Advanced Materials Delivery Successes in CVD Processing

by

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ABSTRACT

As silicon device geometrics become smaller and aspect ratios larger, processing technology is moving from PVD into the area of CVD and OMCVD. Many new source materials are in the research and development stage, and have placed challenging demands on materials delivery technology. This paper will describe the many successes achieved with various delivery methods including thermal, bubblers, pressure-based and Direct Liquid Injection.

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Introduction

In reviewing the recently published *1994 National Technology Roadmap for Semiconductors* and technical journal articles, it becomes apparent that a combination of process problems related to Barrier (Seed) Layers — Metal Interconnects — and Interlevel Dielectrics will pose a real challenge within the next five years. It is predicted that the number of metal levels for DRAMS will remain at 3, while for logic devices it will increase to as many as 7-8. The aspect ratio for contacts/vias will go from 3/1 at 0.25 μm to 6/1 for 0.07 μm for logic devices, and from 4/1 to 10/1 for DRAMS of the same feature size as shown in Figure 1.

Increases in processor speed and reduction in power dissipation require reduced resistance and capacitance in interconnects which translate to higher conductivity wiring and insulators with lower dielectric constants. When improved reliability and tighter CD control in etch processes are added to the puzzle, it becomes clear that device manufacturers' partnering only with a tool (equipment) supplier may not be able to succeed. However, a "teaming" effort between the device manufacturer, tool supplier, university researchers, materials, and materials delivery suppliers will have a much better chance of success.

If one looks at a typical device cross section, as shown in Figure 2, we can see the areas where a barrier is deposited, followed by metal deposition, planarization (CMP) or etch, and interlevel (or intermetal) dielectric deposition. At 0.35 μm geometrics, it is a coin flip to choose whether collimated PVD or a CVD process is best from a cost-of-ownership standpoint. However, upon entering the 0.25 μm world and beyond, it appears that CVD technology will win over PVD.

From a materials delivery standpoint, the requirements are totally different. In PVD, one uses simple inert gases such as argon, while CVD requires a wide variety of source materials ranging from simple gases to very low vapor pressure, temperature decomposition sensitive liquid precursors that cannot be delivered reliably to the processing tool with standard thermal mass flow controllers.

Some of the precursors represent a demanding challenge for the materials delivery equipment suppliers. We will discuss the choices of delivery techniques and various processes for barriers — metals — dielectrics where more advanced delivery methods have proven successful and we look forward to the future for more demanding delivery challenges.

Materials Delivery Techniques

About 90% of the present requirements for the controlled delivery of gas phase source materials are accomplished using thermal mass flow controllers while the remaining 10% require alternative methods.

Thermal Gas Phase Mass Flow Controllers

Shown in Figure 3 is a schematic representation of the traditional thermal MFC. The vapor inlet — usually at pressures above atmospheric, but in some cases as low as 100 Torr — is split in a laminar flow fashion between a heated sensor tube and a bypass and then fed to a control valve. Sensor tube elements have different structures, depending on the manufacturer, but in general allow the inlet vapor to be heated on the upstream end and transfer some of that heat to the downstream end as a function of gas flow rate. This technique is a thermal conduction one, hence the true value of the mass flow rate is dependent on the thermal properties of the particular source material.

The thermal flow characteristics of the sensor tubes were studied by L. D. Hinkle and C. F. Mariano in 1991.[1] This work led to a better understanding of these instruments. In addition, since 1980, much engineering development effort has taken place to improve the long-term performance of thermal MFCs. Extensive reliability, corrosive gas, and surrogate gas testing has been carried out under the auspices of SEMATECH and its MFC Working Group. MFCs no longer carry the stigma of being a poor reliability key component in processing systems. Design rules are nominally 7-year MTBF, calculations in development show 15-year MTBF, and actual field return data show 25-year MTBF for elastomer- sealed units and 15- to 18-year for all-metal MFCs.

Standard thermal MFCs are routinely used when the inlet vapor pressure is > 100 Torr and the operating temperatures are < 50°C for mass flow rates from 0.1 sccm to 200 slm. Special thermal MFCs have been developed for lower inlet pressures down to 5 Torr and for temperatures up to 200°C (with remote electronics). They are being used for the delivery of heated liquid precursors, such as Ti Cl_4, TEOS, TMB, TMP, etc. There are however some important temperature gradient considerations in order to avoid condensation in the delivery lines between the MFC and system.

Thermal Liquid Phase Mass Flow Controllers

The operating temperature of the flow sensor tube in a thermal MFC is normally on the order of 100°C, although the temperature can be reduced to about 60°C for liquid flow applications. Unfortunately, some precursors of interest have serious decomposition problems at these temperatures even in the short periods of time that the sources are in that temperature environment.

There are commercially available LFM (liquid flow meters) that operate at 1 to 5°C above ambient, and take advantage of a Peltier cooling effect to measure flow rate. Hence, these are used for flow metering, then deliver the liquid to some kind of vaporizer to create the source vapor. Discussions with several process development people worldwide have indicated dissatisfaction with LFM/vaporizer systems for reasons not detailed. We have not had the opportunity to test liquid sources first-hand or in a collaborative effort with a chemical supplier, hence cannot comment on the nature of the performance characteristics or related problems.

Bubbler (Saturated Carrier) Systems

A typical bubbler or "saturated carrier" delivery system incorporates a thermal MFC to flow a carrier gas into a carefully temperature controlled container or ampule of the liquid source material. The carrier entrains saturated liquid and carries it to the system. Figure 4 shows such a system including a pressure control loop on the downstream side of the bubbler to enhance the repeatability of the material delivery. Several excellent papers have been written on the dynamics of bubblers and improvements in performance by using the pressure control loop.[2,3] Also, the pressure controllers recently introduced offer small size and excellent performance.

Due to the temperature sensitivity of many of the precursors and the problem in bubblers related to chemistry changes with heating as a function of time, this technique will not lend itself to many of the future low vapor pressure sources. A specific example will be given in the section on Ti N barrier deposition.

Pressure-Based Vapor Delivery

This technique takes advantage of precision pressure measurement and control technology combined with the relative simplicity and predictability of flow in a viscous choked flow regime. Flow through a viscous choked flow orifice depends only on the pressure upstream of the orifice, and not a combination of pressure drop and average line pressure as in the laminar flow regime (Hagen-Poiseuille equation). The flow will be viscous choked provided the pressure upstream is at least twice the downstream pressure. Figure 5 shows a schematic representation of such a system. Examples of the effective use of this method will be given in sections on barriers — metal interconnects — dielectrics.[4]

One area of wide applicability has been in OMVPE wherein metal alkyl sources — Al, In, Ga, Zn, etc. are delivered at low flow rates into systems at operating pressures of 10^{-3} to 10^{-4} Torr (0.1 to 0.01 P).

It should be noted that careful temperature control of interconnecting lines, with a positive going gradient from the source to the system, is a necessity in order to avoid source condensation. Also, that the technique is limited to process operating pressures up to about 5 Torr.

Direct Liquid Injection

In the mid 80's, we began a search for a delivery scheme that could deliver a vapor at flow rates up to 10 cc/min. of liquid (H_20 equivalent) from an unheated liquid source in order not to introduce temperature degradation of the liquid, could work with liquid sources having very low vapor pressures, did not require heated lines with progressive temperature gradients, had high precision and repeatability, was functionally safe, and could even deliver pre-mixed "cocktails" without changing stoichiometry. A review of the literature, commercially available products as well as patent searches, indicated that nebulizers delivered aerosols into the process, HPLC pumps suffered from pulse problems (even though they are advertised as pulse-free), microsyringe pumps[5] had not advanced beyond the laboratory stage, and fast pulse valves had droplet problems.

Several years of intensive development resulted in a two-stage stepper motor driven, piston type precision metering pump with no elastomeric parts delivering liquids in the range of 0.006 to 10.0 cc/min in a truly pulse-free manner to a flash vaporizer with operating temperature up to 250°C as shown in Figure 6.*

Now that we have discussed the various flow metering and control techniques at the disposal of the process equipment engineer, we shall consider some of the challenging process deposition steps in silicon device processing.

Diffusion Barriers

The diffusion barrier, sometimes referred to as a seed or glue layer (adhesion properties), of current high interest is titanium nitride. We shall discuss three processes that are being actively pursued.

Process 1 — Ti Cl_4 + NH_3

This process requires a Ti Cl_4 flow rate of 20 sccm from a source heated to 70°C (vapor pressure ≈ 20 Torr) using a thermal MFC with remote electronics, the MFC temperature controlled at 80°C while the NH_3 flow rate is 500 sccm using a standard thermal MFC[6] (having neoprene seals). Pressure-based MFCs have been very successfully used in this application and offer a viable alternative to thermal MFCs.

*U.S. Patent No. 5,361,800

This process requires heating of the process chamber as well as the pressure transducer and throttle valve to 150°C to preclude buildup of ammonium chloride from the $TiCl_4/NH_3$ reaction.

Process 2 — Titanium Iodide/Hydrogen

A process that appears to have promise for deposition of titanium uses titanium tetraiodide + H_2 in a plasma CVD environment and with NH_3 in a thermal CVD process for Ti N deposition.

The challenge with TiI_4 is that the source is a solid material with a very low vapor pressure, e.g. 2.3 Torr at 150°C. Preliminary results using a very crude delivery system were very good for both Ti and Ti N. Further work is underway on a production delivery technique for such solid sources.

Process 3 — TDMAT or TDEAT

"Chlorine-free titanium nitride films can be produced at relatively low temperatures (less than 400°C) by CVD using metal-organic compounds: CVD Ti N from two of these compounds, TDMAT and TDEAT, is currently being developed for commercial application."[7,8]

Figure 7 shows the vapor pressure curves for $TiCl_4$ and the metal organics TDMAT and TDEAT. We will show that the bubbler method can be used, but the resulting flow and deposition rates are too low for anything other than R & D activities (Figure 8).

However, by using Direct Liquid Injection (DLI) it can be shown that very reasonable TDEAT flow rates and Ti N deposition rates can be routinely achieved (Figure 9).

Figure 10 shows a cross-sectional SEM of Ti N barrier deposition using TDEAT from research done at SUNY-Albany, NY in Dr. A. E. Kaloyeros' Device Physics group.

In summary for TiN deposition — bubblers can be used for R & D with TDMAT/TDEAT resulting in very low precursor flow and Ti N deposition rates. The DLI technique has been proven to be a good solution and can produce production worthy deposition rates.

Metal Interconnects

In the quest to reduce power requirements, copper has been widely investigated. It offers higher conductivity, lower resistivity and less electromigration than aluminum.

Beyond copper alone, a combination of aluminum/0.5% at. wt. copper is being actively pursued. An excellent review paper was published in 1993 by A. E. Kaloyeros and M. A. Fury.[9]

Copper I/Copper II CVD

The materials receiving most attention have been CuI(tmvs), better known commercially as Cupraselect™ (Schumacher) and the β-diketonate solid source material CuII. Figure 11 shows the published vapor pressure curves for these source materials.

Both CuI or Cupraselect, a liquid source, and CuII, a solid dissolved in solvent with a H_2 carrier, have been very successfully delivered into PACVD reactors* and now are producing copper deposition rates in excess of 1000 Å/min for 4:1 aspect ratio vias. It is felt that deposition rates above 2000 Å/min can be achieved in the near future. A cross-sectional SEM of CuI deposition is shown in Figure 12.

Aluminum CVD

CVD deposition of aluminum has been a goal of many process engineers since the 1980's. There are presently two source materials that are actively being pursued — DMEAA and DMAH.

DMEAA — Excellent work was reported by W. Gladfelter and co-workers of University of Minnesota on use of this precursor.[10] We have been able to deliver 5 sccm of a vapor into a commercial reactor operating at 200 mTorr using the pressure-based delivery technique. The computer model is shown in Figure 13, and the cross-sectional SEM of the deposition results is shown in Figure 14.

We have done some preliminary work to deliver DMEAA using the DLI system. This work will renew with much more intensity within the next two months (March 1995).

DMAH — From the open technical literature it would appear that there is considerable R & D activity in the Asian world on DMAH as a viable aluminum source material. A recent paper by Kondoh, et al from Kawasaki Steel, discusses Al-Cu CVD for interconnect formation.[11]

We have confidence that the DLI technique can be used effectively in the delivery of DMAH. This material has a vapor pressure of 1.7 Torr at room temperature, however has a tendency to decompose when the temperature is elevated to produce a higher vapor pressure and flow rates in order to effectively utilize the pressure-based flow method.

*U.S. Patent No. 5,376,409

Interlevel Dielectrics (ILD)

A workshop held in August '94 on ILD's clearly showed that Si O_2 with a dielectric constant of about 4 would not be the material of choice beyond 0.35 μm geometrics. Hence, there is an active effort to find an ILD with as low a dielectric constant as possible (less than 2.5), is structurally solid (i.e. not soft), can withstand processing temperatures up to 400°C, has low H_2O adsorption/desorption properties and can be deposited either by spin-on or CVD methods. Many polymer like materials were discussed at the August conference. We are today, however, still in the world of Si O_2, with many facilities still using silane chemistry, while there has been a serious move toward TEOS as the silicon source and ozone as the oxygen catalyst.

TEOS/Ozone — Si O_2

Several of the mentioned delivery methods have been used to volatilize and deliver TEOS into processing systems operating at pressures from slightly below atmospheric (APCVD) to LPCVD horizontal furnaces at 250 mTorr and single wafer tools at 10-60 Torr.

For APCVD systems (200 Torr to atmosphere), the method of choice has been bubblers. For LPCVD systems, a combination of bubblers and special thermal MFCs (low pressure drop) have been used, but by far the pressure-based technique has been very popular and very successful, since TEOS has a v.p. = 8 Torr @ 50°C and 24 Torr @ 70°C. In the area of single wafer tools at 10-60 Torr operating pressures, bubblers have and are being used, but are excellent candidates for DLI techniques.

Recent results on a production CVD 5000 tool (Applied Materials) using the DLI technique[12] at system pressures of 30 Torr and 280 Torr with TEOS/Ozone flow rates of 80/160 sccm and 20/320 sccm show excellent conformality at aspect ratios of 10 to 1 as shown in Figure 15. In addition to helping to optimize the deposition process for high quality films with excellent step coverage, high deposition rates, and good uniformity, the DLI system has helped to quantify the variables of the system.

To this system will be added a new infrared absorption instrument[13] that we have developed in collaboration with IBM to accurately monitor the partial pressure of ozone to achieve process characterization and repeatability.

Low k Dielectrics

A good overview of organic polymers for ILD applications has been given recently in *Semiconductor International* magazine by P. Singer.[14]

The delivery method will be dependent on the materials characteristics and the method of deposition — spin-on or CVD. We are about to enter into a vertically-integrated

research and development program to develop and commercialize molecularly engineered and structurally reinforced low dielectric constant ($2.0 \leq \kappa \leq 2.5$) materials and associated spin-on and/or vapor phase plasma polymerization processes for incorporation in the 0.25 μm device technology and beyond. This program is expected to begin in the second quarter of 1995.

High κ Dielectrics (Gate Oxides)

On the other end of the dielectric constant spectrum are the high κ dielectrics needed for gate stack capacitors in DRAMS. Figure 16 shows a chart from a paper by Matthews and Fazan of "Micron Semiconductor" presented at the 1994 Spring MRS Meeting. This chart indicates a transition over time from the traditional O-N-O process to the use of higher dielectric constant materials like $Ta_2 O_5$ and further on BST (Barium Strontium Titanate) to achieve capacitance values up to 50fF/μm^2.

Tantalum oxide commonly uses a precursor such as tantalum pentaethoxide, a jelly-like material at room temperature (melting point 21°C) which needs to be heated a few °C above ambient in order to liquify. The DLI method can certainly be used for delivering this material.

BST or Barium Strontium Titanate is an example of materials that have thus far required three separate DLI systems for delivery and good ratio control. Vaporizer temperatures in the 225-250°C range are required for complete vaporization. There is considerable effort by the chemical suppliers to produce a single "cocktail" mix of the three materials dissolved in an appropriate solvent.[15] When this mix is available, a single DLI system should suffice and dramatically reduce the component cost of the materials delivery systems.

Other Materials Possibilities

There are two other interesting and challenging process areas that will need attention within the foreseeable future. Ferroelectrics such as the PZT family for FRAMS and sources for Si C deposition such as MTS (methyl trichlorosilane) since Si C shows up in the device road maps 5-7 years out.

Summary

This paper has discussed the various materials delivery techniques available to process engineers, pointing out the advantages and disadvantages. Specific processes for barriers — interconnects — dielectrics critical to processing beyond 0.35 μm geometrics have been detailed with examples of successes using advanced delivery techniques. We have also mentioned briefly some challenges for the future.

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References

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[13] *IR-5 Infrared Absorption Monitor*, MKS Instruments, Inc., Andover, MA

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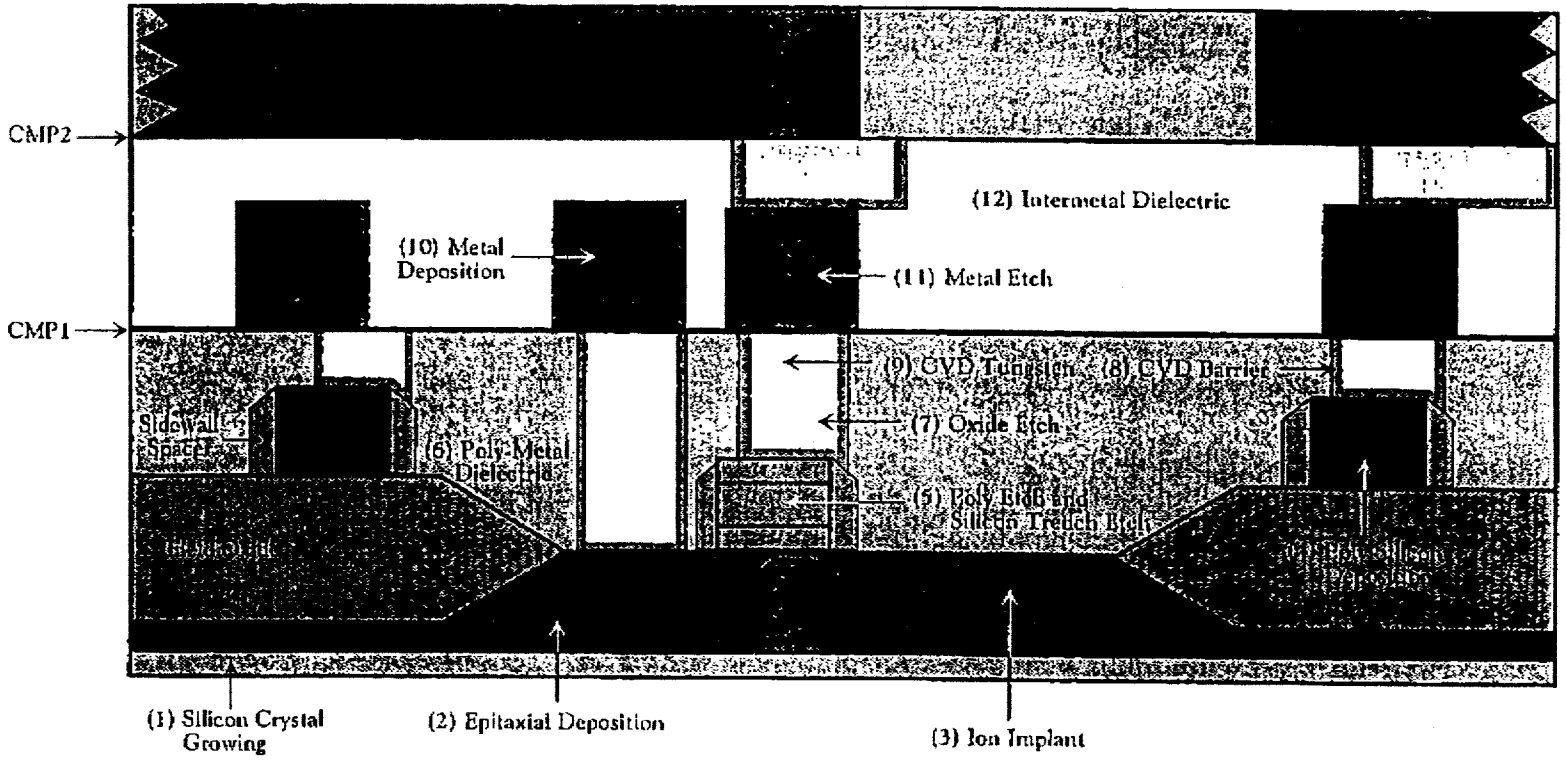
Figure Captions

	1995 0.35 μm	1998 0.25 μm	2001 0.18 μm	2004 0.13 μm	2007 0.10 μm	2010 0.07 μm
No. of metal levels DRAM Logic: Microprocessor	2 4-5	2-3 5	3 5-6	3 6	3 6-7	3 7-8
Contact/Via aspect ratio Logic DRAM	2.5:1 4.5:1	3:1 5.5:1	3.5:1 6.3:1	4.2:1 7.5:1	5.2:1 9:1	6.2:1 10.5:1

Fig. 1



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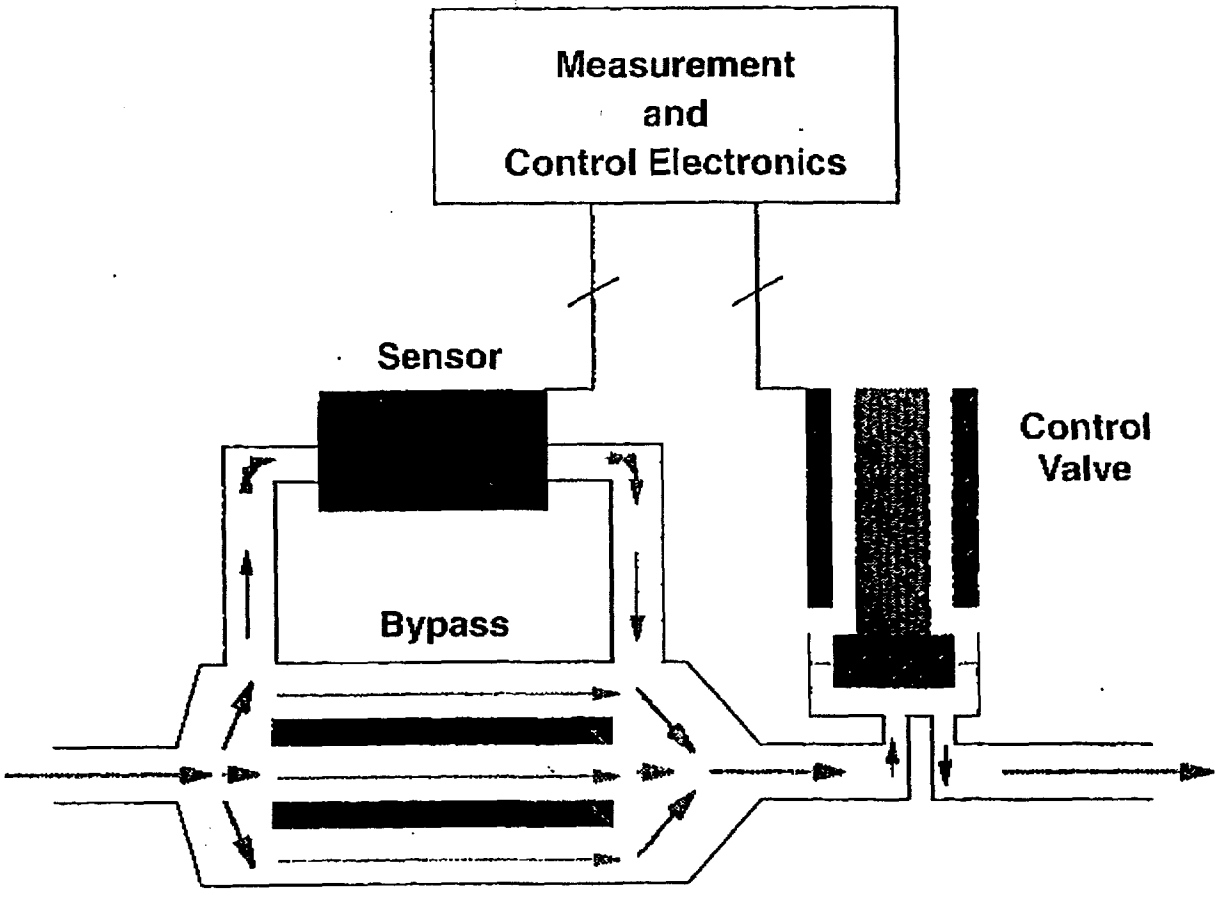

Measurement
and
Control Electronics
Sensor
Control
Valve
Bypass

Fig. 3

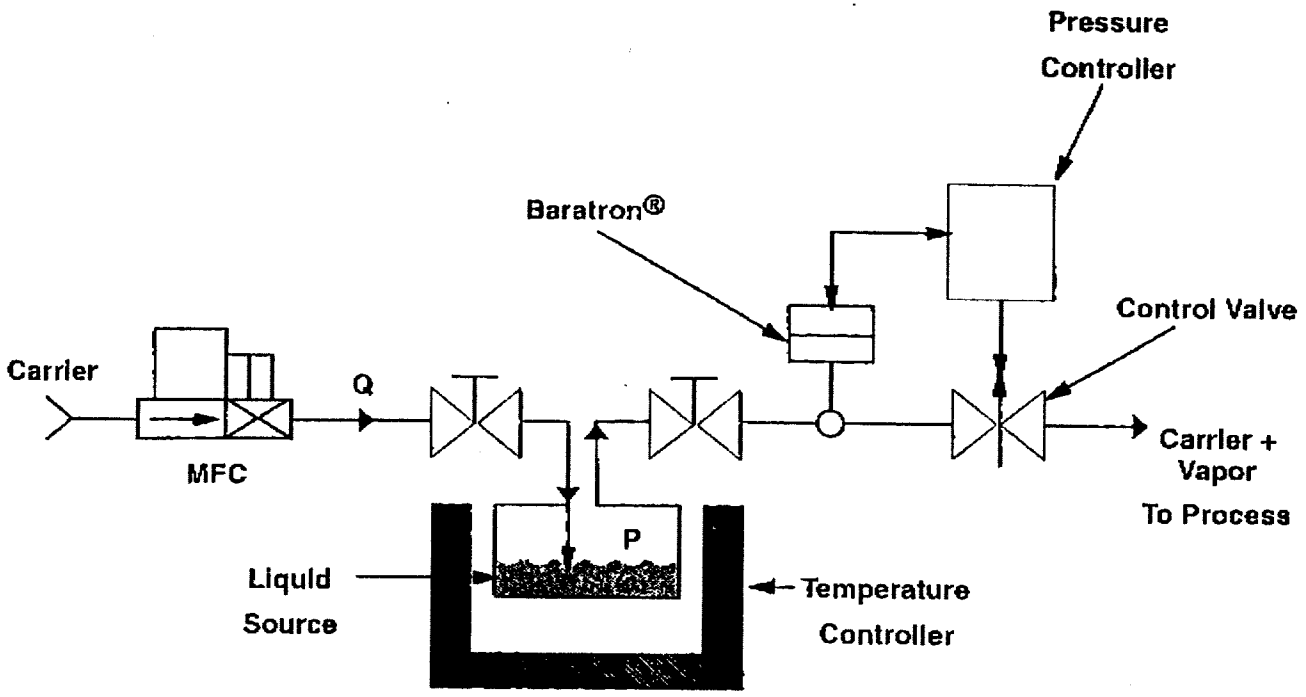
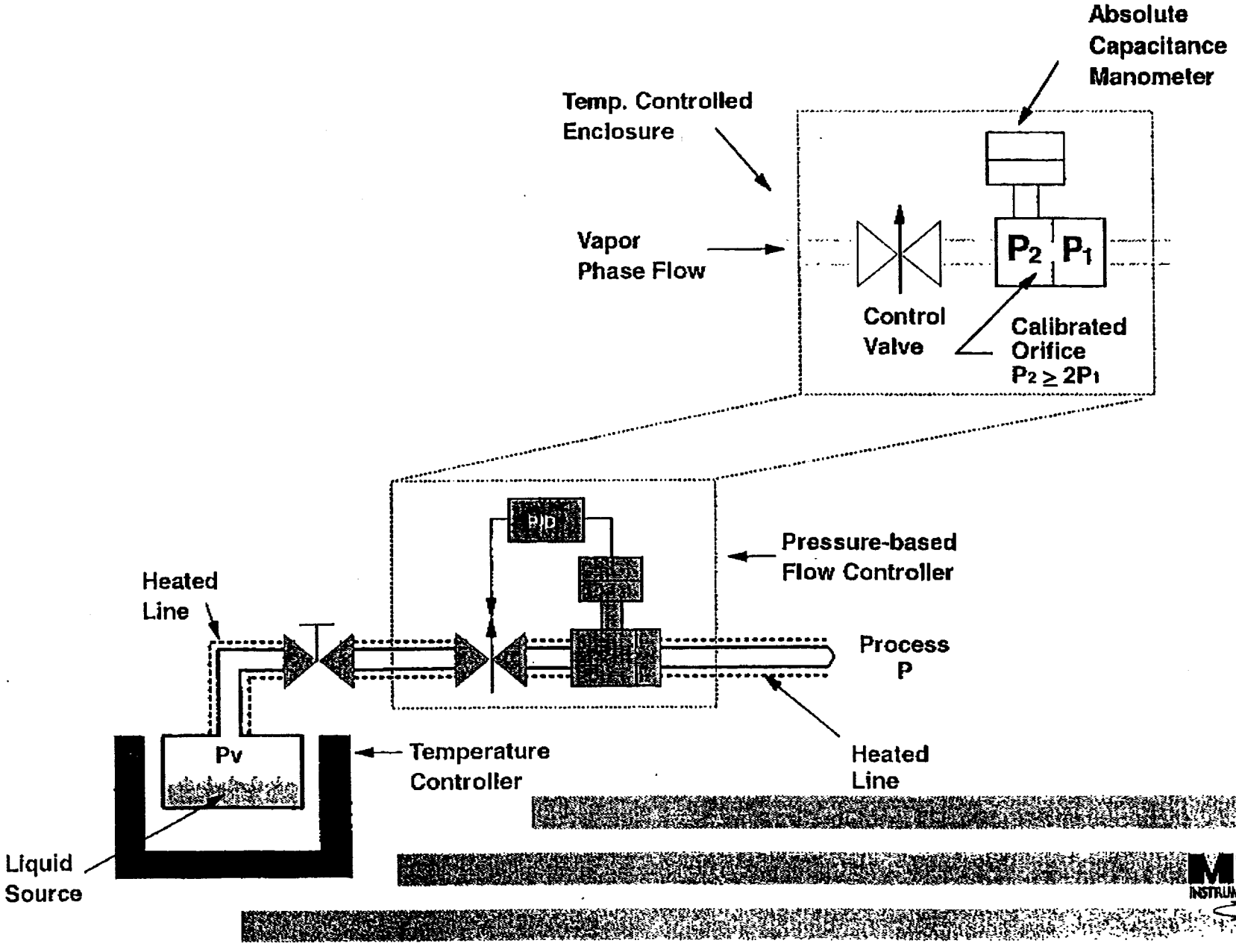
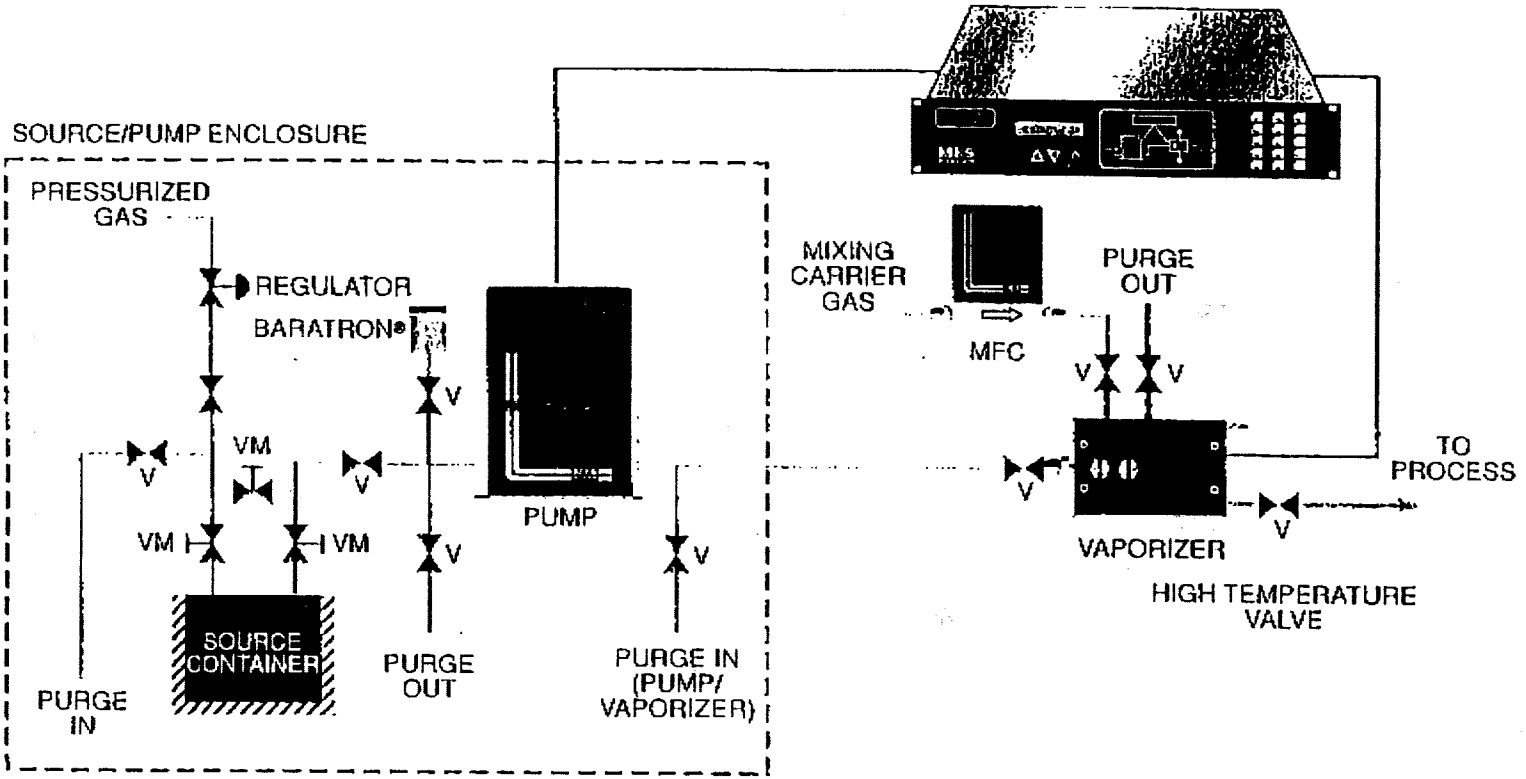

Pressure
Controller
Baratron®
Control Valve
Carrier
Q
MFC
Carrier +
Vapor
To Process
P
Liquid
Source
Temperature
Controller

Fig. 4


Absolute
Capacitance
Manometer
Temp. Controlled
Enclosure
Vapor
Phase Flow
P2
P1
Control
Valve
Calibrated
Orifice
P2 ≥ 2P1
PID
Pressure-based
Flow Controller
Heated
Line
Process
P
Pv
Temperature
Controller
Heated
Line
Liquid
Source


SOURCE/PUMP ENCLOSURE
PRESSURIZED GAS
REGULATOR
BARATRON®
V
VM
V
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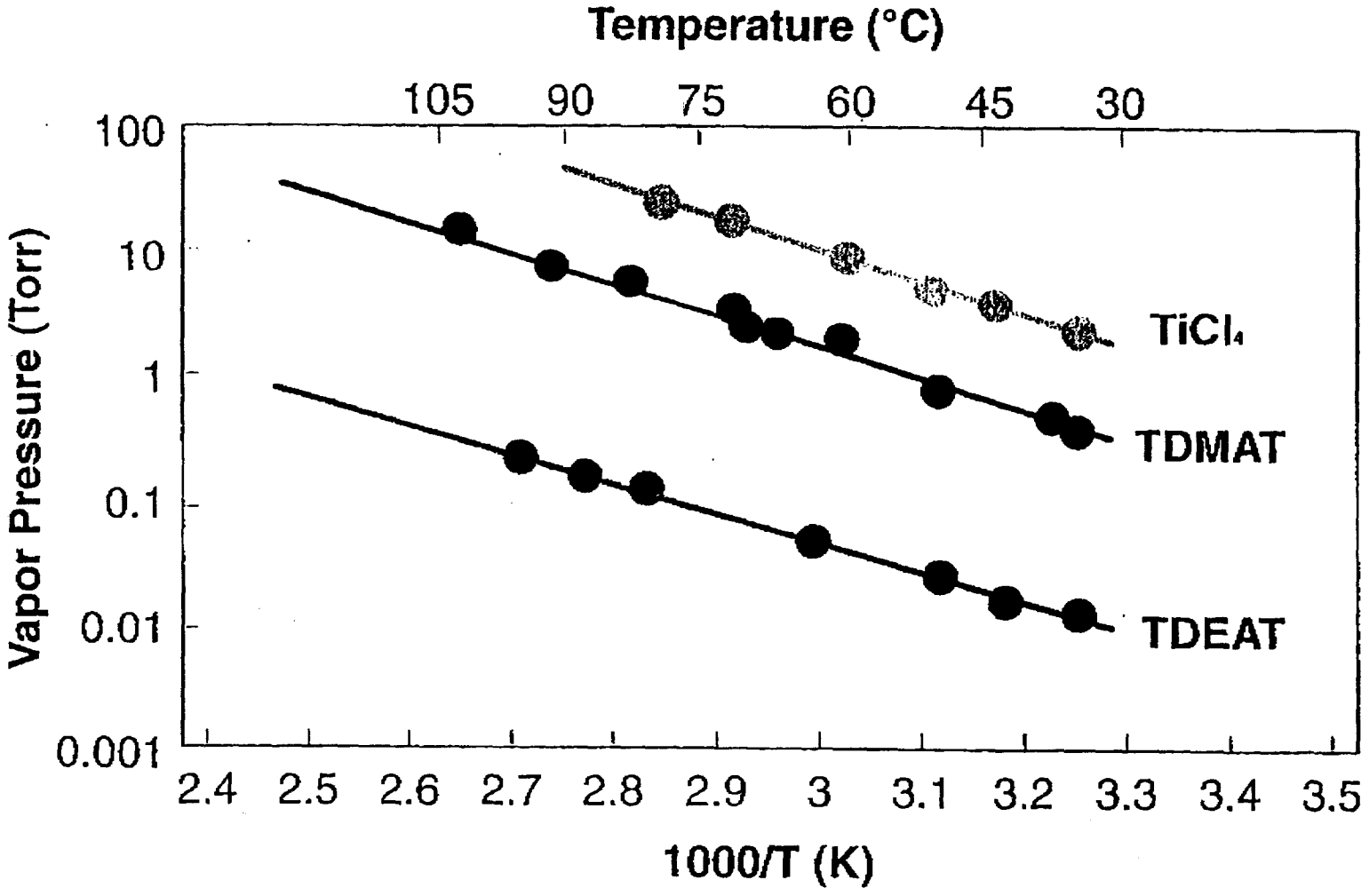
VM
V
V
SOURCE CONTAINER
PUMP
PURGE IN
PURGE OUT
PURGE IN (PUMP/ VAPORIZER)
MIXING CARRIER GAS
MFC
PURGE OUT
V
V
V
V
VAPORIZER
TO PROCESS
HIGH TEMPERATURE VALVE



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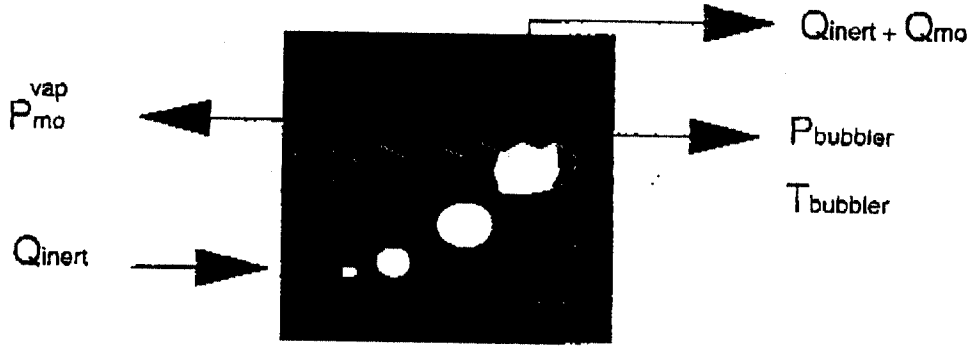
Bubbler Delivery Systems for TDMAT/TDEAT



*$T_{bubbler}$ is limited by thermal decomposition of the metal-organic compound.

TDMAT/TDEAT Example:

$T_{bubbler} = 60°C$
$P_{bubbler} = 50Torr$
$Q_{inert} = 200\ sccm$

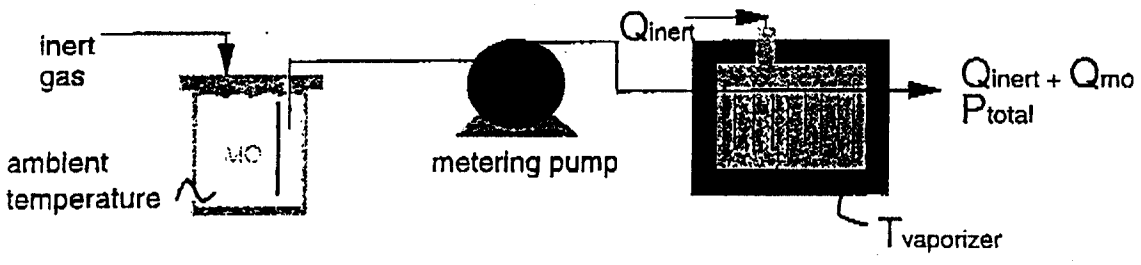
$P_{TDMAT}^{vap} = 1\ Torr$
$P_{TDEAT}^{vap} = .02\ Torr$

$$Q_{mo} = \frac{P_{mo}^{vap} Q_{inert}}{P_{bubbler} - P_{mo}^{vap}} \quad P_{bubbler} >> P_{mo}^{vap} \quad \simeq \quad \frac{P_{mo}^{vap} Q_{inert}}{P_{bubbler}}$$

@ 4 gm/cm^3 TiN, 8" wafers & 20% efficiency

$Q_{TDMAT} = 4$ sccm ~175 Å/min
$Q_{TDEAT} = 0.08$ sccm ~3.5 Å/min

Direct Liquid Volatilization for TDEAT



"Point of Use" Volatilization:

$$T_{vaporizer} > T_{bubbler}$$
$$P_{vaporizer} < P_{bubbler}$$
$$Q_{inert,\ vaporizer} > Q_{inert,\ bubbler}$$

Governing Equation:

$$\frac{Q_{mo}}{Q_{inert} + Q_{mo}} \bullet P_{total} \leq P_{mo}^{vap} \quad \text{or, approximately,} \quad \frac{Q_{mo}}{Q_{inert}} \bullet P_{total} \leq P_{mo}^{vap}$$

So, for

$T_{vaporizer}$ > 90°C, Pvap ~ .2 Torr
Q_{inert} = 10000 sccm = 10 slm
$P_{vaporizer}$ = 40 Torr
Qmo ≤ 50 sccm TDEAT ⟷ 2000Å/min

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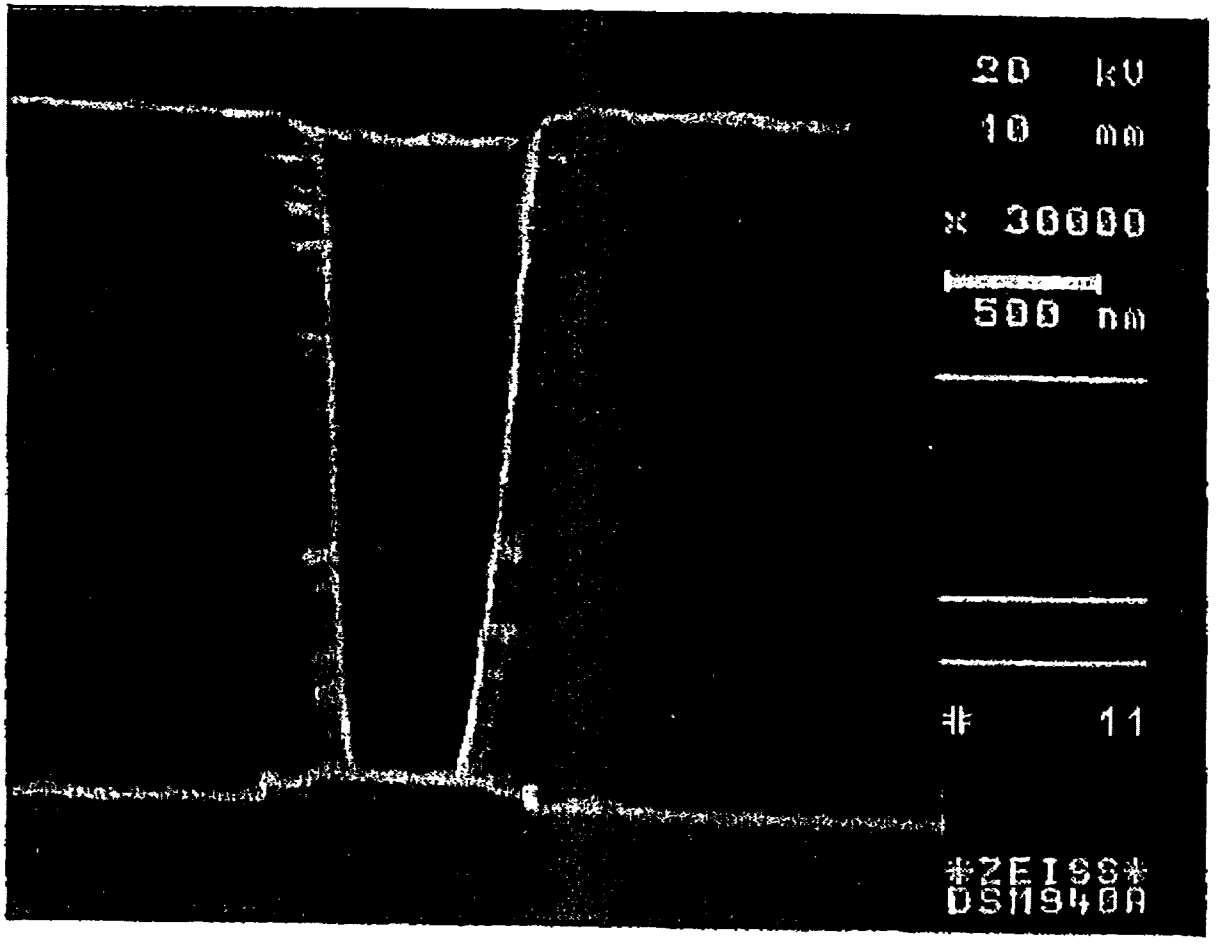
Fig 9


20 kV
10 mA
x 30000
500 nm
11
ZEISS
DSM940A

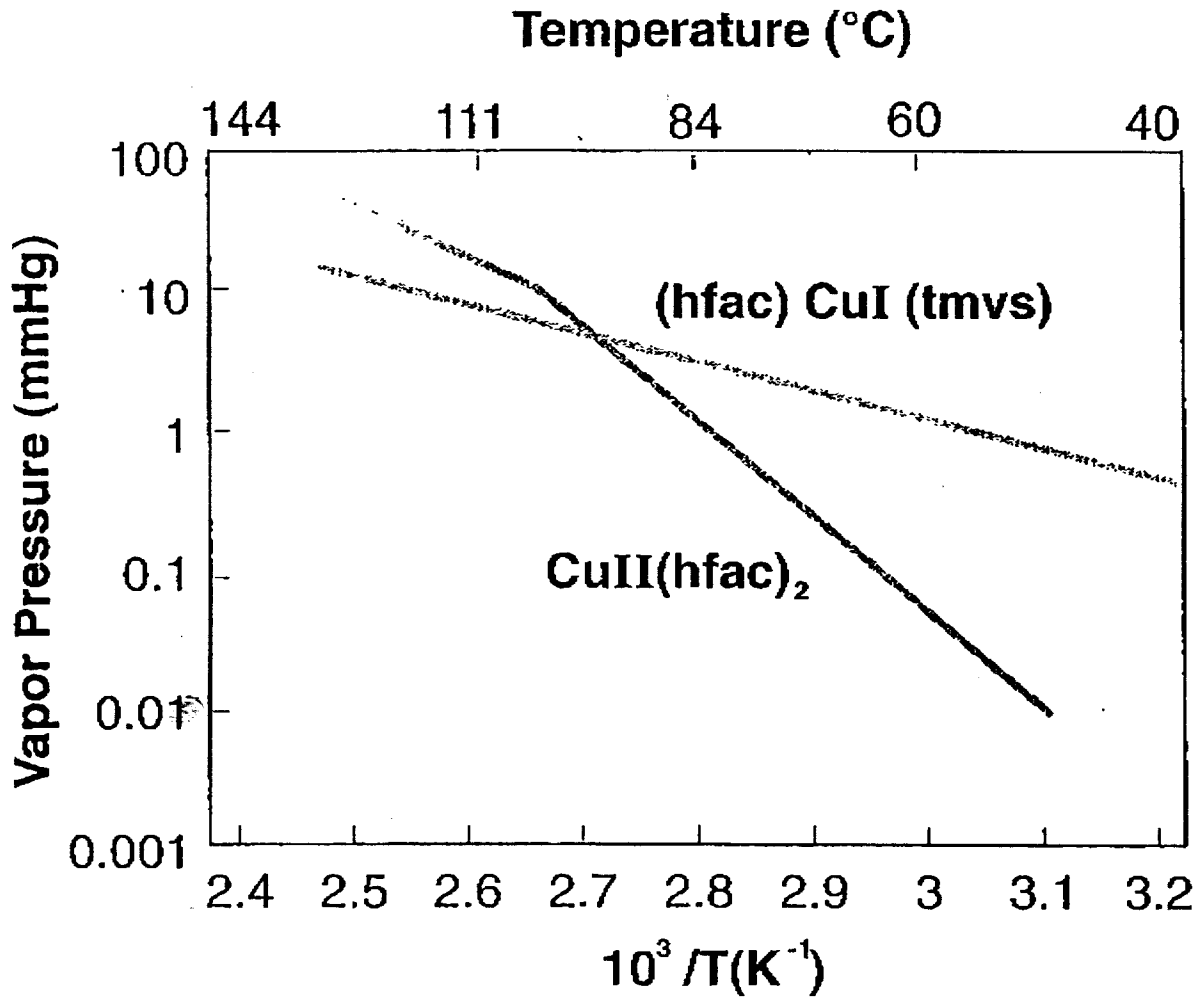
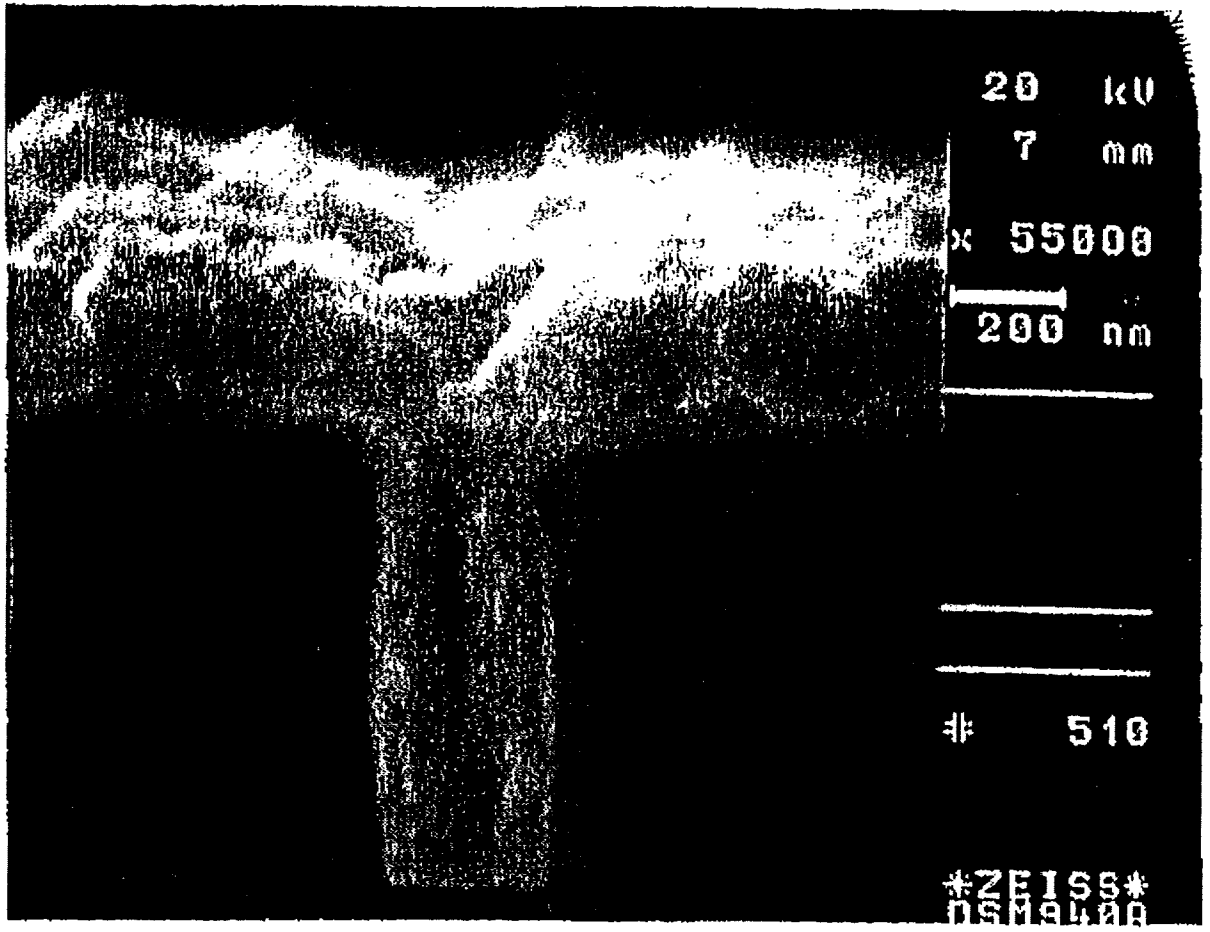
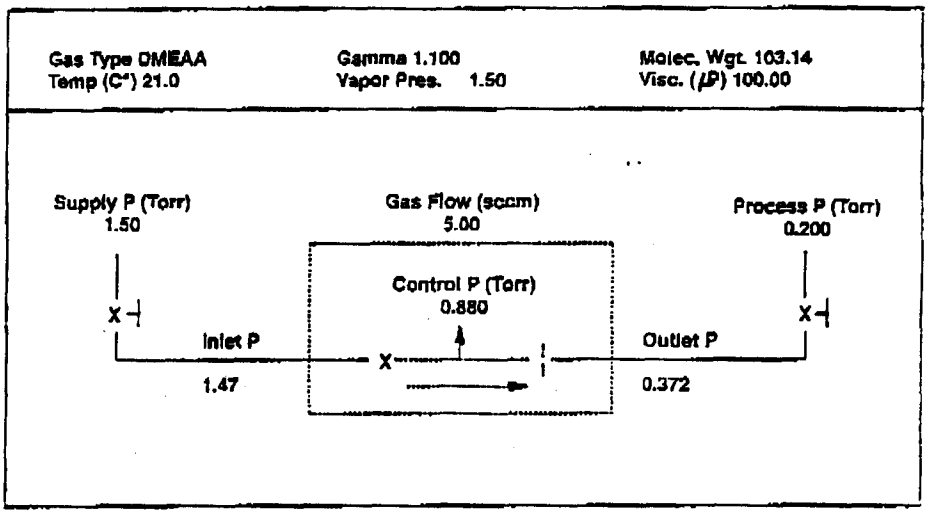
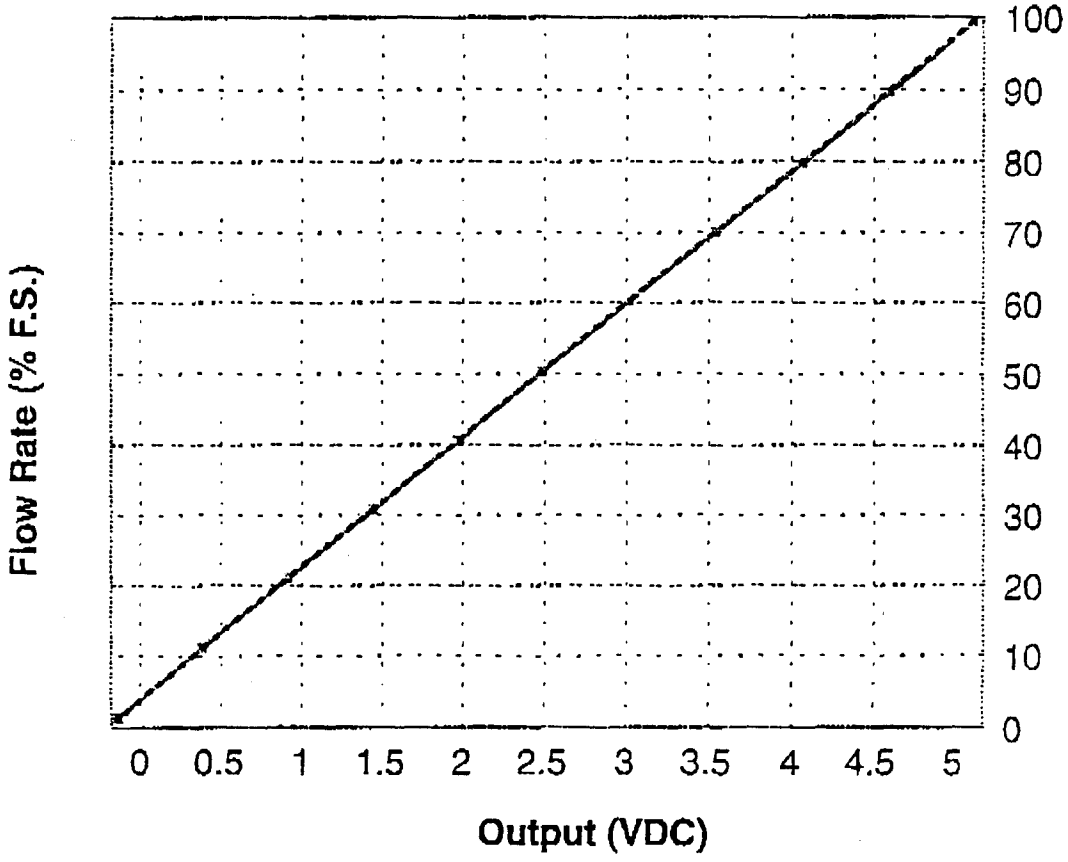

Temperature (°C)
144
111
84
60
40
100
10
1
0.1
0.01
0.001
Vapor Pressure (mmHg)
(hfac) CuI (tmvs)
CuII(hfac)2
2.4
2.5
2.6
2.7
2.8
2.9
3
3.1
3.2
10^3 /T(K^-1)

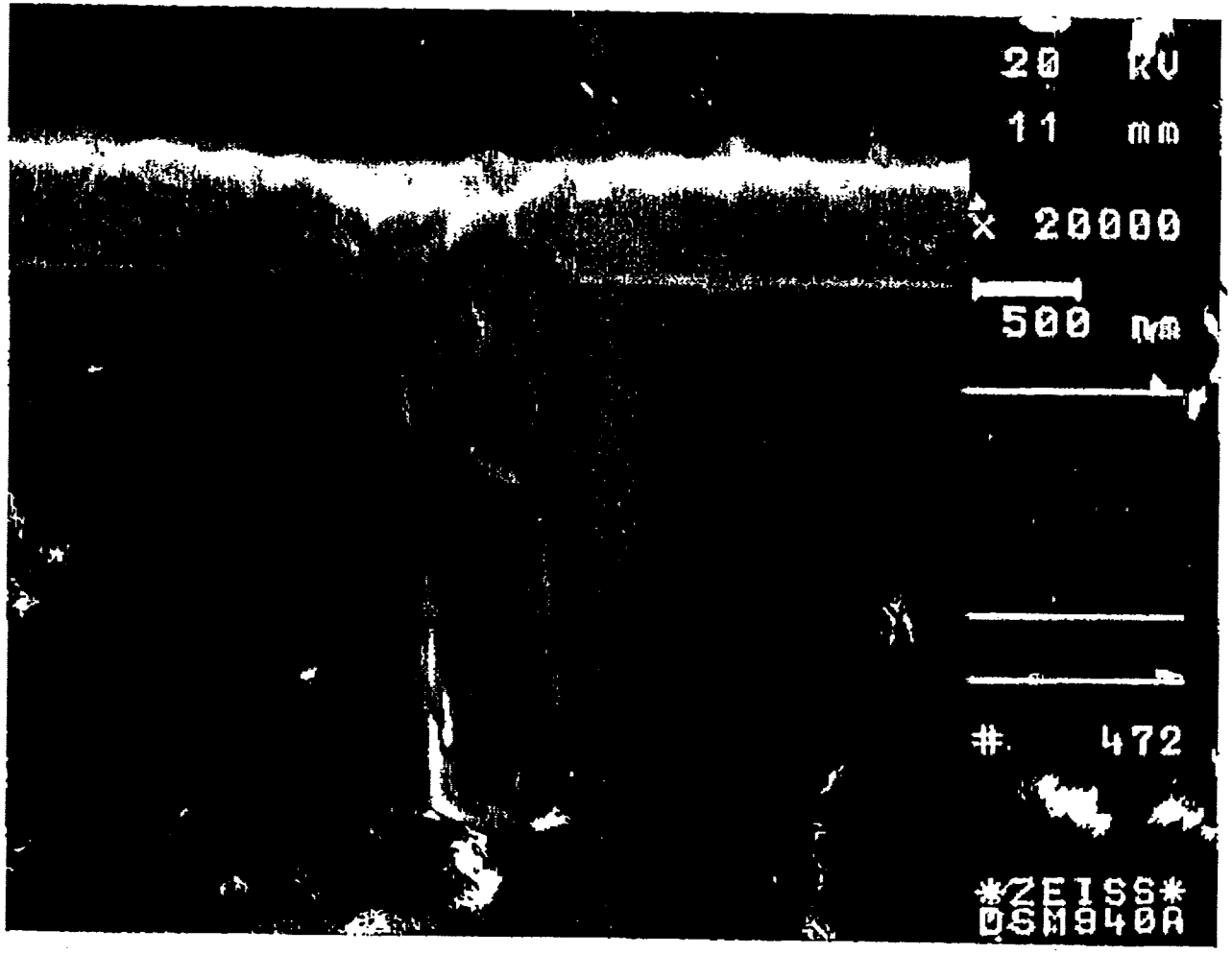
Fig. 11

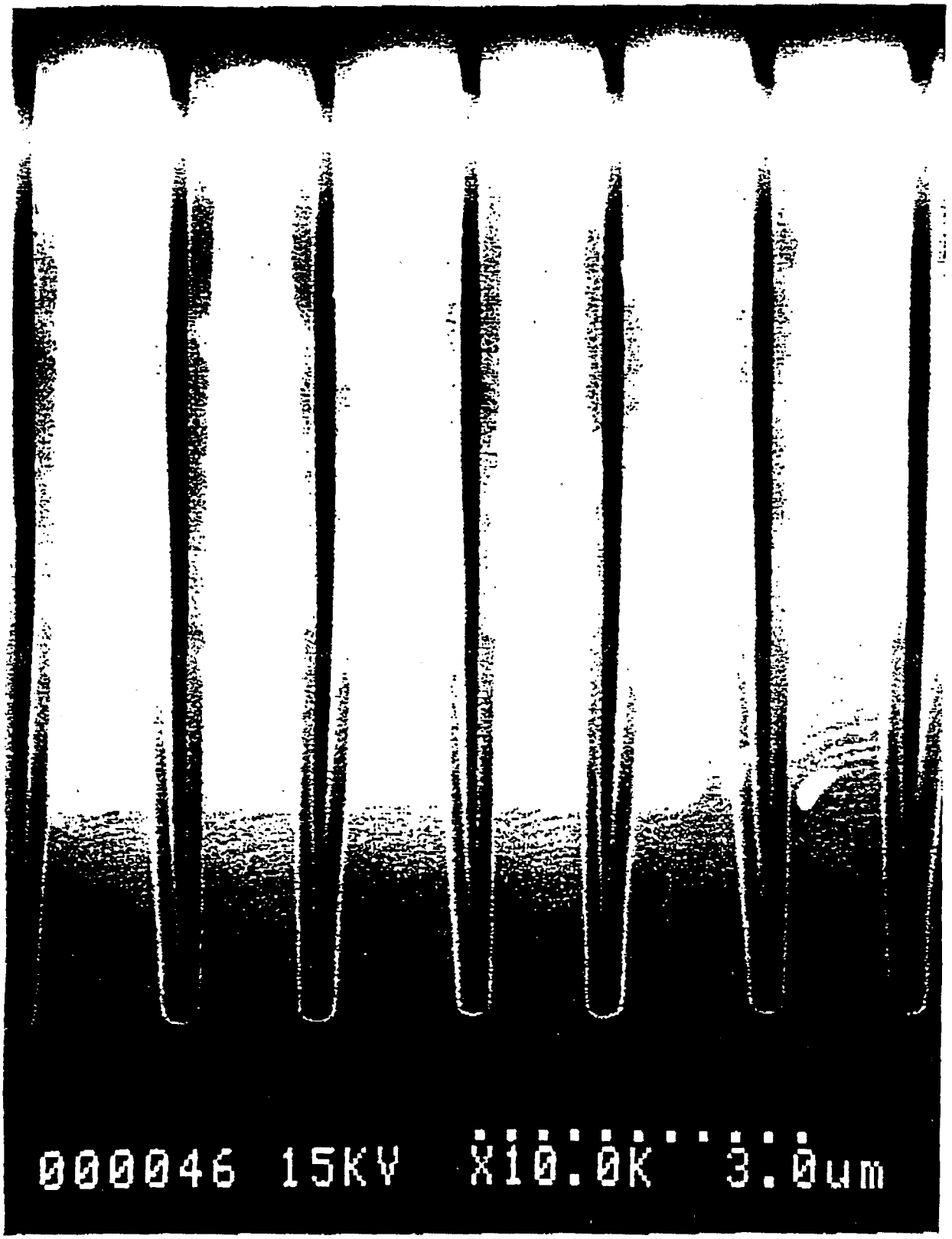

20 kV
7 mm
x 55000
200 nm
510
ZEISS


Gas Type DMEAA
Temp (C°) 21.0
Gamma 1.100
Vapor Pres. 1.50
Molec. Wgt. 103.14
Visc. (μP) 100.00
Supply P (Torr)
1.50
Gas Flow (sccm)
5.00
Process P (Torr)
0.200
Control P (Torr)
0.880
Inlet P
1.47
Outlet P
0.372


Flow Rate (% F.S.)
100
90
80
70
60
50
40
30
20
10
0
0
0.5
1
1.5
2
2.5
3
3.5
4
4.5
5
Output (VDC)

CS-SEM of Via Fill by Al CVD





30 Torr, F-teos=80 sccm, F-O3=160 sccm, F-O2=3000 sccm, F-N2=1000 sccm, T=490 C

Fig 15

DRAMS Capacitance For A Critical Voltage of 1.5V

(Leakage of 1μA/cm^2)

Process/Dielectric	Capacitance (fF/μm^2)
ON (smooth electrode)	6.5
ON with RTN (smooth electrode)	7.0
ONON (smooth electrode)	7.7
ON with HF vapor clean (smooth electrode)	8.6
ON (rough electrode)	12.0
Ta_2O_5 (smooth electrode)	12.0
Ta_2O_5 (rough electrode)	20.4
Barium Strontium Titanate (smooth electrode)	50.0 (expected)

Ref: 1994 Spring MRS Meeting - V.K. Mathews and P.C. Fazan

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Fig.16

Author's Biography

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James F. Loan is presently Operations Manager of the MKS Instruments Materials Delivery Group in Lawrence, MA. He has an extensive background in materials chemistry with an advanced degree from Harvard University. Mr. Loan has many years of experience in high precision electro-mechanical products including military aircraft laser gyro systems, wafer handling robotics while at Brooks Automation, and cluster tool design and fabrication at MRC. Prior to this position at MKS, he was responsible for primary and transfer standard pressure and flow products design and manufacturing.

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John J. Sullivan is Vice President of Marketing at MKS Instruments, Inc. Andover, MA A physicist by training, with a B.S and M.S. from Northeastern University, he spent 9 years in plasma, laser, and vacuum technology research at the U.S. Army Research Laboratories, Fort Monmouth, NJ (1960 - 1969). Later, after 5 years with Granville Phillips, Boulder, CO, he joined MKS as Manager of Technical Sales and Marketing. He has been actively engaged in pressure and flow measurement, control, and calibration instrumentation for a wide variety of scientific disciplines. Mr. Sullivan has published extensively in JVST, Appl. Phys. Lett., J. Appl. Phys., Vacuum, and other technical journals, has been active in American Vacuum Society related activities, serving as Chairman of Recommended Practices for 3 years.