

## Precipitation in Fe–15Cr–1Nb alloys after oxygenation

A. Malfliet<sup>a,\*</sup>, F. Verhaeghe<sup>a</sup>, F. Chassagne<sup>b</sup>, J.-D. Mithieux<sup>b</sup>, B. Blanpain<sup>a</sup>, P. Wollants<sup>a</sup>

<sup>a</sup> Katholieke Universiteit Leuven, Department of Metallurgy and Materials Engineering, Kasteelpark Arenberg 44, 3001 Heverlee, Belgium

<sup>b</sup> ArcelorMittal Isbergues Research Centre, Rue Roger Salengro, BP15, 62330 Isbergues, France

Received 6 July 2009; received in revised form 31 March 2010; accepted 2 April 2010

Available online 27 April 2010

### Abstract

The effect of O on the phase relations at 950 °C in Fe–15Cr–1Nb alloys is experimentally investigated. Fe–15Cr–1Nb alloys are oxygenated by subjecting high-purity Fe–15Cr–1Nb to an O atmosphere at 600 °C. Both the high-purity and the oxygenated Fe–15Cr–1Nb alloys are heat treated for up to 500 h at 950 °C, quenched and investigated by scanning electron microscopy, transmission electron microscopy and electron probe microanalysis. The results show that Fe<sub>2</sub>Nb is in equilibrium with  $\alpha$  (Fe, Cr) with 0.29 at.% Nb in solid solution in the pure Fe–15Cr–1Nb alloy. The presence of a small amount of O induces the precipitation of a Fe<sub>6</sub>Nb<sub>6</sub>O<sub>x</sub> phase with a cubic crystal structure and lattice parameter 1.13 nm, thereby decreasing the Nb in solid solution in  $\alpha$  (Fe, Cr) with increasing O content. © 2010 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

**Keywords:** Stainless steels; Ferritic steels; Non-binary oxides; Wavelength dispersive X-ray spectroscopy

### 1. Introduction

Ferritic heat-resisting steels with high Cr content are used in high-temperature applications. Although basic ferritic grades offer good resistance to stress corrosion cracking, high-temperature oxidation and localized corrosion, they suffer from their susceptibility to intergranular corrosion, low plasticity and limited toughness [1]. However, fluctuating Ni prices have challenged the stainless steel-based sectors to develop ferritic grades which do not suffer from these deficiencies. An important improvement was achieved by stabilizing the ferritic stainless steel through the addition of Ti and/or Nb which form stable carbides and nitrides thereby preventing sensitization. In addition, remaining Nb in solid solution is reported to improve the high-temperature strength [2]. Today, these stabilized grades are strong competitors for the austenitic grades due to their lower and stable cost, comparable high-temperature mechanical properties, limited thermal expansion and excellent corrosion resistance. Nonetheless, to broaden

the use of ferritic grades to more severe working conditions combined with increased life expectancy, the stainless steel industry is continuously forced to develop new grades with improved properties. Some recent investigations have focussed on the addition of W, Mo or Nb to enhance the high-temperature creep strength through the precipitation of intermetallic phases. Although a wide variety exists in their composition, structure, temperature range and stability, some similarities in the behavior of these precipitates can be observed. When M (=W, Mo or Nb) is added to pure Fe–Cr, the hexagonal Laves phase Fe<sub>2</sub>M typically forms. In the presence of C, a phase with stoichiometry Fe<sub>3</sub>M<sub>3</sub>C and crystallographic prototype Fe<sub>3</sub>W<sub>3</sub>C-type is observed in the temperature range 850–1100 °C [3–5]. In particular, Fujita [5] has investigated the Fe–Cr–Nb–C system in the composition range 12.9–19.6 wt.%Cr, 0.40–0.78 wt.%Nb and 0.014–0.016 wt.%C. His results show that at high temperature (900–950 °C) the Fe<sub>3</sub>Nb<sub>3</sub>C phase replaces the Fe<sub>2</sub>Nb phase, thereby forming coarse precipitates which are detrimental for the creep strength. Chassagne [1] observed Fe<sub>2</sub>Nb<sub>3</sub> precipitates in an industrial ferritic stainless steel which are more stable than the Fe<sub>2</sub>Nb precipitates at least up to 900 °C. This Fe<sub>2</sub>Nb<sub>3</sub> phase has a large crystallographic and stoichiometric similarity with the

\* Corresponding author. Tel.: +32 16 32 12 79; fax: +32 16 32 19 91.

E-mail address: [annelies.malfliet@mtm.kuleuven.be](mailto:annelies.malfliet@mtm.kuleuven.be) (A. Malfliet).

$\text{Fe}_3\text{Nb}_3\text{C}$  phase, but C is not mentioned to be present in this phase. The effect of N and O on the precipitation behavior in the Fe–Cr–Nb system is less well investigated. We investigate the nature of the Nb-containing precipitates in oxygenated Fe–Cr–Nb alloys, thereby focussing on the precipitation of a phase similar to  $\text{Fe}_3\text{Nb}_3\text{C}$ .

## 2. Experiments

High-purity Fe–15Cr–1Nb alloy was prepared in a levitation induction furnace. Special care was taken to minimize the content of C, N, O and S. Chemical analysis showed that the alloy contains 15.15 wt.% Cr and 0.99 wt.% Nb, while the C, N, O and S levels are below 10 ppm each. This alloy was hot rolled to a thickness of 1.07 mm. Contamination from the outer surface was avoided by grinding and ultrasonically cleaning the sample before each handling. The high-purity Fe–15Cr–1Nb alloy served as our reference material and also as the starting material to prepare samples with varying O content. The procedure is shown in Fig. 1. First, a piece of Fe–15Cr–1Nb and a certain amount of  $\text{NaClO}_4$  were enclosed in a quartz tube evacuated to  $10^{-6}$  mbar. This tube was kept 96 h at 600 °C. The perchlorate decomposed in NaCl and gaseous O, thereby subjecting the sample to pure O. Next, both the pure alloy and the oxygenated alloys were heat treated. For this purpose they were enclosed in quartz tubes at a pressure of  $10^{-6}$  mbar. For one sample of the pure alloy, a piece of Ti foil was added to capture any remaining O gas inside the tube. The samples were heated at 950 °C for 168 up to 500 h followed by rapid quenching in water, thereby breaking the quartz tubes. An overview of the oxygenation and heat treatment procedure is given in Table 1. The O concentration in Table 1 expresses the amount of O added in the form of  $\text{NaClO}_4$  during the oxygenation treatment relative to the amount of Fe–15Cr–1Nb

Table 1

Experimental conditions for the oxygenation and the subsequent heat treatment of the high-purity Fe–15Cr–1Nb alloy. The O concentration expresses the amount of O added in the form of  $\text{NaClO}_4$  during the oxygenation treatment relative to the amount of Fe–15Cr–1Nb alloy.

Sample	Oxygenation		
	O (ppm)	<i>t</i> (h)	<i>T</i> (°C)
Fe–15Cr–1Nb	1	–	–
	2	–	–
Fe–15Cr–1Nb– <i>x</i> O	3	100	96
	4	500	96
	5	1200	96
	6	2700	96
	7	1000	96
	8	300	96
	9	1800	96
Fe–15Cr–1Nb	Precipitation treatment		
	<i>p</i> (mbar)	<i>t</i> (h)	<i>T</i> (°C)
Fe–15Cr–1Nb	1	$10^{-6}$	500
	2	$10^{-6}$	500
Fe–15Cr–1Nb– <i>x</i> O	+Ti getter		
	3	$10^{-6}$	168
	4	$10^{-6}$	168
	5	$10^{-6}$	168
	6	$10^{-6}$	168
	7	$10^{-6}$	500
	8	–	–
9	–	–	

alloy. The actual O uptake as O in solution plus O in the form of (internal) oxides will be equal or lower than this value considering the possibility that not all O reacted with the sample. The samples Fe–15Cr–1Nb–*x*O8 and Fe–15Cr–1Nb–*x*O9 were not subjected to the heat treatment at 950 °C to investigate the effect of the O treatment at 600 °C.

Finally, the samples were cross-sectioned. One piece was used to measure the total O content in the bulk alloy with an LECO-TC136 Oxygen/Nitrogen Determinator. The remainder of the sample was embedded in resin for scanning electron microscopy (SEM), transmission electron microscopy (TEM) and wavelength dispersive spectroscopy (WDS) preparation. Samples for SEM were prepared by grinding, polishing and electrolytic etching in oxalic acid. SEM was used to visualize the location and distribution of the precipitates and to perform energy dispersive spectroscopy (EDS) analysis on large precipitates. Further characterization, especially of the small precipitates, was performed by TEM using carbon extraction replicas. After grinding and polishing, the samples were etched for 4 min in a picric solution (20 g l<sup>-1</sup> picric acid, 50 ml l<sup>-1</sup> HCl and ethanol) followed by deposition of a carbon nanolayer. This layer, containing the precipitates, was removed from the sample by dissolving the steel matrix in the same picric solution. The carbon replica was positioned on a copper grid before insertion in the transmission electron microscope. Chemical analysis of the precipitates was performed by standardless EDS using a correction factor for thin

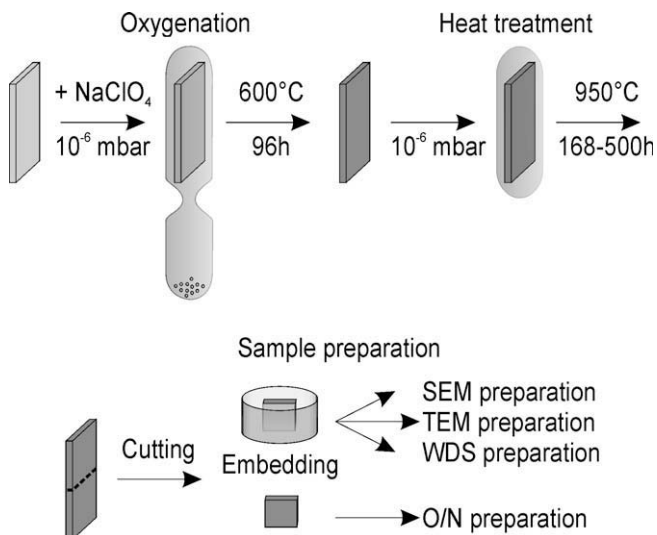


Fig. 1. Flow diagram of the oxygenation, heat treatment and sample preparation of the Fe–15Cr–1Nb alloys.

Download English Version:

<https://daneshyari.com/en/article/1447787>

Download Persian Version:

<https://daneshyari.com/article/1447787>

[Daneshyari.com](https://daneshyari.com)