High Density Plasma Nitriding on Stainless Steel Alloys at PUPR-MC Plasma Machine

Ramón Rivera, Franklyn Colmenares,
Giovanni Lleonart, David Leal,
Jorge Gaudier, Dr. Ángel González-Lizardo,
Dr. Gilmer Burgos, Dr. Edbertho Leal-Quirós
Plasma Physics Laboratory
Department of Development and Scientific Research
Mechanical Engineering Department
Polytechnic University of Puerto Rico

ABSTRACT

A novel method of nitriding at high current levels at Polytechnic University of Puerto Rico Mirror Cusp plasma machine (PUPR-MC) has been performed. Nitrogen ions implantation or plasma nitriding increases the hardness at a surface level of stainless steel because of high plasma temperatures and high ion densities created in the chamber. Nitriding is the process of doping a crystalline metallic matrix with the presence of nitrogen ions; it was previously done in the past utilizing chemical processes with high acid densities at high temperatures. By using plasma nitriding procedure at PUPR-MC surface hardness is increased in 302/304 type stainless steel samples by exposing them to high ion density plasma at high vacuum. This method successfully dopes the surface of the material with strengthening nitrogen ions, without the use of chemical procedures that sacrifice the resistance to corrosion of the given material. A 500 V negative bias is placed on the sample exposed to the nitrogen plasma, where high energy ions are therefore attracted and immersed into the metallic matrix microns of the stainless steel surface. This potential maintains a constant surface temperature at approximately 800°C. Plasma parameters including ion density and plasma temperature were diagnosed using Single Langmuir Probes. The stainless steel samples were then tested using scanning electron microscopy (SEM) and Vickers Micro-Hardness tests to determine the increment in the surface hardness of the material. The SEM showed a significant presence of nitrogen imbedded in the grains of the stainless steel surface.

I- INTRODUCTION

Nitriding, the process of doping a crystalline metallic matrix with the presence of nitrogen ions, was previously done in the past utilizing chemical processes with high acid densities at high temperatures. This process increases hardness, reduces friction, and ads wear resistance to metals in high performance situations under thermal stress, but at the expense of increasing corrosion and causing environmental hazards. A newer and more environmentally safe method of treatment metallic work pieces is called plasma nitriding [6][7].

Plasma nitriding is the process of exposing a work piece to high energy ionized nitrogen gas or plasma. The nitrogen ions are extracted from plasma and attracted to the surface of treated material by a negative floating potential placed on the material surface. The treatments diffuse nitrogen ions into a maximum of 10 microns deep into the surface of a work piece. Traditional plasma treatments can take up to 10 hours and are done at temperatures above 500°C to reach the required conditions.

Previous work done by Pinedo and Monteiro [5] found that by utilizing a low temperature situation with high density plasma, the same effects of satisfactory hardness and wear resistant properties may be achieved without the expense of reducing corrosion resistance. Similar work was done by Li [2][3] at low plasma density with a voltage dependant bias to regulate the surface temperature of the treated material. Due to the requirement of high surface temperatures to achieve a high nitrogen absorption level, corrosion resistance was sacrificed.

Previous work done by Meletis, Singh, and Jiang [4] found that processing the materials above ~500°C on the surface of the sample produces chromium nitride precipitates that increase hardness properties, but cause a decrease corrosion resistance of the given material. Nitrating below 500°C has been found to increase hardness and wear resistance without reducing corrosion. Based on some of the previous works, research with compelling results has been performed by Valencia, Lopez-Callejas, and Muñoz-Castro [8] with toroidal discharges in DC and RF conditions using a pulsed plasma method to achieve the high ion densities to improve nitriding quality. By exposing the material to a constant high density ion flux results show an increase in the nitrogen ions

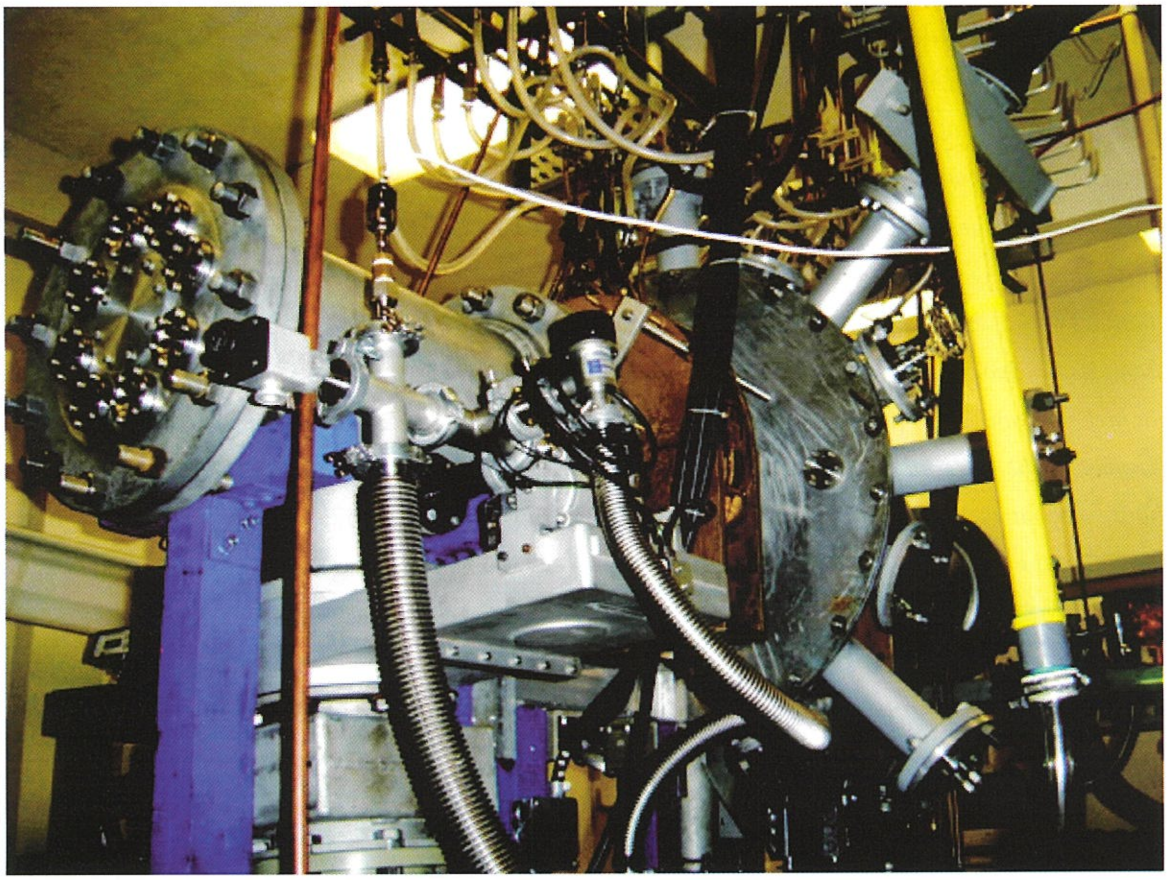

***Figure 1:** PUPR Mirror-Cusp Plasma Machine*

immersed in the matrix of the stainless steel surface. This has been done by using the PUPR-MC to nitride samples of stainless steel.

PUPR-MC plasma machine has the advantage of producing high density ion concentration plasma at regularly low temperatures. Using this device for plasma nitriding will allow the positive effects of high wear resistance and surface hardness without loosing corrosive resistance.

II- PROCEDURE

PUPR-MC plasma machine chamber used for this experiment is 3m long with two 30 cm radius side cylinders and a 50 cm radius central chamber. Using a pair of Helmholtz coils, each one separated 15 cm from the center; a magnetic field is created either in Mirror or Cusp mode. In Mirror mode the ion flow is greater. The magnetic field magnitude is varied by using currents of 620 and 320 Amps into Helmholtz coils. Nitrogen plasma (Figure 2) is then created by superheating the species via electron cyclotron resonance (ECR) after achieving vacuum of 10-4 Torr.

By placing a negative bias on the sample in the form of a probe (Figure 3), the surface of the sample can be changed as more nitrogen ions are attracted to it. These ion densities have a range of 5 x 1015 cm-3 or more. Plasma density is directly proportional to ions saturation current and inversely proportional to ion temperatures. These ion temperatures vary

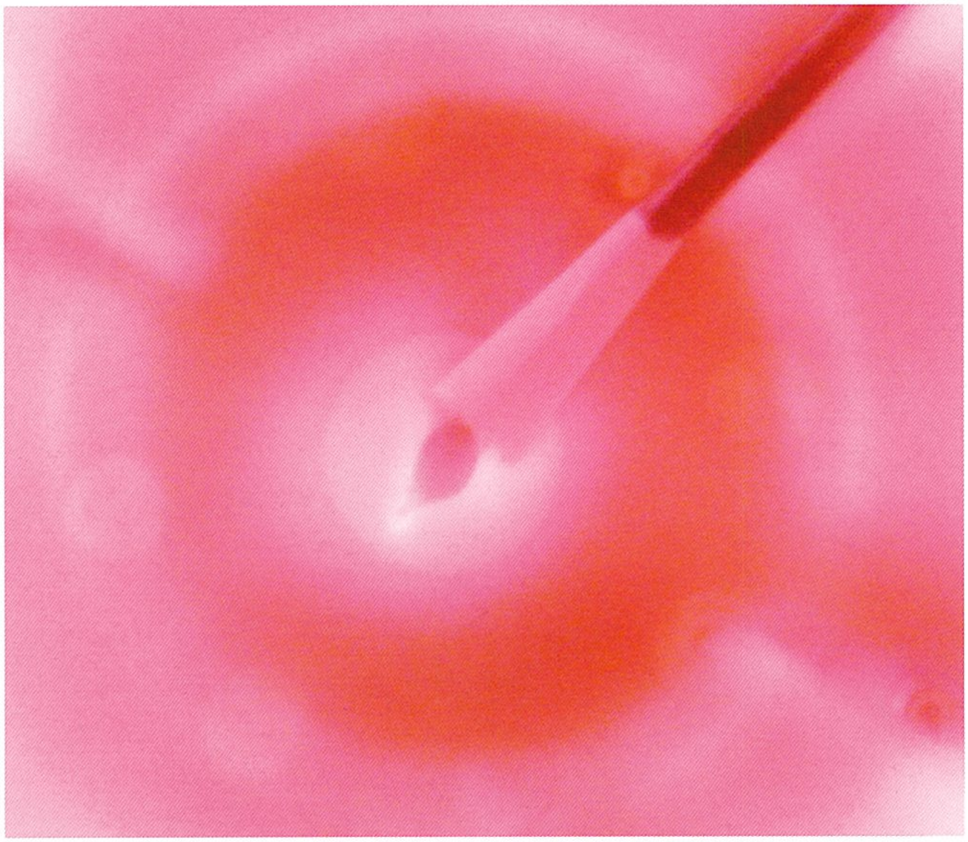

Figure 2: *Treated Samples at High Density Nitrogen Plasma in Mirror Mode*

according to the intensity of the machine magnetic field, and the microwave frequency that produces electron resonance in the chamber.

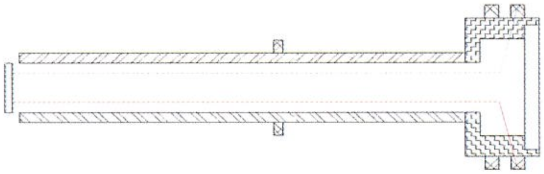

Figure 3: Nitriding Probe Schematic Used for the Experimental Setup

The mounting system used to treat the stainless steel samples in the machine uses a 3/8" diameter stainless steel tube. A micro-coaxial cable runs through the tube, and it is attached to a small mesh at the end, where the stainless steel disks are connected. Liquid ceramic is used to insulate the probe structure and avoid short circuits created by the plasma field. A floating potential of -412 V was applied to the sample. The back of the probe is closed with a stainless steel flange connected to a power supply via a BNC connector.

Samples used in experimentation are Type 302/304 Stainless Steel Shim Discs; 1/2" diameter and .01" thick, will be used to achieve better results.

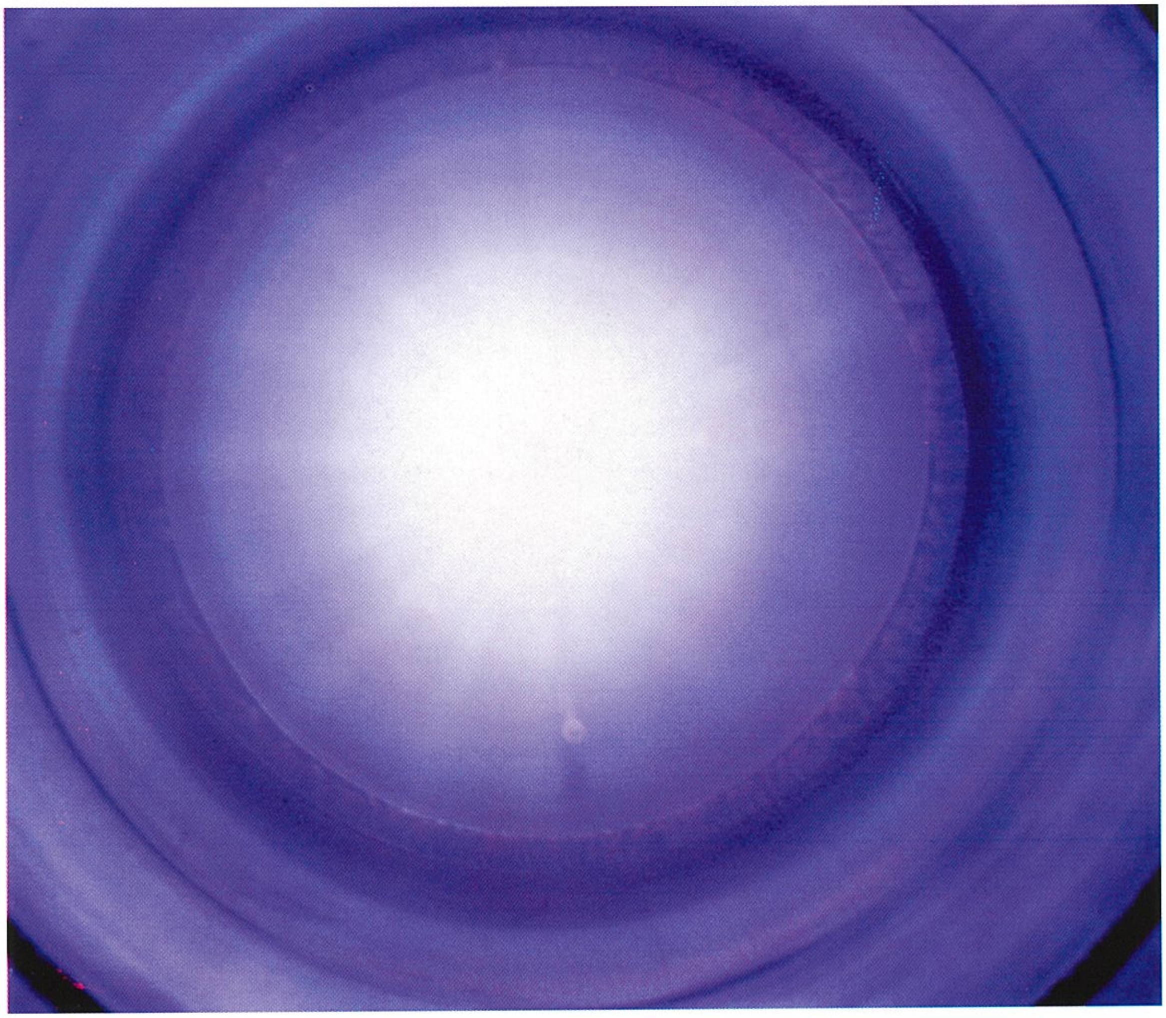

Figure 4: Argon Plasma at PUPR Lab with a Single Langmuir Probe

Figure 5: *Treated Samples*

Samples are placed inside the chamber for a period of 20 minutes. Several batches of tests were done varying chamber coils currents, which are proportional to the magnetic field. Plasma density can be measured by utilizing a Single Langmuir probe or a Double probe [1][9].

The most important parameter for this experiment is the current flowing through the coils, which consequently increases the magnitude of the magnetic field. Plasma density is a function of chamber pressure and of the intensity of the magnetic field provided by the plasma machine current. After materials are treated with nitriding and cleaned, several tests are later performed to display the surface characteristics and results using scanning electron microscopy (SEM), and Vickers Micro-Hardness testing.

III- RESULTS

Several tests were done to understand the effects of plasma treatment on stainless steel samples. This included the Vickers Micro-Hardness tests and Electron Diffraction Spectroscopy analysis. Loads of 500 grams were placed on the surface of samples using a diamond headed point for a period of 15 seconds each. The following table displays Micro-Hardness testing results for a series of samples taken in five situations in which plasma conditions were altered.

Sample 1: Plasma machine with magnetic field current of 620 A, 34.8 V, microwave power of 495 W negative bias of -412 V, high vacuum of 2.9 x 10-4 Torr with a plasma temperature of 3.5 eV and a electron density of 2.1 x 1014 m-3.

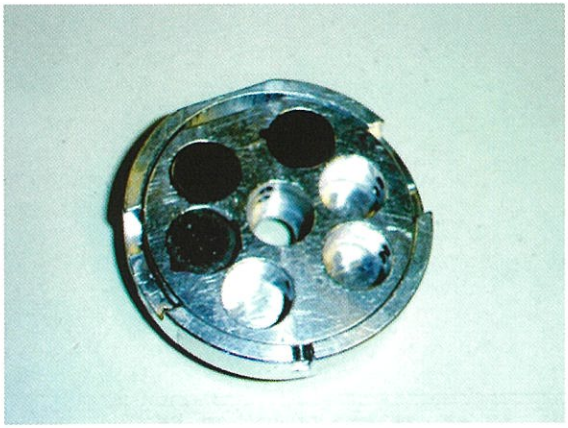

Figure 6: *Treated Samples*

Figure 7: *Scanning Electron Microscopy Equipment*

Sample 2: Plasma machine with magnetic field current of 620 A, 34.8 volts, microwave power of 495 W, no bias, high vacuum of 2.9 x 10-4 Torr with a plasma temperature of 1.99 eV and an electron density of 3.12 x 1014 m-3.

Sample 3: Plasma machine current 320 A, 16.57 V, microwave power of 495 W, negative bias -412 V, high vacuum of 2.9 x 10-4 Torr with a plasma temperature of 6.55 eV and an electron density of 2.187 x 1013 m-3.

Sample 4: Plasma machine current 320 A, 16.57 V, microwave power of 495 W no bias, high vacuum of 2.9 x 10-4 Torr with a plasma temperature of 2.9 eV and an electron density of 2.36 x 1015 m-3.

Sample 5: Control Sample

Table 1: *Micro-Hardness Testing Results*

	VICKER HARDNESS	***BRINELL HARDNESS***
Sample 1	267.8	254.5
Sample 2	255.5	248.8
Sample 3	255.4	243.6
Sample 4	244.0	243.5
Control Sample	241.8	232.4

Micro-Hardness results gave a standard deviation of less than 10 and display an average increment when treated samples are compared to the control sample. Samples placed in the plasma machine under high current and high ion density conditions, yielded an average hardness higher than the control sample in both the Vickers and Brinell testing parameters, with the exception of the final sample. Results also display that the primary and most important quality to

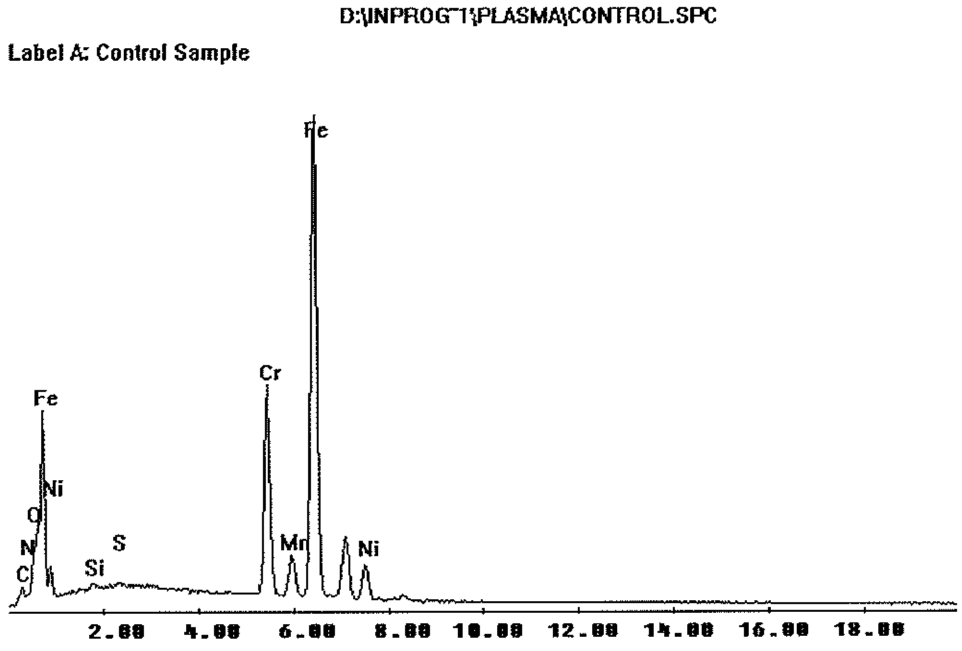

Figure 8: *Electron Diffraction Spectroscopy (EDS)Testing on Control Sample*

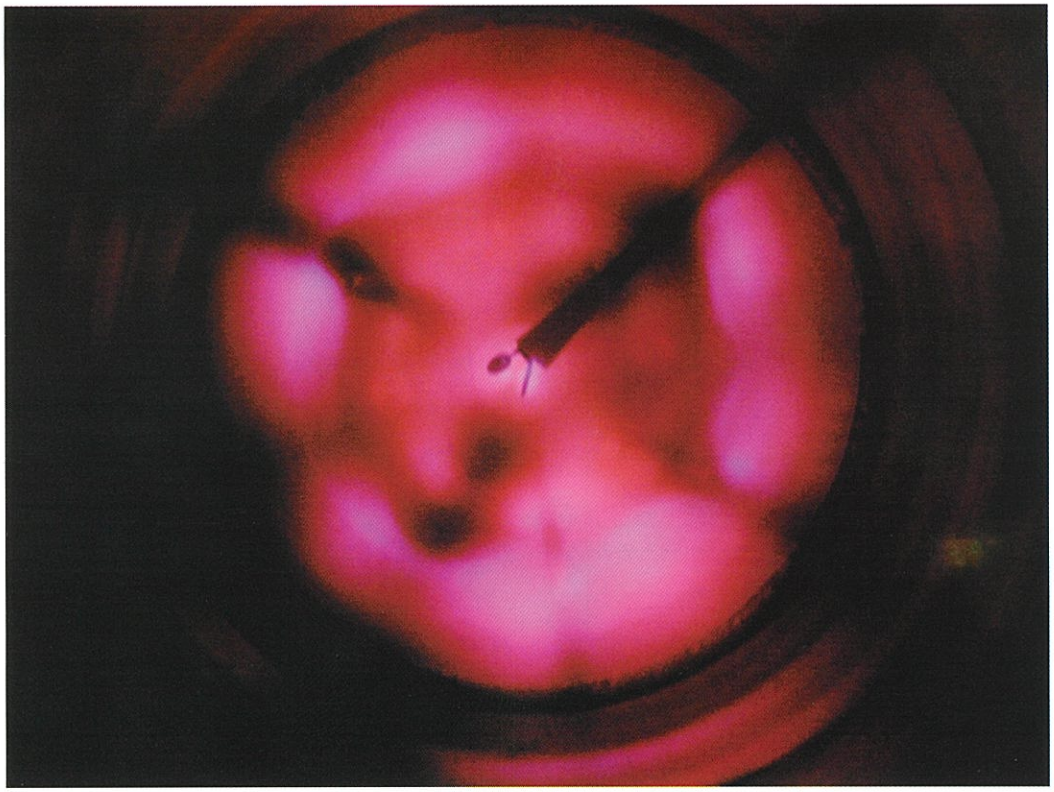

***Figure 11:** Treated Sample at Low Density Nitrogen Plasma in Mirror Mode*

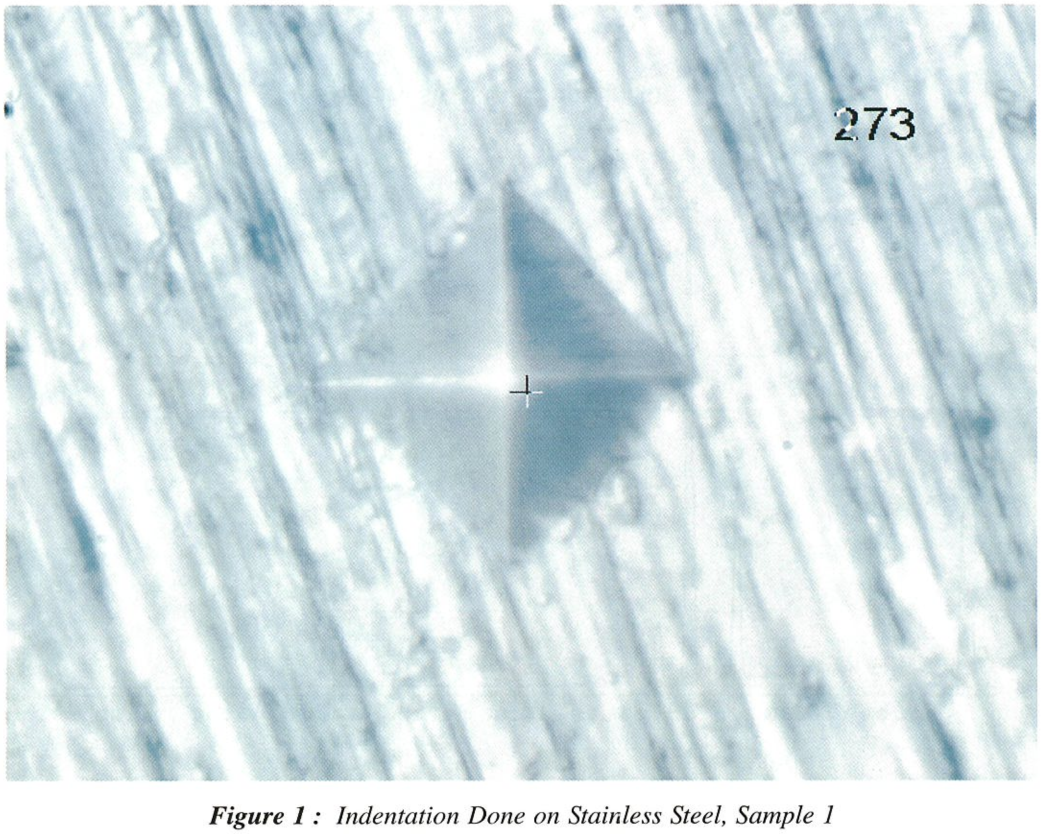

***Figure 1 :** Indentation Done on Stainless Steel, Sample 1*

successfully attract nitrogen ions to the surface of the stainless steel is a negative bias on the sample.

***Table 2:** Nitrogen by Weight in Treated Samples 1 and 3, Using SEM*

	NITROGEN WT%
SAMPLE 1	*2.02*
SAMPLE 3	*1.94*
CONTROL SAMPLE	*1.88*

The EDS results showed (Sample 1) that on the nitrogen weight percentage increased significantly, but made no significant increase on Sample 3. The amount of current flowing through the Helmholtz coils makes a significant difference in the ion densities present in the chamber. That can have a large impact on the nitrogen weight percentage results, and thus on the results of Micro-Hardness testing. In addition, a significant increase in carbon was observed for the treated samples, highly due to external carbon containing impurities from the ceramic containing the probes used to diagnose the plasma properties inside the vacuum chamber.

IV- CONCLUSIONS

In conclusion, nitriding at high current levels in PUPR-MC plasma machine increases the hardness at a surface level of stainless steel, due to high plasma temperatures and high ion densities created in the chamber. The effect of the negative bias on the sample is crucial to the success of the nitrogen process; this

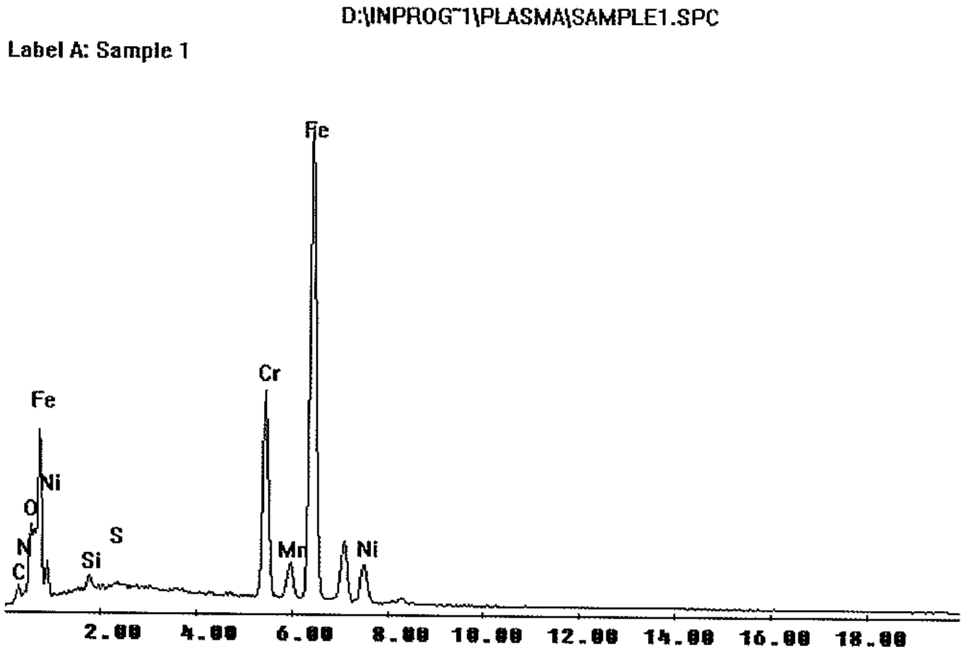

***Figure 9**: EDS Testing on Sample 1*

allows the sample to be charged negatively and to attract positive nitrogen ions. Low current plasma does not substantially increase hardness properties. More testing, repeated tests and improvements on the precision of measurements would increase the quality of the results.

V- REFERENCES

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