New Plasma Source of Hydrides for Epitaxial Growth

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PREFACE

A novel plasma-activated selenium source has been developed in the course of this contract which is significantly different than any other heretofore reported in the scientific literature of the field. It is microwave-excited, magnetically confined plasma sources that is intended to operate under Electron Cyclotron Resonance (ECR) conditions at 2.455 GHz. This source is designed to excite and dissociate the vapor exiting from the aperture of an effusion cell. It combines the effusion cell's elemental vapor flux with a stream of hydrogen and/or helium gas at the resonance point.

The source has been developed to achieve the benefits previously demonstrated using other plasma assisted epitaxial growth techniques without incurring the penalties associated with prior approaches. These benefits include greater safety resulting from the elimination of hypertoxic hydride reactants, greater control of the reaction chemistry achieved by the ability to separately control the ratio of the evaporated element to hydrogen in the plasma, improved material quality at lower epitaxial growth temperatures and reduced interdiffusion at heteroepitaxial interfaces. Furthermore the lower ion energies characteristic of an ECR plasma have been demonstrated to generate negligible ion bombardment damage compared to conventional RF plasmas.

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Approval Signature

Date

SUMMARY

OBJECTIVES

To design, build, and install into a vacuum deposition system a plasma-activated selenium source and demonstrate its operation. The source utilizes elemental selenium and hydrogen as reactants and the system is configured for the growth of CuInSe_2 by elemental evaporation of copper and indium from conventional evaporation sources.

DISCUSSION

A novel plasma-activated selenium source has been developed in the course of this contract which is significantly different than any other heretofore reported in the scientific literature of the field. It is microwave-excited, magnetically confined plasma sources that is intended to operate under Electron Cyclotron Resonance (ECR) conditions at 2.455 GHz. This source is designed to excite and dissociate the vapor exiting from the aperture of an effusion cell. It combines the effusion cell vapor flux with a stream of hydrogen and/or helium gas at the resonance point.

Experiments have been conducted which have successfully demonstrated the achievement of a stable microwave plasma discharge in helium gas. Measurements of the magnetic flux profile of the source suggest, however, that it does not currently operate in an ECR mode as intended. Since power absorption in an ECR plasma is more efficient than the non-resonant absorption which apparently pertains to the source as presently configured, it is anticipated that an improved magnetic confinement design would reduce the 150 watts minimum power currently needed to establish a stable discharge. Furthermore, the lower energy ion distribution characteristic of an ECR plasma is expected to be more beneficial for the minimization or elimination of ion bombardment damage when utilized for plasma assisted epitaxial growth processes. Preliminary calculations indicate that a supplementary solenoidal field coil can be readily incorporated into the design which would substantially increase the resonant excitation volume and enable operation in the desired ECR mode.

CONCLUSIONS

Application of this novel source is anticipated to enable a low temperature, safe process for the growth of epitaxial compound semiconductor films, and in particular CuInSe_2 . The improvements resulting from the demonstration of the technique may be transferrable to other material systems, enabling the reduction in growth temperatures and improvement in material quality of epilayers of GaAs or other III-V compounds utilizing elemental group V reactants instead of their hydrides, with associated improvements in process safety, and hence a reduction in costs. This reduction of epitaxial growth temperatures may enable the fabrication of novel photovoltaic devices which have heretofore been impossible due to the deleterious effects of interdiffusion at heterointerfaces.

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1.0 INTRODUCTION

The history of progress in the development of semiconductor materials can be generally viewed as revolving around two fundamental issues: purity and process temperature reduction. Purity is essential due to the monotonic reduction in charge carrier mobilities which occurs as a consequence of impurity scattering. In a few exceptional cases, semiconductor alloys such as $\text{In}_x\text{Ga}_{1-x}\text{As}$ contain isoelectronic "impurities" resulting in a net increase in carrier mobility due to other effects, such as increases in the curvature of the conduction band dispersion relation's minima (and hence the intrinsic mobility). Even in this case, however, the total mobility is the net difference between an increase due to the latter effect, and a decrease due to the former. Process temperature reduction is inherently important for several reasons. First, low temperatures are often necessary to prevent undesirable interdiffusion of component species at the interface between different materials. Second, lower crystal growth temperatures often effect a reduction in the density of point defects incorporated into the films. This empirical observation is reasonable, even in the case of non-equilibrium growth techniques, in view of the rigorously demonstrable temperature dependence of equilibrium vacancy concentrations (Schottky disorder) [1]. The growth of II-VI compound semiconductor layers is particularly sensitive to high growth temperatures due to the high volatility of *both* the group II and the group VI compounds. This is distinctly different from the case of III-V compound semiconductors, where the group III element is essentially involatile, and this difference manifests itself in a fundamentally different relationship between the flux ratio and growth rate in Molecular Beam Epitaxy (MBE) [2, 3]. Examples of lower deposition temperatures leading to significant advances in semiconductor device technology abound. OrganoMetallic Vapor Phase Epitaxy (OMVPE) has largely supplanted chloride-based VPE as a production process for GaAs in large part due to the fact that it results in lower deposition temperatures. Plasma-Enhanced Chemical Vapor Deposition (PECVD) of silicon nitride has become a standard part of commercial silicon device fabrication technology because of the extremely high temperatures required for its pyrolytic deposition.

From an economic perspective, deposition temperature reduction has a significant impact on the ultimate cost of fabricating high volumes of semiconductor materials as required for mass production of solar cells for terrestrial applications. As processing temperatures drop, the demands on system materials are reduced, enabling the use of lower cost construction materials. For example, at temperatures $\leq 350^\circ\text{C}$ high vacuum systems may be reliably built from aluminum, whereas at higher temperatures stainless steel must be utilized, with concomitant increases in equipment cost.

Plasma-enhanced deposition processes have been proven to be effective in the reduction of temperatures for the epitaxy of many semiconductor materials including GaAs by Physical Vapor Deposition (PVD) [4], OMVPE [5], and Metal-Organic MBE (MO-MBE) [6]; ZnSe by PVD [7] and OMVPE [8]; GaSb, InSb, and InAs by PVD [9], $\text{Hg}_x\text{Cd}_{1-x}\text{Te}$ by OMVPE [10], GaN by OMVPE [11], and ZnO by OMVPE [12]. In *every* case significant reductions in the minimum temperature required for the onset of single crystal epitaxial growth were observed, varying from 150-300°C. There are potential problems with this approach, however, mostly relating to impurity contamination and ion-bombardment damage. We believe the approach developed in the course of this contract will make a significant contribution to low temperature deposition technology by alleviating these problems through a unique approach to plasma excitation of the reactants.

This approach to lowering epitaxial growth temperatures will also result in a significant reduction in process equipment and facilities expenses (and hence photovoltaic costs) by pioneering a new technique for the *in-situ* generation of hydride reactants. The new deposition source can

significantly improve the safety of epitaxial growth processes by eliminating the need for the storage or transport of large quantities (typically gas cylinders) of the hyper-toxic hydride chemicals.

Many approaches to solving this safety and cost problem have been studied, including the use of non-hydride precursors. In OMVPE for example, tertiarybutylarsine has been used to replace arsine for GaAs epitaxy [13], and methylallylselenide has been used to replace hydrogen selenide for ZnSe epitaxy [14]. However, problems persist in this approach with residual carbon impurities and precursor costs. Another approach, pioneered by L. M. Fraas for deposition processes using OM precursors, and routinely used in PVD deposition processes like MBE, is to employ an elemental source such as arsenic for GaAs epitaxy or selenium for ZnSe epitaxy. In the case of OM processes, this approach does not in itself solve the problem of residual carbon contamination.

In the case of plasma-activated PVD processes, it has been shown that the use of hydrogen provides distinctly superior results in the quality of materials grown, when compared to argon [15]. Those results suggest that utilizing hydrogen may have beneficial results under circumstances where the reactants themselves are not sources of carbon contamination. There are several plausible mechanisms which may be suggested for this effect. First, since hydrogen atoms, radicals and ions are all powerful reducing agents, they may effectively getter oxygen or displace chemisorbed oxygen adatoms on the growth surface, increasing their desorption rate; Second, chemisorbed hydrogen may passivate dangling surface bonds, thereby reducing the binding energy of subsequently impinging species, an effect which has been shown to occur in some material systems [16]; and third, since the mass of hydrogen atoms is smaller than that of argon, momentum transfer to the lattice of the crystal, and consequently lattice displacement, is less than the argon case [17]. The approach developed under this contract utilizes elemental selenium in a hydrogen plasma to generate hydrogen selenide and related radical and ionic species *in-situ*.

The novel plasma-activated selenium source developed in the course of this contract is significantly different than any other heretofore reported in the scientific literature of the field. It is microwave-excited, magnetically confined plasma sources that is intended to operate under Electron Cyclotron Resonance (ECR) conditions at 2.455 GHz. This source is designed to excite and dissociate the vapor exiting from the aperture of an effusion cell. It combines the effusion cell vapor flux with a stream of hydrogen and/or helium gas at the resonance point. A non-resonant, higher pressure approach to the *in-situ* generation of arsine from elemental arsenic and hydrogen has been demonstrated in a "downstream microwave plasma" operating mode for OMCVD application [18]. However, all ECR sources reported in the literature, to our knowledge, utilize gas precursors (e.g.: hydrogen or arsine). Thus this modified ECR source enables plasma-assisted epitaxial growth at reduced pressures utilizing the safer elemental reactants instead of the hydrides (in this case selenium instead of hydrogen selenide). Furthermore, the ability to inject mixtures of hydrogen and an inert gas such as helium provides another dimension of control over the relative composition of the flux generated by the source. Presumably a lower ratio of hydrogen to selenium vapor in the plasma will reduce the steady state concentration of hydride species or shift their distribution towards greater concentrations of less completely hydrogenated species. This control over the reactant species distribution is unavailable with ECR sources which utilize hydride precursors and represents another advantage of this novel plasma source.

Conceptually, the source may be viewed as an alternative to a conventional thermal "cracker" utilized to convert the flux of thermally evaporated molecular species to a flux of decomposed molecular species which are more readily incorporated into the crystal lattice (e.g.: As_4 to As_2). A CAD drawing of the final source design, including a coupled effusion cell is shown in figure 1.1.

Figure 1-1. CAD drawing of TE₀₁₁ plasma cracker with coupled effusion cell.

2.0 METHODS

2.1 DESIGN OF SOURCES

The ECR technique has important advantages compared to other techniques of plasma excitation, particularly for crystal growth applications. Excitation at microwave frequencies (e.g.: 2455 MHz) rather than at RF (typically 13.56 MHz) significantly reduces the average ion energies within the plasma since there is less time during which the ions can accelerate and absorb energy within each cycle before the reversal of the force on the ions due to the electromagnetic field's oscillation. Typical ion energies in microwave plasmas are tens of volts, while typical ion energies in RF plasmas are hundreds of volts. Another related consequence of this frequency difference is that the sheath potential of microwave plasmas (i.e.: the potential difference between external ground and the plasma volume) is significantly lower than that of RF plasmas. The sheath potential is a consequence of the higher mobility of electrons than ions in response to the impressed electromagnetic field. Electrons tend to escape to grounded surfaces surrounding the plasma faster than ions, hence in steady state the plasma assumes a positive electrostatic potential with respect to ground in order to establish a dynamic equilibrium between the electron and ion loss and generation mechanisms, thereby maintaining quasi-neutrality in the plasma volume. The importance of these differences for crystal growth applications is extremely significant. The value of ion bombardment of growth surfaces for increasing the surface mobility of adatoms has been clearly demonstrated [17]. However, it is critical for the growth of high quality crystalline materials that the energies of the incident species at the growth surface be sufficiently low to prevent ion bombardment damage, else the growth temperature (or post-growth thermal treatment temperatures) must be sufficiently high to anneal the point and/or extended defects created by the bombardment.

Magnetic confinement is used in conventional RF plasmas as well as microwave plasmas. However, a resonance occurs when the excitation frequency coincides with the natural "cyclotron" frequency (also known as the Larmor frequency) of the electrons' circular rotation around their guiding centers as they follow helical trajectories in a magnetic field, given by:

$$f_{ce} = \frac{eB_0}{m_e} = B_0 * 2.8 \text{ GHz/kG}.$$

A more complete treatment [19] of the coupled electromagnetic wave and plasma system shows that the eigenmodes for the propagation of electromagnetic waves in a plasma along the direction of a static magnetic field are given by the poles of the complex index of refraction in the dispersion relation:

$$\omega = \frac{c}{\tilde{n}} k$$

where the complex index of refraction is given by:

$$\tilde{n}^2 = 1 - \frac{\omega_p^2 / \omega^2}{1 \pm (\omega_c / \omega)}$$

with

$$\omega_p^2 = \frac{n_0 e^2}{m} \quad \text{and} \quad \omega_c = 2\pi f_{ce}$$

defining the plasma and cyclotron angular frequencies respectively; m , n_0 and e are the electron mass, density and charge. The mode characterized by the negative sign in the denominator of this complex index of refraction is called the “whistler” mode (for historical reasons; see [19] § 4.17.1) or R-wave (because it is right-hand circularly polarized), and is the relevant mode to ECR plasma excitation. Hence, unlike conventional RF magnetically confined plasmas, the magnetic field in an ECR plasma creates a resonance which dramatically increases the absorption of energy by the plasma from the power source. The result of this is that ionization efficiencies in ECR plasmas are one to two orders of magnitude higher than in conventional RF plasmas.

Another significant consequence of the stronger coupling between the excitation and plasma in ECR plasmas is that a self-sustaining discharge can be maintained at pressures one to two orders of magnitude lower than conventional RF discharges. Pressures in the 10^{-4} - 10^{-5} torr range can be achieved, and are desirable in this and many other low-pressure techniques where long mean free paths are beneficial.

The extraction of ions from an ECR source results from divergence of the static magnetic field and the fact that ion trajectories (in the absence of collisional scattering) follow the magnetic field lines. Since the magnetic moment enclosed an ion’s helical trajectory in a magnetic field is an adiabatic invariant [19] ions gain kinetic energy when the travel from a region of higher to lower magnetic field. Hence, the energy of ions incident on the growth surface can be directly controlled (over a finite range) by controlling the relative magnetic field at the growth surface compared to the field in the source [20].

The basic source design is a 2.455 GHz resonant microwave cavity placed within a permanent magnet flux shunt assembly to create a “magnetic mirror” plasma confinement volume. Microwave power is coupled to the cavity via a high temperature coaxial microwave cable, and coupled within the cavity to the plasma by a helical antenna designed to couple efficiently to the R-wave eigenmode. The reactants are isolated from the cavity by a sapphire tube in order to prevent unwanted deposition within the source, and to insure that all of the escaping reactant flux is directed toward the substrate. Two sources have been used, a TM_{011} cavity (which is relatively small), and a TE_{011} cavity.

The relative advantages of ECR microwave plasmas compared to conventional and magnetically confined RF plasmas have discussed in this introduction. In summary, they are lower ion energies, control of incident ion energies via control of the magnetic field strength at the growth surface, higher ionization efficiency, and lower pressure operation. The advantage of this ECR source over commercially available ECR sources, or conventional ECR source designs is the utilization of elemental reactants rather than hydride reactants with the concomitant improvements in safety and hence cost. The results of this source development could be applicable to other material systems, such as GaAs, and other growth techniques, such as VCE. Details of specific source design issues will be discussed in more detail in the following subsections.

2.1.1 Antenna Design

Symmetry considerations are extremely important in optimizing the design of the discharge chamber to maximize the power coupled from the power supply into the plasma. The whistler mode is a TEM-type mode propagating parallel to the external static magnetic field, hence the electric and magnetic fields oscillate in the plane perpendicular to the wave’s direction of propagation. An externally impressed microwave field and cavity modes excited by that external field can only couple to this plasma mode to the extent they share this symmetry.

Most commercial ECR sources couple microwaves into a cylindrical ionization cavity by means of an axial, end-coupled TE₁₀ rectangular waveguide. This couples strongly to the cavity if it is designed to resonate in a TE mode at the oscillator's frequency, which can in turn couples strongly to the Whistler plasma mode (presuming an axial static magnetic field is used). This end-coupled waveguide, however, is completely incompatible with an end-coupled effusion cell and any other hollow waveguide coupling geometry would not efficiently couple the external power with the cavity modes.

Hence, an antenna had to be designed which would effectively couple to both a cavity TE mode and the plasma whistler mode. The choice of antenna coupling to the cavity immediately forces the use of coaxial waveguides to deliver power to the antenna. This created two challenging issues: a microwave cable and power feedthrough for the vacuum chamber. This section will discuss the significant issues and solution to each of these three problems.

Helical antennas have long been used for microwave applications since their invention in 1946, and a great deal of empirical and analytical information is available regarding their impedance and radiation characteristics in less complex applications [21]. When embedded in a resonant cavity, analytical calculation of their effective radiation impedance becomes an intractable problem. Nevertheless, the techniques developed for impedance matching of helical antennas to coaxial transmission lines [22] have been implemented in the final antenna design to the extent feasible given manufacturing constraints. Specifically, the antenna is peripherally fed via a bulkhead feedthrough in the base of the cavity and dielectrically isolated from the ground plane by a boron nitride disc against which it rests (pitch=0) for the first half-revolution, creating a $\lambda/2$ transmission line coupling. The pitch increases linearly over the second half-revolution to its final value determined by the radius and the "one integral wavelength per revolution" constraint described next.

One goal of the overall source design is to maximize the interaction probability in order to achieve the greatest possible degree of molecular dissociation, excitation and ionization. In order to maximize the transverse electric field strength along the central axis of the cavity a helical antenna design was selected with the helix diameter and pitch chosen to give a path length along the antenna of one wavelength at 2.455 GHz per revolution. This insures that the electric field vector in the *interior* of the helix is predominantly radial. The wavelength is fixed by the oscillator frequency at:

$$\lambda_{2.455 \text{ GHz}} = \frac{c}{\nu} = \frac{2.998 \times 10^{10} \text{ cm/sec}}{2.455 \times 10^9 \text{ cycles/sec}} = 12.21 \text{ cm} = 4.807''$$

The general expression for the path length, s , of a helix of pitch l and with $\theta/2\pi$ rotations is:

$$s^2 = (\theta r)^2 + l^2 \leftrightarrow l = \sqrt{s^2 - (\theta r)^2}$$

Hence the maximum radius of a helical antenna at this wavelength subject to the "one integral wavelength per revolution" constraint is found by setting the pitch (l) to zero, or:

$$s^2 = (2\pi r)^2 \leftrightarrow r_{\text{max}} = \frac{12.212 \text{ cm}}{2\pi} = 1.944 \text{ cm} = 0.79''$$

These design choices and constraints led to the selection of a 1" outside diameter sapphire tube as the reaction vessel, when combined with the smallest physical dimensions of standard miniature microwave cable connectors (type SMA). Two antenna types were built, one with a radius of 0.6" and one wavelength pathlength; the other six wavelengths pathlength with a radius of 0.7"; for use with effusion cells with or without water cooling, respectively.

These antennas, the coaxial microwave cable and microwave vacuum power feedthroughs were custom built utilizing materials and techniques developed originally for advanced radar systems for demanding military aircraft applications. Several iterations of design were necessary to eliminate inadequacies which resulted in failure of the various components during the earlier technology development phases of this contract. Design features which were found to be important include venting of cable connectors inside the vacuum to prevent virtual leaks and localized plasma breakdown, the replacement of all brazed joints in the antenna assembly with laser welded or mechanically constrained connections to prevent thermal runaway and melting, and the use of hermetically sealed, silica-filled high temperature stainless-clad copper coaxial conductor cables.

2.1.2 Cavity Design

A resonant microwave cavity load has the effect of storing electromagnetic field energy, thereby increasing the strength of the fields. It can be shown that in the absence of an interior conductor, only two types of resonant modes can be sustained in a right circular cylindrical waveguide or cavity, those with radial magnetic field vector (TM) and those with radial electric field vector (TE) [23, p. 95]. Calculating the dependence of these mode frequencies on the geometric dimensions of ideal cavities is straightforward, but provides only a first order estimate of the resonant frequencies of a practical cavity design for this application, which includes perturbations due to apertures on either end, dielectric loading (the sapphire discharge chamber tube and boron nitride insulators) and interior conductive surfaces (the helical antenna and plasma column itself during operation). Calculating the magnitude of these perturbations is possible using finite-element analysis, but empirical quantification of these deviations from ideality is simpler, cheaper and quicker. The effect of the perturbations was correctly anticipated to be a reduction in the mode frequencies when compared to the idealized cavity calculations.

Experimentally, each cavity was originally constructed with a smaller inside diameter than would be calculated for the ideal mode at the pump frequency, microwave impedance measurements of the cavity were conducted to measure the deviations of the cavity from ideality, and then the cavities were re-machined to larger inside diameter in an iterative process which finally converged on the required dimensions. Other key considerations in the choice of cavity dimensions were maximizing the frequency separation of the desired mode from competing modes (which could result in mode-hopping and instability) and maximization of the quality factor (Q) of the desired cavity mode. Resonant mode frequencies were calculated using the expression [23, p. 328]:

$$f_{nm1} = \left[\left(\frac{x_{nm}}{a} \right)^2 + \left(\frac{l\pi}{h} \right)^2 \right]^{1/2} \frac{c}{2\pi}$$

where a is the cavity radius, h is its height, c is the speed of light and for TE modes $x_{nm} = p'_{nm}$ (the n^{th} root of the equation $J'_m(x)=0$ where $J'_m(x)$ is the first derivative of the Bessel function $J_m(x)$) whereas for TM modes $x_{nm} = p_{nm}$ (the n^{th} root of the equation $J_m(x)=0$).

The initial TE₀₁₁ mode cavity design was based on the general observation that the Q is maximal for approximately equal height and diameter, combined with a plot of “isofrequency” contours for the various neighboring modes at a frequency corrected by the loading and non-ideality shifts previously measured on a smaller cavity. In this context “unloaded” means the cavity was measured *without* the sapphire discharge tube inserted and “loaded” means measurement *with* the tube inserted (operational mode). The non-ideality shifts are the difference between the calculated mode frequencies for an ideal cavity and those measured for the unloaded cavity, which is still perturbed by the antenna, boron nitride insulators, and endface apertures. These results are shown in figure 2-1.

Figure 2-1. Calculated resonant frequency contours of TE_{011} and neighboring modes as a function of diameter and height of an empty ideal right circular cylindrical cavity.

Note that the total shifts of the different modes vary greatly, far more than their resonance bandwidths, as will be shown in section 3. Furthermore, the resonant frequency of the mode of desired symmetry, the TE₀₁₁ mode, is relatively insensitive to the cavity length. This makes accurate tuning relatively easy but conversely requires a very careful choice of cavity diameter if the effective tuning range is to be in the frequency domain of interest.

Table 2-1 Calculated mode frequencies of semifinal TE₀₁₁ cavity design at *minimum* tuning length limit.^a

Character	n	m	l	Bessel root (n,m)	f (GHz)
TE	1	1	1	1.841	1.5380
TM	0	1	0	2.405	1.5446
TM	0	1	1	2.405	1.8312
TE	2	1	1	3.054	2.1942
TE	1	1	2	1.841	2.2952
TM	1	1	0	3.832	2.4610
TM	0	1	2	2.405	2.5011
TM	1	1	1	3.832	2.6503
TE	0	1	1	3.832	2.6503
TE	2	1	2	3.054	2.7779
TM	1	1	2	3.832	3.1506
TE	0	1	2	3.832	3.1506

^adiameter = 5.850", length = 6.000"

Table 2-2 Calculated mode frequencies of semifinal TE₀₁₁ cavity design at *maximum* tuning length limit.^b

Character	n	m	l	Bessel root (n,m)	f (GHz)
TE	1	1	1	1.841	1.4994
TM	0	1	0	2.405	1.5446
TM	0	1	1	2.405	1.7989
TE	2	1	1	3.054	2.1673
TE	1	1	2	1.841	2.1907
TM	1	1	0	3.832	2.4610
TM	0	1	2	2.405	2.4056
TM	1	1	1	3.832	2.6281
TE	0	1	1	3.832	2.6281
TE	2	1	2	3.054	2.6923
TM	1	1	2	3.832	3.0754
TE	0	1	2	3.832	3.0754

^bdiameter = 5.850", length = 6.400"

Results of the ideal cavity mode analysis prior to the last design iteration of the TE₀₁₁ cavity are shown in tables 2-1 and 2-2 for the upper and lower length limits of the cavity's tuning range, respectively. Experimental data will be presented and compared with these calculations in section 2.2.

2.1.3 Magnetic Flux Shunt Design

As described in earlier sections, one goal of the overall source design is to maximize the interaction probability in order to achieve the greatest possible degree of molecular dissociation, excitation and ionization. Another means of achieving this goal is to use magnetic confinement techniques to increase the residence time and thereby the steady-state concentration of ions in the plasma volume. A magnetic mirror is a field arrangement originally developed for plasma research which utilizes two annular magnets or solenoids with their magnetic fields parallel to their common axis, and creates a region of lower magnetic field flux between them. Ions generated in this lower field region will travel in helical trajectories along the flux lines, but due once again to the adiabatic invariance of the magnetic flux enclosed in that helix they will *decelerate* as they approach the *higher* flux region, and given sufficiently high field gradients most will reflect back into the interior of the plasma volume. In the context of an ECR source, most primary ion generation will occur in the resonance field region, so it is important that this resonant field strength occur inside the microwave cavity and that higher fields exist near the entrance and exit apertures of the source.

Permanent magnets were chosen for this magnetic mirror design due to their relative compactness compared to sufficiently strong electromagnets. The tradeoff of greatest consequence was the requirement for substantial heat shielding, water cooling, and thermal isolation from the effusion cell and discharge cavity to prevent thermal demagnetization of the permanent magnets. In order to isolate their strong magnetic fields from the rest of the vacuum system instruments and to prevent their uncontrolled interaction with other magnetizable system components, these magnets were enclosed in a "flux shunt," an enclosure of highly susceptible magnetic materials which have the effect of "channeling" the magnetic field between the two permanent magnet annuli.

Samarium-cobalt magnets with a $\text{Sm}_2\text{Co}_{17}$ alloy composition and 26 Oersted-Gauss B-H field energy product were chosen. The cylindrical magnetic flux shunt is comprised of end-plates fabricated from a 49% nickel, 49% cobalt, 2% vanadium alloy sold under the trade name of "2V-Permandur," connected via a cylindrical coil of 49% iron, 49% cobalt, 2% vanadium alloy sheet metal sold under the trade name of "Permandur." These magnetic components were pre-assembled onto water-cooled copper heat spreaders which became the primary structural components of the entire source. The exterior of the copper components were nickel plated to prevent corrosion due to selenium vapor.

2.2 MICROWAVE CHARACTERIZATION

Microwave cavity impedance measurements were conducted throughout the course of this source development effort to provide feedback to the iterative design optimization process described in section 2.1.2. All of these measurements were made using a Hewlett-Packard 8753 network analyzer with a 8753B and 85047A modules for the measurement of forward and reflected power. The results of these measurements were plotted as reflected signal attenuation (in decibels) versus frequency. The measured data corresponding to the ideal cavity calculation results presented in tables 2-1 and 2-2 are shown in figures 2-2 and 2-3, respectively.

Figure 2-2. Measured impedance of semifinal TE₀₁₁ cavity design at *minimum* tuning length limit.

Figure 2-3. Measured impedance of semifinal TE₀₁₁ cavity design at maximum tuning length limit.

The assignment of modes and determination of shifts due to non-ideality were conducted by comparing the calculated and empirical data for unloaded cavities, as demonstrated with table 2-3 for the data extracted from tables 2-1, 2-2 and figures 2-2 and 2-3. In general it was found that mode identification was best made on the basis of shifts in frequency accompanying changes in diameter and length of a cavity than on the basis of the absolute frequency measurement, since the frequencies of the various modes shift by different amounts, sometimes resulting in empirically different ordering of the modes than were calculated.

Note that there are more experimentally measured modes than calculated. Some of these unpredicted resonances are clearly due to splitting of degenerate modes resulting from symmetry violation. Such modes are identifiable because they nearly perfectly track one another. Another resonance, labelled "antenna" is assigned as such because it does not shift with *any* change in cavity geometry. Finally, some of the modes may be unpredicted modes of TEM character since the introduction of the antenna in the interior of the cavity removes the previously mentioned proscription against TEM modes, which applies strictly only to cavities without interior conductors. On the other hand, no resonance was found in cavities of any dimension which correspond to the TE₁₁₂ mode predicted by the general theory. The reason for its absence is unknown.

**Table 2-3 Compilation of theoretical calculations and experimental data
demonstrating unloaded semifinal^a cavity mode assignments.**

Cylindrical Cavity Calculated Modes	f (GHz)		tuning shift Δf [ideal]	tuning shift Δf [data]	f (GHz)		Assignment of Measured Modes	Non-ideality Shift (measured-ideal)	
	height= 6.0000"	height= 6.4000"			height= 6.00"	height= 6.40"		height= 6.00"	height= 6.40"
TE 111	1.5380	1.4994	0.0386	0.0260	1.3925	1.3665	TE 111	-0.14549	-0.13292
TM 010	1.5446	1.5446	0.0000	-0.0070	1.5680	1.5750	TM 010	0.02343	0.03043
TM 011	1.8312	1.7989	0.0323		1.7590				
TE 211	2.1942	2.1673	0.0269	0.0310	1.8410	1.8100			
TE 112	2.2952	2.1907	0.1045	0.0190	1.8540	1.8350	TM 011	0.02284	0.03611
TM 110	2.4610	2.4610	0.0000	0.0230	2.1730	2.1500	TE 211	-0.02119	-0.01733
TM 012	2.5011	2.4056	0.0955	0.0780	2.4970	2.4190	TM 012	-0.00411	0.0134
TM 111	2.6503	2.6281	0.0222			2.4280			
TE 011	2.6503	2.6281	0.0222	-0.0010	2.4350	2.4360	TM 110	-0.02603	-0.02503
TE 212	2.7779	2.6923	0.0857	0.0120	2.5290	2.5170	TE 011	-0.12131	-0.11112
TM 112	3.1506	3.0754	0.0753	0.0020	2.5590	2.5570	antenna		
TE 012	3.1506	3.0754	0.0753			2.6730	TE 212		-0.01925
				0.0270	2.8580	2.8310			
				0.0550	2.9100	2.8550			
				0.0490	2.9260	2.8770			

^adiameter = 5.850"

The determination of shifts due to loading the cavity with the sapphire discharge chamber tube were conducted by comparing the empirical data for loaded and unloaded cavities, as shown in table 2-4 for the TE₀₁₁ mode. Note that the loading shift of this individual mode is quite reproducible and constant. Also, note that the lowest TE₀₁₁ mode frequency for this semifinal cavity design, corresponding to the loaded cavity at maximum tuning length, is too high.

Table 2-4 Comparison of frequency shifts of the TE₀₁₁ mode due to dielectric loading of the cavity^a at several different lengths.

length (in):	loaded f (GHz)	Δf	unloaded f (GHz)	Δf	Loading Shift
6.0000	2.468		2.529		-0.061
		-0.008		-0.009	
~6.35	2.46		2.52		-0.06
		-0.004		-0.003	
6.4000	2.456		2.517		-0.061

^adiameter = 5.850"

Figure 2-7. Final cavity design, tuned and fully loaded, *in-situ* TE₀₁₁ mode impedance measurement.

The final TE₀₁₁ cavity design, compensated for all the perturbations, loading effects, and deviations and from ideality described above, has an inside diameter of 5.875" and tuned to the microwave power supply frequency at an inside length of ~6.03". Its measured microwave impedance and derived parameters are shown in figure 2.7. Note that the bandwidth is only 1.74 MHz, quite narrow when compared to its tuning range (cf: Table 2-3) of ~12 MHz. The quality factor (Q) of 1409 is proportional to the ratio of the time-average energy stored in the cavity to the energy loss per second, which is $Q/2\pi$ or 224 [23, p. 314]. The return loss of -14.644 dB is equivalent to an 81% attenuation of the input voltage in the reflected wave amplitude. These characteristics are quite adequate for this materials processing application.

2.3 MAGNETIC PROFILING

To discover whether the source design would achieve the requisite ECR resonance condition, the magnetic field strength distribution of the completed assembly was measured *in-situ*. The data was collected using an F.W. Bell Model 4048 Gaussmeter with their model A-4048-002 axial magnetic flux probe. Thus, the data compiled in table 2-5 is a measurement of only the *axial* component of the magnetic field vector. Two sets of measurements were made, one at a radius approximately 1mm inside the sapphire discharge tube, the other along the center axis of the tube. Distances are measured from the exit aperture of the discharge tube downward toward the effusion cell. Note that both profiles achieve the critical field value of 875 Gauss for ECR resonance at 2.455 GHz outside of the interior of the resonant cavity which begins at a distance of 4.4 cm from the exit aperture. This is not a desirable configuration since the TE₀₁₁ mode has 0 azimuthal, 1 radial and 1 axial maxima in field strength and a transverse electric field z-dependence proportional to $\sin(\pi z/d)$. Hence, the ideal TE₀₁₁ cavity mode transverse electric field strength falls to zero at the endplates of the cylindrical cavity. In this configuration only fringing fields extending outside the cavity through the endplate apertures or propagated by a plasma column excited by another absorption mechanism can effectively excite the plasma in the resonance zone.

Table 2-5 Axial magnetic field strength profiles of the final source assembly.

axial distance (cm) =	Axial Magnetic Field Strength (Gauss) at											
	0	0.5	1	1.5	2	2.5	3	3.5	4	4.5	5	5.5
radial distance = 0 cm	146.2	55.1	383	569	765	908	873	634	292	-370	-237	
radial distance = 1.1 cm	112.7	35	340	599	923	993	952	597	151	-197	-358	-448

3.0 TECHNICAL DISCUSSIONS

3.1 SOURCE INSTALLATION

The TE₀₁₁-mode cavity and flux shunt assembly described in section 2 constitute the plasma "cracker" which is attached to the exit orifice of a commercial, water cooled, low temperature effusion cell, the EPI-20MLT manufactured by EPI Systems, Inc. The sapphire discharge chamber and effusion cell crucible are coupled by a small pyrolytic boron nitride disc incorporating a socket into which a 1/8" stainless gas delivery line is inserted. Microwave power is coupled to the microwave cavity antenna bulkhead fitting by the high-temperature hermetically sealed microwave cable described previously which attaches at the opposite connection to a 1" baseplate feedthrough with impedance-matched microwave coaxial couplers on either side of the vacuum seal. Power is provided by an Astex S-250 microwave power supply connected to the feedthrough via a circulator, water cooled dummy load and double-slug tuner. These latter features permit tuning of the load impedance to match the source and protect the power supply from excessive reflected microwave power which might otherwise damage the klystron.

The source is mounted onto a water-cooled baffle inside a rotating disc reactor which incorporates both copper and indium effusion sources as well as EIES sensors and quartz crystal oscillators for monitoring and controlling the deposition process. The system is designed for load-lock operation and incorporates custom radiant heaters for substrate temperature control.

3.2 SOURCE OPERATIONAL CHARACTERISTICS

Experiments were conducted to demonstrate operation of the source and test for microwave leakage from the system subsequent to its installation. The source was tested using helium gas. A steady state discharge was established for ~20 minutes at a forward power of 150 watts, reflected power of 2 watts and a system background pressure of 5×10^{-5} torr of helium (indicated pressure of 2.6×10^{-4} torr with calibration of the vacuum gauge controller for nitrogen and a sensitivity ratio of 0.18 [24]). The strong blue emission characteristic of recombination emission from helium plasmas was clearly observable under these conditions.

These operational characteristics are puzzling considering the inadequacy of the magnetic profiles for ECR resonance as discussed in section 2.3 above. Inadequate plasma characterization data is available to unequivocally identify the nature of the observed discharge. Nevertheless it seems likely that the observed plasma is predominantly a spacecharge wave resulting from the interaction of the plasma with charge accumulations at the surface of the sapphire discharge tube. This type of wave is not possible in the theory of idealized spatially infinite plasmas, but has been studied extensively in plasma research because of its common occurrence in practical experimental configurations, and in particular in systems with precisely this geometry. Considerable difficulty is entailed in distinguishing these spacecharge modes in finite plasmas bounded by dielectric surfaces from whistler-mode waves because both waves are slow and may have similar passbands. Furthermore, nearly any antenna in a finite plasma column has fringing fields that can excite the spacecharge wave and both modes require close antenna coupling to the plasma [25, p. 182]. In this case the axial component of the helical antenna's near (interior) field is the component oriented in the direction required to couple to these spacecharge wave modes whereas the radial component is oriented in the direction required to couple to the whistler mode.

4.0 CONCLUSIONS

A novel plasma-activated selenium source has been developed in the course of this contract which is significantly different than any other heretofore reported in the scientific literature of the field. It is microwave-excited, magnetically confined plasma source that is intended to operate under Electron Cyclotron Resonance (ECR) conditions at 2.455 GHz. This source is designed to excite and dissociate the vapor exiting from the aperture of an effusion cell. It combines the effusion cell vapor flux with a stream of hydrogen and/or helium gas at the resonance point. The source is mounted onto a water-cooled baffle inside a rotating disc reactor which incorporates both copper and indium effusion sources as well as EIES sensors and quartz crystal oscillators for monitoring and controlling the deposition process. The system is designed for load-lock operation and incorporates custom radiant heaters for substrate temperature control.

Experiments have been conducted which have successfully demonstrated the achievement of a stable microwave plasma discharge in helium gas. Measurements of the magnetic flux profile of the source suggest, however, that it does not currently operate in an ECR mode as intended. Since power absorption in an ECR plasma is more efficient than the non-resonant absorption which apparently pertains to the source as presently configured, it is anticipated that an improved magnetic confinement design would reduce the 150 watts minimum power currently needed to establish a stable discharge. Furthermore, the lower energy ion distribution characteristic of an ECR plasma is expected to be more beneficial for the minimization or elimination of ion bombardment damage when utilized for plasma assisted epitaxial growth processes. Preliminary calculations indicate that a supplementary solenoidal field coil can be readily incorporated into the design which would substantially increase the resonant excitation volume and enable operation in the desired ECR mode.

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ABSTRACT

A novel plasma-activated selenium source has been developed in the course of this contract which is significantly different than any other heretofore reported in the scientific literature of the field. It is microwave-excited, magnetically confined plasma sources that is intended to operate under Electron Cyclotron Resonance (ECR) conditions at 2.455 GHz. This source is designed to excite and dissociate the molecular vapor evaporating or subliming from a heated solid or liquid reservoir. It can combine an effusion cell vapor flux with a stream of hydrogen and/or helium gas, enabling the *in-situ* generation of hydrides for use in low pressure growth techniques where long mean free paths are desirable. Experiments were conducted to demonstrate a stable discharge within the source and measures identified to improve its operational characteristics. Application of this novel source is anticipated to enable a low temperature, safe process for the growth of high quality epitaxial compound semiconductor films. This reduction of epitaxial growth temperatures may enable the fabrication of novel photovoltaic devices which have heretofore been impossible due to the deleterious effects of interdiffusion at heterointerfaces resulting from the high temperatures required for growth of adequate quality material using conventional processes.

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