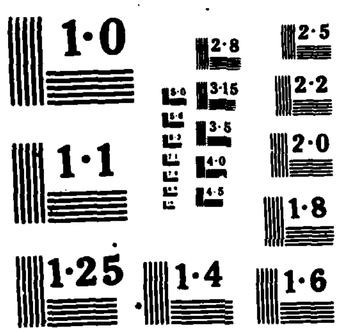
AD-A168 332 INTERFACIAL PROPERTIES OF GERMANIUM NITRIDE DIELECTRIC 1/1
LAYERS IN GERMANIUM(U) CALIFORNIA UNIV SAN DIEGO LA
JOLLA DEPT OF ELECTRICAL ENGINEE L G MEINERS 1986
UNCLASSIFIED N00014-84-K-0459 F/G 20/3 NL



NATIONAL BURFAU OF S
MICROCOPY RESOLU TEST

Final Report

Contract No. N00014-84-K-0459

Office of Naval Research (Mr. Max N. Yoder, Electronics Division

800 North Quincy Street, Arlington, Virginia 22217)

Interfacial Properties of Germanium Nitride Dielectric Layers in Germanium

Principal Investigator: L. G. Meiners

Department of Electrical Engineering and Computer Sciences, C-014

University of California, San Diego

La Jolla, California 92093

ABSTRACT

The enclosed report represents work performed at UCSD on Contract N00014-84-K-0459 entitled "Interfacial properties of germanium nitride dielectric layers on germanium" and provides a full account of the results obtained during the contract period: July 1, 1984 to June 30, 1985. The paper "Simple low-cost microwave plasma source has been published in the Review of Scientific Instruments, volume 57, page 164(1986). The remainder of this report describes the fabrication of the deposition system for the germanium nitride layers.

The first year's effort on this project has been primarily devoted to the design and construction of a low-pressure chemical vapor deposition system for growth of the germanium nitride layers. The gas manifold layout is shown schematically in Fig. 1, the reactor assembly is shown in Fig. 2, and the vacuum pumping assembly is shown in Fig. 3. The construction and operation of the microwave plasma source is described in the enclosed technical article which has been published. The remainder of the system is described below.

DESIGN AND FUNCTIONAL DESCRIPTION

Gas Manifold

Control and composition of the gases to be delivered to the reactor assembly is accomplished by the gas manifold. Plumbing fixtures are mounted on an aluminum plate held by a supporting frame. This assembly is then placed into a vented extension built on to the existing fumehood. Reactant gases are received into the cabinet from external supply, and have pressure relief safety valves in line prior to connection to the gas manifold. The manifold provides two independent, simultaneous supply lines to the reactor assembly.

The right hand side of the gas manifold schematic depicts the supply line of the gas inlet to the resistive heated furnace. N_2 and NH_3 gases are initially passed through a bubbler containing a liquid azeotrope of gallium and indium, with aluminum added, to scavenge oxygen and water. Total flow rate and composition of an ammonia-nitrogen mixture is accomplished by adjustment of the small metering valves while monitoring the flowmeters. A second nitrogen line is equipped with an ethylene bromide bubbler and purge capability to allow for <u>in-situ</u> etching.

The left side of the schematic depicts the supply line to the microwave resonant cavity, the gas flow rate and composition is determined by adjustment of small metering valves while monitoring the flowmeters. An additional nitrogen line is provided without flowmeter restriction to allow reactor assembly purge.

Reactor Assembly

The microwave resonant cavity is the excitation source for production of a plasma discharge in the flowing ammonia gas. The cavity is assembled from aluminum end plates supporting a cylindrical brass sheet which forms the sidewall. Circular brass plates attached to threaded pipe on each end form the end plates. The knurled knob in the figure is threaded to allow approximately one millimeter of displacement of the end plate per revolution. Moving both end plates allows choice of cavity length and relative distance of the magnetron position from the end plate. Not shown is a boron nitride tube placed, such that, the plasma will be isolated from contact with the quartz tube preventing any interaction.

Two gas inlet nozzles are provided where the quartz conduit widens and enters the reaction chamber. The resistance heated furnace is equipped for optional two-temperature zone operation. Separate, zone-centered thermocouples are provided for temperature control.

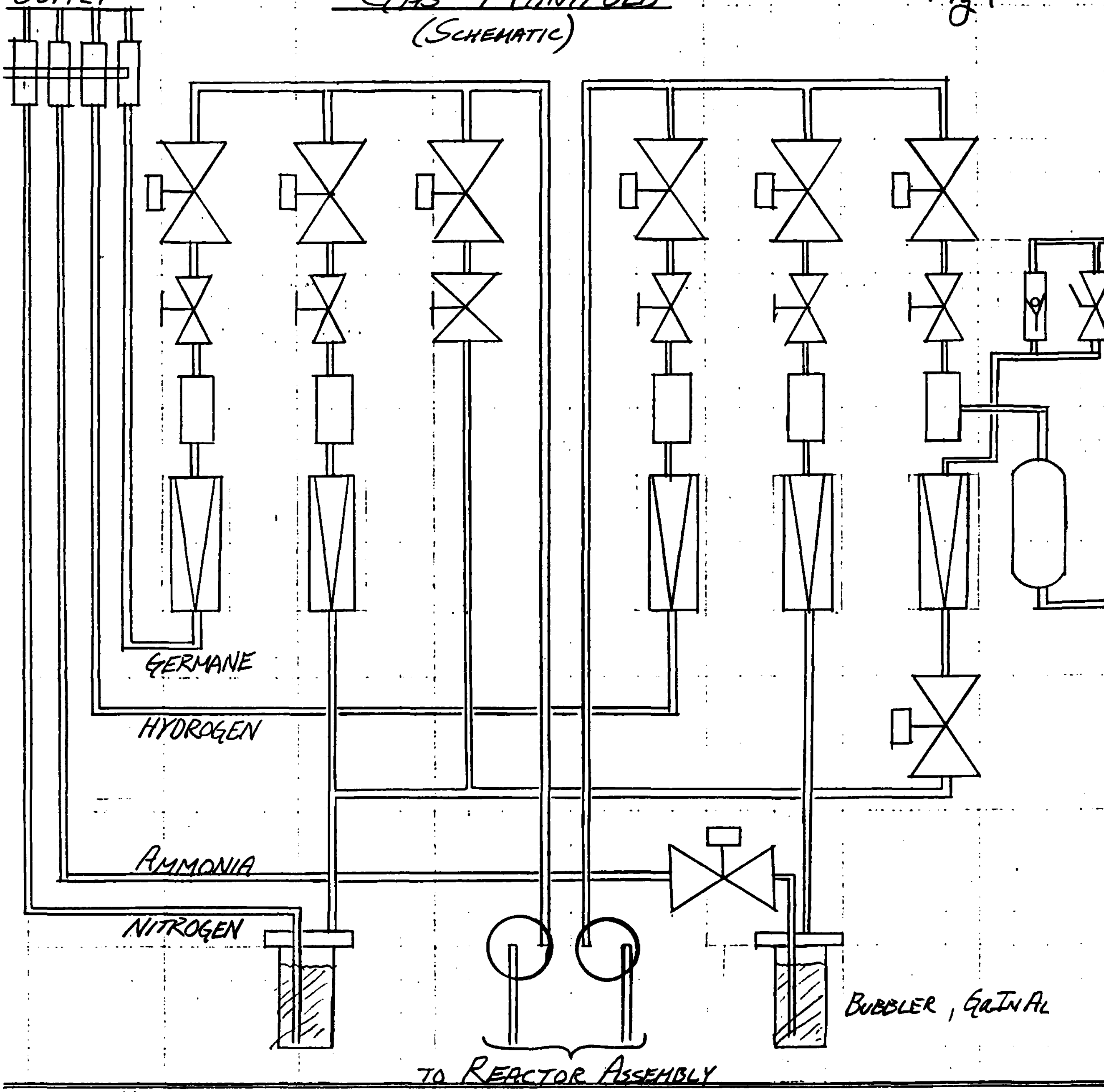
Substrate loading is accomplished after removal of an 90°C elbow connecting the reaction chamber to the vacuum system.

Vacuum System

Gases leaving the reaction chamber are conducted through a coaxial trap, vibration bellows and angle valve to the inlet port of the rotary vane pump. A small vacuum shunt is provided to prevent excess turbulance around the substrate as the angle valve opens. The pump is equipped with inert gas ballast for pumping condensable vapors. An external oil purifier, as recommended for vane pumps, was selected with attention to the ammonia service required.

GAS MANIFOLD
(SCHEMATIC)

Fig. 1



VENTED, PRESSURE RELIEF

PNEUMATIC, NC BELLOWS VALVE

MANUAL METERING VALVE, MEDIUM

MANUAL METERING VALVE, SMALL

MANUAL OPEN-CLOSED VALVE

CHECK VALVE, PRESSURE REL

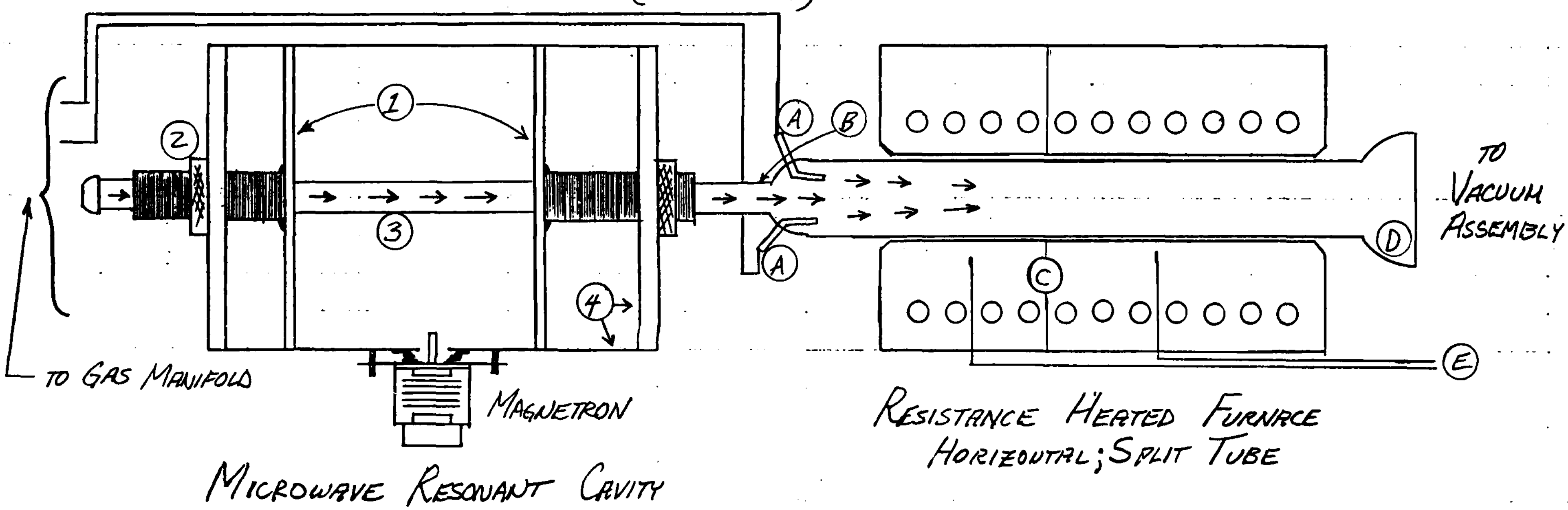
7 MICRON FILTER

250 ml S.S. CANISTER

FLOW METER

MILLIPORE WAFER-GUARD FIL

REACTOR ASSEMBLY
(SCHEMATIC)

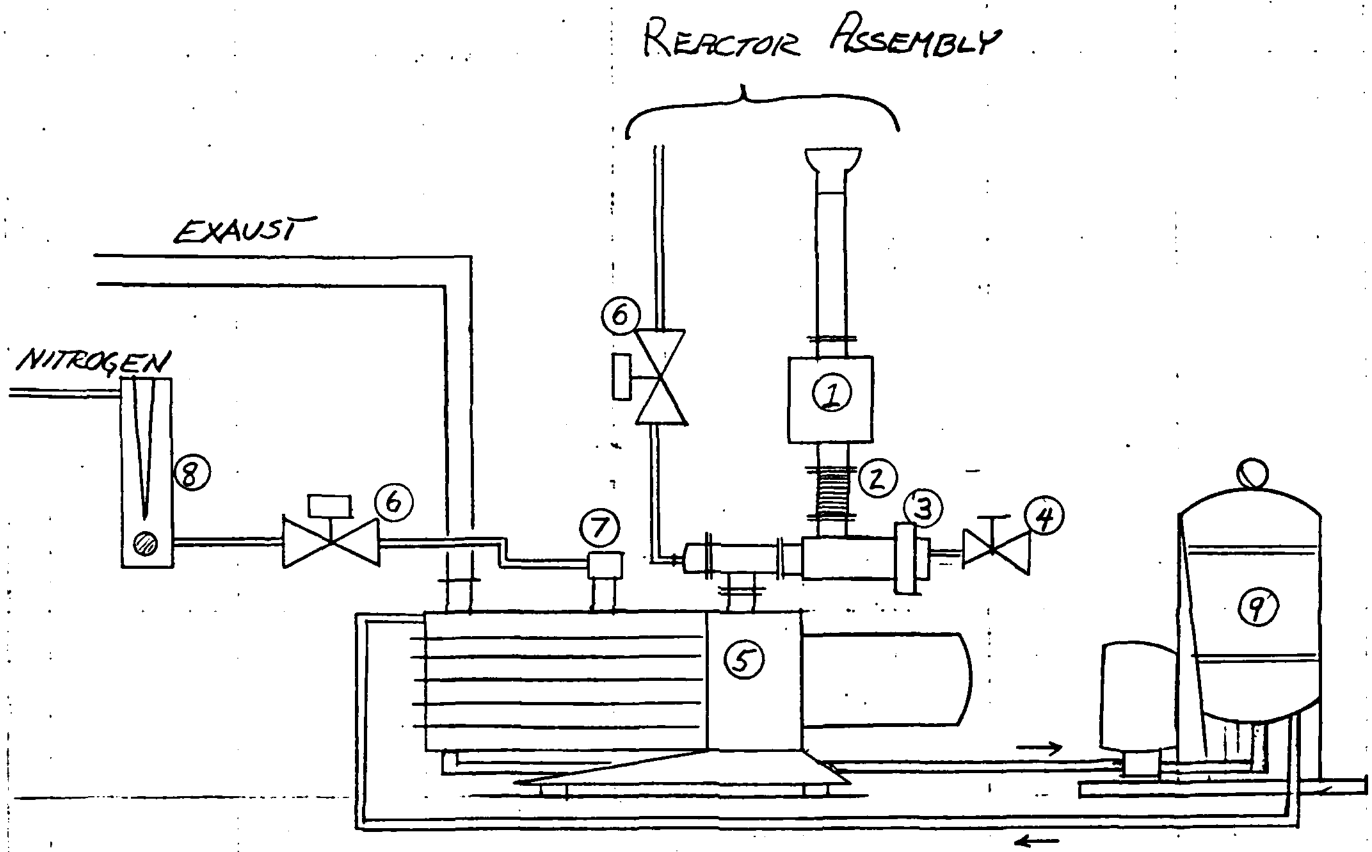


1. SLIDING SHORT
2. POSITIONING NUT (KNURLED KNOB)
3. QUARTZ TUBE (CONDUIT)
4. RIDGID OUTER FRAME
   ALUMINIUM END-PLATES
   BRASS SIDEWALL

A. GAS INLETS FROM GAS MANIFOLD
B. QUARTZ TUBE - PLASMA INLET
C. ZONE BOUNDARY - OPTIONAL DUAL TEMP
D. SAMPLE LOADING PORT - VACUUM DISCONNECTED
E. THERMOCOUPLES, ZONE CENTERED

Fig. 2

Vacuum Assembly
(Schematic)



1. Coaxial Trap
2. Bellows Section
3. Angle Valve
4. Manual, Metering Valve
5. Rotary Vane Pump
6. Pnuematic, N.C. Bellows Valve
7. Inert Gas Ballast Connection
8. Flowmeter with meter valve
9. Oil Purifier

Fig 3

Simple low-cost microwave plasma source

L. G. Meiners and D. B. Alford

Department of Electrical Engineering and Computer Sciences, University of California, San Diego, La Jolla, California 92093

(Received 26 August 1985; accepted for publication 4 October 1985)

A generator-cavity system is described which is capable of delivering 0–600 W of microwave power at 2.45 GHz. The power generating section has been constructed from components contained in a portable home microwave oven and the cavity was assembled from easily machinable pieces. The cw magnetron source was mounted directly on a cylindrical microwave cavity. The plasma was contained in an on-axis 20-mm o.d. quartz tube. Design tradeoffs and operating information are discussed.

INTRODUCTION

A type of apparatus being used with increasing frequency for chemical processing in the gaseous state depends in its operation on the production of excited gas particles in a radiofrequency (rf) plasma. The system that is used to provide the rf energy for the plasma varies greatly depending on the frequency of the rf source and the application at hand. The use of microwave sources offers advantages in that the plasmas can be maintained at higher gas pressures and flow velocities than are possible when operating at lower (0.1–30 MHz) frequencies. These advantages have in the past been partially offset by the fact that the design of microwave coupling cavities can be described as an empirical art at best and by the usually greater cost of the microwave power sources. Used medical diathermy units which operated at 2.45 GHz and produced about 100 W[1] were sometimes adapted for laboratory use, however, these are no longer readily available. An obvious choice of a power source for an experiment of this type would be a cw magnetron of the type that has been developed for use in microwave ovens. These devices typically operate at 2.450 GHz, have power outputs of 500 W or more, and usually are designed so as not to self-destruct when a large amount of power is reflected back into the magnetron tube. This paper describes the construction and operation of a microwave plasma source using one of these tubes.

I. MICROWAVE GENERATOR

One of the first considerations in the design was the development of a means whereby the power delivered to the microwave plasma could be continuously adjusted. A common method for the modulation of microwave power to a load is to operate the source at full power and shunt off the excess power to a dummy load. This can be accomplished in waveguide by combining a circulator or isolator with a variable attenuator, one or more dummy loads, and other miscellaneous waveguide components to monitor the output and reflected power so that the sections can be tuned properly.[2,3] Since the dummy load must be capable of accepting the full source power at zero output to the load, rather large air-cooled or water-cooled units are required to dissipate the 500–1500 W which they may receive. Although this is a possible way to construct a circuit for adjusting the power output to a plasma source, it did not really satisfy our objective of fabricating a simple, inexpensive unit which could be used for chemical processing. The power adjustment on a typical home microwave oven is achieved by simply turning off the ac line voltage to the magnetron power supply every few seconds to yield a lower average power output. This was not deemed an acceptable solution for the present problem because the plasma would be extinguished each time the magnetron was shut off. The magnetron requires a low-voltage–high-current ac filament supply and a high-voltage–low-current dc cathode supply. The microwave output power is directly related to the dc input power; however, this is usually not considered a good way to modulate the power output when the tube is used for communications purposes. When the cathode voltage is only slightly above the threshold value required for the maintenance of stable oscillations, the spectral purity of the output markedly degrades. This is unacceptable if the power is being radiated under the usual conditions which place strict limitations on the amount of out-of-band radiation which can be emitted. However, for the problem at hand it seemed as if this would present no large shortcoming since the microwave energy would be completely contained, and a small amount of broadband energy in the source would not affect the plasma production efficiency, which was our main concern in any event.

We purchased a small portable microwave oven capable of providing 600 W of microwave power at 2.45 GHz[4] plus an extra transformer for the magnetron power supply, a variable autotransformer (120 V, 10 A) to supply current for this transformer, an ac wattmeter (1000 W) to monitor the power input to the cathode dc supply, and miscellaneous hardware. Figure 1 illustrates the completed circuit. The essential modification is the means to supply constant current to the magnetron filament while allowing for an adjustable level of the dc voltage applied to the magnetron cathode.[5] Preliminary testing with the magnetron tube still in place in the microwave oven indicated that the tube output could be adjusted from zero to full power by adjusting the variac.

The next problem to be considered in the design was the method of coupling energy from the magnetron into the microwave cavity. Typically this is done by mounting the magnetron into a waveguide assembly with appropriate match-

wer out-
›bjective
l be used
a typical
ıg off the
very few
; was not
blem be-
the mag-
·voltage–
·low-cur-
wer is di-
ιs usually
:r output
es. When
old value
the spec-
unaccep-
ıal condi-
of out-of-
the prob-
ırge short-
ompletely
:rgy in the
efficiency,

en capable
GHz[4] plus
ply, a vari-
ent for this
the power
hardware.
essential
rent to the
ble level of
..[5] Prelimi-
lace in the
›uld be ad-
ariac.
ıgn was the
nto the mi-
ıg the mag-
ate match-

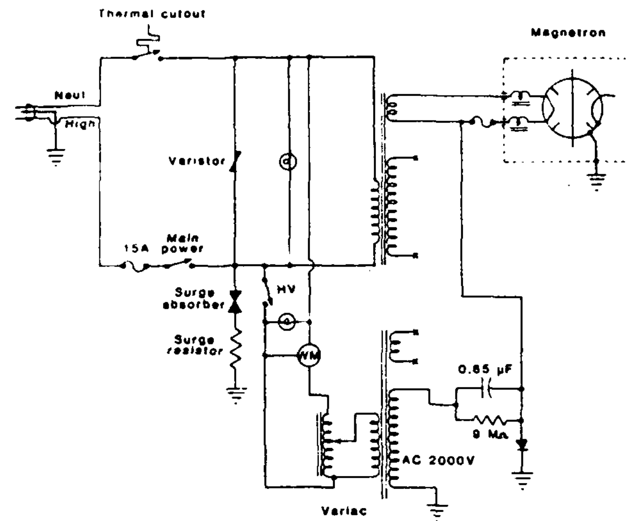


FIG. 1. Schematic diagram of microwave power source.

ing stubs and at the receiving end matching the waveguide to the cavity containing the plasma. Since it seemed inevitable that at least under some conditions (such as during tuning) the cavity would not be matched to the source, it would be necessary to introduce an isolator to absorb the power reflected from the cavity. Otherwise, the cavity would be extremely difficult to tune since one would have reflections not only from the cavity but also from the magnetron. Unfortunately high-power isolators are not only large and heavy (they contain large permanent magnets) but are also expensive. The prospect of adding this extra hardware to the system seemed at variance with our original goal. As we studied various cavity designs[6,7] we realized that there would be nothing to prevent us from mounting the output voltage probe of the magnetron directly into the cavity. We would give up some desirable features by doing this. The microwave power input to the cavity could not be monitored. One would only be able to measure the dc power input to the magnetron tube itself. The magnetron tube in a typical home microwave oven is usually protected from overheating by placing a thermal cutout switch close to the tube. Following a conservative approach, we mounted the switch immediately adjacent to the tube instead of several inches away as in the original oven design. Also the microwave power incident to and reflected from the cavity could no longer be used as a guide in obtaining proper tuning of the cavity. These restrictions seemed acceptable for our purposes. The dc power input to the magnetron would seemingly be a parameter which could be reset to give reproducible conditions in the plasma. For determination of the cavity adjustment which provided the optimum plasma tuning we could rely on photometric indications of the plasma brightness.

II. CAVITY

The cavity configuration was similar to that described by Asmussen *et al.*[2] and is shown in Fig. 2. The cavity walls were constructed from 1/16-in. brass sheet which was rolled to the appropriate diameter. The end pieces were made from 1/2-in. thick aluminum plate and the remaining plates and threaded pieces were constructed of brass. The magnetron was mounted in the wall of the cavity with a circular washer of metal braid around the cavity opening to prevent leakage of microwave radiation. The axial position of the voltage probe of the magnetron was chosen to allow maximum flexibility in cavity tuning. The cavity end plates could be adjusted over wide ranges to accommodate large variations in the loading presented by the plasma. The end-plate adjustment allows 1 mm of travel for each revolution of the knob and the position may be verified by direct observation through ventilation holes in the cavity walls. The magnetron tube was connected to the power supply via a pair of stranded #10 wires to reduce to an acceptable value the voltage drop produced by the approximately 14 A of filament current. Extra insulation for these leads was achieved by feeding them through 1/4-in. i.d. Tygon tubing. The wires were enclosed in a braided copper shield which eliminated the possibility of any rf radiation and provided a ground return path for current from the plate of the magnetron. The transitions at the

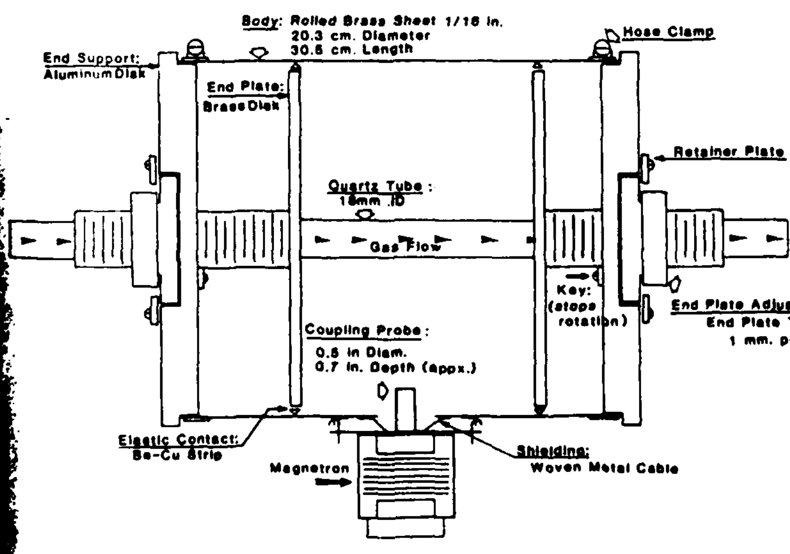


FIG. 2. Microwave cavity for plasma source.

metal enclosures were carefully insulated to accommodate the approximately 3-kV dc present on the filament leads. A small muffin fan was mounted directly to the magnetron to provide cooling and the connector for this was interlocked to the high-voltage power supply.

III. OPERATION

For our application the input ac power to the magnetron supply has been usually limited to less than 600 W. Under these conditions it has been possible to operate with no additional cooling of the cavity (it resides in a chemical fume hood in an approximate 0.5 m/s air flow). The cavity is used to provide excited nitrogen species generated from ammonia which are fed to an indirect plasma enhanced system for dielectric deposition. Typical deposition times are less than 15 min and the temperature rise of the cavity walls during that time interval is less than 20 °C. During the initial start up of the system optimum cavity tuning was achieved at lower power levels and the power increased gradually as the cavity was retuned to accommodate the change in the plasma conductivity.

The dimensions of the resonant cavity were chosen such that the radial distance between the cavity side wall and the plasma column would accommodate only a single half-wavelength. Thus, only one resonance mode is allowed for each tuning length selected. Start up was accomplished by setting the tuning length to 9.5 cm, longer than that reported by Asmussen *et al.*[2] for the first two transverse electric modes TE_{011} and TE_{111} for a cavity of similiar dimensions.

Optimizing the tuning length consisted of incrementally reducing the cavity length with the pressure and flow rate appropriate to our application, held constant. Initially the magnetron heated rapidly, even with a plasma present, due to the large amount of reflected power being returned to the magnetron. Thermal cutout would result in less than 10 min if operated at full power under this condition. As the tuning length was reduced to a final setting of approximately 7 cm the cavity walls heated during operation; the magnetron remained cool. Optimal length was detected by direct observation of plasma brightness, length of plasma extension beyond the cavity, and increased meter readings on the power supply.

Maximizing the plasma extension at the outlet, with either no change or reduction at the inlet was achieved by parallel movement of the cavity end plates in the upstream direction, locating the probe approximately 2.5 cm from the end plate associated with the outlet.

Pressures in the neighborhood of 1.0 Torr required the cavity length set at approximately 7.1 cm. This corresponds to a TE^{*}_{111} hybrid resonance mode described by Asmussen *et al.*[2] A plasma can be achieved over a rather large range of pressures (10–0.01 Torr) and flow rates. The system appears to be reliable and the results reproducible. The total cost of the electronic ports and other materials used for the construction of the system came to less than $900.

ACKNOWLEDGMENTS

We thank D. E. Dixon for assembly of the magnetron power supply. This work was supported by a grant from the Office of Naval Research.

[1] J.R. Brandenberger, Rev. Sci. Instrum. 42, 1537 (1971).
[2] J. Asmussen, R. Mallavarpu, J.R. Hamann, and H.C. Park, IEEE Proc. 62, 109 (1974).
[3] H.S. Ho and W.P. Edwards, Radio Sci. 12 (65), 131 (1977).
[4] NE-6650A, Panasonic Company, One Panasonic Way, Secaucus, NJ 07094.
[5] S. Chilukuri and W. L. Lichten, Rev. Sci. Instrum. 50, 256, 1979.
[6] F. C. Fehsenfeld, K. M. Evenson, and H. P. Broida, Rev. Sci. Instrum. 36, 294 (1965).
[7] A. Ding, J. Karlau, and J. Weise, Rev. Sci. Instrum. 48, 1002 (1977).

END
DTIC
7-86