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High Temperature Oxidation of AISI 439 Ferritic Stainless Steel in Synthetic Air Atmosphere

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Maria de Fatima Salgado[a,b,c*], Jackeline Macêdo de Sousa Santos[a], Giscard Eanes Dias Viana[a], João Alberto Santos Porto[a,b], Gabriel de Souza Veras Fontinele[a], Esah Hamzah[c], Farid Nasir Ani[c]

[a]Department of Math and Physical, Maranhão State University, Morro do Alecrim, 65.600-380 Caxias, Maranhao, Brazil – CESC/UEMA
bPrograma de Pós-Graduação Eng. de Materiais do Instituto Federal do Piauí – Teresina - PPGEM / IFPI[2]
[c]Faculty of Mechanical Engineering, Universiti Teknologi Malaysia, 81310 UTM Johor Bahru, Johor, Malaysia

*Corresponding author
mariasalgado@professor.uema.br

Graphical abstract

Abstract

Stainless steels may be used and exposed to aggressive gases at high temperatures. The oxidation behavior of AISI 439 ferritic stainless steel, was investigated by oxidation treatment at 850 °C and 950 °C, for 50h in Synthetic Air with 20% O_2 atmosphere in a tubular oven and in a thermobalance. The oxidation kinetics of films are determined by measuring the mass versus oxidation time. The microstructure and chemical composition of the oxides were determined by Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDS). Chemical analysis by EDS showed that films formed on AISI 439 stainless steel exhibited Cr as the principal element in the oxide film, in proportions to form the chromium oxide (Cr_2O_3) and the following elements: Mn, Fe, Ti and Si. Based on the oxidation kinetics, it was observed that steel oxidation follows the parabolic behaviour with increase in temperature and it produced the highest oxidation rate at 950 °C and the lowest rate at 850 °C.

Keywords: AISI 439 Stainless Steel, Synthetic Air, Oxidation, Ferritic stainless steel, Chromium Oxide

Abstrak

Keluli tahan karat boleh digunakan dan terdedah kepada gas gas yang agresif pada suhu tinggi. Prestasi pengoksidaan AISI 439 keluli tahan karat ferit, disiasat dengan rawatan pengoksidaan pada suhu 850 °C dan 950 °C, untuk 50 jam dalam udara sintetik, suasana 20% O_2 dalam ketuhar tubular dan termobalance. Kinetik pengoksidaan filem ditentukan dengan mengukur jisim berbanding dengan masa pengoksidaan. Struktur mikro dan komposisi kimia oksida ditentukan oleh mikroskopi pengimbasan elektron (SEM) dan spektroskopi X-ray dispersif tenaga (EDS). Analisis kimia menunjukkan bahawa filem-filem yang terbentuk pada keluli tahan karat AISI 439 mempamerkan Cr sebagai unsur utama dalam filem oksida, dalam perkadaran untuk membentuk kromium oksida (Cr2O3)dan unsur-unsur berikut: Mn, Fe, Ti dan Si. Berdasarkan kinetik pengoksidaan, ia dapat diperhatikan bahawa pengoksidaan keluli mengikut tingkah laku parabola dengan peningkatan suhu dan menghasilkan kadar pengoksidaan tertinggi pada shu 950 °C dan kadar paling rendah pada suhu 850 °C.

Kata kunci: AISI 439 keluli tahan karat, pengoksidaan, udara sintetik, keluli tahan karat ferit, kromium oksida

1.0 INTRODUCTION

Ferritic stainless steels have progressively replaced austenitic stainless steels due to their lower costs as a consequence of the near absence of nickel [1]. Recent studies show that the oxidation kinetics of the AISI 439 ferritic stainless steel follows a parabolic law in the temperature range of 750-900 °C in dry air [2]. It means that the growth rate of the protective oxide film, mainly composed of Chromia, formed on the surface of the steel is controlled by inward oxygen ions diffusion from atmosphere or by outward cation diffusion from metallic substrate or by both cation diffusion and oxygen ions diffusion [3]. The kinetics of growth and composition of the oxide films formed on the ferritic stainless steel AISI 409 between temperatures 850 °C and 950 °C in synthetic air atmosphere containing 20% of O_2 was recently studied [4]. Chemical analysis described that chromium is the major chemical element in 850 °C formed in the film at other temperatures in which Fe is predominant. Other alloying elements such as silicon and titanium are found in small amounts up to 900 °C. When comparing rates of oxidation, it was found that the AISI 409 steel has gained mass in response to increasing temperature.

The kinetics of growth and composition of the oxide films formed on the ferritic stainless steel AISI 444 between temperatures 850 °C and 950 °C in synthetic air atmosphere containing 20% of O_2 was recently studied [5]. Chemical analysis described that chromium is the majority chemical element at all temperatures, and it was verified that the parabolic increase of the films of oxide formed contain predominantly chromium. A high content of iron was detected in the regions in which the detachment occurred the most.

Salgado *et al*. [6] studied high temperature oxidation behavior of ferritic stainless steel type AISI 441, which was submitted to oxidation treatment at 850 °C and 950 °C over 50 h in two different atmospheres: Synthetic Air in a tubular oven and Argon, containing 1 ppm of O_2. Chemical analysis showed that films formed on AISI 441 stainless steel exhibited mostly chromium oxide and the following elements: Cr, Mn, Fe, Ti and Si. Concerning the oxidation kinetics, it was observed that in Synthetic Air, the steel oxidation grows gradually with the increase of temperature, but in Argon atmosphere, it produced the highest oxidation at 900 °C and the lowest at 950 °C.

The effect of oxygen partial pressure and temperature on the oxidation behavior of unstabilized and Nb-stabilized AISI 430 ferritic stainless steel was investigated [7] over the temperature range of 850 °C - 950 °C in air, Ar +1 ppm O_2 or Ar–H_2–H_2O atmospheres. Nb-stabilized AISI 430 steel is more resistant to oxidation than unstabilized AISI 430 under all tested conditions, except above 900 °C in Ar–H_2 –H_2O. While the oxidation behavior of unstabilized AISI 430 is strongly affected by the atmosphere composition, Nb stabilized AISI 430 oxidation rates do not depend strongly on the atmosphere. For both steels, the chemical analysis shows Cr_2O_3 as the main phase in the oxide scales, but solid solutions such as $FeFe_{2\text{-}x}Cr_xO_4$ and $MnCr_2O_4$ formed in almost all scales for atmospheres of Ar +1 ppm O_2 or in Ar –H_2–H_2O. Fe_2O_3 and Mn.5Cr.5O_4, are also observed for oxidation in air.

AISI 439 is a ferritic stainless steel with ferrite structure and does not change phase as the others stainless steels in the same range [8]. Other works in the literature study about steel oxidation at high temperature in the range 850-950 °C in air atmosphere were used for comparison. The parabolic oxidation kinetics of the AISI 439 steel and present of Cr as major metallic component at some temperatures in both work conditions, indicates that the growth rate of the protective chromia layer formed on the steel surface should be controlled by cation or/and oxygen ion diffusion through the scale layer [9, 25].

The present study aimed to investigate the effect of the equipment, a tubular oven and a thermobalance used on the AISI 439 stainless steel oxidation. As well as comparate the kinetics of oxidation, the micrographs and chemical composition of the films formed on the steel when oxidized in different equipment. That is the first time this research is carried out

2.0 METHODOLOGY

The AISI 439 stainless steel was supplied by Arcelor Mittal Inox of Brazil. The chemical composition is: C (0, 0060%); Mn (0,18%); Si (0,42%); Cr (17,01%); Ni (0,23%); P (0,033%); S (0,0010%); Nb (0,17%); Ti (0,15%); N (122ppm) and Fe (balance). The samples were grinded with 1100, 1200 and 2000 SiC paper, and then polished with a diamond paste of 3 and 1 μm [10]. This procedure is normally used before the oxidation test of stainless steel at high temperature [4]. The oxidation testes were carried out in a dynamic atmosphere of synthetic air with 20% O_2 in a tubular furnace for 2, 4, 8, 16, 32, 40 and 50h to grow a chromia layer on the polished surface of the steel. The samples were cut into dimen¬sions of (10 x 10 x 0.6) mm3. For continuous oxidation, samples with dimensions of (5x 5x 0,6) mm3 were used. The treatments were performed in a thermobalance SETARAM TGDTA 92, with sensibility equal to ± 1 μg between 850 °C and 950 °C for 50h maintained under total pressure of 1 atm.

The oxidation kinetics were established by measuring the mass gain per unit area ($\Delta M/S$) as function of oxidation time (t). The microstructures of the oxide films were examined by Scanning Electron Microscope (SEM) and the chemical composition were determined by Energy Dispersive X-ray Spectrometer (EDS).

3.0 RESULTS AND DISCUSSION

3.1 Oxidation Kinetics

The results of mass gain per unit area (ΔM/S) as a function of oxidation time (t). Were ploted to show to oxidation behavior of the steels. The AISI 439 stainless steel oxidation behavior in synthetic air between 850 °C and 950 °C are shown in Figures 1 and 2. It is observed that AISI 439 exhibited gradual increase in mass gain as the temperature increases. It is observed that the mass gain at all temperatures increases very rapidly at the initial stage probably due to rapid nucleation of Mn spinell phase. The result is similar to that determined at the same temperature for the oxide growth [11, 12].

Is showed in the Figure 1, the thermogravimetric analysis of the stainless steel after oxidation at 850 °C, 900 °C and 950 °C in air atmosphere for 50 hours.

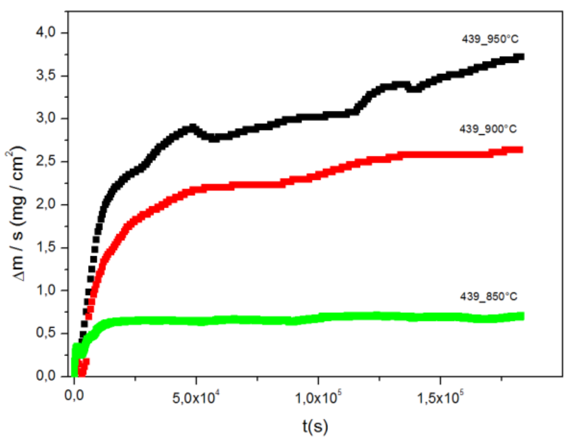

Figure 1 Oxidation AISI 439 in thermobalance

The curves of mass gain as a function of oxidation Time obtained in the analysis conducted in a tubular oven between 850 °C and 950 °C in synthetic air atmosphere are shown in Figure 2.

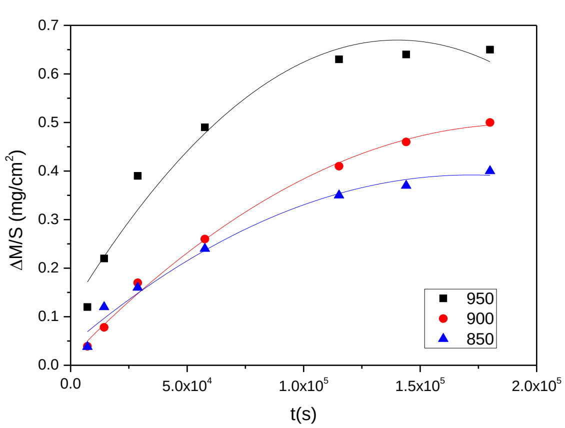

Figure 2 Oxidation AISI 439 in tubular furnace

The oxidation kinetics exhibit only one stage, and the variation of mass as a function of time is very small and independent of temperature. Both oxidations behavior follow parabolic kinetics, indicating that kinetic oxidations are controlled by cations and/or by oxygen diffusion through oxide film[9].

The parabolic kinetics during oxidation are described by the ratio: $(\Delta M/S)^2 = kpt + ko$, where kp is the parabolic oxidation constant and ko is a constant. The constant (kp) was established in graphs of (M/S)2 as a function of the time.

When the kinetic oxidation follows a parabolic law, the graph of $(\Delta M/S)^2$ as a function of oxidation time (t) provides a straight line, in which the angular coefficient is equal in value to the parabolic constant of an oxidation named "kp". The graphs of $(\Delta M/S)^2$ as a function of oxidation time (t) were plotted in order to determine the constant oxidation values, which are illustrated in Table 1.

Table 1 Parabolic constant kp ($g^2.cm^{-4}.s^{-1}$) after isothermal oxidation of AISI 439 Stainless steel for 50 h in two different furnaces

Kp ($g^2.cm^{-4}.s^{-1}$) - AISI 439 Stainless steel - Synthetic air		
Temperature (°C)	**Tubular Furnace**	**Thermobalance**
850	$7{,}51 \times 10^{-08}$	$2{,}67 \times 10^{-12}$
900	$1{,}10 \times 10^{-07}$	$3{,}83 \times 10^{-11}$
950	$2{,}42 \times 10^{-07}$	$7{,}26 \times 10^{-11}$

These results are in agreement with the work of other researchers affirming that the constant parabolic oxidation, (kp), varies with temperature [18].

3.2 Microstucture of Oxide Films

The SEM images shown in Figures 3 to 8 of the samples oxidized at temperature 850 °C and 950 °C in Synthetic Air atmosphere exhibit the growth of a continuous oxide film at all temperatures [13].

Figures 3, 4 and 5 show the SEM of the steel when oxidated in Thermobalance. It can be seen that at 850 °C, preferential oxidation occurs in grain boundary, where as at 900 °C a more uniform film is seen and at 950 °C a really compact film is shown covering all grains.

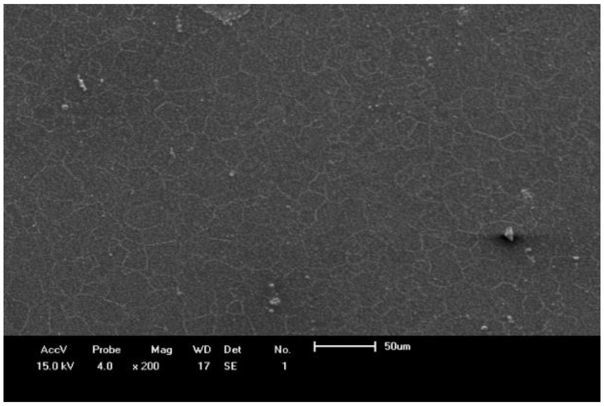

Figure 3 SEM micrograph, temperature 850 °C, atmosphere: synthetic air – thermobalance

The oxide scale formed on the 439 steel is continuous but at 850 °C can be seen preferential oxidation of grains.

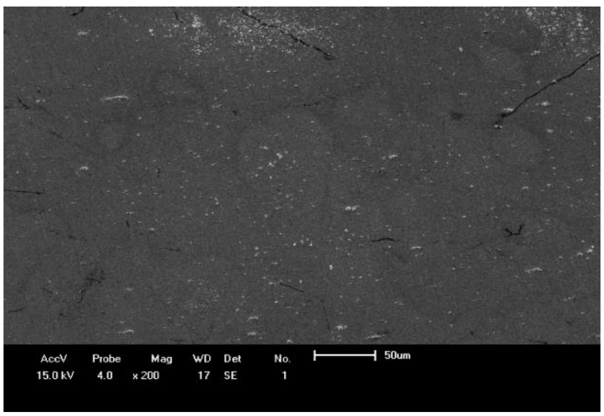

Figure 4 SEM micrograph, temperature 900 °C, atmosphere: synthetic air thermobalance

At 900 °C there is the formation of spinel and chrome oxide several clusters.

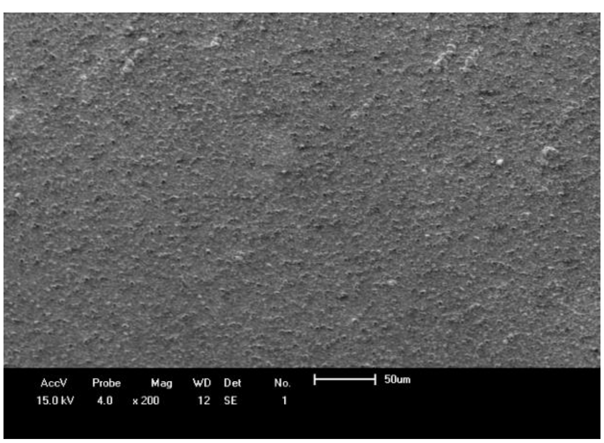

Figure 5 SEM micrograph, temperature 950 °C, atmosphere: synthetic air thermobalance

Finally was observed that the chromium and oxygen diffusivities in grain boundaries are greater than the corresponding volume diffusivities under the same experimental conditions, showing that the grain boundaries are the preferred ways for the ion diffusion in the oxide films formed on the AISI 439 steel, for all temperatures as shown in Figures 6, 7 and 8. the results agree Malheiros [26]. The oxide scale formed on the 439 steel is continuous for all temperatures.

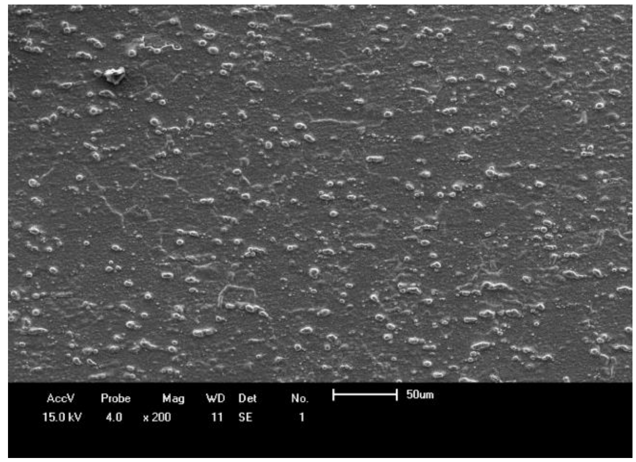

Figure 6 SEM micrograph, temperature 850 °C, atmosphere: Synthetic Air AISI 439_tubular furnace

At 850 °C there are spherical particles of about 3 – 6 μm in diameter dispersed on the oxide scale as shown in Figure 6.

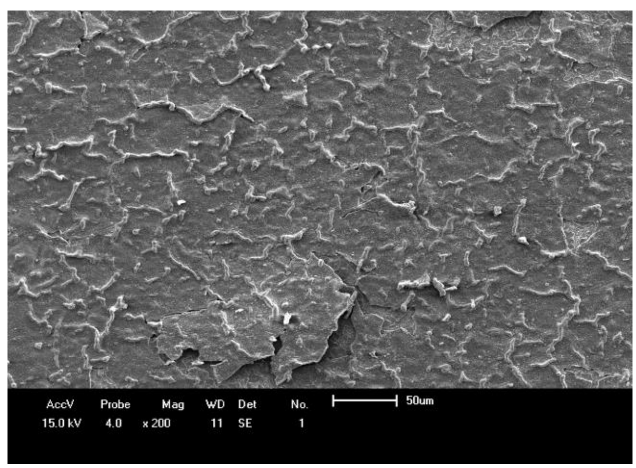

Figura 7 SEM micrograph, temperature 900 °C, atmosphere: Synthetic Air AISI 439_ tubular

In some grain-boundary an increase in stress at 900 °C was observed to occur in the detailed region where the embrittlement of the film was detached in the SEM analysis as shown in Figure 7 and by [14].

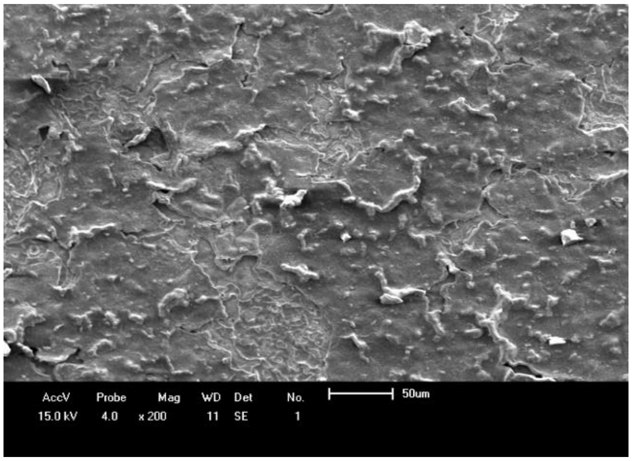

Figure 8 SEM micrograph, temperature 950 °C, atmosphere: Synthetic Air AISI 439_ tubular furnace

In the Figure 8 was showed an adherence and continuous film on the sample. These results was observed by other researches [1, 6, 7, 19].

It can be seen in the images of the oxidized samples that thermobalance provides more uniformity than those oxidized in tubular oven. This may be because the samples were introduced into the tubular furnace when the latter was already at the temperature specified for carrying out the oxidation. As far sample was introduced into the thermobalance when it was at room temperature and passed through the process of oxidation during heating of the thermobalance, thereby resulting in the small difference in the surface morphology of the film.

3.3 Chemical Composition of Oxide Films - EDS Analysis of Oxide Film Formation

The formation of the chromia-rich scale with chromium-manganese spinel is the commonly observed situation when Stainless Steel Ferritic are oxidised at high temperature [3, 4, 5, 6, 7, 11,12]. The spectrum shown in Figures 9 to 14 correspond to the micrographs and chemical composition of oxide films formed at 850 °C, 900 °C and 950 °C for oxidation of the AISI 439 steel in air. The EDS analysis show the Cr as the major metallic element on oxide films formed in all experimental conditions. Lower quantities of Mn, Ti and Fe were also observed in the oxide films.

The micrographs and spectrum shown in Figures 9 to 11 correspond to oxidation of the AISI 439 steel in air in the thermobalance. The micrograph obtained at 850 °C is shown in Figure 9a. The EDS plots of chemical composition as shown in Figures 9a and 9b present two distinct points observed where at point 2 is showns an intense peak of Ti element indicating the formation of a structure called sand rose which was also observed by Resende *et al*. [1].

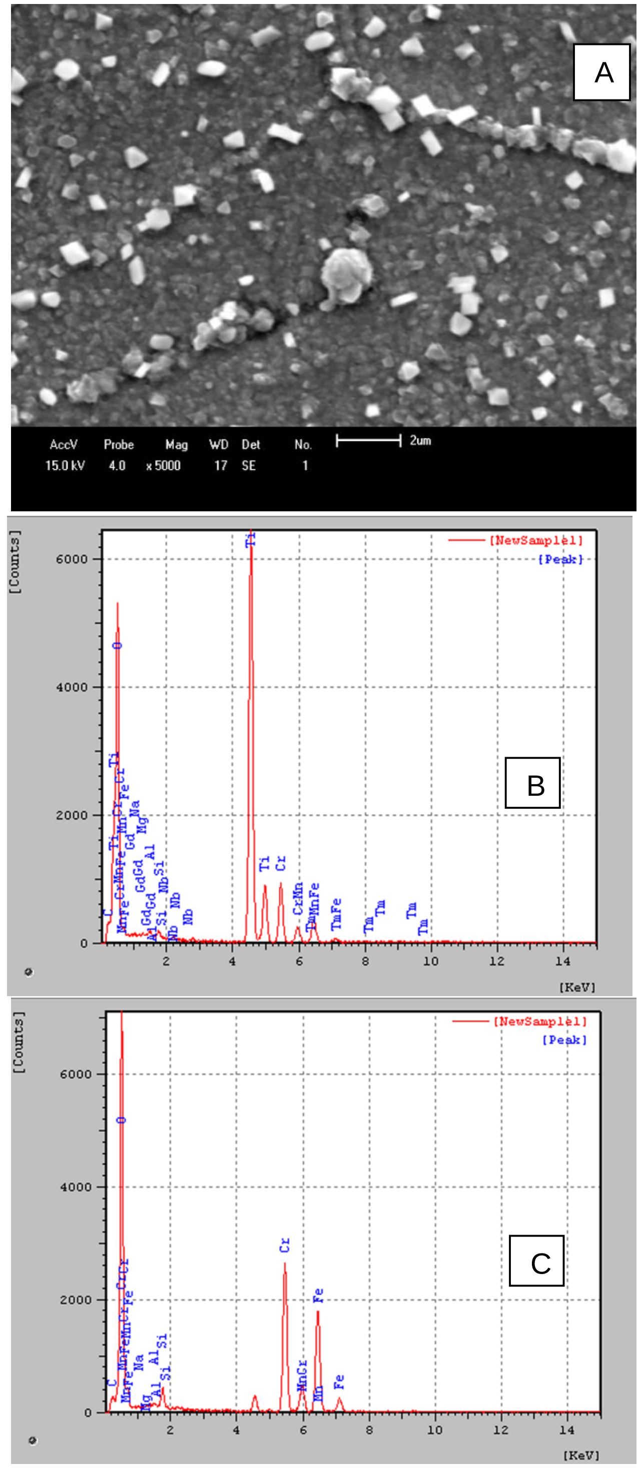

Figure 9 a) AISI 439 Steel, temperature 850°C, atmosphere: Synthetic Air; b). EDS - 850 °C, thermoblance-point 1; c). EDS_439 Steel, temperature 850 °C, atmosphere: Synthetic Air. thermoblance-point 2

Sand roses structure probably form rapidly and they do not last long. They are short-lived creations in geological terms and are very similar to the polymetallic nodules found on the deep ocean floor when concentric layers of iron and manganese hydroxides precipitate around seeds. Overall, these two quirks of nature, one continental and the other marine, are ephemeral geological processes that occur at the interface between water and sediments [15]. The same titanium oxide complex structure appears at a temperature of 850 °C when the steel AISI 439 is oxidized in Synthetic Air atmosphere.

The EDS in Figure 10 shows no difference is chemical composition of Point a in all samples because the film is very homogeneous.

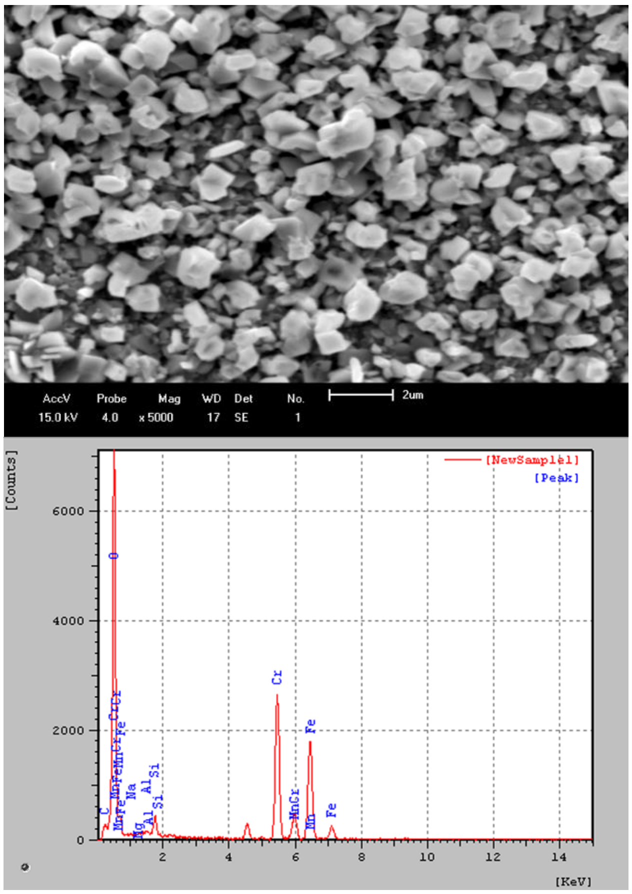

Figure 10 Microstructure and EDS of the oxide film formed on AISI 439 oxidized in air at 900 °C for 50 h, in thermobalance

With temperature increase to 950 °C an increase of some structures was observed as shown in Figure 11.

However, the EDS shows that there is no difference in chemical composition between points 1 and 2. An increase in the amount of Fe element relative to the previous EDS samples may have been caused by iron ions from the iron oxides formed at the beginning of the oxidation, which can spread through the chromium oxide film favoring the formation of phases containing iron, as observed by [2, 17].

The micrographs and spectrum shown in Figures 12 to 14 correspond to the image and chemical composition of oxide films formed at 850 °C, 900 °C and 950 °C for oxidation of the AISI 439 steel in air in Tubular furnace. These figures show that the surface morphology of stainless steel AISI 439 apparently does not change with temperature of 900 °C and 950 °C but change was notable at 850 °C. It could be observed that Titanium oxide appeared on the surface as presented in Figure 12. Similar results were obtained by Resende at al [1]. But Titanium oxide don´t appeared by Serra *et al*. [16].

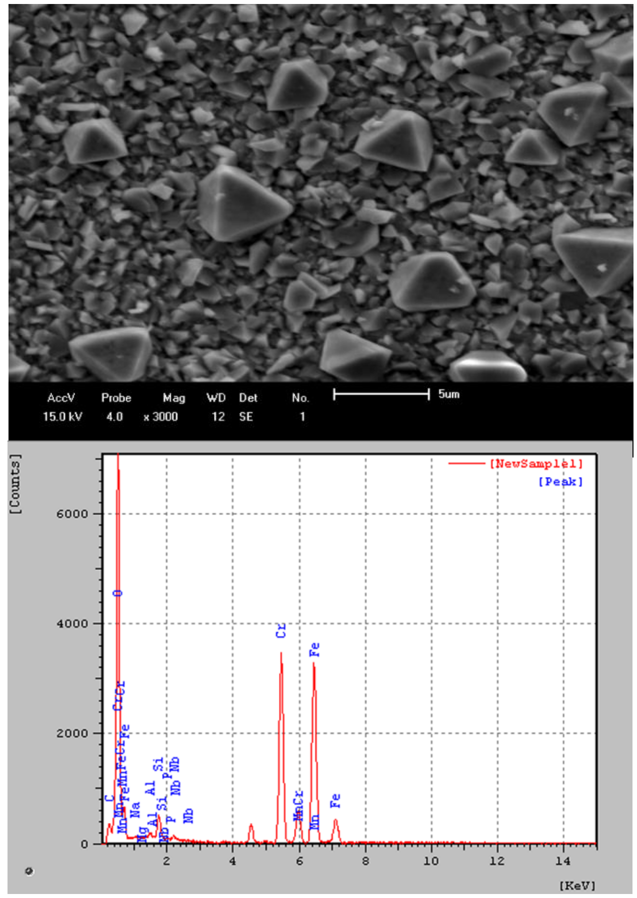

Figure 11 Microstructure and EDS of the oxide film formed on AISI 439 oxidized in air, at 950 °C for 50 h, in thermobalance

Figure 13 presents the surface morphology of AISI 439 oxidised at 900 °C over 50 h, and spectrum EDS with small facets are visible on the surface. The FeMn and CrMn peaks have been suggested and analyzed as spinel MnCrFe and MnCr phase. The oxidation layers were continuous, adherent to the metallic substrate, and mainly consist chromia (Cr_2O_3) [11].

As shown in Figure 14, after oxidation of the stainless steel AISI 439 at 950 ° C, there is formation of a ondulations (buckling) on the surface, which occur due to the detachment of the metal oxide film from the metal substrate. Similar results were obtained in the case of oxidation study on stainless steel 439 in various oxygen partial pressures [1]. The intensity of dominant peaks of Ti were reduced at 950 ˚C.

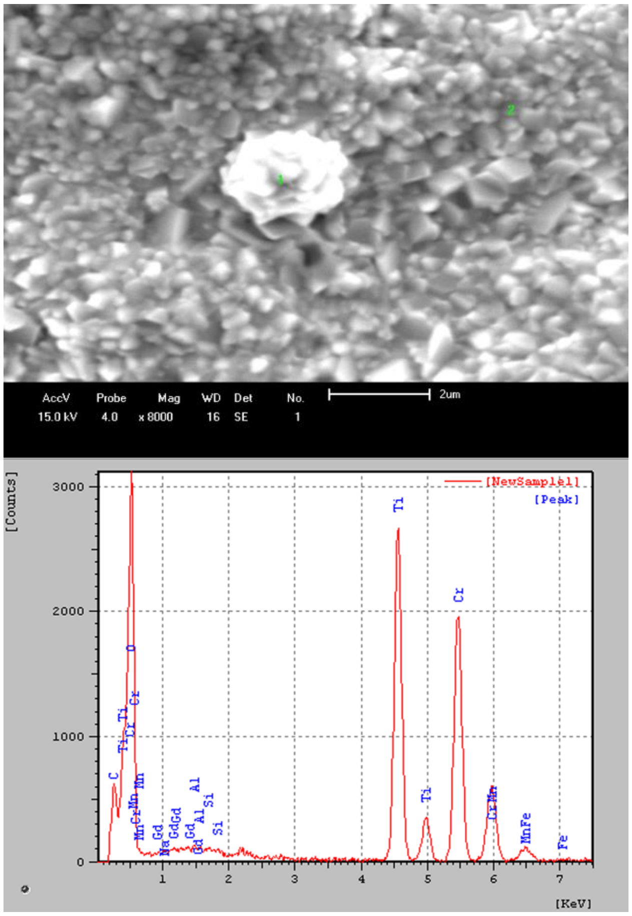

Figure 12 Surface morphology and EDS of AISI 439 at 850 °C

In synthetic air atmosphere, the oxide films formed are continuous with equiaxial grains. EDS showed that the composition of these particles was similar to that of the oxide scale, except at 850 °C which was found to be complex titanium oxide. For oxide film formed on the 439 steel, study indicates that the higher the temperature, the higher the Cr content. The Mn content in the oxide scale also increased as the temperature increases. On the other hand, the particle size was progressively enlarged as the temperature increases, which indicates that the detected $(Mn,Cr)_3O_4$ spinel is formed on the top layer such as [11]. At some points analyzed Cr and Mn were also found as shown in Figures 12, 13 and 14, which suggests the formation of $MnCr_2O_4$ spinel particles on the outer surface. The presence of Mn on chromia forming alloys was recently discussed by Sabioni *et al.* [20]. The presence of titanium and manganese was detected in the oxide scale at 850 °C, and these elements together with chrome produced mixed oxides in the structure as shown in Figure 12. The spinels play a decisive role in the quasi-ternary Cr-Mn-Ti oxide system [21, 27]. The spinel MnCr2O4 may be regarded as the central connection [21]. Part of the chromium can be replaced by trivalent titanium at low pressures, and the formation of a solid solution with the spinel Mn2TiO4 is possible in all cases [21]. The mixed oxides with titanium appeared more significantly in the film of the AISI 439 formed on samples oxidized at 850 °C. Based on their study of Mn diffusion in chromia films, they suggested that Mn-rich particles on the outer surface results from the initial oxidation, due to the fact that manganese affinity for oxygen is higher than chromium affinity. This is followed, in a second stage, by manganese ion diffusion through chromia towards the outer surface of the scale. The amount of Mn on the top layer of the film is limited by the small amount of Mn in the steel compared to chromium content, and may form a spinel oxide.

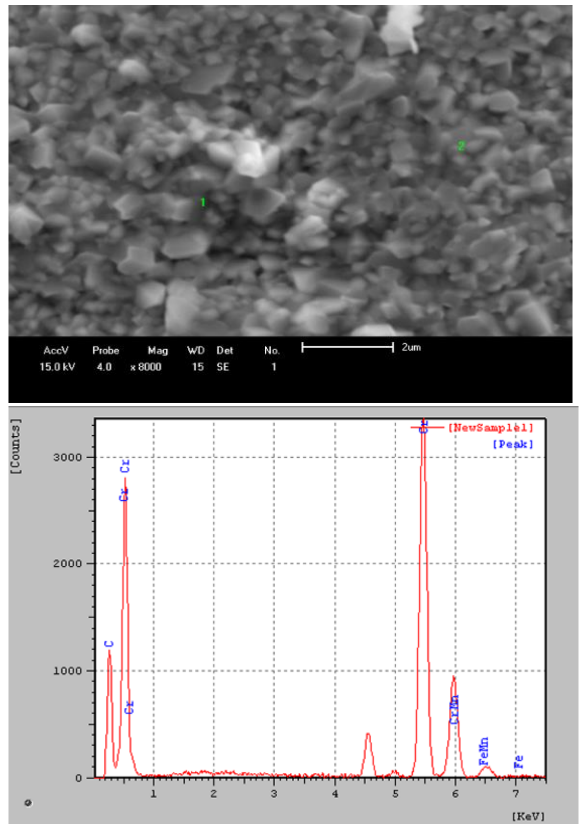

Figure 13 Surface morphology and EDS of AISI 439 at 900 °C

Chromia is the main metallic element in an oxide films and with the present of Mn, Ti, and Fe but in lower quantity. These four metallic elements are the components of the phases detected on oxide films as shown in Figures 9 to 14, considering that chromium oxide does not act as a barrier to manganese and iron diffusion. Manganese and Iron ions, from the manganese and iron oxides formed at the beginning of the oxidation, can spread through the chromium oxide film favoring the formation of phases containing manganese or iron, as observed by other researchers [2, 20]. The elements: Ti, Mn, Si, Cr and Fe were identified on the steel surface of all samples as shown in Figures 9 to 14 indicated the formation of chromia and spinels in the oxide layer according to literature [22, 23, 25] and important research was developed to study the metal/oxide interface at short high temperature oxidation duration and an oxidation model was proposed [24].

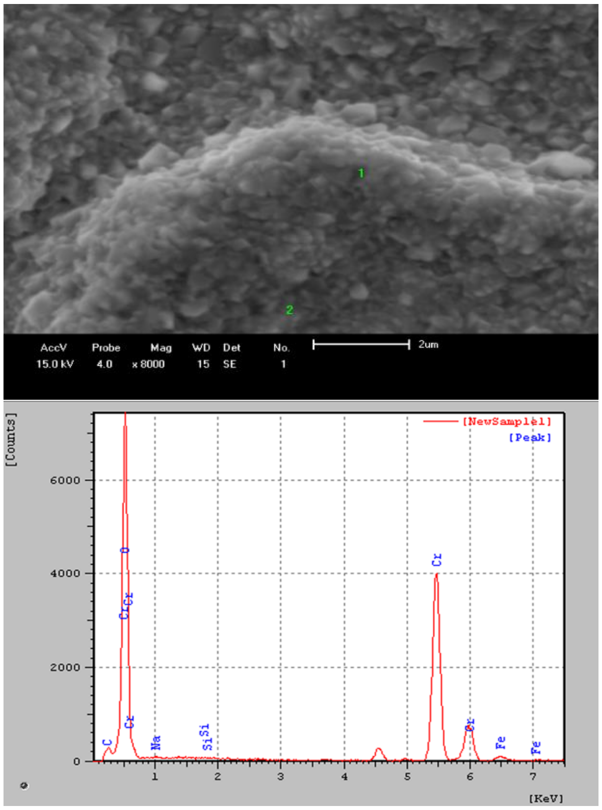

Figure 14 Surface morphology and EDS of AISI 439 at 950 °C

4.0 CONCLUSION

The AISI 439 ferritic stainless steel is resistant to oxidation at high temperatures due to the formation of a protective surface film of Cr_2O_3. In a tubular furnace and the thermogravimetric study of this steel, at temperatures of 850 and 950°C in Synthetic air, it was shown that the oxide film growth kinetics follow a parabolic law. According to Wagner's theory of oxidation, the growth of a protective oxide film according to a parabolic law indicates that the oxidation is controlled by the ionic diffusion, thus from the chromium and/or oxygen through the film.

At 850 °C, the AISI 439 steel presents better resistance towards oxidation. The oxidation kinetics of the steel occurs only in one stage between 850 °C and 950 °C. The parabolic constants of steel oxidated in tubular furnace are higher compared with those in Thermobalance, but the same reaction product is formed in the different equipment.

Analysis by EDS confirm Cr as the principal element in the oxide film, in proportions to form the chromium oxide (Cr_2O_3) and the stabilizer elements, such as: Mn and Ti forming complex oxides for both the furnaces used. At 850 °C it was revealed that the presence of the chemical element Ti indicating the formation of titanium oxide which for us sand rose structure.

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