Study of Concentration-depth Profiles of the Titanium and Nitrogen Ions by SRIM/TRIM Simulation*

Estudio de perfiles de concentración por profundidad de iones de titanio y nitrógeno por medio del *software* SRIM/TRIM

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Abstract

In this research, the concentration-depth profiles reached by titanium and nitrogen particles, on the surface of AISI/SAE 1020 carbon steel substrates, by using of ion implantation technique, are studied. The ions are surface deposited by means of high voltage pulsed discharges and electric arc discharge under high vacuum conditions. The concentration and position distribution of the metallic and non-metallic species are obtained by simulation of the interaction of ions with the matter, stopping and ranges of ions in the matter, by the computer program transport of ions in matter. The implantation dose is calculated from the discharge data and the previously established study parameters in this work. From the simulation results, the depth profiles demonstrated that titanium and nitrogen ions may reach up to 300 Å and 600 Å and concentrations of 1.478 x 10^{16} ions⁄cm^2 and 2.127 x 10^{16} ions⁄cm^2, respectively. The formation of titanium microdroplets upon the surface of the substrates is identified from the micrographs obtained by the scanning electron microscopy technique; furthermore, the presence of titanium and nitrogen implanted on the surface of the substrate is verified through the elemental composition analysis by the energy dispersive spectroscopy, validating the effect of ion implantation on ferrous alloys.

Keywords: Carbon steel, dose, electric arc, ion implantation, surface modification, transport of ions in matter.

Resumen

En este trabajo se obtuvieron perfiles de concentración por profundidad de partículas de titanio y nitrógeno implantadas, en la superficie de un acero al carbono AISI/SAE 1020, por medio de la técnica de implantación iónica tridimensional. Las partículas fueron ionizadas por medio de descargas pulsadas de alto voltaje y de arco eléctrico a bajas presiones. La distribución de posición y concentración de las especies metálicas y no metálicas son adquiridas mediante un programa informático que analiza la interacción y el transporte de iones en la materia. La dosis de los iones implantados se calcula teóricamente a partir de los parámetros experimentales y los datos adquiridos de las descargas durante el proceso de modificación superficial. Los perfiles de profundidad demostraron que los iones de titanio y nitrógeno pueden llegar a implantarse a una profundidad de 300 Å y 600 Å, y conseguir concentraciones de hasta 1,478 x 10^{16} ions⁄cm^2 y 2,127 x 10^{16} ions⁄cm^2, respectivamente. La formación de microgotas de titanio sobre la superficie del material implantado fue identificada mediante micrografías obtenidas por microscopia electrónica de barrido; además, la presencia de titanio y nitrógeno implantado en la superficie de los substratos fue verificada mediante el análisis de composición elemental por espectroscopia de energía dispersa, validando el efecto de la implantación iónica sobre aleaciones ferrosas.

Palabras clave: acero al carbón, dosis, arco eléctrico, implantación iónica, modificación superficial, transporte de iones en la materia.

Introduction

The surface modification by ion implantation technologies via plasma is an alternative method to improve the physicochemical properties of metallic materials such as surface hardness, wear and corrosion resistance. It is based on the bombardment of ions onto a target to enrich the solid surface by generating films and producing modifications and defects inside the crystalline network of the material without considerable changes in its internal properties either its dimensions. Ion beam implantation (IBI), ion implantation by immersion in plasma (PIII) and ion implantation by plasma source (PSII) are some of the plasma-techniques broadly studied in the development of the science and engineering of the surface of materials [1]–[6].

Indeed, such an enhancement of the physical and chemical properties in metallic materials is due to advances in processes, procedures, equipment among other technological factors involved in the development and manufacture of new materials. In addition to this, it is of great importance to devote an attention to the development of computational simulation since it has become an indispensable tool for the study and comprehension of materials, specifically when is studied the collision of the ion with the atoms that make up a metallic solid and its effect by penetrating into the crystal lattice [7]–[9].

From several ion beam data analysis software packages available as shown in [8], the Stopping and Ranges of Ions in Matter-TRansport of Ions in Matter (SRIM/TRIM) computing package has become an attractive option for the study of ion distribution in solids, where it is possible to obtain the depth, distribution and composition profiles of doping particles into a blank substrate [7]–[12]. Therefore, the purpose of the present work is by implementing this software package, to study the concentration-depth profiles of the titanium (Ti) and nitrogen (N) ions in the AISI/SAE 1020 steel, from the previously established parameters in the experimental process and support these findings by microscopic and spectroscopic characterization.

Materials and Methods

The experimental methodology which comprises the selection and preparation of the ferrous alloy, apparatus description, dose estimation method, SRIM/TRIM simulation and characterization tests is described in the sections outlined below.

Material

AISI/SAE 1020 steel coupons were implemented in this study. Prior the surface treatment, the samples were prepared by grinding then polishing with silicon carbide papers down to 600 grit according with ASTM E3-95 Standard [13]. The substrates were distributed upon a coupon holder. Then, the latter is placed on the cathode region of the Joint Universal of Plasma and Ion Technologies Experimental Reactor (JUPITER) chamber, ready to receive a further surface preparation by sputtering [2]–[5].

Ion Implantation

Ion implantation was performed following the procedure proposed by Jaime Dulce [4] for the usage of the JUPITER reactor. Once the coupon holder is placed on the cathode inside the JUPITER chamber, "high vacuum" conditions are reached by a pressure system as shown in [2]–[5]. At this stage, a high voltage pulsed discharged is activated at 5 keV for 15 minutes together with an argon (Ar) gas supply in order to complete the surface preparation by sputtering. After the sputtering process, the electric arc discharge is activated, evaporating the titanium cathode and simultaneously ionizing the particles by the effect of the high voltage discharge set this time at 10 keV during 10 mins; achieving the ion implantation of metallic particles onto AISI/SAE 1020 carbon steel substrates. Likewise, the implantation hybrid treatment with Ti + N ions is carried out. The same energy and time treatment are applied but the difference is only the nitrogen gas supply inside the JUPITER chamber, allowing the implantation of both metallic and non-metallic species onto the samples.

Estimation of the Implantation Doce

Data of the pulse of the discharge are obtained by the Tektronix TDS 20028 oscilloscope. The calculation of the dose of implanted ions in some metallic materials depends mainly on factors such as: Duration of treatment, ion current density, repetition frequency, pulse duration, ion-electron secondary emission coefficient of the material and the cathode area [2]–[3].

The model used to calculate the implanted dose was reported in [9]–[16] where it is necessary to define the ion current value, as shown in equation (1).

$$I_T = I_e + I_i \tag{1}$$

Where I_i is the ion current, I_T is the total current obtained from the area under the curve of pulse discharge during the implantation, and I_e is the electronic current which can be known as it follows in equation (2).

$$I_e = \gamma I_i \tag{2}$$

Where γ is the ion-electron coefficient. Replacing equation (2) in equation (1) we can calculate I_i. Then, with the purpose of knowing the ion current density we have equation (3).

$$J_i = \frac{I_i}{A_T} \tag{3}$$

A_T is the total exposed area of the target on the cathode during the implantation. This ion current density value allows to estimate the implanted ions flux (Φ) upon the target surface by means of equation (4).

$$\Phi = \frac{J_i}{e^-} \tag{4}$$

Where e^- is the electron charge. Finally, the surface density of the implanted ions equation, which depends on the previously mentioned parameters, is calculated by equation (5).

$$D_i = \Phi \times \tau \times f \times t \tag{5}$$

Where D_i is the implantation dose, Φ ion flux, τ is the pulse duration, f is the repetition frequency and t is the discharge or treatment time.

Ion-solid Interaction

The SRIM/TRIM software developed by Ziegler *et al*. [7], was used to simulate the implantation of metallic and non-metallic species ions onto steel substrates carbon. SRIM/TRIM is based on the Monte Carlo computational method, which predicts the trajectory behavior of the colliding ions with the atoms of the structure of the metallic material up to nanometric scales [7]–[9].

Surface Characterization

The morphological characterization was performed by means of scanning electron microscopy (SEM) with a thermionic emission microscope Carl Zeiss EVO MA10 acquiring SEM images by secondary and retro-projected electrons. The identification of elements and its distribution upon the images were performed by and energy dispersive spectroscopy (EDS) with a detector Oxford X-act coupled to SEM. In the analysis was established a work distance of 9.5 mm and an acceleration voltage of 20 kV. It was possible to obtain black and white images on the surface of the material AISI/SAE 1020 steel implanted with Ti and T + N ions.

Results

The profiles obtained from the ion-solid interaction simulation, the theoretical estimation of the implantation dose and morphological surface characterization were carried out to identify changes and evaluate the effect of ion implantation upon the surface of the low carbon steel in question.

Ion Implantation

In figure 1 is shown the state AISI/SAE 1020 carbon steel substrates after the ion implantation process with (a) Ti and (b) Ti + N particles. It is evident some changes upon the substrate surfaces, such as the formation of a silver-colored metallic Ti layer in figure 1a, and a golden metallic shine in figure 1b due to the hybrid treatment, indicating the incorporation of nitrides into the surface.

Figure 1. Implanted substrates with (a) Ti and (b) Ti + N ions

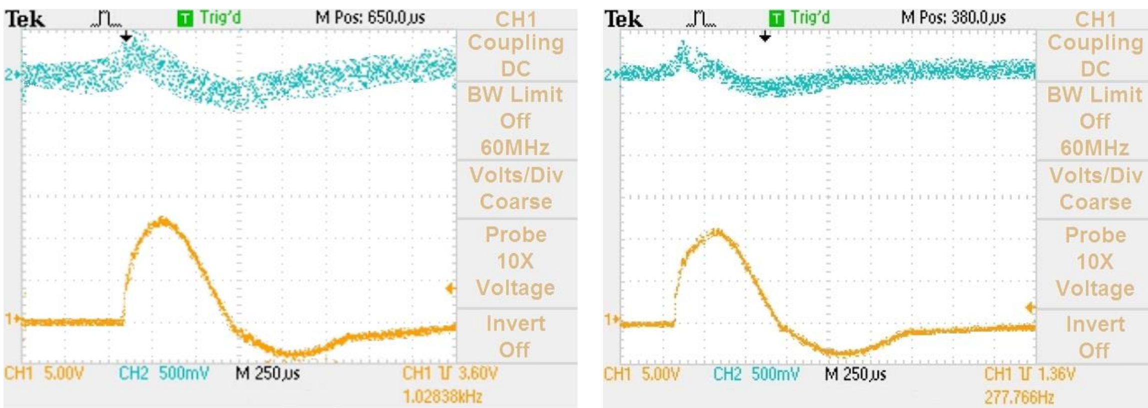
(a) **(b)**

Source: Own elaboration

Estimation of Implantation Dose

Definition of parameters of the ion implantation processes upon metallic substrates such as the estimation of the implanted dose, allows to determine the effect of the treatment and/or behavior of the material on its physicochemical properties. Within the literature, implantation dose values within the range of $1 \text{ x } 10^{15}\ \text{ions/cm}^2$ and $1 \text{ x } 10^{17}\ \text{ions/cm}^2$ are commonly reported and by several authors [17]–[23].

Figure 2. Current and voltage pulses discharge of (a) Ti ion implantation, and (b) Ti + N hybrid treatment



(a) **(b)**

Source: Own elaboration

Once the experimental parameters are known, the values of the implantation dose of Ti and both Ti + N particles on the carbon steel surface in question, are obtained when replacing them in the equation (5). The pulse duration and repetition frequency were established at 2.5 x 10^{-4} s and 30 Hz, respectively. The value of the ionic current is obtained from the area under the curve of the pulse discharge shown in figure 2. The ion-electron coefficients were obtained from those reported by J. R. Conrad *et al.* [24] for an AISI 304 steel cathode and an energy of 10 KeV (γ = 3.0). The results obtained from the model implemented to estimate the density of metallic and non-metallic implanted ions on an AISI/SAE 1020 carbon steel, are in agreement with those doses reported by other authors in the literature [17]–[23]. The values of the dose calculations implanted with Ti and the hybrid treatment Ti + N are shown in the table 1.

Table 1. Calculation of implantation dose

Parameter	Treatment with Ti	Hybrid treatment (Ti+N)
Ion current (A)	0.045	0.034
Total exposed area (cm^2)	85.54	85.54
Ions flux(ion/cm^2s)	$3.284x10^{15}$	$2.463x10^{15}$
Dose($ions/cm^2$)	$1.478x10^{16}$	$2.127x10^{16}$

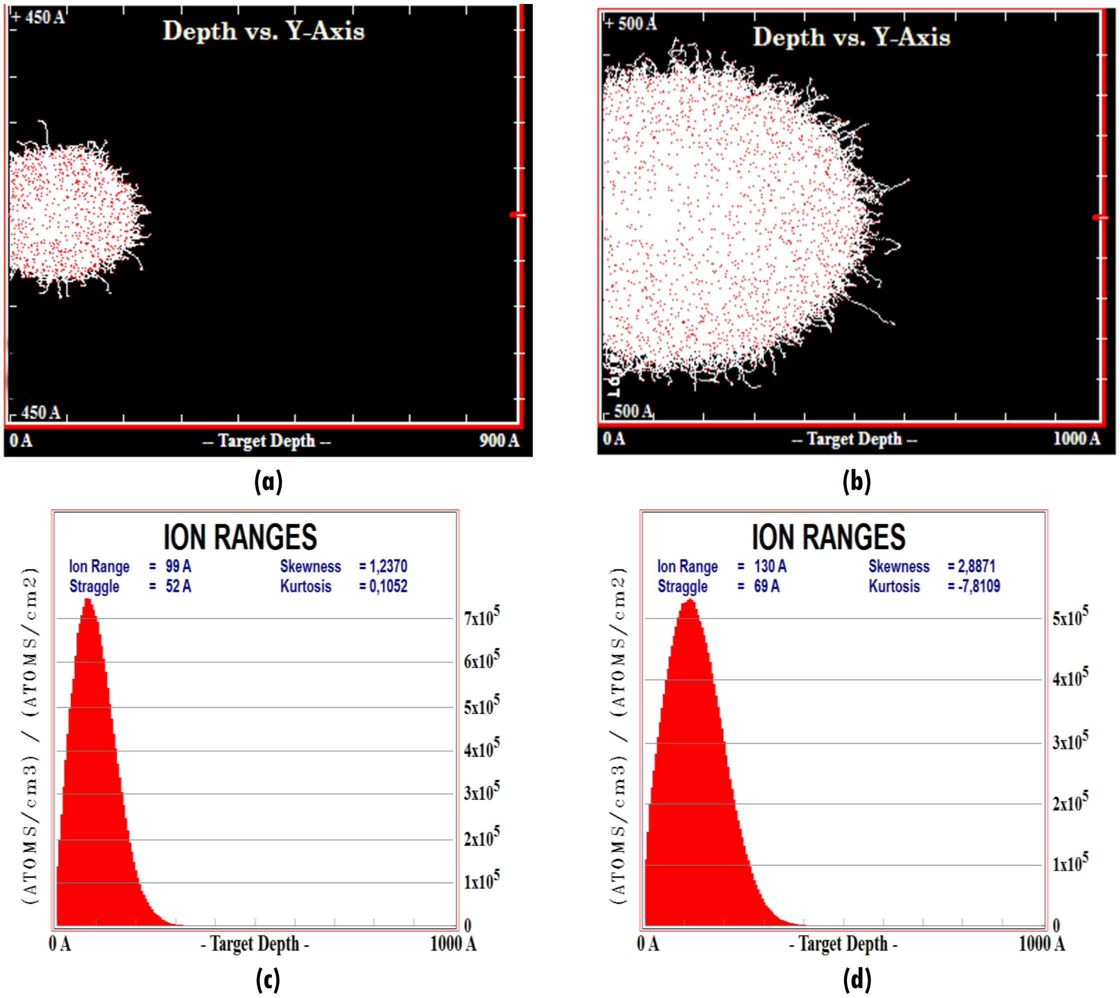
Source: Own elaboration

SRIM-TRIM Simulation Results

As to the simulation by using SRIM/TRIM, it was performed from an ions flux at $3.2 \text{ x } 10^5$ $ions/cm^2$ and $7.2 \text{ x } 10^5$ $ions/cm^2$ of Ti and N, respectively. Such magnitudes correspond to a certain implanted dose fraction or a theoretical ion density estimated in the previous numeral. Likewise, this calculation basis was also established in order to have a rough idea of the behavior of ions and not to extend in the calculation time during the simulation. Titanium and Nitrogen ions were accelerated perpendicularly onto the surface of a carbon steel at a 10 keV energy, obtaining trajectory and depth-concentration profiles of ionized species by electric arc (Ti) and by a hybrid treatment with high voltage pulsed discharges (Ti + N).

In figure 3 it is shown the distribution profile and the path of the Ti (figure 3a) and N (figure 3b) ions accelerated at a 10 keV voltage upon the surface of an AISI/SAE 1020 carbon steel whose elemental composition, an input variable required by the software, was based on the results obtained by optical emission spectroscopy in other works reported by Sanabria [25], [26]. It is observed the irregular trajectories due to the polycrystalline structure of the base material and the magnitude of the applied voltage; the path of implanted ions reaches depths in the material up to 300 Å for Ti molecules and 600 Å for N molecules. Likewise, in figure 3c and figure 3d, it is illustrated the 2D depth-concentration profiles of metallic and non-metallic ions implanted at 10 keV. It is noticed that the highest concentration of Ti and N ions were found at 99 Å (figure 3c) and 130 Å (figure 3d), respectively. This indicates that ionized Ti particles by electric arc will be located in the more superficial layers of the material, obtaining a greater concentration of metallic species in the outermost regions of the material compared with the non-metallic species in the hybrid treatment.

Figure 3. Concentration-depth profiles of (a) Ti and (b) N, and ions trajectory of (c) Ti and (d) N into a carbon steel target

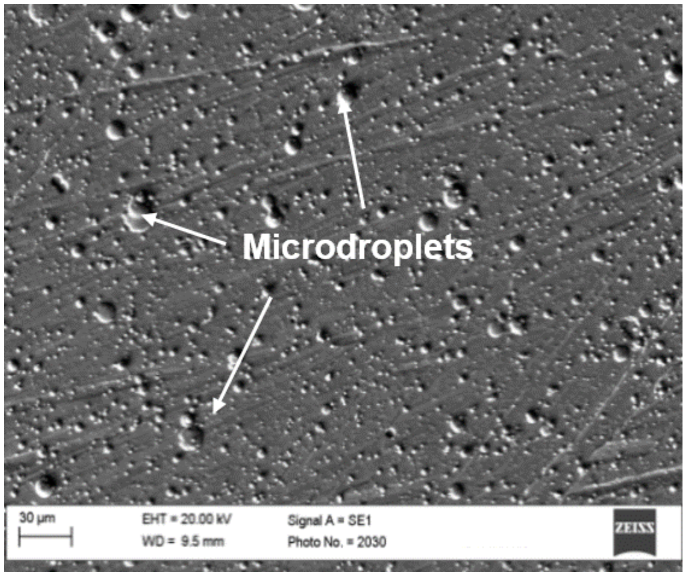


Source: Own elaboration

Surface Morphology Analysis

From the SEM photomicrograph of AISI/SAE 1020 steel surfaces in figure 4, is noticed a coating of Ti with multiple microdroplets, product of the non-ionized species (clusters formed during evaporation of the Ti cathode) and of the operating characteristics of the electric arc system implemented for evaporation of metals. Generally, the production of the microdroplets is due to the instability of the cathodic spot, which can be controlled from experimental parameters such as electric current as mentioned by Tsygankov *et al.* [27], [31]; Mussada and Patowari [28]; Parada *et al.* [29]; Valbuena *et al.* [30], and Dugar-Zhabon *et al.* [32].

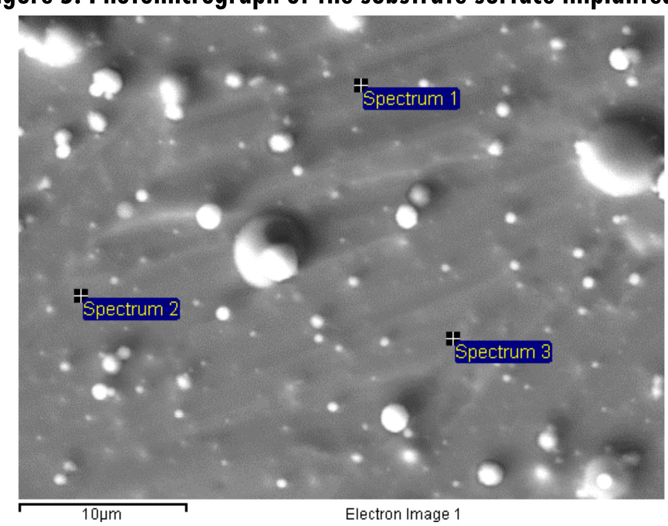
Figure 4. Photomicrograph of AISI/SAE 1020 steel surface modified with Ti ions



Source: Own elaboration

Figure 5 illustrates the photomicrographs obtained by EDS technique together with the results of the elemental composition analysis reported in table 2 whose values correspond to the regions of interest (spectrum 1-3) distributed upon the substrates implanted with Ti ions. In addition to the typical alloy elements present in this type of carbon steel, the spectra also detected the presence of a high Ti content and a low iron (Fe) content upon the surface due to the thickness of the coating produced by the electric arc discharge, which validates the effect of the ion implantation technique upon ferrous alloy by enriching the solid surface with metal species.

Figure 5. Photomicrograph of the substrate surface implanted Ti



Source: Own elaboration

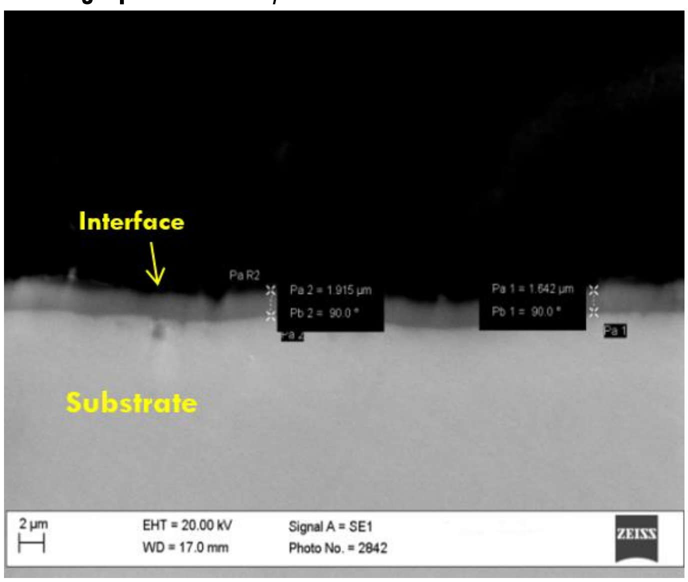
Table 2. Elemental analysis by EDS of the substrate surface implanted with Ti

Element	Spectrum (wt. %)		
	1	2	3
Fe	1.33	1.29	1.27
Al	0.36	0.36	0.43
Ti	98.31	98.35	98.30

Source: Own elaboration

As for the results obtained by a cross-sectional photomicrograph and depicted in figure 6, it is shown the substrate-coating interface, where is observed the thickness of the implantation zone which varies between 1.6 micrometers and 1.9 micrometers. Such thickness of the formed amorphous layer can be attributed to the ionization energy in the implantation process; allowing the deposition of the dopant particles onto the near-surface layer of the substrate.

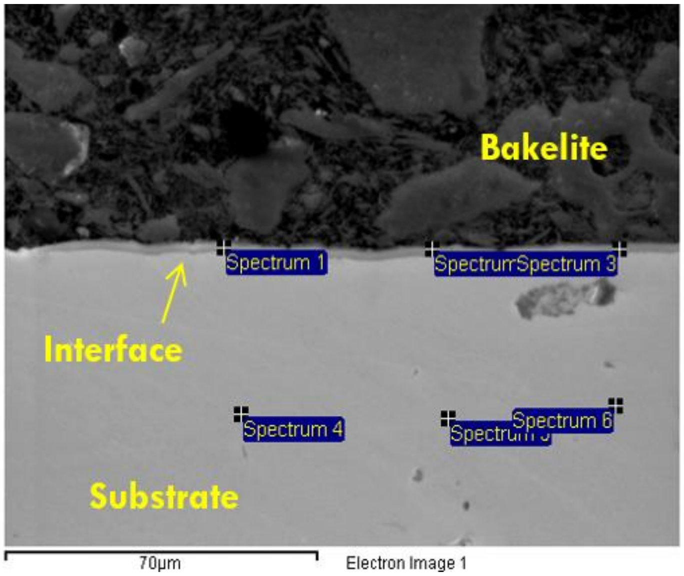
Figure 6. Photomicrograph of the AISI/SAE 1020 steel cross-section modified with Ti ions



Source: Own elaboration

Figure 7 and table 3 shows the EDS results for a cross section of the AISI/SAE 1020 steel implanted with Ti ions. In the coating-substrate interface, a region with a high Ti content (spectrum 1 to spectrum 3) is evidenced and gradually reduced at deeper layers, while the interior of the metal is mainly constituted by Fe (spectrum 4 to spectrum 6), validating the fact that only the bombarded species onto the surface will be placed in the outer layers of the implanted material.

Figure 7. Photomicrograph of the cross-section of the substrate implanted with Ti



Source: Own elaboration

Table 3. Elemental analysis by EDS of the cross-section of the substrate implanted with Ti

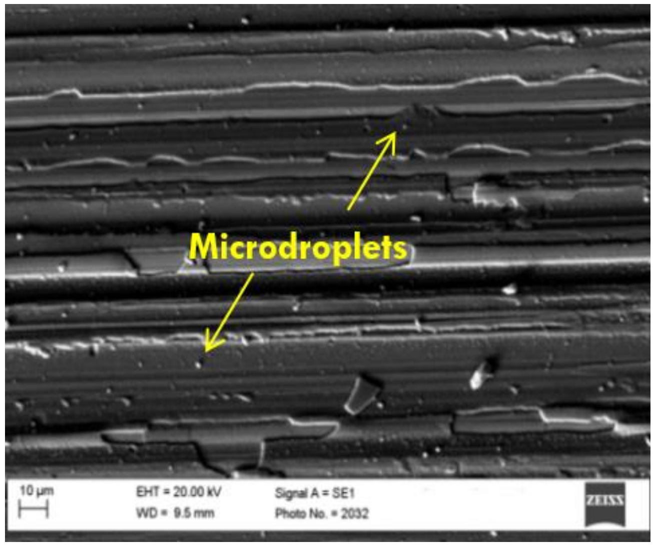
Element	Spectrum (wt. %)					
	1	2	3	4	5	6
Fe	2.32	2.05	1.93	99.30	99.33	99.33
Al	0.75	0.97	0.84	-	-	-
Mn	-	-	-	0.70	0.67	0.67
Zn	0.60	2.28	-	-	-	-
Si	-	0.87	-	-	-	-
Ti	96.33	93.83	97.23	-	-	-

Source: Own elaboration

Turning to the hybrid treatment, the figure 8 illustrates the photomicrograph of the surface of the carbon steel substrate implanted with Ti + N ions. The laminar distribution of the coating mainly depends on the surface finish obtained from the metallographic surface preparation of the substrates prior the ion implantation process [33]–[35].

Additionally, the photomicrograph shows a decrease in microdroplets compared with that presented in figure 6 (surface modified with Ti ions). The decrease in microdroplets is due to the presence of a combined atmosphere of metallic (Ti) and non-metallic (N_2) species that during the ionization of Ti atoms and N_2 molecules, produced by the electron flow of the high voltage pulsed electrical discharge at low pressures, reduces the formation of Ti clusters achieved by means of the electric arc discharge [27]–[31].

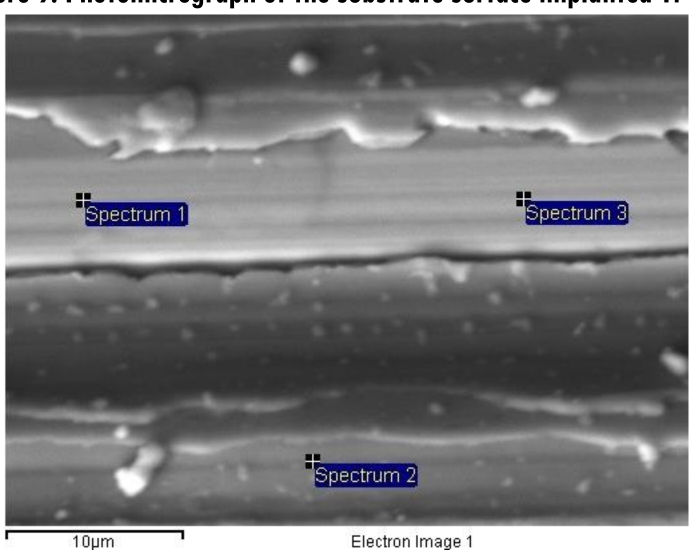
Figure 8. Photomicrograph of AISI/SAE 1020 steel surface modified with Ti + N ions



Source: Own elaboration

Figure 9 and table 4 shows the EDS results made across the region of interest upon the surface of AISI/SAE 1020 steel substrates implanted with Ti + N ions. Spectrum 1 to spectrum 3 on the figure 9, reveal the presence of Ti, N, and Fe which are, as expected, the most abundant elements of this ferrous alloy. The Ti and N contentment is due to the simultaneous bombardment of metallic and non-metallic species implanted by the hybrid treatment when exposing the samples to both electric arc and high voltage pulsed discharges at low pressures.

Figure 9. Photomicrograph of the substrate surface implanted Ti + N



Source: Own elaboration

Table 4. Elemental analysis by EDS on substrate surface implanted with Ti + N

Element	Spectrum (wt. %)		
	1	2	3
Fe	51.99	9.47	60.83
Mn	0.40	0.36	0.48
Si	-	-	0.28
Ti	26.42	68.38	19.17
N	21.19	21.79	19.24

Source: Own elaboration

Conclusions

From the experimental parameters defined in this work, it was determined that the dose of Ti and N ions, implanted upon AISI/SAE 1020 steel substrates at a 10 keV energy, correspond to 1.478×10^{16} $\mathrm{ions/cm^2}$ and 2.127×10^{16} $\mathrm{ions/cm^2}$, respectively. These values are in agreement with the ranges reported in the literature by implementing ion implantation technologies for surface protection.

The distribution and depth-concentration profiles obtained from the simulation in the SRIM/TRIM, provide an idea of the distance travelled by ions implanted in the material at a certain energy. The depths achieved by Ti and N ions applying a 10 keV voltage were approximately 300 Å and 600 Å, respectively, demonstrating that non-metallic ions reach a greater depth than metallic species into a ferrous alloy.

The formation of microdroplets upon the coating formed upon the surface of AISI/SAE 1020 steel when it is implanted with Ti ions were identified, being greater than the surface treatment implanted with both Ti and N ions. The elemental composition spectra obtained from EDS analysis, detected the presence of Ti and N particles on the surface of the carbon steel in question. In addition to this, it was obtained a comparable compositional gradient between the compositional spectra by EDS analysis and the compositional distribution profiles of the implanted species in the simulation results.

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