Diagnostics for plasma processing (etching plasmas) (invited)

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Plasma processing diagnostics play two different roles—characterization and control. The goal of plasma characterization is to establish connections of data with external parameters and to verify models. The goal of control diagnostics is to make noninvasive *in situ* measurements of relevant processing parameters. Diagnostics used in semiconductor etching are considered. These include Langmuir probes, laser induced fluorescence, optical emission spectroscopy, infrared and Fourier transform infrared absorption spectroscopy, mass spectrometry, microwave interferometry, and radio frequency diagnostics. An example is given of the use of many diagnostics in characterizing SiO_2 and Si etching by fluorocarbons. © *1997 American Institute of Physics.* [S0034-6748(97)51001-X]

I. INTRODUCTION

Plasma processing employs partially ionized plasmas with cold electrons (≈ 3 eV) and covers the pressure range 10^{-4} Torr$<p<$ several atmospheres. With increasing pressure, the subject shifts from physics issues to chemistry issues to heat transport issues. With increasing pressure, neutral species become increasingly more important. Diagnostics must be provided for both charged particles and for neutral species. In many cases, plasma processing makes use of the interaction of plasma with material surfaces. This means that the processing depends on plasma properties at the substrate surface and only indirectly on the bulk plasma properties. Many of these interactions are difficult to measure *in situ*.

This article concentrates on one type of plasma-surface interaction, semiconductor processing. Processing consists of a combination of steps involving etching of material from the surface and deposition on the surface. At the present time, plasma etching usually takes place at several hundred mTorr using capacitively coupled etch tools, but within a few years semiconductor processing will be carried out in "high density-low pressure" ($\leqslant 10^{12}$ cm^{-3}) etch tools operated in the range 1/2 to 20 mTorr. Plasma etching can be thought of as physics assisted chemistry and consists of a combination of etching and deposition. This article looks to the near future and considers the constraints, needs, and present solutions for high density-low pressure plasma diagnostics. In this regime, physics issues have some importance.

In order to get an idea about what needs to be measured and how to carry out the measurements, the conditions in a typical plasma processing device should be considered. The plasma environment for etching plasmas consists of plasma densities in the range of 10^{10} to 10^{12} cm^{-3}, electron temperatures ≈ 3 eV, B fields <1 kG, ion beam energies <150 eV, and radio frequency (rf) usually at 13.56 MHz or 2.45 GHz. A large variety of free radical atomic and molecular species as well as both positive and negative ion species are also present. Etching competes with polymer deposition on surfaces and deposition on "dust" particles in the plasma. Radio frequency is employed rather than direct current (dc) because the polymer deposition leaves most surfaces as insulators.

The characteristic lengths are: Debye length $\lambda_D \approx 10^{-3}$ cm, ion neutral collision lengths $\lambda_{in} \approx 1-10$ cm, skin depths $\delta = c/\omega_{pe} \approx 0.5-1$ cm, presheath length $\approx \lambda_{in} \approx 1-10$ cm, minimum pattern size 0.2 μm. Collisionless sheath thicknesses equal $(eV_B/T_e)^{3/4}\ \lambda_D \approx 10^{-2}$ cm. Generally, λ_{in} are somewhat smaller than device dimensions. The critical etching issues in plasma etching are: radial uniformity in etch rate, anisotropy, and selectivity, where anisotropy refers to the ratio of etch rate perpendicular and parallel to the wafer surface and selectivity is the ratio of etch rates of various surface species (e.g., Si to SiO_2, SiO_2 to photoresist).

Industrial processing tool manufacturers have resisted the use of *in situ* diagnostics and most industrial processing tools are not equipped with diagnostics. In fact, industrial tools are *not designed* to be used with diagnostics. Etching chambers are designed to accommodate wafers 200 mm in diameter with 300 mm under discussion. Side, top, and bottom boundaries are devoted to plasma production (e.g., inductive coils), wafer handling equipment, wafer chucks which provide wafer cooling and biasing, gate valves, etc. Little space is normally provided in the transverse dimension which are often only the order of 100 mm or less. This means there is little space for diagnostics and very limited diagnostic access available.

Diagnostics are needed for two different roles in processing plasmas, characterization and control. The goal of plasma characterization is to verify models and to establish connections with external control parameters such as input power, flow rate, neutral pressure, gas species, etc. Somewhat surprisingly, the goal is not to make measurements during processing and often measurements are not made on processing tools but rather on model systems which are only somewhat related to the processing plasmas. The goal of control diagnostics and sensors is to make *in situ*, noninvasive measurements of the relevant charged and neutral species or of the process itself. Characterization is often satisfied with measurements of all available parameters while control only need concentrate on rate limiting steps. In principle, it is not necessary to measure all parameters and it may not be useful to do so.

Favored diagnostics for characterization include optical emission spectroscopy, infrared absorption and reflection spectroscopy, optical interferometers for monitoring real

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time etch rate, and selectivity. Boundary diagnostics include interferometers, ion energy analyzers, mass spectrometers, and probes.

Sensors for end point monitoring are well established and currently efforts are under way to establish sensors suitable for real time control of etch plasmas. Control can be run to run or real time with real time being preferable. In run to run, the condition of the finished product may be used to improve the process. Examination of test wafers after specified processing steps is now the single most important diagnostic technique in industrial plasma etching. This approach can be quite time consuming.

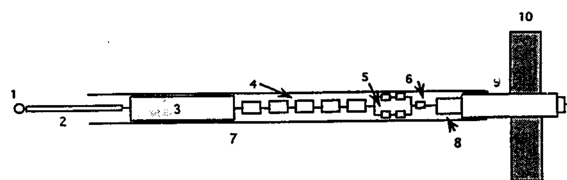
Real time *control* requires "sensors" of the plasma process that are *in situ* diagnostics. However, there is much concern in industry about the introduction of impurities during processing. For example, class 1 clean rooms (filtered to contain only one dust particle per cubic foot) are employed to reduce contamination. Therefore, noninvasive *in situ* diagnostics are needed for control. Diagnostics must also be suitable for the "shop floor," i.e., be simple to use and rugged.

What can be measured? There are three categories: (1) machine parameters (power, pressure, flow rate, etc.); (2) plasma parameters (potentials, densities, temperature, radical concentration, etc.); and (3) process parameters (e.g., in plasma etching: etch rate; anisotropy; selectivity; uniformity, etc.). Most machine parameters are easily obtained, but the information that they provide about the etching process is limited because the relationship between the machine parameters and the desired process parameters is usually not well understood. Gas phase measurements of local species concentrations and surface processing parameters provide more direct information about the etching process. Measurements are most useful if they are made *in situ* at or near the wafer surface. We will now consider specific diagnostic techniques and describe how they are used for characterization or control.

II. PROBES

Probes are typically used for characterization of high density-low pressure etching plasmas. They have the advantage of providing point measurements of plasma parameters. The presence of rf, polymer deposition, magnetic fields, secondary electron emission, negative ions, etc. complicates the interpretation of probe data. In the presence of rf, both filter (see Fig. 1)[1] and following techniques[2] have been employed to allow measurement of plasma potential, electron temperature, and plasma density. Measurements without filters have been useful. These probe $I-V$ traces can be either rapid ($\ll \omega^{-1}$) or time averaged. Time average $I-V$ characteristics are useful in keeping track of the range of potential fluctuations in the plasma and in identifying electrons trapped by nonlinear waves.[3] In high density–low pressure tools, the fluctuating fields are more important because the ion sheath transit time $\approx \omega_{\mathrm{pi}}^{-1}$, so ions at plasma boundaries can follow the rf when exiting the plasma.

Probe perturbations to the plasma can be a problem. When a probe is biased at the plasma potential ϕ_p, the electron current density at a probe is approximately $\sqrt{m_i/m_e}$ $\approx$ 300 times larger than the wall current density, which is retarded by the plasma wall sheath. This means the effective size of the probe is 300 times larger than its geometric area. Langmuir probes biased at ϕ_p can then represent a significant perturbation to the loss area and especially when operated in plasmas with relatively short electron collision mean free paths, can deplete the electrons in a local volume. This problem has led to the choice of cylindrical probes (with radius $a \ll \lambda_D$) in the relatively high pressure, relatively low density, capacitively coupled plasmas. Cylindrical probe theory[4] based on orbital motion is much harder to apply than planar probe theory[5] which applies for probes used in high density–low pressure plasmas in which small probes can be chosen which satisfy $a \gg \lambda_D$. Polymer deposition on probes (and windows and walls) is a serious problem for particle collecting probes. One solution is to use a fast injection probe[6] which reduces the time the probe spends in the plasma. The remainder of the time the probe must be stored in a probe "garage."



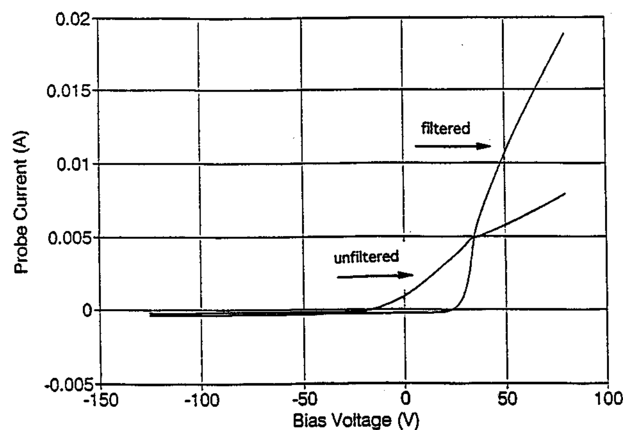


FIG. 1. Typical probe I/V characteristics for filtered and unfiltered probes, together with schematic of a filtered probe.

In processing tools with dc magnetic fields, electron saturation current is reduced resulting in electron to ion saturation current ratios I_e^*/I_i^* the order of 10–20. This behavior is similar to that of probes in the scrape off layer of tokamaks and can be understood using models developed by Stangeby.[7] In addition, instabilities are driven when the probe draws current in a B field. The perturbation by the probe reduces the plasma density and with it the plasma potential so the "knee" in the probe $I-V$ characteristic is not a good measure of the plasma potential in the absence of the probe. Plasma potential measurements are best carried out with emissive probes. Probes are operated in the limit of zero

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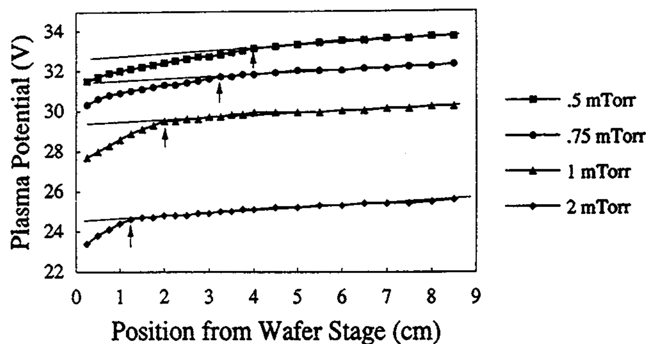

FIG. 2. The plasma potential vs position from the wafer stage in an 800 W, Nitrogen discharge, measured with an emissive probe.

emission.[8] The inflection point of the $I-V$ curve is monitored as the emission is decreased to zero. An example of this technique is the measurement of the presheath near the wafer in an (ECR) tool (see Fig. 2).[9]

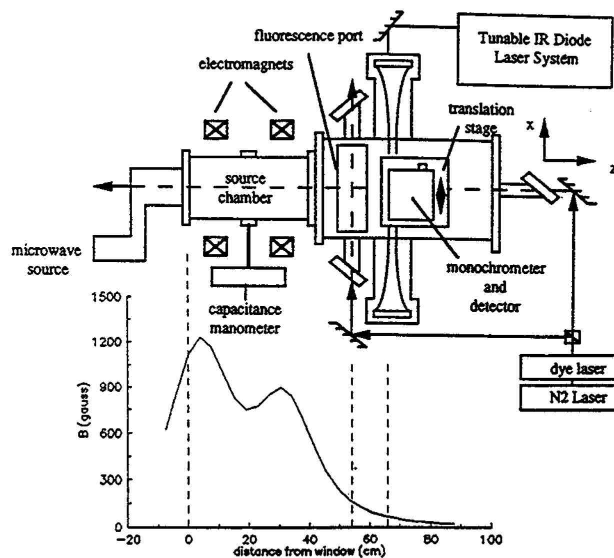

FIG. 3. Schematic of the experimental setup showing the locations of the LIF and IR ports with respect to the plasma source.

III. LASER INDUCED FLUORESCENCE

Etch anisotropy depends on the angle of incidence of ions at the wafer. This angle (measured with respect to the normal to the surface) depends on the ratio of the parallel ion velocity distribution function ($E_{\parallel}$) and the perpendicular ion temperature ($T_{\perp}$) at the presheath-sheath boundary. Laser induced fluorescence[10,11] provides a noninvasive technique to measure ion velocity distribution functions, and from them $E_{\parallel}$ and $T_{\perp}$.

Laser induced fluorescence (LIF) employs tunable lasers to resonantly excite atomic or molecular levels and the fluorescence light is detected. LIF normally employs pumped dye lasers. Fluorescence is detected by a monochromator, a filter+photomultiplier, or an optical multichannel analyzer. The technique is sensitive and can detect densities as low as 10^{8} cm^{-3}. It has good spatial resolution (determined by the intersection of the laser beam with the optics) and can give good temporal resolution. LIF has been used to measure free radical and ion relative densities and velocity distribution functions, gas temperatures, and electric fields when suitable levels exist. Absolute density measurements require separate calibration.

LIF was useful in the study of capacitive discharges[12] because of its sensitivity. However, the plasmas that were characterized were not processing plasmas. At the time of those measurements, the parallel plates were often moved (and sometimes the entire chamber) while the optics remained stationary. Clearly, the emphasis was to characterize the discharge. At the same time, the emphasis of most industrial efforts were to determine chemical recipes for achieving good wafer etching with capacitive discharges. An example of the use of LIF in a high density–low pressure plasma is given in Fig. 3. Den Hartog *et al.*[10] determined the ion temperature $T_{\perp}$ perpendicular to the magnetic field in an electron cyclotron resonance plasma from Doppler profiles of a N_2^+ transition. The beam diameter was 3 mm, the plasma density 1.8×10^{11} cm^{-3}. Measurements with LIF have shown that the ion drift parallel to the B field and far from a substrate are mobility limited and that ions do not take advantage of the ambipolar potential associated with the expanding magnetic fields in the ECR source (see Fig. 4).[13]

Although it is noninvasive, LIF requires significant adjustment and is neither simple to use nor rugged. Therefore, it is not suitable for use as a control diagnostic. LIF is limited to species with states that can be reached by optically allowed transitions from the ground or metastable state. In many cases, suitable levels are not available. When it can be used, it can provide valuable data on ion and neutral distribution functions. Unfortunately, strong electric fields are restricted to sheaths a fraction of a mm in thickness, much smaller the laser beam diameter and are not readily studied.

The pitch angle diagnostic (PAD)[14] can also be used to provide measurements of $T_{\perp}/E_{\parallel}$ in the presence of an axial magnetic field. PAD consists of a moveable ion collector which can be slid along the axis of a cylindrical tube parallel to the magnetic field. The variation of the collected current with ion collector axial position gives $T_{\perp}/E_{\parallel}$. Comparison of LIF and PAD results show that the two techniques are comparable. PAD can be mounted at the wafer position in ECR

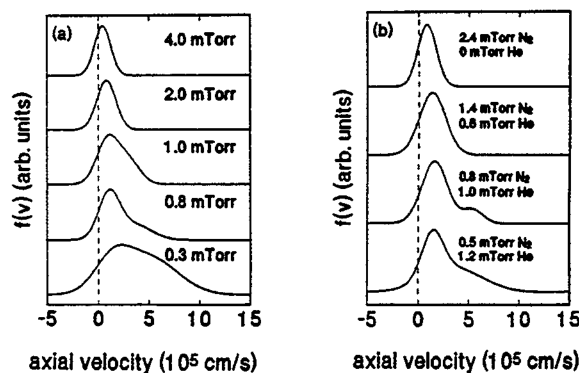

FIG. 4. Ion velocity distribution functions at (a) several pressures in pure nitrogen, (b) several nitrogen/helium mixture ratios.

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or helicon tool if there is a B field at the wafer. PAD could be used as a control diagnostic but because PAD requires a B field, it will not work in an unmagnetized inductively coupled plasma (ICP).

IV. OPTICAL EMISSION SPECTROSCOPY

Optical emission spectroscopy (OES) has been used over a number of years to provide measurements for operations such as end point detection and the presence of impurity gases. It is also being tested for various real time and run to run control schemes one of which is run to run control of radial plasma/etch uniformity.

OES can be used for end point detection. In etching plasmas, as one layer is etched away and an underlying layer becomes exposed to the plasma, the neutral gas in the plasma changes. The underlying layer has an entirely different product neutral or simply etches more slowly. This causes the product neutral concentrations to change and their respective emission line intensities to change. Time dependent OES can then be used as an indicator that the layer is etched and the process can be stopped to avoid substantial overetching.[12]

OES can be used to indicate the presence of impurities in the plasma. An OES ''signature'' of a tool that is working properly is often taken and when the tool is not working correctly another spectrum is taken. The difference between the two spectra can give an indication of what is wrong, e.g., if there is an unsuspected air leak in the system, a particular mass flow controller is putting in too much or too little additive gas, the system is ''dirty'' and impurity emission such as oxygen and hydrogen from water etc. is present. Commercial optical channel analyzers are readily available and present day spectral software makes this ''analysis'' painless. However, viewing windows get coated or etched, and so the light gathering power of the instrument can change. Also, T_e can be modified by impurities, so that the whole spectra can change. However, OES has been found to vary with impurities or tool seasoning.[15]

OES can be used to obtain a measure of plasma uniformity and thereby etch uniformity by using multiple path length measurements. Optical emission typically measures emission along a line of sight. It does not give a point measurement such as a Langmuir probe. However, since wafers are discs and the plasma plus the rate limiting chemistry needs to be uniform over the wafer surface to obtain uniform processing, several line of sight measurements across the diameter of the wafer can be used to give a measurement of the etch radial uniformity. Anderson *et al.*[16] has shown correlation between multiple OES signals acquired by viewing various positions across the top of a wafer and the radial uniformity of the etching process. His group proposes to use these measurements in control systems so that tools can dynamically change plasma parameters and thereby control (eliminate) radial variations of the process.

Direct quantitative measurements of neutral, ion, and radical concentrations from line emission require accurate knowledge of the electron energy distribution function and electron density along the observation path. Since only the tail of the electron distribution function in these low electron temperature plasmas is involved with emission, localized variations of the electron distribution can grossly affect the measured line integrated, line emission but will hardly affect the base plasma parameters. An OES technique that provides a quantitative measure of atomic ion species concentrations in a plasma is called actinometry. This method employs adding an actinometer gas, usually a noble gas like argon to the plasma at a known concentration (measured by a capacitive manometer or an RGA). The actinometer gas is picked to have a particular emission line whose emission varies with electron velocity in a manner-similar to the chemically active atomic ion line emission whose concentration is to be measured. Then, the concentration of that atomic ion is equal to the ratio of the atomic line emission to the actinometer gas line emission multiplied by the argon density. In low pressure discharges, an RGA needs to be used to dynamically measure the argon concentration because the plasma has been found to affect the noble gas density.[17]

V. ABSORPTION SPECTROSCOPY

Absorption spectroscopy is a useful technique that enables one to measure the absolute concentrations of selected molecular radicals and neutrals and atomic metastable states in a plasma chamber more quantitatively than OES. For this measurement, there is no need to accurately know the electron distribution function along the line of sight. Molecules and some atoms have useful absorption lines in the visible, infrared, or microwave frequency ranges. Absorption spectroscopy requires a sufficiently powerful and constant source that can be directed through the sample and a sensitive detector along with a technique to accurately determine the frequency of the absorbed radiation.

CF_4 and CHF_3 have particularly strong absorption bands in the infrared region. Therefore, single pass absorption using an FTIR spectrum analyzer can be used to measure the break up of these molecules in etching tools. The FTIR sends two beams of infrared radiation through the plasma and then measures the signal. The beams which originally come from the same intense broadband source, are split by a beam splitter and travel a known range of path length differences (determined by a moving mirror), are then reunited and sent out through the sample and detected. The multiple measurements at different path lengths are converted to wave number by fast Fourier transformation techniques. Measurements[18] in an ECR tool show that the absorption spectra of CF_4 is reduced by a factor of 4 after the plasma is turned on so the plasma has significantly broken up the parent gas. Molecular concentrations and absorption coefficients for the daughter molecules, CF, CF_2, and CF_3 are such that multipass IR absorption is required (see Fig. 3).[12] A cryogenically cooled diode laser with a small frequency width ($0.001\ cm^{-1}$) is used as the source. Amplitude modulation and lock-in amplifier techniques are used on the detected signal to capture the small amount of absorption as the diode laser frequency is swept over the absorption lines. A Heriott cell is used to allow multiple passes of the beam through the plasma. It was found that the concentration of CF_2 is of the order of $10^{11}\ cm^{-3}$ over a broad range of input plasma powers and neutral pressures.

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More recently, microwave absorption techniques have been used to measure molecular concentrations (e.g., SiO) in a low pressure ECR deposition plasma. The technique is very similar to IR and FTIR absorption except a narrow band microwave source is used along with microwave detectors and quasioptical collection optics.[19] Visible light techniques have been used to measure concentrations of metastable argon atoms in a low pressure inductive plasma[20] and silicon deposition system.[21]

A. Ion mass and energy analyzers

Measurements of ion and radical fluxes to a simulated wafer surface can be made by putting a small aperture in the surface and sampling the fluxes using ion and neutral mass and energy analysis techniques in a differentially pumped space on the other side of the aperture. The low pressure environment is needed to increase the mean free path of the sampled species and helps to prevent any changes in their charge state and chemical composition. Gridded energy analyzers,[22] curved electrostatic plates, and quadrupole mass analyzers, singly or in combination have been used to measure the acceleration of ions through a variable frequency rf generated sheath,[23] the angular direction of the ions,[24] and the mass distribution of the impinging ions[25] and neutrals.[26]

B. Microwave interferometers

Microwave interferometers have received only limited use even in high density plasma tools. The need for reasonable access (5–8 cm diam windows even for 60 GHz systems) or internal launching devices, e.g., horns, frequency sources stable over hours, and minimal interference from multiply reflected beams in plasmas only 5–10 cm across has relegated these devices to only a few development laboratories.[27] Conceptually, microwave interferometry can provide an unambiguous measure of n_e but in practice it is not used where it could possibly do the most good—in production systems.

C. Radio frequency diagnostics

Plasma characteristics, electron density, and collisionality, affect the plasma impedance as seen by the rf generator through the matching circuit for both capacitive and inductive rf systems. Commercial units are now available to unambiguously detect the changes in plasma impedance.[28] These measurements are somewhat simple to add to a tool and do not require additional access to the plasma chamber. This technique is being more broadly used as a sensor of the tools performance and has been used in initial studies of real time control experiments.

D. Final example

As an example of the use of diagnostics, we consider experiments at Wisconsin on ECR etching of SiO_2 and Si. Experiments with CF_4 found that the SiO_2 and Si etch rate could be described by a simple expression which depended on only the ion energy flux to the wafer and the fluorine concentration.[29] The ion energy flux was determined by taking the product of the ion saturation current to a probe mounted close to the wafer and the ion energy which was determined from the plasma potential measured by Langmuir and emissive probes and the wafer bias voltage. Extensive probe measurements were made to characterize the plasma. Earlier experiments with LIF had established that almost all the ion energy was acquired in the sheath and presheath.[10] The fluorine concentration was measured by argon actinometry calibrated with an RGA.[16] Concentrations of neutral CF_x radicals, where $x=1$, 2, and 3, measured with multipass infrared absorption and FTIR proved not to be important contributors to the etch rate. Experiments with CHF_3 Si etching indicated that film deposition was an important additional process. By measuring the mass composition of the ionic flux with the QMS system, absolutely calibrating the flux, and adding it to the deduced neutral flux from the measured radical concentrations, it was found that *ions* were serving as polymer precursors unlike lower density-higher pressure discharges where neutral flux serves as the polymer precursors.[30] During the course of these experiments, real time *in situ* electron density measurements were made with a microwave interferometer. In addition, the composition of the deposited films were investigated with an FTIR.

ACKNOWLEDGMENT

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