In situ measurement of the electric conductivity of plasma deposited films using Langmuir probes

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Single probes can be used to study in situ the electrical properties of thin films deposited in plasma processes either on the probe itself or on the counter electrode or an equivalent substrate. This is demonstrated and discussed for rf discharges in C_2H_2 and HMDSO/O_2. Kinetics of deposition processes and the role of ions are investigated using the kinetic probe proposed by G.K. Vinogradov [1].

Probe characteristics discussed in this contribution are recorded in a capacitively coupled rf discharge at 13.56MHz. The vacuum vessel is a cylindrical glass tube of 200mm length and 90mm inner diameter, the Al-flanges at the ends are the grounded and the driven electrode, respectively. Measurements were made in C_2H_2 and HMDSO/O_2 plasmas at 0.1 - 0.2mbar pressure and 10 - 20W power. The heatable probe, inserted in the midplane between the electrodes, is made from 0.12mm Ir or 0.1mm Pt90/Ir10 wire. For SiO_2 containing coatings probe temperatures of more than 1300° C are necessary to prevent probe contamination.

In C_2H_2 plasmas we found changes of the characteristics due to the non-linear voltage drop across low conductivity layers deposited on the surface of the cold probe similar to those described earlier [1,2]. The characteristics are flattened and show an additional curving at potentials negative with respect to the floating potential U_{fl}.

However, our measurements reported in Refs. [1,2] were performed in large metalllic reactors, where the large wall acted as pickup electrode for the return current in the probe circuit. In the present experiment the grounded electrode is much smaller. Thus the resistivity of coatings of this electrode influences the probe characteristic, too. This can be recognized by a shift of the floating potential of the heated probe and a decrease of the electron current to the probe with deposition time, while the ion current remains nearly constant - effects not observed in discharges in metallic vessels [1,2] serving as a large grounded electrode in contact to the plasma.

To separate the effects of probe coating and electrode coating we have built a heatable grounded electrode in addition to the heatable probe. In C_2H_2 plasmas deposited films could thus be removed from the electrode. In Fig. 1, characteristics of the coated (cold) probe against the clean (hot) electrode and that of the clean (hot) probe against the clean electrode as well as against the coated (cold) electrode are compared.

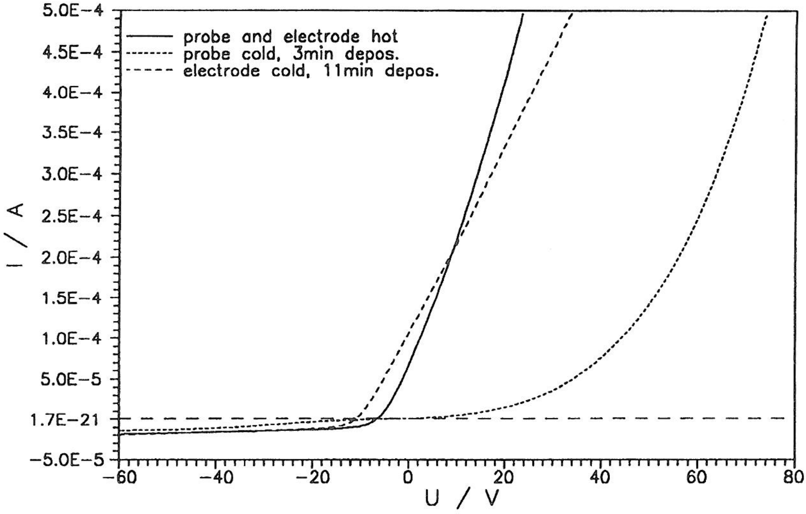

Fig. 1: Langmuir probe characteristics measured in an C_2H_2 plasma at 0.10mbar, 10Watt. Full line: both probe and electrode are heated, no deposition; dashed lines: deposition on either the cold electrode or the cold probe.

The floating potential U_{fl} of the hot probe (with respect to ground) is shifted to more negative values after switching off the electrode heating, and to more positive values when switching it on again (Fig. 2). Probably, this shift corresponds to the shift of the plasma potential due to enhanced secondary electron emission from the coated electrode. This shift of U_{fl} is more serious in dc coupled rf discharges [3]. Besides of this, the rf compensation seems to be less effective for probe measurements with coated electrode. This would also result in a shift of U_{fl}.

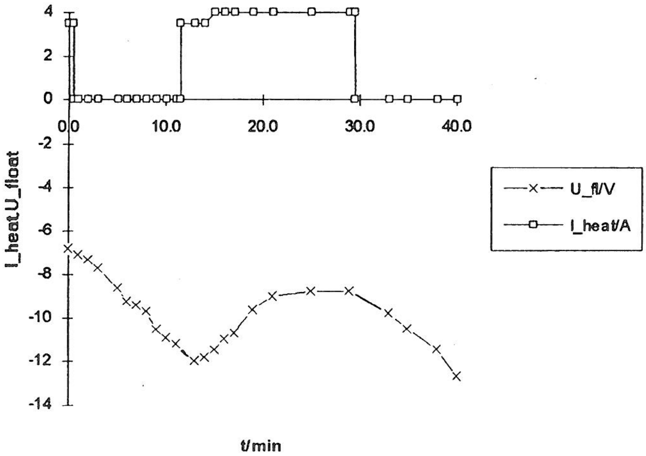

Fig. 2: Floating potential U_{float} of the hot probe in an C_2H_2 plasma at 0.1mbar, 10Watt, in relation to the heating current I_{heat} of the electrode.

Preventing coating of the electrode the electric current voltage characteristic of the probe coating can be derived by comparing the characteristics of the hot and the cold probe as described in Ref. [2]. It turns out that the current voltage characteristics of these films are highly nonlinear.

Coating of the counter electrode changes the characteristics of a clean probe also in HMDSO/O_2 plasmas used for the deposition of SiO_2 layers, but more slowly than in C_2H_2 plasmas. After running the discharge for several days, the characteristics of the single probe change to curves looking like double probe characteristics (Fig. 3). The conductivity of the layer is too small for sustaining the electron current. At probe voltages higher than 33V breakdowns through the layer can be observed, sparks are flickering in front, and the electron current steeply increases. By cleaning the electrode mechanically, the probe characteristic gets back its "normal" shape. (Removing these SiO_2 containing layers by heating the electrode was not yet tried.)

Nevertheless, coatings on the probe surface can be removed almost completely by direct heating of the probe, see Fig. 4. The first characteristic (full line) is recorded at a heating current of 1.9A, which corresponds to temperatures of more than 1300° C (pyrometer reading through the glass wall), after that the probe is cooled down and becomes coated, and then the heating current is increased again up to 1.9A. Both characteristics measured at 1.9A are close to each other. We are not sure, wether the small differences are due to a thin film remaining on the probe surface or to a slight coating of the electrode (see above). For heating currents of 2.4A or more, the probe emits electrons.

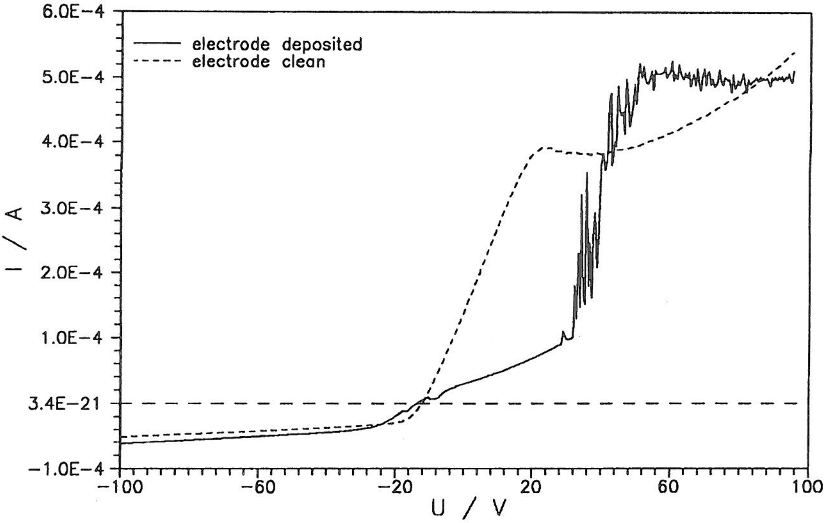

Fig. 3: Probe characteristics recorded in Ar discharges with the grounded electrode deposited in HMDSO/O_2 plasmas during several days running and after mechanical cleaning of the electrode.

We found that only Ir is suited for directly heated probes for use in O_2 containing plasmas. Probes made from other materials we tested (Pt, Pt90/Ir10) are blown out within a few seconds when heated to red glow.

If it is possible to remove probe layers totally by direct heating, one can use hot Langmuir probes with clean surfaces to measure the electric conductivity of a layer deposited on the substrate. To do this, the substrate must be used as the counter electrode of the probe circuit. This might be a new way to control plasma deposition processes in situ.

For studying the kinetics of the deposition process, i.e. especially the influence of the voltage drop across the space charge sheath, the kinetic probe or constant current probe as proposed and successfully demonstrated by G.K. Vinogradov [4] is advantageous. Here, the probe current is kept constant during deposition, and thus the voltage between the plasma and the surface of the coated probe remains constant. Therefore, the change of probe voltage during deposition of the probe gives directly the voltage drop at the growing film. This method allows the study of film growth in situ under stable sheath conditions.

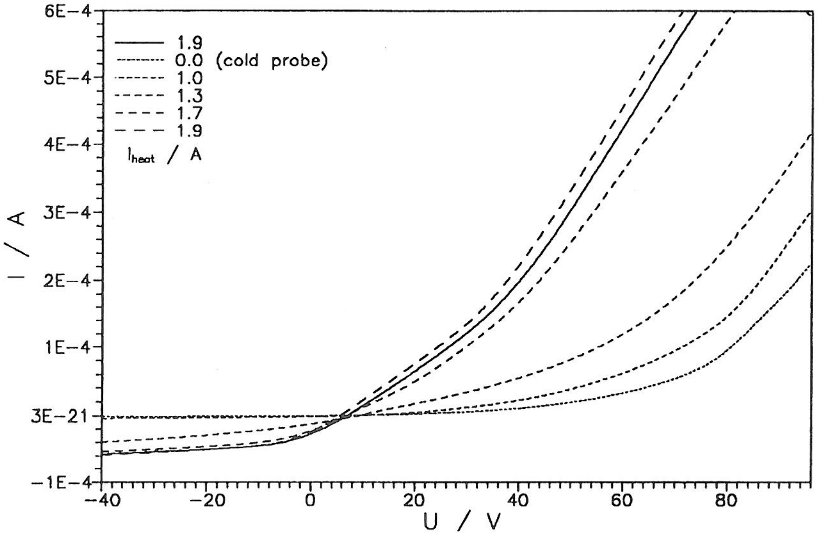

Fig. 4: Probe characteristics measured at different probe heating currents in an rf plasma at 15W, 0.01mbar HMDSO, 0.08mbar O_2 .

We have built for this purpose a constant current source stabilizing currents of 0.01 – 1 mA at voltages up to ±350 V. Measurements of *U(t)* for different positive and negative probe currents are depicted in Fig. 5. For the C_2H_2 plasma we found the voltage to increase slowly for about 30s after switching off the heating current, i.e. during this time the temperature decreases and a complete coverage of the probe is constituted. After that, the voltage increases almost linearly with rates not very much dependent on the probe current and thus the corresponding voltage drop at the space charge sheath in front of the probe. This might be due to the fact, that the layer is formed mainly by neutrals. Positive ions seem to play no important role in this deposition process.

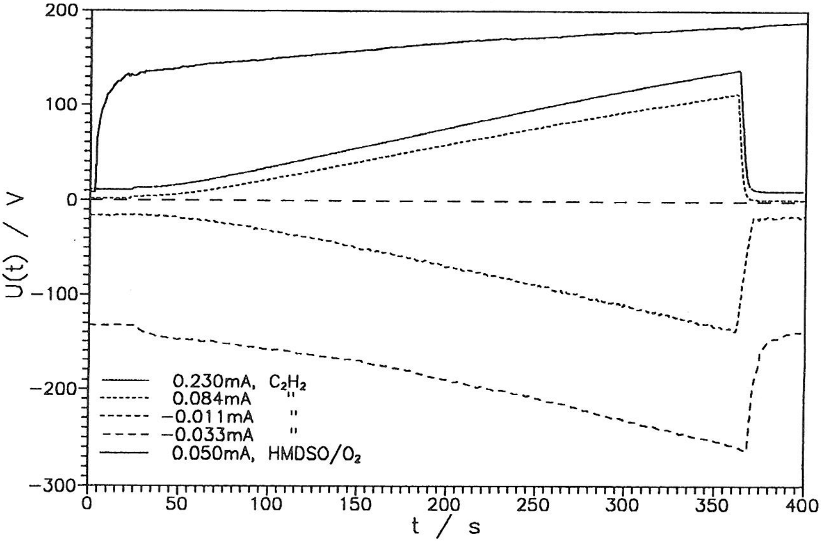

Fig. 5: Measurements of the total voltage drop *U(t)* between probe and ground electrode at different constant probe currents with the kinetic probe. In C_2H_2 the heating of the probe was switched off after 30s and switched on at about 370s. In HMDSO/O_2 the probe heating was switched off after a few seconds.

When depositing SiO_2 containing layers of high resistivity the voltage increases very fast immediatly after cooling down the probe surface. After that it varies only slowly. Further investigations at different process and plasma parameters must be performed in order to study in situ the kinetics of plasma deposition. To find a correlation between the voltage drop at and the thickness and quality of the deposited layer more informations on conduction mechanisms of very thin films are necessary.

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