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A METHODOLOGY OF AL-CU ALLOY NITRIDING BY ELECTRICAL DISCHARGES

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***Abstract**. Different efforts have been made to find a method that can take advantage of the lightness of aluminum alloys for the manufacture of mechanical components subjected to high wear. Nitriding is a physicochemical process that promotes the formation of nitrides in a material, the creation of a surface layer of AlN can mean an increase in hardness and therefore the reduction of surface wear of different mechanical components. This article presents the methodology used for the nitriding of Al-Cu alloys using electric discharges generated by the EDM process and shows the various methods for the analysis of the mechanical properties.*

***Keywords:** Nitriding, Duraluminum, EDM, Methodology.*

1. INTRODUCTION

As engineering advances, the importance of component weight reduction in mechanical designs is evident. This creates the need to produce new composite materials or treatments for improving the surface resistance of some material while maintaining the weight and composition of its matrix. Various surface treatments have been used to improve the performance of materials for different applications, surface hardening, for example, decreases surface wear and increases the shelf life of the treated material. Other factors such as the reduction of the coefficient of friction or the resistance to fatigue are influenced by the superficial treatments. Nitriding is a physical-chemical treatment that promotes the creation of nitrides, in this case superficially in the form of a layer, keeping the substrate with its normal properties and the surface hardened. In the aluminum nitriding process, various techniques such as plasma nitriding, gas nitriding, salt bath nitriding (or liquid nitriding) and ion implantation are used, resulting in thin layers up to 20 µm, as in the case of Okumiya et al. (2009). In the Table 1 several authors are presented who were able to nitrate the aluminum through different methods and the results obtained in each case.

The Electrical Discharge Machining (EDM) process utilizes electric discharges between two electrodes (part and tool) submerged in a dielectric fluid for subsequent removal of material by sublimation (ALBINSKI, 1996). This process has emerged as one of the most common in the manufacture of metal molds and dies (SANTOS et al., 2016). It has been demonstrated that the plasma channel generated in the process promotes the ion exchange between both the electrodes and the suspended particles in the dielectric fluid. The use of alternative dielectric fluids and mixtures with powdered material shows ionic deposition, in which case a mixture of deionized water with urea (CH_4N_2O) was used.

In the current work, the nitriding of the aluminum AA 2011 (aluminum-copper alloy) was carried out through the sink electrical discharge machining (EDM). A methodology of nitriding through electric discharges produced by the EDM process is presented, which shows the influence of the state of the alloy and the generation of discordances in the crystalline lattice promoted by the creation of hardening precipitates (θ’ and θ’’) due to aging.

Table 1. Features of creating AlN layers with different processes

Method	Author(s)	AlN layer thickness	Temperature and process
Plasma nitriding	H. Chen et al. (1994)	3 µm	>723 K, >8 h
PIII (Plasma-Immersion Ion Implantation)	T. Czerwiec et al. (2000)	10 – 15 µm	723 – 823 K, >5 h
Plasma nitriding	S. Gredelj et al. (2002)	2 – 3 µm	575 °C, 180 min.
Ion nitriding	T. Fitz (2002)	Até 1 µm	Pre-spray cleaning with

			argon (Ar). 400 °C by 2 h
Plasma nitriding	P. Visuttipitukul et al. (2006)	3 µm	Refining the microstructure by Bulk Mechanical Alloying (BMA)
Plasma/barrel nitriding	M. Okumiya et al. (2009)	20 µm	Combined process of barrel nitriding for 5 h at 903 K and plasma nitriding for 2 h at 853 K.
EDM nitriding	D. M. Souza (2016)	104 µm	Aging treatment a 524 °C

2. METHODOLOGY

The method was to implement surface enrichment by nitrides in duralumin using electrical discharge machining (EDM). The samples were tested after artificial aging treatment in a muffle furnace at a temperature of 160 ° C for 14 hours with cooling in the oven for another 12 hours, the heat treatment scheme is shown in Fig. 1. In this process, an unconventional dielectric fluid, a solution of deionized water with urea was used, with urea being the source of nitrogen. In the research conducted by Santos (2013) observed that the urea concentration in the deionized water has no significant influence on the creation of the nitride layer or the thickness of the same nitridation of a carbon steel AISI 1040. This indicates that the amount of nitrogen introduced in the crystal lattice by the plasma channel is limited. According to the results obtained by Santos (2016), the samples do not present physical or mechanical changes with different concentrations of urea and the Vickers microhardness values obtained are very close to each other.

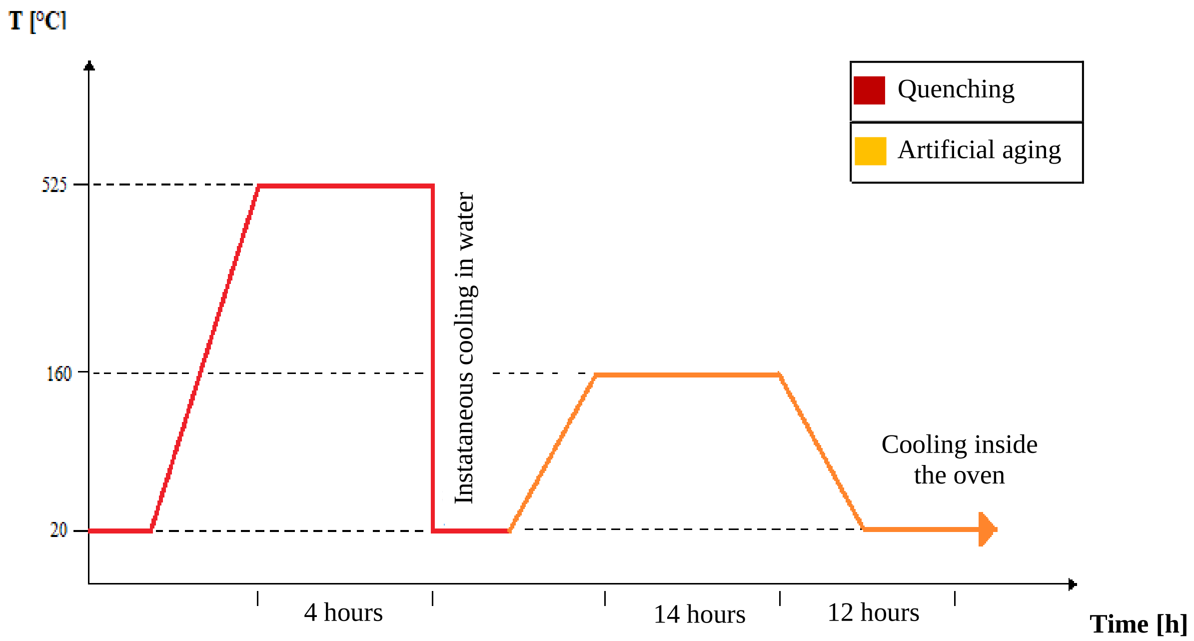


Figure 1. Heat treatment scheme for artificial aging on AA 2011 aluminum alloy

The operating parameters for the use of the EDM machine are: the polarity of the tool electrode, voltage, current, pulse time (Ton), the relationship between the pulse time and the total time (DT), the gap (gap), sensitivity, time erosion, periodic removal of the electrode tool, interval between erosion and remoteness.

3. EXPERIMENTAL PROCEDURE

The electrode used was made of ultrafine grain graphite with a diameter of 10 mm and a length of 150 mm. In order to perform the nitriding process by electrical discharges it was necessary to prepare the samples to be submitted to the process, they were taken from an aluminum bar AA 2011 and cut to a standard size of 20 mm x 20 mm x 10 mm and were subjected to several heat treatments in a muffle furnace before nitriding to prove the influence of distortion generation due each treatment on the crystalline network of the material in the process.

In the process of nitriding by electrical discharges, the main tank of the machine was not used, but an auxiliary vessel of stainless steel of 150 ml of capacity, which was filled with the solution of water deionized with urea (5 g/l).

The parameters with the greatest influence on the process are current (i), pulse time (t_{on}), working time (Dt), electrode material, dielectric fluid, polarity and erosion time. These parameters were defined according to the production of electric discharges in the process, several combinations of parameters were tested to choose the one that produces the most electric discharges in 1 minute of erosion time, thus producing a more stable plasma channel. The parameters used on electrical discharge nitriding process (EDN) are shown in Table 2.

Table 2. Parameters used on EDN process for AA 2011 aluminum alloy

Parameter	Concept
Current (i)	30 A
Pulse time (t_{on})	100 µs
Working time (Dt)	50 %
Dielectric fluid	Deionized water + urea (5 g/l)
Erosion time	60 s

All samples were subjected to metallographic analysis of cross-section after EDN. The metallographic preparation consisted of the cross-section of the machined area, followed by manual sanding with silicon carbide sanding grit mesh in the following sequence: 320, 400, 600, 1000 and 2000, followed by polishing with alumina oxide and last chemical etching with reactive Keller for the development of grain boundaries and areas of interest.

After the metallographic preparation the samples were taken under optical microscopy, scanning electron microscopy (SEM) and X-ray diffraction (XRD). Table 3 shows the parameters parameters used in XRD analysis. In optical microscopy the different phases created in the process were evaluated, due to the melting and re-solidification of the surface of the material.

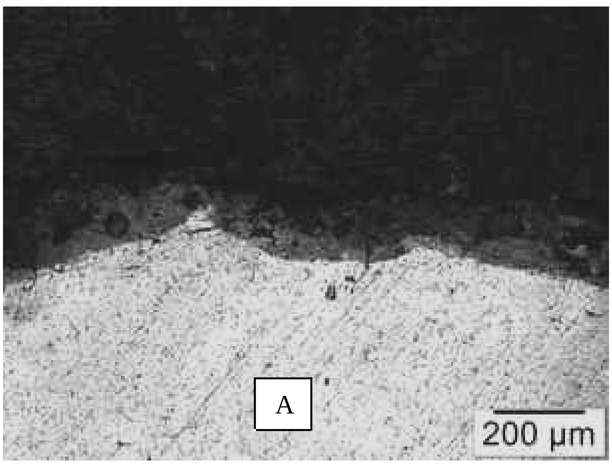
For the evaluation of the surface hardness, a Vickers (HV) microhardness equipment with a load of 40 gf was used, analyzing the remelted zone and the matrix for comparison.

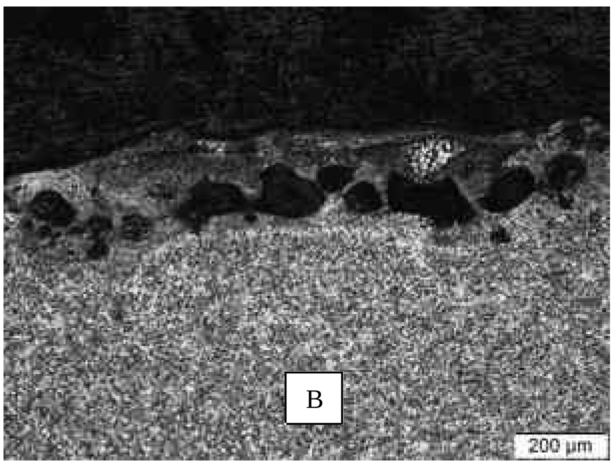
Table 3. Technical parameters for XRD (θ-2θ)

Radiation	CuKα
Voltage	40,0 kV
Current	30,0 mA
Scanning mode	Continuous scan
Spacing	0,02 deg
Scanning speed	1,0 deg/min
Initial and final angle (2θ)	20° - 80°

4. RESULTS AND DISCUSSIONS

Figure 2 shows the images acquired by optical microscopy of the samples submitted to the nitriding process by electrical discharge for each condition.





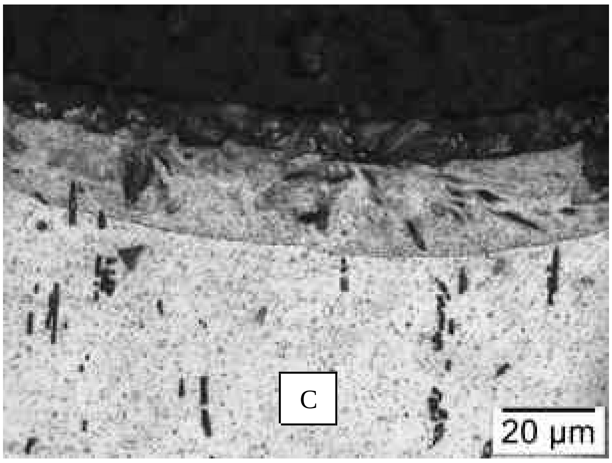


Figure 2. Optical microscopy images of the cross section for each condition of the alloy of the duralumin AA 2011 submitted to the EDN process. Chemical attack with Keller reagent to reveal microstructure. A). Solubilized; B). Annealed; C). Aged;

Through the images obtained with optical microscopy shown in Fig. 2 the different phases created in the process can be differentiated due to the melting and re-solidification of the material. The remelted zone (RZ) layer thickness for each condition after EDN process are presented in Table 3.

Table 3. Remelted zone thickness for each condition after EDN process

	RZ thickness (µm)
Solubilization	151 ± 20
Quenching	192 ± 38
Aging	68 ± 12

The X-ray diffractogram for the aged sample is shown in Figure 3 and it is possible to identify some low intensity peaks of α-AlN with a hexagonal crystalline system at the following 2θ (Bragg's) angles: 36,21 ° [0 0 2] and 37,91 [1 0 1]. The data were correlated with datasheets extracted directly from the PCPDFWIN database that the machine has.

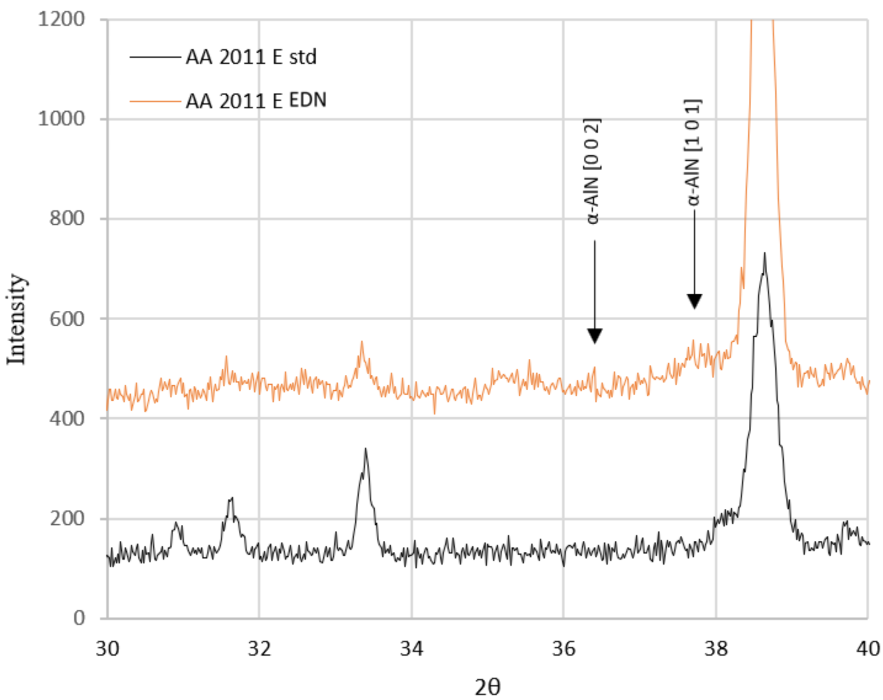
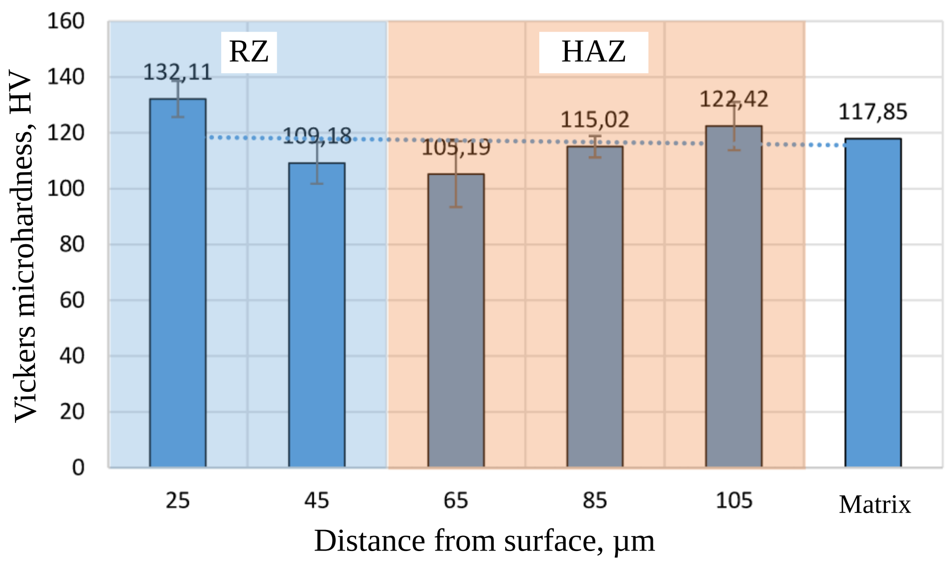


Figure 3. X-ray diffraction of the surface submitted to the EDN process in the aged state of the alloy

After confirming the presence of AlN in the alloy, microhardness tests were performed to prove the influence of nitriding in the EDN process. The results of microhardness test for aged stat of alloy are presented in Figure 4 and this shows that there was no variation of microhardness between the process result layer and the alloy matrix. Failure to change the physical properties could mean that the amount of nitride created in the process was small.



The results of microhardness test for each condition is shown in Table 4, In all the conditions a significant variation in the microhardness of the recast layer was observed. in the zone affected by the heat has a small variation in the microhardness due to the high temperatures suffered in this area, making changes in the properties of the material due to the tempering.

Table 4. Microhardness test ressults for each condition of AA 2011 Aluminum alloy

	RZ	**HAZ**	**Matrix**
Solubilized	87,8 ± 2,3	83,1 ± 1,2	83,4 ± 0,2
Annealed	56,9 ± 1,4	55,6 ± 1,5	58,0 ± 1,0
Aged	117,8 ± 6,2	106,5 ± 5,0	117,8 ± 0,3

5. CONCLUSIONS

The methodology used in this work with the purpose of nitriding the duralumin AA 2011 by means of electric discharges demonstrated that it is possible to create a layer of nitride in this material after aging with the use of the EDM machine.

The use of a solution of deionized water with urea as a dielectric fluid was effective as a source of nitrogen for the process. The plasma channel generated between the electrodes (workpiece (+) and tool (-)) energizes the N_2 ions suspended in the dielectric fluid, which are accelerated against the surface of the material and introduced through the crystalline lattice of material.

The state of the alloy of the material, product of the analyzed thermal treatments, influences the effectiveness of the nitriding by electrical discharge process. The distortion of the crystalline lattice caused by the creation of coherent and semi-coherent precipitates due to the aging process promoted the incorporation of the nitrogen in the surface layer generated by the process. The RZ created by the process with aging treatment has a thickness of 68 ± 12 µm for this alloy state, and this varies according to the treatment thermal analysis.

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