STUDY OF THE INFLUENCE OF ELECTRICAL AND PLASMA PARAMETERS ON NIOBIUM ANNEALING

Nícolas Virgilli Guimarães, virgilli@inpi.gov.br
Sílvio Francisco Brunatto, brunatto@ufpr.br
Universidade Federal do Paraná - Departamento de Engenharia Mecânica.
Cx. 19011, CEP 81531-990, Centro Politécnico (Campus II), Bairro Jardim das Américas, Curitiba, PR, Brazil.

Abstract. *This work comprises a study of the influence of electrical and plasma parameters on niobium annealing. The study characterized to utilize plasma devices to annealing treatments of niobium metal samples at 900 ºC and 60 minutes, in 90% reduction cold-rolled, were performed in heating using hollow cathode discharge configuration where the sample's heating is performed by means of the ion bombardment in an annular glow discharge. The annealing treatments of niobium metal utilizing plasma devices and the influence of electrical and plasma parameters on niobium recrystallization were investigated. Samples micro structure was characterized considering the rolling direction and the cross-section of the specimens. The recrystallization rate was determined confronting the hardness and the softening rating of the annealed and cold-worked samples. Results indicate that electrical and plasma parameters on niobium recrystallization have influenced in the recrystallization process and the ion bombardment plays an important role in the recrystallization process activation.*

Keywords: Niobium, Plasma Annealing; Hollow Cathode Discharge; DC Abnormal Glow Discharge.

1. INTRODUCTION

Abnormal glow discharges have been applied in various metallurgical processes, as plasma nitriding (Shohet, 1991) and Hirvonen *et al.* (1996) and plasma sintering Muzart *et al.* (1997) and Batista *et al.* (1998). Electric discharges with annular cathode cavities lead to the effect known as hollow cathode discharge (Timanyuk and Tkachenko, 1989). Taking advantage of the high ionization rate, hollow cathode discharges of gas mixtures containing hydrogen, nitrogen and methane were also used to surface treat steels in carburizing or carbonitriding Terakado *et al.* (1996) and nitriding Benda *et al.* (1997). This discharge configuration has aroused in a growing technological interest, inasmuch as it can be utilized not only as an alternative processing technique of surface treatments but also in new applications and developments, being the case of hollow cathode discharge sputtering device for uniform large areas thin film deposition Koch *et al.* (1991).

The abnormal glow discharge containing hydrogen and argon for sintering purposes is characterized by full covering of the cathode by the glow region (Chapman, 1980), supplying an uniform treatment. The principle of heating is based on the ion and fast neutrals bombardment of the sample. A negatively biased voltage was applied to the sample, which worked as the cathode of the abnormal glow discharge, generating an electric field in the cathode sheath, where ions are strongly accelerated. Collisions between ions and argon atoms or hydrogen molecules of the gas discharge in the cathode sheath result in a flow of fast neutrals toward the cathode. The bombardment of ions and fast neutrals heat the sample (cathode). The modified linear abnormal glow discharge, using the hollow cathode configuration, also results in the bombardment of the cathode by ions and fast neutrals, consequently causing its heating. Using the hollow cathode geometry, the ionization rate is higher than that of the linear abnormal glow discharge. According to Engel (Engel, 1994), the ionization rate of the former is around ten times higher than that of the latter considering an argon discharge generated at 300 V, under 100 Pa in a configuration whose distance inter-cathode is 5 mm. As a result, an increased heating efficiency is obtained in the same proportion as compared to the linear discharge.

In the photography of the Fig. 1, a weak luminosity corresponding to the linear discharge can be observed on the outside of the external (hollow) cathode and an intense glow in the inter-cathode region, which resulted from the high ionization rate, attained in the hollow cathode configuration (see the annular discharge). This configuration produces a plasma-confined geometry and has been used as an efficient process for sintering pressed samples, which are placed in the central cathode Brunatto *et al.* (2001, 2003 and 2005a).

In recent work Brunatto *et al.* (2005b), a strong grain growth effect in the iron sample processed in hollow cathode discharge was observed. This behavior could be related to the activation of the sintering mechanisms when the plasma sintering technique is utilized. In the same way, comparatively to the conventional sintering process, it was observed in Batista *et al.* (1998) a more effective pore rounding effect, for iron samples sintered in linear plasma. In the attempt to explain this result, the authors suggest a possible additional material transport mechanism improving the diffusion in the powder particles contact region of the compact ones (or, in the necks). Such mechanism would be attributed to the propagation of phonons along the material, being then produced by transference of momentum as a consequence of the sample bombardment by the plasma species. The hypothesis presented in Batista *et al.* (1998), *id est* the propagation of phonons, allied to the mechanism of disappearance of grain boundaries Peterson (1983) and Berry *et al.* (1991), could be also valid in the attempt to explain the strong effect of the grain growth verified in the iron samples sintered in hollow cathode discharge, in Brunatto *et al.* (2005a).

So, an alternative annealing process based in the use of plasma could be expected to perform the recrystallization of metallic materials. This assumption is related to the possible additional material transport mechanism improving the diffusion when plasma is utilized, as observed in previous works Batista *et al.* (1998) and Brunatto *et al.* (2005b).

This work comprises a study of niobium annealing utilizing plasma devices. Niobium is a refractory metal, which presents potential applications in the nuclear, space, superconducting and bioengineering industry Keith *et al.* (2007), Ribeiroa *et al.* (2006) and Catani *et al.* (2006). In addition, niobium has been also of great interesting in a wide range of applications in corrosive, erosive and wear environments Barzilai *et al.* (2006 and 2005), being the case of the production of niobium carbide (NbC) by annealing process of niobium coatings deposited on graphite, due the combination of the physical and mechanical properties of it.

In this work, annealing treatments in 90% reduction cold-rolled niobium samples were performed using hollow cathode discharge (HCD) configuration where the sample's heating is performed by means of the ion bombardment in an annular glow discharge. The influence of electrical and plasma parameters on niobium recrystallization was investigated. The samples microstructure was characterized considering the rolling direction and the cross-section of the specimens, and the recrystallization rate was determined confronting the hardness and the softening rating of the annealed and cold-worked samples.

2. RECRYSTALLIZATION OF NIOBIUM

Recrystallization of niobium has been studied by different authors (Fotedar and Monteiro, 1977), Sandim *et al.* (2003) and Jiang *et al.* (2006). In accordance with Wilkinson (Wilkinson, 1970), for niobium recrystallization purpose, temperatures ranging between 900 and 1200 °C are recommended.

Fotedar and Monteiro (Fotedar and Monteiro, 1977) studied the effect of fast neutron irradiation on the recovery and recrystallization of niobium in the temperature range 25-1200 °C for 1 h. The sigmoidal recovery curves showed an initial increase in microhardness in the temperature range 25-300 °C, which is related to the migration of interstitial impurity atoms O, C and N to dislocations and defect agglomerates produced by cold work and irradiation. It is stated the recrystallization in cold worked niobium proceeds by subgrain growth (controlled by subgrain coalescence) and strain induced grain boundary migration. The results indicated the fast neutron irradiation facilitates the formation of recrystallization nuclei by subgrain coalescence and thus accelerates the initial nucleation process in cold worked and irradiated specimens by about 150°C.

Sandim *et al.* (2003) has given special attention on the recrystallization behavior of a cold-rolled niobium bicrystal. A high-purity coarse-grained niobium bicrystal was 70% cold rolled in multiple passes. It was verified the deformation occurred in an inhomogeneous manner in both grains giving rise to a banded structure. In consequence, highly misoriented boundaries were developed in the microstructure in a wide range of misorientations, many reaching about 55°. It is stated these boundaries act as effective nucleation sites for recrystallization. Upon annealing at 800 and 900°C, the new recrystallized grains were nucleated preferentially at deformation heterogeneities and in the vicinity of the prior grain boundary in this bicrystal.

Jiang *et al.* (2006) have investigated the effect of texture evolution on recrystallization as a function of rolling strain in 50%, 70%, 80%, 90% deformed high purity niobium samples, which was rolled at room temperature with multiple passes in the same direction without lubricant and then annealed at 750° C for 1 h. In accordance with the authors, niobium has a tendency to generate shear bands, which causes heterogeneous local rotations that lead to a much wider range of crystal orientations, and which apparently weakens the classical bcc deformation and recrystallization texture characteristics after Nemat-Nasser *et al.* (2000) in Jiang *et al.* (2006). Additionally, it is stated recrystallization textures in IF steels can be accounted for based upon a strain energy release criterion based upon elastic anisotropy. Niobium has the opposite elastic anisotropy as iron after Simmons and Wang (Simmons and Wang, 1971) in Jiang *et al.* (2006), so the driving force to favor particular orientations in recrystallization may be opposite or nonexistent.

Finally, a very detailed study for pure niobium recrystallization is presented on Siciliano Jr. (Siciliano Jr., 1993). The author shows the annealing heat treatment results performed in a resistive conventional oven, at temperatures of 700, 800, 900, 1000, 1100, 1200 and 1300 °C, times of 2, 4 and 6 h, for 63.5%, 79.7%, 92.3% and 98.6% cold rolled samples in multiple passes. Results indicate the samples 92.3% cold rolled and treated at 800 °C, for times of 2, 4 and 6 h did not present recrystallization, however considerable recovering was evidenced. The softening relative to the initial hardness was 23.5%, 29.3% e 30.5%, respectively. This result is explained by the high niobium stacking-fault energy, which allows annihilating excess dislocations, decreasing the thermodynamic potential for material recrystallization. In addition, to the samples partially recrystallized, the softening determined in percentage values was always higher than the recrystallized volumetric fraction (in percentage), indicating a significant recovering occurrence and the consequent decreasing of the boundary migration's thermodynamic potential during the recrystallization process. The occurrence of the recovering decreasing the recrystallization's thermodynamic potential, and the insufficient thermal activation to the high angle boundaries' migration could explain why the recrystallization did not occur entirely. This fact emphasizes the importance of the recovering phenomena in the high stacking-fault energy metals as niobium, and it was evidenced to the samples 92.3% cold rolled and treated at 900 °C, for times of 2, 4 and 6 h, which presented recrystallization volumetric fractions of 23.9%, 40.7% and 51.2%, respectively. Otherwise, full recrystallization was evidenced to the samples treated at temperatures equals or higher than 1000 °C, and the smallest average grain size was verified to the samples 92.3% cold rolled, 4 h treated, which presented average grain size of 33 μm.

3. EXPERIMENTAL PROCEDURE

The annealing heat treatments were realized in the same discharge chamber utilized in previous works Brunatto *et al.* (2001, 2003 and 2005a). A detailed description of the experimental apparatus is available in earlier reports Brunatto *et al.* (2003). Figure 1 presents a schematic representation of the discharge chamber and an *in situ* photography of the hollow cathode discharge. The discharge chamber consisted of a 350 mm diameter, 380 mm high stainless steel cylinder to attached steel plates and sealed with O-rings at both ends. The system was evacuated to a residual pressure of 1.33 Pa (10^{-2} torr) using a two-stage mechanical pump. The gas mixture consisting of 80% argon (99.999% pure) and 20% hydrogen (99.998% pure) was adjusted using two datametrics mass flow controllers whose full scale value was 8.33 x 10^{-6} m^3s^{-1}. The pressure in the vacuum chamber was adjusted with a manual valve and measured with a capacitance manometer of 1.33 x 10^4 Pa (100 torr) in full-scale operation.

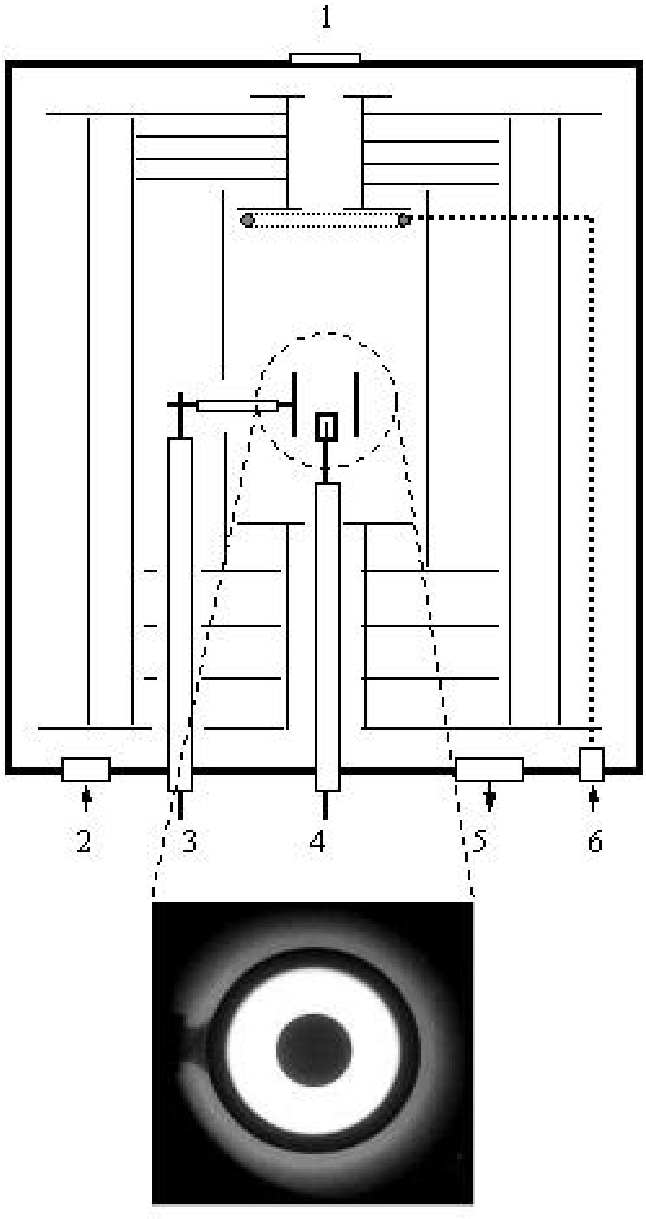


Figure 1. Schematic representation of the discharge chamber and an *in situ* photography of the hollow cathode discharge (1 = view window; 2 = outlet to manometer; 3 = external electrode; 4 = central electrode; 5 = outlet to vacuum pump; and 6 = gaseous mixture inlet).

Niobium samples were machined-made from a 10 mm diameter niobium cylindrical bar presenting 98.9% purity. The niobium bar was manufactured from an electron-beam melted 250 mm diameter ingot, cold-forged to 100 mm diameter, vacuum-annealed and then cold-rolled to a total reduction in thickness of 90% in multiple passes. Niobium samples 10 mm in diameter and 10 mm in height were placed on a pure niobium support (10 mm in diameter and 12 mm height), which worked as the central electrode (Fig. 2). To ensure a uniform bombardment of ions, leading to a thermal equilibrium between the parts, and in order to generate an uniform electric field and consequently a homogeneous annular discharge, a cylindrical niobium part (10 mm in diameter and 3.5 mm height) was placed at the top of the sample.

Annealing was performed at 1173 K (900 ºC) for 60 minutes, with a gas mixture flow of 5 x 10^{-6} m^3s^{-1}, at a heating rate of 0.38 $K.s^{-1}$ (0.38 $ºC.s^{-1}$), and all samples were cooled under gas mixture flow. Pressure of 400 Pa (3 torr) was utilized in accordance with the type of heating condition, in the case to the hollow cathode discharge.

Annealing treatments were realized using hollow cathode discharge configuration (Fig. 2) where the sample's heating is performed by means of the ion bombardment in an annular glow discharge. The experiment condition was repeated three times to guarantee the reproducibility of results.

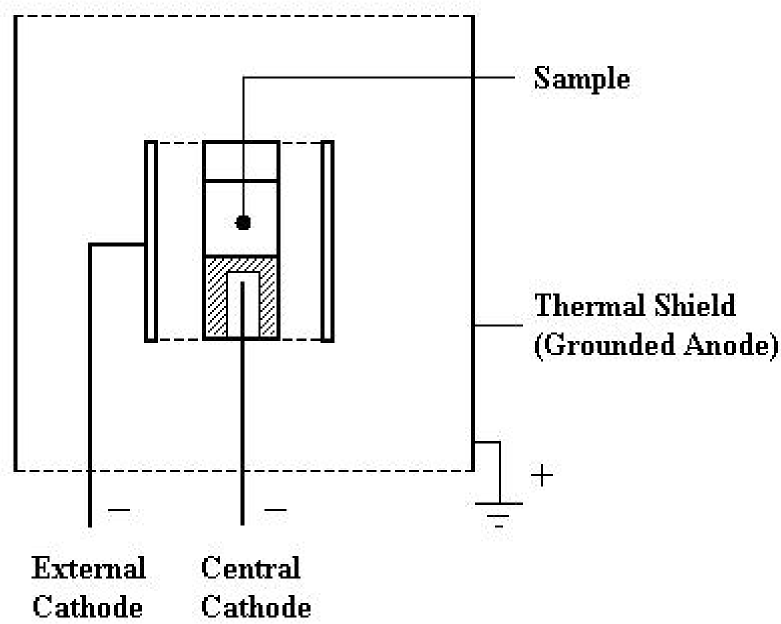


Figure 2. Schematic representation of the hollow cathode discharge configuration.

At the hollow cathode discharge configuration, both the inner cathode and the outer cylinder are negatively biased at the same voltage, using a square waveform pulsed power supply of 5.0 kW. The voltage was varied between 540 and 640 V, in accordance with the studied condition, and the power transferred to the plasma was adjusted by varying the time switched on (t_{ON}) of the pulse. To ensure a stable discharge, an electrical resistance adjusted to 50 Ω was connected in series between the power supply and the discharge chamber. The t_{ON} of the pulse could be varied from 10 to 200 μs and the on- plus off-time was 240 μs. The sample temperature was varied by adjusting the on/off time of the pulsed voltage and was measured using a chromel-alumel (type K) thermocouple. This thermocouple was protected with a stainless steel cover, 1.5 mm in diameter, electrically isolated with Al_2O_3 and inserted 8 mm into the sample holder.

In the heating condition using the hollow cathode discharge configuration (Fig. 2), the cylindrical surface of the sample was exposed to the plasma, and its heating to the annealing temperature was a direct consequence of the ion bombardment effect. In this condition both the electrodes (central and external) were negatively biased, acting as cathodes.

The external cathode was made from an AISI 310 stainless steel tube (composition in atom.%: 25% Cr, 16% Ni, 1.5% Mn, 1.5% Si, 0.03% C and balance of Fe). In the heating condition using the hollow cathode discharge configuration (Fig. 2), the external cathode was machined to 21 mm internal diameter, 2 mm wall thickness and 25.4 mm height, resulting in an inter-cathode distance of 5.5 mm.

Microstructure characterization of the niobium samples was performed using standard metallographic procedures for specimen preparation, in an Olympus BX51 Optical Microscope. Specimens were cut out along the rolling direction and at cross-section using the electrical discharge machine technique. Samples were grinding using 220, 400, 600, 1200, and 2400 SiC sand-paper and were polished using 0.6 μm diamond paste and 0.5 μm Al_2O_3 solution. Samples' etching was realized using a solution of 30 ml HF, 15 ml HNO_3, and 30 ml HCl (in accordance with ASTM 66 standard). Microhardness Vickers were determined using a Shimadzu HMV-2T apparatus, using 0.2 kg load. The grain size characterization was determined in accordance with ASTM E-112 standard. The ASTM grain-size number is defined by the relationship Eq. (1), where n is the number of grains per square inch as seen in a specimen viewed at a magnification of 100 times, and N is the ASTM grain size number.

$$n = 2^{N-1} \quad (1)$$

4. RESULTS AND DISCUSSION

Figure 3 shows the heating curve obtained to reach the specified annealing temperature for the studied condition. Despite the average heating rate was 22.5 ºC/min (hypothetical constant rate), the sample heated in the hollow cathode discharge configuration (HCD) was slightly submitted to higher temperatures (35 °C/min until 500 °C, 11°C/min until 750 °C and 50 °C/min until 900 °C). So, the highest permanence at 500 to 900 ºC temperature range could significantly influence the annealing process as a consequence of the recovering and recrystallization processes competition, and since niobium presents great facility to the recovering, as seen, a decreasing in the thermodynamic potential for material recrystallization could be expected.

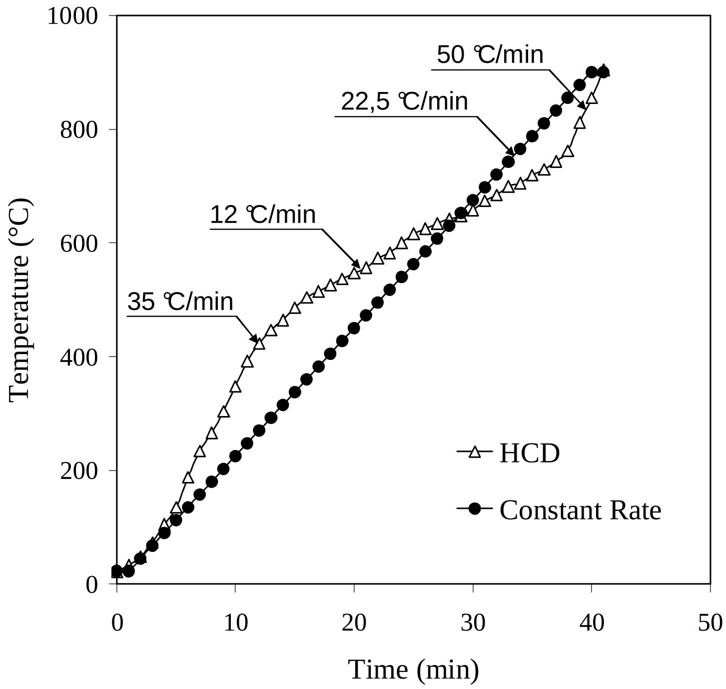


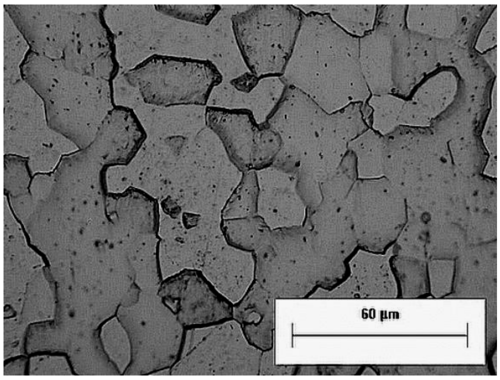
Figure 3. Heating curve obtained to reach the specified annealing temperature for the studied condition.

Table 1 depicts the variation of the macroscopic discharge parameters of heating configuration. It presents the average and standard deviation values at 1173 K (900 ºC) annealing temperature, along the 60 min annealing time. The plasma assisted heating conditions exhibit different behaviours as a function of the central electrode design (Fig.2). It must be pointed out in the HCD configuration an intense ion bombardment effect occurs in the central cathode, so the annealing temperature was achieved using 3 Torr pressure, about three times lower than the pressure which would be necessary to obtain the same heating effect by ion bombardment, using a linear glow discharge Brunatto *et al.* (2000). A study by Timanyuk and Tkachenko (1989) supports this assumption. Their findings revealed that the dark space in the central cathode of an annular discharge is thinner than that observed on the inner surface of the external cathode. Therefore, lesser collisions between ions and neutral species tend to occur in the central cathode sheath, resulting in a higher kinetic energy of the ions reaching the cathode surface and, hence, in a higher heating efficiency of the sample. The higher heating efficiency to the 1173 K (900 ºC) annealing temperature can be verified when the parameters values used in the discharges are confronted.

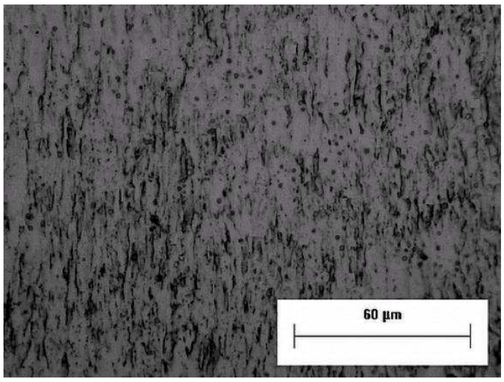
Table 1. Average and standard deviation of the macroscopic discharge parameters at the isothermal annealing stage for heating configuration (I_{CC} = central cathode current; and I_t = total discharge current).

Heating configuration	p (torr)	I_{CC} (mA)	t_{ON} (μs)	Volt. (V)	It (mA)
HCD	3	176 ± 9	62 ± 4	536 ± 11	438 ± 9

Figure 4 presents the microstructure characterization of the HCD (Fig. 4a) annealed specimens, and in the cold-rolled condition (Fig. 4b). At the start condition (Fig. 4b), samples present a typical deformed structure, which is characterized by share and deformation bands aligned in the rolling direction. As can be seen in the Table 2, 90% cold-rolled samples present 127 $HV_{0.2}$ average microhardness and 0% softening rate. These values are in accordance with those presented by Siciliano Jr. (Siciliano Jr., 1993), for similar cold-rolling condition, which certifies the start condition of the 90% cold-rolled niobium samples.



4(a)



4(b)

Figure 4. Microstructure of the samples annealed in: a) HCD configuration; b) as non-processed, in the 90% reduction cold-rolled condition;

Table 2. Evolution of the recrystallization determined by means of the microhardness (average and standard deviation) and softening rate.

Condition	Microhardness ($HV_{0.2}$)	Softening rate (%)
Full annealed Nb (theoretical ASM[(1)])	80	100
HCD processed sample	79 ± 5	100
90% reduction cold-rolled Nb	127 ± 3	0

[(1)] (ASM, 1990)

The microstructure obtained for the sample processed in the HCD configuration (Fig. 4a) qualitatively indicates that full recrystallization occurred, since a structure of not-aligned crystals (or grains) are present and there is no more evidence of the share and deformation bands' presence. This affirmative is supported considering the grain size determination, whose measurements indicate an average grain size of 35 µm and an ASTM number between 6.5 and 7.0 (Table 3). In addition, besides the full recrystallization, a slight grain growth occurred. This assumption is based on two results obtained by Siciliano Jr. (Siciliano Jr., 1993):

a) 92.3% cold-rolled niobium samples annealed in resistive conventional oven at 900 ºC during 2 hours presented a partially recrystallized microstructure, with 23.9 vol.% recrystallized fraction; and
b) 92.3% cold-rolled niobium samples annealed in resistive conventional oven at 1000 ºC during 2 hours presented a fully recrystallized microstructure, with average grain size of 32 µm.

Note that similar results were obtained in the HCD configuration (35 µm versus 32 µm average grain sizes) using an 1 hour annealing time and 100 ºC annealing temperature lower than those used by Siciliano Jr. (Siciliano Jr., 1993).

Table 3. Determination of the grain size for niobium sample annealed in the hollow cathode discharge configuration, according to the ASTM E-112 standard.

Heating configuration	n (100x)	N	Grain size (µm)
ASTM E-112	45.25	6.5	37.8
HCD	55.75	6,8	35.4
ASTM E-112	64.00	7.0	31.8

Results showed in the Table 2 confirm the above-mentioned. Note the HCD processed sample presents 79 $HV_{0.2}$ average microhardness, and 100% softening rate, which is expected for full recrystallized niobium. The results obtained could indicate the strong ion bombardment effect, which is an intrinsic characteristic of the hollow cathode discharge, has an important role on the mechanisms that involve the thermally activated metallurgical transformations, where the atomic diffusion is present. Considering the growing interest of metal-mechanic industry in plasma assisted manufacturing, further studies are being conducted to deepen the knowledge of this effect on the metallic materials characteristics.

5. CONCLUSIONS

This work involved a comparative study of niobium annealing utilizing plasma and conventional devices. The influence of electrical and plasma parameters on niobium recrystallization was investigated. Annealing treatments were performed in hollow cathode discharge with ion bombardment.

Our results demonstrate that the design of the central cathode can influence the discharge parameters necessary to heat niobium samples in hollow cathode discharge, which could be related to the intrinsic characteristics of this kind of discharge, as the higher ionization rate and higher ion bombardment energy. Besides, the metallurgical events assisted by atomic diffusion, which is typically the case of the recrystallization and grain growth processes, proof to be strongly dependent of the ion bombardment effect. This conclusion is valid at least for the hollow cathode discharge configuration.

This study opens up a new field of research involving materials processing assisted by plasma. Considering the growing interest of industry in high temperature metallic materials, as niobium, however, further studies are being conduced to determine the influence of hollow cathode discharge and abnormal glow discharge processing on the mechanical properties and on the microstructure evolution of the material.

6. ACKNOWLEDGEMENTS

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7. REFERENCES

ASM International Handbook Committee, 1990, "ASM Handbook", 10th ed., v.2, pp 1144-1145.

Barzilai, S., Frage, N. and Raveh, A., 2006, "Surface & Coatings Technology", 200, 4646-4653.

Barzilai, S., Frage, N. and Raveh, A., 2005, "Vacuum", 79, 171-177.

Batista, V. J., Binder, R., Klein, A. N. and Muzart, J. L. R., 1998, "The International Journal of Powder Metallurgy", 34, 8, 55-62.

Benda, M., Vlcek, J., Cibulka, V. and Musil, J., 1997, "J. Vac. Sci. Technol. A", 15, 5, 2636-2643.

Berry, R. S., Bernholc, J. and Salamon, P., 1991, "Appl. Phys. Lett.", 58, 6, 595-597.

Brunatto, S. F., 2000, "Estudo e desenvolvimento do processo de sinterização de compactados de ferro com enriquecimento superficial simultâneo de elementos de liga em descarga elétrica de cátodo oco". Doctorate Thesis, UFSC, Brazil.

Brunatto, S. F., Guimarães, N. V. and Muzart, J. L. R., 2005b, "Proceedings of the 18th International Congress of Mechanical Engineering", ABCM, Brazil.

Brunatto, S. F., Kühn, I. and Muzart, J. L. R., 2001, "Materials Research", 4, 4, 245-250.

Brunatto, S. F., Kühn, I. and Muzart, J. L. R., 2003, "Sintering iron using a hollow cathode discharge", Materials Science and Engineering A, Volume 343, 163-169.

Brunatto, S. F., Kühn, I. and Muzart, J. L. R., 2005a, "Journal of Physics D: Applied Physics", 38, 2198-2203.

Catani, L., Cianchi, A., Lorkiewicz, J., Tazzari, S., Langner, J., Strzyzewski, P., Sadowski, M., Andreone, A. Cifariello, G., Di Gennaro, E., Lamura, G., Russo, R., 2006, "Cathodic arc grown niobium films for RF superconducting cavity applications". Physica C 441 130-133.

Chapman, B., 1980, "Glow Discharge Processes, 406", New York, John Wiley & Sons.

Engel, A. V., 1994, "Ionized Gases", 2nd ed., 325, New York, American Institute of Physics.

Fotedar, H. L. and Monteiro, W. A., June-July 1977, "Journal of Nuclear Materials", Volume 67, Issues 1-2, Pages 24-32. Effect of fast neutron irradiation on the recovery and recrystallization of niobium.

Hirvonen, J. P., et al., 1996, "Surface and Coatings Technology", 84, 600-604.

Jiang, H., Bieler, T. R., Compton, C., Grimm, T.L., 2006, "Cold rolling evolution in high purity niobium using a tapered wedge specimen", Physica C 441, 118–121.

Koch, H., Friedrich, L. J., et al., 1991, "J. Vac. Sci. Technol. A", 9, 4, 2374-2377.

Leonard, Keith J., Busby, Jeremy T., Zinkle., Steven J., 2007, "Aging effects on microstructural and mechanical properties of select refractory metal alloys for space-reactor applications.", Journal of Nuclear Materials, v. 366, pp. 336–352.

Muzart, J. L. R., Batista, V. J., Franco, C. V. and Klein, A. N., 1997, "Proceedings of Advances in Powder Metallurgy & Particulate Materials", MPIF, 1997, 3, 77-84.

Nemat-Nasser, S., Guo, W. G., 2000, "Flow Stress of Commercially Pure Niobium Over a Broad Range of Temperatures and Strain Rates", Materials Science and Engineering A, Volume 284, 1.

Peterson, N. L., 1983, "International Metals Reviews", 28, 2, 65.

Ribeiroa, R., Ingolea, S., Ustab, M., Bindalc, C., Ucisikd, A.H., Lianga, H., 2006, "Tribological characteristics of boronized niobium for biojoint applications", Vacuum 80, 1341–1345.

Sandim, H. R. Z., Lins, J. F. C., Pinto, A. L. and Padilha, A. F., 2003, "Materials Science and Engineering A", 354, 217-228.

Shohet, J. L., 1991, "IEEE Transactions on Plasma Science", 19, 5, 725-733.

Siciliano Jr., F., 1993, "Estudo comparativo entre a recristalização do nióbio puro e da liga Nb-1%Zr" M.Sc. Dissertation, USP, São Paulo, Brazil.

Simmons, G., Wang, H., 1971, "Single Crystal Elastic Constants and Calculated Aggregate Properties: A Handbook", 2nd Edition, MIT Press, Cambridge.

Terakado, K. et al., 1996, "Metallurgical and Materials Transactions A", 27A, 401-405.

Timanyuk, V. A. and Tkachenko, V. M., 1989, "Sov. Phys. Tech. Phys.", 34, 7, 832-834.

Wilkinson, W.D., 1970, "Fabrication of Refractory Metals", New York-London-Paris, Gordon and Breach Science Publishers, Inc., pp 184-195.