# PLASMA CHARACTERIZATIONS OF AN ELECTRON CYCLOTRON RESONANCE MICROWAVE PLASMA PROCESSING REACTOR

by

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A Thesis
Submitted to the Faculty of Graduate Studies
in Partial Fulfillment of the Requirements
for the Degree of

DOCTOR OF PHILOSOPHY

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# Abstract

This thesis presents a plasma-physical characterisation of an electron cyclotron resonance (ECR) microwave plasma processing reactor. These experiments are intended to provide an empirical connection between the reactor and the plasma as well as the phenomenological roots of a model of reactor-plasma interactions. First, a brief presentation of pertinent background materials and a critical review of the literature concerning plasma characterisations of divergent magnetic field ECR reactors is given. It is concluded that the current understanding of these systems is poor, and that extensive experimentation is required before useful models can be developed.

Several plasma diagnostics are first adapted for use in the ECR system. The stability of the plasma is characterised using visual inspection, reflected versus incident microwave power characteristics, and dynamic measurements of the microwave power or the floating voltage of a probe inserted into the plasma. It is shown that the plasmas occur in the form of stable, quiescent "plasma modes" characterised by unique shapes and power characteristics, and continuous dependences on system variables. Transitions between modes are noisy, discontinuous and often bistable. It is proposed that the plasma modes resulted from the mixing of electromagnetic waveguide modes due to changes in the refractive index of the plasma.

The above diagnostics are then used to select a stable operating regime of the divergent field ECR configuration for detailed characterisation. A computerised data acquisition and analysis system based on a cylindrical orbital-motion limited Langmuir probe is used to measure the axial variation of the plasma density, electron temperature, plasma potential and floating voltage as functions of microwave power, pressure and magnetic field strength. The density is observed to depend primarily on diffusion along the magnetic field away from a source region located at ECR. Increasing the power increases the density, and increasing the pressure reduces the axial diffusion length. The electron temperature shows a spike at ECR due to efficient heating inside the ECR zone, and inelastic collisional cooling outside. Increasing either the power or pressure decreases the temperature slightly. Varying the magnetic field strength simply shifted the electron temperature profile axially along with the location of the ECR zone. The density profile moved only partially since it also depended on the locations of the chamber walls. The potentials were shown to depend on the density and electron temperature.

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# CHAPTER 1

# INTRODUCTION

*Basic research is when I'm doing what I don't know what I'm doing.*
- Werner von Braun

The subject of this thesis is a plasma-physical analysis of an electron cyclotron resonance (ECR) microwave plasma processing reactor. In this chapter some background material is given, as well as a discussion of the factors which motivated this work. The objectives and organisation of the thesis are also described.

## 1.1. PLASMA PROCESSING

In general, plasma processing refers to the synthesis, decomposition or modification of materials using a plasma. More specifically, this term usually implies the use of an electrical discharge to perform various surface modification procedures during the fabrication of microelectronic circuits. These procedures can be grouped into four categories:

(1) *Cleaning*: A plasma is used to chemically and/or physically remove unwanted surface deposits such as adsorbed contaminants, native oxides or protective coatings.

(2) *Etching*: A plasma is used to cut specific patterns into a surface through the selective removal of material. This is accomplished through the use of protective masks and plasma conditions which only remove certain substances. While similar to cleaning in principle, it is quite different in intent.

(3) *Growth*: A plasma is used to generate reactive chemical species which combine directly with the surface to form a modified surface layer (oxidation, for example).

(4) *Deposition*: A plasma is used to either chemically (through homogeneous or heterogeneous chemical reactions) or physically (through sputtering or beam formation) generate species which will deposit a thin-film (adsorb, nucleate/react, coalesce) on a surface. The essential difference between growth and deposition is that in growth the plasma species react *with* the surface, while in deposition they react *on* the surface.

By far the greatest use of plasma processing takes place in the microelectronics industry; etching applications alone sustain a multi-billion dollar equipment market.[1] The recent adoption of VLSI (very large scale integration) integrated circuit technology has greatly increased the use of plasma processing in integrated circuit fabrication. VLSI circuits require sub-micrometer features which necessitate the use of highly directional and selective etching processes only attainable with plasma techniques.[2] Small feature sizes also mean that conventional growth processes cannot always be used because the high temperatures often involved induce excessive diffusion of, or structural instabilities in, some materials.[3] Plasma deposition can be used to overcome these problems, since energy is coupled into the plasma rather than the surface, allowing films to be grown at much lower surface temperatures.[2] In addition, some materials can *only* be produced by plasma techniques, such as hydrogenated amorphous silicon, which is used in solar cells and xerographic drums.[4] Plasma processing is compatible with other important vacuum techniques used in integrated circuit fabrication (molecular beam epitaxy, ion implantation), and is safer to use, cleaner and less polluting than older methods, many of which rely on the use of liquid chemicals.[5]

## 1.2. MOTIVATIONS

For mainly historical reasons, the most commonly used plasma processing technique is capacitively coupled, radio-frequency (typically 13.56 MHz) glow discharge (RFGD) with internal parallel-plate electrodes.[6] Such systems are capable of etching features smaller than 1 μm with a high degree of anisotropy and selectivity.[7] They can also produce thin-films of many of the important materials used in integrated circuit fabrication.[8] These reactors achieve anisotropic etching through the production of high energy ions, which bombard the sample surface in the presence of chemically reactive species,[9,10] activating their reaction with the surface. These energetic ions are produced by the large DC electric fields in the electrode sheaths and can damage the etched surfaces.[3,11] This damage (in the form of defect creation, contamination, layer mixing and/or surface roughening), is often not tolerable in VLSI or GaAs circuits. Similar considerations apply to deposition, with the added problem that the substrate temperatures required to produce quality materials are often still too high.[3,12] Thus there appears to be room for improvement, primarily in reducing ion energies and lowering the substrate temperatures required for good thin-film quality.

ECR plasma processing systems are thought to offer improvements over RFGD and other methods in both of these areas.[3,12,13,14,15,16] ECR reactors can perform highly controllable, selective, sub-micron, directional etching with little surface damage.[14] They have also been used to produce quality thin-films at low substrate temperatures and at good deposition rates.[3,12,17,18] In particular, ECR systems are capable of single-pass planarisation (the filling of trenches to produce a flat surface), a process which requires several passes through RFGD systems.[3] ECR reactors typically generate high plasma densities at low pressures which results in high ion fluxes at low energies. These are often desirable properties for both etching and deposition. High densities imply high dissociation rates which, in turn, are often credited with allowing high processing rates to be maintained at low substrate temperatures without sacrificing the quality of the process. High ion fluences appear to activate surface reactions, especially in etching[6] and also perhaps in deposition. If desired, ion energies can be increased separately with DC or RF sample biases,[19,20] a process more versatile and controllable than possible in RFGD, where the bias is inseparable from the method of plasma production.

It therefore appears that ECR plasma processing may be of great importance in the effort to push integrated circuit feature sizes into the submicron regime. This view is reflected in the recent surge of interest in ECR systems in the U.S.A.*, which had

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* Consider, for example, the 12 million dollar (over 5 years) Center for Plasma-Aided Manufacturing started in 1988 at the University of Wisconsin-Madison. Funded mostly by industrial contributions, one of the four key areas targeted for extensive (and largely catch-up)

largely ignored this technology throughout its developmental stages. In Canada, Bell-Northern Research has been studying the application of ECR plasmas to silicon nitride deposition and GaAs etching for several years.[21] Their parent company, Northern Telecom, is now using commercial ECR systems for prototype device fabrication[†]. Mitel S.C.C. is also planning pre-production trials of commercial ECR reactors in 1990 to evaluate their use in advanced IC fabrication processes.[22]

Given that plasma processing is vital to advanced integrated circuit fabrication and that ECR systems are becoming important plasma processing tools, it should be obvious that understanding how ECR plasma processing systems work and what they can do is of great importance. This is the general motivation for this work, as well as the applications research which has been performed in our laboratory for the last six years.

### 1.3. OBJECTIVES

Plasma processing is, by necessity, a largely empirical science[*]. All plasma processing systems have a large number of system variables which influence the plasma in a host of complex ways, and which also interact with each other via the plasma. System and applications development *must* proceed primarily by trial-and-error because currently there are no models of these highly complicated systems. However, because of the widespread use and commercial importance of capacitive RFGD systems, considerable effort has recently gone into developing models of these systems. This has been made possible by the appearance of powerful, inexpensive computers with which to perform the extensive numerical computations required for such models, and a large body of experimental data concerning the physical and chemical properties of RF plasmas. Such data is conspicuously absent in the field of ECR plasma processing, a result of the novelty and rarity of these systems. Some ECR plasma characterisation studies have been performed recently (and will be reviewed in chapter 2), but these have only scratched the surface of the immense parameter space available to these machines. Only very recently have any models concerning ECR plasma processing been proposed.[23,24]

The ECR microwave plasma processing reactor that is the subject of this thesis was designed and built by Sergio Mejia and Bob McLeod in order to fabricate amorphous hydrogenated silicon (a-Si:H) thin-films intended for use in solar cells.[25,26,27] The fabrication of microcrystalline silicon ($\mu$c-Si:H) thin-films, as a potential replacement for polycrystalline silicon in VLSI integrated circuits, was also studied,[28,29,30,31] as was the etching of Si and $SiO_2$.[32,33] Recent efforts have focussed on low-temperature deposition of $SiO_2$ thin-films for use as a general-purpose dielectric in Si and GaAs integrated circuits, and perhaps as a gate dielectric in VLSI and sub-micron MOSFET's.[17,18,34,35,36] Until this thesis, however, no detailed plasma characterisations

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research is plasma processing of semiconductors by ECR.

[†] From employment advertisements and NT job interviews of colleagues.

[*] Consider, for example, the following comment contained in the brochure advertising the Massachusetts Institute of Technology's summer course (No. 10.61s, 1989) on plasma processing: "Plasma processes have been developed, largely, by a trial-and-error approach with minimal understanding of the plasma physics and chemistry".

have been undertaken.

The overall purpose of this thesis is to take some of the first serious steps in developing a detailed understanding of the physical processes important in ECR and sub-ECR magnetised microwave processing plasmas. This is to be done through a number of basic plasma-physical (as opposed to plasma-chemical, which are application dependent and thus not considered in this thesis) experiments on a proven research ECR plasma processing reactor, with the intent of answering as many of the following questions in as much detail as possible:

(1) What kind of information about these plasmas is most important, and what techniques must be used to obtain this information?

(2) What are the fundamental properties of these plasmas, and how do they depend on important system variables?

(3) Which plasma mechanisms are, and which are not, important and how do they influence the properties and behaviours of the plasmas?

(4) What are the basic components that a theoretical model or description of these plasmas should possess?

Specifically, the experiments are to consist of the most basic and informative plasma diagnostic (characterisation) techniques. Some of these will be developed as part of this thesis while others will be standard techniques, modified so that they will function reliably under the conditions found in these plasmas. The aim will be to measure the most immediately informative properties of the ECR plasmas. These include those properties that are both obviously important yet widely neglected, such as visible shape and microwave power absorption, as well as the standard parameters of plasma density and electron temperature. Relative trends as functions of the most important system variables (microwave power, pressure, magnetic field strength and position) will be of primary interest.

Whenever possible, the results will be interpreted in terms of basic theoretical concepts in order to develop a more rigorous understanding of ECR plasma processing systems. The results of these efforts will then be used to make recommendations towards design improvements and process optimisation procedures.

## 1.4. ORGANISATION

This thesis begins with an introduction to ECR plasma processing and an outline of the motivations and objectives of this thesis. In chapter 2, the background material important to this thesis is presented. A general review of basic plasma physics is given first, followed by a brief description of the design of ECR plasma processing reactors. The last part of chapter 2 consists of a critical review of the literature concerning plasma characterisations in ECR processing reactors.

The specific ECR system studied in this thesis is described in detail in chapter 3, along with the Langmuir probe diagnostic system used to characterise the plasmas. Additional theory pertinent to the system technologies and diagnostic techniques is also given.

Early experiments, which were critical in defining the objectives and methods of this thesis, are presented in chapter 4. Chapter 5 then describes a set of three complementary, qualitative diagnostics which were found to be very useful in characterising the overall behaviour of ECR and magnetised microwave processing plasmas.

The heart of this thesis is contained in chapter 6. This chapter describes the results of a detailed Langmuir probe characterisation of divergent field ECR processing plasmas, with emphasis on their axial structure and responses to system variables. The theoretical and practical implications of these results are also discussed.

Finally, the conclusions and recommendations of this thesis are presented in chapter 7.

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# CHAPTER 2

# REVIEW OF ECR PROCESSING PLASMAS

*There is something fascinating about science.*
*One gets such wholesale returns of conjecture*
*out of such a trifling investment of fact.*
- Mark Twain, *Life on the Mississippi.*

The purpose of this chapter is to introduce the basic physical concepts important in ECR microwave processing plasmas, to describe existing ECR plasma processing reactors and to review the literature concerning the physical plasma properties of these systems.

Throughout this thesis it will be assumed that the reader has at least a passing familiarity with the kinetic theory of gases. In particular, the fact that knowledge of the energy (or velocity) distribution function (EDF) of a gas enables one to calculate virtually all of its macroscopic properties is of fundamental importance. In plasmas of the type used in processing applications it is the electron gas which determines the majority of the plasma properties, so knowledge of the electron EDF (EEDF) is essential in any efforts to model the physics or chemistry of such plasmas. Its is also often true in these plasmas that the EEDF is well approximated by the equilibrium *Maxwellian* EDF. This distribution is uniquely specified by two parameters; the particle *density* per unit volume, $n$, and the *temperature*, $T$, which is a measure of both the average kinetic particle energy and the variance of the distribution about this value. Therefore, knowledge of the electron density, $n_e$, and temperature, $T_e$, is of fundamental importance in any efforts to model either the plasma or the processes which it is used to perform. Fortunately these are relatively easy parameters to measure, and their determination has been the primary focus of most plasma characterisations of ECR processing plasmas to date, this thesis included.

## 2.1. SOME BASIC PLASMA PHYSICS

The primary purpose of any processing plasma is to generate chemical reactions within a gas or between a gas and a surface. This is accomplished through the generation of free electrons, ions and photons, as well as molecular, atomic and radical species of varying degrees of excitation. The chemical nature of these products determines the kinds of plasmas that must be used; electron temperatures in the range of 1 to a few tens of eV are required to create viable chemical species and induce reactions, while moderate electron densities of $n_e > 10^{14}$ m$^{-3}$ are needed to produce significant reaction rates.

ECR plasma processing systems produce chemical plasmas using low pressure, flowing, bounded, magnetised microwave discharges. The resulting plasmas are generally *quasi–neutral* and *weakly ionised*. Quasi-neutrality is a defining property of plasmas[1] which requires that $n$ be large enough that the charged particles are able to