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Abstract. *When a solid is immersed in plasma, two different types of energy transfer occur: by radiation and collision of the particles. In the first case, the energy is transferred uniformly and parallel to the surface. In the second case, it is transferred to the vicinity of the micro regions where the thermal spikes are generated. To study these two effects, samples of the steel AISI M35, whose values hardness are strongly sensitive to the temperature tempering, were used as micro thermal sensors. By correlating values hardness with temperature, it was possible to obtain the thermal profile of the samples steel AISI M35 treated by plasma. It was verified that plasma treated samples presented layer in the surface and in the region in contact with sample port, which is not present in the samples tempered in resistive furnace. Furthermore, it has been verified that the conditions that showed the higher thermal gradient in the region exposed by the plasma, were the parallel discs treated by configuration in hollow cathode with a pressure of 2 mbar.*

Keywords: *thermal gradient, hollow cathode, rapid steel AISI M35, plasma.*

1. INTRODUCTION

Among the new energy sources for materials processing, the plasma has distinguished itself as an alternative to the industry due to its versatility. In certain applications such as in the steel industry (smelting, reduction in the oxides), metallurgy (thermochemical treatments, sintering, welding and cutting) or even in the processing of the hazardous waste (waste organometallic, electroplating sludge, sludge of the aluminum production, incineration ashes and hospital waste), the understanding in the form of the heating by plasma and their effect about the microstructure and properties in the materials thus treated are crucial to the development of the materials by this new technology. (Yamazaki, Risbud *et al.*, 1996). However, the modeling mathematical and measurements experimental to a real situation of the heating of solid immersed in plasma, is very complex. We know that in the plasma, the energy transfer occurs by radiation of the plasma to the solid surface, too as from the thermal peaks created in the surface due to particle bombardment. (Kersten, Deutsch et al., 2001).

This way, it is expected that the plasma-treated materials causes the appearance thermal peaks, differently from other conventional forms of heating. Is know of literature review that in the region heated by plasma produces a change in the microstructure by promoting alterations in the hardness and mechanical strength, once the plasma is capable of producing high heating rates of the treated parts (Akamatsu e Yatsuzuka, 2003).

It was observed that a gradient of the porosity was obtained in compact samples in the powder microalloyed of the aluminum bronze, from the surface to the interior region (Alves C, Hajek et al., 2003). The explanation given for this phenomenon, was the local fusion of the powder particle, produced by generated thermal peaks, and the consequent percolation for the internal pores, thus increasing the surface porosity. The literature shows that, depending in the configuration of the plasma, the effect thermal gradient can be more accentuated (Galvão, Costa et al., 2008). When the geometric characteristic of the conventional glow discharge (configuration planar) is modified to configuration hollow cathode, the ionization rate is increased in more than 100 times, generating a plasma with high collision rate (Barankova, Bardos et al., 1996). The samples treated with this configuration depend of distance between cathodes, gas pressure and voltage (Alves Jr, Hajek et al., 2003). Although there are records about the thermal gradient generated in solids immersed in plasma, little is known about these values.The temperature measurement local is the largest obstacle to the quantification.

The present work proposes a methodology to determine the temperature in different micro-regions and with that obtain the thermal profile. Is know that some quenched steels have a hardness highly sensitive to the tempered temperature. Among these steels one can cite the steel AISI M35. This steel when quenched and tempered in resistive furnace, forms a curve in the sigmoidal behavior of the hardness measure with relation to the tempered temperature. This occurs because the finely dispersed carbides existing in the quenched structure pass through a coalescence process during the tempered. The tempered process occurs due to structural changes thermally sensitive, and that the time for that to occur in this steel is around 2 hours (Bayer, Becherer e T, 1989; Bayer e Walton, 1990). That is, even if the process was reversible, the time for cooling to room temperature is less than the time of tempering. It turns out that in the tempering, the structure that will remain is that which was obtained with the highest temperature.

Thus, in this work we used samples of the steel AISI M35 to determine from the microhardness profile when immersed in plasma, and indirectly, determine the temperature profile.

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2. EXPERIMENTAL METHODOLOGY

The probe used to determine the thermal profile was constructed with the AISI M35 steel discs (diameter D of 33.45 mm and height H of 1 mm). The disks were initially heated until the temperature 1230 °C, followed by rapid cooling (quench). Then, part of the samples were tempered in resistive furnace with temperatures 550, 580, 610, 640, 700 and 750 °C to obtain the graph of the hardness versus tempering temperature. The results shown in this graph, Fig. 3, were used to determine the Eq. 1 and this allowed obtain the temperature profile of the steel discs immersed in plasma, from the hardness profile. The samples immersed in plasma also were previously tempered under the same conditions. Two types of the configurations were used, planar cathode discharge (DCP) and hollow cathode discharge (DCO). In figure 1 is shown the plasma equipment used and the two configurations detailed.

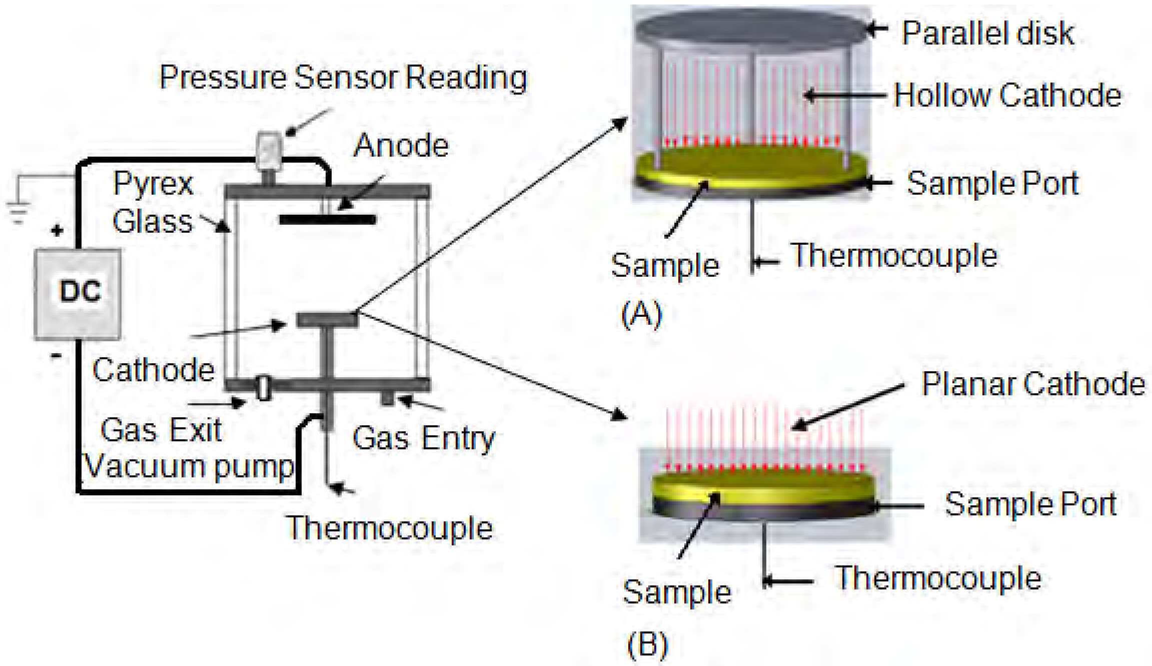

Figure 1 - Schematic plasma equipment used for tempered the steel discs of quenched steel AISI M35. (A) Configuration Hollow Cathode, (B) Configuration Planar cathode.

The cylindrical reactor of the borosilicate, posses a height in the 300 mm and diameter in the 180 mm. It is closed by two stainless steel flanges, sealed by gaskets of the nitrile-profile L. In the top flange is the connection the anode, pressure sensor and a device for breaking of the vacuum. In the bottom flange is inserted the cathode, thermocouple and input and output of the gases. The cathode is composed of the tube stainless steel, by where passes a thermocouple, in your extremity has a platform what supported the ceramic structure. For the planar configuration is placed only one table (platform of supported) of the stainless steel, with a diameter (33.45 mm) that serve as a sample port. For the configuration hollow cathode is added in parallel disk to the sample port, with the same diameter, supported by a tripod of wire (1.2 mm thickness), then forming a hollow cathode with distance between cathode of 12 mm. In this configuration the sample is placed between the two cathodes. The use of this parallel disk (configuration hollow cathode) increases the ion density, making more effective the transfer of the energy by collision.

To revenir the samples in plasma was considered the temperature of the thermocouple fixed in the sample port as reference. Was used for tempering the reference temperature of the 550 ° C, in argon atmosphere and pressure of 4 mbar for sample treated without confinement. In the configuration hollow cathode was used pressure of 2 mbar and also of 4 mbar. The time of the tempering in all the conditions lasted 2 h.

After of the tempering in plasma, microhardness essays were performed in cross section of the samples, as well too the tempering samples in furnace resistive. The prints were made in the samples using a load of the 10 gf, at form equidistantly from one another, with spaced in the 0.02 mm in the vertically and in the horizontally, from the edge to the center of the sample, as shown in Figure 2.

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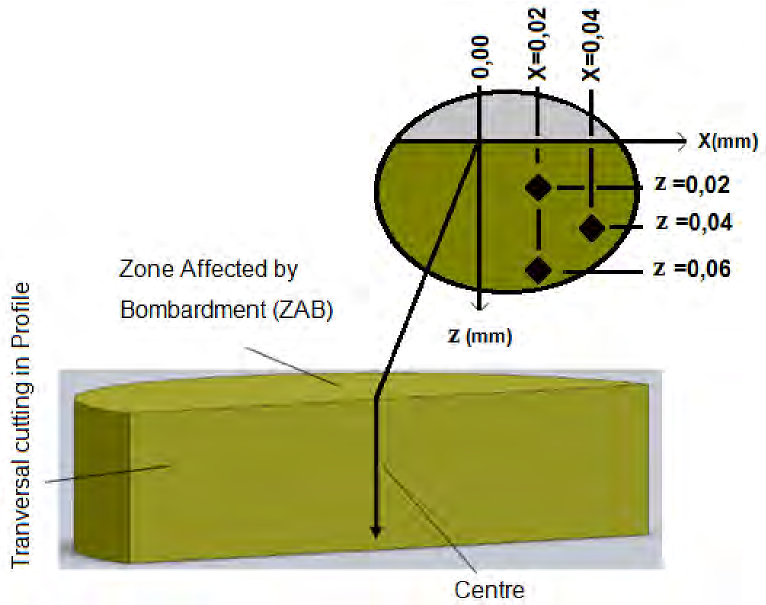

Figure 2 – Representation of the profile cutting of the sample, indicating the region and the direction, in which the microhardness essays were performed.

From the obtained hardness values, these are inserted in the graph temperature versus hardness. This allows obtained a approximate function called of calibration function, as shown in Figure 3, allowing to determine indirectly, the temperature values to construct the thermal profile of the samples tempered in plasma. Were calculated the values average hardness of the samples tempered in resistive furnace for different tempering temperatures the 550, 580, 610, 640, 700 and 750 °C, originated a function of the hardness versus tempered. This function was used to obtain Eq. 1 and it was determined the temperature reached in the samples during the plasma treatments.

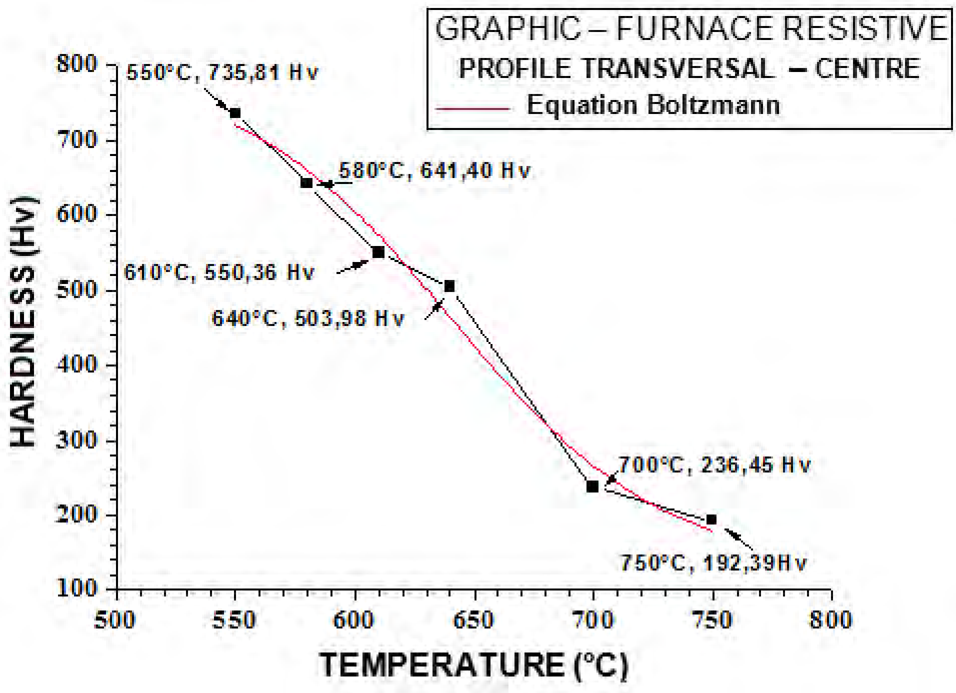

Figure 3 - Curve hardness versus tempering temperature for samples treated in resistive furnace.

An adjustment of the curve was realized through the trend line sigmoidal, based in the Boltzmann model given by the equation:

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$$T(°C) = Ln\left(\frac{(A1 - A2)}{D(Hv) - A2} - 1\right) d_X + x_0 \qquad (1)$$

Where T(°C) is the tempered temperature, D(Hv) represents the Value of the Hardness applied; dx is the width of the track or level scattering where occurs the largest variations in hardness; A1 represents the lower asymptotes; A2 is the top asymptotes and X0 represents the inflection point of the graph.

3. RESULTS AND DISCUSSION

3.1. Hardness and Temperature Profile

The behavior of the hardness profile with the depth of the tempered samples in plasma was performed and perceived that, unlike the uniformity found in the tempered samples in furnace resistive, there was a significant variation of the hardness value along the depth of the samples. Generally these samples immersed in plasma showed values of the hardness in surface smaller than in the interior. The figure 4 shows the hardness profiles for the samples treated in plasma in the configuration hollow cathode for 2 mbar (Fig. 4a) and 4 mbar (Fig. 4b), as well the hardness profile for the parallel disks the samples (5c and 5d) respectively.

As can be seen in both samples and parallel disks, hardness increases from surface to interior and then decrease. In the opposite side the same behavior occurs. Comparing this configuration with the planar cathode, to pressure of 4 mbar, it is observed that the gradient of the hardness was less intense than in those samples treated hollow cathode (fig. 6).

As the hardness is related with the sample temperature (Eq. 1), we can obtain the graph of the thermal profile of the samples immersed in plasma. They were thus constructed, from the curves of the figure 4,5 and with the aid of the Eq. 1, the curves of Figure 7.

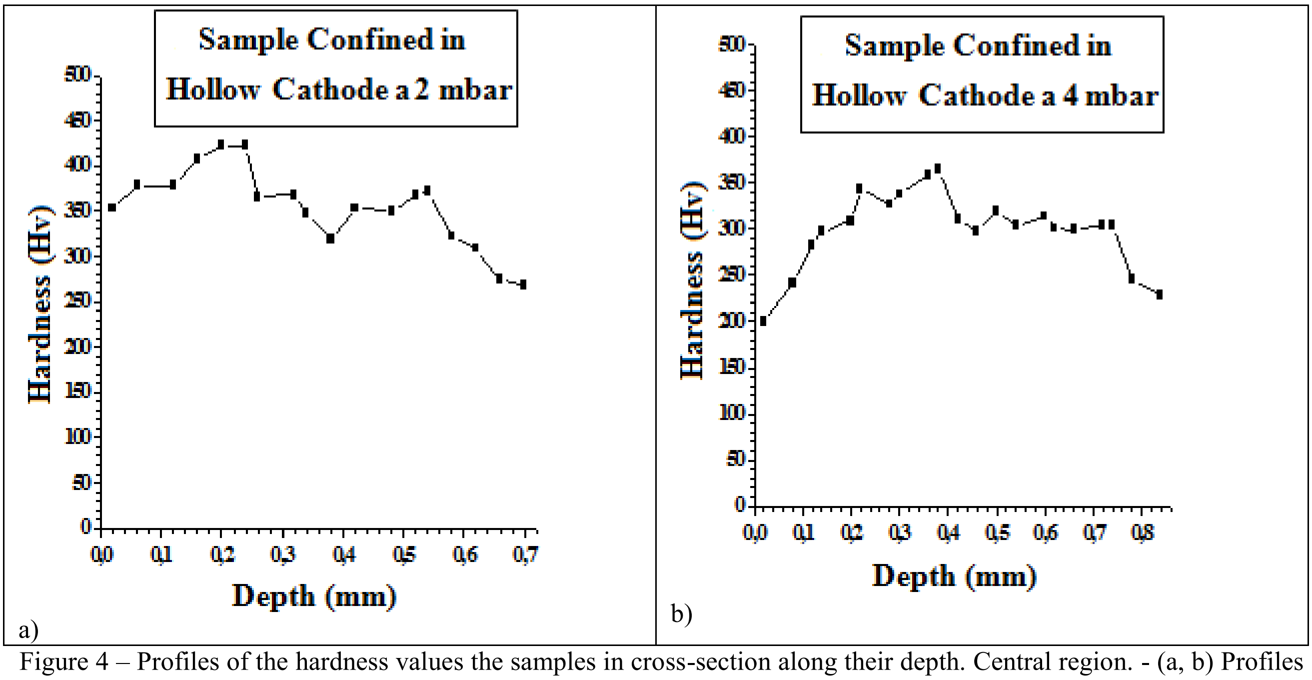

Figure 4 – Profiles of the hardness values the samples in cross-section along their depth. Central region. - (a, b) Profiles samples tempered in plasma by configuration hollow cathode in a pressure of 2 and 4 mbar, respectively.

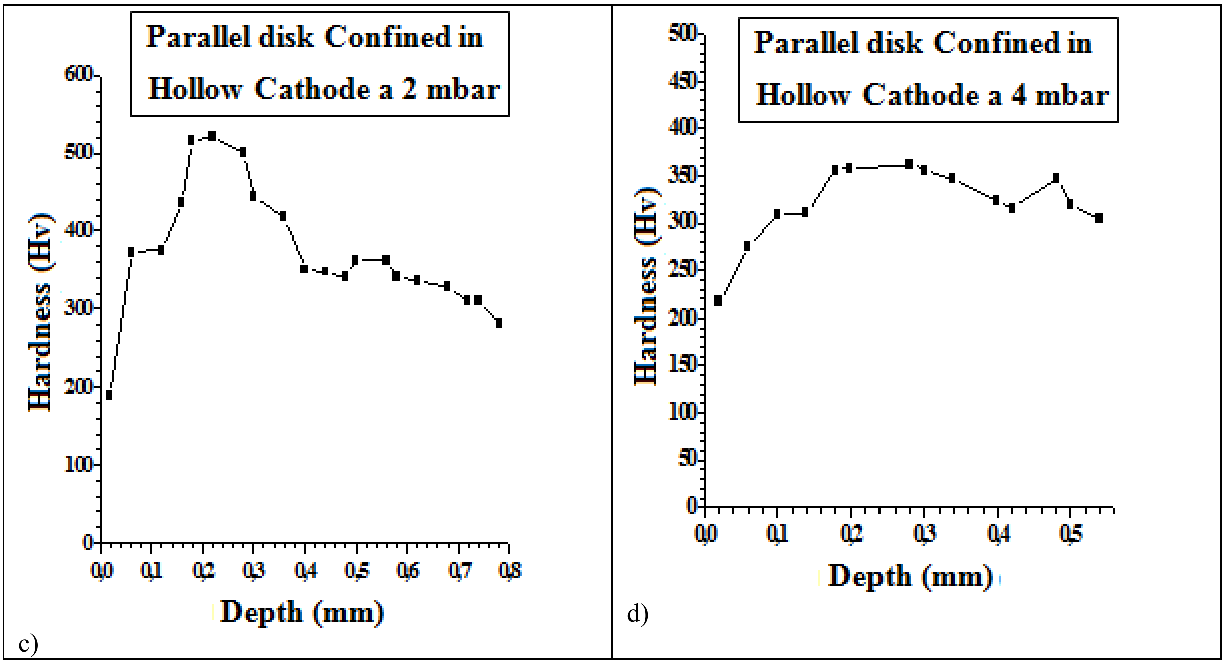

Figure 5 – Profiles of the hardness values the samples in cross-section along their depth. Central region. - (c, d) Profiles of the disks parallel tempered in plasma by configuration hollow cathode at a pressure of 2 and 4 mbar, respectively.

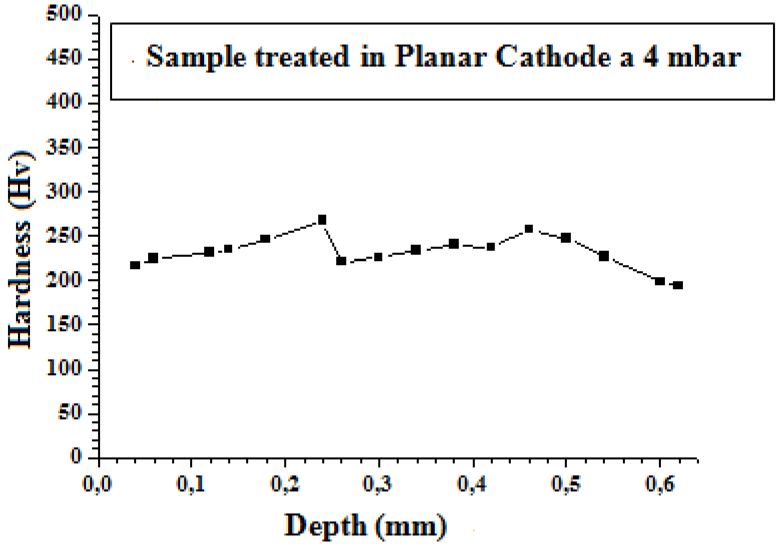

Figure 6 - Profile of hardness in the cross section of the sample tempered in plasma by cathode planar configuration, in the pressure of 4 mbar along their depth (central region).

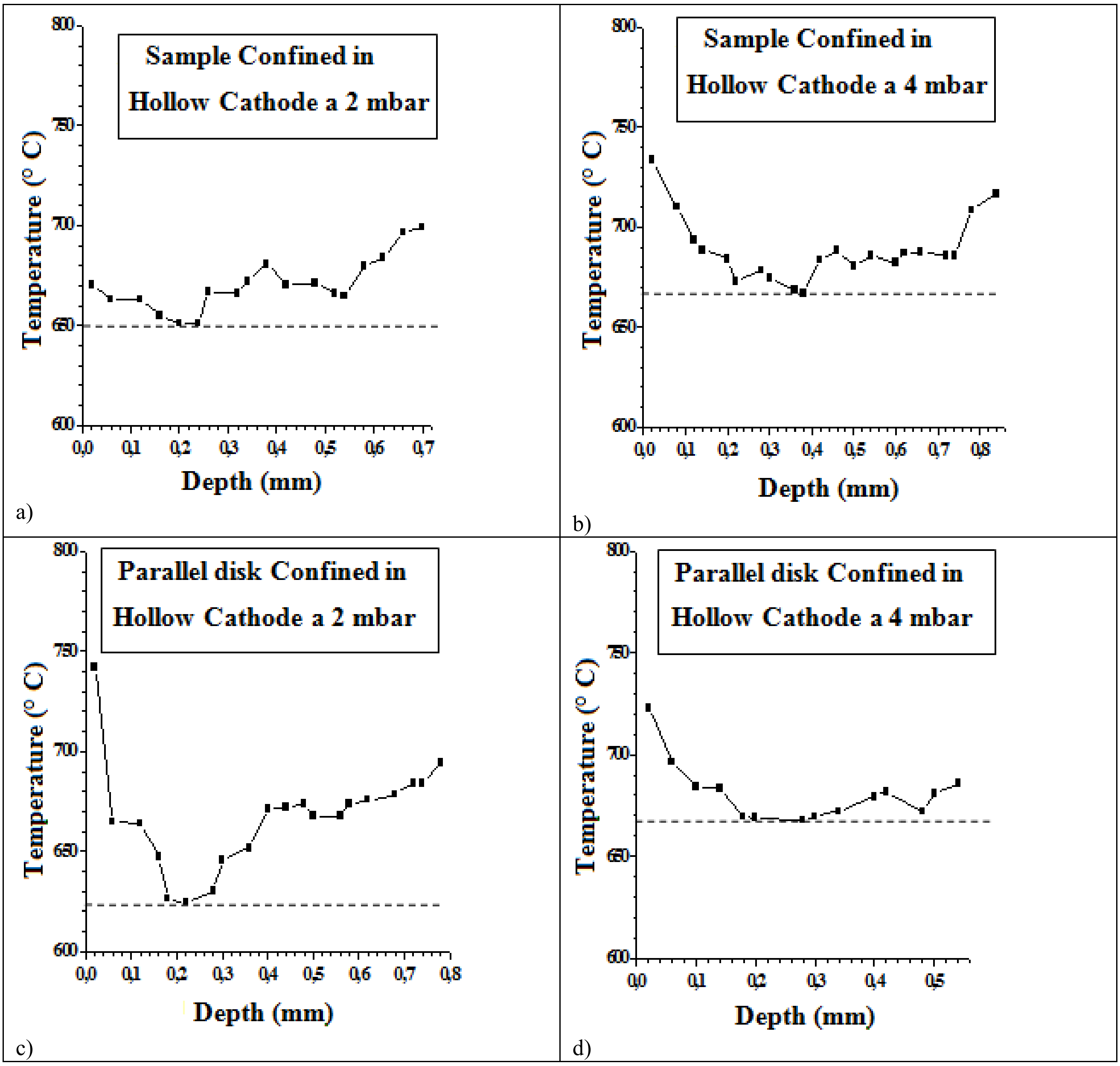

Figure 7 - Profiles of temperature values the samples in cross-section along their depth. Central region. - (a, b) Profiles of samples tempered in plasma by hollow cathode configuration in a pressure of 2 and 4 mbar, respectively. - (c, d) Profiles of disks parallel tempered in plasma by configuration hollow cathode at a pressure of 2 and 4 mbar, respectively.

These graphs indicate that there was a variation in temperature between the surface and the center of the samples. For the case of the sample tempered in the configuration hollow cathode in 2 mbar, as shown in Figure 7 (a), showed a temperature variation of the 670.37 ° C in the surface exposed in the plasma and 651.03 ° C in the center of steel disc.

Already of the sample tempered in the configuration hollow cathode, in the pressure of 4 mbar, as shown in Figure 7 (b), was found that the temperature values in the surface exposed to the plasma was of 733.56 ° C, and 668 60 ° C in the center of the sample. These phenomena can be explained due the effect of the ion bombardment in the surface of the material treated in plasma. Is known that in the plasma, the heat transfer to the surface if does primarily by radiation and by ions bombardment and energetic neutral species (Kersten, Steffen et al., 1995). In the process of the bombardment, each collision produces a peak termic, being after diffused to the neighborhood (Kersten, Steffen et al., 1995). Depending of the rate collision and of the thermal conductivity of the materials, the characteristics of the layer may vary.

For samples treated by discharge in configuration hollow cathode, due the plasma confinement, the rate of the ionization will be greater, resulting in higher rate of the bombardment. For lower pressures, the energy by ion will be higher, implying higher thermal peaks and consequently higher thermal gradient. But it did not happen when

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comparing the samples treated in 4 mbar and 2 mbar. It is noted that the sample treated at 4 mbar has a higher gradient than that treated at 2 mbar. Analyzing the graph of the temperature of the latter sample, it is found that the surface in contact with the sample port has a higher temperature than the surface exposed to the plasma. Once the surface is protected from ion bombardment occurs and heat flux from the plasma to the outside through of the sample port, it was expected that the temperature lower than or equal to that of the interior of the sample.

Carefully observing the micrograph in this surface (Fig. 8), is possible verify the presence of the points that suggest micro mergers and that may explain the observed phenomena. As the surface is not completely flat and the contact with the sample port is not complete, the contact points are subject the high voltages that generate micro-arcs, which can generate a region of the fusion local around in point of the contact (Kudryavtsev, Parton et al., Solids Mech 1982). The presence of the micro arc also explains why the temperature at the surface exposed to plasma of the sample in 4 mbar was greater that 2 mbar. The micro arc occur when contact between two surfaces is not perfect, only a few points in the surface are in contact with the sample holder. This means that the sample this a potential lower than the cathode, which reduces the collision rate of the ions to the surface, resulting in a lower temperature.

To validate this hypothesis we analyzed the thermal profile of the parallel disks, as shown in Fig. 7c and 7d, since these discs have no contact with the sample port and, therefore, must be free of the such micro-arcs. One of the surfaces is exposed in plasma with ion high density (inner side of the hollow cathode) and the other surface exposed in plasma the ion low density. For the pressure of 4mbar (Fig. 7d), verified that the temperature is highest of the surface exposed in the plasma, being of 723 ° C and decreasing to 667 ° C inside in the disc, and then increased to 685 ° C in the opposite surface. For the Parallel disk tempered in 2 mbar (Fig. 7c), verified a similar behavior but with higher thermal gradients. Contrary to what happened to the samples on the sample port, these results are coherent. The region irradiated and bombed by plasma in the configuration hollow cathode (configuration with high ion density) reached a highest temperature that interior the disc. The other side of the disc, the surface exposed the plasma in planar configuration (low ion density), the temperature was higher than that in the interior, because this surface of the sample is irradiated and bombed by the plasma species planar. As this configuration has a inferior ion density that the first, the surface temperature is lower than that exposed of the plasma in the configuration hollow cathode.

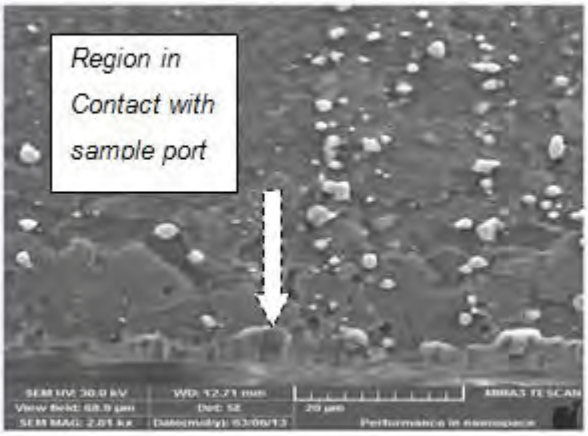

Figure 8 - Micrograph of the sample tempered in plasma and confined at a pressure of 2 mbar showing micro welds obtained with the scanning electron microscope (SEM). Increase of 2000 times.

The other situation studied was of the samples exposed the plasma with low ion density (planar configuration). In the Figure 9 is shown a thermal profile obtained from of the Fig. 6, using Eq. 1. It refers the sample tempered in 4 mbar, in the planar configuration. Found that the temperature difference between the surface and the interior of the sample is in approximately 20°C, that is approximately the value found for the exposed side of the disc parallel to the planar plasma in 4 mbar (Fig. 7d).

A summary of the results of the thermal profile obtained in samples heat treated plasma with different discharge configurations can be shown through of the Fig. 10. This mapping was realized through of the values of the result inserted for the temperature T, of the plasma treated samples along in the depth (z), swept in a certain distance arbitrary (x), shown in the form of the color gradient.

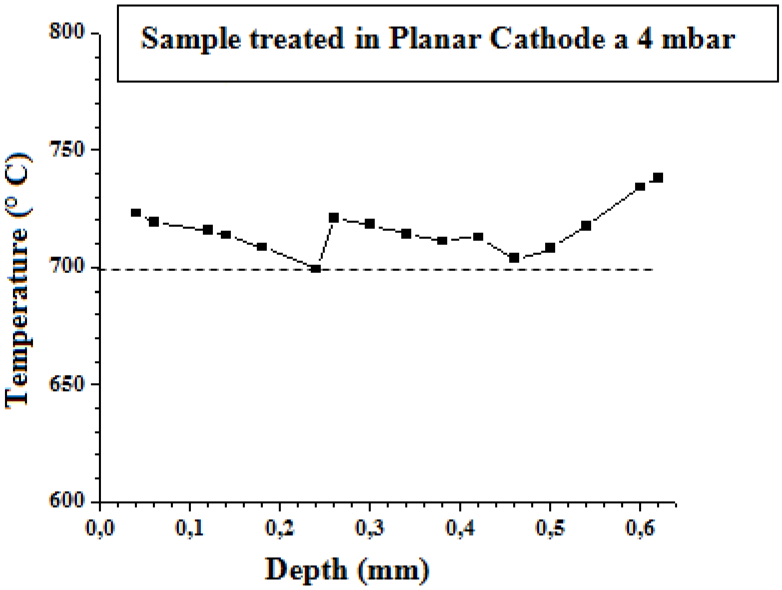

Figure 9 - Thermal profile in the cross section of the sample tempered in plasma by cathode planar configuration, the pressure of 4 mbar along their depth. Central region.

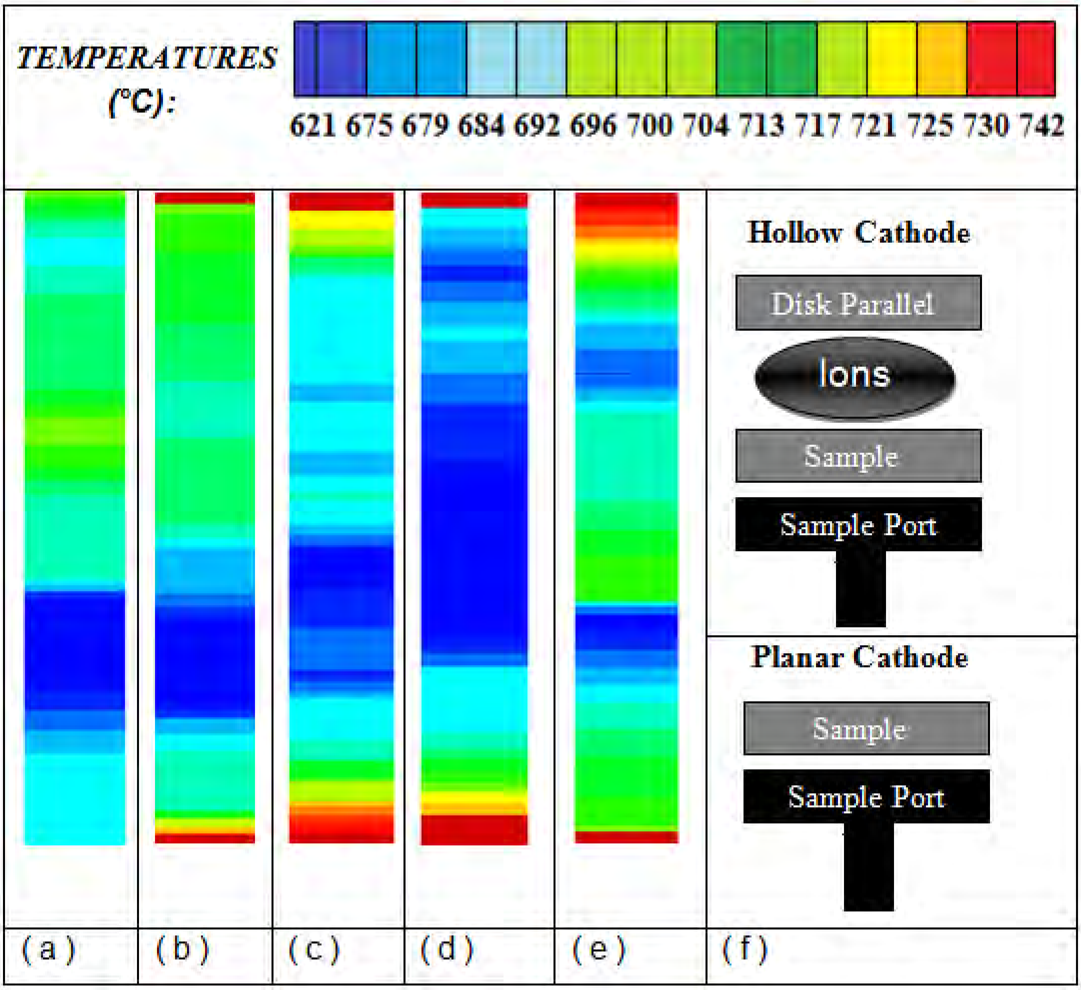

Figure 10 - Behaviour of the temperature profile along the cross section of the samples treated in plasma, where: (a) Sample confined the 2mbar, (b) parallel disc confined the 2mbar (c) Sample confined the 4mbar (d) parallel disc confined the 4mbar (e) Sample treated in planar plasma the 4mbar.

4. CONCLUSIONS

The tempered samples in plasma showed layers in surface and in the region in contact with sample port containing a high rate of the thermal heating. The presence these rates contributed to changes in microstructure and in the hardness of the samples. These differences, were not observed in the samples tempered in resistive furnace along of the your transverse section. The microhardness values in samples tempered in plasma varied considerably between the region exposed to plasma, as well in the region in contact with the sample port, with the your material centre.

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