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Plasma Immersion Ion Implantation with a 4kV/10kHz Compact High Voltage Pulser

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Abstract. Development of a 4 kV/10 kHz Compact High Voltage Pulser and its application to nitrogen plasma immersion ion implantation (PIII) of different materials as Si, Al alloys, SS304 stainless steel and Ti alloys are discussed. Low voltage (1-5 kV) pulses at high frequencies (up to 20 kHz for 2 kV) were obtained with maximum power delivered at 5 kV, 7 kHz. These conditions were not sufficient to reach temperatures above 200ºC in the samples because of short duration of the pulses. However, very shallow implantations of nitrogen in Si, Al5052, SS304 were observed by Auger electron spectroscopy and improved corrosion resistance was obtained for Al5052 when it was treated by nitrogen PIII at 2.5 kV, 5µs and 5 kHz pulses.

Keywords: plasma immersion ion implantation, compact high voltage pulser, Si, Al5052, SS304.
PACS: 52.77Dq

INTRODUCTION

Compact High Voltage Pulsers (CHVP) are important electronic devices that are being developed and improved with the aim to applying them in as diverse fields as aerospace, nano and microelectronics [1], low voltage plasma immersion ion implantation (PIII) nitriding [2], surface enhancement of polymers [3] and optoelectronic materials [4], medical [5], etc. Their light-weight and compactness in comparison to commonly used large and heavy high voltage pulsers are the reason of their possible success in the above mentioned fields that require somewhat modest voltages (1 to 5 kV) but high frequencies (over 5kHz). We have utilized semiconductor switching and step-up transformers instead of hard tube system for this pulser downsizing [6, 7, 8].

PIII is a relatively newly developed technique for surface modification of materials, specially suited for industrial components. This treatment consists of immersing the samples or workpieces in a plasma and applying a negative high voltage pulses to the target. Ions of interest are then extracted from the plasma, accelerated towards the surface (mainly at normal incidence) and implanted tridimensionally.

Normally, plasma implantation process imposes several requirements on the power supplies such as square wave high voltage pulses (in the range of 5kV to 40 kV), short rise-times (less than 1.0 µs) and pulse duration varying from 5 to 50µs at a certain repetition rate (typically between 300Hz and 1kHz). Compared with conventional ion beam implantation (IBI), PIII process has several advantages such as no need of beam guiding or target manipulation and ability of implanting objects with complex and irregular forms. In addition, by using this technique it is possible to obtain an improved conformal implantation if the ion sheath is completely conformal around the target. In practice, however, corners and edges of the real objects increase the non-uniformity of implant, especially if the plasma sheath is too thick. One way of circumventing this problem is to apply lower voltage pulses (1 kV to 5 kV) of shorter duration (1 µs to 5 µs) for producing thinner plasma sheath, which improves the uniformity of ion implantation of treated pieces (especially those with irregular shape) but at the cost of decreasing the thickness of the implanted layer.

However, in certain applications as ultra-shallow junctions, very thin treated layers are being pursued. Also in some applications in biomedical engineering, thin layers of modified materials with enhanced properties can be obtained through low energy PIII.

Therefore, we devised for plasma implantation systems a solid-state compact modulator, in which a small capacitor discharges through a forward converter composed of a low blocking voltage IGBT switch (of only 1 kV) and three step-up pulse transformers, rather than employing comparable hard-tube devices such as

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in conventional implant pulsers that are of large size, expensive and cumbersome [8].

Previously, we have reported mainly on the electrical performance of CHVP with 4kV/2A/5kHz nominal capability [8]. In the present paper, emphasis will be given to the surface modification results obtained by using an improved version of CHVP for plasma immersion ion implantation. With a maximum output of 4kV and operating at 10kHz, this pulser was applied to nitrogen PIII of Al5052, SS304, Si, polymers and Ti6Al4V, showing shallow implantation of N, as expected. In the Al alloy, a significant improvement of its corrosion resistance was measured even with such thin modified layer. Comparison of such material surface performances obtained at different operating conditions of CHVP are discussed in this paper, confirming the usefulness of this kind of low cost pulsers in PIII research.

EXPERIMENTAL METHODS AND APPARATUS

The details of the circuit and the electronic performance of the developed CHVP solid-state pulser are discussed elsewhere [8, 9].

For the implantation tests, the pulser was connected to a PIII system described in Fig. 1. The DC glow discharge is powered by simple power supply capable of providing 900V, 0.6 A maximum output. The nitrogen plasma is initiated between the cylindrical stainless steel rod and the neck connected to the PIII cylindrical chamber with 27 cm diameter and 47 cm length, as shown in the same figure. The stainless steel rod is connected to a cooper feed-through to apply the negative high voltage pulse. We typically run the discharges at powers of 180 W, with corresponding discharge voltages of 600 V and currents of approximately 0.3 A. Under these conditions, the plasma potential near the center of the PIII plasma chamber reaches 350 V, which in some cases can be deleterious for PIII processing due to the sputtering of the surface under treatment. To reduce plasma potential, an electron shower hot filament is used. By adjusting the power of this electron source, the plasma potential can be controlled from zero to 350V [10].

For the measurements of the microstructure of the nitrogen implanted materials, Philips PW 1830, x-ray diffractometer in the standard 2θ scan mode was used. For the Auger Electron Spectroscopy (AES) measurements, used for the determination of elemental concentration in the treated samples, a spectrometer from FISONS Instruments Surface Science, model MICROLAB 310-F was used.

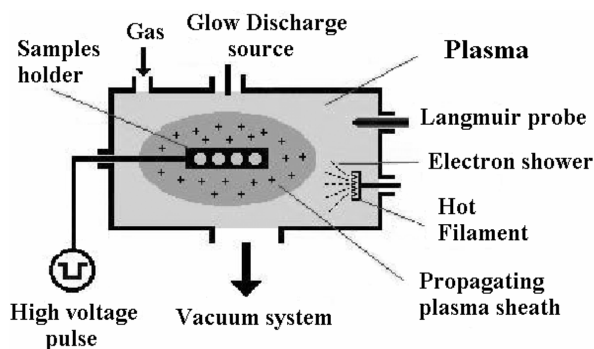

FIGURE 1. Schematics of the plasma immersion ion implantation experiment.

The corrosion resistance of the treated materials was measured by a potenciostat/galvanostat AUTOLAB, model PGSTAT30, with three electrode configuration inside an electrolytic cell. The cell contained a solution with 3.5% ppm of NaCl with pH equal to 6. The potentials were measured with respect to a reference electrode made of Ag/AgCl.

For the rocking curve measurements, a Philips X-PERT MRD, high resolution x-ray diffractometer was used.

RESULTS AND DISCUSSION

The experimental results concerning the electronic performance of CHVP in the present configuration are discussed shortly. Tests in the resistive load of 2 kΩ showed that pulses of 5 μs duration with rise and fall times of 1 μs each were obtained at frequencies of up to 10 kHz. Currents of up to 10 A were obtained during the pulses. With a nitrogen plasma load, we obtained pulsed voltages with square forms and currents with opposed peaks at the start and the end of the pulses with a plateau in between. For 2.5 kV, 5 μs pulses we achieved current plateau of 0.15 A, while pulsing at 5 KHz. We have performed many materials treatments under this condition.

We also performed parameter survey of the plasma and the pulser conditions. We were able to achieve conditions of very high frequency of 20 kHz but with lower voltages, below 2 kV. On the other hand, 5 kV pulses were maintained at 7 kHz, while pulse currents in plasma as high as 0.4 A were attained. We summarize the PIII operating conditions with CHVP in Table I.

In Fig.2 we show the profiles of implanted nitrogen and impurity oxygen, for the sample of Si treated under condition 1. The nitrogen penetration in this case was less than 10 nm, with peak concentration of about 15%, at around 5 nm. The contamination of

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oxygen was very high (up to 70% at the very surface) due to native oxide layer or oxygen residual gas in the chamber during PIII treatment. This situation could be improved by using better pumping systems and argon cleaning discharges.

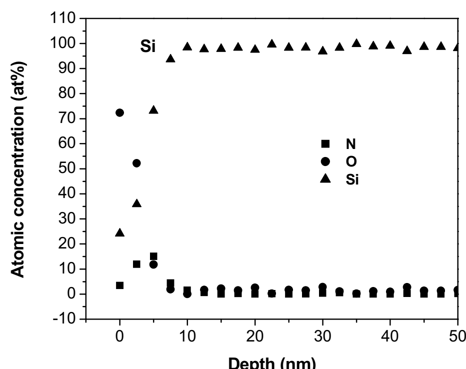

FIGURE 2. Atomic concentration profiles for the samples of Si treated by nitrogen PIII

In Fig. 3 we show the profiles of implanted nitrogen and impurities for the Al5052 sample treated under condition 1. Oxygen was again the predominant impurity but C and Fe were also present in this case. Nitrogen peak concentration was 15% and the reach was near 30 nm. Other elements shown are the usual constituents of the alloy.

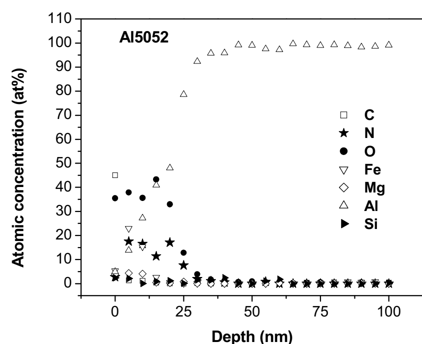

FIGURE 3. Atomic concentration profiles for the samples of Al5052 treated by nitrogen PIII

Samples of SS304 implanted with nitrogen treated under condition 1 showed AES profiles as in Fig.4. The peak concentration was near 25% while the maximum reach of nitrogen was less than 20 nm. Oxygen contamination was also high (near 40%) while some carbon was present near the surface. Chromium concentration was reduced at the surface but nickel concentration was increased (segregation effect?).

These implantations resulted in the following changes for the SS304: a) As was seen in the XRD results (not shown here), we could only observe the formation of the α-phase, indicated by a diffraction

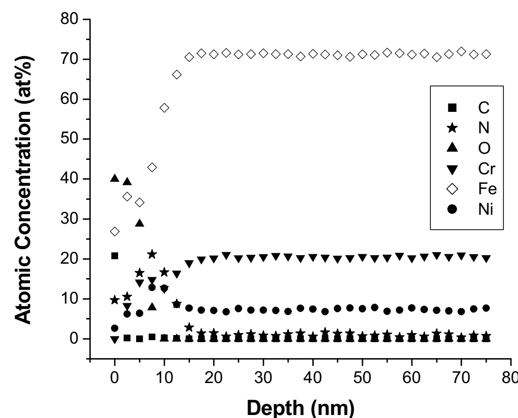

FIGURE 4. Atomic concentration profiles for the samples of SS304 treated by nitrogen PIII

peak to the right of the γ(111) peak at 2θ= 44°. No changes were seen for the corrosion resistance or microhardness of the surface.

Meanwhile, for the Al alloy treated under condition 1, there was a significant improvement in the corrosion behavior of the surface. As shown in Fig.5, the corrosion current density was reduced by an order of magnitude. Furthermore, the corrosion potential was improved from -680 mV to -250 mV and the alloy was passivated in the range of -250 mV to 1.5 V, after the implantation.

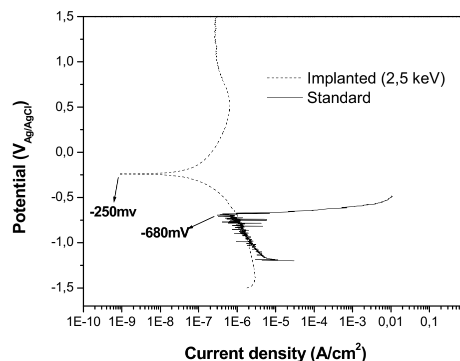

FIGURE 5. Polarization curves of Al5052 for untreated and nitrogen PIII treated samples, respectively

As for Si treated with nitrogen PIII under condition 1, it showed clear distortion in its rocking curve compared to the untreated one, as shown in Fig.6. This distortion to the left of the Si diffraction peak is indicative of strained layer due to the nitrogen implantation or defects resulting from the bombardment by those ions. Samples of materials treated under other conditions listed on Table I are being analyzed presently.

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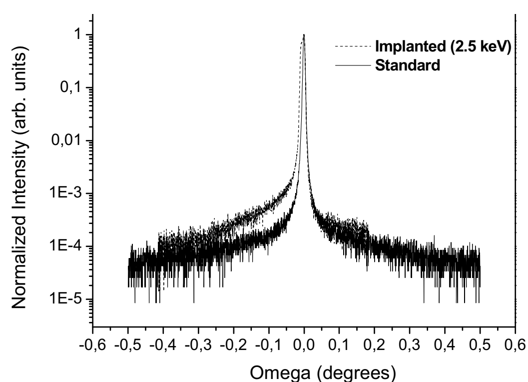

FIGURE 6. Rocking curves of untreated and nitrogen PIII treated samples of Si

CONCLUSIONS

A compact high voltage pulser (CHVP) nominally rated 4kV/5μs/10kHz was tested in terms of electronics and successfully applied to surface modification of different materials by plasma immersion ion implantation (PIII) at low energies (1- 5 kV) at high frequencies (up to 20 kHz at 2 kV). Our CHVP can stand continuous operation at such conditions for up to 1 to 3 hours, routinely. Plateau currents in plasma of up to 0.4 A were attained at 5 kV, when frequencies of 7 kHz were used. The CHVP combining IGBT switching with step up transformers (x3) allowed to reach such parameters in a very compact, lightweight, low cost configuration.

As a first application, this CHVP was applied to the nitrogen PIII treatment of different materials: Si, Al5052, SS304, Ti6Al4V. Polymers are also being tested. Because of short pulse length (5 μs), and relatively low voltages, very conformal PIII treatments of real workpieces are expected. Furthermore, very shallow implantations of nitrogen were achieved: less than 10 nm in Si, less than 20 nm in SS304 and 30 nm in Al5052.

For Si targets, this result is encouraging for shallow ion implantations needed for new frontiers in nano and microelectronics. For Al5052, despite of such thin treated layer, we observed substantial improvement of the surface against corrosion, when compared to the untreated specimens. In the case of SS304, only a transformation to alpha-phase was observed for the near surface layer, when 2.5 kV and 5 kHz (the same conditions of treatments for the Si and Al5052 also) was applied. We are presently pushing further the parameters of CHVP to achieve a thin layer of γ_N-phase in SS304 which presents nobler properties than its alpha-phase.

ACKNOWLEDGMENTS

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TABLE 1. Summary of operating conditions of CHVP for materials treatment by PIII processing

Treatment conditions	Implantation voltage (kV)	Pulse frequency (kHz)	Pulse duration (μs)	Implantation time (min)
1	2.5	5.0	5.0	60.0
2	4.0	10.0	5.0	60.0
3	2.5	10.0	5.0	180.0
4	2.0	20.0	5.0	60.0
5	5.0	7.0	5.0	60.0

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