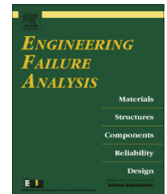




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Failure of pyrolysis coils coated with anti-coking film in an ethylene cracking plant [☆]

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ABSTRACT

This paper presents a specific kind of failure in ethylene cracking coils coated with anti-coking film. It investigates a case in which the coils made of 35Cr 45Ni high temperature alloy failed within two years of operation. The damage occurred due to heavy oxidation in localized regions of the coil resulting in the formation of blisters, which eventually failed by cracking. The mechanism involved was determined by studying the oxidized samples under a scanning electron microscope with an energy dispersive system and is attributed to the presence of rare earth metals in the anti-coking film and inherent casting defects in the base alloy. The cerium present in the anti-coking film diffused preferentially to a defect site in the parent alloy thereby resulting in its segregation which further led to embrittlement.

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1. Introduction

Ethylene production in a petrochemical plant is carried out in a cracking furnace. These furnaces comprise of radiant coil assemblies in which a pre heated mixture of hydrocarbons and steam is cracked at high temperatures of around 950–1150 °C and at moderate pressure of 2–3 bars [1]. Such conditions demand the use of high metallurgy alloys which can demonstrate excellent mechanical and physical properties at elevated temperatures. To fulfill this requirement the furnace coils are usually made of the Fe–Cr–Ni alloy system. These alloys exhibit excellent properties in terms of oxidation resistance, carburization resistance, high temperature creep and thermal expansion.

Further to this, many significant improvements have been made to reduce the damage in the furnace coils by reducing the extent of carburization and oxidation. One of the ways that has been implemented worldwide in the last few years is by the use of specially coated coils [2].

These coils are internally coated with a thin and adherent anti-coking film which is made up of composites with metallic and ceramic constituents along with the presence of rare earths (RE) like yttrium, zirconium and cerium [3,4]. The rare earths present in these coatings improve oxidation resistance and the low concentration of nickel, iron and other metals in the coating (which act as substrate for the formation of filamentous coke) enhances the carburization resistance of the furnace coils [5–8]. Thus these anti-coking films serve a dual purpose.

[☆] This document is a collaborative effort.

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1.1. Effect of adding rare earth elements

The protective oxide layers of chromium and aluminum, which form at high temperatures in these alloys, act as a barrier between the substrate and the environment thereby reducing further oxidation. But upon exposure to oxidizing atmospheres for extended periods at high temperatures the chromium oxide layer tends to become non-protective in nature. The internal stresses which increase as the thickness of the oxide layer increases [9] and the formation of volatile CrO_3 at high temperatures leads to the formation of internal cracks and decreased oxide scale adhesion. An improvement in this has been achieved by the addition of rare earths in the alloy. These can be (1) pre-alloyed with the base metals, or (2) alloyed with the anti-coking films that are applied on the internal surface of the furnace coils [10,11].

The rare earth ions present in the anti-coking film diffuse towards the base alloy and contribute in forming a diffusion barrier. The mechanism most widely accepted attributes the improvements to segregation–diffusion of the rare earth ions to oxide grain boundaries [12]. The oxygen potential gradient, which extends from the gas interface into the substrate, sets up the path for the diffusion of the RE ions, which first segregate to the metal–scale interface and then diffuse inwards into the scale. The RE ions follow the fastest path to the gas interface i.e. the scale grain boundaries. The ionic radii of the rare earths are significantly higher than that of the elements in Fe–Cr–Ni alloys, and their presence at the alloy or oxide grain boundary impedes cation diffusion. This in turn transforms regular anion diffusion into the predominant diffusing species and thus determines oxide growth [13–17].

Introducing very low amounts (0.1%) of rare earth elements to alloys has been proven to be very effective in improving the reliability of the oxide scale. Although the action of rare earth elements is not fully understood, several mechanisms have been proposed [18], including but not limited to the following:

- Improving the scale adhesion through the development of oxide pegs into the alloy acting as mechanical keying agents.
- Reducing the accumulation of voids at the alloy/scale interface leading to better adhesion.
- Imparting higher spallation resistance by enhancing the scale plasticity through the modification of the oxide structure.

The outward cationic diffusion, which has been observed in Fe–Cr–Ni alloys, generates vacancies that, upon accumulation, may lead to the formation of voids at the alloy/oxide interface (lack of adhesion) and eventually spallation [18]. However, the formation of the voids could be prevented by adding rare earth elements like Y, Ce or Sc, that are believed to act as a sink for the vacancies.

2. Investigation

The radiant coil assembly of the ethylene furnace investigated at Reliance Industries petrochemical complex (Gujarat, India) is as shown in the figure and consists of four passes (W-shaped, Fig. 1) in which all the passes are made up of 35% Cr–45% Ni alloy. The coils were internally coated with an anti-coking film to increase decoking time by reducing coke formation. During operation a leak was detected in a second pass tube and the furnace was taken off the service for detailed inspection and repairs.

The leak from the coil was observed in the form of a crack which had opened up to one-third of its circumference (see Fig. 2a). The region surrounding the crack appeared blistered (see Fig. 2b) and recorded high magnetic permeability values. Further investigation revealed similar blisters at six different locations with each of them being located on the inlet pass of the furnace coil assembly. Visually these blisters appeared similar to the blister in the cracked tube except for the fact that these had not yet cracked completely.

3. Experimental results

The failed coil samples were subjected to various non-destructive testing and microstructural analysis.

3.1. Macrostructural examination

The macrostructural examination reveals that the damage is highly localized in nature and the affected region had taken the form of a blister (see Fig. 3a and b).

The depth of the blistered region is comparable to the thickness of the radiant tube. The nature of attack seems to be external and slightly greenish layers formed in the blistered region suggest the presence of oxides of chromium. It appeared as if the outer surface of the tube had undergone oxidation and due to poor oxidation resistance the subsequent layers were oxidized as well.

3.2. Liquid penetrant testing

The liquid penetrant test revealed the presence of cracks in all the cases (see Fig. 4a and b). Coil samples in which the cracks were observed on both the inner and outer surfaces reflect a higher extent of oxidation damage in such a way that

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