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*EXPLORATORY STRESS-CORROSION
CRACKING TESTS ON SOME LOW-ALLOY
HIGH-STRENGTH STEELS*

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G. J. BIEFER

PHYSICAL METALLURGY DIVISION

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EXPLORATORY STRESS-CORROSION CRACKING TESTS ON SOME
LOW-ALLOY HIGH-STRENGTH STEELS

by

G. J. Biefer*

ABSTRACT

Using a test rig in which the test specimens are small bent strips, held in this configuration under spring loading, stress-corrosion cracking (SCC) tests were performed in several media upon four alloys resembling AISI Type 4320 but with different Si and Mn contents. One of the steels had the composition of "HY-Tuf"**, a proprietary steel said to have high resistance to hydrogen-embrittlement cracking.

A steel containing 1.48% Si and 0.68% Mn showed the best all-round resistance to SCC in tests in normal sulphuric acid (with and without a cathodic "poison"), boiling calcium-ammonium nitrate, and 3.5% NaCl solution, outperforming the steel having the "HY-Tuf" composition.

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Direction des mines

Bulletin technique TB 111

ESSAIS DE FISSURATION PAR CORROSION SOUS
TENSION PRATIQUÉS SUR CERTAINS ACIERS
FAIBLEMENT ALLIÉS À HAUTE RÉSISTANCE

par

G. J. Biefer*

RÉSUMÉ

Au moyen d'un appareil d'essai utilisant des éprouvettes sous forme de petites bandes recourbées, retenues en place par un ressort, des essais de fissuration par corrosion sous tension ont été pratiqués dans diverses solutions sur quatre alliages ressemblant à l'acier AISI de type 4320 mais ayant une teneur différente en Si et en Mn. L'un des aciers avait la composition du "Hy-Tuf"**, un acier de spécialité censé posséder une haute résistance à la fissuration due à la fragilisation par l'hydrogène.

Un acier ayant une teneur de 1.48 p. 100 en Si et de 0.68 p. 100 en Mn a manifesté la meilleure résistance à la fissuration par corrosion sous tension à tous points de vue lors des essais dans l'acide sulfurique (avec et sans "empoisonnement" cathodique), dans le nitrate de calcium-ammonium en ébullition et dans une solution de NaCl à 3.5 p. 100. L'acier en question s'est avéré plus résistant que l'acier de composition "Hy-Tuf".

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INTRODUCTION

One of the factors preventing the wider application of high-strength steels is their tendency to fail, under high operating stresses, by stress-corrosion cracking (SCC) or hydrogen-embrittlement cracking (HEC) (1). It was decided, therefore, to undertake long-term studies of these phenomena at the Physical Metallurgical Division (PMD). The primary object of this work was to evaluate the SCC and HEC behaviour of high-strength steels of current technological importance. A secondary object was to gain an increased understanding of cracking mechanisms, with the ultimate goal of producing high-strength steels having increased resistance to cracking.

It had been reported that "HY-Tuf"* high-strength steel is unusually resistant to HEC(2). It was decided, therefore, as a first step in the development of satisfactory laboratory techniques, to carry out measurements of SCC** on a steel of this composition.

For purposes of comparison, it was decided to investigate, in addition, the behaviour of three other high-strength steels with compositions and tensile strengths resembling those of "HY-Tuf" steel, but differing from it in their silicon and manganese contents.

Concurrently with the work described here, R.D. McDonald, of the Ferrous Metals Section, PMD, was carrying out HEC tests on the same steels(3).

*Crucible Steel Co. of America, Pittsburgh, Pa. - Chemical analysis: C, 0.25%; Ni, 1.80%; Mn, 1.30%; Mo, 0.40%; Si, 1.50%; Cr, 0.35%

**For simplicity, SCC will be used as a general term (including HEC) for the remainder of this report, unless specified otherwise.

EXPERIMENTAL

Materials

Four steels were prepared as 50-lb aluminum-killed heats in an induction furnace. Their chemical analyses appear in Table 1. After forging to 1-in.-thick plate, the steels were given a normalizing treatment at 900°C (1650°F) for a half-hour, followed by air cooling (3).

Subsequently, portions of the plate were rolled to a thickness of 0.060 in., then given a stress-relieving treatment of 1 hour at 590°C (1094°F). Rectangular SCC standard specimens in the form of 1.75 x 0.25 x 0.04-inch strip were then prepared at the machine shop so that the major axes of the specimens lay in the rolling direction.

The as-machined specimens were given an austenitizing heat-treatment for 15 min at 927°C (1700°F), followed by an oil quench. This temperature was that recommended for AISI Type 4320-H steel in reference 4.

Some of the quenched specimens were then tempered for 1 hour at each of three selected temperatures, giving a range of tensile strengths; water quenching followed the tempering.

Hardness determinations, carried out upon heat-treated metal specimens of the four steels, yielded the average hardnesses listed in Table 2. This table also contains approximate tensile strengths obtained from the hardnesses, using the data of reference 5.

Prior to tests, the surfaces of the heat-treated specimens were lightly abraded on 120-grit silicon carbide, to remove surface oxide and provide a standard surface condition. Thorough washing, degreasing, and quick drying in a jet of hot air followed this.

Apparatus

The apparatus used in the experiments was patterned after that employed by Uhlig and Sava (6), and was designed to hold the strip specimens in a bent configuration under spring loading. Because of the spring loading, the initiation of cracking led to stress intensification, and a fairly rapid propagation to complete failure, in contrast to the stress-relieving

effects sometimes resulting from crack initiation with "constant strain" bent-beam tests in which spring loading is not employed.

One of the stress-corrosion rigs is reproduced in Figure 1. A bent specimen is shown in position, insulated from the Inconel frame and the moveable upper jaw by means of ceramic inserts. The times-to-break were recorded accurately by a timing device which was deactivated when the bent specimen had cracked sufficiently to allow the upper jaw to move, to open a microswitch. The time to this first definite movement of the upper jaw of the rig was taken as equal to the breaking time, provided that the specimen then proceeded to a more visible failure.

In all cases, prior to insertion in the rig the specimens were bent under 4-point loading and placed between the jaws of the rig without allowing spring-back. Bending was then continued between the jaws of the rig until the ends of the specimen were separated by a standard distance of 1.4375 in. Stresses were not known accurately, but were apparently beyond the elastic limit, as specimens bent in this way, then removed from the rig, showed a permanent set.

Test Solutions

The following test solutions were employed:

- A. Normal sulphuric acid at room temperature.
- B. As above, but containing 5 drops per litre of cathodic poison composed of 2 g phosphorus dissolved in 40 ml CS₂(7).
- C. Boiling calcium-ammonium nitrate solution. The solution had the composition

860 g Ca(NO₃)₂.4H₂O

30 g NH₄NO₃

110 ml H₂O

and was similar to that used by Uhlig and Sava(6).

- D. 3.5% NaCl solution at room temperature.

Solutions were always discarded after testing, and each specimen was tested individually in a freshly made solution. In the tests in sulphuric acid, 1 to 3.5 litres of acid were used for each specimen, whereas 500 ml of solution were used for each test in the standard nitrate solution. In the tests in sodium chloride solution, 1 to 4 specimens were immersed in a jar containing 15 litres. In the long-term tests in sodium chloride solution, distilled water was added periodically to make up for losses due to evaporation, and the entire solution was changed every 1000 hours.

RESULTS

A. Normal Sulphuric Acid at Room Temperature

The results obtained in SCC tests in this solution appear in Table 3. It can be seen that failures were extremely rapid, except for the steels given the 538°C (1000°F) tempering. These last-mentioned steels corroded at rather high rates, in the neighbourhood of 200 mg/cm²/day. They were observed to yield under the spring loading, simply due to loss of metal; subsequent to testing, an examination of specimen surfaces failed to disclose any cracks.

B. Normal Sulphuric Acid plus Cathodic "Poison"

The results obtained in tests in this solution appear in Table 4. It was found that the cracking mode differed according to the alloy and heat treatment, as follows:

1. As-quenched specimens. In the majority of the cases these specimens showed their failure cracks at locations removed from the centre of the specimen. In addition to the failure crack, alloys 6010, 6012 and 6027 showed many other parallel cracks. Alloy 6028, in contrast, showed only a few or no cracks in addition to the main failure crack.
2. Specimens tempered at 218°C (425°F). The appearance of specimens of the different alloys was generally the same as that of the corresponding as-quenched specimens.

3. Specimens tempered at 371°C (700°F). In the majority of cases the specimens showed their failure cracks at locations other than the centre of the specimens. Alloys 6010 and 6028 showed only the failure cracks, whereas alloys 6012 and 6027 showed additional cracks parallel to the failure cracks, 6027 to a greater extent than 6012.
4. Specimens tempered at 538°C (1000°F). The specimens, when they failed, usually cracked at or near the centre of the specimen, and this was the only crack they showed.

C. Boiling Calcium-Ammonium Nitrate.

The results obtained in SCC tests in this solution appear in Table 5.

D. 3.5% NaCl at Room Temperature.

The results obtained in SCC tests in this solution appear in Table 6. Only the as-quenched specimens were observed to crack; the specimens in other heat-treatment conditions endured about 2000 hours of exposure without showing any signs of cracking.

It was observed that the specimens had not corroded evenly, but showed shiny (cathodic) and rusty (anodic) areas. In two of the as-quenched specimens, it was noticed that the convex central area, where tensile stresses are maximum, had been cathodic; i.e., it had remained shiny and uncorroded during the immersion. This had shifted the failure crack to an anodic (rusty) location removed from the centre, i.e. to a point of lower stress. This appeared to give longer lives to these two specimens as compared with similar specimens which had failed near their centres; this is shown in Table 6.

Aside from the two specimens referred to above, failures were generally near the specimen centres. Generally the failure crack was the only one to be seen, though, in three of the failed specimens, there was some hint of shallow subsidiary cracking parallel to the failure crack.

DISCUSSION OF RESULTS

In both sulphuric acid solutions, the failure mechanism is probably HEC(7). In the normal sulphuric acid solution which did not contain the cathodic "poison" (Table 3), specimens either failed rapidly or did not fail at all but, instead, corroded rapidly. In this test medium there was a clear discrimination between the 538° C (1000°F) temper, in which specimens were not susceptible, and the other tempers, in which specimens

were susceptible to cracking. Perhaps in the case of this "resistant" temper, metal was simply being removed by corrosion more rapidly than cracks could initiate and proceed. Though differences between the alloys were slight, it appeared that they could be ranked, in order of resistance to SCC, as follows: 6028, 6027>6012, 6010.

In the "poisoned" sulphuric acid solution (Table 4), corrosion rates were lower and the alloys showed marked differences in behaviour. Steel 6028 was consistently the most resistant alloy at all heat-treatment conditions, though it was equalled by alloy 6010 in the 538°C (1000°F) temper. Increased resistance to SCC was observed to be correlated with a decrease in the number of visible cracks; in order of general resistance to SCC, the alloys could be ranked 6028>6010>6012>6027.

Steel 6028 also showed the best resistance to SCC, at all tempering temperatures, in the boiling calcium-ammonium nitrate solution. However, in this solution, the order of resistance of the other alloys had changed so that the ranking, in terms of resistance to SCC, was 6028>6027>6010>6012.

In the 3.5% NaCl solution, SCC was confined to the as-quenched condition, and there were, therefore, much fewer data available to compare the alloys. In addition, in this medium there appeared to have been an unpredictable interference because of uneven corrosion, as described previously. Tentatively, it appears that Steel 6028 probably was the most resistant, and that the ranking, in order of SCC resistance, was probably 6028>6012>6010>6027.

SUMMARY AND CONCLUSIONS

1. A stress-corrosion cracking test rig, constructed of Inconel, in which small bent strips of metal are held in this configuration under spring loading, performed satisfactorily in exploratory SCC tests on low-alloy high-strength steels. This fulfilled the primary objective of the work.

2. SCC susceptibility of the four steels tested was found to be related to their differences in chemical composition, and also to the test media. Performance was generally inconsistent from one medium to the next, e.g., alloy 6010 (at the 538°C (1000°F) temper) was highly susceptible in calcium-ammonium nitrate but resistant in "poisoned" sulphuric acid.

Additional research, not contemplated at present, would be needed to explain the differences in SCC behaviour noted.

3. It is noteworthy that steel 6028, containing 1.48% Si and 0.68% Mn, was found to show the best all-round resistance to SCC, outperforming an alloy with the composition of "HY-Tuf" steel (Crucible Steel Co. of America). It is of interest, however, that in parallel tests of HEC carried out by R.D. McDonald(3), these two high-silicon steels were both equally resistant to HEC and superior to the two lower-silicon alloys.

ACKNOWLEDGEMENTS

Mr. B.G. Olivier of the Corrosion Section, PMD, carried out most of the SCC tests, with some assistance from Mr. H.M. Weld of the same Section in the early stages of the work. The chemical analyses were supplied by the Mineral Sciences Division, Mines Branch, Department of Energy, Mines and Resources.

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TABLE 1
Chemical Analyses of Steels
 (Per Cent)

Steel No.	C	Mn	Si	S	P	Ni	Cr	Mo
6010*	0.26	0.61	0.26	0.030	Tr.	1.82	0.50	0.32
6012	0.235	1.32	0.30	0.030	0.007	1.86	0.48	0.41
6027**	0.21	1.40	1.48	0.026	0.001	1.82	0.44	0.42
6028	0.22	0.68	1.48	0.027	0.001	1.92	0.45	0.43

* The composition of this steel resembles that of AISI Type 4320.

** The composition of this steel resembles that of "HY-Tuf"
 low-alloy, high-strength steel (Crucible Steel Co. of America).

TABLE 2

Hardness and Approximate Tensile Strengths* of the Steels after
Various Heat Treatments

Steel No.	Mn, %	Si, %	Oil-quenched from 927°C (1700°F)		Oil-quenched from 927°C (1700°F), then tempered 1 hour at					
			Hardness, R _c	UTS, kpsi	218°C (425°F)		371°C (700°F)		538°C (1000°F)	
					Hardness, R _c	UTS, kpsi	Hardness, R _c	UTS, kpsi	Hardness, R _c	UTS, kpsi
6010	0.61	0.26	51	250	45.5	220	42	200	33.5	150
6012	1.32	0.30	49	240	45	210	41.5	200	33.5	150
6027	1.40	1.48	48	230	45.5	220	45	210	37	170
6028	0.68	1.48	46	220	44	210	44	210	37	170

*According to Reference 5.

TABLE 3

Stress-Corrosion Cracking Behaviour in Normal Sulphuric AcidSolution at Room Temperature

(Time to failure, hr)

Steel No.	Oil-quenched from 927°C (1700°F)		Oil-quenched from 927°C (1700°F), then tempered 1 hour at					
			218°C (425°F)		371°C (700°F)		538°C (1000°F)	
6010	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	>20*	>5*
6012	0.1	<0.1	<0.1	<0.1	<0.1	<0.1	>20*	>5*
6027	<0.1	0.1	<0.1	0.3	0.1	0.1	>20*	>5*
6028	<0.1	0.1	0.1	0.1	0.1	0.1	>20*	>5*

* Had not cracked after indicated immersion.

TABLE 4

Stress-Corrosion Cracking Behaviour in "Poisoned" Normal
Sulphuric Acid Solution at Room Temperature

(Time to failure, hr)

Steel No.	Oil-quenched from 927°C (1700°F)			Oil-quenched from 927°C (1700°F), then tempered 1 hour at								
				218°C (425°F)			371°C (700°F)			538°C (1000°F)		
6010	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.2	0.7	>22.3*	18.5	>22.0*
6012	<0.1	<0.1	0.1	0.1	0.1	<0.1	0.1	0.1	<0.1	1.5	1.2	0.8
6027	<0.1	0.1	0.1	0.1	0.1	0.2	0.1	0.1	0.1	0.3	0.2	0.4
6028	0.6	1.4	0.4	8.0	1.9	4.5	>18.3*	11.2	2.5	17.6	>23.3*	14.9

* Had not cracked after indicated immersion.

TABLE 5

Stress-Corrosion Cracking Behaviour in Boiling Calcium-
Ammonium Nitrate Solution

(Time to failure, hr)

Steel No.	Oil-quenched from 927°C (1700°F)	Oil-quenched from 927°C (1700°F), then tempered 1 hour at								
		218°C (425°F)			371°C (700°F)			538°C (1000°F)		
6010	<0.1 <0.1 <0.1	<0.1	<0.1	<0.1	0.2	0.2	0.2	0.7	1.1	0.8
6012	<0.1 <0.1 <0.1	<0.1	<0.1	<0.1	0.2	0.1	0.1	0.3	0.3	0.1
6027	0.1 0.1 0.3	0.2	0.2	0.1	0.2	0.3	2.1	15.1	36.9	19.9
6028	0.1 0.1 0.1	0.2	0.2	0.2	2.9	2.1	5.1	65.8	>150*	>150*

* Had not cracked after the indicated immersion.

TABLE 6

Stress-Corrosion Cracking Behaviour of Steels in 3.5% Sodium Chloride Solution at Room Temperature

(Time to failure, hr)

Steel No.	Oil-quenched from 927°C (1700°F)	Oil-quenched from 927°C (1700°F), then tempered 1 hour at					
		218°C (425°F)		371°C (700°F)		538°C (1000°F)	
6010	753 572	>2010*	>2033*	>2000*	>2014*	>2014*	>2014*
6012	1273** 553	>2033*	>2033*	>2000*	>2012*	>2012*	>1969*
6027	281 775**	>2010*	>2012*	>2010*	>2009*	>2010*	>2013*
6028	>2000* 1269	>2011*	>2011*	>2009*	>2010*	>2009*	>2014*

* Had not cracked after the indicated immersion.

** Off-centre break.

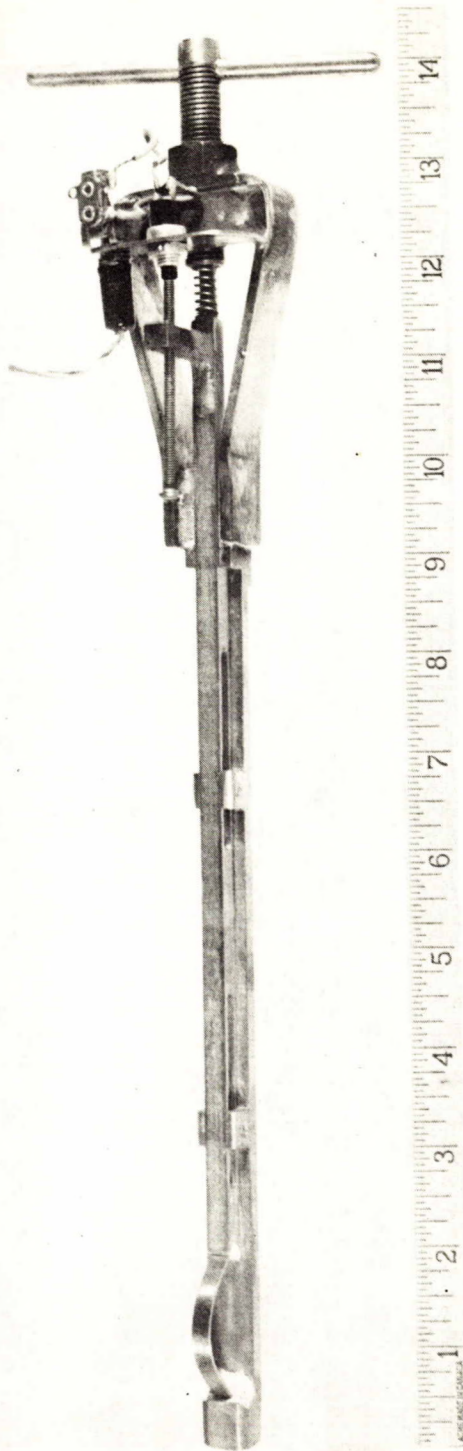


Figure 1. Inconel rig used to carry out stress-corrosion cracking tests on small bent-strip specimens under spring loading.

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