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Reduction Of Coking
With Minimox® Self-Protective Alloy Treatment

Summary

Surfaces treated with Minimox® Self-Protective Alloy Treatment, followed by thermal oxidation, are anticipated to exhibit reduced coking. This is because Minimox treatment (1) Reduces oxide scale cracking, (2) Changes the composition of the oxide, and (3) Halts diffusion of metal to the surface though oxide scales.

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Coking Reduction

I. Background

Minimox® Self-Protective Alloy Treatment is a patented process that changes the structure and/or chemistry of the thermal oxide of a wide range of alloys when compared to the thermal oxide of untreated alloys.

A. The Minimox Process

The patented Minimox® process is not a coating, per se, but is more accurately described as a "surface treatment" or "surface doping." The solution contains primarily rare earth nanoparticles that are superficially applied to stainless steels, nickel alloys, aluminum alloys, titanium alloys, magnesium alloys and many superalloys. The particles are not continuous in coverage. There is no measurable coating thickness. The nanoparticles cause resulting thermal oxides to have a different chemistry and structure when compared to untreated materials. The thermal oxides are significantly thinner, but more impervious with an ultrafine grain size. The treatment plus oxidation causes the alloy to become "self-protective."

As an example of “self-protection”, substantial improvement in the high temperature oxidation properties of martensitic stainless steel have been demonstrated. When heated in air, 410 stainless steel can exhibit catastrophic oxidation. When Minimox®-solution treated 410 stainless steel is heated in air, the oxide is thin and stable. Figures 1-2 compare macrophotographs of 410 stainless steel heated in air to 850°C for 24 hours. The oxide in Figure 1 is primarily Fe_2O_3. In contrast, the oxide in Figure 2 (Minimox-treated) is a combination of Cr_2O_3 and $MnCr_2O_4$. There is no Cr or Mn in the Minimox solution. The surface diffusion profiles have changed dramatically.

Figure 1. Surface of untreated 410 stainless steel alloy after oxidation. Surface is primarily Fe_2O_3 (via XRD).

Figure 2. Surface of Minimox-solution treated 410 stainless steel after oxidation. Via XRD, oxide is a combination of Cr_2O_3 and $MnCr_2O_4$. There is no Cr or Mn in the Minimox solution.

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As another demonstration for the effectiveness of Minimox treatment, Figures 3-4 show the topography of the thermal oxide of treated and untreated Ni600 alloy. The surface of the oxidized, untreated alloy has blisters and flakes. The surface of the treated and oxidized alloy is relatively smooth and dense.

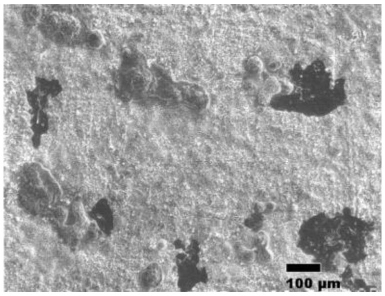

Figure 3. Surface of untreated Ni 600 alloy after oxidation. SUBSTANTIAL FLAKING. Untreated and oxidized surface has exposed metal to catalyze coking.

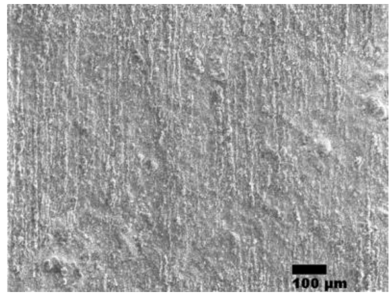

Figure 4. Surface of Minimox-solution treated Ni 600 alloy after oxidation. NO FLAKING IS PRESENT – therefore, there is no exposed metal and coking is not nucleated.

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II. Rationale for the use of Minimox in Coking Applications

According to literature sources,[1] *"It is found that surface scales developed initially on pure Ni and Fe-Cr-Ni-Mn alloy surfaces have no catalytic effect on deposition of filamentary coke. But metal or alloy substrates under cracked scales strongly catalyze nucleation and growth of filamentary coke along the cracks."*

This research implies:

- Oxides do not have a catalytic effect for coke deposits.
- Cracked scales are very deleterious.

Therefore, the ***thin, stable, uncracked oxides on Minimox-treated surfaces are anticipated to reduce coking.*** The next page illustrates the results of related laboratory tests.

[1] Wu, X.Q., et al, *Journal of Materials Science*, **35** (2000) 855-862.

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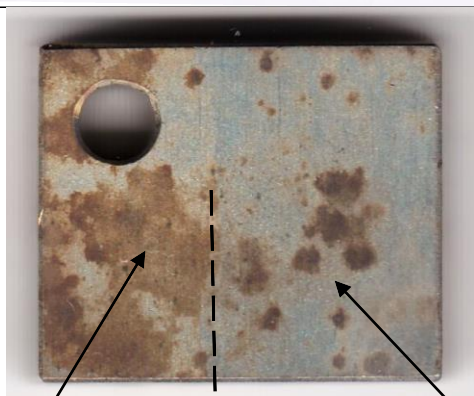
III. Treated Samples Exposed to Pack Carburization

Experiments were conducted on 330 stainless steel. Samples were partially treated with Minimox and were preoxidized to 600°C for 10 hours. These half-treated samples as well as virgin surfaces were inserted into a pack carburization system at 593°C for 600 hours. Because the coated samples were only half treated with Minimox, the effect of mere oxidation was also compared. The samples are shown in the images below. Although this is not a formal coking experiment, it is anticipated the results would be similar.

	330, virgin, machined, after pack carburization tests. Adherent graphite residue is apparent.
 Oxidized without Minimox / Oxidized with Minimox	330, machined, right side treated with Minimox, after pack carburization tests. The Minimox-treated side shows decreased graphite adhesion and decreased corrosion.

III. Summary

Minimox® treatment:

- Stabilizes the thermal oxide scale, thereby mitigating cracking
- Restricts diffusion of metal to the surface.
- Reduces graphite adhesion and corrosion.

This thin, dense oxide is anticipated to reduce coking catalysis.

Contact our office for full details of the experiments conducted.

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