Engineering Failure Analysis 66 (2016) 526–532

Contents lists available at ScienceDirect

Engineering Failure Analysis

journal homepage: www.elsevier.com/locate/engfailanal

Embrittlement of a FeCrAlMo alloy exposed to high temperature

Jorge Stella *, Aurora Molina, Adalberto Rosales

Universidad Simón Bolívar, Departamento de Ciencia de los Materiales, Caracas 1080, Venezuela

ARTICLE INFO

Article history:
Received 21 December 2015
Received in revised form 5 May 2016
Accepted 9 May 2016
Available online 13 May 2016

Keywords:
FeCrAlMo alloys
Embrittlement
ODS
Aging

ABSTRACT

A metallurgical evaluation of a FeCrAlMo alloy was carried out after observing low temperature fracture in several purge lines made of this material. The unexpected material embrittlement was attributed to high temperature phase transformations occurred during a previous exposure of the components at 800 °C. The microstructural analysis showed extensive Mo-rich precipitates and Cr-rich carbides at grain boundaries associated with the failed components. The study also showed by means of compression tests carried out at room temperature evident loss of ductility in the samples exposed at high temperatures as a consequence of the material aging.

1. Introduction

FeCrAl alloys have been widely employed during the past years in high temperature applications. Their main application fields involve chemical and metallurgical processes where temperatures above 1000 °C are commonly reached in strongly oxidizing or carburizing environments [1–4]. FeCrAl alloys are recognized to form protective external Al_2O_3 layers against oxidation and metal dusting and to exhibit acceptable creep resistance. Since the above reasons, these alloys have received high acceptability in industrial processes that demand materials with reliable performance at high temperatures.

Novel powder metallurgy (P/M) FeCrAl based alloys have been introduced in the market since two decades ago. Some of these materials possess interesting modifications aimed at increasing creep resistance such as addition of molybdenum as alloying element and small quantity of dispersed oxides in the metallic matrix [5–7]. Nevertheless, up to now mechanical properties of these materials have not been studied in depth. This paper presents a study related to recurrent failures observed in a direct reduction plant. In this plant frequent fractures were detected in a novel material employed in purge lines previously exposed at 800 °C for time periods of approximately 90 days in contact with a gas mixture of composition: H_2-71.35; CO-9.42; CO_2-11.50; N_2-0.13; CH_4-7.60 (vol%). In these systems brittle fracture has been repeatedly identified in tubular components made of a P/M FeCrAlMo alloy during maintenance activities usually carried out in scheduled plant shutdowns. Material fracture has been observed to occur at room temperature as consequence of low intensity impacts employed in order to remove adhered deposits on the affected components. The main aim of this study is the characterization of the microstructural changes that took place during the material exposure at high temperature in order to find the causes of embrittlement.

Failures originated by embrittlement after aging in iron–base alloys have been widely reported, especially in processes that involve operations or manufacture steps that are carried out at high temperatures. Many failure analyses of components [e.g. 8–11] have revealed formation of Cr and Mo-rich secondary phases, which promote significant loss of ductility and eventual fracture of the components. Sigma phase (σ) has probably been claimed as the most deleterious Cr-rich phase related to embrittlement in

* Corresponding author.
E-mail address: jstella@usb.ve (J. Stella).

http://dx.doi.org/10.1016/j.engfailanal.2016.05.016

iron–based alloys, especially in stainless steels with relative high Mo contents such as duplex and superduplex steels [12–15]. Moreover, other secondary phases (such as R, χ, Laves, μ) associated with high Cr and Mo contents, have been recognized as embrittlement promoters [16–18]. It is well known that these phases are formed after aging in very low volumetric fractions; nevertheless, their influence on the mechanical properties of the material can become extremely harmful. Most fundamental studies concerning secondary phases have been conducted in stainless steels and high alloyed steels, however, to date, few or none characterization concerning aging of FeCrAl and FeCrAlMo alloys have been reported, hence studies based in failures originated by aging in these group of materials may be a relevant contribution for development and improvement of new iron-based alloys employed at high temperatures.

2. Experimental analysis

As summarized in Table 1 the failure analysis involved the unexposed material (M0) and samples in ex-service condition (after 90 days exposure at 800 °C). Microstructural characterization of samples in ex-service condition (M1 and M2) was performed in two zones: the fracture zone and an adjacent area which is separated 15 cm from the fracture zone. Microstructure analyses were carried out by means of scanning electronic microscopy (SEM), and energy dispersive X-ray spectroscopy (EDS). Sample microstructures were revealed by metallographic etching employing a solution with the following chemical composition: HNO_3: 25; CH_3COOH: 25; HCl: 37.5; Glycerin: 10; HF: 2.5 (vol%). Furthermore, X-ray diffraction (XRD) characterization was also conducted in samples without previous exposure and in ex-service condition. The diffraction data was collected employing a Bruker-D8 Advance diffractometer, using a Cu-Kα radiation ($\lambda = 0.154$ nm) and a Bragg-Brentano configuration. XRD scans were carried out in the θ-range of 2–100° (θ: angle between the specimen plane and the incident X-ray) in steps of 0.02°.

In order to qualitatively evaluate the grade of ductility of the material compression tests were carried out. Due to the available geometry of the samples, the tests were done following non-standardized conditions on 3.34 cm diameter and 1 cm thick rings. A hydraulic compression machine (Perkin Elmer) was employed for the compression tests. The rings were loaded until fracture or until the samples reached a high level of deformation. The maximum applied load was 1700 kgf.

3. Results and discussion

3.1. Characteristics of the unexposed material

The evaluated material is an ODS (oxide dispersion strengthened) alloy obtained by P/M. Its nominal composition is shown in Table 2. The microstructure mainly consists of a ferritic matrix with moderate concentration of yttria, zirconia and hafnia particles finely dispersed in the matrix as shown in Fig. 1. The ferritic matrix presents a wide distribution of grain sizes with diameters between 20 and 400 μm. No secondary metallic phases in the unexposed samples were identified by metallographic examination.

3.2. Morphological aspects of the material in ex-service condition

Fig. 2 shows the cross section microstructure of a failed component in near and far regions from the fracture areas. Both regions exhibit similar characteristics regarding grain size and precipitate distribution. Although a low fraction of precipitates is located inside the ferritic grains, it is clear from Fig. 2 that precipitates are mainly concentrated at grain boundaries. According to the morphological details and chemical analysis of the precipitates (Fig. 3) two kinds of particles can be identified as follows:

a) Faceted particles of diameter between 5 and 25 μm. According to the EDS analysis and the observed particle morphology this phase corresponds to a Cr-rich carbide. EDS analysis shows higher concentration of Cr (50–70 in wt%) in this phase and relative lower Mo values (~11%).
b) Molybdenum rich particles with a mean diameter lower than 5 μm. While Mo weight percents are typically over 30%, Cr contents are lower than 15%.

As observed in Figs. 2 and 3, the apparent occupied volume by the small particles (Mo-rich) is considerably higher than that observed for the carbide particles. Interestingly, Mo-rich particle formation was mainly observed at the carbide boundaries. The chemical element distribution and location of Mo-rich particles suggest that the formation of these particles is originated from the previous transformation of Cr-rich carbides, which contain a higher Mo concentration than the matrix.

It is also noticeable the fact that Mo-rich particles precipitation takes place uniformly along the whole cross section of the component following no gradients related to internal or external walls. This suggests that the environmental condition had little or none influence on the aging of the material.

Table 1
Characteristics of the analyzed samples.

Material	Previous exposure	Sample location in the component
M0	Unexposed	Random
M1	Exposed approximately 2160 h (90 days)	Fracture zone
M2		15 cm separated from the fracture zone