



# Material-related effects during ion beam treatment by an end-Hall ion source



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## ABSTRACT

Reproducibility and accuracy of dry etching processes are a challenge for the operators of end-Hall ion sources with hot filament (HF) neutralizer. The variation of the neutralization current  $I_N$ , e.g. due to filament aging as well as the properties of the substrate material lead to changes in the resulting etch rate of the substrate. Therefore a special controller setup was developed to automatically neutralize the ion beam of an end-Hall ion source with HF by measurement of the substrate current and control of an introduced difference current ( $I_D$ ). In this study, the influence of conductive (amorphous CuTi-film), semi-conductive (B-doped Si(100)) and non-conductive material (128°YX LiNbO<sub>3</sub>) on  $I_D$  is demonstrated. Additionally, the etch rate for a wide process regime ( $V_A$  and  $I_A$ ) for Si(100), the surface roughness for defined under- and overneutralization and the properties of the etched sidewalls were investigated. The effect of the gas distributor ring material on the etch rate is also shown.

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## 1. Introduction

End-Hall ion sources [1] developed by Kaufman and Robinson in 1989 were typically used for ion beam treatment of various substrates such as ceramics, metals and polymers [2]. The field of application stretches from pre-cleaning over structuring processes to complex surface engineering [3–5]. In recent years, the performance of these end-Hall ion sources have been further improved by means of gridding, water cooling and ring shaped filaments [6]. Indeed, the operation principle of the end-Hall ion source is quite simple and has already been described in literature [6–13]. However, important technological aspects including several details of process control in case of insulating or piezoelectric substrates are not presented yet but are relevant to many applications for surface pre-treatment or ion beam sputtering especially in surface acoustic wave (SAW) technology. The present method (measuring of the substrate potential) is covered by a patent [9] but results were not verified for hot cathode ion sources experimentally. The neutralization effect was described for ion sources with cold cathode, e.g. anode-layer type [14,15] or gridded sources [16]. The electrons generated in an end-Hall ion source for electron impact ionization

are produced by thermionic emission at the hot filament cathode [7,17]. These thermal electrons form an electron current  $I_E$  split into three parts. The first part is accelerated towards the positive anode and forms the electron current  $I_G$  while the second part is conducted to the grounded chamber wall and forms the electron current  $I_W$  and the third part is forced by ions towards the substrate surface and feeds the electron current  $I_S$ .

Theoretically, if an excess of electrons near the filament is reached, the ion beam flux towards the substrate can partially or fully be neutralized [6]. In the case of Mark I<sup>+</sup> ion source (Veeco Instruments Inc.), an electron current value  $I_E$  of 5–20% of the total anode current  $I_A$  [10] is recommended to obtain such a fully neutralized beam. Nevertheless, for insulating substrates such as ceramics, glasses or polymers, charging can still occur over time due to an underneutralized ion beam [18]. Thereby, a change of the ion beam profile i.e. of the density and energy distribution of the ions within the ion beam is expected, associated with beam focusing and widening [6]. Especially for piezoelectric substrate materials, like quartz, LiNbO<sub>3</sub> or LiTaO<sub>3</sub> which are typically used in SAW technology, local differences in the substrate surface potential can result in arc discharge effects which can damage or destroy the SAW structures [19].

The focusing and widening of the ion flow towards the substrate is controlled by the electric field accompanying the flow. Effective neutralization of the ion beam suppresses the electric field [20]. In

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case of insufficient neutralization (underneutralization), the ion beam spreads and the ion energy as well as the ion density distribution become broader while the mean energy  $E_I$  of the ions decreases. This means, the ratio of the average ion energy  $E_I$  and the anode voltage ( $V_A$ ) can decrease below a value of  $E_I/eV_A \approx 0.6$  [6,18]. In fact, the mean ion energy and thereby the etch rate  $A_R$  are directly influenced by the state of neutralization. In case of an excess of thermal electrons at the cathode (overneutralization), the ion beam is focused to a smaller cross section area. The ion energy distribution becomes sharper, and the  $E_I/eV_A$  ratio is shifted to a higher value ( $E_I/eV_A \approx 0.94$ ). In both cases the ion energy as well as the ion density vary which lead to a change of the etch rate of the substrate material. It should also be noted that aging of the filament, e.g. by reducing its diameter due to thermal evaporation or ion sputtering of filament material, can strongly influence the efficiency of thermionic emission. Thus a sensitive control of the neutralizing electron current is needed to keep the etching parameters constant over a long process time.

The influence of different substrate materials on the neutralization effect and the resulting process parameters especially the etch rate has been studied in this work. Further, it's demonstrated how to balance between the ion and the electron current at the substrate surface for charge neutralization [8] and how it could be maintained over a long process time. Moreover, we have determined the etch rates and surface roughening for a wide range of significant process parameters such as anode voltage ( $V_A$ ) and anode current ( $I_A$ ), exemplarily for the Si(100) reference material in detail and additionally for aCuTi and LiNbO<sub>3</sub>.

## 2. Materials and methods

An end-Hall ion source (Mark I<sup>+</sup>, Veeco) was arranged in the centre of a vacuum chamber (500 mm diameter) and vertically aligned at a distance of 150 mm to the substrate surface. The chamber was evacuated by a turbo molecular pump (Turbo 301 Navigator 360 l/s, Varian) to a base pressure below  $4 \cdot 10^{-7}$  mbar.

The process gas (Ar or O<sub>2</sub>) is supplied via a perforated gas distributor plate fabricated of stainless steel (for Ar or O<sub>2</sub>) or pyrolytic carbon (for Ar). A ring metal plate used for shielding was arranged around the substrate opposite to the ion source and electrically connected with the precision resistor  $R_m$  as well as the metallic substrate carrier. The conductive shield surrounding the substrate carrier ensures a current flow in case of an insulating substrate as well.

Experimentally, the influence of the electrical conductivity of different substrate materials on the etch rate and on process parameters (anode voltage, anode current) was studied for: a) Boron-doped Si(100) wafers of a thickness of about 525  $\mu\text{m}$  (bulk resistivity value 0.01 ...0.02  $\Omega\text{cm}$ ), b) piezoelectric 128°YX LiNbO<sub>3</sub> wafers with 525  $\mu\text{m}$  thickness (bulk resistivity value  $> 2 \times 10^{10}$   $\Omega\text{cm}$ ), and c) conductive amorphous Copper–Titanium (aCuTi) thin film with 1  $\mu\text{m}$  thickness (measured resistivity  $< 2.1 \times 10^{-4}$   $\Omega\text{cm}$ ) sputtered on a thermally oxidized Si(100) wafer.

The etch rate was determined on amorphous material (aCuTi) and on single crystalline materials (Si(100), LiNbO<sub>3</sub>) to ensure a homogenous erosion over time. For this purpose, a cross of two photoresist lines was dispensed on each of the (10 × 10) mm<sup>2</sup> shaped samples using a 3-axis dispensing workstation (Ultimus V, Nordson Corp.) and baked at 100 °C for 600 s. After ion beam treatment and subsequent resist removal, the etched steps were analyzed (more than ten profile measurements each) with a mechanical profiler (Alpha-Step 500, Tencor). The measured step height in relation to the etch time gives the resulting etch  $A_R$ .

Cross sections were prepared by FIB (focused ion beam, 1540XB

CrossBeam, Zeiss). Surface roughness  $R_q$  (RMS, root mean square) of etched Si(100) was systematically determined by atomic force microscopy measurements in tapping mode (Nanos AFM, Bruker Corp. and Dimension 3100, Veeco Instruments Inc.) on a (2 × 2)  $\mu\text{m}^2$  scanning range. Image corrections and roughness calculations were made with Gwyddion v.2.36 analysis software tool. Raw data was only corrected by mean plane subtraction and first order line-correction to correct the vertical drift of the piezo tube over time, which is important to determine the roughness of very smooth samples (RMS < 5 nm).

The magnetic flux density was measured with a Gaussmeter (455 DSP, Lake Shore) to be 70 mT on the magnets surface and 36 mT in the anode region 10 mm from the gas distributor for stainless steel, carbon or without gas distributor ring. Comparable results were given by Oudini et al. [21] for a Mark-series Veeco ion source.

X-ray photoelectron spectroscopy (XPS) depth profiles were determined in a XPS system (PHI 5600-CI, Physical Electronics) with a 16-channel multi-channelplate. Sputtering cycles (without sample rotation) with 60 s duration at typical ion beam energies of 2.5 keV under an angle of incidence of 60° to the surface in a scan region of about 1 mm<sup>2</sup> were performed. The acquisition was done using a Mg K $\alpha$  (1253.6 eV) at 15 keV and 350 W with a pass energy of 29 eV. The samples were transported from the ion etching machine to the XPS tool without vacuum interruption in a special transfer chamber [22] to avoid surface contamination.

The operation principle of the Mark I<sup>+</sup> end-Hall ion source was first described in 1987 [17] and patented in 1989 [1] including a detailed theoretical consideration by Oudini et al. [21]. The ion end-Hall source was extended by a resistor  $R_m$  for measuring the difference current  $I_D$  to ground (Fig. 1).

## 3. Theory

The circuit diagram (Fig. 1) can be explained as follows: the alternating current of magnitude  $I_C$  through the filament cathode heats up the filament to a temperature at which thermionic emission ( $I_E$ ) occurs. These thermal electrons are accelerated towards the anode by the voltage  $V_A$ , forming the electron current  $I_C$ . As a result of the magnetic field of the permanent magnet arranged at the backside of the conically shaped anode or, more precisely of the gas distributor plate, the electrons of  $I_C$  follow the magnetic field lines towards the anode [23] (and simulated by Oudini et al. [21]). By this helical electron trajectory, the probability of inelastic collisions with gas atoms per unit volume is increased, leading to more efficient ionization in front of the anode. Both,  $I_C$  and the current  $I_B$  of secondary electrons, which are released during ionization form the anode current  $I_A$ . Typically, the electrical potential of the insulated gas distributor plate is at floating potential  $\phi_F$ . This leads to a voltage drop between this plate and the anode ( $V_A$ ) as well as the plasma potential ( $\phi_P$ ) [21]. The cathodes potential ( $\phi_C$ ) is near 0 V, because of the transformers center tap [24].

Most of the positive ions are accelerated out of the anode towards the cathode by a voltage ( $\phi_P - \phi_C$ ) and the residual ions are accelerated towards the gas distributor ring by a voltage ( $\phi_P - \phi_F$ ). The ions remove material from the filament and the gas distributor plate, if their kinetic energy is high enough for sputtering. In this case, contamination of the substrate surface with filament and ring material would be expected.

Measurement of the potential of the gas distributor plate (to ground) has shown that  $\phi_F$  is close to ground potential (<5 V). In order to describe the electrical behavior of the end-Hall ion source in detail, a charge carrier loss i.e. an ion current loss  $I_L^+$  and an electron current loss  $I_L^-$  can be introduced into the circuit diagram (Fig. 1). This includes the positive ion loss as well as electron loss at

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