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Spatially resolved high resolution interferometry

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Abstract. The construction of a cheap but very stable and accurate Michelson interferometer is described. To avoid (expensive) lock-in techniques a DC feedback loop has been implemented. A major advantage of this system is that the signal is proportional to the optical path length change itself, and not to the sine thereof. The frequency stabilization of the He–Ne laser used—necessary to attain the desired high resolution—is outlined. The Abel inversion procedure, used in a cylindrically symmetrical situation to convert lateral measurements into radial profiles, is discussed. A length-equivalent accuracy of about 0.1 nm has been accomplished. The system has been used to measure the radial profile of the refractive index of RF plasmas in CF₄ and Ar.

1. Introduction

The refractive index of a plasma is very sensitive to a number of phenomena. Free electrons are known to decrease its value, whereas atoms increase it due to their (positive) polarizability. Furthermore, atoms can show dispersion of radiation in the vicinity of a spectral line. If the plasma contains molecular species, the situation becomes even more difficult, since the contribution of different species has to be added. Interferometry is often used as a diagnostic method to measure the refractive index (Timmermans *et al* 1985). Usually the aim is to determine the electron density. However, in the case of RF plasmas in CF₄ used for plasma etching, on which, as an example, we will focus in this paper, it is not clear *a priori* what the dominant phenomenon will be with respect to the refractive index n . Based on the experiment, further analysis is required. An experimental difficulty is that usually the changes in optical path length induced by these plasmas are very small: of the order of a few nm.

In this paper we describe a simple interferometer system, based on a Michelson interferometer, that offers the possibility of detecting these small changes in path length. The system consists of a DC feedback loop: the interferometer signal is used to compensate the path-length changes. This, as well as the rigid construction, ensures high accuracy.

We will commence by describing the experimental method and the instrument in detail. Then the Abel inversion procedure is introduced, followed by a discussion of the various contributions to the refractive

index of a plasma. Finally some results are given from measurements in RF plasmas in CF₄ and Ar.

2. Experimental details

The reactor for plasma etching which is used for these experiments is described elsewhere in detail (Bisschops 1986). Basically it consists of a set of two parallel, cylindrical electrodes surrounded by a vacuum system. One of the electrodes is grounded, the other one is powered by a RF generator (13.56 MHz). The pumping system comprises a 250 m³ h⁻¹ Roots blower and a two-stage roughing pump. The gases (Ar and CF₄) are fed to the reactor through mass flow controllers. The pressure is measured by an MKS Baratron pressure gauge.

To allow for the measurement of very small changes of the optical path, the Michelson interferometer has to be of a very rigid construction. Also the mechanical connection to the plasma must be very stable. Furthermore the coherence length of the He–Ne laser used should be larger than the size of the interferometer arms by a factor of about 1000; this in principle allows for the detection of a path change of one millifringe.

To meet the first demand, all the components of the interferometer have been attached to a rigid metal plate, that can be mounted on top of the reactor. Mirror 2 is placed on a heavy C-shaped piece of aluminium, which encircles the plasma and is screwed onto the interferometer plate (see figure 1). The whole system is thermally isolated from the surroundings to achieve good temperature stability.

To meet the demand of a large coherence length, a frequency-stabilized He–Ne laser was constructed. The

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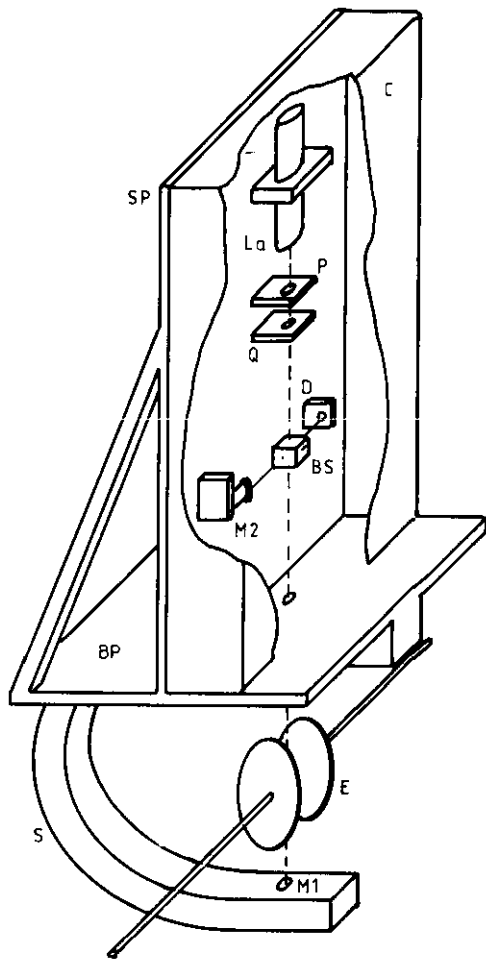


Figure 1. Outline of the experimental set-up, viewed through a cut in the cover (C). The complete interferometer is mounted on a supporting plate (SP), which is itself fixed perpendicularly to the bottom plate (BP). The bottom plate rests (vacuum sealed) on the top of the vacuum vessel (not drawn in the figure) containing the electrodes (E). In order to avoid feedback, the beam of the stabilized He-Ne laser (La) passes through a polarizer (P) and a quarter-wave plate (Q) before entering the actual interferometer at beam splitter BS. Reference mirror M2 is mounted on a piezo-element. The beam reflected there interferes on detector D with the plasma passing beam reflected by mirror M1. Mirror M1 is mounted on a rigid C-shaped support hanging around the plasma.

two modes of an unpolarized Hughes 3121H He-Ne laser of 22 cm tube length (see figure 2) drift through the Doppler profile either due to temperature changes of the laser tube or due to mechanical vibrations. Since the modes are linearly polarized in orthogonal directions they can be separated with a beam splitter and two polarizers. The cavity length can be influenced by a piezo-element which has been mounted on the laser tube. The principle of stabilization is shown in figure 3. The changes in cavity length cause changes in the intensity ratio of the two modes. These changes are fed to an integrator together with a reference signal; after high voltage amplification, the signal is fed back to the laser cavity by means of the piezo-element. Using this stabilization method, frequency stabilities of 1 MHz

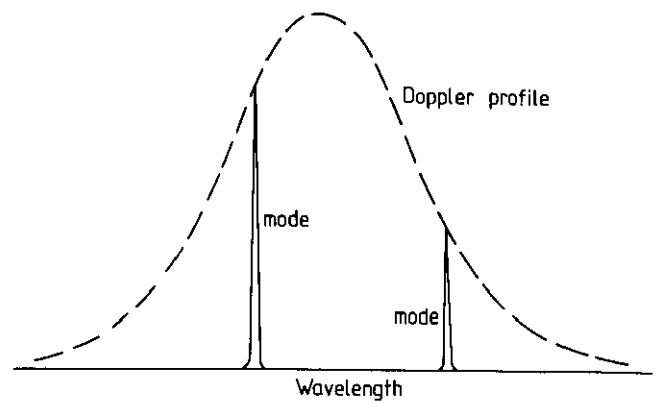


Figure 2. The modes of a He-Ne laser in the Doppler profile. The Doppler profile is the total broadened line profile, the modes are selected by the resonance condition for the laser cavity. They are linearly polarized in perpendicular directions.

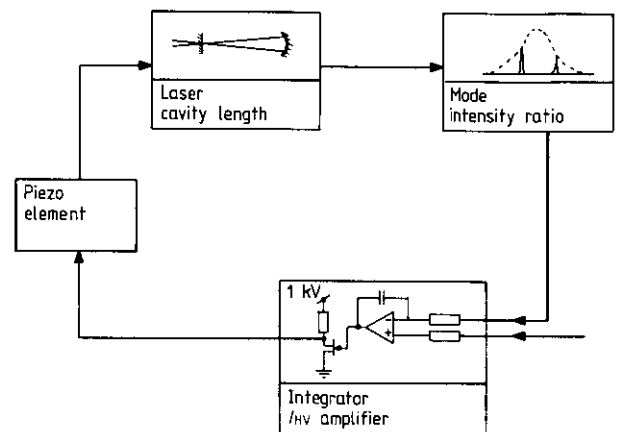


Figure 3. Stabilization principle for the He-Ne laser. The changes in the cavity length cause a change in the mode intensity ratio, which is fed back to the cavity length using an integrator and a piezo-element.

long-term (hours) and 10 kHz short-term (minutes) have been achieved. This implies coherence lengths of 300 m and 30 km respectively. Since the time needed to perform one measurement is of the order of minutes, the coherence length is more than sufficient for our application.

The interferometer signal is (see equation (1) below) proportional to the cosine of the path difference. This gives rise to certain inaccuracies and ambiguities, especially near the maxima and minima of the fringes. Therefore the interferometer signal is kept constant with a feedback loop (figure 4): the interferometer signal is fed to an integrator together with a reference signal. The integrated signal is amplified by a high voltage amplifier. The high voltage controls a piezo-element on which the reference mirror of the Michelson interferometer is mounted. In this way the optical path difference is fed back. An external movement of any of the mirrors or another change of the optical path difference will now be compensated by an equally large displacement of the reference mirror. Since the length change of a piezo-

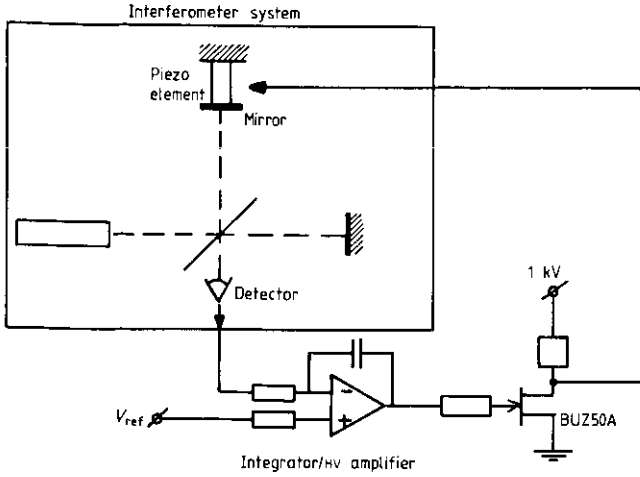


Figure 4. Feedback system for the interferometer. The interferometer signal is fed back to the optical path length difference by means of an integrator and a piezo-element.

element is proportional to the voltage applied to it, the change in optical path difference, i.e. the quantity which we want to measure, is now proportional to the change of the output voltage of the high voltage amplifier.

This voltage is sampled with a 12-bit ADC incorporated in an interface system controlled by an M68000 microcomputer (Voskamp *et al* 1989). The complete measurement is computer-controlled: the plasma is switched on and off automatically and the effects of the plasma are extracted from those of other phenomena such as slow variations in the room temperature. In figure 5 a typical time profile of the piezo control signal is given, and the procedure to separate the plasma effect from other effects is illustrated.

The relative accuracy of the complete system is determined by the hysteresis of the piezo-element and

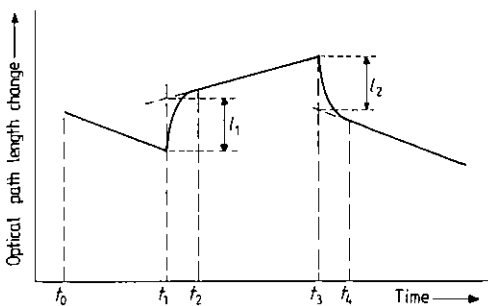


Figure 5. Typical example of a time-dependent measurement of the interferometer signal. From $t = t_0$ to $t = t_1$ no plasma is present. The path-length difference changes gradually due to minute temperature changes of the environment. At $t = t_1$ the plasma is switched on. From $t = t_2$ the situation is stable: the integration time of the integrator has passed. Now a different heating pattern results: the C-shaped mirror support is heated by the plasma. At $t = t_3$ the plasma is switched off. After $t = t_4$ the situation is stable again. The heating pattern is the same as before $t = t_1$. The lengths l_1 and l_2 are equal, and they represent the path change due to the presence of the plasma.

amounts to about 5%. The absolute accuracy, determined mainly by the mechanical stability of the interferometer and by the coherence length of the laser, is about 0.001 fringe (~ 0.3 nm).

3. Method

The total intensity $I(l)$ measured by the detector depends on the difference l between the optical path lengths of the two interferometer arms:

$$I(l) = I_0 [1 + \cos(2\pi l/\lambda)]. \quad (1)$$

The optical path length difference l depends on the refractive index of the medium in the measurement arm integrated along the laser beam. Using the refractive index $n(x, y)$ of the plasma as a function of the spatial coordinates x and y (see figure 6), the path length l is a function of the lateral coordinate y :

$$l(y) = \int n(x, y) dx. \quad (2)$$

If the plasma is assumed to be cylindrically symmetric, the refractive index $n(x, y)$ can only be dependent on the radial coordinate r :

$$n(x, y) = n(r). \quad (3)$$

The plasma radius R is defined as the radius beyond which the refractive index becomes constant. If the plasma is switched off, the refractive index $n(r)$ changes by $\Delta n(r)$, and the optical path difference $l(y)$ by $\Delta l(y)$. It can be shown that

$$\Delta l(y) = \int \Delta n(x, y) dx = 2 \int_y^R \frac{\Delta n(r)r dr}{\sqrt{(r^2 - y^2)}}. \quad (4)$$

As $\Delta n(r) = 0$ for $r \geq R$, Abel inversion may be applied (Lochte Holtgreven 1968):

$$\Delta n(r) = -\frac{1}{\pi} \int_r^R \frac{\partial \Delta l(y)/\partial y}{\sqrt{(y^2 - r^2)}} dy \quad (5)$$

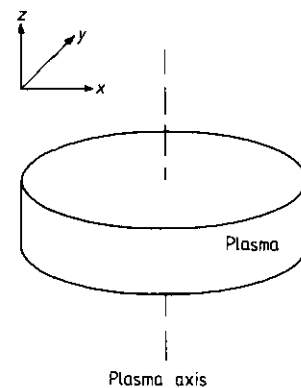


Figure 6. Illustration of the coordinate system. The cylinder represents the circumference of the plasma, the x and y directions are parallel and perpendicular to the line of sight (the laser beam) respectively.

from which the change in the refractive index can be evaluated.

4. The refractive index of a plasma

The refractive index n of a plasma is determined by the densities and polarizabilities of the particles present in the plasma. The contribution $(n - 1)_{el}$ to the refractive index n of the free electrons is given by

$$(n - 1)_{el} = -\frac{e}{8\epsilon_0 m_e \pi^2} \lambda^2 N_e \quad (6)$$

where λ is the wavelength of the light, N_e is the electron density, ϵ_0 the dielectric constant and m_e the electron mass. With an electron density of about 10^{16} m^{-3} (Bisschops 1986), this results in a value of $(n - 1)_{el}$ of 1.6×10^{-12} .

The contribution $(n - 1)_h$ of the heavy particles (neutral molecules and radicals, negative and positive ions) can be represented by

$$(n - 1)_h = 2\pi \sum_i N_i \frac{\alpha_i}{4\pi\epsilon_0}, \quad (7)$$

where the index i runs over all vibrational and electronic states of all heavy particles present in the plasma, N_i is the density of species i , and α_i is the polarizability of species i . The polarizability of CF_4 in the ground state, which is by far the most dense species, is given by

$$\left(\frac{\alpha}{4\pi\epsilon_0}\right)_{\text{CF}_4} = 3 \times 10^{-30} \text{ m}^3. \quad (8)$$

With a pressure of 100 Pa (1 mbar) and a temperature of 300 K this results in $(n - 1)_{\text{CF}_4} \approx 1 \times 10^{-7}$.

It is evident that the contribution of the free electrons can safely be neglected. The refractive index n_{pl} of the plasma will be given by

$$(n - 1)_{pl} = \frac{1}{2\epsilon_0} \sum_i N_i \alpha_i. \quad (9)$$

5. Measurements

To test the interferometer system, it was used to measure the polarizabilities of Ar and CF_4 at room temperatures. Using (4) and (9), and the equation of state $p = nkT$, the polarizability is extracted from a measurement without plasma of the change in optical path length l as a function of gas pressure (see figure 7 for an example). The polarizabilities that result are $(\alpha/4\pi\epsilon_0)_{\text{Ar}} = (1.7 \pm 0.1) \times 10^{-30} \text{ m}^3$ and $(\alpha/4\pi\epsilon_0)_{\text{CF}_4} = (3.1 \pm 0.3) \times 10^{-30} \text{ m}^3$. This is in very good agreement with the literature values in Landolt-Börnstein (1962).

In a plasma the lateral profile of the optical path change $l(y)$ as a function of the lateral position y has been determined for several pressures and power densities. A typical example is shown in figure 8. The measured profile is fitted to the exponential of an even-powered polynomial using a least-squares method, and then Abel

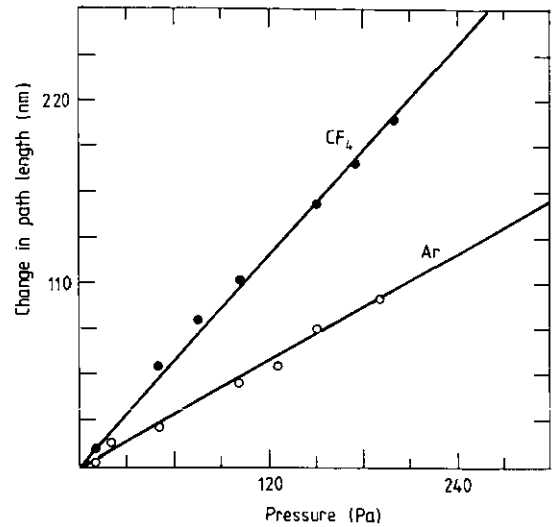


Figure 7. The optical path length change as measured by the interferometer as a function of gas pressure. The polarizability can be determined from the slope.

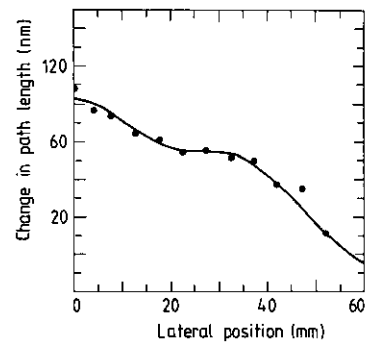


Figure 8. Lateral profile of the optical path change caused by the presence of the plasma.

inversion is performed. The radial profile of the refractive index change results (cf equation (5)). In figure 9 examples of typical radial profiles of CF_4 plasmas and Ar plasmas are compared.

All measurements yield an increase of the refractive index in the entire plasma. Since the existence of a pressure gradient is very unlikely, due to the low values of the gas flow (50 sccm), and since furthermore an increase of the gas temperature would lead to a decrease of the refractive index, the increase of the refractive index has to be attributed to an increase of the average polarizability of the gas as soon as the plasma is ignited. In the case of CF_4 there are two candidates: dissociation of the molecules into radicals, and vibrational excitation. An estimation of the polarizability of CF_x radicals using a quantum mechanical approximation proposed by Hirschfelder *et al* (1967) leads to an increase in the averaged molecular polarizability by a factor of 4 if a CF_4 molecule is dissociated. With an expected dissociation degree of the order of 1% (Bisschops 1986) this yields an increase of the refractive index by a factor of 1.04. This is not even by approximation enough to explain the measured increase of a factor of 2.

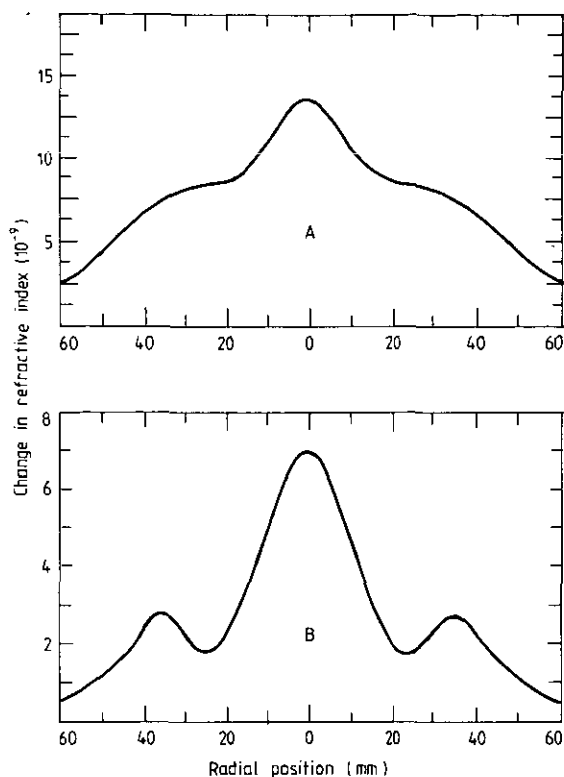


Figure 9. Radial profile of the refractive index of an RF plasma in CF_4 (A) and Ar (B). They are constructed from lateral profiles as in figure 8 using the Abel inversion.

It is therefore probable that vibrational excitation of the CF_4 gas is the cause of the measured large increase. Generally the polarizability of a vibrationally excited molecule is larger than that of a molecule in the ground state: a factor of around 3 is not uncommon for hydrogen compounds (Kolos and Wolniewicz 1976; Paudey and Sautry 1979). It might very well be that a large fraction of the CF_4 molecules is vibrationally excited. Indications for this are also found using IR absorption spectroscopy (Haverlag *et al* 1989). As the polarizability of vibrationally excited CF_4 is not known exactly, an accurate estimation of the excitation degree cannot be given, but several percent to tens of percent seems a likely range.

The increased polarizability of the argon plasmas may have a similar explanation, although the role of vibrationally excited species is taken over by metastable atoms: a metastable argon atom has a polarizability factor of ~ 100 larger than in the case of ground state atoms (Huddleston 1965).

6. Conclusion

A special Michelson interferometer based on a frequency-stabilized He-Ne laser has been constructed and shown

to be operational. The ambiguity of conventional interferometers with regard to the sign of the path-length change is eliminated by the implementation of a feedback loop. This loop also yields a signal that is directly proportional to the path-length change (no sine-dependency). The optimum resolution is of the order of 0.1 nm. Measurements performed in an RF plasma in CF_4 indicate that the change of the refractive index is positive, and of the same order of magnitude as $(n-1)$ without a plasma. Up to now, no solid explanation for this large effect has been found. Vibrational excitation seems a good candidate.

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References

- Bisschops T H J 1986 Investigations on RF plasmas related to plasma etching *PhD Thesis* Eindhoven University of Technology, The Netherlands
- Haverlag M, Kroesen G M W and de Hoog F J 1989 *In situ* measurement of C_nF_m densities in an RF CF_4 plasma using infrared absorption spectroscopy *Proc. 1Xth Int. Symp. Plasma Chemistry (Bari, Italy)*
- Hirschfelder J O, Curtiss C F and Bird R B 1967 *Molecular Theory of Gases and Liquids* (New York: Wiley)
- Huddleston R H 1965 *Plasma Diagnostic Techniques* (New York: Academic)
- Kolos W and Wolniewicz L 1976 Polarizability of the hydrogen molecule *J. Chem. Phys.* **46** 1426
- Landolt-Börnstein 1962 *Zahlenwerte und Funktionen aus Physik, Chemie, Astronomie, Geophysik und Technik* 6th edn vol II-8 *Eigenschaften der Materie in ihren Aggregatzustände: Optische Konstanten* (Berlin: Springer)
- Lochte Holtgreven W (ed) 1968 Evaluation of plasma parameters *Plasma Diagnostics* (Amsterdam: North-Holland) pp 184-6
- Paudey P and Sautry D 1979 Vibrational contribution to molecular polarizabilities and hyperpolarizabilities *J. Chem. Phys.* **73** 2899
- Timmermans C J, Rosado R J and Schram D C 1985 An investigation of non-equilibrium effects in thermal arc plasmas *Z. Naturf.* **40a** 810-25
- Voskamp J H, van Nijmegen F C and van der Wal A J 1989 Phydas, een interactief systeem voor data-acquisitie en besturing van fysische experimenten *Nederlands Tijdschrift voor Natuurkunde* **A55** 50-5