Oxidation of 1020 steel in the abnormal glow discharge

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Abstract. 1020 steel is a material very used for surface treatment in the abnormal glow discharge. Because the composition of the gaseous atmosphere has an important influence on the results of plasma treatment, in this work the oxidation process of 1020 steel is verified on the abnormal glow discharge under different concentrations of air (20% to 100%) at temperatures of 600°C and 900°C. For each atmosphere used mass variation is measured during the process of surface oxidation, the structure and microstructure of the oxide film formed is observed and also its mechanical properties through its microhardness.

1. Introduction

Steel is one of the manufacturing and construction materials more versatile, more adaptable and of broad industrial use. At a relatively low price, steel combines the strength and the possibility of being worked, allowing various methods for manufacturing parts [1]. In addition, steel properties can be controlled according to specific needs by heat treatments, mechanical work or by varying their chemical composition [2]. The steel may be subjected to different thermochemical treatments in the plasma of an abnormal glow discharge (AGD), exploiting synergistic effects of photons, electrons, ions and free radicals presents in the AGD, which activate the plasma-surface interactions promoting its reactivity [3]. One of the processes that can be studied in the plasma of the AGD is the oxidation of steel which, under suitable conditions of temperature and oxygen partial pressure, as described in the phase diagram Fe-O [4], can form various iron oxides wustite (FeO), magnetite (Fe₃O₄) and hematite (Fe₂O₃). In this work the oxidation of 1020 steel [5] is made in the AGD in atmospheres containing varying concentrations of air at temperatures of 600°C and 900°C.

2. Methodology

1020 steel cylindrical samples of 10mm diameter and 4mm in height were sanded and then polished to obtain mirror shine following standard metallographic procedures for sample preparation [6]. Next, samples were placed for 40 minutes on the cathode of the AGD of pressure 2torr, 600°C, flows of the mixture of air and hydrogen of 2scc/s and of variable proportion of air from 20% to 100%. The confined anode geometry [7] was used as the electrode configuration and the discharge was initiated with a DC pulsed source. Mass variations (in percent) of these samples treated at 40 minutes, for each air content, were measured with a digital scale with a resolution of 0.0001g. Because changing the percentage of air of 60% for 40% a negative mass variation is observed, testing of these same concentrations of air at 900°C were performed for 24 minutes to verify this behaviour with changing temperature. Heating rates used were 100°C/min. Samples were analysed by XRD using cobalt K. radiation with wavelength corresponding to 1,789Å. Samples oxidized at 900°C was conducted to microhardness analysis.
3. Results
Maintaining a constant temperature of 600°C, treatment time constant in 40 minutes and the flow of the air-H₂ mixture also constant in 2scc/s, mass variation (Δm in percent) versus air content (in percent) in the gaseous mixture as shown in Figure 1 was obtained.

![Figure 1](image-url)

**Figure 1.** Sample mass variation as a function of air content (in percent) during the 1020 steel oxidation in AGD (Flow=2scc/s, T=600°C and t=40 minutes).

In Figure 1, mass loss in the samples is observed for low air contents. This is due to sputtering which is favoured by the large amount of hydrogen present in the atmosphere [8]. In an intermediate point between 40% and 60% of air content, a transition of the mass variation from loss to a gain occurs. This is due to the bigger iron oxides formation, in the sample surface, at bigger air content in the gaseous atmosphere. Subsequently, a maximum is observed in the mass gain of the sample corresponding to the least amount of hydrogen used and then it decreases in the absence of hydrogen in the gaseous mixture. This behaviour is due to a better efficiency of air discharges with small amounts of hydrogen as observed in other studies for nitrogen [9] and oxygen [10]. The behaviour of voltage and current, depending on the air content in the AGD during oxidation of 1020 steel under the conditions of Figure 1, is shown in Figure 2. In Figure 2, it can be seen that the voltage necessary to maintain the temperature of 600°C decreases with increasing air content in the discharge. This is due to increased heavier species that transferred their momentum to the cathode and to the presence of hydrogen that improves discharge efficiency by allowing the generation of more active species [11]. Conversely, the discharge current increases gradually with the air content to its maximum when the least amount of hydrogen to the gaseous atmosphere is supplied. In fact, as mentioned above by increasing the amount of air in the discharge heavy particles are increased and also the presence of hydrogen improves the efficiency of the same finding maximum efficiency for small amounts of hydrogen. In this case, one can observe the minimum current when the hydrogen of the gaseous mixture of the AGD is removed. Due to the change of sign of the mass variation from negative to positive for air concentrations of 40% and 60%, respectively (see Figure 1), the same tests are performed at a temperature of 900°C and t=24 minutes in order to verify the influence of temperature on this behaviour. Results of these tests are shown in Table 1. It can be seen (Table 1) the same behaviour present in Figure 1 where the variation of mass passes from loss for concentrations of 40% air to gain for air concentrations of 60%, due to the greater amount of iron oxides formed in the latter condition. Because it to increase the temperature of the sample was also necessary to increase the voltage of discharge (see Table 1), it is also obtained a greater mass loss due to sputtering with 40%
air in the atmosphere of the discharge with respect to seen in Figure 1, although the process be performed for a shorter time (24 minutes). In addition to the concentration of 60% air at the discharge, in Table 1 (at lower treatment time) a greater mass increase than that presented in Figure 1 is reported, which agrees with the endothermic nature of the reactions for the formation of iron oxides. [12].

![Figure 2](image)

**Figure 2.** AGD voltage and current versus air content during 1020 steel oxidation (P=2torr, Flow=2scc/s, T=600°C and t=40 minutes).

| Air(%) | Δm(%) | V(V) | I(mA) |
|-------|-------|------|-------|
| 60    | 0.019 | 665  | 157   |
| 40    | -0.021| 725  | 149   |

**Table 1.** Mass variation depending of air content during 1020 steel oxidation in the AGD (P=2torr, Flow=2scc/s, T=900°C and t=24 minutes).

Figure 3 shows micrographs at 1000X of 1020 steel samples oxide in AGD at 900°C for 24 minutes with air percentages of 40% and 60% in the Air + H₂ mixture of flow 2scc/s and pressure of 2torr. Micrographs of Figure 3 show a similar microstructure with larger deposits in the sample treated with 60% air in the gas atmosphere (see Figure 3(b)) due to the increased presence of oxygen interacting with the ferrous surface. In Figure 3(a) (40% air), however the sample having a mass loss (see Table 1) due to sputtering during the process, it has also been deposited a thin oxide film on the steel surface which is confirmed XRD as discussed below.

Diffractograms, using K. cobalt line (λ=1,789 Å), of 1020 steel before subjected to oxidation in AGD and after oxidation at 900°C for 24 minutes, 2torr pressure and flow rate of 2scc/s of Air+H₂ atmosphere with air percentages 40% and 60% are shown in Figure 4. In diffractograms of samples subjected to oxidation in the AGD with air concentrations of 40% and 60% (diffractograms of Figure 4(c) and 4(b), respectively), besides the peaks corresponding to 1020 steel (diffractogram of Figure 4(a)) present peaks associated with iron oxides Fe₃O₄ and Fe₂O₃ [13], with the absence of the corresponding FeO phase as reported for iron oxidation at 150°C in atmosphere of high purity oxygen [14]. In another study the formation of the three iron oxides (FeO, Fe₃O₄ and Fe₂O₃) at a temperature
of 400°C and the formation of single Fe₃O₄ and Fe₂O₃ at a temperature of 600°C was reported [15]. Consistent with the results obtained for the oxidation of 1020 steel in AGD at 900°C, presented here, the formation of only iron oxides Fe₃O₄ and Fe₂O₃ was obtained, under the pressure conditions, gaseous atmosphere and time used.

**Figure 3.** Micrographs at 1000X of 1020 steel samples subjected to the AGD at 900°C for 24 minutes with total flow of 2scc/s, 2torr of pressure of air +H₂ atmosphere with air contents of: (a) 40% (b) 60%.

**Figure 4.** DRX with K, cobalt line (λ=1.789Å), of 1020 steel (a) and of 1020 steel after AGD oxidation at 900°C during 24 minutes, at pressure 2torr and total flow of 2scc/s of the air +H₂ atmosphere with air contents of 60% (b) and 40% (c).
Table 2 shows microhardness measures (HV), made with an applied load of 25gf, depending on the percentage of air for the oxidation of 1020 steel in the AGD at temperature of 900°C (Flow=2scc/s and t=24 minutes). The measurements represent the average of 5 measurements made on the sample surface. In this table the value of microhardness for 1020 steel before subjected to oxidation treatment in the AGD is also included.

| Air (%) | Microhardness (HV) | Deviation (HV) |
|---------|--------------------|----------------|
| 1020 steel | 130 | 4.0 |
| 60 | 415 | 120 |
| 40 | 580 | 17 |

Microhardness of 1020 steel is enhanced by the oxide formed on its surface as can be seen in Table 2. Microhardness of samples for AGD with air contents of 60% and 40% is in the range characteristic for magnetite phase [16,17]. Although also hematite (Fe₂O₃) is formed on the steel surface in both air concentrations (see Figure 4), the microhardness does not correspond to that of this phase [18] which could be due to non-formation of a consistent hematite layer due, to reduced treatment time of the samples in the AGD (24 minutes).

4. Conclusions
The 1020 steel surface oxidation has been made in the AGD with different air contents in the air + H₂ atmosphere at pressure of 2torr at temperatures of 600°C and 900°C. First, the change in mass of the samples increases with the content of air in the gaseous atmosphere finding a transition from mass loss to a mass gain in the range between 40% and 60% air, also it reaches a maximum when the least amount of hydrogen was used in the atmosphere and then decrease when the content of hydrogen in the gaseous atmosphere (100% air) is removed. Secondly, the potential to maintain the temperature of the cathode discharge decreases with air content in the atmosphere, namely, with the amount of heavy species in the gaseous atmosphere. On the other hand, the discharge current exhibits the same behaviour, as a function of air content, that the mass variation, which is increased with small amounts of hydrogen in the gaseous atmosphere. Finally, the surface of samples treated with air contents between 40% and 60%, have a similar structure and microstructure, constituted by both iron oxides corresponding to magnetite (Fe₃O₄) and hematite (Fe₂O₃) phases. The microhardness measured in these two types of samples is also similar and corresponds to the accepted value for magnetite.

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