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High-Temperature Gaseous Corrosion

Revised by Russell D. Kane, InterCorr International, Inc.

Introduction

WHEN METAL IS EXPOSED TO AN OXIDIZING GAS at elevated temperature, corrosion can occur by direct reaction with the gas, without the need for the presence of a liquid electrolyte. This type of corrosion is referred to as tarnishing, high-temperature oxidation, scaling, or gaseous corrosion. As shown in Fig. 1 (Ref 1), the rate of attack (in terms of weight gain) increases substantially with temperature for many steels and stainless steel alloys. The surface film typically thickens as a result of reaction at the scale/gas or metal/scale interface due to cation or anion transport through the scale, which behaves as a solid electrolyte. For continuous, nonporous scales, ionic transport through the scale is the rate-controlling process. The thermodynamic stability, the ionic defect structure, and certain morphological features of the scale formed are key factors in determining the resistance of an alloy to a specific environment.

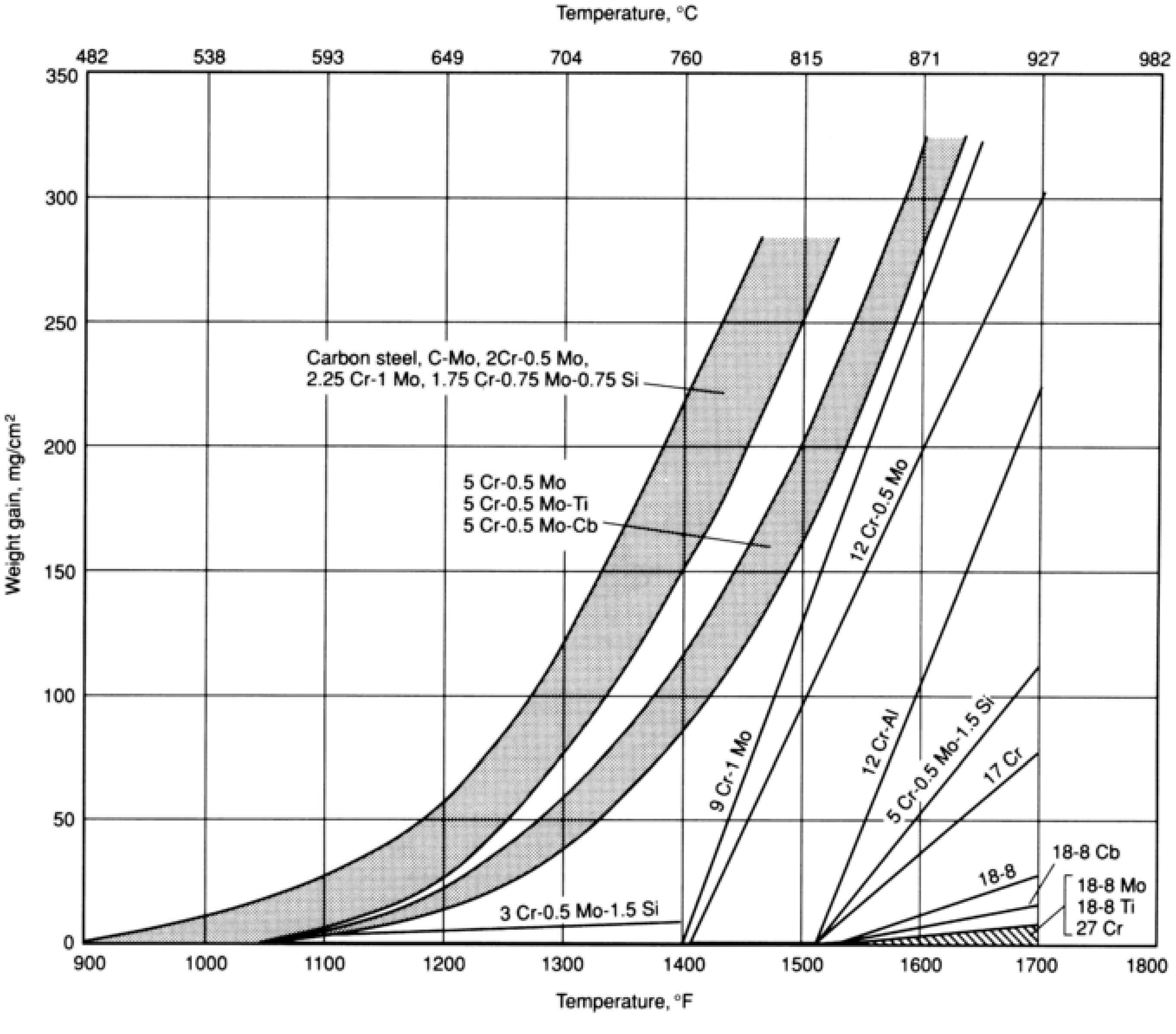

Fig. 1 Oxidation resistance of carbon, low-alloy and stainless steels in air after 100 h at temperatures from 590 to 930 °C (1100 to 1700 °F). Source: Ref 1

Initial film growth is usually very rapid. If the scale is a nonporous solid and completely covers the metal surface, the reaction rate will decrease when the thickness reaches a few thousand angstroms as the transport of reactive species through the film becomes rate controlling. The subsequent corrosion rate depends on the details of this transport mechanism, which may be due to electrical potential or concentration gradients or to migration along preferential paths, and so may correspond to any of several rate laws, as shown in Fig. 2. Where a diffusion process is rate controlling, the kinetics usually follow a parabolic rate law, in which the rate progressively decreases with time (see Fig. 3) (Ref 2). Figure 4(a) illustrates the compact, continuous protective scale of essentially chromium oxide (Cr_2O_3) formed on alloy 800. If the scale is porous (or is formed as a vapor species) or does not completely cover the metal surface, a linear rate is usually experienced.

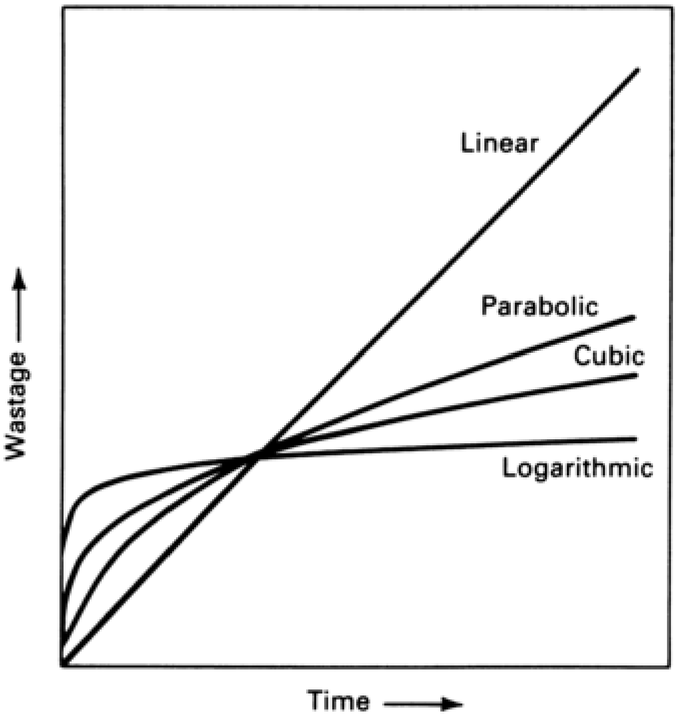

Fig. 2 Forms of kinetic curves that represent various thermal degradation processes

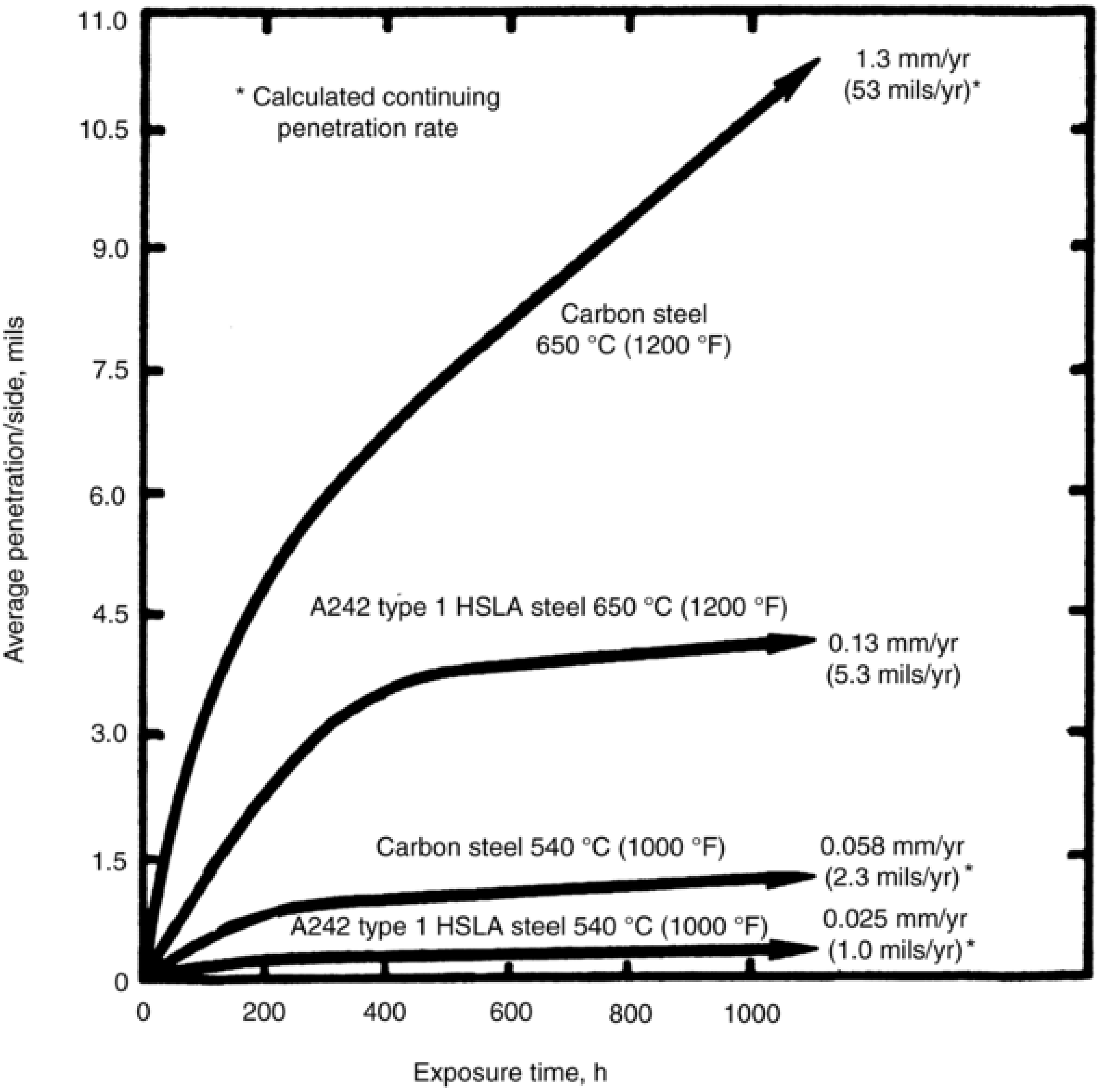

Fig. 3 Oxidation of carbon steel and high-strength low-alloy (HSLA) steel in air. Source: Ref 2

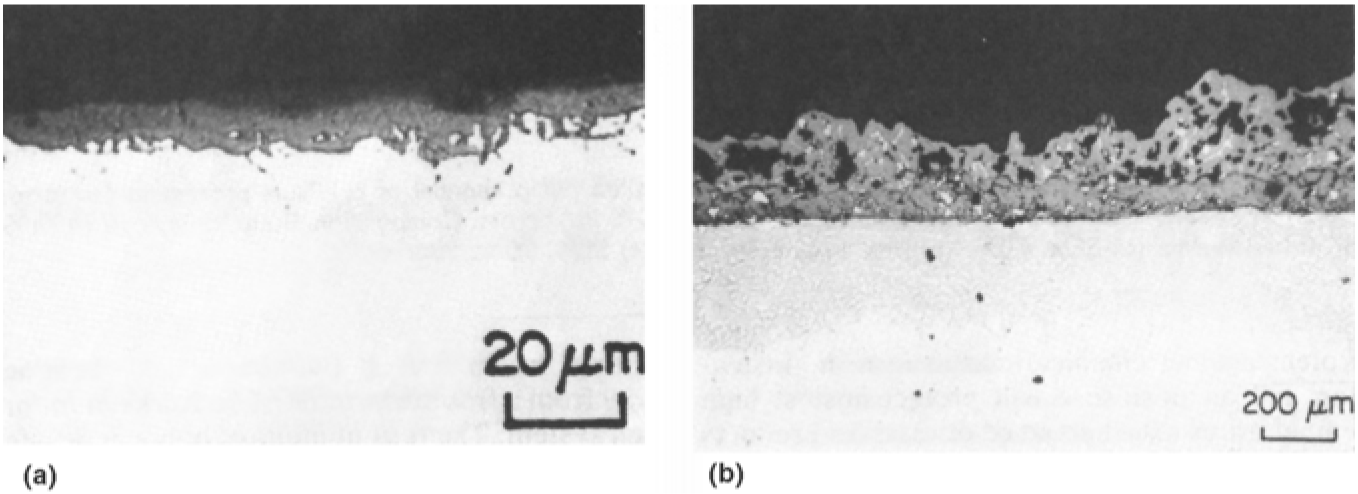

Fig. 4 Protective and nonprotective scales formed on alloy 800. (a) Cr_2O_3-base protective oxide scale formed in sulfur-free oxidizing gas. (b) Sulfide-oxide scale formed in reducing conditions containing hydrogen sulfide. Courtesy of I.G. Wright, Battelle Columbus Division

The latter circumstance can be assessed from the Pilling-Bedworth ratio, which is the ratio of the volumes of oxide produced to that of the metal consumed by oxidation; values of 1.0 or greater result in complete surface coverage by oxide and, usually, protective behavior. This is not a complete or foolproof measure for assessing the likelihood of protective scaling behavior. At high temperatures, the growth of nominally protective oxides may be sufficiently rapid that the compressive stresses resulting from a Pilling- Bedworth ratio greater than 1 become sufficiently great that the scale (or alloy) deforms, resulting in spalling that acts as a stress-relief mechanism; in some cases, the protection offered by such scales may be low at this point, as shown in Fig. 5.

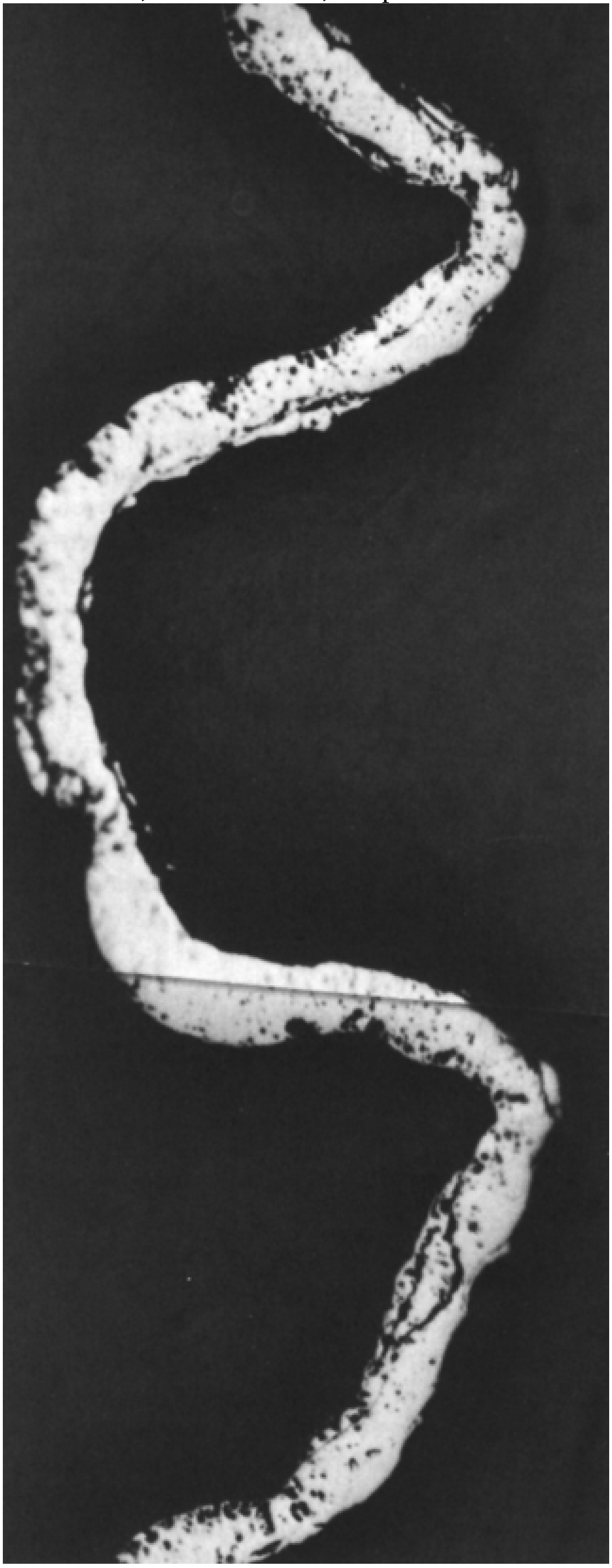

Fig. 5 Cr_2O_3 scale formed on pure chromium at 1100 °C (2012 °F). A Pilling-Bedworth ratio of 2.0 results in high compressive stress in the scale, which is relieved by buckling and spalling. Courtesy of I.G. Wright, Battelle Columbus Division

The desired characteristics for a protective oxide scale include:

- High thermodynamic stability (highly negative Gibbs free energy of formation) so that it forms preferentially to other reaction products possible under the exposure conditions
- Low vapor pressure so that the oxide forms as a solid and does not evaporate into the atmosphere
- Pilling-Bedworth ratio greater than 1.0 so that the oxide completely covers the metal surface
- Low coefficient of diffusion of reactant species (metal cations and corrodent anions) so that the scale has a slow growth rate
- High melting temperature
- Good adherence to the metal substrate, which usually involves a coefficient of thermal expansion close to that of the metal, and sufficient high-temperature plasticity to resist fracture from differential thermal expansion stresses that can result particularly from thermal cycling

High-temperature scales are usually thought of as oxides, but may also be sulfides, possibly carbides, or mixtures of these species. Oxides and sulfides are nonstoichiometric compounds and semiconductors.

There are essentially two types of semiconductors: *p*-type (or positive carrier)—which may have vacancies in its metal lattice or an excess of anions contained interstitially—and *n*-type (or negative carrier)—which may have an excess of metal ions contained interstitially or vacant anion lattice sites. For diffusion-controlled scaling, the rate of scale growth can be altered by modification of the concentration of the particular defects involved. For example, *p*-type oxides exhibit increased cationic transport rates (increased oxidation rates) at increased oxygen pressures, while transport in *n*-type oxides is essentially independent of oxygen pressure. Both types of oxide can be doped by the addition of specific ions to the oxide lattice. For *p*-type metal deficit oxides, for example, the addition of cations of higher valence than the native cations results in an increase in the number of cation vacancies and therefore an increase in the oxidation rate, while lower-valence cation additions have the opposite effect.

Sulfides typically exhibit an intrinsically greater rate of transport of anions and cations than the oxides of the same metal and so provide scales that are significantly less protective than oxides.

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1. H.E. McGarrow, Ed., *Making, Shaping and Treating Steel,* United States Steel Corp., 1971. p 1136

2. R.T. Jones, in *Process Industry Corrosion,* National Association of Corrosion Engineers, 1986, p 373

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High-Temperature Oxidation

Alloys intended for high-temperature applications are designed to have the capability of forming protective oxide scales. Alternatively, where the alloy has ultrahigh-temperature- strength capabilities (which is usually synonymous with reduced levels of protective scale- forming elements), it must be protected by a specially

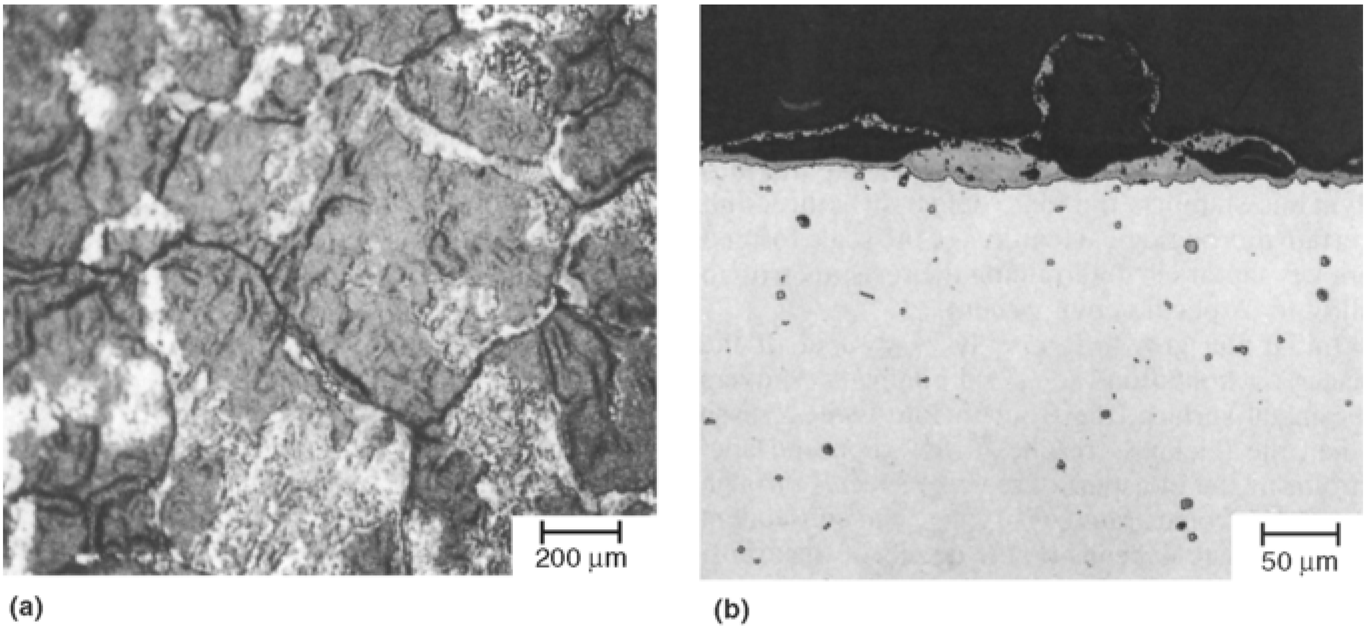

Fig. 8 Topography (a) and cross section (b) of oxide scale formed on Fe-18Cr alloy at 1100 °C (2012 °F). The bright areas on the alloy surface (a) are areas from which scale has spalled. The buckled scale and locally thickened areas (b) are iron-rich oxide. The thin scale layer adjacent to the alloy is Cr_2O_3, which controls the oxidation rate. Courtesy of I.G. Wright, Battelle Columbus Division

Because these protective oxide scales will form wherever the alloy surface is exposed to the ambient environment, they will form at all surface discontinuities; therefore, the possibility exists that notches of oxide will form at occluded angles in the surface, which may eventually serve to initiate or propagate cracks under thermal cycling conditions. The ramifications of stress-assisted oxidation (and of oxidation assisting the applied stress) in the production of failure conditions are not very well understood, but can constitute important considerations in predicting corrosion performance in actual operating systems and be of benefit in practical failure analysis.

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Sulfidation

When the sulfur activity (partial pressure, concentration) of the gaseous environment is sufficiently high, sulfide phases, instead of oxide phases, can be formed. The mechanisms of sulfide formation in gaseous environments and beneath molten-salt deposits have been determined in recent years. In the majority of environments encountered in practice by oxidation-resistant alloys, Al_2O_3 or Cr_2O_3 should form in preference to any sulfides, and destructive sulfidation attack occurs mainly at sites where the protective oxide has broken down. The role of sulfur, once it has entered the alloy, appears to be to tie up the chromium and aluminum as sulfides, effectively redistributing the protective scale-forming elements near the alloy surface and thus interfering with the process of formation or re-formation of the protective scale. If sufficient sulfur enters the alloy so that all immediately available chromium or aluminum is converted to sulfides, then the less stable sulfides of the base metal may form because of morphological and kinetic reasons. It is these base metal sulfides that are often responsible for the observed accelerated attack, because they grow much faster than the designed coating. Oxides that effectively meet the criteria for protective scales listed previously and can be formed on practical alloys are limited to Cr_2O_3, alumina (Al_2O_3), and possibly silicon dioxide (SiO_2). In the pure state, Al_2O_3 exhibits the slowest transport rates for metal and oxygen ions and so should provide the best oxidation resistance.

Alloying requirements for the production of specific oxide scales have been translated into minimum levels of the scale-forming elements, or combinations of elements, depending on the base alloy composition and the intended service temperature. Figure 6 represents the oxidation rate of iron-chromium alloys (1000 °C, or 1832 °F, in 0.13 atm oxygen) and depicts the types of oxide scale associated with various alloy types in this range of chromium content. Figure 7 illustrates the morphology of a semiprotective scale formed on a cobalt-chromium alloy. Alloys based on these minimum specifications will form the desired protective oxide upon initial exposure, but because of the accompanying depletion of the scale-forming element, they will probably be unable to re-form the protective layer in the event of loss or failure of the initial scale.

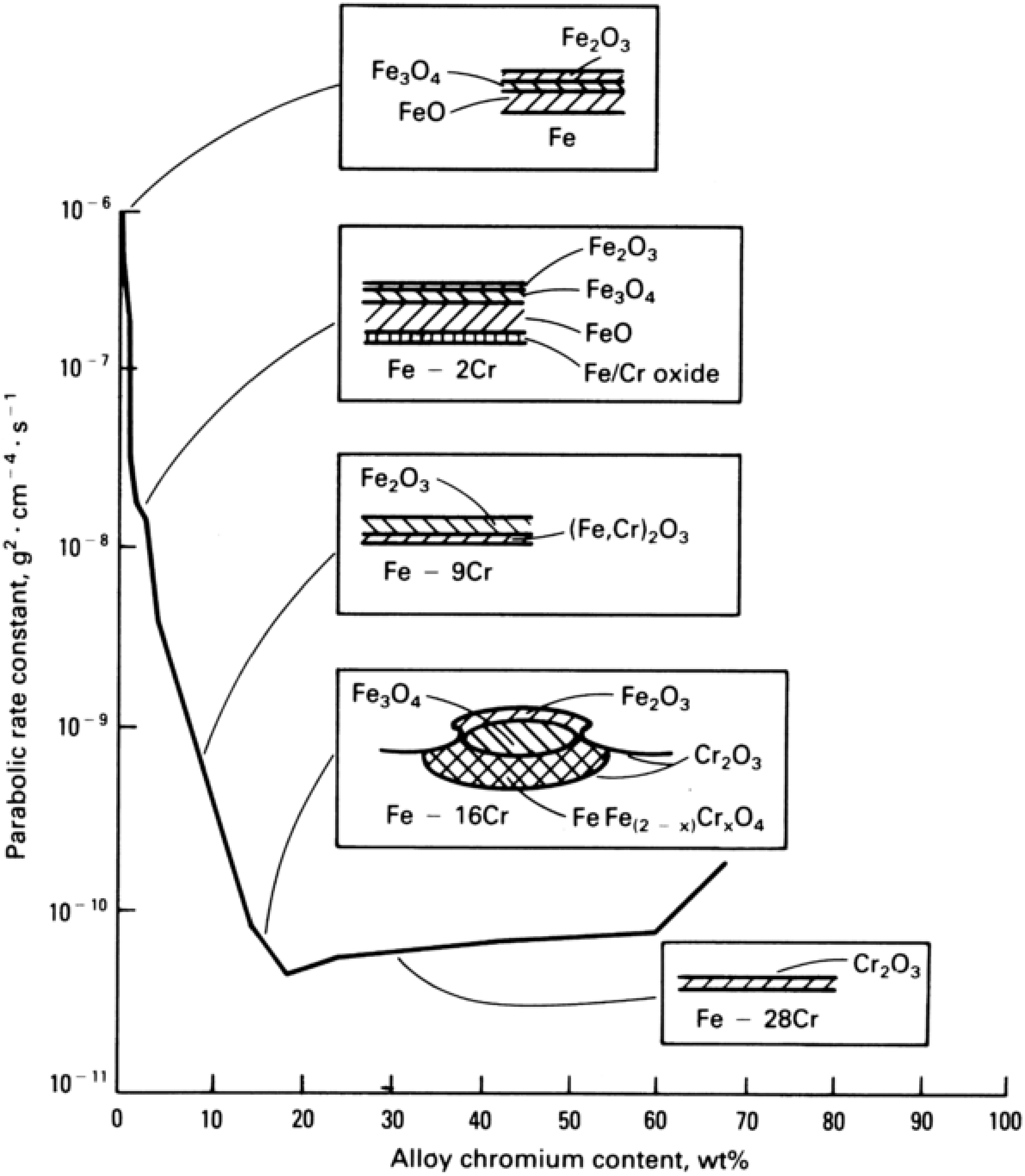


Fig. 6 Variation of the oxidation rate and oxide scale structure with alloy chromium content (based on isothermal studies at 1000 °C, or 1832 °F, in 0.13 atm oxygen)

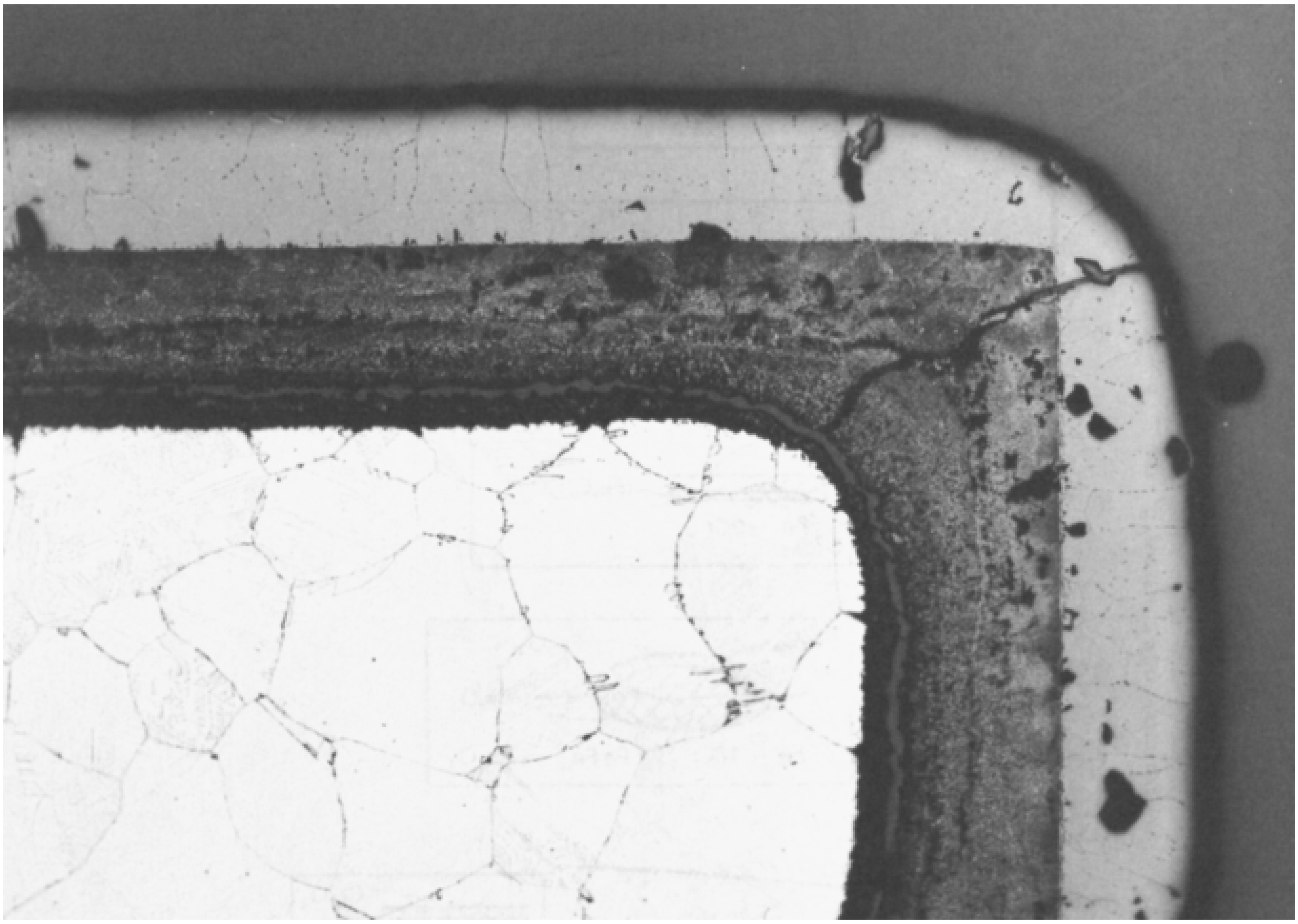


Fig. 7 Multilayer oxide scale formed on Co-10Cr alloy at 1100 °C (2012 °F). Outer layer is CoO; inner (mottled gray) layer is CoO containing dissolved chromium and particles of Co-Cr spinel. The chromium level in this alloy is insufficient to form a fully protective Cr_2O_3 scale. Courtesy of I.G. Wright, Battelle Columbus Division

A useful concept in assessing the potential high-temperature oxidation behavior of an alloy is that of the reservoir of scale-forming element contained by the alloy in excess of the minimum level (around 20 wt% for iron-chromium alloy at 1000 °C, or 1832 °F, according to Fig. 6). The more likely the service conditions are to cause repeated loss of the protective oxide scale through spalling, erosion, wear, or mechanical stresses, the greater the reservoir of scale-forming element required in the alloy for continued protection. Extreme cases of this concept result in chromizing or aluminizing to enrich the surface regions of the alloy or in the provision of an external coating rich in the scale-forming elements.

The breakdown of protective scales based on Cr_2O_3 or Al_2O_3 appears, in the majority of cases, to originate through mechanical means. The most common is spallation as a result of thermal cycling, or loss through erosion or abrasion. Typical scale structures on an Fe-18Cr alloy after thermal cycling are shown in Fig. 8. Cases in which the scales have been destroyed chemically are usually related to reactions occurring beneath deposits, especially where these consist of molten species. An additional mode of degradation of protective Cr_2O_3 scale is through oxidation to the volatile chromium trioxide (CrO_3), which becomes prevalent above about 1010 °C (1850 °F) and is greatly accelerated by high gas flow rates.

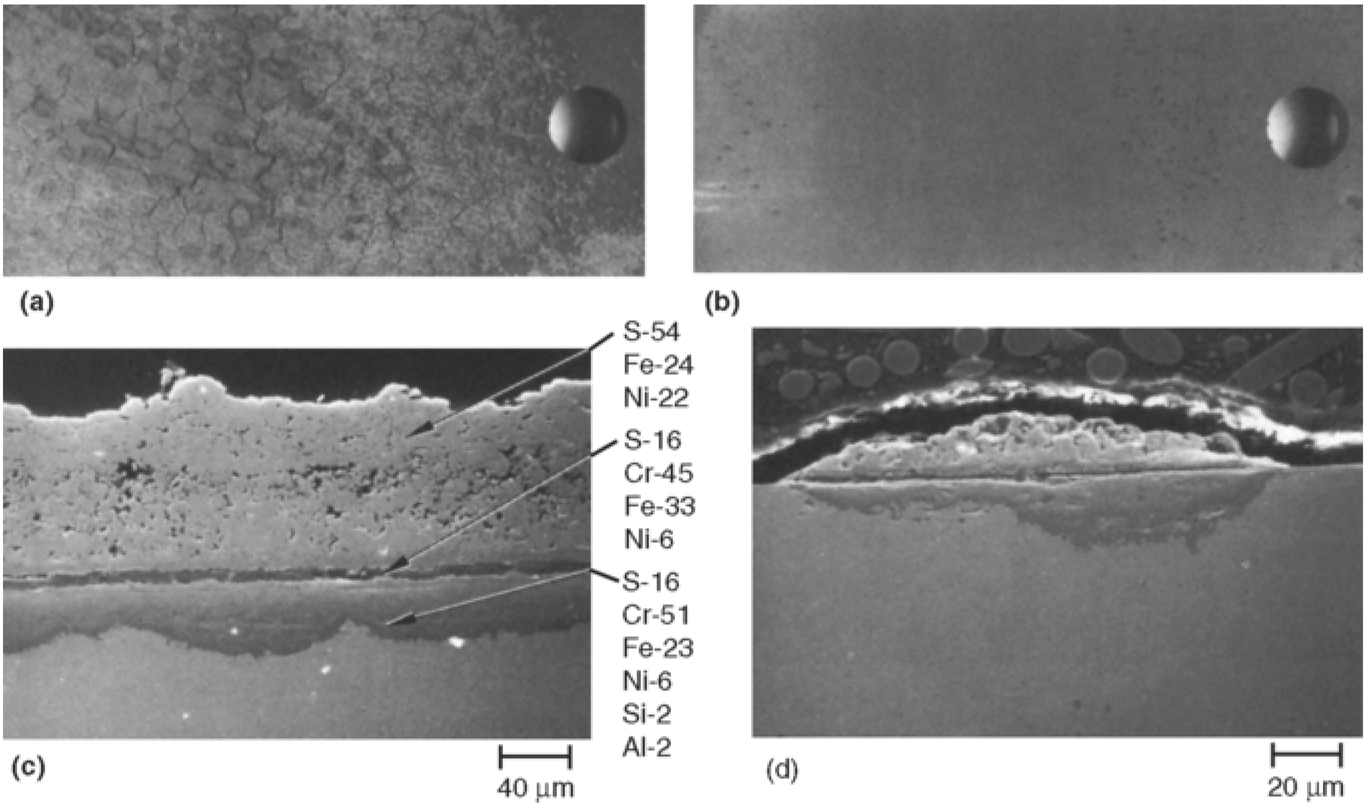
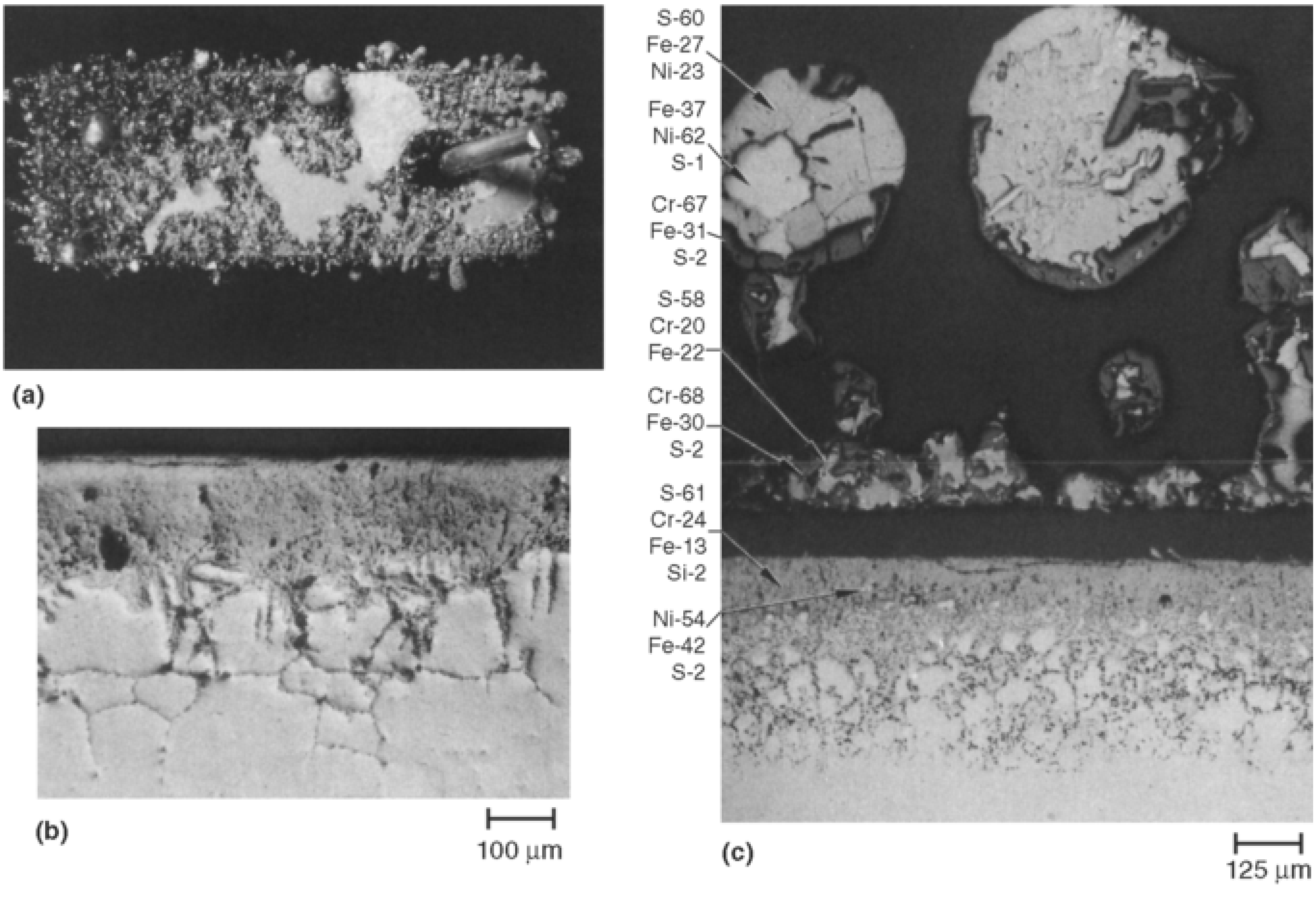


Fig. 10 Alloy 800 test coupons with a 0.254 mm (0.01 in.) diam grain size exposed to a coal-gasifier environment for 100 h. (a) and (c) Tested at 650 °C (1200 °F) and oxygen and sulfur partial pressures of 3 × 10^{-24} atm and 1 × 10^{-8} atm, respectively. (b) and (d) Tested at 650 °C (1200 °F) and p_{O_2}= 3 × 10^{-24} atm and p_{S_2}= 1 × 10^{-9} atm. Scanning electron micrographs show sulfide scale (c) and an external sulfide formation (d). (a) and (b) ~2×. Courtesy of G.R. Smolik and D.V. Miley, E.G. & G. Idaho, Inc.



oxides or sulfides of chromium or aluminum; in addition, they have relatively low melting points, so that molten slag phases are often possible. Figure 4 compares a protective (oxide) scale and a nonprotective (sulfide) scale formed on alloy 800. It is precisely this effect that provides the requirement for materials with higher alloy content of oxide-forming elements to impart increased sulfidation resistance.

Sulfur can transport across continuous protective scales of Al_2O_3 and Cr_2O_3 under certain conditions, with the result that discrete sulfide precipitates can be observed immediately beneath the scales on alloys that are behaving in a protective manner. For reasons indicated previously, as long as the amount of sulfur present as sulfides is small, there is little danger of accelerated attack. However, if oxidizing conditions exist, once sulfides have formed in the alloy, there is a tendency for the sulfide phases to be preferentially oxidized by the encroaching reaction front and for the sulfur to be displaced inward, forming new sulfides deeper in the alloy, often in grain boundaries or at the sites of other chromium- or aluminum-rich phases, such as carbides. In this way, fingerlike protrusions of oxide/sulfide can be formed from the alloy surface inward, which may act to localize stress or otherwise reduce the load-bearing section. Such attack of an austenitic stainless steel experienced in a coal-gasifier product gas is shown in Fig. 9. The sulfidation behavior of alloy 800 at temperatures and oxygen and sulfur potentials representative of coal-gasification processes is illustrated in Fig. 10, Fig. 11, Fig. 12. Additionally, as the oxygen and sulfur activities may vary in service, the tendencies for sulfidation and oxidation to persist or coexist change as well. Figure 13 (Ref 3) shows a stability diagram for the Cr-S-O system. This indicates how the dominant corrosion mechanism can change from oxidation or sulfidation depending on the availability of sulfur and oxygen. This figure also indicates the predominance of oxidation under all but the highly reducing conditions provided by very low oxygen partial pressure.

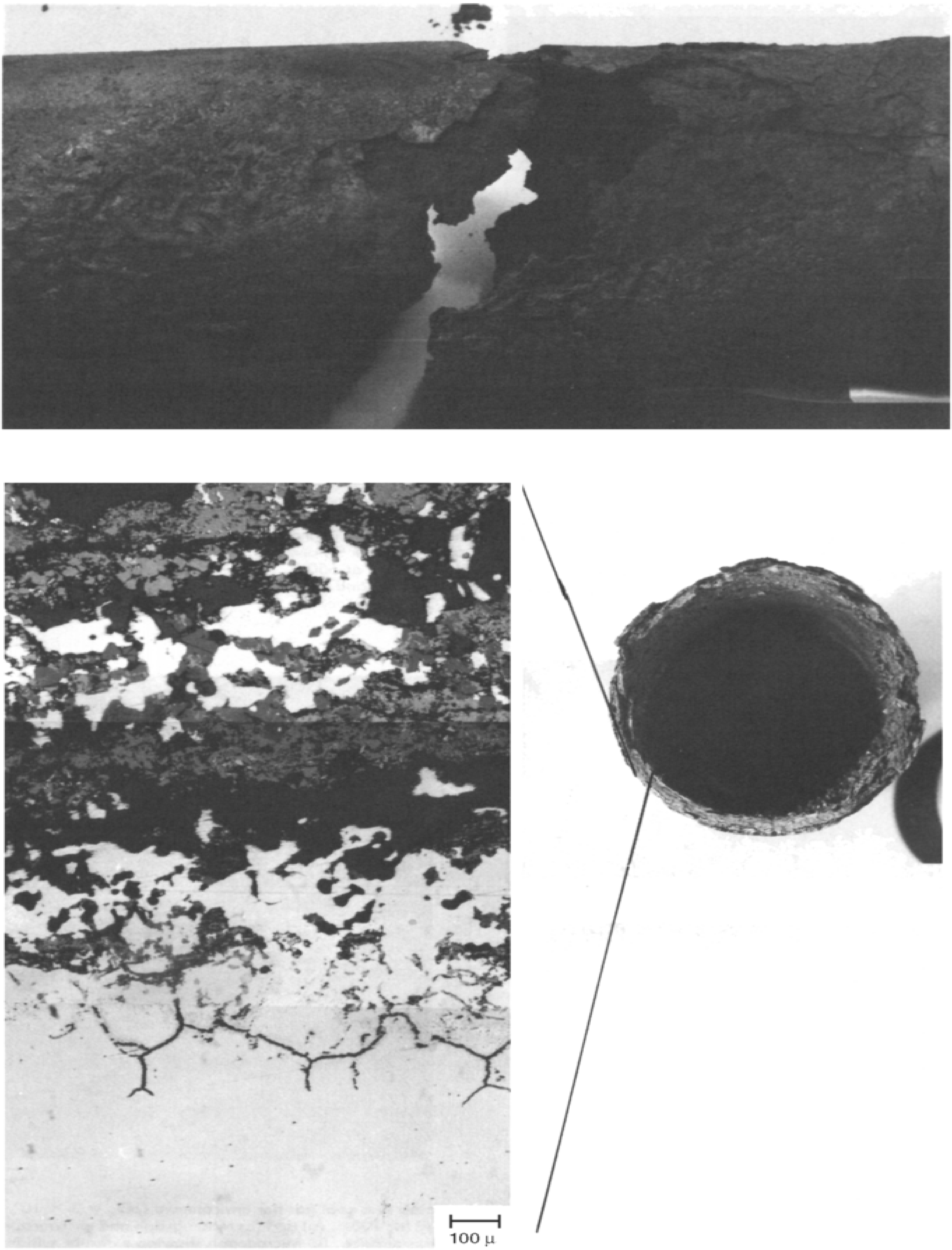


Fig. 9 Example of high-temperature sulfidation attack in a type 310 heat-exchanger tube after ~100 h at 705°C (1300 °F) in coal-gasifier product gas

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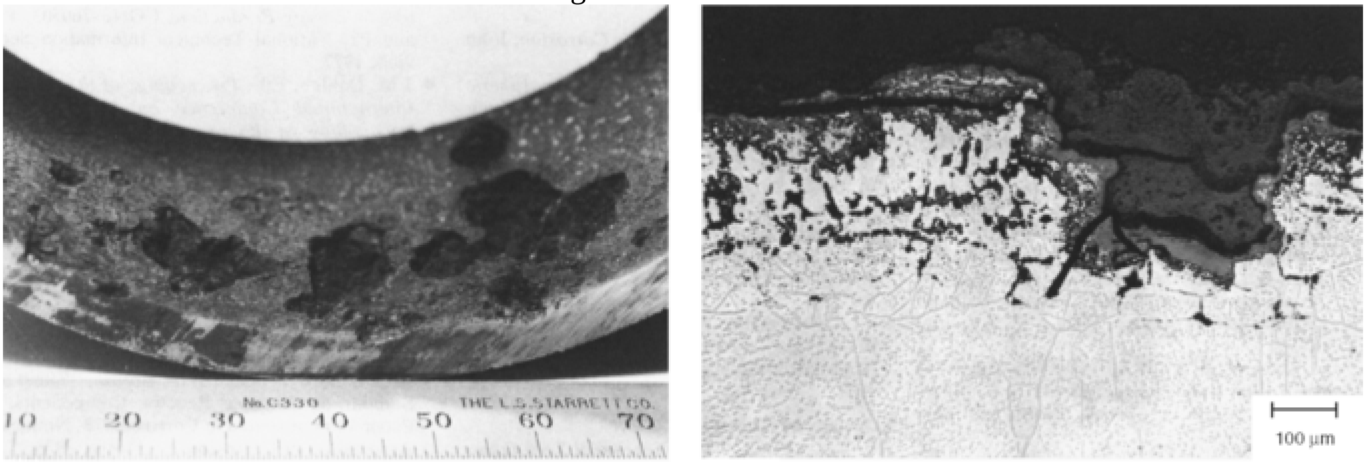
Carburization

As in the case of sulfide penetration, carburization of high-temperature alloys is thermodynamically unlikely except at very low oxygen partial pressures, because the protective oxides of chromium and aluminum are generally more likely to form than the carbides. However, carburization can occur kinetically in many carbon-containing environments. Carbon transport across continuous nonporous scales of Al_2O_3 or Cr_2O_3 is very slow, and alloy pretreatments likely to promote such scales, such as initially smooth surfaces or preoxidation, have generally been found to be effective in decreasing carburization attack. In practice, the scales formed on high-temperature alloys often consist of multiple layers of oxides resulting from localized bursts of oxide formation in areas where the original scale was broken or lost. The protection is derived from the innermost layer, which is usually richest in chromium or aluminum. Concentration of gaseous species such as carbon monoxide in the outer porous oxide layers appears to be one means by which sufficiently high-carbon activities can be generated at the alloy surface for carburization to occur in otherwise oxidizing environments. The creation of localized nonoxidizing, microenvironments is also possible under surface deposits that create stagnant conditions not permeable by the ambient gas, resulting in local regions of low oxygen activities where carburization can persist.

Once inside the alloy, the detrimental effects of the carbon depend on the location, composition, and morphology of the carbide formed. Austenitic steels should carburize more readily than ferritic steels because of the high solubility of carbon in austenite. Iron-chromium alloys containing less than about 13% Cr contain various amounts of austenite, depending on temperature, and should be susceptible to carburization, while alloys with 13 to 20% Cr will form austenite as a result of absorption of small amounts of carbon. Iron-chromium alloys containing more than ~20% Cr can absorb considerable amounts of carbon before austenite forms, becoming principally $(CrFe)_{23}C_6$ and ferrite. An example of rapid high-temperature carburization attack of an austenitic stainless steel is shown in Fig. 14.



(a) (b)

Fig. 14 Example of high-temperature carburization attack pitting in type 310 reactor wall after ~4000 h exposure to coal-gasification product gas. The pits were formed during operation under conditions of high-carbon activity in the gas. (a) Overall view of pitting. (b) Section through a pit. Courtesy of I.G. Wright, Battelle Columbus Division

Alloying elements can exert an influence on the susceptibility to carburization of various alloys. In particular, silicon, niobium, tungsten, titanium, and the rare earths have been noted as promoting resistance to

Fig. 11 Sulfidation attack of alloy 800 test coupons exposed to a coal-gasifier environment ($p_{O_2} = 3 \times 10^{-20}$ atm and $p_{S_2} = 1 \times 10^{-7}$ atm) at 870 °C (1600 °F) for 100 h. (a) and (b) Macrograph and micrograph, respectively, of a test coupon with a 0.254 mm (0.01 in.) diam grain size. (c) Micrograph showing external sulfides, sulfide scale, and intergranular sulfidation of a test coupon with a 0.022 to 0.032 mm (0.0008 to 0.0013 in.) diam grain size. (a) ~1.5×. Courtesy of G.R. Smolik and D.V. Miley, E.G. & G. Idaho, Inc.

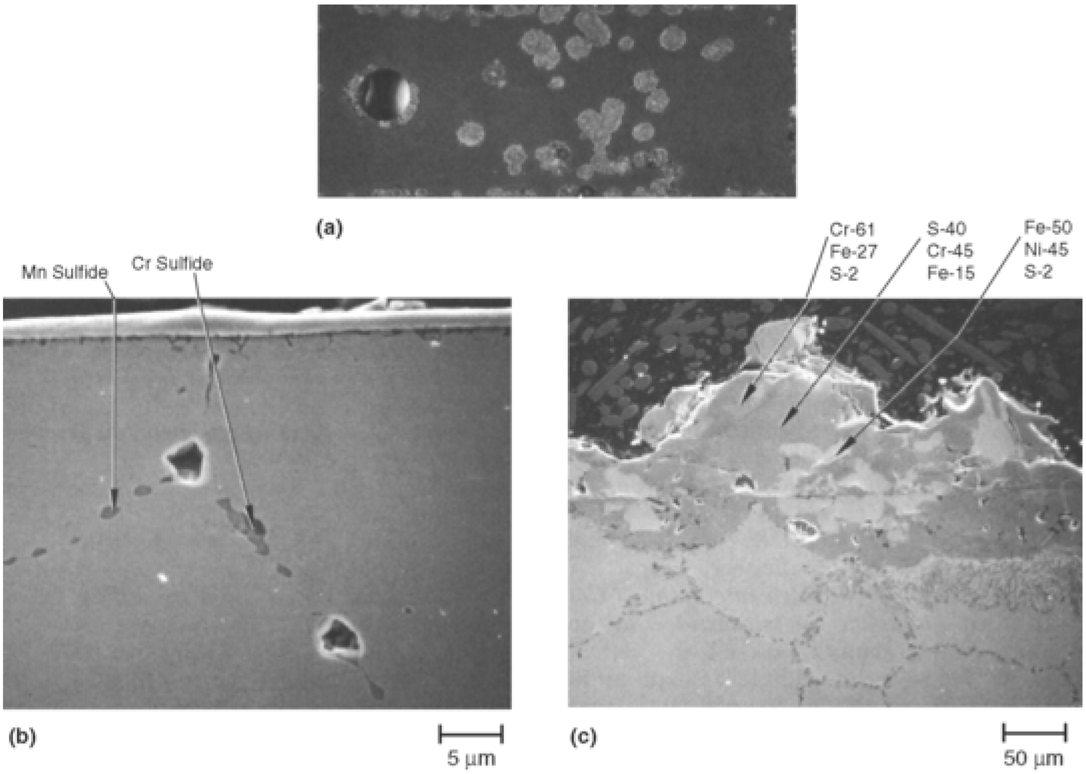


Fig. 12 Macrograph (a) of an alloy 800 test coupon with a 0.254 mm (0.01 in.) diam grain size exposed to a coal-gasifier environment ($p_{O_2} = 3 \times 10^{-19}$ atm and $p_{S_2} = 1 \times 10^{-7}$ atm) at 870 °C (1600 °F) for 100 h. ~1.5×. Micrographs (b) and (c) show cross sections through the Cr_2O_3 layer and disrupted oxide region having external sulfides. Courtesy of G.R. Smolik and D.V. Miley, E.G. & G. Idaho, Inc.

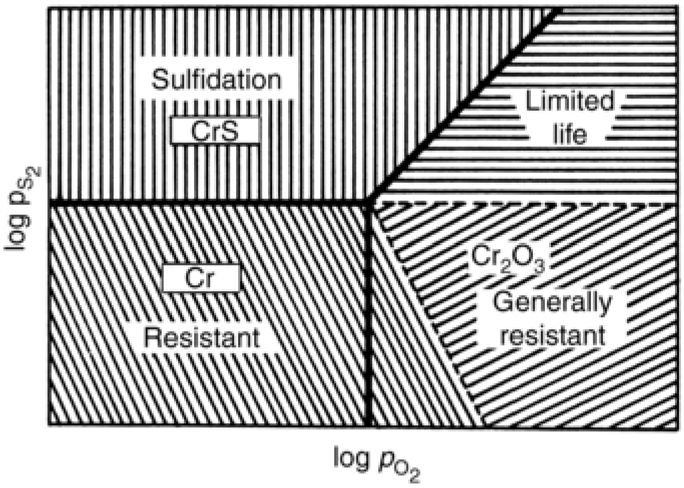


Fig. 13 Stability of the Cr-S-O system Source: Ref 3

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3. H.W. Eiselstein and E.N. Skinner, in *Effect of Cyclic Heating and Stressing on Metals at Elevated Temperature,* STP 165, American Society for Testing and Materials, 1954, p 162

susceptible to this form of attack due to the greater stability of chromium carbide versus Fe_3C found in carbon steels. However, as the partial pressure of hydrogen and/or the temperature of exposure increases, a greater amount of these alloying additions are required to prevent such attack. One example is 2.25Cr-1Mo, a steel that undergoes some decarburization in high- temperature high-pressure hydrogen, but is less likely to fissure than carbon steel. However, as the conditions increase in severity, higher-alloy steels such as 5Cr-0.5Mo or 9Cr-1Mo may be required. The susceptibility of steels to attack by hydrogen can be judged from the Nelson Curves, which indicate the regions of temperature and pressure in which a variety of steels will suffer attack.

Another alloy susceptible to hydrogen attack is copper containing small amounts of cuprous oxide. This oxide reacts to form steam within the alloy, resulting in significant void formation.

Hot corrosion generally refers to a form of accelerated attack experienced by the hot gas path components of gas turbine engines. Two forms of hot corrosion can be distinguished; most of the corrosion encountered in turbines burning liquid fuels can be described as type I hot corrosion, which occurs primarily in the metal temperature range of 850 to 950 °C (1550 to 1750 °F). This is a sulfidation-based attack on the hot gas path parts involving the formation of condensed salts, which are often molten at the turbine operating temperature. The major components of such salts are sodium sulfate (Na_2SO_4) and/or potassium sulfate (K_2SO_4), apparently formed in the combustion process from sulfur from the fuel and sodium from the fuel or the ingested air. Because potassium salts act very similarly to sodium salts, alkali specifications for fuel or air are usually taken to be the sum total of sodium plus potassium. An example of the corrosion morphology typical of type I hot corrosion is shown in Fig. 16.

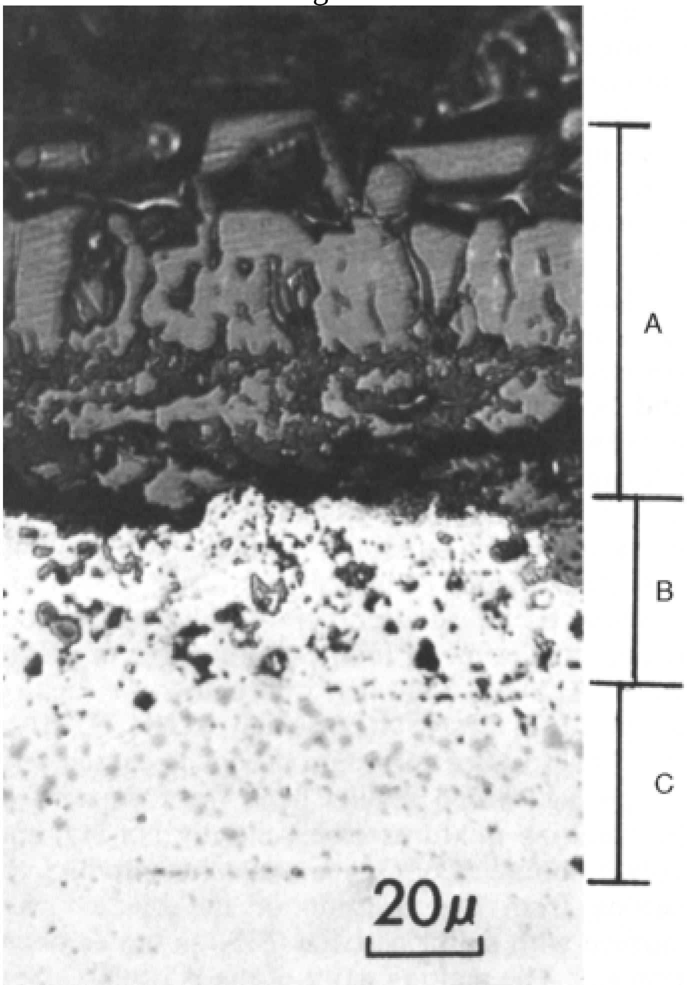

Fig. 16 Ni-20Cr-2ThO$_2$ after simulated type I hot-corrosion exposure (coated with Na$_2$SO$_4$ and oxidized in air at 1000 °C, or 1832 °F). A, nickel-rich scale; B, Cr$_2$O$_3$ subscale; C, chromium sulfides. Courtesy of I.G. Wright, Battelle Columbus Division

Very small amounts of sulfur and sodium or potassium in the fuel and air can produce sufficient Na_2SO_4 in the turbine to cause extensive corrosion problems because of the concentrating effect of turbine pressure ratio. For example, a threshold level has been suggested for sodium in air of 0.008 ppm by weight below which hot corrosion will not occur. Type I hot corrosion, therefore, is possible even when premium fuels are used. Other fuel (or air) impurities, such as vanadium, phosphorus, lead, and chlorides, may combine with Na_2SO_4 to form mixed salts having reduced melting temperature and thus broaden the range of conditions over which this form of attack can occur. This is one of the major problems in combustion of impure fuels and incineration of municipal wastes. Also, agents such as unburned carbon can promote deleterious interactions in the salt deposits.

Research in the 1970s and 1980s led to greater definition of the relationships among temperature, pressure, salt concentration, and salt vapor- liquid equilibria so that the location and rate of salt deposition in an engine can be predicted. Additionally, it has been demonstrated that a high chromium content is required in an alloy for good resistance to type I hot corrosion. The trend to lower chromium levels with increasing alloy strength has therefore rendered most superalloys inherently susceptible to this type of corrosion. The effects of other alloying additions, such as tungsten, molybdenum, and tantalum, have been documented, and their effects on rendering an alloy more or less susceptible to type I hot corrosion are known and mostly understood.

Although various attempts have been made to develop figures of merit to compare superalloys, these have not been universally accepted. Nonetheless, the near standardization of such alloys as alloy 738 and alloy 939 for first-stage blades/ buckets, and FSX-414 for first-stage vanes/nozzles, implies that these are the accepted best compromises between high-temperature strength and hot-corrosion resistance. It has also been possible to devise coatings with alloying levels adjusted to resist this form of hot corrosion. The use of such coatings is essential for the protection of most modern superalloys intended for duty as first-stage blades or buckets.

Type II, or low-temperature hot corrosion, occurs in the metal temperature range of 650 to 700 °C (1200 to 1300 °F), well below the melting temperature of Na_2SO_4, which is 884 °C (1623 °F). This form of corrosion produces characteristic pitting, which results from the formation of low-melting mixtures of essentially Na_2SO_4 and cobalt sulfate ($CoSO_4$), a corrosion product resulting from the reaction of the blade/bucket surface with sulfur trioxide (SO_3) in the combustion gas. The melting point of the Na_2SO_4-$CoSO_4$ eutectic is 540 °C (1004 °F). Unlike type I hot corrosion, a partial pressure of SO_3 in the gas is critical for the reactions to occur. Knowledge of the SO_3 partial pressure-temperature relationships inside a turbine allows some prediction of where type II hot corrosion can occur. Cobalt- free nickel-base alloys (and coatings) may be more resistant to type II hot corrosion than cobalt-base alloys; it has also been observed that resistance to type II hot corrosion increases with the chromium content of the alloy or coating. Results from corrosion tests over several commercial alloys over a range of temperatures using a mixed-ash composition of Na_2SO_4 and K_2SO_4 is shown in Fig. 17 (Ref 5). It is the role of alloy composition and service temperature to define conditions for acceptable corrosion resistance and alloy use.

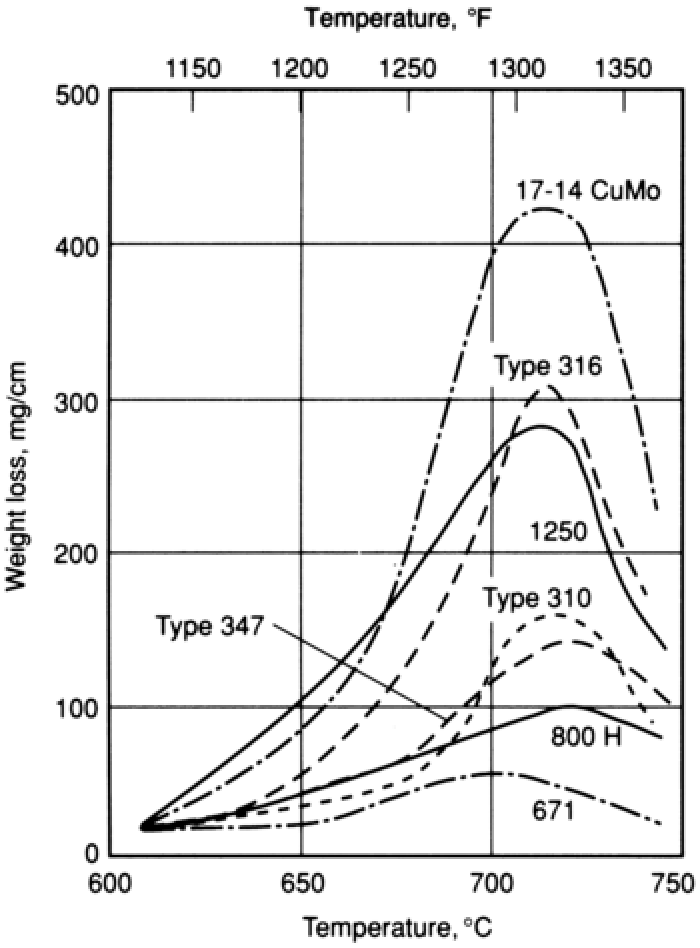

Fig. 17 Results of laboratory tests conducted in synthetic flue gas ($80N_2$-$15CO_2$-$4O_2$-$1SO_2$, saturated with H_2O) with synthetic ash (37.5 mol% Na_2SO_4, and 25 mol% Fe_2O_3) covering samples. Exposure was 50 h. Source: Ref 5

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carburization. The influence of silicon is particularly noteworthy as shown in Fig. 15 (Ref 4). It shows particular benefits in terms of reducing weight gain over the range of 1.5 to 2.5% in the bulk for Fe-Ni- Cr alloys of nominally 24 to 28% Cr and 20% Ni. Minor alloying additions of niobium in Ni- Cr-Fe alloys have been shown to reduce carburization rates by up to 25% over those without niobium additions. Experience with aluminum and manganese has been varied, although aluminum additions and diffusion coatings have shown particular promise in being able to set up a barrier to carbon ingress, thereby increasing resistance to carburization. Studies of alloy ratio of nickel and chromium have shown that alloys based on 50:50 nickel-to-chromium composition have generally better resistance than alloys where this ratio is 0.5 or 0.25. However, the presence of lead, molybdenum, cobalt, zirconium, and boron are considered detrimental to carburization resistance.

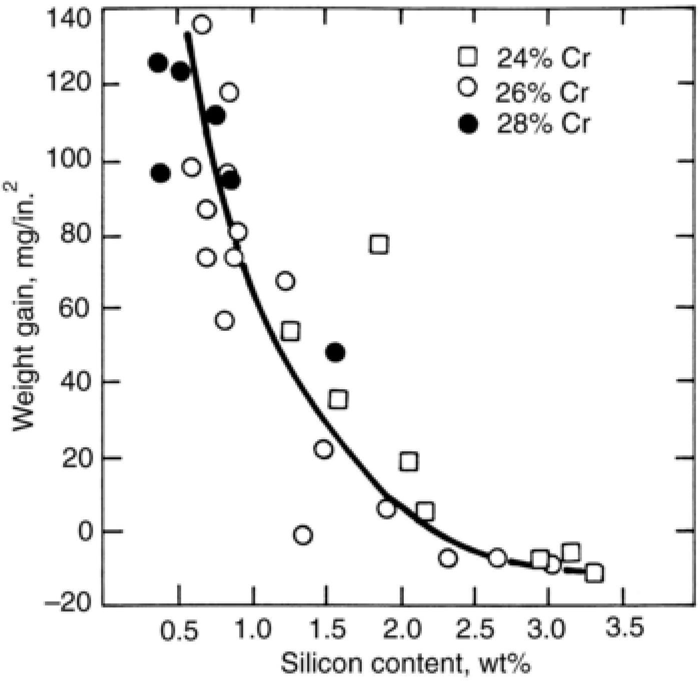

Fig. 15 Effect of silicon on the carburization resistance of cast Fe-20Ni-Cr alloys tested at 1090 °C (2000 °F) for 24 h in wet ethane Source: Ref 4

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Other Forms of High-Temperature Corrosion

Hydrogen Effects. In hydrogen environments at elevated temperatures and pressures, there is increasing availability of atomic hydrogen that can easily penetrate metal structures and react internally with reducible species. An example is the attack experienced by carbon and low-alloy steels, in which atomic hydrogen reacts with iron carbide to form methane, which then leads to loss of strength from decarburization, formation of voids, and fissuring of the steel. Alloy steels with stable carbides, such as chromium carbides, are less

R.D. Kane, High-Temperature Gaseous Corrosion, *Corrosion: Fundamentals, Testing, and Protection*, Vol 13A, *ASM Handbook*, ASM International, 2003, p 228–235

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