# The role of Cr, Al and Mn in the resistance of refractory steels to hot salt corrosion from combustion deposits

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#### Abstract

Molten salts from combustion residues assume differentiated compositions depending on the type of fuel and the environmental variables. The choice of resistant materials should be made on the basis of more specific criteria with respect to those adopted in hot air oxidation. Three different kinds of steel (a) Fe-Cr-Al-Y, (b) Fe-Mn-Al, (c) Fe-Cr-Ni-Mn were tested in a basic mixture of molten salts (KCI-K<sub>2</sub>SO<sub>4</sub>-Fe<sub>2</sub>O<sub>3</sub>) occurring in the furnace of ferrous mineral agglomeration and in an acid one (Na<sub>2</sub>SO<sub>4</sub>-V<sub>2</sub>O<sub>5</sub>) typical of combustion systems using liquid hydrocarbons. This proved how chlorides are dangerous, particularly towards chromium-rich scales or matrices. Vanadium pentoxide is dangerous if it is associated with ferrous oxide. Manganese in the alloy helps with protection, particularly in sulphur-bearing atmospheres, also in the presence of chlorides, provided that there is no  $V_2O_5$ . Alloying with manganese is preferable in aluminium steels rather than in chromium steels, because of the high diffusivity of the element in Cr<sub>2</sub>O<sub>3</sub> scales.

#### Riassunto

## Sui ruoli del Cr, Al e Mn nella resistenza degli acciai refrattari alle corrosioni da depositi salini di combustione

I sali fusi, prodotti da residui di combustione, assumono composizioni differenziate a seconda dei combustibili e ambienti coinvolti. La scelta dei materiali resistenti deve essere fatta sulla base di criteri più specifici rispetto a quelli adottati nell'ossidazione a caldo in aria. Tre diversi tipi di acciai: a) Fe-Cr-Al-Y, b) Fe-Mn-Al, c) Fe-Cr-Ni-Mn sono stati provati in una miscela basica di sali fusi (KCI-K<sub>2</sub>SO<sub>4</sub>-Fe<sub>2</sub>O<sub>3</sub>) ricorrente in forni di agglomerazione di minerale ferroso e in una acida (Na<sub>2</sub>SO<sub>4</sub>-V<sub>2</sub>O<sub>5</sub>) tipica dei sistemi di combustione di idrocarburi liquidi. Le prove hanno messo in evidenza come i cloruri siano dannosi soprattutto su scaglie e matrici a base di cromo. Il pentossido di vanadio è dannoso se associato a ossido di ferro. Il manganese in lega concorre alla protezione, soprattutto in ambienti solforati, anche in presenza di cloruri, ma purché in assenza di V<sub>2</sub>O<sub>5</sub>. L'alligazione del manganese è da preferire in acciai all'alluminio, piuttosto che in quelli al cromo, data l'alta diffusività dell'elemento nelle scaglie di Cr<sub>2</sub>O<sub>3</sub>.

## Introduction

Hydrocarbon products and pit coal used as fuels always contain elements which are chemically detrimental to construction materials of steam generators; sulphur, a sodium (and/or potassium in the coal) and vanadium. The elimination of these elements is limited, for economic reasons, to the treatment of natural gas only. A first type of corrosion from combustion gas affects the *low-temperature* area of steam generators, namely air preheaters, flue ducts and stacks (1). Mainly responsible for this attack is the SO<sub>3</sub> aqueous condensate from oxidation of the SO<sub>2</sub> normally contained in combustion gases. The mechanism of SO<sub>3</sub> formation lies in the reaction

$$SO_2 + O \rightarrow SO_3$$

which occurs at burner flame temperatures due to the presence of atomic oxygen. The excess air in combustion is the controlling parameter (2). By limiting the excess to only 2-3%, the quantity of SO<sub>3</sub> is drastically reduced. An excess rise in combustion air particularly favours the oxidation of vanadium, which is contained in fuel oils, to high valency oxides (V2O5) forming vanadates, which catalyse the  $SO_2 \rightarrow SO_3$ reaction. On the other hand, a low excess of air limits vanadium oxidation to the form V<sub>2</sub>O<sub>3</sub> (approx. 1970°C m.p.) and V<sub>2</sub>O<sub>4</sub> (approx. 1967°C m.p.) which, with respect to V<sub>2</sub>O<sub>5</sub> (approx, 680°C m.p.), besides not promoting SO<sub>2</sub> oxidation, are higher melting, and less reactive to alkaline sulphates, thereby preventing the formation of dangerous, low-melting compounds. A practical mean of corrosion control consists in ensuring that the minimum working temperature in the cold parts of the system (economizers, air preheaters and, particularly, stacks, funnels) is never below the dew point of the combustion gas. The dew point and

the sulphur content are linked by a linear relationship in the interval 0.5 + 4% sulphur in fuel oils (3):

$$T_{dew'}(^{\circ}C) = 8 \times \%S + 132$$

A second practical measure is in the combustion stage and consists in limiting the excess air and/or using additives, such as MgO, which have a neutralizing effect on the environment. The materials normally used are carbon steels: it is convenient to substitute steel parts from time to time, rather than using materials more resistant to sulphuric attack, or steels coated with ceramic or vitreous compounds (4).

The problem of identifying materials which are intrinsically more resistant to corrosion is of much interest, on the other hand, for the high-temperature part of the system, where oxygen, SO<sub>2</sub>/SO<sub>3</sub>, and vanadium and chloride oxides are, specifically, the agents most involved in the related corrosion. The percentage of each component varies from case to case, depending on the type of installation; an atmosphere with low oxygen activity and high sulphur activity is typical of systems for converting pit coal, while in gas turbines there are normally in play oxidizing atmospheres containing SO<sub>3</sub>. In steam generators, on the fume side of superheaters, the corrosive consumption of exposed materials noticeably increases above 500°C, reaching a maximum at 600°C, then falling again beyond 700°C (5). The reason for this lies in the fact that, within the range 500°C-600°C, condensed phases are formed (sulphates-vanadates-chlorides), which tend to volatilize above 700°C.

As said above, the  $V_2O_5$  content is linked to the excess air used for combustion. This compound, which is acid in character, tends to shift the volatilization interval towards higher temperatures, as well as to lower the fusion temperature of the mix. Chlorides further aggravate the situation: NaCl and Na<sub>2</sub>SO<sub>4</sub> form eutectic at 650°C. Moreover, chlorides are particularly aggressive to oxides, including the protective ones based on  $Cr_2O_3$  (6).

A high V/Na ratio, e.g. 350/50 ppm, rather than a high V + Na content, seems to further aggravate corrosion (7). The presence of a continuos deposit of melted salt on the exposed metallic surface can only increase the kinetics of corrosion in relation to the activities of aggressive species and their diffusivities.

A large number of experimental investigations so far carried out aimed at testing and comparatively evaluating commercial materials generically resistant to oxidizing atmospheres ( $p_{O_2} > 10^{-1}$  bar), rather than at formulating alloy steels specifically resistant to sulphurous media. The latter is always the best solution as an alternative to coats whether metallic or non-metallic, which do not offer long-term reliability for various reasons.

In formulating a potentially resistant alloy, the problems linked with hot oxidation in a specific medium, and consequently, the role of the elements constituting the same alloy, should all be taken into account. Chromium and aluminium, which form protective scales able to inhibit sulphur attack, can be foreseen in the composition. However, regarding chromium, a

substantial reservation of poor effectiveness can exist in the presence of chlorides in aggressive media. In this connection, alumina offers better protection, but the overriding problem concerns the formation of a continuous coating through the selective oxidation of aluminium in steels which contain this element in low quantities (more compatible with workability), besides the problem of resistance to spalling due to temperature changes.

Nickel, which generally has only a structural function, has showed to be a dangerous element, especially if its content in the steel is greater than the minimum enrichment required to obtain an austenitic structure (8). However, it would be rather easy to think to replace the nickel with manganese which, besides the structural effects connected with gammagenic properties, is considered for its capacity of resistance to sulphurous media and for the resistance to erosion offered by steels which contain it.

The present work is focused precisely on this last aspect, with the aim of clarifying, even in view of the forseeable development of steels with a high manganese content, the role of this element in corrosion systems in molten salts. In a previous work (9), the behaviour of Cr-Al, Cr-Ni-Mn and Mn-Al steels was investigated in mixtures of molten salts, with slight basicity, made up from KCI-K<sub>2</sub>SO<sub>4</sub>-Fe<sub>2</sub>O<sub>3</sub> of recurring composition in the furnaces of mineral agglomeration for ironmaking. Under test conditions, identical to those of the present work, mechanisms of selective internal sulphidation, which could kinetically control the formation of scales of external oxide, were evident. The manganese in such conditions diffuses from the substrate to the scale interface and explains the function of the sacrificial element, given its greater affinity for sulphur.

In the present paper, the investigation is extended to acid sulphate mixtures by adding  $V_2O_5$ , in relation to the mechanism of attack by molten salts resulting from hydrocarbon combustion. In the simulation of such mixtures, we assumed the composition 54,5%  $V_2O_5$  + 45,5%  $Na_2SO_4$  (with a V/Na ratio of around 2) representative of those normally met in practice (10). To evaluate more specifically the action of the individual components of the mixture on the various alloying elements, further tests were carried out, under the same conditions, with the following binary mixtures:

- a) 50% KCI + 50% Fe<sub>2</sub>O<sub>3</sub>
- b) 50% K<sub>2</sub>SO<sub>4</sub> + 50% Fe<sub>2</sub>O<sub>3</sub>

c) 
$$50\% V_2 O_5 + 50\% Fe_2 O_3$$

The first two mixtures refer to salt melts typical of agglomeration, the third to salt melts from hydrocarbons combustion gases. The addition of iron oxide is justified by the almost constant presence of such a compound in practical systems.

## **Materials and procedures**

Chemical analysis of the alloys studied is given in Table 1. The alloy Fe-Cr-AI-Y is a ferritic steel of the AISI type 446, with a microaddition of yttrium, to increase the adhesion of the aluminium film to the matrix, and fusion is obtained under a vacuum with subsequent hot rolling to a thickness of about 2 mm. The alloy Fe-Mn-Al is an austenitic steel, produced by melting Armco iron, electrolytic manganese, pure aluminium and graphite in an induction furnace under an argon atmosphere. After soaking for 1 hour at 1050°C, this ingot was hot-rolled to a test thickness of 2.5 mm. The alloy Fe-Cr-Ni-Mn is an austenitic steel, cast directly into bars of about 39 x 30 mm, and tested in the resulting cast structure. Specimens of the above-mentioned alloys, around 6 ×  $5 \times 2$  mm, obtained by cutting with a diamond wheel, where metallographically polished with 1  $\mu$ m alumina, degreased and weighed and their final dimensions were accurately measured. The tests for attack in saline mixtures were conducted isothermally at 750  $\pm$  5°C for 30 hours. For this, we used alumina crucibles, each one containing 3 cm<sup>3</sup> of mixture in which the specimen was immersed. The composition, by weight, of the various mixtures were as follows:

- 1) 50% KCl, 10% K<sub>2</sub>SO<sub>4</sub>, 40% Fe<sub>2</sub>O<sub>3</sub>, indicated below as (CI-S).
- 54.5% V<sub>2</sub>O<sub>5</sub>, 45.5% Na<sub>2</sub>SO<sub>4</sub>, indicated below as (V-S).
- 3) 50% KCl, 50% Fe<sub>2</sub>O<sub>3</sub>, indicated below as (Cl).

## TABLE 1 - Chemical composition of the steels used

Kinds of steel	weight %										
	С	Si	Mn	Cr	Ni	AI	Mo	Cu	Nb	Ti	Y
Fe-Cr-Al-Y	0.11	1.41	0.95	24.21	_	1.36					0.005
Fe-Mn-Al	0.95		23.30			7.40		·			
Fe-Cr-Ni-Mn	0.42	1.23	5.73	25.94	12.15		0.29	0.1	0.58	0.01	

4) 50%  $K_2SO_4$ , 50%  $Fe_2O_3$ , indicated below as (S). 5) 50%  $V_2O_5$ , 50%  $Fe_2O_3$ , indicated below as (V).

After each test, the sample was mechanically separated from the solidified mixture and finally submitted to ultrasonic treatment. Sections were examined by optical microscopy in order to determine the residual thickness and the morphology of attack, and by SEM-EDS to obtain a better metallographic view of the corroded area and the chemical analysis of the phases.

## Results

In Fig. 1, losses of metal thickness, the depth of internal

Fig. 1 - Histogram of losses in thickness, attack penetration and/or thickness of oxide in the tests in mixtures (CI-S) and (V-S).

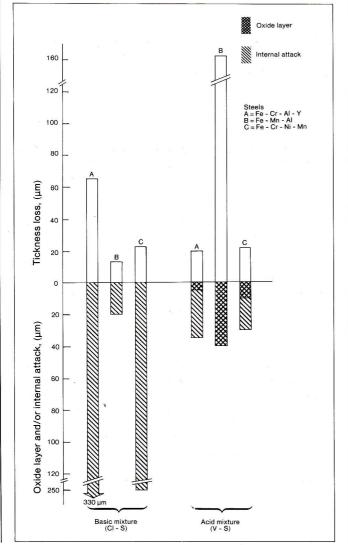
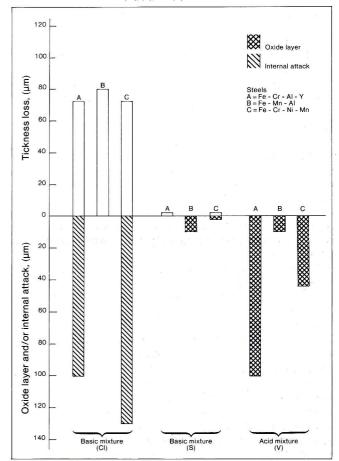


Fig. 2 - Histogram of losses in thickness, attack penetration and/or thickness of oxide in the tests in mixtures (CI), (S), and (V).



attack and the thickness of the oxide layer, whenever present, are given for the above-mentioned test samples, in mixtures 1 and 2. In Fig. 2, the same parameters are given for mixtures 3, 4 and 5. Examination of the data in Fig. 1 shows the inverse behaviour of Cr-AI-Y and Mn-AI steels, on passing from mixture (CI-S) to (V-S). In the first mixture, the ferritic steel with chromium only, clearly suffers the most corrosion of the two, while in mixture (V-S), the austenitic steel Mn-AI seems the weakest. The Cr-Ni-Mn steel, with a behaviour halfway between the two in the (CI-S) mixture, shows a greater resistance to attack in the (V-S) mixture.

**Mixture 1** (50% KCl, 10%  $K_2SO_4$ , 40%  $Fe_2O_3$ ) The results already described in reference (9) indicate (Figs. 1 and 3) a strong internal attack in the Cr-Al-Y steel which appears to be chromium-depleted. A similar but considerably reduced internal attack is observed in the Cr-Ni-Mn steel. Only a generalized non-uniform corrosion results in the Mn-Al steel, which shows to be manganese-depleted in the matrix near the surface (at 10 µm from the metal-slag interface the Mn content is reduced to only 5% the bulk value).

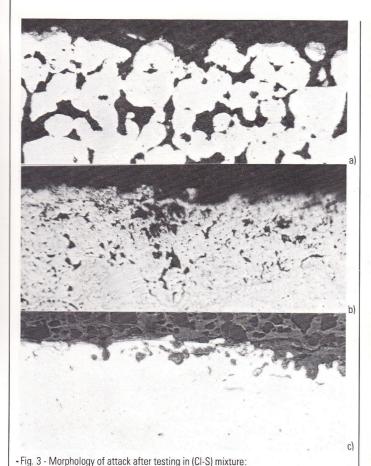


Fig. 4 - Morphology of attack after testing in (V-S) mixture of Fe-Mn-Al alloy. x1000.

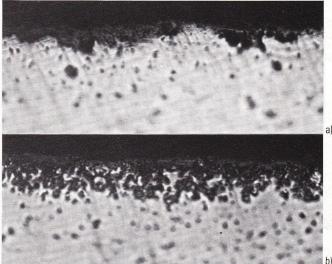


Fig. 5 - Morphology of attack after testing in (V-S) mixture: a) Fe-Cr-Al-Y; b) Fe-Cr-Ni-Mn. x1000.

other two steels, the loss is increased by about 3-4 times. As in the case of (CI-S) mixture, the attack in alloys containing chromium is mostly intergranular (Fe-Cr-Al) or interdendritic (Fe-Cr-Ni-Mn). Fig 6 shows a metallographic section of the Cr-Al-Y steel after testing: line scan analysis of the chromium indicates a total lack of this element in the attack zone, and a behaviour similar to that in mixture (CI-S), in which, as said before, the chromium content in the attack zone is reduced by 80% in respect of the bulk alloy value. The Mn-Al steel, on the other hand, undergoes a generalized but intense attack. Line scan analysis of the manganese points out a decreasing concentration in the direction from the metal matrix towards the outside

a) Fe-Cr-Al-Y; b) Fe-Cr-Ni-Mn; c) Fe-Mn-Al. x500.

#### Mixture 2 (54.5% V<sub>2</sub>O<sub>5</sub>, 45.5% Na<sub>2</sub>SO<sub>4</sub>)

Metallographic examination of the sample sections after testing indicated a rather deeper attack in the Mn-Al steel (Fig. 4); the scale is compact and thick: EDS analysis revealed an increased amount of sulphur in it as we progress inwards, until a value of 8-10% is reached near the matrix. The scale appears to be uniformly enriched in aluminium up to 10-14%; near the scale, the matrix is not depleted in manganese, while aluminium content is lowered to only 3-4%. The Cr-Al-Y steel and the Cr-Ni-Mn steel show thinner scales (Fig. 5a, 5b) which, on analysis, are seen to be made up of oxides of Fe, Cr and Al in the case of the Cr-Al-Y steel, and oxides of Fe, Cr, Ni and Mn in the case of Mn-Al steel. Slight presence of sulphur (0.5%-1%) and vanadium (1-2%) was noted only in the scales of the Cr-Al-Y steel.

## Mixture 3 (50% KCl, 50% Fe<sub>2</sub>O<sub>3</sub>)

The corrosion rates are rather high and almost equal for all three steels. It must be particularly pointed out that, with respect to the mixture (CI-S), the loss in thickness is almost equal for the Cr-Al-Y steel, whereas for the

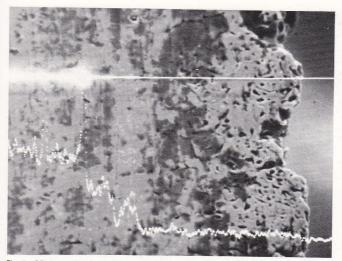
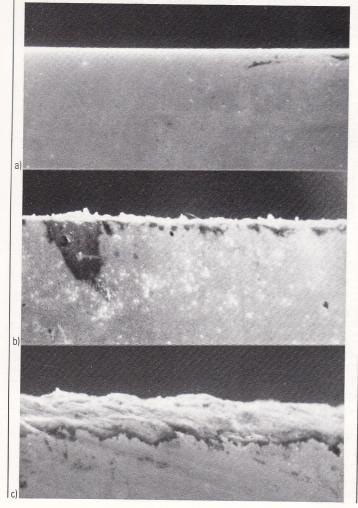


Fig. 6 - SEM morphology of attack and linear analysis of the chromium after testing in mixture (CI) of Fe-Cr-Al-Y alloy. x500.

Fig. 7 - SEM morphology of attack after testing in mixture (S): a) Fe-Cr-Al-Y; b) Fe-Cr-Ni-Mn; c) Fe-Mn-Al. x1000.



of the oxide. In mixture (CI-S), the Mn-AI steel showed the same morphology of attack, but in a less intense way.

## Mixture 4 (50% K<sub>2</sub>SO<sub>4</sub>, 50% Fe<sub>2</sub>O<sub>3</sub>)

Unlike the other mixtures, which are liquid at the testing temperature, (750°C), this is in a sticky state, which contributes to limit its aggressiveness. Of the alloys rich in chromium, the Cr-Al-Y steel is almost immune, while the Cr-Ni-Mn steel shows a light attack. The Mn-Al steel presents a compact oxide layer (16% Al, 30% Mn, 21% Fe) and a sub-layer with a greater alumina content (23% Al, 11% Mn, 55% Fe) (Fig. 7). The matrix seems devoid of manganese (10%) near the layer, while the aluminium content is almost the same as originally.

Fig. 8 - SEM morphology of attack after testing the Fe-Cr-AI-Y alloy in mixture (V). x500.

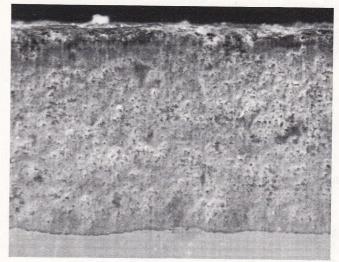
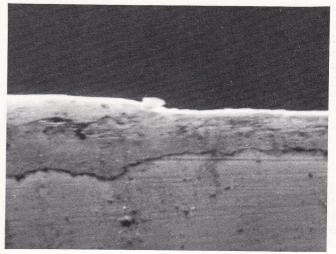


Fig. 9 - SEM morphology of attack after testing the Fe-Mn-Al alloy in mixture (V). x4500.



**Mixture 5** (50% V<sub>2</sub>O<sub>5</sub>, 50% Fe<sub>2</sub>O<sub>3</sub>)

In this mixture, all three steels form an adherent compact layer, as the micrograph in Figs. 8 and 9 show. The Cr-Al-Y steel shows a thicker oxidized layer with a spinel base, resulting from the interaction of chromium oxide and iron oxide in the mixture. The vanadium content remains the same throughout the oxide thickness (2-3%). The Cr-Ni-Mn steel shows a thinner layer than Cr-Al-Y steel, rich in  $Cr_2O_3$  on the inside, with Mn oxides and vanadates on the outside. Mn-Al steel undergoes the least attack; in the layer a high, uniform content of vanadium, equal to 29%, was confirmed.

## Discussion

In basic studies of corrosion in mixed gaseous systems, we often use diagrams of thermodynamic stability of the condensed phases, of the Ellingham-Pourbaix type, calculated for each component at a given temperature. In sulphurous and/or oxidizing atmospheres, we are limited to M-O-S diagrams (Fig. 10) derived from the thermodynamic equations:

> $(a_{O})_{eq} = p_{O_2}^{1/2} = exp (-\Delta G_{MO}^{O}/RT)$  $(a_{S})_{eq} = p_{S_2}^{1/2} = exp (-\Delta G_{MS}^{O}/RT)$

were M is the bivalent metal,  $a_0$  and  $a_s$  are the activities of oxygen and sulphur respectively, in equilibrium.

Such diagrams cannot be used to predict the products of reaction. Indeed, except for the approximation at 1 for the activity of metals and of oxides and/or sulphides, the diffusive phenomena create, locally in the zones of phase growth, environmental conditions which are different from those of the gaseous mixture. In other

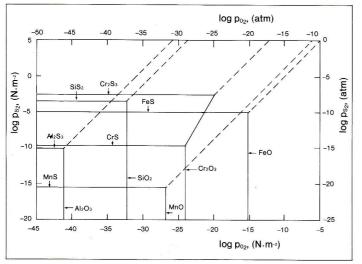


Fig. 10 - Diagram of the thermodynamic stability of some oxides and sulphides at  $750^{\circ}\text{C}.$ 

words, an M-O-S diagram can only indicate if a condensed phase of a specific metal can be in equilibrium with a gas of given composition. Considering a generic reaction,

$$MS + 1/2 O_2 = MO + 1/2 S_2$$

the diagram can indicate if it is likely to proceed to the left (when  $(a_S/a_O)$  gas >  $(a_S/a_O)$  eq), or to the right (when  $(a_S/a_O)$  gas <  $(a_S/a_O)$  eq).

Under the test conditions of the present work, oxygen activity in the molten salt mixtures is such as to fall within the field of oxide stability for all metals. However, transport phenomena of metal cations and S<sup>=</sup> ions across the oxide layer can distort from a kinetic point of view every thermodynamic forecasting. For the case investigated, selective oxidation in air would lead to the formation of an Al<sub>2</sub>O<sub>3</sub> film for the Cr-Al-Y steel, of a Cr<sub>2</sub>O<sub>3</sub> for the Cr-Ni-Mn steel, while for the Mn-Al steel, at the testing temperature, the oxide would be a mixture of manganese and iron oxide. Chromium oxide grows at the scale-gas interface, controlled by Cr<sup>+++</sup> ion diffusion, more selective the higher the concentration of metal in the alloy. However, the dissolution of sulphur in the chromium oxide increases the rate of cation migration towards the exterior (11). In increasing order of diffusivity, Ni, Fe and Mn can also migrate, infirming the protectiveness of the scale. Wild (12) has studied Mn migration through chromium oxide films formed on stainless steels: the element diffuses at a rate two orders of magnitude greater than that of chromium. A certain incompatibility between the two elements in the alloy can be seen in sulphurous atmospheres (13). Basing on these considerations, it can be understood why Cr-Ni-Mn steel undergoes a light attack in S mixture, unlike the Cr-Al-Y steel in which aluminium contributes greatly to protection; in the (V-S) mixture, the behaviour of the two steels containing chromium is the same. The presence of chlorides, as in mixtures (CI-S) and (CI), is dangerous for steels containing chromium, for which the attack is localized in intergranular or interdendritic paths at the expense of this element, which is lost as volatile CrCl<sub>3</sub>. The presence of V<sub>2</sub>O<sub>5</sub> seems to inhibit the sulphidizing action in the Cr-Al-Y steel and also, despite the presence of nickel, in the Cr-Ni-Mn steel in the (V-S) mixture; indeed, the pentoxide, because of its acidic character, explicates a negative effect on the solubility of SO3 in the mixture. Moreover, inside the oxide scales, an oxygen-supply function can be recognized to  $V_5O_5$  with consequent ability of increasing oxygen activity in the scale. This mechanism, on the one hand, has little influence on the two alloys containing chromium, but appears responsible for the higher rate attack which the Mn-Al steel undergoes in the (V-S) mixture with respect to (S). Note that the formation of manganese sulphide at the metal-scale interface,

(where thermodynamic conditions of low  $P_{O_2}$  and high manganese activity exist) is followed in the outer oxide layers by the reaction

### $MnS + O^{=} = MnO + S^{=}$

which results in sulphur recycling and consequent attack of the matrix. The pentoxide by itself (mixture 5) brings the attack towards a configuration analogous to that in air oxidation.

The behaviour of the Mn-Al steel demonstrates that it is not so much the presence of  $V_2O_5$  to be harmful in itself, but the combustion of sulphur and pentoxide.

## Conclusions

1. In alloys whose resistance to sulphurous media is based on the formation of layers of chromium oxide, or still better aluminium oxide, the resistance is lowered by the presence of chlorides, although the presence of vanadium is not noticeably compromising. However, the presence of  $V_2O_5$  introduces an aggravating element in the presence of ferrous oxide in the salt mixture, owing to the capacity of the latter compound to form spinels with chromium or aluminium oxides.

2. Alloys whose resistance in sulphurous media is based on the sacrificial action of manganese, which has a high affinity for sulphur, give promising results even in the presence of chlorides, but not in the presence of vanadium.

The above makes it clear that the expectation of alloys resisting a wide spectrum of hot corrosion in various media is excessively ambitious. This, in fact, involves besides the need for a high number of highly priced alloying elements also the risk of incurring rough errors in application. It seems more appropriate to study for each individual environment the alloy which gives the greatest guarantee of corrosion resistance. It can be foreseen rather that new formulation steels (Fe-Mn-Al) can be equivalent or positively superior, in specific media, with respect to more expensive steels.

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