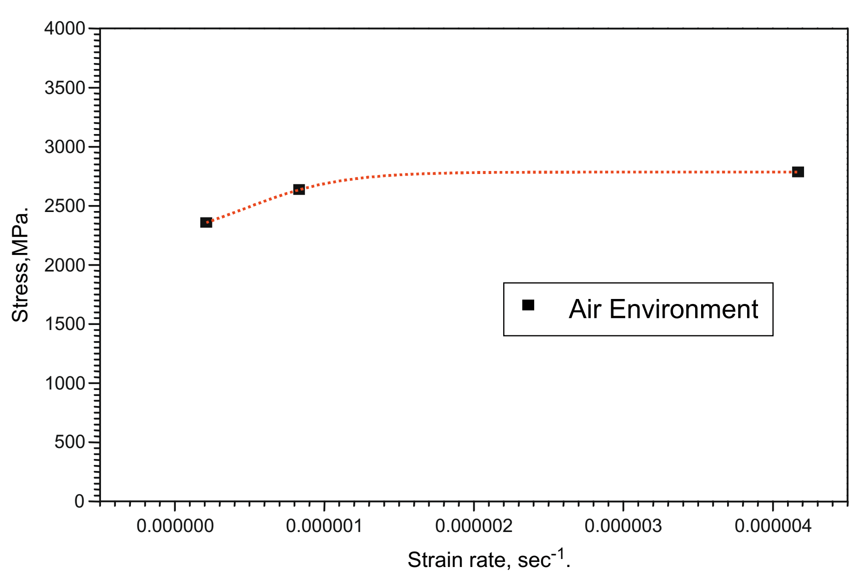


Fig. 3. NTS vs. strain rate for tests performed in 30% RH air; $K_t = 3.35$.

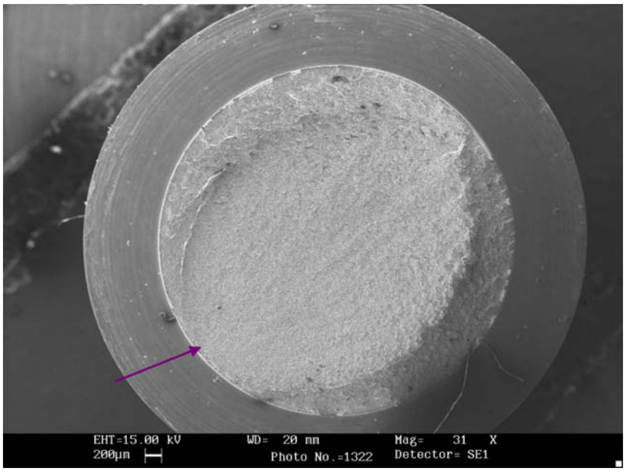


Fig. 4. Low magnification view of the fracture surface of the specimen No. 9 tested in 30% RH air.

is a typical high magnification view of the flat fracture surface. No intergranular facets were noticed on the fracture surface.

The dependence of NTS on K_t can be explained by assuming that hydrogen in the environment is playing a role in the fracture process. A higher K_t associated with the root of the notch is expected to lead to a higher degree of accumulation of atomic hydrogen at the notch tip, higher degree of embrittlement and consequently lower applied stress level at which failure occurs. Hardie and Liu [4] studied the effect of K_t on NTS of a low alloy steel heat treated to a very high strength level (ultimate tensile strength of 1720 MPa) in a hydrogen environment and found that NTS decreased with increasing K_t. They concluded that increasing severity of the notch leads to increasing degree of HE in the hydrogen environment.

The apparent dependence of NTS on strain rate can also be explained based on the assumption that hydrogen is playing a role in the fracture mechanism. It has been well documented for the high strength steels that the extent of HE increases with decreasing strain rate [18]. Bradhurst and Heuer [5] carried out tensile tests in air over the strain rate range 5×10^{-4}–$5 \times 10^{-7}\ s^{-1}$ on unnotched samples of 18Ni2400 steel and found that fracture strength decreases with decreasing strain rate. The level of humidity in the air was not reported. The authors explain the behavior in terms of slight HE effect.

The question then to be answered is whether HE of 18Ni2400 steel is possible in 30% RH air environment. There are several pieces of evidence to support the view point that it is possible. For the lower strength cobalt-free 18%Ni maraging steel T-250 (yield strength ~ 1700 MPa), Zhang et al. concluded [9] that HAC takes place in constant displacement tests in air with RH $\geqslant$ 30%. For the cobalt-bearing 18%Ni maraging steel with yield strength level of ~1700 MPa, it was concluded [11] that moisture in the air can cause HE. The steel grade under study has a much higher yield strength level (2400 MPa) in the condition in which it is investigated. It has been well established that the tendency to HE increases with increasing strength level of the steel. Extending the same logic, within the family on 18%Ni maraging steels, the higher the strength the more is to be the susceptibility to HE. Further in high strength steels subjected to an applied tensile stress, if a stress concentrator is present on the surface of the specimen, HAC is likely to originate at the stress concentrator [3]. The notch in notched specimens used in the present study acts as the stress concentrator and fractographic examination has indeed shown that crack has initiated at the root of the notch. It is believed that SCC is not coming into picture in the tests done in 30% RH air, as it is a benign and not corrosive environment. Based on the foregoing it is concluded that a mild HE has occurred in 30% RH air environment.

3.2. Slow strain rate testing in synthetic seawater environment

Table 2 shows the NTS value as a function of strain rate for tests carried out on specimens with K_t in the range – 3.04–3.09. This range being narrow, effect of variation in K_t on NTS can be ignored as far as results in Table 2 are concerned. There are test results available on two specimens for the intermediate strain rate $8.33 \times 10^{-7}\ s^{-1}$, whereas only one result is available for the other two strain rates. The NTS value obtained at the lowest strain rate is the least of all the NTS values obtained in the present study. There is high scatter of NTS values, as can be seen from the two test results available for the intermediate strain rate, emphasizing that it is necessary to test more than one specimen for each combination of test parameters, to arrive at an average/representative value. However, for want of adequate test material, this aspect could not be taken care in the present study. While this limitation exists, it seems likely that the NTS in synthetic sea water tests decreases with decreasing strain rate. That increasing degree of environmentally assisted damage takes place with decreasing strain rate during the SSRT runs is only to be expected. It is worth noting in this context that the results of Bradhurst and Heuer [5] also show that fracture stress of 18Ni2400 decreases with decreasing strain rate when tested in 3.5 wt.% NaCl solution under cathodic conditions.

Table 3 shows the NTS values obtained with two different K_t values, when testing was done at the strain rate of $4.17 \times 10^{-6}\ s^{-1}$. The NTS value corresponding to K_t of 3.5 (1468 MPa) is much smaller, compared to the NTS value (1092 MPa) when K_t is 2.82. These are only two results; it is possible that the effect of the severity of the notch on the NTS value in tests is similar to that noticed in 30% RH air tests. It has been reported that as with high strength steels, the more severe the notch, the greater is the susceptibility to SCC of 18%Ni maraging steels [3].

Table 4 brings out a comparison of the NTS and time to failure values obtained in air and synthetic seawater environments for the strain rate $4.17 \times 10^{-6}\ s^{-1}$, where a one to one comparison is pos-

Table 1
Grouping of specimens tested based on stress concentration factor.

Specimen	No.	Stress concentration factor (K_t)
Group A	8	4.80
Group B	7	3.81
Group C	12	3.50
Group D	2	3.35
	6	3.35
	9	3.35
Group E	1	3.04
	11	3.09
	13	3.08
	14	3.08
Group F	4	2.82
	10	2.82

Table 2
NTS as a function of strain rate for tests carried out in synthetic seawater; K_t in the range 3.04–3.09.

Strain rate (s^{-1})	NTS (MPa)	Specimen no.
4.17×10^{-6}	1086	14
8.33×10^{-7}	1131	11
8.33×10^{-7}	627	13
2.10×10^{-7}	595	1

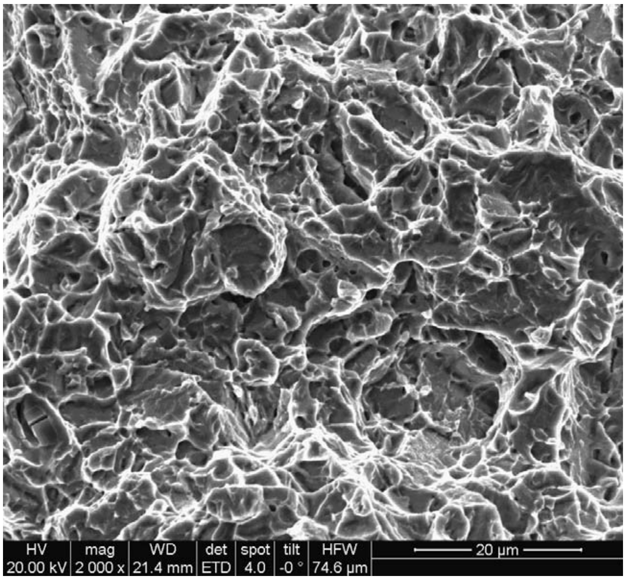


Fig. 5. Typical high magnification view of the elliptical flat fracture surface of specimen No. 9.

Table 3
Effect of K_t on NTS in tests carried out in synthetic seawater; strain rate: $4.17 \times 10^{-6}\ s^{-1}$.

K_t	NTS (MPa)	Specimen no.
2.82	1468	4
3.50	1092	12

sible with the data available. The values obtained in seawater are much smaller. While the data is very limited, it appears that seawater environment leads to a drastic reduction of NTS and t_f. Ratios in the range 0.8–1.0 normally denote high resistance to EAC. The ratio in the present study is 0.53 for the NTS and 0.57 for t_f at the highest strain rate used. This shows that the maraging steel grade tested is highly susceptible to corrosive damage in seawater environment.

Fracture surfaces were studied using the SEM technique. Crack initiation occurred in all cases at the root of the notch. The fracture surface consisted of essentially two regions: (i) the region next to the crack initiation point, where material separation occurred as a result of EAC and (ii) region where failure occurred as a result of overload fracture. Fig. 6 illustrates this for the case of specimen No. 13 tested at the strain rate of $8.33 \times 10^{-7}\ s^{-1}$. Region A failed by EAC and region B by overload fracture. Fig. 7 shows the SEM picture of typical fracture appearance of the region A at high magnification. It can be seen that material failed by intergranular fracture. The figure also shows incidence of secondary cracking along grain boundaries. For all the specimens tested in synthetic seawater, regardless of the strain rate, intergranular fracture dominated the EAC region. Microvoid coalescence and quasicleavage comprised the fracture modes in the region B as illustrated by Fig. 8, showing the region B of the fracture surface of specimen No. 13 at a high magnification.

Table 4
NTS and t_f ratios – synthetic seawater to 30% RH air environment.

	Specimen no. 4 (30% RH air)	Specimen no. 10 (synthetic seawater)	Ratio
K_t	2.82	2.82	
NTS (MPa)	2768	1468	0.53
t_f (h)	5.24	3.00	0.57

There is strong evidence that the resistance of high strength steels to SCC in many corrosive media decreases as the strength increases. Maraging steels, like other ultra high strength steels,

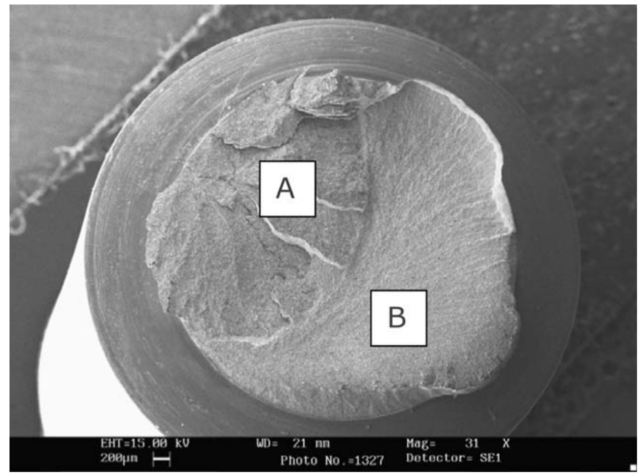


Fig. 6. SEM fractograph showing two distinct regions on the fracture surface of sample tested in seawater (Strain rate = $8.33 \times 10^{-7}\ s^{-1}$).

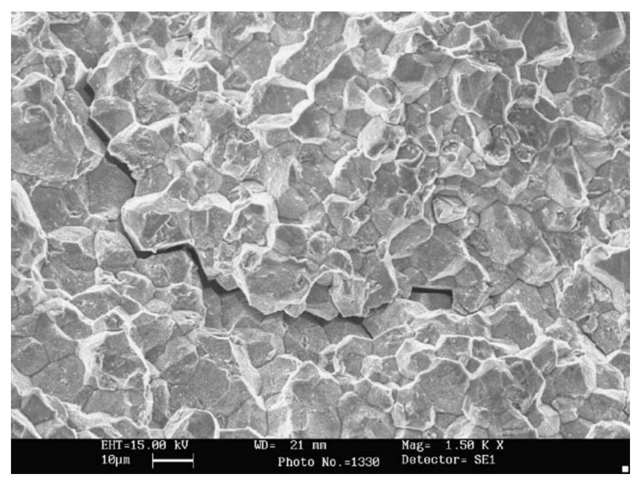


Fig. 7. Typical high magnification view of the fracture surface generated by EAC in seawater tests.

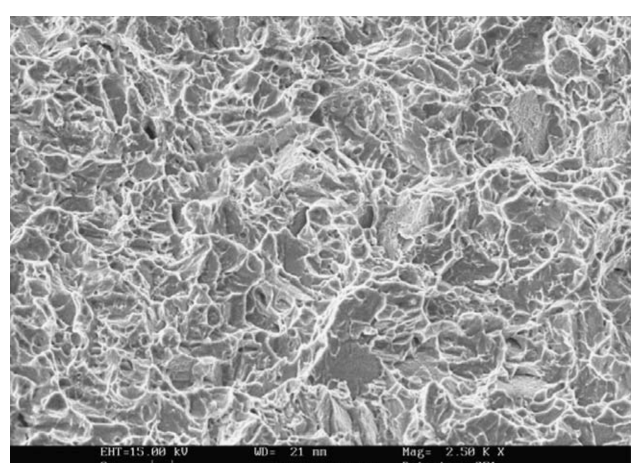


Fig. 8. SEM fractograph showing typical appearance of region B of the fracture surface of specimen No. 13.

are susceptible to SCC in most aqueous environments. A yield strength of about 1400 MPa has been mentioned as the threshold value, above which the vulnerability to catastrophic cracking can be expected [19]. For example, the maraging steel grade 18Ni1700 when tested in solution treated condition with a yield strength of ∼1000 MPa shows little or no susceptibility to SCC in 3.5% NaCl solution. The same steel in aged condition with a yield strength of ∼1700 MPa shows strong susceptibility, the K_{ISCC} value dropping to ∼50% of the fracture toughness K_{Ic}. The steel being studied here is in the peak-aged condition and comes under the ultra high strength category. This explains the observed high degree of susceptibility to corrosive damage in seawater environment. The mechanism of cracking operating in maraging steels under free corrosion conditions is still debated [19]. Detailed treatment of the underlying mechanism(s) is not in the scope of this paper.

4. Summary and conclusions

To study the tendency of aged 18Ni2400-maraging steel to EAC, slow strain rate tensile tests were carried out on notched round specimens. The experiments were conducted in 30% RH air and synthetic seawater at three different strain rates. Testing was conducted in the peak-aged state. It appears that there is a mild HE occurring in the steel in 30% RH air environment at the strain rate of $2.10 \times 10^{-7}\ s^{-1}$. It appears that the EAC is sensitive to the severity of the notch.

Testing in synthetic seawater appears to, based on the very limited data available, lead to drastically reduced NTS and time to fracture (t_f) values compared to testing in air. The very high strength of the steel in the tested condition, it is believed, has importantly contributed to the high susceptibility to cracking in seawater. Testing in synthetic seawater promoted intercrystalline fracture in the region in which fracture occurred by EAC.

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