JOURNAL OF APPLIED ELECTROCHEMISTRY **22** (1992) 396–403

Slow strain rate and constant load tests of A 285 and A 516 steels in Bayer solutions

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Received 3 July 1989

Stress corrosion cracking (SCC) experiments were carried out on A 285 and A 516 steels by slow strain rate and constant load techniques. The influence of the impurities contained in Bayer solution on the susceptibility to SCC was studied. The results obtained by both techniques indicated that aluminate ions promote passivation but the oxide layer includes numerous defects and the SCC susceptibility increases. The slow strain rate experiments provided a rapid method for investigating caustic stress corrosion cracking and intensified the susceptibility to SCC compared to the results obtained by the constant load technique. In the passive potential region, some shallow cracks were detected only when the experiments were conducted at slow strain rate in solutions containing aluminate ions. The presence of such cracks indicated that anodic protection could not entirely prevent SCC in the Bayer solution.

1. Introduction

In the Bayer process for extracting alumina from bauxite ores, welded reaction vessels and pipes made of carbon steel frequently suffered from stress corrosion cracking (SCC) during service [1–3]. This resulted from corrosion in the caustic environment which is used for dissolving hydrated aluminum oxides in the bauxite ores. Numerous investigations on caustic embrittlement have been made during the last 50 years [1–5]. However, very limited information exists on corrosion in caustic aluminate solutions. Some research work has been carried out for determining the influence of AlO_2^- ions and other impurities dissolved in Bayer solutions on the SCC phenomenon.

The aim of the present work is to study the susceptibility to SCC of two carbon steels used for pressure vessels, namely the A 285 and A 516 steels (ASTM standards) in media containing AlO_2^- ions and other impurities of Bayer solution. The embrittlement of the steels was evaluated by slow strain rate and constant load tests, two techniques frequently employed to study stress corrosion. The comparison of the results obtained by these two methods helped to investigate both crack density and crack propagation. The failures were examined by scanning electron microscopy. The goal of this study is to relate the caustic embrittlement obtained in the laboratory by different techniques to that found practically. Some protective methods such as anodic protection are discussed.

2. Experimental details

2.1. *Material*

Studies were carried out on two carbon steels, A 285 and A 516 (ASTM standards – presure vessel quality) with the chemical composition presented in Table 1.

The tensile specimens were cut from the steel plates in the as-received condition which consisted of a ferrite plus pearlite microstructure and machined to yield cylindrical specimens of 25 mm gauge length and 4 mm diameter. The end of the test pieces were threaded to receive tensile grips. Prior to testing, they were polished with emery paper of grit 600, rinsed in water, alcohol, distilled water and dried. Teflon tape was wrapped over the surface above and below the gauge length.

2.2. *Electrolytes*

Test environments included:
(a) 2.25 M NaOH solution
(b) Bayer liquor containing 3.6 M NaOH (2.25 M NaOH as free caustic soda), 0.67 M Al_2O_3, 0.46 M Na_2CO_3, 0.02 M Na_2SO_4 and 9 g l^{-1} organic carbon species.

For studying the influence of each impurity of the Bayer solution, the caustic electrolytes having 2.25 M NaOH as free caustic soda, were dissolved with each species separately to obtain:
(c) caustic aluminate solution: 3.6 M NaOH and 0.67 M Al_2O_3;
(d) caustic carbonate solution: 2.25 M NaOH and 0.46 *M* Na_2CO_3;
(e) caustic sulphate solution: 2.25 M NaOH and 0.02 M Na_2SO_4;
(f) a mixed solution of 3.6 M NaOH, 0.67 M Al_2O_3, 0.46 M Na_2CO_3 and 0.02 *M* Na_2SO_4 for the study of the effect of organic species;
(g) a concentrated solution of 7.73 M NaOH; and
(h) a concentrated solution of 7.73 M NaOH saturated by Al_2O_3.

All the solutions except the Bayer liquor, were prepared from distilled water and analytical grade chemicals. The Bayer solution was obtained from a

Table 1. Chemical composition of A 285 and A 516 steels (wt %).

Steel	*C*	*Mn*	*P*	*S*	*Si*	*Cu*	*Ni*	*Cr*	*Mo*	*Sr*	*Fe*
A 285	0.19	0.71	0.020	0.016	0.01	0.007	0.004	0.033	0.004	0.002	balance
A 516	0.24	1.15	0.008	0.007	0.25	0.044	0.025	0.033	0.004	0.002	balance

precipitator of the Alcan plant at Arvida (Canada). All the solutions were deaerated by argon before and during the experiments. They were heated and maintained at 100 ± 2° C.

2.3. *Slow strain rate experiments*

These were conducted with a MTS machine or an apparatus of Cortest Inc (Cortest Series 3400 CERT equipped with a MSC-10 motor speed controller) at a rate of $2.6 \times 10^{-6}\,s^{-1}$. A two compartment cell made of Teflon surrounded the gauge length of the specimen. The construction of the cell was similar to that described previously for electrochemical studies [6, 7]. A platinum counter electrode was employed and the potential of the working electrode (tensile specimen) was measured with respect to a room temperature saturated calomel electrode. The reference electrode compartment was connected to the main cell by a Luggin capillary. Heating was achieved by an electrical resistance mantle wrapped around the main compartment. The potentials wer controlled by a Tacussel model RT 20-2 potentiostat.

2.4. *Constant load experiments*

These were carried out with an apparatus designed in house for operating in the plastic region [8], since plastic deformation is essential for caustic embrittlement. The constant load was applied through a pneumatic cylinder and a lever loading system. The procedure and cell were similar to those explained previously for the slow strain rate tests.

2.5. *SCC measurements*

The susceptibility of the steels to SCC was evaluated by the reduction of area (ROA), the elongation of the gauge length (ε%) and the time to failure (t_f). For these purposes, the diameter of the section and the gauge length were measured before and after each test, and were compared to those obtained from an experiment in oil at 100° C where corrosion does not occur.

The SCC of the steels was also verified by the detection of the secondary cracks along the gauge length by scanning electron microscopy using a JEOL-2553 apparatus. One half of the broken specimens was mounted in epoxy resin, grounded to the longitudinal mid-plane, polished and etched by nital 3%. The cracks were observed and the longest crack was then measured. The fracture surface was also examined.

Duplicate tests were run for reproductibility. Dispersion of the results was less than 10%, except for the constant load tests where it was of the order of 15%.

3. Results and interpretation

3.1. *Slow strain rate experiments*

The experiments were conducted at three levels of potential:

- the open circuit potential (o.c.p.) which was recorded throughout the test
- a critical potential for SCC situated in the active-passive zone (Table 2); this potential was determined by the transient method described elsewhere [6].
- a potential located in the passive zone to simulate anodic protection, close to that used practically [9], i.e. −250 mV/SCE.

3.1.1. Open circuit potential. The recorded open circuit potential of the two steels in different electrolytes are presented in Fig. 1a and b. The two steels showed the same passivation tendency with a noble shift of the o.c.p. The potential, located initially from −1.120 to −1.200 mV/SCE, reached a value of −250 to −500 mV/SCE at the end of the test, depending on the presence of impurities in the soluton and the studied steel.

In 2.25 M, the final o.c.p. was more noble than that recorded in other solutions containing Bayer solution impurities or a higher concentration of caustic soda. However, the time necessary to achieve that passivity was much longer. When aluminate ions AlO_2^- were present, the passivation was easier and took less time than in the previous cases, but the potential at that passive state was 70 to 100 mV more negative, indicating that the steel was more active. Carbonate ions, sulphate ions and organic carbon species dissolved in the electrolytes had less influence on the passivation

Table 2. Critical potentials to SCC found by the transient method [*6*]

*Electrolyte**	(*a*)	(*b*)	(*c*)	(*d*)	(*e*)	(*f*)	(*g*)	(*h*)
A 285	−920	−950	−950	−915	−920	−950	−975	−975
A 516	−925	−950	−950	−920	−920	−950	−975	−1000

* (a) 2.25 M NaOH; (b) Bayer solution; (c) 3.6 M NaOH + 0.67 M Al_2O_3; (d) 2.25 M NaOH + 0.46 M Na_2CO_3; (e) 2.25 M NaOH + 0.02 M Na_2SO_4; (f) 3.6 M NaOH + 0.67 M Al_2O_3 + 0.46 M Na_2CO_3 + 0.02 M Na_2SO_4; (g) 7.73 M NaOH; and (h) 7.73 M NaOH saturated by Al_2O_3.

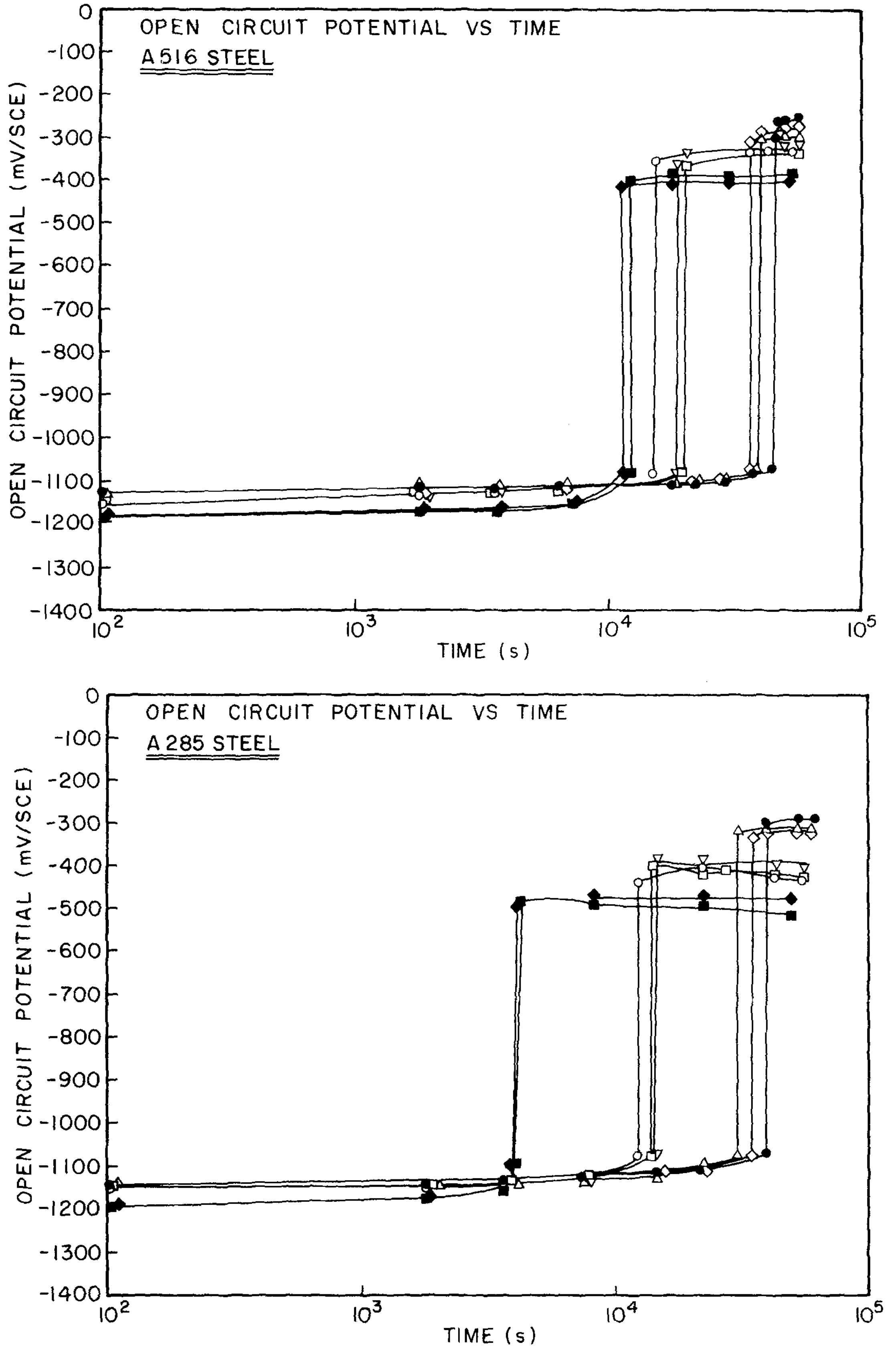

Fig. 1. Open circuit potential against time curves in different electrolytes (a) A 516 steel. Key: (●) 2.25 M NaOH; (○) Bayer; (□) 3.6 M NaOH + 0.67 M Al_2O_3; (△) 2.25 M NaOH + 0.46 M Na_2CO_3; (◇) 2.25 M NaOH + 0.02 M Na_2SO_4; (▽) 3.6 M NaOH + 0.67 M Al_2O_3 + 0.46 M Na_2CO_3 + 0.02 M Na_2SO_4; (■) 7.74 M NaOH; (◆) 7.74 M NaOH saturated by Al_2O_3. (b) A 285 steel. Key: (●) 2.25 M NaOH; (○) Bayer; (□) 3.6 M NaOH + 0.67 M Al_2O_3; (△) 2.25 M NaOH + 0.46 M Na_2CO_3; (◇) 2.25 M NaOH + 0.02 M Na_2SO_4; (▽) 3.6 M NaOH + 0.67 M Al_2O_3 + 0.46 M Na_2CO_3 + 0.02 M Na_2SO_4; (■) 7.74 M NaOH; (◆) 7.74 M NaOH saturated by Al_2O_3.

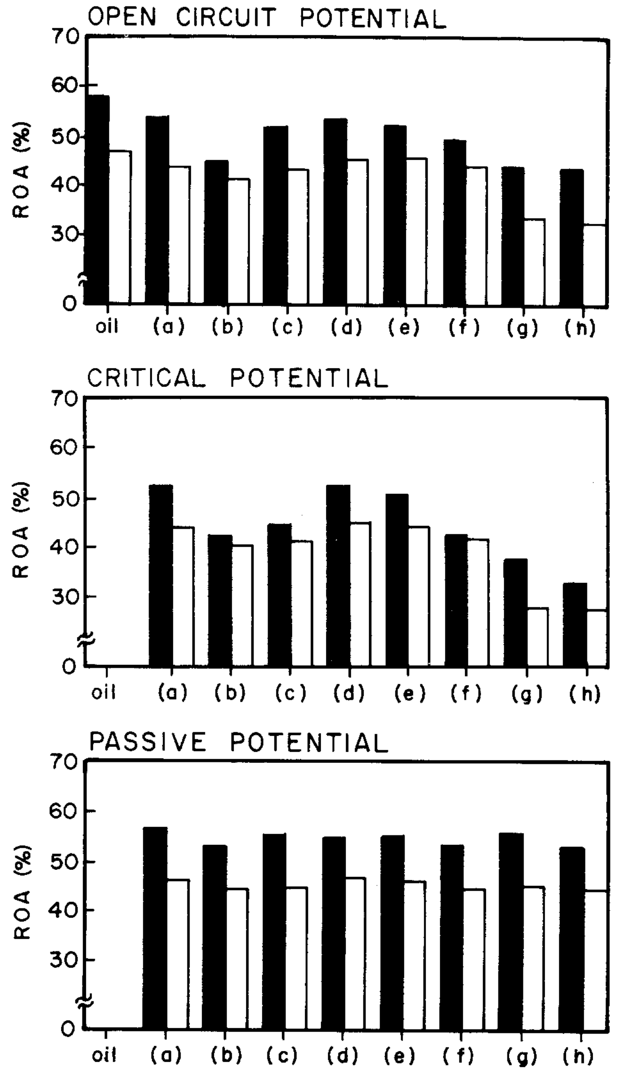

Fig. 2. Slow strain rate results (%ROA) of A 285 and A 516 steels (a) at the open circuit potential, (b) at the critical potential, (c) at the passive potential. The electrolytes are described in Table 2.

than AlO_2^- ions. With an increase in NaOH concentration, the influence of the dissolved AlO_2^- ions was not as significant as in dilute NaOH solution (curves f and g). Figure 1a and b indicated also that the increase of NaOH concentration had a more significant effect on the passivation of A 285 steel than that of A 516 steel.

The results obtained by slow strain rate experiments (ROA, ε and t_f) are in accordance with the above observations on the influence of each impurity on caustic embrittlement. Some susceptibility to SCC was noted particularly in concentrated caustic solutions or when AlO_2^- ions were present in the electrolytes. The susceptibility did not increase with the addition of CO_3^{2-} or SO_4^{2-} ions in the solutions. Results of the ROA are reported in Fig. 2a. The percentage of elongation and the time to failure did not vary greatly with the presence of the different studied impurities. The average values were about 14.2% and 15.8 h, respectively.

3.1.2. Critical potential. The critical potentials were situated for both steels between -920 and -1000 mV/SCE in the active-passive zone. An increase of NaOH concentration or the presence of AlO_2^- species shifted the critical potential to more active values, about 75 mV and 25 mV, respectively (Table 2). Compared to the results obtained at the o.c.p., the suceptibility to SCC at critical potentials increased noticeably (Fig. 2b): the ROA increased more than 3%, while both elongation and time to failure decreased about 2% in the solutions containing 2.25 M free sodium hydroxide with or without dissolved impurities. The average values of ε% and t_f were 14.1% and 15.2 h, respectively. In the concentrated solution 7.73 M NaOH, the %ROA was increased more than 10%. The effect of AlO_2^- ions was more evident at this level of potential than at the o.c.p., while the other Bayer solution impurities had very little influence on the susceptibility to SCC of both steels.

It was also noted during the experiments, that the recorded current was initially anodic corresponding to the imposed anodic potential. The current decayed with time and after less than 1 hour, the current became cathodic. A very weak current density less than $0.1\,\mu A\,cm^2$ was recorded. This change of the current from anodic to cathodic coincided with the formation of a passive film at the surface of the specimen and a shift to the passive potential. The o.c.p. of the passive surface became more noble than the imposed potential.

3.1.3. Passive potential. At -250 mV/SCE, in the electrolytes which did not contain AlO_2^- ions, only slight differences were observed in the results (ROA, ε and t_f) compared to those obtained in oil. Therefore the steels seem to be protected at this anodic potential which lies in the region of anodic protection used in practice (Fig. 2c). The average values of elongation and time to failure were 15.2% and 16 h, respectively.

Particularly, when AlO_2^- ions were dissolved in the electrolytes, some susceptibility to embrittlement was observed. In these experiments, when the anodic potential was applied, the anodic current decayed rapidly coinciding with the formation of the passive layer. But the current remained anodic till the end of the test and did not become cathodic as in the cases with the critical applied potential.

3.2. *Constant load experiments*

All the specimens were deformed initially about 25% before being mounted in the cell. A load corresponding to 95 or 100% of the yield strength was then applied. Some tests were also conducted with notched specimens in order to shorten the time to cracking. The results are given in Fig. 3 and Table 3.

3.2.1. In the active-passive region. The experiments showed that the period of crack initiation was very long and it could take more than 95% of the cracking processes. During this time, the relative deformation was insignificant ($< 0.10\%$). In Table 3, we designated the time of initiation of the crack the period where a linear portion of the elongation versus time curve was

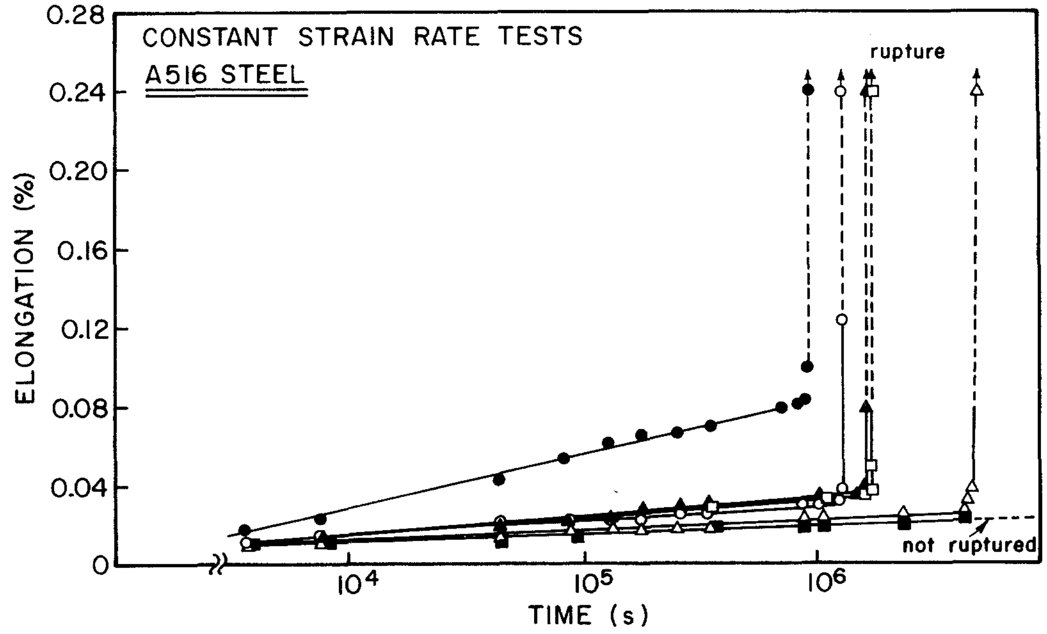

Fig. 3. Constant load tests carried out on A516 steel. Key: (Y. S. electrolyte, E_{imp}/mV(SCE)): (○) 95% (g), −975 mV; (● 100% (g), −975 mV; (△) 100% (a), −925 mV; (▲) 100% (b), −950 mV; (□) 100% (c), −950 mV; (■) 100% (b), −250 mV.

recorded, just before the curve became broken up (Fig. 3). The last portion of the curve corresponds to the propagation of the crack to rupture and was very fast (about 7200 s or 2 h).

An increase of the applied load from 95% to 100% of the yield strength reduced the duration of the experiment by over 20% in the same experimental conditions. If the specimens were notched, the tests with an applied load of 100% Y.S. were then accelerated by about 17 to 27% (Table 3). The results obtained in the tests carried out in Bayer and caustic aluminate solutions show that the caustic embrittlement was accelerated in these environments. Particularly, AlO_2^- ions were responsible for over 85% of the cracking effects produced by all the impurities of Bayer solution. These results were verified for both steels.

Similarly to the slow strain rate experiments, the change of the current density from anodic to cathodic was also recorded in the constant load tests but the time where the steel remained anodic was about 10% longer than that observed in the slow strain rate experiments.

3.2.2. *In the passive region.*

All the experiments carried out at −250 mV/SCE with an applied load corresponding to 100% of the yield strength did not rupture over five weeks. In the plain NaOH solutions (2.25 and

Table 3. Constant load test results

Steel	*Load*	*Electrolytes*	$E_{imposed}$ / mV(SCE)	*Initiation time* /h	*Time to failure* /h
A516	95% Y.S.	(g)	−975	345.5	348.2
A516	100% Y.S.	(g)	−975	248.2	250.8
A516	100% Y.S.	(g)	−250	n/a	not ruptured
A516	95% Y.S.	(a)	−925	over 5 weeks	not ruptured
A516	100% Y.S.	(a)	−925	5 weeks	840 (≈ 5 weeks)
A516 notched	100% Y.S.	(a)	−925	485.0	486.5
A516	100% Y.S.	(a)	−250	n/a	not ruptured
A516	95% Y.S.	(b)	−950	512.0	515.6
A516	100% Y.S.	(b)	−950	440.0	142.5
A516	100% Y.S.	(c)	−950	365.0	367.3
A516	100% Y.S.	(b)	−250	n/a	not ruptured
A516	95% Y.S.	(c)	−950	5 weeks	840 (≈ 5 weeks)
A516	100% Y.S.	(c)	−950	410.0	412.4
A516	100% Y.S.	(c)	−950	325.0	327.3
A516	100% Y.S.	(c)	−250	n/a	not ruptured
A285	100% Y.S.	(a)	−950	375.5	377.5
A285	100% Y.S.	(b)	−950	350.0	352.6

Table 4. Crack density and crack length of A 516 steel in different electrolytes at 100° C

Solution	*Potential* / mV (SCE)	*Number of cracks*	*Crack length* /μm
(a)	−925	18	50
(b)	−950	40	80
(c)	−950	36	80
(d)	−920	32	75
(e)	−920	30	70
(f)	−950	37	80
(g)	−975	44	250
(h)	−975	46	400

7.74 M), the surface of the gauge length was recovered by a thick black oxide layer and no cracks were detected by the scanning electron microscope. In Bayer and caustic aluminate solutions, the oxide layer became more adherent than the previous cases and was reddish. The examination by scanning electron microscopy of the gauge length allowed obervation of some rare shallow cracks. This verifies the results obtained using slow strain rate.

3.3. *Scanning electron microscopy*

For the slow strain rate experiments, the secondary intergranular cracks increased in depth and number from the open circuit potential to the active-passive transition zone, or when impurities, particularly AlO_2^- ions, were present in the solutions. The cracks observed on the A 285 steel seemed larger but less numerous than those observed on the A 516 steel. In the passive region (−250 mV/SCE), no susceptibility to SCC was noted in the plain NaOH electrolyte. However, with the addition of species contained in Bayer solution in the electrolyte, some shallow cracks were observed with a blunt crack tip and appeared like pits. Table 4 indicates the crack density and the longest secondary crack noted for A 516 steel in different studied electrolytes.

In the 7.74 M NaOH solution, the fracture surface of A 516 steel showed an intergranular region of about 250 μm depth that formed an outer annulus of SCC and the inner zone was ductile due to the overload fracture. When aluminate ions were present in the solution, the intergranular attack of SCC was concentrated at numerous points of the surface and did not create an annulus of regular depth. The different intergranular regions could have reached up to 400 μm depth (fig. 4a and 4b) in some cases. The secondary cracks were also deeper and less numerous than in 7.74 M NaOH solution.

The constant load tests revealed practically the same observations as those above, but some differences should be noted: the cracks are generally less numerous and more shallow than those obtained by the slow strain rate tests. For example, in 7.74 M NaOH the longest intergranular secondary cracks was about 180 μm deep as compared to the 250 μm intergranular region obtained in slow strain rate experiments.

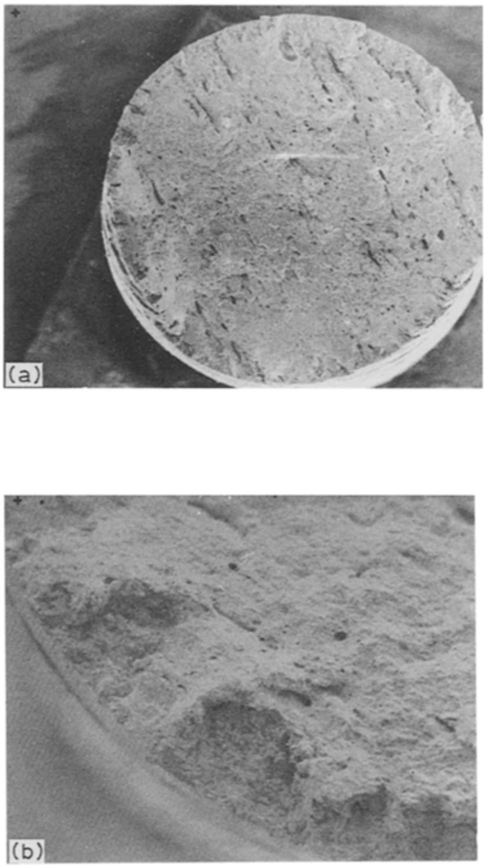

Fig. 4. Faciès of rupture of A 516 steel in 7.74 M NaOH solution saturated by Al_2O_3 obtained by the slow strain rate technique, showing different spots of intergranular corrosion attack. (a) magnification: 25 × (b) magnification: 70 ×

4. Discussion

4.1. *Effect of Bayer solution impurities*

Among these impurities, aluminate ions (AlO_2^-) have an important influence on the passivation and the susceptibility of the carbon steels to SCC, while carbonate and sulphate ions have little effect on the embrittlement. The passive layer containing aluminate species is porous and may have many defects. Some electrochemical studies made previously [7, 10] have assumed the formation of a $Fe_{3-x}Al_xO_4$ layer ($x \leqslant 2$). Reinoehl [4] assumed that aluminate falls into the class of anodic inhibitors which promote formation and repair of a protective film. Incomplete inhibition results in an unfavorable cathode to anode area ratio which promotes rapid selective attack at the local anodes. The large cathode represents the protective but not quite continuous film, and the small anodes constitute film discontinuities. The embrittlement thus appears worse than in the absence of amounts of "inhibitor". The micrographs of the rupture surface from the concentrated NaOH solution saturated with Al_2O_3 tend to confirm this (Fig. 5a and b).

On the other hand, carbonates and sulphates fall into the class of cathodic inhibitor which can promote stress corrosion cracking depending on the amount

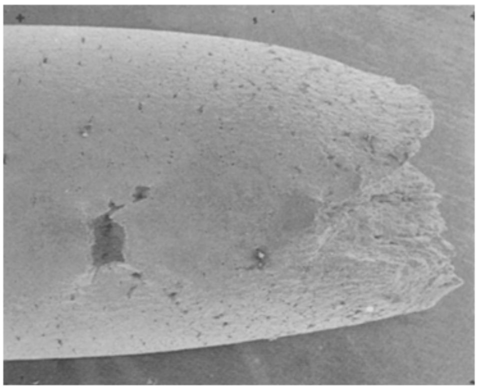

Fig. 5. View of the ruptured gage length of A 516 steel in Bayer solution obtained by the constant load experiments — (magnification 25 ×).

which dissolves in the solution. The inhibition of SCC occurs only when these species hinder localized attack by favouring a more generalized form of corrosion, thereby rendering the cathode to anode area ratio less unfavourable. These inhibitors generally induce the precipitation of a compound which is relatively insoluble in caustic solutions.

4.2. *Anodic protection*

Among the protective methods such as stress relieving, nickel lining, and cathodic protection employed in the alumina industry, anodic protecton has not yet been applied widely in North American alumina plants. Anodic protection has been applied for preventing SCC of equipment in the alumina industry in Europe and it is a reliable method at moderate temperature and NaOH concentration [12]. However, this method has been recently installed on some pressure vessels in Canada where operating conditions such as temperature, pressure, and NaOH concentration are generally higher. The present investigation on the passive region indicates that the Bayer solution caused some initiation of SCC while in plain NaOH solutions, protection can be achieved. However, anodic protection is still beneficial in lowering the crack propagation rates.

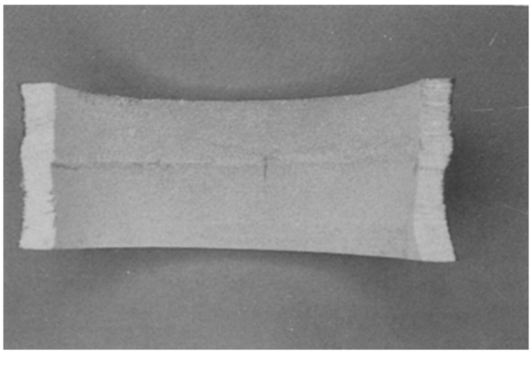

Fig. 6. Piece of reducer embrittled by SCC (cracks perpendicular to the weld are indicated by the arrows).

4.3. *Comparison between slow strain rate technique, constant load technique and practical caustic embrittlement*

Slow strain rate and constant load techniques are used frequently for investigating stress-corrosion cracking. The influence of Bayer solution impurities dissolved in the electrolyte, the effect of the applied potential and the strain are studied by both methods and the results are almost the same. Both techniques show a higher susceptibility of A 285 steel than A 516 steel, probably due to a higher phosphor and sulphur content in the A 285 steel. The segregation of such elements at grain boundaries, which accelerates SCC, is demonstrated extensively by other workers [13].

The stress corrosion susceptibility is noticeably lower in experiments carried out with a constant load than in experiments with a slow strain rate. This is in accordance with other works [14, 15] which also demonstrated that both methods lead to a similar classification of the susceptibility to SCC of stainless steels, without modifying the cracking mechanisms. However, some authors [16, 17] consider that the slow strain rate technique is unsuitable for the study of caustic embrittlement because the results are not reproducible in practice.

The present results confirm that constant load and slow strain rate tests are both reliable methods for investigating stress corrosion cracking of carbon steels. The increase of the susceptibility to embrittlement by the slow strain rate technique is only a supplementary indication of the possiblity of the steel cracking in more severe conditions. In addition, the primary advantages of the slow strain rate technique are to rapidly provide an evaluation of SCC susceptibility and the results are positive in that specimen failure occurs either in a ductile manner (no environmental effects) or in a brittle mode (environmental effect) when SCC occurs. In the constant load experiments, the initiation of the cracks can take a very long time and there is high scatter of the results.

The intergranular secondary cracks observed on the gauge length of the specimen are the main features of SCC obtained by both methods. These cracks are always perpendicular to the tensile axis and, in practice, the same situation is observed. For example, in a welded part (piece of reducer) as shown in Fig. 6, cracks are observed perpendicular to the weld, i.e., perpendicular to the hoop stresses. Hoop stresses play a major role in the SCC of pipes and tanks. They are particularly active at the weld-parent metal junction because the weld acts as a circumferential restriction. The level of hoop stress is normally well below the stress level applied in the laboratory, but it can increase locally due to stress risers on the surface, such as weld defects, metallurgical structures in the heat affected zone or impurities (slag, mill scale, oxides . . .). The cracks are initiated perpendicular to the hoop stress, i.e. perpendicular to the weld. They propagate and can form a continuous crack parallel to the weld, mainly due to mechanical forces and this signals imminent, complete failure.

5. Conclusions

1. Caustic cracking occurs in the active-passive transition zone of potential. The embrittlement increases when impurities contained in Bayer solution are present in the electrolyte and among these impurities, aluminate ions are the main species which promote significant SCC susceptibility.
2. Anodic protection is a reliable method for preventing SCC in plain NaOH solution. However, in the presence of dissolved AlO_2^- ions, the danger of embrittlement still exists, although the initiation and the propagation of the cracks are lowered by an imposed potential in the passive range.
3. Slow strain rate and constant load tests are two reliable methods for investigating SCC. The slow strain rate method provides a quick laboratory technique for assessing caustic embrittlement but the susceptibility of the steel is intensified by this method compared to the constant load technique. Some similarity of crack initiation and propagation between laboratory experiments and practice was recorded.

Acknowledgments

The authors are grateful to the National Science and Engineering Research Council of Canada (NSERC) for providing financial support.

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