

Characterization of the oxides formed at 1000 °C on the AISI 304 stainless steel by X-ray diffraction and infrared spectroscopy

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Abstract

The aim of this work is to show the contribution of the infrared spectroscopy (FT-IR) to the identification of the oxides formed on the AISI 304 stainless steel during isothermal oxidation at 1000 °C, in air. This work focuses on the differentiation of spinel type AB_2O_4 structures and corundum type M_2O_3 structures. It is shown that after 100 h oxidation, the scale is composed of two subscales. The structural analyses were performed both on the adherent subscale and on the external subscale, which spalled off during cooling to room temperature. In the spalled subscale, the infrared spectroscopy (FT-IR) and X-ray diffraction (XRD) analyses exhibit the presence of two spinel phases: $Mn_{1.5}Cr_{1.5}O_4$ and $FeCr_2O_4$, as well as hematite Fe_2O_3 . The FT-IR and XRD analyses of the adherent subscale enable us to distinguish the spinel $Mn_{1.5}Cr_{1.5}O_4$ and $FeCr_2O_4$ structures which are difficult to identify by XRD alone. Chromia Cr_2O_3 appears to be slightly present in the adherent subscale. According to our results, the parabolic regime of the kinetic curve corresponds to a scale growth mechanism governed by an inward oxygen diffusion. Published by Elsevier B.V.

Keywords: AISI 304 stainless steel; XRD; FT-IR; High temperature oxidation

1. Introduction

High-temperature stainless steels of AISI 304 type are frequently used in power plants, gasification systems, pipes used in petroleum refining plants, combustion processes and in aerospace applications. Depending on the application, materials are subjected to corrosive atmospheres and thermal cycling [1,2].

Before high temperature treatment, one should notice the initial presence of α' martensitic phase in the XRD analysis (Fig. 1) of the 304 steel, which comes from its elaboration and is induced by polishing treatments.

Several studies highlight that, at high temperature, chromia-forming alloys such as AISI 304 stainless steels present an initial oxide growth leading mainly to chromia Cr_2O_3 and spinel type oxide formation [3–18]. However, *in situ* X-ray diffraction studies have shown that after 10 h oxidation at 1000 °C, a change in the structural composition of the layer

appears with time (Fig. 1) [19,20]. Then, it seems that the oxide scale is composed of iron containing oxides such as Fe_2O_3 and $FeCr_2O_4$, which are supposed to be responsible for the change of the oxidation kinetic. Nevertheless, it appears that XRD alone does not permit to clearly characterize the spinel or corundum phases present in the outer and the inner oxide subscales.

The aim of this work is to better differentiate the spinel type AB_2O_4 structures and corundum type M_2O_3 structures, present in the oxide scale, by the use of the FT-IR spectroscopy [21–23]. This study can also be related to the work of McDevitt and Baun [24] who employed this technique to identify oxides exhibiting various structures.

2. Experimental

2.1. Synthesis of reference oxide for FT-IR analyses

In order to investigate the scale structure by FT-IR, we firstly collected a data base of FT-IR spectra of synthesised oxides such as the iron chromite ($FeCr_2O_4$) [25] and manganese chromite ($Mn_{1.5}Cr_{1.5}O_4$) [26]. We also recorded the FT-IR

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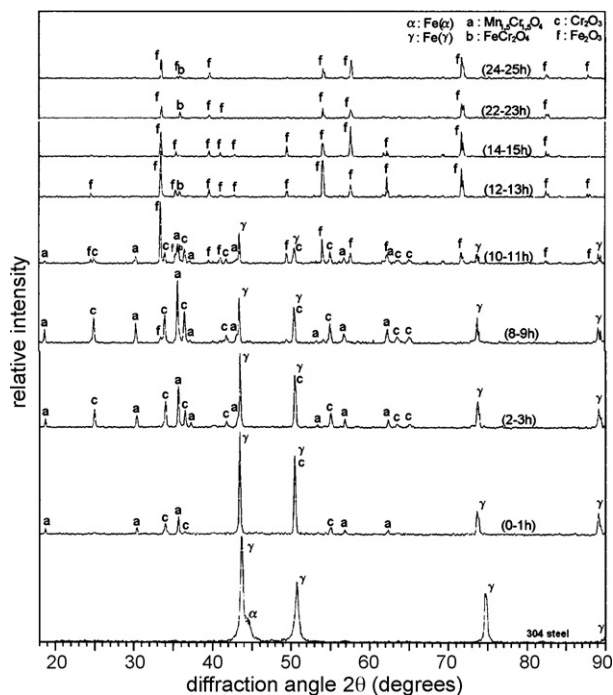


Fig. 1. In situ XRD performed on AISI 304 steel during isothermal oxidation, at 1000 °C, in air [19,20].

spectra of commercial oxides such as Cr_2O_3 and Fe_2O_3 , which are the oxides potentially formed on the AISI 304 stainless steel. FeCr_2O_4 was synthesized owing to the data collected on the Cr–Fe–O phase diagram [25]. The synthesis was performed under argon atmosphere at 1300 °C in a SETARAM TGA 92-1600 apparatus after mixing pure iron (Fe) with Cr_2O_3 and Fe_2O_3 according to the following Eq. (1):



We have also synthesized the $\text{Mn}_{1.5}\text{Cr}_{1.5}\text{O}_4$ spinel oxide following the indications of the Cr–Mn–O phase diagram [26]. The synthesis was realized by mixing Mn_2O_3 with Cr_2O_3 in an alumina crucible placed in a muffle furnace at 1000 °C in air according to the Eq. (2):



The reference oxides of the corundum type (Cr_2O_3 [from ALDRICH] and Fe_2O_3 [from CARLO ERBA]) are commercial high purity chemicals.

The FT-IR spectra, obtained on commercial and synthesized oxides, were collected on a NICOLET IMPACT 400D spectrometer. The characteristic absorption bands obtained on the reference oxides are listed in Table 1

2.2. Sample preparation

AISI 304 specimens provided by Imphy were polished on SiC paper up to the 800 polishing grade, then washed with ethanol and finally dried under a hot draught just before isothermal oxidation at 1000 °C. The alloy composition is given in Table 2.

Table 1
IRTF characteristic bands of various reference oxides

Oxides (synthesised and commercial)			
FeCr_2O_4	$\text{Mn}_{1.5}\text{Cr}_{1.5}\text{O}_4$	Cr_2O_3	Fe_2O_3
Characteristic bands (cm^{-1})			
613	658	684	587
574	609	648	540
504	500	621	469
		582	
		568	

2.3. Procedure to collect the adherent oxide scale

Pendse and Stringer [27] proposed a method to remove the metallic substrate present under an oxide scale in order to analyze the oxide scale alone. We thus prepared a solution of 10% bromine in absolute ethanol. Then, we placed the oxidized samples in a becher containing the Br_2 alcoholic solution during three days at room temperature. After dissolution of the metal, we rinsed the samples with ethanol in order to clean all separated oxide sheets finally collected on a filter. The recovered oxide particles were rinsed with ethanol in order to eliminate all the remaining bromine and finally dried with air in the filter. Then, Infrared Spectroscopy and XRD could be used to analyze the oxide particles.

2.4. Experimental techniques

The FT-IR spectroscopy was carried out with a Nicolet Impact 400D spectrometer. The spectra were obtained with a resolution of 4 cm^{-1} . The wave number was ranging from 400 to 800 cm^{-1} . To prepare the tablet used for FT-IR analyses, 0.2 mg of oxides particles are mixed with 100 mg of dried KBr. The mixture was crushed to prepare a 100 mg tablet finally place in the FT-IR spectrometer. Before mixing with KBr, we have sieved the crushed oxide scale to obtain a particle size less than $38 \mu\text{m}$. As it was mentioned by McDevitt and Baun [24] that the oxide particle size should be as small as possible in order obtain well defined infrared bands.

The kinetic results under isothermal conditions were recorded by means of a Setaram TG-DTA 92-1600 microthermogravimetric balance, during 100 h, at 1000 °C, in air. The characterization of the oxide scales was carried out in an X-Ray Philips X' PERT MPD diffractometer (copper radiation,

Table 2
Weight % composition of the AISI 304 obtained by ICPMS

Fe	Bal.
Cr	17.9
Ni	9.05
Mn	1.52
Si	0.48
Co	0.22
Mo	0.15
Cu	0.10
C	0.05
Ti	0.01
S	0.01

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