THE HISTORY AND DEVELOPMENT OF DUPLEX STAINLESS STEELS

"ALL THAT GLISTERS IS NOT GOLD"

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ABSTRACT

Duplex stainless steels are being used extensively in the oil, gas and petro-chemical industries because of their unique combination of mechanical and corrosion resistance properties. This paper briefly traces the history and development of duplex stainless steels and considers in detail the role of certain alloying elements in improving the corrosion performance and mechanical properties of these stainless steels.

Field service experience has shown that some duplex stainless do not perform as well as others and the need to incorporate more stringent compositional controls into specifications to achieve consistent corrosion performance is highlighted. The influence of microstructure on the properties of duplex stainless steel and on the general and localised corrosion performance is also discussed. A comparison of the pitting and crevice corrosion performances of certain commercially available duplex, austenitic, super-austenitic and super-duplex stainless steels in sea water environments is also presented.

Introduction

While the duplex stainless steels have been popular in the Communist world for many years, the use of duplex stainless in the Western world has mainly been limited to specialists applications. One of the early developments was in the 1930's (1) when it was found that the introduction of ferrite into austenitic stainless steel increased the resistance to sensitisation. In those days this was significant in view of the high carbon content prevalent in stainless steels at that time. Another early use of ferrite in austenitic stainless steels was to improve the proof stress of castings. The use of controlled ferrite contents in specially developed grades of 'austenitic' castings enabled significant strength advantages to be achieved (2). The use of small amounts of ferrite in wrought austenitic steels was not as popular in view of the poor hot workability properties that resulted. However, systematic studies of plasticity in relation to temperature and the relationship between the ferrite and austenite phases made it possible to optimise the conditions for the hot plastic deformation of ferritic-austenitic stainless steels.

Nevertheless it was not until the 1950's when nickel shortages were being experienced, that duplex stainless steel development programmes gained some impetus. It was realised that significant improvements in the strength properties and corrosion resistance of the alloys could be obtained. Alloys developed in the early 1950's contained typically 25-26wt% chromium, approximately 4-5wt% nickel and 3wt% copper. However, owing to the lack of understanding of the physical metallurgical principles governing these alloys, they possessed poor toughness and ductility properties. It should be stressed that the heat treatment applied to such alloys included a relatively slow air cool, water quenching being considered too severe for practical applications. It was considered essential to improve the ductility of the steel in the air cooled condition and work was undertaken to study the effect of chromium and nickel variations on ductility. Since the maximum nickel content envisaged at the time was 5wt% it was found that the best ductility was obtained in alloys containing Cr22-23 wt%⁽³⁾. The nickel shortages however, receded and development work on the duplex alloys virtually ceased.

In the late fifties the Alloy Casting Institute was developing a duplex stalatess steel subsequently designated CD4MCU. This was a 25-wtCr-5wt%Ni-2wt% Mo-3wt%Cu duplex stainless steel with excellent strength and good corrosion resistance. However, problems of brittle failure were encountered during manufacture and in service. This resulted in a lowering of the maximum chromium content to optimise the microstructural phase balance. It was found that the ductility was improved if alloy was solution heat treated and water quenched (4,5)

However, it was not until the late 1960's and early 70's, at the time of another nickel shortage, that the full potential of the duplex alloys became appreciated. It was recognized that conventional austenitic stainless steels were inherently low strength alloys and could suffer from pitting and crevice corrosion in the presence of chloride ions, due to localised breakdown of the passivating surface film. Austenitic alloys were also prone to stress corrosion cracking in chloride environments. Increased activity in the off-shore oil industry demanded a material to combat the severe conditions encountered in marine environments such as North Sea Oil fields.

This led to the development of several high chromium duplex ferritic-austenitic alloys and interest in such stainless steels, for use in marine industries, has increased during the last decade. Duplex stainless steels with ferrite-austenite microstructures have been studied extensively and several papers exist for background reading (3, 6-9). It can be deduced from this literature that duplex stainless steels can offer several advantages, when compared to conventional austenitic stainless steels, and some of these are listed below:

- Proof strengths are substantially higher than those of austenitic types while still retaining good levels of toughness.
- The concentrations of those elements giving improved resistance to pitting and crevice corrosion may be increased without undue increase in nickel content.
- An attractive balance between resistance to pitting in chloride solutions and general corrosion in acids is attainable in one alloy.
- Resistance to sensitisation is good even without stabilisation.
- Resistance to chloride stress corrosion cracking is significantly better than that of the austenitic grades.

Improved Duplex Alloy

Notwithstanding these comments it has been found desirable to develop a duplex alloy possessing even better corrosion characteristics and improved mechanical properties, castability and weldability, yielding a general purpose alloy having wide acceptance and application in the marine engineering industry. To achieve this objective it was

necessary to study the effect of various alloying elements on the corrosion performance and mechanical properties of duplex stainless steels. Changes in alloying element content which are possibly desirable from the point of view of establishing attractive corrosion resistant characteristics, can have a marked effect on the metallurgy of duplex stainless steel. Consequently, this can effect the physical and mechanical characteristics, thus placing a constraint upon the type and degree of alloying which can be used practically. The establishment of certain desirable properties such as high strength with acceptable toughness and ductility is dependent upon the control of the microstructure and this may limit the corrosion resistance attainable Alloying elements in solid solution, manganese sulphide inclusions, various phases which can precipitate giving chromium and molybdenum depleted zones around the precipitates can all have a profound influence on the microstructure, the mechanical properties of the alloy and on the maintenance and breakdown of passivity.

A logical way to handle such a complex array of metallurgical variables is to examine how each variable effects passivity, microstructure and the mechanical properties and to incorporate this knowledge into new alloy development programmes, fabrication and heat treatment schedules.

Reference to the anodic polarisation curve shown in Figure 1, schematically summarises the effect of certain alloying elements in enhancing the passivity of stainless steel (10). Several studies have been undertaken to determine the effect of certain alloying additions to duplex stainless steels and these are summarised below:

Effect of Chromium

Stainless steels derive their passivity from alloying with chromium. Alloying iron with chromium moves the primary passivation potential Epp (figure 1)(10) in the active direction, this expands the passive potential range and reduces the passive current density ipass. In chloride solutions increasing the chromium content of duplex stainless steel raises the pitting potential Ep, thereby expanding the passive potential range. The beneficial effect of chromium on the pitting characteristics of duplex steel is illustrated in figure 2,(11) and similar results have been obtained by other investigators (12). An increase in chromium, which is a ferrite stabiliser, can be balanced by increased nickel in order to retain the correct microstructural phase balance. However, there is a maximum limit to the level that Cr can be increased without enhancing the rate of sigma phase formation, which could reduce the ductility, toughness and corrosion properties of the alloy.

Effect of Molybdenum

For a given chromium content, molybdenum has a strong beneficial influence on passivity. As shown in figure 1⁽¹⁰⁾ Mo additions move the pitting potential in the more noble direction thus extending the passive potential range. The exact mechanism by which molybdenum exerts its beneficial influence is not fully understood (13-16). Increased molybdenum content also lowers imax and thus molybdenum improves the resistance to general corrosion, pitting corrosion and crevice corrosion in chloride solutions.

In duplex alloys, (figure 3)⁽¹⁷⁾ it can be seen that more than 3wt% molybdenum is essential for the prevention of crevice corrosion in high temperature sea water. However, this effect can

diminish markedly during a weld cycle if the precipitation of chromium nitride/carbide occurs in the ferrite. The maximum level of molybdenum which can be added to duplex stainless steels is restricted to around 4 wt% since molybdenum additions widen the sigma phase field and can extend it to temperatures in excess of 1000°C. Consequently, during the casting of thick sections it would be difficult to avoid sigma phase formation with a resultant loss of ductility and toughness.

Effect of Nitrogen

One of the most significant improvements in the localised corrosion performance of duplex stainless steels is obtained by increasing nitrogen levels in the region of 0.2wt%-0.3wt% (figure 4)(18). Nitrogen moves the pitting potential in the noble direction and extends the passive potential range (figure 1). The effect of molybdenum and nitrogen in improving the pitting resistance of duplex stainless steel has been demonstrated by Sakai et al (12) as shown in (figure 5). Trueman et al (19) have shown that the beneficial effects of nitrogen appear to be enhanced in the presence of molybdenum.

The mechanistic role of the alloying elements molybdenum and nitrogen in determining passivity remains to be established. Preliminary research suggests that nitrogen may reduce elemental partitioning of chromium in duplex stainless steels (20) and also modify the passive film thus improving localised corrosion performance.

The main experimental difficulty is that the processes occurring at the instant of destruction of a passive film proceed very quickly. Surface analysis can at best only give an indirect indication of the enrichment or depletion processes

that may be related to alloying effects. Lu et al (21) used Auger electron spectroscopy to show that high nitrogen concentrations were obtained at the metal-passive film interface, whereas molybdenum concentrations did not show any increase. Enrichment of elemental nitrogen has been shown to develop during prolonged passivation of stainless steel in acid solutions. However, since this process is relatively slow it can only be used to explain enhanced localised corrosion resistance in areas where enrichment would occur more rapidly e.g. at "weak" points or breakdown sites in the passive film.

In order to initiate a pit in stainless steel the local dissolution rate (anodic current density) must be extremely high, many amps cm⁻². In recent work carrying out electrochemical experiments in H Cl solutions, simulating the solution in a pit nucleus, it has been shown that alloying a stainless steel with nitrogen "reversibly" impedes anodic dissolution from reaching these extremely high values. The dissolution rate at which this phenomenon occurs is in the region of 0.5A cm⁻², which is probably in the same range as the current densities required to sustain pitting in small cavities. The inhibition probably arises because the reaction for the dissolution of nitrogen, $(N + 4 H^{\dagger} + 3e =$ NH4⁺) which is a cathodic reaction, becomes too slow above a certain potential to keep pace with the anodic dissolution of the metal. This results in a surface enrichment of N2 atoms which produce the observed blocking effect (22). In fact, no oxide passivation is involved in this process.

Nitrogen levels of 0.2wt%-0.3wt% also indirectly improve the ductility and toughness of duplex stainless steels, since nitrogen is an austenite forming element. Furthermore the nitrogen in

solid solution increases the strength of the alloy.

In previous work, high levels of nitrogen have necessitated high levels of manganese (e.g. = 4wt%) to ensure the solid solubility of the nitrogen is not exceeded. Chance et al⁽¹⁸⁾ (figure 6) followed this theory in the development of a duplex stainless steel which exhibited improved localised corrosion performance. While manganese is generally considered detrimental to the pitting resistance of stainless steel due to the formation of manganese sulphides, the combined additions of molybdenum plus nitrogen tend to override this effect and the pitting potentials exhibit high noble values. However, it is well established that manganese sulphide inclusions are a most favourable site for pit initiation in stainless steels (23). Thus maintaining manganese to extremely low levels (e.g 0.5%) results in the preferential formation of chromium sulphides which render pit initiation more difficult.

Effect of Nickel

Nagano et al⁽¹⁷⁾ reported that a nickel level of around 7wt% in duplex stainless steels is effective in reducing crevice corrosion of sensitised alloys (figure 7)⁽¹⁷⁾. The effect of nickel in reducing pitting corrosion of duplex stainless steels is demonstrated by Sakai et al⁽¹²⁾, (figure 8). They show that nickel contents should be maintained between 4% and 8% for 25%Cr alloys and 4-7% for 22%Cr alloys in order to obtain optimum pitting resistance. Nickel is seen (figure 1) to move the pitting potential in the noble direction thereby extending the passive potential range and also reduces the passive current density.

If nickel contents are significantly in excess of the optimum for pitting resistance, so that the austenite content increases markedly above 50%, the residual more highly alloyed ferrite, as a result of partitioning will more readily transform to the brittle sigma phase at temperatures in the range 650-950°C. This will adversely affect the hot working characteristics of wrought steels, impact toughness, ductility and weldability of cast duplex stainless steels (11). Conversely if the nickel content is reduced below the optimum level for pitting resistance, leading to high ferrite contents, low toughness will result. This is because the delta ferrite formed immediately on solidification tends to have low dugtility associated with larger grain size (11).

Effect of Tungsten

Tungsten at a level of approximately 0.6wt% has also been added to duplex stainless steels to extend the passive potential range and reduce the passive current density. (24). Nagano et al (17) state the tungsten increases the immunity potentials to crevice corrosion of duplex stainless steels in high temperature chloride solutions to levels above which crevice corrosion starts as shown in (figure 9). ESCA studies of the passive film indicate that tungsten is adsorbed without modification of the oxide state and that the tungsten is present in the passive layer. (25). In acid chloride solutions it would appear that tungsten probably passes directly from the metal into the passive film, by interaction with water and formation of insoluble WO3 rather than through a dissolution then adsorption process. Whereas in neutral chloride solutions, the beneficial effect of tungsten is interpreted by the interaction of WO3 with other oxides, resulting in enhanced stability and enhanced bonding of the oxide layers to the base metal.

It can be inferred that tungsten will act in a similar manner to molybdenum with respect to enhanced sigma phase formation. Consequently there is a limit on the level of alloying that is practicable otherwise the sigma kinetics are enhanced potentially reducing the ductility and toughness of the alloy.

Effect of Cooper

The beneficial effect of copper additions on the corrosion resistance of stainless steels in non-oxidising media is well known. If approximately 0.5wt% of copper is added, the active dissolution rate of duplex stainless steels in boiling hydrochloric acid and the crevice corrosion loss in chloride solutions are both decreased. Guha and Clark (11) state that the addition of copper required to achieve the optimum corrosion resistance in 70% HoSO4 at 60°C decreases from 1.5% in a 25%Cr duplex stainless steel to 1% in a 28%Cr duplex stainless steel. Figure 10⁽¹¹⁾ shows the effect of copper in improving pitting and the general corrosion resistance of duplex stainless steet.

Additions of copper have been claimed to be of particular advantage in sea water contaminated with H₂S at high flow velocities where cavitation erosion can occur (26) although this has not been borne out by subsequent field service experience. The improvement in the corrosion performance is said to come from the formation of a copper layer on the stainless steel surface during the active dissolution rather than changes in the intrinsic passive behaviour (27).

The maximum level of copper utilised in wrought duplex stainless steel is limited to approximately 2wt% since

higher contents reduce hot ductility. Furthermore, additions of copper have been reported to promote precipitation hardening in duplex stainless steels (11).

Effect of Other Alloving Flements

Among the other alloying elements that move the pitting potential in the noble direction, thereby extending the passive potential range, are vanadium and silicon (figure 1). Wilde (28) has patented compositions of high silicon (3.5-5.5wt%) duplex stainless steels that are reported to be immune from stress corrosion cracking using the boiling magnesium chloride solution tests. However silicon contents in excess of approximately 1.0wt% disproportionately enhance sigma phase formation and can lead to poorer corrosion resistance in duplex stainless steel retained in the as welded condition.

The carbon content of most wrought duplex stainless steel is restricted to 0.030wt% maximum to ensure good hot workability. The low carbon concentrations inevitably suppress carbide precipitation and further reduce the susceptibility of duplex stainless steel to localised corrosion in the as-welded condition. Modern melting techniques also ensure that extremely low sulphur and phosphorus contents are obtained to improve the cleanness of the steel and hence the overall corrosion performance.

Pitting Resistance Equivalent

It is evident from the foregoing that a number of alloying elements in stainless steels move the pitting potential in the noble direction. These beneficial effects are complex and interactive and attempts have been made to use compositionally derived empirical relationships for pitting resistance indexes, (PRE: PITTING RESISTANCE EQUIVALENTS). The general formulae for nitrogen containing duplex stainless steels is as follows (10).

 $PREN = %Cr + 3.3\%Mo + 16\%N_2$

It is generally regarded that PREN values of >35 are necessary to determine whether an alloy will display good pitting performance in deaerated seawater. However in warm raw oxygenated seawater PREN values of >40 are necessary to guarantee the localised corrosion resistance of stainless steels. There are reservations concerning the utilisation of such formulae in total isolation. The formulae do not take account of the beneficial effects of other elements which improve pitting performance such as tungsten. The equations totally ignore the effects of microstructural factors on the breakdown of passivity by pitting or crevice corrosion. Nevertheless by incorporating minimum PREN values into specifications enables a more consistent corrosion performance to be obtained.

To demonstrate the important influence of PREN value on the localised corrosion performance of stainless steel, (pitting and crevice corrosion), a number of accelerated corrosion tests have been undertaken in sea water using potentiostatic polarisation techniques. (29) Figure 11 shows a graph of the critical pitting potential versus temperature and is representative of the results obtained on the commercially available alloys listed in Table 1. From these results the alloys can be ranked in terms of their critical pitting temperature in sea water as follows:

(* These alloys were tested but are not shown in Figure 11)

The results of the critical pitting temperatures obtained on these stainless steel alloys have been plotted against their respective PREN values as shown in Figure 12. A regression analysis has been undertaken on this data and shows that a good correlation exists between the critical pitting temperatures and the PREN values. Similarly an excellent correlation is found if the critical crevice temperatures determined for these alloys are plotted against their PREN values as shown in Figure 13. The ranking of these alloys in terms of their critical crevice temperature in sea water is as follows:

Zeron 100 (most resistance) > DP3 > UNS S31254 (254 SMO) > Sanicro 28 > 2RK65 > Zeron 25 > Ferralium > SAF 2205 > 316L. It is also worthy of comment that the data points for the tungsten containing stainless steels (Zeron 100 and DP3) would lie closer to the regression line if the PREN formula was to take account of tungsten. Indeed if tungsten is assume it to have a similar potency to molybdenum then the data points would be shifted by an amount equivalent to that shown in figures 12 and 13.

The crevice corrosion resistance of stainless steels and nickel alloys can also be determined using a mathematical modelling technique which simulates the corrosion process (30). The technique takes account of many factors including crevice type, crevice geometry, crevice solution, alloy composition, bulk solution etc. An

example of the ranking produced by this technique is shown in figure 14. (31). From these results it can be seen that Zeron 100 shows excellent resistance to crevice corrosion. Indeed the lowest result ever recorded for Zeron 100 shows as good a resistance to crevice corrosion as the best 6Mo super austenitic grades of stainless steel. This result further confirms the superior crevice corrosion performance of the super duplex and super-austenitic grades of stainless over the traditional austenitic grades of stainless such as UNS \$8904, 316 and the 304 types. Although not represented on Figure 14, the conventional duplex stainless steels generally give a crevice corrosion performance which varies in the region of approximately 500-700 on the ranking scale. This is not altogether surprising since only one duplex stainless steel manufacturer includes minimum PREN values into its specifications. It is possible to obtain various heats of particular grades of the traditional duplex stainless steels which exhibit markedly different crevice corrosion performances. To take a specific example, the PREN value of UNS S31803 (2205) can vary from 30.53 to 37.75 according to the level of alloying. even though the material conforms to the overall compositional specifications. Clearly this difference in PREN will significantly effect the resultant crevice corrosion performance of the alloys. Indeed, according to figures 12 and 13 the critical pitting temperature will increase from approximately 30°-60°C and the critical crevice temperature from approximately 25-50°C as the PREN value changes from 30.53 to 37.75.

The ranking shown in Figure 14 has been quantified in terms of crevice gap since after the level of alloying, this is one of the more important parameters in determining whether or not crevice

corrosion will occur. The data in figure 14 can be used to define a critical gap for each alloy below which crevice corrosion will occur. Previous studies have reported that average gaps in the range 0.2 - 0.5 um are typical in practice while gaps of less than 0.2 um are extremely unlikely (32). This means that Incomel 625, Zeron 100 or UNS S31254 are acceptable for aerated sea water applications, whereas UNS S8904, conventional duplex stainless steels, type 316, type 304 or the 400 series stainless steels are not. Thus to provide a stainless steel which exhibits freedom from crevice corrosion in aerated sea water. it is necessary to use a super-austenitic stainless steel such as 254SMO or a super-duplex stainless steel such as Zeron 100.

CONCLUSION

A resume of the beneficial effects of various alloying additions to duplex stainless steels has been given. It can be concluded that the optimum corrosion performance is obtained from a super-duplex stainless steel which has a minimum PRENvalue of 40. The super duplex stainless steel should contain low carbon and be alloyed with optimum levels of Cr, Ni, Mo, Cu, W and N to achieve the correct mechanical and corrosion resistance properties. Mn, Si, S and P should also be controlled to optimise the overall corrosion performance of the steel. In view of the varicorrosion performance experienced in the traditional duplex stainless steels it would be advisable for manufacturers to incorporate minimum PREN values into their specifications to guarantee a more consistent localised corrosion performance. The new breed of super-duplex stainless steels (Zeron 100) give a localised corrosion performance in aerated seawater which is at least equivalent to the best grades of the 6 Mo super-austenitic stainless steels while giving all the strength advantages etc of the traditional duplex stainless steels. Consequently, the super-duplex stainless offer a more cost effective solution to the materials problems of the oil, gas and petro-chemical industries.

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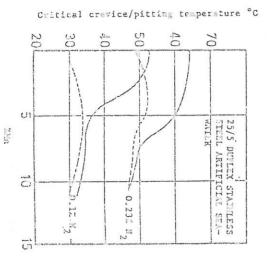
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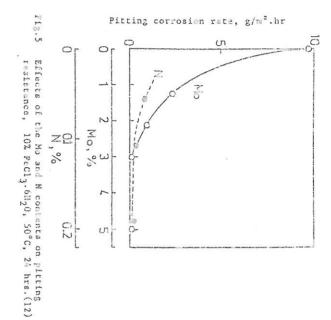
CHEMICAL COMPOSITIONS OF SPECIFIC STAINLESS STREE TABLE 1:

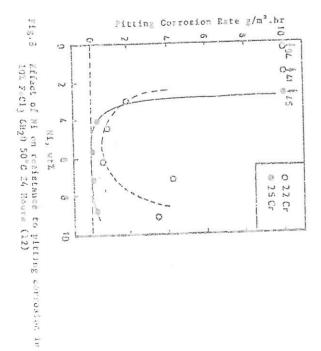
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H NOMINAL COMPOSITION

Fig. 6 Variation in critical pitting and crevicing temperatures with manganuse for two nitrogen levels (18)







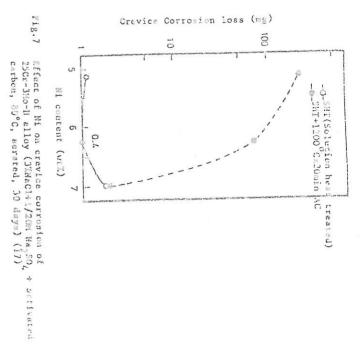
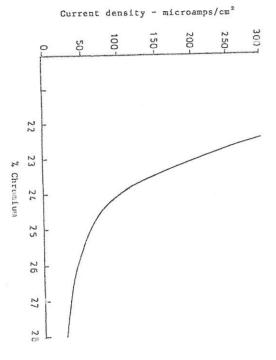


Fig. 2 Effect of chromium on pitting current density after 16 hours of Congress (S.C.F.) in 3% NaCl at 36°C (11)



Epp Cr,Ni,Wi

Epp Cr,Ni,Wi

Epp Cr,Ni,Wi

Epp Cr,Ni,V, ii

Epp Cr,Ni,V, ii

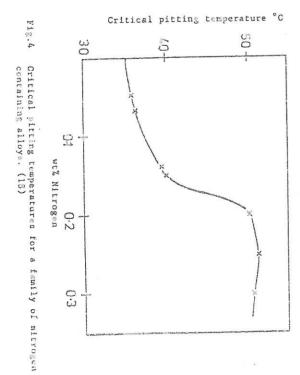
Epp Cr,Ni,V, ii

Epp Log Current Density

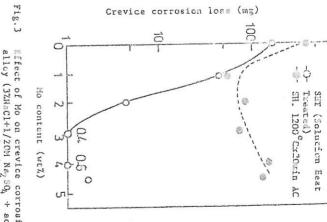
Log Current Density

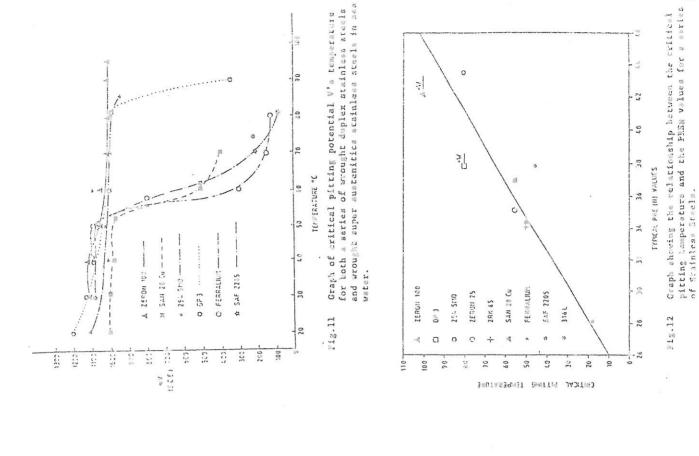
Schematic summary of the effects of alloying elements in Stainless Steels on the anodic polarization curve (10)

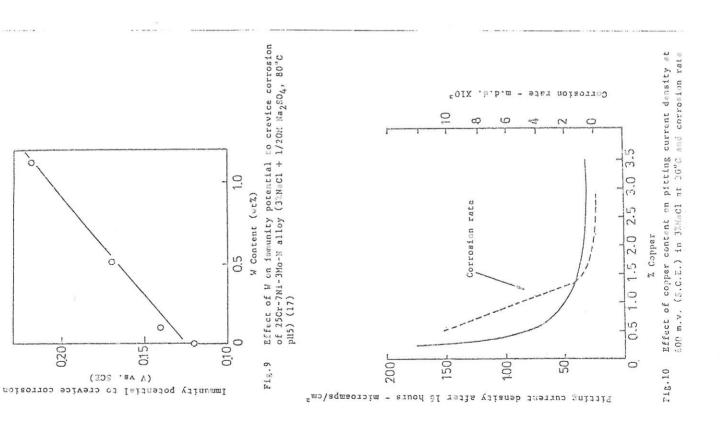
NOBLE











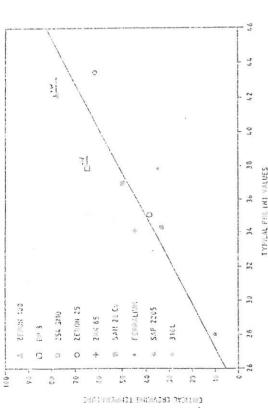
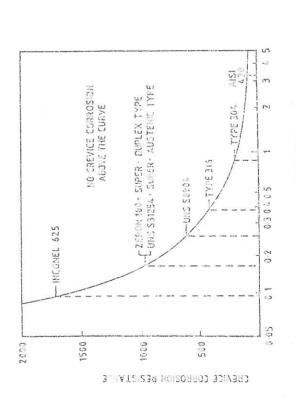


Fig.13 Graph showing the relationship between the critical crevicing temperature and the PRE values for a series of Stainlass Steels.



AVERAGE CREVICE SAP (UM)

Fig.14 Prediction of whether or not crevice corrosion vill occur in ambient temperature sessuater in a Sma deep crevice of varying widths.