

# The corrosion behaviour of mild steel, 3CR12, and AISI type 316L in synthetic minewaters

by N. MURSALO\*, M. TULLMIN†, and F.P.A. ROBINSON‡

## SYNOPSIS

The corrosion resistance of mild steel, 3CR12, and AISI type 316L stainless steel were assessed by accelerated electrochemical corrosion testing in synthetic minewaters containing different permutation levels of chloride and sulphate ions. The general corrosion resistance of mild steel was adversely affected by the presence of both the chloride and the sulphate ions. In contrast, sulphate ions in the water had a beneficial passivating effect on the stainless alloys and improved their resistance to both general and localized corrosion. Sulphate ions in the water would thus tend to offset the detrimental effects of insidious chloride ions on stainless steels.

## SAMEVATTING

Die korrosiebestandheid van sagte staal, 3CR12 en die AISI-tipe vlekvrystaal 316L is bepaal deur versnelde elektrochemiese korrosietoetse in sintetiese mynwater wat verskillende vlakke van chloried- en sulfaatione bevat. Die algemene korrosiebestandheid van sagte staal is nadelig beïnvloed deur die aanwesigheid van sowel die chloried- as die sulfaatione. Aan die ander kant het sulfaatione in die water 'n voordelige passiverende uitwerking op die vlekvrystaal gehad en hul bestandheid teen sowel algemene as gelokaliseerde korrosie verbeter. Sulfaatione in die water sal dus geneig wees om te kompenseer vir die nadelige uitwerking van verraderlike chloriedione op vlekvrystaal.

## Introduction

Most industrial waters contain both chloride and sulphate ions. In the case of South African minewaters, these two anions, derived from soluble salts, usually make up the bulk of the total solids in solution. Histograms showing the distribution of chloride and sulphate ions in minewaters covering the entire geographical region of the South African goldfields<sup>1</sup> are shown in Figs. 1 and 2.

A certain amount of research has been conducted<sup>1-3</sup> on the variables affecting the corrosivity of South African minewaters, and a data base of the corrosion resistance of mild steel and stainless steels in these waters has been compiled<sup>4</sup>. However, with the exception of Capendale's research<sup>3</sup>, these studies have failed to specifically isolate the chloride and sulphate ion contents as the principal variables affecting the corrosion resistance of these alloys. Although indicating possible trends, these studies included other variables affecting corrosivity, such as pH value and nitrate or ammonia content. Both laboratory studies and field experience have indicated that, while sulphate ions have a passivating influence on stainless steels by improving their resistance to localized corrosion, the corrosivity towards mild steel appears to be increased. These contrasting effects have thus given rise to a certain degree of confusion about the functions of higher sulphate contents in minewater.

The present study was undertaken to determine and clarify the influence of chloride and sulphate ions on the

general corrosion resistance of mild and stainless steels, and also on the resistance of stainless steels to pitting and

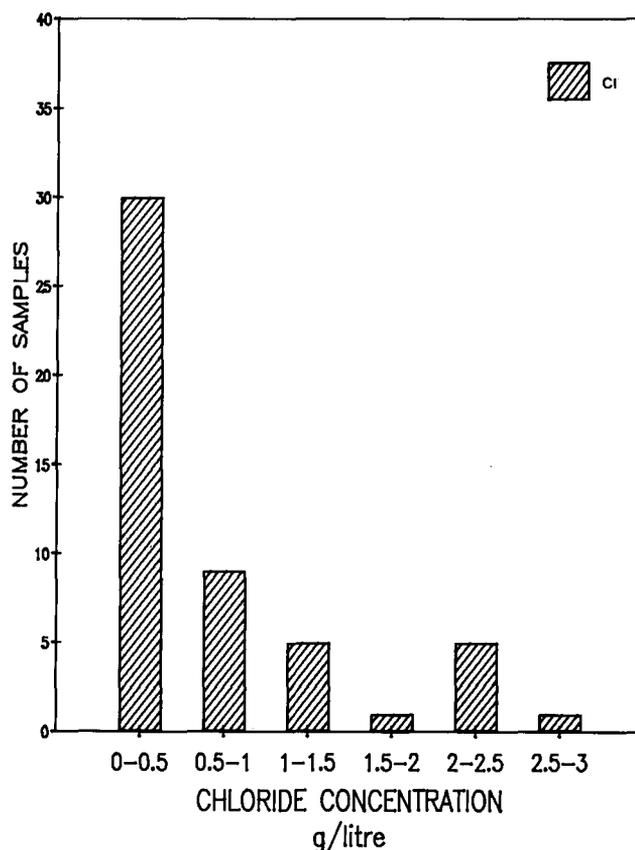


Fig. 1—Distribution of chloride in South African minewaters (after Higginson and White<sup>1</sup>)

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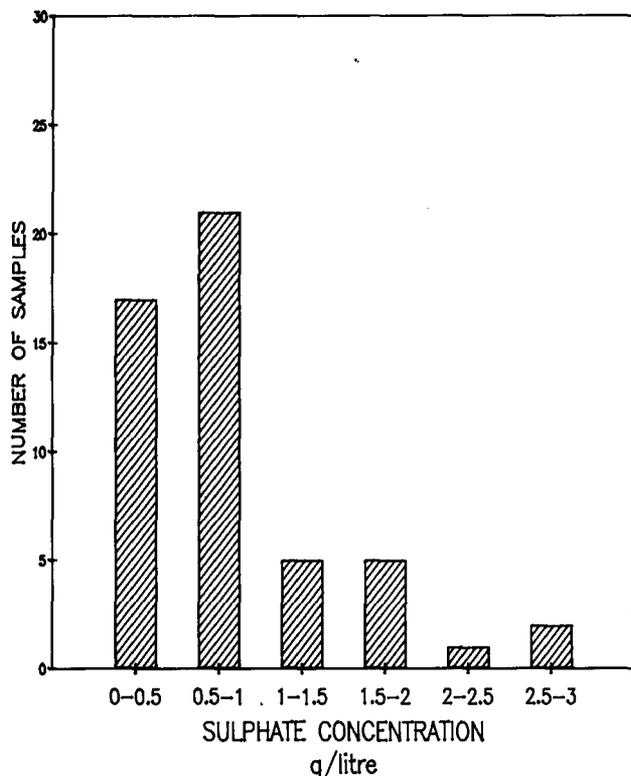


Fig. 2—Distribution of sulphate contents in South African minewaters (after Higginson and White<sup>1</sup>)

crevice corrosion. To investigate these effects unambiguously, synthetic minewaters were used in which only the ratios of chloride to sulphate ions were varied. The corrosion resistance of mild steel, 3CR12 steel, and AISI type 316L stainless steel at various chloride and sulphate levels was assessed by electrochemical polarization, which is a technique that has been widely employed in similar studies.

Mild steel has been used extensively as a material of construction for components in contact with South African minewaters, mostly with suitable coatings for improved corrosion resistance. More recently, however, materials with higher inherent corrosion resistance, such as the corrosion-resisting steel 3CR12 and the more highly-alloyed stainless austenitic alloys, e.g. AISI type 316L, have found increasing application. Mild steel tends to corrode by uniform or general corrosion in South African minewaters. General 'rusting' over the entire surface is characteristic of this form of attack. In corrosive minewaters, which are usually associated with low pH, high contents of dissolved oxygen and total dissolved solids, and high temperatures, the general corrosion rate of mild steel may become excessive in the absence of coatings, such as galvanizing, paint systems, or a 'duplex' combination of the two. However, the suitability of such coatings is substantially limited if synergistic corrosive and abrasive conditions prevail.

In contrast, the general corrosion rates for stainless steels in South African minewaters are usually extremely low and rarely cause for concern, since these alloys are in a passive state, which is afforded by the alloying additions, notably chromium and molybdenum. Ironically though, the very presence of a protective passive surface film on these alloys may render them susceptible to

localized corrosion in the form of pitting or crevice corrosion. It is indeed these two forms of localized corrosion that have led to the vast majority of failures of stainless alloys in service, including those in South African minewaters. It is thus not surprising that the pitting and crevice corrosion resistance of stainless alloys such as 3CR12 and AISI types 304L and 316L are the prime requirements when they are being considered for application in a particular minewater.

The deleterious effect of high levels of chloride ions in minewaters on the resistance of the most common South African stainless alloys such as 3CR12, 304L, and 316L to pitting and crevice corrosion is relatively well-known and accepted. The effect of sulphate ions appears to be less understood and is possibly often neglected in the prediction of corrosion resistance. The importance of sulphate ions on the corrosivity of industrial waters is apparent from the example presented in Fig. 3. The pitting potential of 3CR12, a measure of the resistance to pit initiation, was significantly higher in water of higher sulphate content, even though the level of chloride ions and the temperature were also significantly higher.

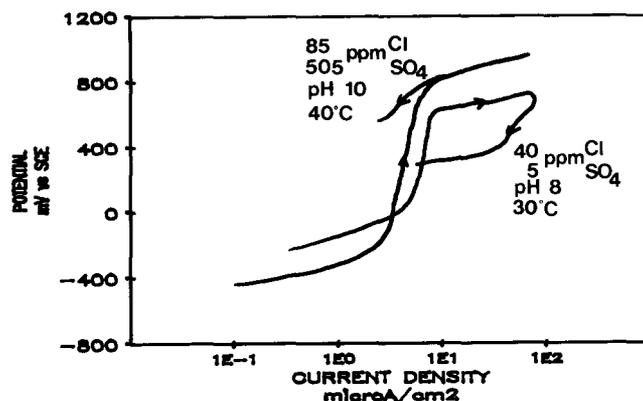


Fig. 3—Pitting curves of 3CR12 in two industrial waters of different sulphate and chloride contents (the unit for current density is  $\mu\text{A}/\text{cm}^2$ )

#### Experimental Procedures

The actual chemical composition of the mild steel and the nominal compositions of the 3CR12 and 316L used in the present corrosion studies in synthetic minewaters containing various amounts of chloride and sulphate ions are listed in Table I. The three alloys selected for testing represent the three main categories of alloys used in the mining industry in increasing order of corrosion resistance, namely low-carbon or mild steels, corrosion-resisting alloys (3CR12), and a true stainless steel (316L) having relatively high pitting resistance.

#### Preparation of the Solutions

Two stock solutions containing 3000 p.p.m. of sodium chloride and 3000 p.p.m. of sodium sulphate were made up with doubly distilled water in order to ensure consistency in the dilution, and mixing of synthetic waters containing various permutations of chloride and sulphate ions. The solutions for corrosion testing were made up so as to approximate the chloride and sulphate contents found in a variety of actual minewaters. These solutions contained from 30 to 3000 p.p.m. of chloride ions and from 0 to 1500 p.p.m. of sulphate ions. The pH values

TABLE I  
CHEMICAL COMPOSITIONS\* OF THE STEELS TESTED

Steel	C	Cr	Ni	Mn	Mo	Si	P	S
Mild steel	0,08	0,16	0,12	0,34	0,02	0,1	0,01	0,016
3CR12	0,03 max	11-12	1,5 max	1,5 max		1,0 max	0,03 max	0,03 max
316L	0,03 max	16-18	10-14	2,0 max	2-3	1,0 max	0,05 max	0,03 max

\*All values in mass percentages, the balance being Fe

of the solutions were adjusted to a constant value of 8 by the addition of appropriate amounts of sodium hydroxide. A pH value of 8 was selected because this value lies close to that observed in the majority of minewaters, as shown by the pH distribution of the samples of South African minewaters given in Fig. 4<sup>1</sup>. The solutions were maintained at  $30 \pm 1^\circ\text{C}$ , and were de-aerated by a stream of ultra high-purity argon. In this way, the only variables affecting the corrosivity of the synthetic minewaters were the different contents of chloride and sulphate ions. All the other variables that could influence corrosion were kept constant.

#### General Corrosion Studies

Potentiodynamic polarization studies extending into both the cathodic and the anodic regions were carried out on the three alloys in the synthetic minewaters. The potentiodynamic polarization curves of potential versus current density were obtained with a computerized Princeton model 273 corrosion-measurement system and model 332 software. The general corrosion rates of the mild steel

and stainless alloys were determined from these potentiodynamic curves. The curves also give an indication of the overall corrosion behaviour at various potentials, i.e. active or passive potential ranges can be identified, and also the regions of pitting and general corrosion.

The corrosion cell and the test procedure documented in the ASTM G5 standard were employed in the general corrosion tests. The sample-holder was a modified version using flat specimens machined from round bar and ground to a final finish of 1200 grit. The standard ASTM sample-holder has been criticized worldwide, since crevice corrosion is likely to occur in holders of this design. To obviate this drawback, the edges of the specimens were coated with Fortolac lacquer. The specimens were allowed to corrode freely in the de-aerated waters for 1 hour before polarization commenced, by which time a stable free-corrosion potential ( $E_{\text{corr}}$ ) had been established. The potentiodynamic scans were initiated in the cathodic region 100 mV below  $E_{\text{corr}}$  and usually terminated at 1200 mV. All the potentials were measured relative to SCE, and a scan rate of 1 mV/s was used throughout the tests.

The polarization-resistance technique was used to determine the general corrosion rates of mild steel, 3CR12, and 316L from the data in the range  $E_{\text{corr}} \pm 20$  mV. In order to apply this technique with accuracy, the anodic and cathodic Tafel constants have to be established by a separate method. In the present study, the Tafel constants were determined with software for Parcalc Tafel analysis. An iterative computer procedure is used in this analysis, in which an equation of the following form is fitted to the experimental data with a 'best-fit criterion':

$$I = I_{\text{corr}} \left( e^{2.3DE/\beta A} - e^{-2.3/\beta C} \right)$$

where  $I$  = measured current density at potential  $E$   
 $I_{\text{corr}}$  = corrosion current density  
 $\beta A$  = anodic Tafel constant  
 $\beta C$  = cathodic Tafel constant  
 $DE$  = overpotential ( $E - E_{\text{corr}}$ ).

The general corrosion rates of the mild steel and the two stainless alloys were determined by the procedure set out above, and the pitting potential behaviours of the 3CR12 and 316L alloys were recorded from the potentiodynamic polarization curves. These differed slightly from those determined in the actual pitting scans, since the latter excluded the cathodic polarization step to avoid interference with the passive film.

#### Studies of Pitting and Crevice Corrosion

Scans of pitting polarization were conducted in the synthetic minewaters for the two stainless alloys 3CR12 and 316L so that the influence of different contents of chloride and sulphate ions on the resistance to pitting and crevice corrosion could be assessed.

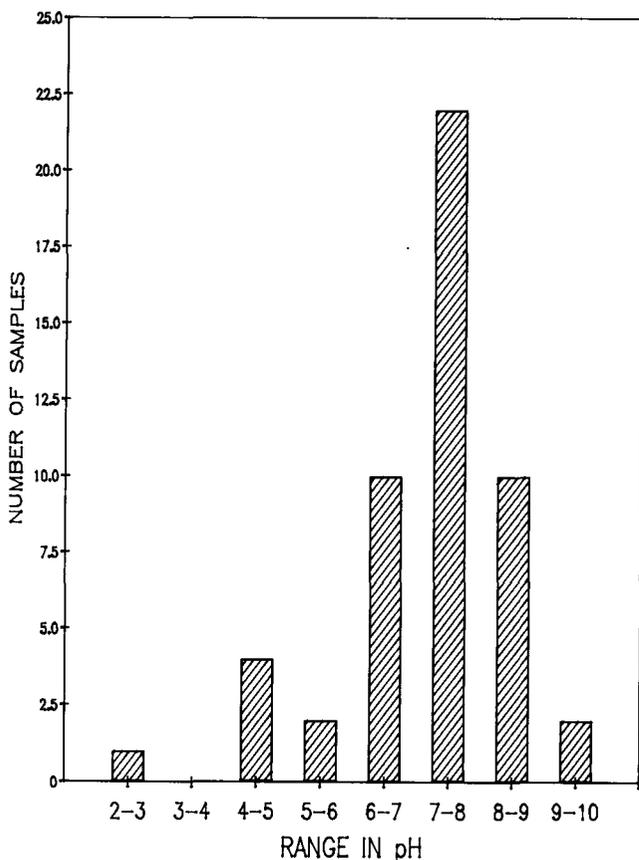


Fig. 4—Distribution of pH values in South African minewaters (after Higginson and White<sup>1</sup>)

The pitting scans were obtained according to the guidelines given in the ASTM G61 standard. The specimens were again ground to a 1200 grit finish, and the edges were coated with Fortolac lacquer to avoid crevice corrosion. In this way, pitting potentials could be established with a reproducibility of  $\pm 20$  mV. The scans were started at  $E_{corr}$ , after a stabilization period of 1 hour, at a scan rate of 1 mV/s. The scans were usually reversed at a threshold current of  $8 \times 10^4 \mu\text{A}/\text{cm}^2$ , although the different corrosivity of the various waters made it impossible to establish a specific value of threshold current suitable for all cases. The solutions were also de-aerated with UHP argon and maintained at  $30 \pm 1^\circ\text{C}$ .

The pitting resistance of the 3CR12 and 316L alloys was characterized by the pitting potential ( $E_p$ ) and the protection potential ( $E_{pp}$ ). The pitting potential signifies the onset of pit initiation. Pre-existing pits may still grow below the pitting potential when pit initiation does not occur. The protection potential is defined as that potential below which neither pit initiation nor growth takes place, and is the potential at which the reverse pitting scan intersects the forward scan. As in the case of the pitting potential, high values of the protection potential are a clear indication of improved pitting resistance. The resistance of a variety of stainless alloys to crevice corrosion has been found to be directly proportional to the difference between the  $E_p$  and the  $E_{pp}$  values<sup>5</sup>. However, this correlation appears to be mainly of an empirical nature.

#### Experimental Results and Discussion

In all the synthetic minewaters tested, the mild steel underwent active corrosion. Corrosion proceeded uniformly, and the general corrosion rate increased steadily with increasing potential. On the other hand, as expected, the 3CR12 and 316L alloys passivated spontaneously in all the solutions. There were no active peaks nor extensive passive ranges over which the current was very low. Pitting occurred at high potentials, the onset of pitting being characterized by a sharp increase in current.

In the case of the mild steel, the general corrosion resistance was found to be adversely affected by increases in the concentrations of both the chloride and the sulphate ions. An example of the detrimental effect of increasing sulphate ions is shown in Fig. 5. At corresponding potentials, the current densities (a direct measure of corrosion rate) were significantly higher in the water containing the additional 300 p.p.m. of sulphate ions. The general corrosion rates of mild steel are shown as a function of the total concentration of chloride and sulphate ions in Fig. 6. Higginson and White<sup>1</sup> have shown that, in the case of mild steel, the dominant cathodic reaction in de-aerated minewaters is diffusion-controlled oxygen reduction provided the pH exceeds a value of 5. De-aerated minewaters were found to contain oxygen concentrations of less than 0,1 mg/l. It is evident in the present work that the corrosion rate increased steadily with the total concentration of anions. The results are consistent with those of Rawat<sup>6</sup>, who demonstrated the corrosive nature of both chloride and sulphate ions towards mild steel in the environments of Indian coal mines. The increased corrosion rate of the mild steel with increasing chloride and sulphate ions is believed to be mainly due to the resulting

increase in solution conductivity. Higginson and White<sup>1</sup> demonstrated the essentially linear relationship between total dissolved solids and conductance in the waters of South African gold mines (Fig. 7).

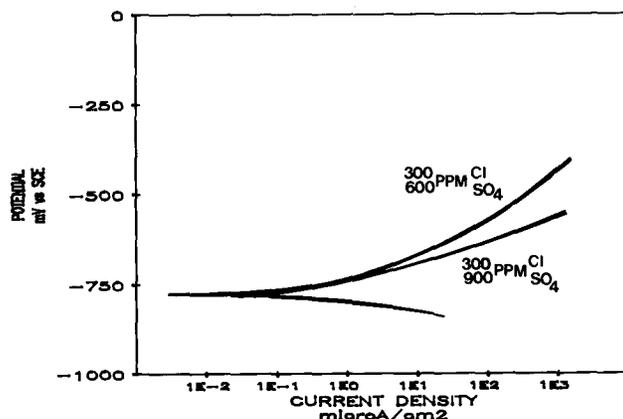


Fig. 5—Potentiodynamic polarization curves of mild steel at different sulphate levels

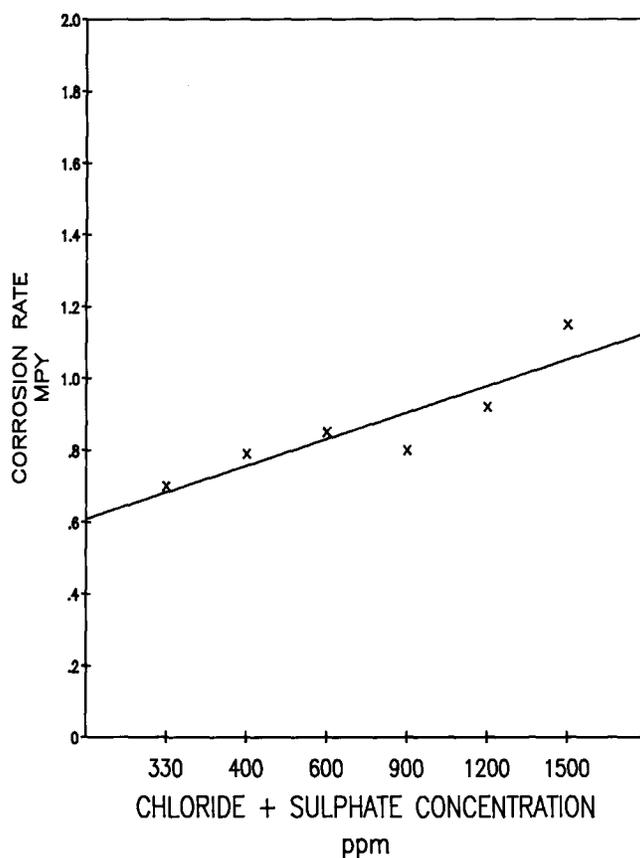


Fig. 6—General corrosion rate of mild steel as a function of combined chloride and sulphate contents

In contrast to the detrimental effects observed for the mild steel, the potentiodynamic polarization curves revealed distinct beneficial effects of sulphate ions on the corrosion resistance of the two stainless alloys 3CR12 and 316L. The beneficial effects of increased sulphate ions are clearly demonstrated in Figs. 8 and 9. Increased sulphate levels (at a fixed chloride content) decreased the passive current densities of both alloys, and also increased

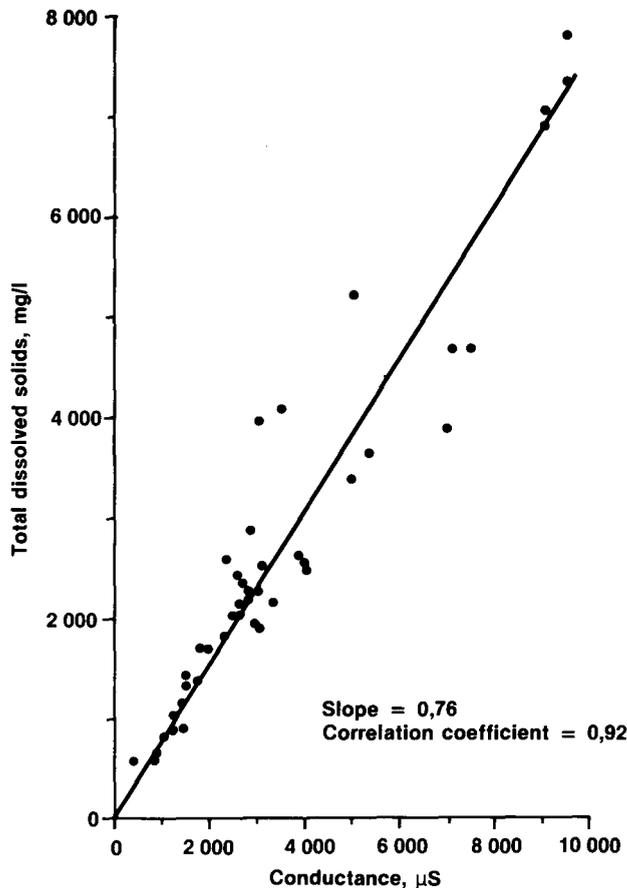


Fig. 7—Variation of total dissolved solids with conductance (after Higginson and White<sup>1</sup>)

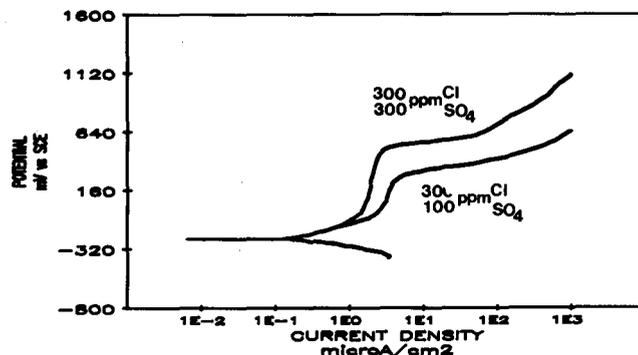


Fig. 8—Potentiodynamic polarization curves of 3CR12 at different sulphate levels

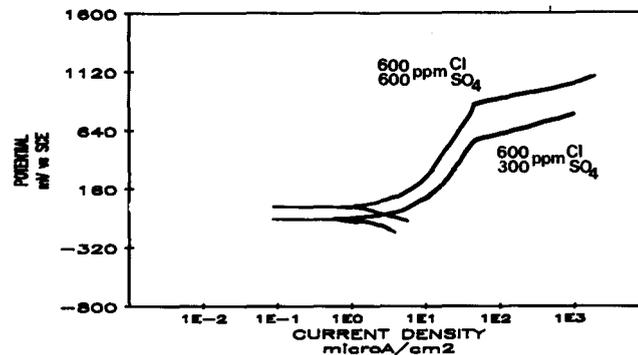


Fig. 9—Potentiodynamic polarization curves of 316L at different sulphate levels

the range of passivity preceding pit initiation. In view of the finding that the passive current densities were decreased by sulphate additions, it would be expected that the general corrosion rates of 3CR12 and 316L would also have decreased, since these alloys became corroded in the passive state in the waters concerned. This was indeed the case, as shown by the general corrosion rates in Fig. 10, in which high ratios of sulphate to chloride ions distinctly decreased the general corrosion rates of both the 3CR12 and the 316L. The deleterious influence of high levels of chloride ions may be explained by the hypothesis that (a) the chloride ions enter the passive film and thereby enhance the conduction of metal cations through the film, or (b) the adsorption of chloride ions around metal cations leads to enhanced dissolution over non- or aquo-complexed cations. From the results in Fig. 10, it is evident that sulphate ions mitigate the detrimental effects of chloride ions on the general corrosion resistance of chromium-containing iron alloys.

It should be noted that the general corrosion rates of both the 3CR12 and the 316L were extremely low (well below 1 million parts per year) and, in fact, very similar. This is to be expected since both alloys passivated spontaneously in the solutions. The general corrosion rates of the 3CR12 and 316L alloys are usually of little concern in applications involving South African minewaters by virtue of the passivation phenomenon. Of significantly more importance, however, is the effect of sulphate ions on the resistance of these alloys to pitting and crevice corrosion, as described below. The lower passive current den-

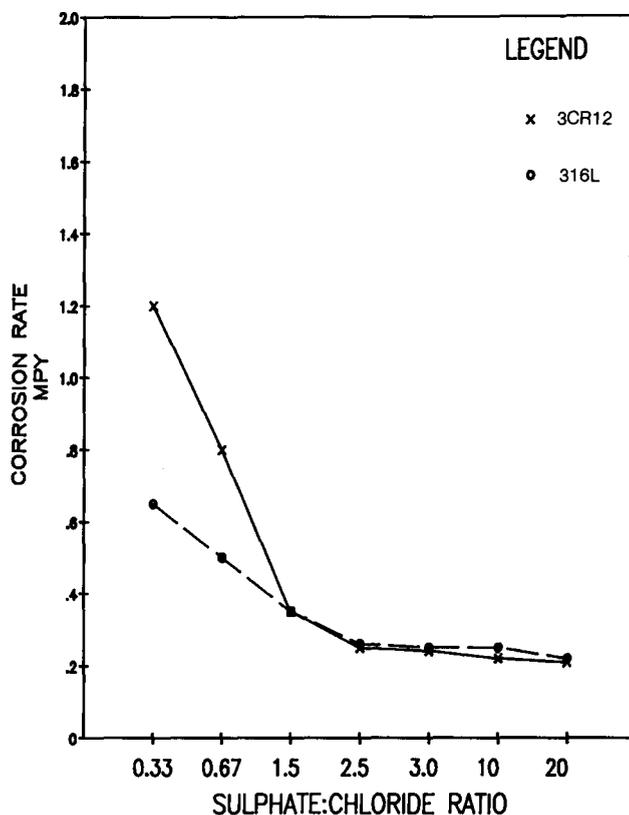


Fig. 10—General corrosion rates of 3CR12 and 316L as a function of sulphate-to-chloride ratio

sities and lower general corrosion rates recorded at high ratios of sulphate to chloride ions indicates that the stability of the passive film is enhanced at higher sulphate levels, and hence the pitting potentials can also be expected to increase.

The beneficial effects of increasing sulphate levels on the pitting resistance of the 3CR12 and 316L alloys at a fixed level of chloride ions (600 p.p.m.) are shown in Fig. 11. Significant increases in the pitting resistance (as predicted by pitting potentials) were recorded for both alloys. As expected, the pitting potentials of the 316L were significantly higher than those of the 3CR12. The superior performance of the 316L obviously results from the higher chromium content and the presence of 2 to 3 per cent molybdenum in the alloy. The results in Fig. 12 confirm the improved pitting resistance at high ratios of sulphate to chloride ions. Pitting potentials obtained by the potentiodynamic technique followed very similar trends.

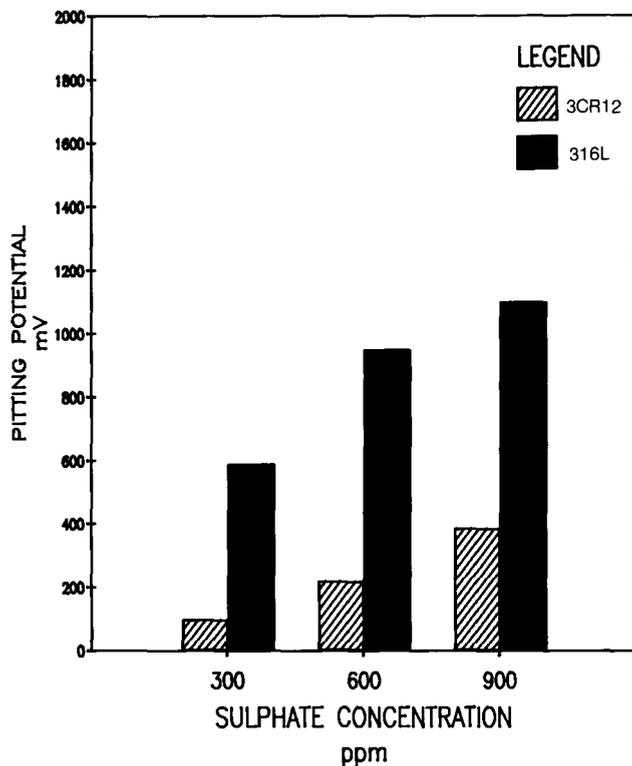


Fig. 11—Pitting potentials of 3CR12 and 316L at increasing sulphate contents with chloride level fixed at 600 p.p.m.

The detrimental effects of chloride ions in lowering the pitting potentials can usually be defined by the following relationship:

$$E_p = A - B \log[Cl],$$

where A and B are constants and

[Cl] is the concentration of chloride ions.

In the event of sulphate ions improving the pitting resistance and counteracting the effects of chloride, a relationship of the form

$$E_p = A - B \log\left(\frac{[Cl]}{[SO_4]}\right)$$

should apply. Such a relationship was indeed confirmed

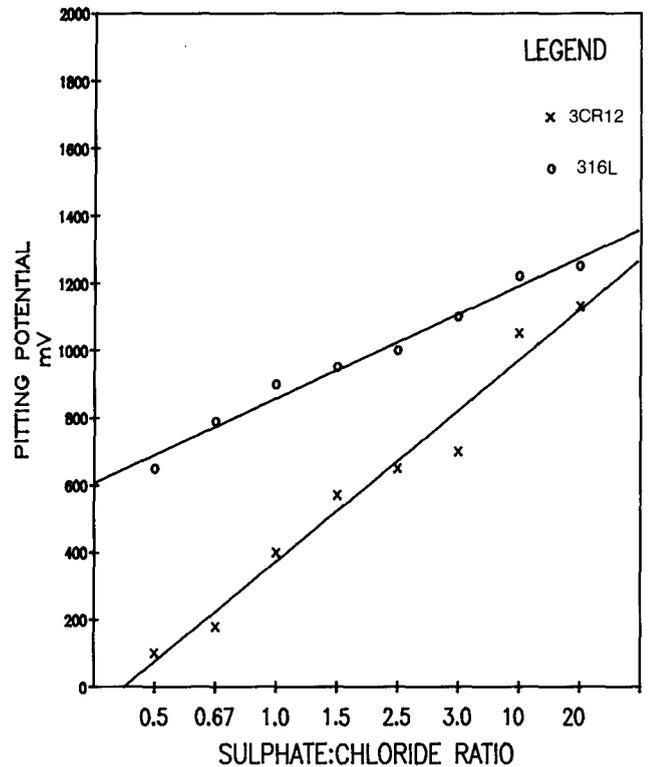


Fig. 12—Pitting potentials of 3CR12 and 316L as a function of the sulphate-to-chloride ratio obtained from pitting scans

in the pitting scans, as shown in Fig. 13 for the 3CR12 and 316L alloys. The straight-line correlation coefficients amounted to 0,98 for the 3CR12 and 0,95 for the 316L. Similar results were obtained from the pitting potentials of the potentiodynamic scans.

The influence of sulphate ions on the pit repassivation tendencies were studied by reversing the scanning direction in the pitting scans. Typical curves for the 3CR12 and 316L alloys at high and low ratios of sulphate to chloride ions are shown in Figs. 14 and 15 respectively. At high sulphate levels, no hysteresis loops were recorded, the reverse scans returning to the left of the forward scans. This behaviour indicates that pit growth above  $E_p$  was relatively slow, and the small pits formed in the forward scans repassivated spontaneously in the reverse scan without the formation of a hysteresis loop. At the low levels of sulphate to chloride ions, hysteresis loops with associated protection potentials were recorded for both the 3CR12 and the 316L. Under these conditions, pit growth in the forward scan was relatively severe, so that these pits repassivated well only below the pitting potential in the reverse scan. The hysteresis loops also indicate that the resistance to crevice corrosion is significantly reduced as the ratio of sulphate to chloride ions changes from high to low values.

The results presented above clearly demonstrate the beneficial passivating effects of sulphate ions on the general, as well as the localized, corrosion resistance of the two stainless alloys 3CR12 and 316L. This is in direct contrast to the effect on mild steel, for which sulphate (as well as chloride) ions reduce the general corrosion resistance. Similar beneficial effects of sulphate ions on the pitting resistance of stainless alloys have been reported<sup>3,6,7</sup>. Capendale<sup>3</sup> reported the inhibitive effects of

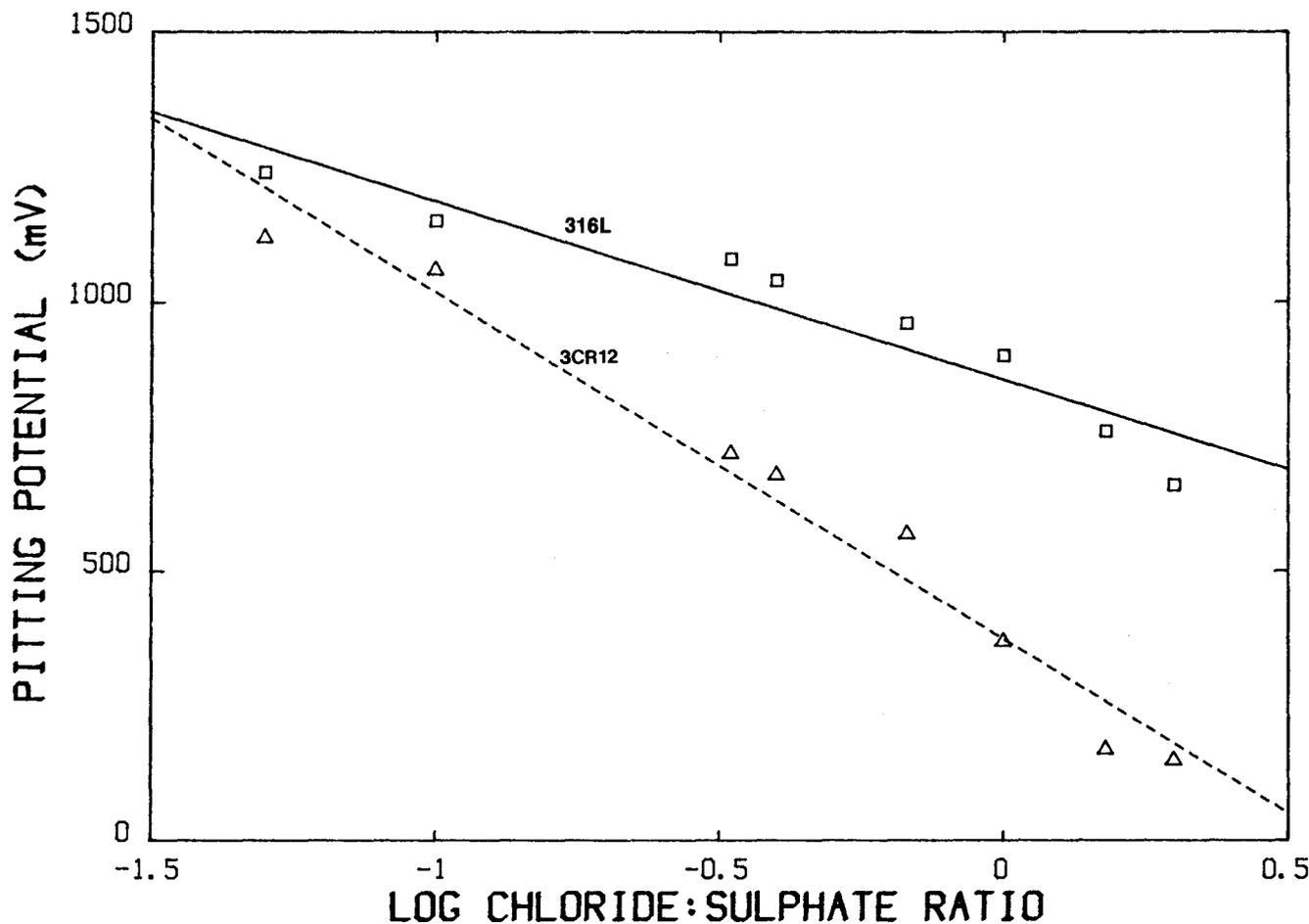


Fig. 13—Linear dependence of pitting potential on log (chloride:sulphate)

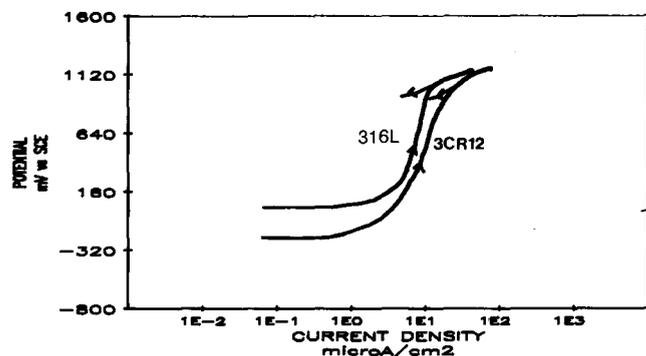


Fig. 14—Typical pitting curves of 3CR12 and 316L at high ratios of sulphate to chloride

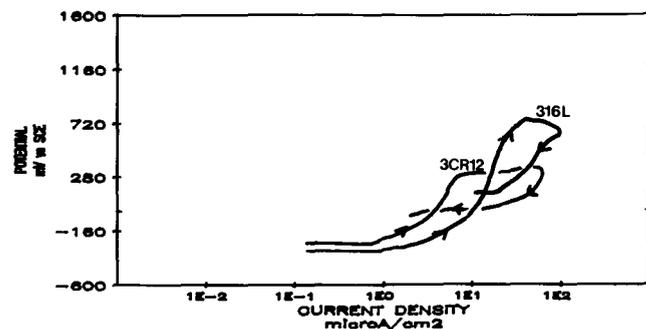


Fig. 15—Typical pitting curves of 3CR12 and 316L at low ratios of sulphate to chloride

sulphate ions on the pitting corrosion of AISI 431 stainless steel, but showed that their inhibitive power was weaker than that of nitrates on a weight-for-weight basis. Apart from demonstrating that the critical chloride concentration required for pitting corrosion was increased through sulphate additions, Capendale<sup>3</sup> demonstrated that the inhibitive effects of nitrates and sulphates can vary considerably in magnitude, depending on the pH value.

Leckie and Uhlig<sup>7</sup> reported the beneficial effects of increased sulphate levels in increasing the pitting resistance of AISI type 304 stainless steel. At fixed levels of chloride ions, the pitting potentials were significantly increased by additions of sulphate ions. Increased sulphate levels also tended to decrease the passive current densities of the 304 alloy marginally, as also recorded for the 3CR12 and 316L alloys in the present investigation. The beneficial effects of sulphate ions were ascribed to a competitive adsorption mechanism. Adsorbed oxygen, rather than metal oxide, is considered to make up the passive film, with aggressive chloride ions competing with oxygen for sites on the surface of the stainless steel. Sulphate ions also become adsorbed on the surface, thereby effectively excluding damaging chloride ions. In the presence of sulphate ions, a shift of potential to more noble values is required, so that a critical concentration of chloride ions in the double layer is reached to destroy passivity locally. Palumbo *et al.*<sup>8</sup> also observed enhanced pitting resistance in a stainless alloy at higher sulphate concen-

trations in neutral chloride-sulphate synthetic waters and also in tests on lake water. Increased levels of sulphate resulted in more-noble pitting potentials and less-extensive pitting attack on polarized samples. The beneficial effects of sulphate ions were rationalized in terms of the competitive adsorption theory proposed by Leckie and Uhlig<sup>7</sup>.

In contrast to the above mechanism, and based on the more modern concept that pitting resistance is mainly controlled by the repair of a passive oxide film, rather than by its ability to resist breakdown, Newman *et al.*<sup>9</sup> proposed that, even in the earliest stages of pit nucleation, the electro-transport of sulphate ions would slow down the accumulation of harmful chloride ions at the metal-solution interface. It was suggested that sulphate ions inhibited pitting corrosion by reducing the interfacial concentration of chloride ions, thereby stimulating conditions for film repair.

### Conclusions

The following conclusions were drawn from the accelerated electrochemical corrosion tests carried out in synthetic minewaters.

- (1) The mild steel underwent general corrosion in chloride-sulphate solutions and did not passivate. The corrosion resistance of the mild steel did not improve at higher concentrations of sulphate, but was significantly decreased by both chloride and sulphate ions.
- (2) Increased sulphate levels tended to lower the passive current densities and reduce the general corrosion rates of both the 3CR12 and the 316L specimens in the waters tested. The general corrosion rates of these two alloys were lower than that of the mild steel, since they corroded in the passive state.
- (3) Of most importance was the finding that increasing concentrations of sulphate ions significantly improved the resistance of the two stainless alloys 3CR12 and 316L to pitting (and also crevice) corrosion. Sulphate ions thus effectively counteracted the deleterious effect of insidious chloride ions. As expected, the more highly alloyed 316L had superior resistance to localized corrosion than the lower-alloyed 3CR12.

It is evident from the results obtained that sulphate ions play a major role in the corrosivity of South African minewaters, a fact that is possibly often overlooked when the levels of chloride ions are being considered. In the case of stainless steels, sulphate ions exert a definite beneficial passivating effect by increasing the pitting potential ( $E_p$ ). They also increase the protection poten-

tial ( $E_{pp}$ ). Pitting corrosion will occur in minewaters if the free corrosion potential,  $E_{corr}$ , exceeds either  $E_{pp}$  (in the case of pre-existing pits) or  $E_p$  (in which case pre-existing pits will grow and new pits will initiate on the surface). The desirability of high  $E_p$  and  $E_{pp}$  values thus becomes apparent as  $E_{corr}$  in an actual minewater may be shifted to noble potentials by oxidizing ions such as  $Fe^{3+}$  and  $Cu^{2+}$  or by galvanic action. In addition,  $E_p$  and  $E_{pp}$  may be reduced under crevice conditions where local acidity and chloride ions can accumulate, by mechanical surface damage, or when the operating temperature is increased. In view of the above, the desirability of maintaining high levels of sulphate ions and low levels of chloride ions to limit pitting attack on stainless steels in minewaters is apparent. Unfortunately, in many treatments of recirculating minewaters, all the ions are removed in equal proportions, without selective removal of the chloride ions<sup>3</sup>. At first sight, an effective water treatment would involve the addition of inhibitive sulphate ions. However, this measure could have severe drawbacks in the presence of sulphate-reducing bacteria. These bacteria produce highly corrosive species, such as sulphide ions from sulphate ions, which tend to cause extremely severe pitting corrosion in mild and stainless steels, including 316L. The activity of sulphate-reducing bacteria would have to be closely monitored, and possibly controlled, through the addition of biocides, before any thought could be given to the addition of sulphate ions as corrosion inhibitors.

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