

## Interoffice Correspondence

Date: 1/19/96



To : Phil Barnett  
Rob Davenport  
Tom St. Dennis  
Jim Dunnam  
Howard Grunes  
Bob Kruse  
Steve Lin  
Paul Lindstrom  
Jaim Nulman  
Jim Pasichuke  
Sass Somekh  
Avi Tepman

CC: Galiso-Nuvac  
Anne Barr  
George Drazic  
Michael George  
Dan Gockel  
Mike Grivet  
Imran Hashim  
David Huo  
Jon Ivester  
John Lechler  
Andy Nordhoff  
Terry Reed  
Scott Seamons  
Binxi Sun  
Gerald Yin

From: Jiaxiang(Joe) Zhou  
Steve Dasso  
Zbigniew Golinski

Subject: Report of hot argon cycle purge experiment at PVD chamber work center

### **A faster way to receive UHV in a PVD chamber**

#### **I. Introduction**

In order to further reduce the chamber test cycle time, the PVD chamber work center has been working with an outside company, Galiso-Nuvac, since July of 1995 to introduce a hot gas purge technique, developed by Galiso-Nuvac and labeled "HGS (Hot Gas Sweep)", into Applied Materials at Austin.

At earlier stages of the experiment, an argon recirculation machine was used with a PVD chamber to speed up the bakeout process. The results did not show a noticeable reduction in bakeout time. The probable reason is the gas delivery setup that was available at the time did not meet Nuvac's requirement for UHP Ar gas.

Galiso-Nuvac proposed a hot argon sweep method to achieve UHV in a PVD chamber. The first positive result came on November 15, which showed that PVD chamber bakeout time could be reduced by more than 50% in comparison to the presently specified procedure. After two more chamber tests to see how far this technique could be taken with PVD, the work center decided to purchase the experimental equipment including software.

In order to test the reliability of the technique and equipment, six widebody PVD chambers with almost identical configurations have been rigorously tested during the last two weeks before Christmas shutdown. A clean, very well baked Leybold Inficon RGA was also used to monitor chamber contamination. The test results show that hot argon cycle purge on the PVD chamber is a repeatable technique. It will reduce chamber bakeout time by at least 20 - 25 hours.

This report starts from a baseline - present PVD chamber vacuum qualifying process, then briefly describes the hot argon cycle purge experimental process. A detailed result of all test chambers and typical pump down curves, together with typical RGA scans will be given. Some discussion will follow.

## II. Baseline - Present PVD Chamber Vacuum Qualifying Process

### 1. Chamber Configuration

- A virgin PVD chamber (Standard, Widebody or Watercooled)
- A process kit
- A source with Cu or Al backing plate

### 2. Pumping System

- CTI - 8T Cryogenic pump
- The cryo-shield is installed to reduce heat and radiation into the cryopump
- A pair of standoffs are installed to increase pumping speed during vacuum test
- A Leybold leak detector is used as a roughing pump

### 3. Bakeout Elements

- Two bakeout lamps with 500 W each
- Center heater (Size 5", 6" or 8")

### 4. Main Bakeout Process

- Purge the chamber with 20 psi dry nitrogen for 1 - 2 hours
- Roughing down to about 100 millitorr, then open the gate valve
- Pump down to less than  $3.0E-6$  torr, turn on the bakeout lamps
- After 32 hours of lamp bakeout, turn on the heater for four more hour's bakeout (lamps stay on)
- Turn off both lamps and heater after the 32 + 4 hours of bakeout, cool down without cooling water

### 5. Qualifying Specification (at chamber test work center)

- Base pressure:  $6.0E-9$  torr
- Rate of rise:  $1.5E-6$  torr/2min.

### III. Hot Argon Cycle Purge Experiment

The layout of the hot argon cycle purge experiment is shown in Fig. 1. Compared with normal PVD chamber test, the main differences are:

- (1) A dry pump is directly connected to the roughing line of PVD chamber
- (2) An ultra-pure argon gas line is connected to the chamber through heater and/or bypass gas line
- (3) A controller (HGS-1) is used to control the argon line valve, roughing valve and gate valve
- (4) Computer software is used as an interface between the PVD chamber and the controller, automatically opening or closing valves and controlling the argon cycle purge time

The purity of argon gas is very important for this technique. The critical parameter is the water partial pressure in the argon gas. In this experiment, the commercially available ultra-pure liquid argon contains less than one ppm (part per million) of water. An argon purifier is used in the line to further reduce the water partial pressure to a level of less than 10 ppb (part per billion).

A 1000 torr baratron in addition to the 0.1 torr baratron, which was originally used with the PVD system is used to monitor the pressure in the chamber during argon purge. Two rod-type thermocouples are also used to monitor the external temperature of the chamber body.

Before turning on the bakeout lamps, the chamber is filled with the ultra-pure argon and pumped down to the 50 millitorr for three times (Cold purge). Then the chamber is pumped down to high E-7 torr and the bakeout lamps and heater are turned on. Then the chamber is pumped by the dry pump instead of the cryogenic pump. The argon valve is periodically opened and closed for about 20 cycles. During this period of about three hours, the heater is ramped up from 100 °C to 400 °C (Ramp purge). A hot argon purge of up to 10 cycles follows. The dry pump and the cryogenic pump are alternately used to pump the chamber. During the cryo-pumping period, the ion gauge can be turned on and the pressure reading and a rate of rise measurement can be used as indicators to show if the chamber is ready to cool down (Hot purge).

When the chamber is ready to cool down, the argon gas is turned off and then the gate valve is opened. All heating elements are turned off at this point except the ion gauge baking jacket. The Neslab cooling water can be used to cool the heater and the source as long as the pressure in the chamber is below 1.0E-6 torr. The cooling water will reduce the cool down time by 5 - 10 hours. TempScan software is also used to record the pumpdown curve through the ion gauge reading.

#### IV. Results

1. "Feasability Test" period: Nov. 14 - Nov. 30, 1995

Equipment owner: Galiso-Nuvac (One-chamber controller)

Quantity of test chamber: Three

Brief information:

Type	Cryo-Shield	Standoff	Bakeout-time	Qual. or Fail
(1) W/B, 8"	On	On	14 hours	6.0E-9 torr
(2) W/B, 8"	Off	Off	8 hours	6.0E-9 torr
(3) W/B, 8"	On	Off	4.5 hours	Failed

Detailed information is shown on the Table 1.

2. "Repeatability Test" period: Dec. 5 - Dec. 17, 1995

Equipment owner: AMET chamber (One-chamber controller)

Quantity of test chamber: Six

Brief information:

Type	Cryo-Shield	Standoff	Bakeout-time	Qual. or Fail
(1) W/B, 8"	On	On	12.5 hours	6.0E-9 torr
(2) W/B, 8"	On	On	13 hours	3.0E-9 torr
(3) W/B, 8"	On	On	10.5 hours	Wafer Lift Leak
(4) W/B, 8"	On	On	11 hours	Gate Valve Leak
(5) W/B, 8"	On	On	14 hours	3.0E-9 torr
(6) W/B, 8"	On	On	11 hours	2.0E-9 torr

Detailed information is shown on Table 2.

3. "Process Verification" period: Dec. 21,96 - Feb. 96

Equipment owner: AMET chamber (Three-chamber controller)

Quantity of test chamber: Thirty (proposed)

Brief information:

Type	Cryo-Shield	Standoff	Bakeout-time	Qual. or Fail
(1) W/B, 8"	On	On	11 hours	5.0E-9 torr
(2) W/B, 8"	On	On	11 hours	2.0E-9 torr
(3) W/B, 8"	On	On	11 hours	Ion gauge defect

.....

Detailed information will be provided after the experiment finished

#### V. Discussion

1. Experiments performed at the PVD chamber work center have shown, after leaving out three chambers with unrelated part failures, that the hot argon cycle purge process is faster and is a repeatable way to obtain ultra-high vacuum in a PVD chamber. Starting from the present baseline, without any configuration change in the chamber, it will reduce bakeout time by at least 20 hours per chamber. If the cooling water is used to cool the heater and the source, at least 5 more cooling hours will also be reduced. With this new bakeout technique, the capacity of the chamber work center will be potentially increase by about 85% without additional test controllers and test

ports (see the attached chamber test capacity analysis). The ultra-pure argon and dry pump used in this technique allow an ultra-high vacuum chamber, like the PVD chamber, to be pumped in a much higher pressure region so that the water vapor and other contaminants can be more efficiently removed from the vacuum chamber. A detailed theoretical explanation will be given shortly in a separate paper.

2. There are three failed chambers which involved defective parts, including a leak at the wafer lift and gate valve and damage of ion gauge filament. Once the defective parts had been identified and replaced, all three chambers were easily qualified after 1 - 2 hours of rebaking. These results show that water and other contamination have been efficiently removed from these chambers during the hot argon cycle purge. The failure reason of the only chamber which did not involve a defective part was probably due to bakeout time that was too short (4.5 hours). This chamber was also tested using a hot argon purge of the cryopump while the gate valve was opened. The failure may suggest that hot argon purge of the cryopump is not a good idea, and it has been eliminated from all following tests.

3. During the "Repeatability Test" period, a well-baked RGA head has been used to monitor all six test chambers. The RGA scan data shows that this new bakeout technique does not cause any additional contamination. Typical RGA data including background and after-bakeout scan have been included along with this report.

4. When the chamber work center purchased the test equipment, request of automation of argon purge process and modification of the single-chamber controller into multi-chamber controller have been discussed. Now the three-chamber controller has been tested for the first three chambers, and it operates satisfactory. The software for automation of the process has been tested several times. In order to solve an occasional lock-up problem of the interface computer and ensure a safe work environment, the work center is working with Galiso-Nuvac to debug the software and to install an uninterruptable power supply (UPS) into the interface computer. Concurrently, technologist training in this new technique is also scheduled for the chamber test module.

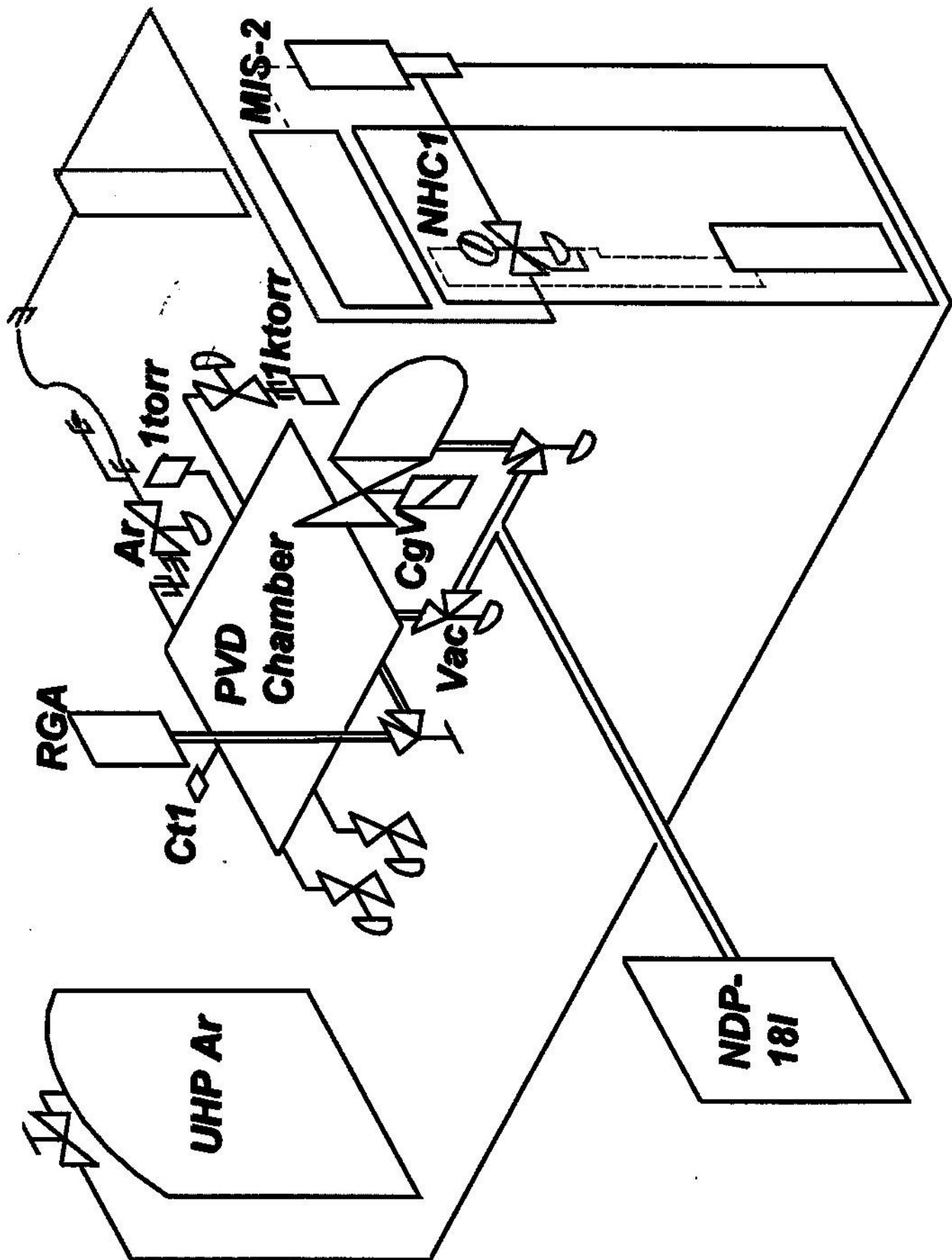
5. Further Testing: The work center is planning to test a variety of chambers, currently being tested in the chamber test module, utilizing the hot argon cycle purge technique in the near future. This will involve a 101% chamber which is not equipped with a heater, a PVD chamber which is equipped with a heater but not equipped with the heater argon line, a watercooled chamber which is equipped with a large cryopump elbow and a HTHU heater which needs a much longer bakeout time, a preclean chamber and a clamped-degas chamber. The resulting bakeout time and cooldown pressure and ROR for

each of these chambers needs to be determined during the testing. The possible impact of this technique to chamber qualification will also be investigated. The experiment results will be provided as soon as possible.

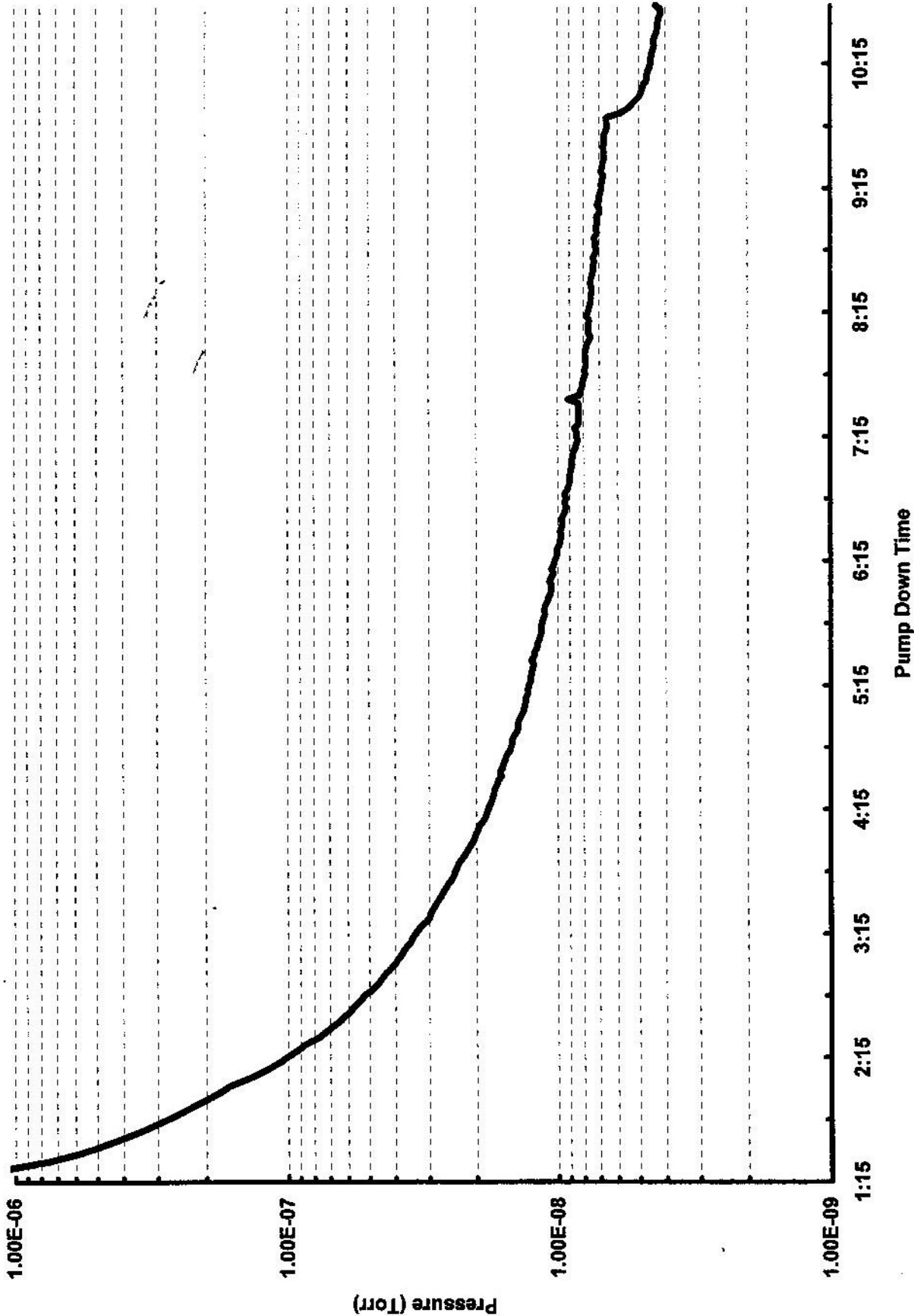
#### Appendix

1. Figure 1: Layout of the hot argon cycle purge experiment
2. Figure 2: Typical pump down curve after hot argon cycle purge
3. Table 1: Test results of first three "Feasability Test" chambers
4. Table 2: Test results of the six "Repeatability Test" chambers
5. Figure 3: RGA background scan (LeyBold - Head 4530)
6. Figure 4: RGA scan of the qualified chamber S/N 1262 using hot argon cycle purge (LeyBold - Head 4530)
7. PVD chamber test/capacity analysis





# Pump Down Curve After Ar Cycle Purge (s/n 1302)





List of ultra-pure argon cycle purge test										
No. Date	Chamber	Source & Process Kit	Heater	Cryo-Shield	Standoff	Bakeout Tim	Cool Tim	Total Tim	QF Pressure	Rate of Rise
1	Nov. 14-16	1089 W/B Yes	8"	On	On	14 hours	15 hours	29 hours	6.0E-9 torr	7.9E-7 torr/2m.**
Hot argon purge the chamber only, no chill water during cooldown Test personnel: Chuck Granci, Carl Hoover(Nuvac) Joe Zhou, Zbigniew Golinski(AMET)										
2	Nov. 17-19	1282 W/B Yes	8"	Off	Off	8 hours	4 hours	12 hours	6.0E-9 torr	N/A
Hot argon purge the chamber only, chill,water flow through the target and heater during cooldown										
*	Re-QF	Yes	8"	On	On	2 hours	20 hours	22 hours	6.0E-9 torr	9.3E-7 torr/2m.
Test personnel: Chuck Granci, Carl Hoover(Nuvac) Steve Dasso, Zbigniew Golinski (AMET)										
3	Nov. 28-30	1250 W/B Yes	8"	On	Off	4.5 hours	N/A	N/A	Failed 2.9E-8 torr	N/A
Hot argon purge both chamber and cryopump while the gate valve opened, chill water colldown Test personnel: Chuck Granci, Carl Hoover(Nuvac) Joe Zhou, Zbigniew Golinski(AMET)										
Present AMET procedure of chamber bakeout										
*		Yes	5",6",8" or 101	On	On	36 hours (32 + 4)	15 - 18	51 - 54	6.0E-9 torr	1.5E-6 torr/2m.

List of Hot Argon Cycle Purge Reliability Test

Test Number	2	3	4	5	6
Test Date	6/12 - 7/12	10/12 - 11/12	12/12 - 13/12	14/12 - 15/12	16/12 - 17/12
Chamber Type - Heater	Wide Body - 8"	Wide Body - 8"	Wide Body - 8"	Wide Body - 8"	Wide Body - 8"
System Number, S/N	3685-4, 1262	3687-2, 1302	3688-2, 1288	3690-1, 1290	3692-4, 1225
Backing Plate	Cu	Cu	Al	Al	Al
Process Kit	Yes	Yes (G-12)	Yes	Yes (G-12)	Yes
Shuttler Arm ?	No	No	No	Yes	No
With Cryoshield ?	Yes	Yes	Yes	Yes	Yes
With Standoff ?	Yes	Yes	Yes	Yes	Yes
Bakeout Time (hours)	12.5	13	11	14	11
Cooling With Water ?	Yes **	Yes **	Yes	Yes	Yes
Time to 6.0E-9 torr	11.5	8	N/A	8.5	5
Time to Qualifying	11.5	10	N/A	10	8.5
Base Pressure *	6.0E-9 torr	3.0E-9 torr	3.0E-9 torr	1.0E-7 torr	2.0E-9 torr
Rate of Rise	1.4E-6 torr/2m.	1.4E-6 torr/2m.	3.0E-6 torr/2m.	1.7E-6 torr/2m.	1.5E-6 torr/2m.
Qualified ?	QF	Failed	Failed	QF	QF
Start to Finish Time	25	24	N/A	25	20
Software Through ?	Yes	Yes	No	Yes	No
Auto or Semi-auto	Semi-Manual	Semi-Auto	Auto	Auto	Auto
If Fail, What's Reason	N/A	N/A	Wafer lift leak	GV leak	N/A
Re-Bakeout Time	N/A	N/A	1.5 Hours	1 Hour	N/A *
Final Pressure	N/A	N/A	2.0E-9 torr	6.0E-9 torr	N/A
Test Personnel	Joe Z.	Joe Z.	Joe Z.	Joe Z.	Joe Z.
	Zbigniew G.	Lindsey(galiso)	Lindsey(galiso)	Zbigniew G.	Zbigniew G.
		Steve D.	Zbigniew G.		
		Zbigniew G.			

\* Stable Ion Gauge for all six test chambers    \*\* Cooling facility failure noticed during cooldown

Hot Ar Cycle purge Test -- 10/1 W/O-S

3/11 1262

RGA 4530

RGA back ground

PL(20) = 1.0E-9 for

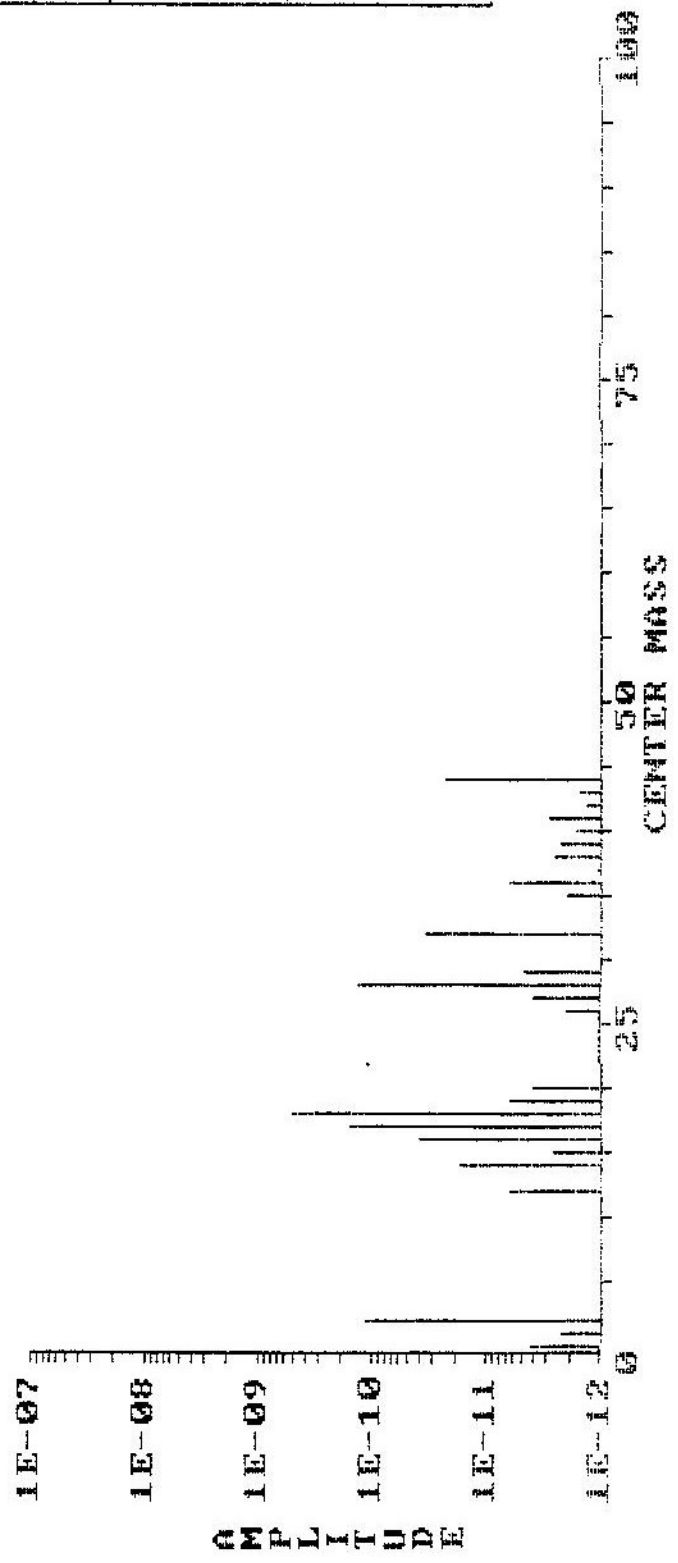
PRINT	GAIN <	GAIN >	DISPLAY	EXIT
			AXIS	INFO

REP CRGM925  
H102

11:14:10  
Fri Dec 01 1995

EMULS ON  
DEGAS OFF  
RELS OFF  
CIS OFF  
PPM OFF

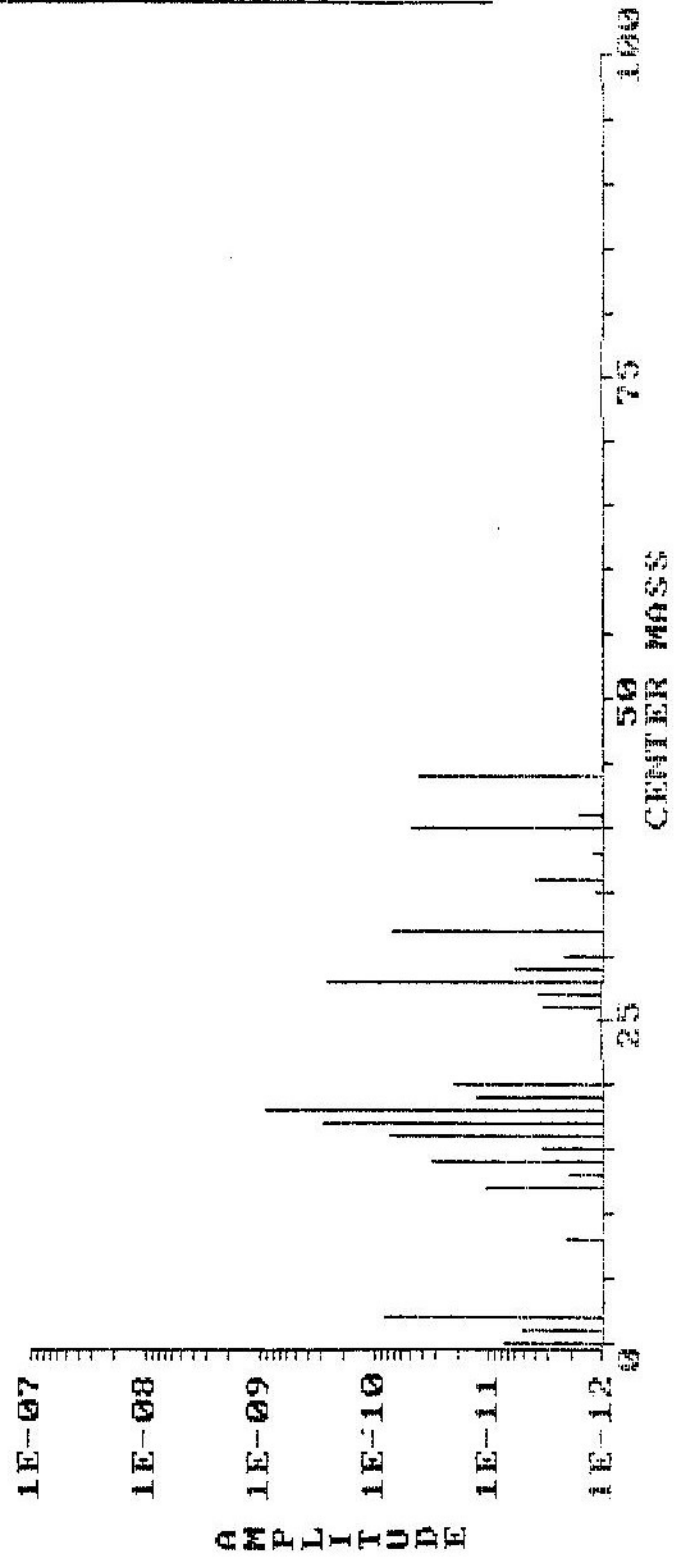
GAIN = 1E-09 A. S  
SCAN = N 5.7 S  
EMULT = 1000  
MASS = 50 AMU  
WIDTH = 100 AMU



140T Ar Cycle purge Test 106 W/B-8  
 Final Qualify's print (14 hours basement  
 11 hours control room)  
 RGA 4530  
 Monitor 22°C  
 P<sub>6</sub> = 6.0 E-7 Torr ROR = 1.4 E-6 Torr/2m.

SCAN >	C. MASS	WIDTH	GAIN >	DISPLAY	CONTROL
SCAN <	EMULT		GAIN <	AXIS	INFO

Bar HI00  
 16:11:52  
 Thu Dec 07 1995  
 10308 KORE  
 GAIN = 1E-09 A.S  
 SCAN = N 5.7 S  
 EMULT = 1000  
 MASS = 50 AMU  
 WIDTH = 100 AMU  
 EMIS ON  
 DECAL OFF  
 RELAY OFF



**PVD CHAMBER TEST CAPACITY ANALYSIS/NUVAC  
Q1 (91 SYSTEMS)**

VARIABLES	Current	Nuvac		
SET-UP	0.5	0.5		
LEAK CHECK	0.5	0.5	ADD'L HRS FOR NSO	24
COOL CRYO	4	4	ADD'L HRS FOR TC wafer	24
INSTL PROC KIT	0.5	0.5		
N2 PURGE	2	0		
PUMP DOWN	2	0		
BAKE OUT	32	14		
B/O W-HEATER	4	0		
COOL DOWN	18	8		
FINAL TEST	0.5	0.5		
MAT ISSUES	8	8		
MISC OTHER	0	0		
1st PASS HRS	72	36		
2nd PASS HRS	24	24		

SYSTEM	SYS QTY	CHM PER	WKS QTR	QTY WK	% YLD	1st PASS HRS	2nd PASS HRS	TOT HRS REQ
ENDURA	86	3.8	13	25.14	90%	1810	60	1870
CENTURA	5	2.3	13	0.88	90%	64	2	66
NSO	39	1	13	3.00	90%	216	7	223
TC wafer test	22	1	13	1.65	90%	40	4	44
MISC OTHER	13	1	13	1.00	90%	72	2	74
CHMs PER WEEK =								29.02
Weighted Hrs./chamber=								78.47
								2277

ENDURA	86	3.8	13	25.14	90%	905	60	965
CENTURA	5	2.3	13	0.88	90%	32	2	34
NSO	39	1	13	3.00	90%	108	7	115
TC wafer test	22	1	13	1.65	90%	66	7	72
MISC OTHER	13	1	13	1.00	90%	36	2	38
CHMs PER WEEK =								29.02
Weighted Hrs./chamber=								42.21
								1225

Number of Nuvac ports: 3

CONTROLLERS	4	4	4	4	4	4
PORTS	19	20	21	22	23	24
DAYS/WK	6.5	6.5	6.5	6.5	6.5	6.5
HRS/DAY	21	21	21	21	21	21
HRS/WK PER PORT	137	137	137	137	137	137
Standard ports	16	17	18	19	23	24
Nuvac ports	3	3	3	3	3	3
Max. number of Ch./wk (Std.)	28	30	31	33	40	42
Max. number of Ch./wk (Nuvac)	10	10	10	10	10	10
Total max. Number of Ch./wk	38	39	41	43	50	51
Utilization	77%	74%	71%	68%	58%	56%