ION IMPLANTATION ON NICKEL TARGETS BY MEANS OF A REPETITIVE PLASMA FOCUS DEVICE

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Riassunto
Sono stati fatti test per verificare il possibile uso di una macchina Plasma Focus come implantatore di ioni. L'impianto utilizzato è il Plasma focus ripetitivo in funzione presso il Centro ENEA del Brasimone. Il campione da implantare è un foglio di nickel con una superficie di circa 20 cm$^2$ inserito in una struttura rigida la cui distanza può essere variata con riferimento all’estremo superiore dell’anodo. La macchina Plasma Focus è operata a 0,5 Hz con una pressione di riempimento di Azoto a 4 mbar. Dopo l’irradiazione, il campione è analizzato per mezzo di spettroscopia Auger che fornisce la concentrazione dei vari elementi a differenti profondità d’implantazione. Il risultato dell’analisi dimostra che il Plasma Focus è un’efficace sorgente per implantazione ionica, anche per applicazioni metallurgiche. I risultati sono confermati dalla misura della durezza del campione prima e dopo l’implantazione.

Abstract
Some test has been done in order to assess the possible use of a Plasma Focus as an implanter. The device utilized is the repetitive Plasma Focus operating in the ENEA Brasimone Center. The implanted sample is a sheet of Nickel with a surface of about 20 cm$^2$ inserted in a rigid sample at a variable distance from the top of the anode. The Plasma Focus device is operated at 0,5 Hz using a Nitrogen filling gas at a pressure of 4 mbar. After irradiation the sample is analyzed with Auger spectroscopy that provides the surface concentration of the various elements on the sample at different implantation depths. The result of the analysis shows that the Plasma Focus is an effective implantation source, even for metallurgical applications. Those results are confirmed by the measurement of the hardness of the sample before and after the implantation.

Key words: Plasma Focus, Ion Implantation, Auger spectroscopy, Metallurgical hardness
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1 INTRODUCTION

The Plasma Focus (PF) is a device consisting in two coaxial electrodes in vacuum connected to a fast high voltage capacitor bank and separated by an insulator. When the HV is applied to the electrodes trough a spark gap, an electrical breakdown develops on the surface of the insulator and the discharge is driven by the Lorentz force to run along the gap between the electrodes at a speed of some $10^7$ cm/s and when reaches the end of the inner electrode collapses on the axis of the device focusing in a hot blob of plasma (Plasma Focus). In this phase strong electric fields are generated that produce intense ion beams. The duration of each discharge is of the order of 10 $\mu$s.

Normal implanters are basically made of an ion source, an acceleration stage and an extraction stage. They are expensive and require high vacuum. Normally the sample is implanted inside a large vacuum chamber. As a matter of fact most of the treatments are done on relatively small samples (tools, prostheses, catheters). In our opinion the advantage of the PF respect the existing ion sources can be in the easiness of the operation and in the reliability. For instance the device operates at pressures of the order of ten mbar allowing the connection to large objects to be implanted without putting them in a large vacuum chamber.

The study of the characteristics of the ion beams emitted by the PF machines has been and is currently the object of a wide set of experiments that have produced a large amount of papers in the literature [1-7]. Our work is focussed not on the study of the characteristics of the beams or on optimizing at a fine level the production, but on the gross characteristics of the
emission measured in terms of the effective capability of modifying the properties of the target samples. For normal applications, the order of magnitude of the implantation density required is: $10^{14}$-$10^{15}$ ions/cm$^2$ for semiconductor technology; $10^{16}$ ions/cm$^2$ for ion mixing technologies; $10^{17}$ ions/cm$^2$ for metallurgical applications.

2 EXPERIMENTAL SET-UP AND PROCEDURE

We operate our PF at 6 kJ, 21 kV and with central electrode of positive polarity (anode). The discharge current is of the order of 0.5 MA. The central hollow electrode, made of tungsten, has a diameter of 34.5 mm and a length (outside the insulator) of 7.5 cm. The outer electrode is a cylinder made of stainless steel with a radius of 4.5 cm and a height equal to that of the total central electrode (electrode + insulator length). A turbo pump provides the initial vacuum and no particular care has been taken to assure the cleanliness of the vacuum. The gas to be implanted is in the vacuum chamber at a pressure typically of 4 mbar. The insulator, made of quartz, is a cylinder of 8.6 cm height and the total surface exposed to the plasma is 54 cm$^2$. The implanted sample is a sheet of Nickel with a surface of about 20 cm$^2$ and a thickness of 0.1 mm. The sample was inserted in a rigid sample-holder made of SS that keeps it parallel to the electrode at a distance that can be varied. The discharges are made using pure Nitrogen. The distance of the sample was initially set on the axis of the machine at a distance of 0 cm from the upper part of the central electrode. The sample was immediately destroyed evidently by the shock due to the impact of the plasma sheet. The distance sample-electrode has been then progressively increased up to a value of 20 cm where no visible damage was produced by large series of discharges. The sample was removed after 62 discharges (at 0.5 Hz and at a filling pressure of 4 mbar) and analysed with Auger spectroscopy. It should be noted that after the series of discharges the central electrode presented just on axis a clear hole showing that material was removed (probably by the impact of high energy electron beams produced in the PF).

The Auger technique is perhaps the most versatile method to analyse surface atomic composition, because it has good sensitivity for a wide range of elements and can be reasonably quantitative. The depth resolution of Auger technique is only few angstroms and deeper regions of the sample are analysed by progressively removing the deposited material with an Ar ion beam.
3 EXPERIMENTAL RESULTS

Figure 1 shows the results of the Auger analysis. In Figure 2 it is put in evidence the behaviour of N implantation from Fig. 1. In Figures 1 and 2 there are shown the measured concentrations of the various elements found on the surface progressively eroding the sample at a rate of one mono-atomic layer.

A measure on a mono-atomic layer volume is made each 70 nm in the erosion process. It should be mentioned that some traces are present of Fe, O and C that we attribute to the preparation procedure of the sample. The Auger measurements have been performed on four zones of the sample, located respect the PF axis at P_1=1 cm, P_2 = 0.75 cm, P_3 = 1 cm and P_4=2.25 cm in different azimuthally directions. No significant variations of the results are observed in the four zones. The results reported in Fig. 1 and 2 refer to the zone P_1.

Looking in particular to the Nitrogen implantation, upon integration of the concentration up to the maximum penetration (about 900 nm), and being the inter-atomic distance of the Ni substrate equal to 2.2 Å, it is found that the surface content of nitrogen after implantation in the four measurement zones is N_{P_1}=4.7 \times 10^{17} \text{ atoms/cm}^2, N_{P_2}=3.6 \times 10^{17} \text{ atoms/cm}^2, N_{P_3}=4.1 \times 10^{17} \text{ atoms/cm}^2, N_{P_4}=3.7 \times 10^{17} \text{ atoms/cm}^2. The maximum variation from the average of the content is so less than 17%.

\begin{figure}[h]
\centering
\includegraphics[width=0.4\textwidth]{Fig1.png}
\includegraphics[width=0.4\textwidth]{Fig2.png}
\caption{Figure 1 - Surface concentration of W, Ni and N found with Auger analysis on the Ni irradiated target. Figure 2 - Surface concentration of N on the Ni irradiated target (same of Fig 1 on an expanded scale).}
\end{figure}
4 DISCUSSION

The main difficulty in interpreting the experimental results is due to the presence of a large amount of W. On the surface it reaches a concentration greater than 50% and it is present on the sample up to a depth of 850 nm with a concentration of about 10%, lower detectable limit for such element with our Auger apparatus. Two different mechanisms can produce the presence of W on the sample.

The first deals simply with the implantation of the W extracted by the electron beam generated by the PF from the centre of the anode where it is clearly visible that material has been removed by the discharges. The W ions should be accelerated toward the sample by the same electric field (and voltage) causing the relativistic electron beam. An evaluation of the W energy spectrum from the implantation range into the sample, in the hypothesis of single ionized ions, made with the same procedure that in the following is performed for the implantation of Nitrogen, indicates a spectrum extending from 1.5 to 8 MeV. In the literature it is reported that the electron beams in the PF have energies of the order of 1 MeV. Moreover multiple ionized ions (up to 8) are reported reaching energies of 12-14 MeV [1,8]. The intensity of such beams is nevertheless quite low. We cannot so exclude such mechanism but we consider it unlikely.

The second mechanism deals with the melting of the sample surface hit by the splashing of the plasma sheet in the late part of his life when it is strongly doped by the material coming from the electrodes (practically a W plasma). Consequently an alloy is formed between W and the sample material. The fact that the sample was systematically macroscopically damaged when placed at a distance less than 20 cm from the electrode is an indication that the acting mechanism is the second.

It is anyway clear that the deposition of W on and inside the sample is cumulating with the number of discharges. The composition of the sample is so varying al long as the PF discharges go on. This fact is important in deducing the energy spectrum of the N ions implanted on the sample by the measure of the projected range, which depends upon the sample composition. In evaluating the spectrum we use the following approximate formula [9]:

\[
E_{[keV]} = \frac{R_{[nm]} \cdot \rho_{[g/cm^3]} \cdot Z_f^{2/3} \cdot (3 \cdot M_1 + M_2)}{39 \cdot (M_1 + M_2)}
\] (1)
Where $R_p$ is the projected range of the implanted ion (i.e. the projection of the range along the normal to the sample surface), $E$ his energy, $\rho$ the density of the target material, $M_2$ is the atomic weight of the target (sample) atoms, $M_1$ and $Z_i$ are the atomic weight and the atomic number of the implanting ions.

Equation (1) is valid if the average energy loss of the ions into the target material results from elastic collisions with the target atoms (nuclear stopping or elastic energy loss). The alternative mechanism of loss is toward the electrons of the target atoms (electronic stopping or inelastic energy loss). The use of (1) represents well the ion energy spectra reported in the following for energies greater than about 100 keV for Nitrogen impinging ions.

Taking into account the approximations and the intrinsic range straggling, it descends that the precision of the deduced energy spectrum is quite poor. Anyway this is a general limit of the method unless very refined measurements are not performed. We evaluate that the precision of the deduced energy spectrum is of the order of 30%.

Figure 3 reports the energy spectrum deduced with the elaboration of Fig. 2 for the implantation of Nitrogen. As the implantation takes place on a sample having a composition varying as the series of discharges is going on, in Fig. 3 the two different spectra are reported, corresponding to the implantation on a sample having the initial composition (i.e. pure nickel) and on a sample having the final composition, i.e. that of Fig. 1. The real spectrum is then laying somewhere between the two reported spectra. Anyway this result is acceptable considering the above-mentioned small precision of the method. In evaluating the energy spectrum of the N ions impinging on the alloy Ni+W the following relationship for the projected range has been used [8]:

\[
N_{\text{ion energy}} \text{ (keV)}
\]

\[
\text{Surface Concentration } \times 10^{13} \text{ (atoms/cm}^2)\]

\[
0 \quad 10 \quad 20 \quad 30 \quad 40 \quad 50
\]

\[
\text{N on Ni}
\]

\[
\text{N on Ni+W}
\]

\[
0 \quad 200 \quad 400 \quad 600 \quad 800 \quad 1000 \quad 1200 \quad 1400
\]

\[
\text{Figure 3 - N implantation on two different backgrounds.}
\]
\[ R_p(A_xB_y) = (x \cdot N_A + y \cdot N_B) \cdot \left( \frac{R_p(A)}{N_A} \cdot \frac{R_p(B)}{N_B} \right) \]

(2)

Where \( x \) and \( y \) are respectively the relative number of atoms of the two components \( A \) and \( B \) normalized to 1 (\( x+y=1 \)) and \( R_p(A) \), \( R_p(B) \), \( N_A \) and \( N_B \) are the projected ranges and the atomic densities of pure hypothetical substrates of the two elements.

The energy spectrum on Ni+W of Fig. 3 is obtained by applying Eq. (2), using the composition of Fig. 1 in steps of 70 nm.

5 METALLURGICAL TEST

As mentioned in the Introduction the measured density of the implanted N atoms implies that modifications of the metallurgical properties of the sample have to take place. A measure of such properties is then a method to assess the correctness of our measurement (at least in order of magnitude). We have measured the hardness of the sample before and after the implantation. This metallurgical characteristic shows before the implantation a value of 142 in the Vickers scale. The results of the measurement after the implantation are shown in Fig. 4.

The result is perfectly consistent with the implantation density measurement showing an increase of the hardness up to 55% on a surface of about 20 cm².

![Figure 4 - Hardness of the Ni sample after the N implantation. Scanning along three parallel lines.](image-url)
6 CONCLUSIONS

Our results show that the PF is an effective source for implantation even for metallurgical applications (i.e. about $10^{17}$ ions/cm$^2$ and depth of some 100 nm). The implantation appears uniform within 17% on an area of 16 cm$^2$.

The analysis of the experimental results shows nevertheless that the PF beam presents a broad energy spectrum, a characteristic not compatible with semiconductor technology applications.

Besides the implantation of the filling gas ions, some macroscopic modification takes place on the sample surface due to the material of the electrode impinging on it. The mechanism is not clear but it can be due to the melting of the sample surface upon the impingement of the plasma sheet with the consequent formation of a surface alloy on which the filling gas ions are implanted. Depending on the applications the possibility of creating under controlled conditions a surface alloy or a convenient substrate on the target material can have a positive impact on the usefulness of the PF as an implanter.
REFERENCES


