NASA

Hydrogen Leak Detection

Technical Interchange Meeting

Held at:

Kennedy Space Center
Cape Canaveral, Florida

April 29–30, 1992

Proceedings

Florida Tech

INQUIRIES

FOR TECHNICAL INFORMATION

REGARDING TOPICS PRESENTED AT THIS

HYDROGEN LEAK DETECTION TECHNICAL INTERCHANGE MEETING

PLEASE CONTACT THE FOLLOWING:

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APRIL 29, 1992

HYDROGEN LEAK DETECTION TECHNICAL INTERCHANGE MEETING
KENNEDY SPACE CENTER
APRIL 29-30, 1992

AGENDA - Wednesday, April 29

9:00 a.m.	SECTION 1	Introduction Overview-Florida Tech Study	Ron Barile
	SECTION 2	Welcome / Key Note Address H2 Leak Detection Today & Tomorrow at NASA KSC	William Helms
	SECTION 3	H2 Leak Detection at NASA-MSFC	William Powers
	SECTION 4	H2 Sensor Work at NASA Lewis	William Nieberding
		Break - Refreshments	
	SECTION 5	Leak Detection at Stennis Space Center	Jay D. Hunt
	SECTION 6	Recent Advances in Solid State Hydrogen Sensors	Robert Hughes
	SECTION 7	Sandia Robust Wide-Range Hydrogen Sensor Module	Paul McWhorter
	SECTION 8	Microfabricated Point Source Hydrogen Sensor	Robert Powers
	SECTION 9	Optical Leak Detection of H2	Robert Hindy
	SECTION 10	Qualifications of Metal-Insulator-Semiconductor Sensors for H2 Leak Detection	Steve Pyke
12:00		Lunch at HQ Cafeteria	
1:00		Facility Tours - o Shuttle Landing Field o Launch Control Center o Vehicle Assembly Building o Shuttle Launch Pad o Orbiter Processing Facility	
	SECTION 11	A Hydrogen Microsensor Concept	Thurman Henderson
	SECTION 12	SSME Leak Detection Using Sequential Image Processing	Montgomery Smith

HYDROGEN LEAK DETECTION TECHNICAL INTERCHANGE MEETING
KENNEDY SPACE CENTER
APRIL 29-30, 1992

AGENDA - Thursday, April 30

9:00 a.m.		Introduction	Ron Barile
	SECTION 13	Hydrogen Detection at AEDC. A Presentation of Low Pressure Time Response Tests of Catalytic Sensors	Ben Hartsfield
	SECTION 14	Leak Detection for Launch Vehicle Applications	Darby Makel & Roy Sakabu
	SECTION 15	Remote Detection & Imaging of Hydrogen Concentration by Raman Lidar	Robert Martin
		Break - Refreshments	
	SECTION 16	Raman Lidar Measurements	S. H. Melfi
	SECTION 17	Remote Laser-based Detection of H2 by Laser-exited Amplified Spontaneous Emission	Andy Sappey
	SECTION 18	Hazardous Gas Detection System Design Requirements for National Launch System	Jacqueline Guerrero
	SECTION 19	US Navy Needs for Hydrogen Measurement	Jeffrey Wyatt
	SECTION 20	Hydrogen Piping Specification-New CGA Publication G5.4	Michael Deakter
12:00		Lunch at HQ Cafeteria	
1:00		Tour Labs - o Hazardous Gas Detection; o Optical Instrumentation; o Transducer Instrumentation	
	SECTION 21	Hydrogen Sensing, Transport & Storage	Konrad Colbow
		Informal Group Discussions	All Attendees

HYDROGEN LEAK DETECTION TECHNOLOGY

KSC GRANT TO FLORIDA TECH

MARCH, 1992 THROUGH FEBRUARY, 1993

* PI: RONALD G. BARILE, CHEM. ENGRG, FL. TECH.

* CONTACTS: BILL HELMS, GSE BRANCH CHIEF

  DAVE COLLINS, INSTRUMENTATION SECTION CHIEF

  BILL LARSON, TRANSDUCERS SECTION

* WORK PLAN:

  1. REVIEW AND REPORT H_2 DETECTION R & D
  2. DEFINE DEVICE REQUIREMENTS WITH KSC
  3. SET UP COMMUNICATION WITH JSC-TIM PARTICIPANTS, OTHER WORKERS AND USERS, INCLUDING SITE VISITS
  4. CONDUCT TIM AT KENNEDY SPACE CENTER
  5. TEST PROMISING PROTOTYPES AT KSC STARTING JUNE, 1992

HYDROGEN LEAK DETECTION TIM

June 4-5, 1991
Johnson Space Center

HYDROGEN LEAK DETECTION PRESENTATIONS

In-Flight Hazardous Gas Detection
(NASA JSC/Nancy E. Munoz)

Space Shuttle Hydrogen Leak Detection at KSC
(NASA KSC/William R. Helms)

H2 Leak Detection (LeRC)
(NASA LeRC/Bill Nieberding)

Leak Detection Overview (MSFC)
(NASA MSFC/W.T. Powers)

Hydrogen Safety
(Vitro Corporation/Willam A. Price)

Leak Detection at Stennis Space Center
(NASA SSC/Tom Koger & Curtis Campbell)

Ground Support System Design & Operation
Concepts for the National Aerospace Plane / X-30
(Air Force Flight Test Center/Kurt D. Buehler)

H2 Detection at Arnold Engineering Development Center
(Sverdrup/Ben Hartsfield)

NASP Flight Instrumentation
(Rocketdyne Division, Rockwell Inernational/Robert N. Hindy)

Automated H2 Leak Detection for Rocket Engine
Health Monitoring
(Aerojet Propulsion Division/Randy L. Bickford)

Airco Hydrogen Leak Testing Methods
(Airco Industrial Gases/Joe Green)

Hydrogen Leak Prevention & Detection
(Air Products/George E. Schmauch)

Hydrogen Leak Detection
(Linde, Union Carbide Industrial Gases/Tom G. Halvorson)

H2 Leak Detection Sensors
(Los Alamos National Lab/Walter Stuart)

Thin Film Hydrogen Sensor
(Oak Ridge National Laboratory/Barbara S. Hoffheins)

University of Cincinnati Leak Detection Activities
(University of Cincinnati/Peter Disimile)

Optical Non Intrusive Leak Detection for the
Space Transportation System
(Rocketdyne Division, Rockwell International/Robert N. Hindy

NASA Headquarters H2 Leak Detection Requirements
(NASA Headquarters/Addison Bain)

HYDROGEN LEAK DETECTION AT KSC TODAY AND TOMORROW

William R. Helms
Chief, GSE Branch
Kennedy Space Center
April 29, 1992

CURRENT SYSTEMS

- Hydrogen Leak Detection in air
- Hydrogen Leak Detection in N2 or He
- Hydrogen Fire Detection
- Primary Hazardous Gas Detection (HGDS)
- Backup Hazardous Gas Detection (B/U HGDS)
- Hydrogen Umbilical Mass Spectrometer (HUMS)

TECHNOLOGY DEVELOPMENT PROJECTS

H2 Leak and Fire Detection

- H2 Leak Detection for N2/He purged areas
- Cryogenic H2 Leak Dynamics
- UV / IR H2 Flame Detection
- Hydrogen Flame Simulator / Calibrator
- Multispectral Television (MTV)

Leak Location and Evaluation

- Schlieren Imaging of H2 Leaks
- Ultrasonic Leak Location
- Ultrasonic Imaging of Leaks
- Millimeter Wave Imaging of H2 Leaks
- Plume (Condensed H2O) Image Processing

HAZARDOUS GAS DETECTION (MULTIGAS SENSORS)

- Gas Analysis by Optical Spectrometry
- Miniature Ruggedized Mass Spectrometers
- Advanced Hazardous Gas Detection Systems

H2 LEAK DETECTION IN AIR

Requirement:

- Detect cryogenic or gaseous hydrogen leaks in Ground LH2 Loading System at flanges, valves, etc., during propellant fill and drain

Met by:

- Approximately 60 Detector Electronics (Rexnord) catalytic combustion sensors above strategic flanges, valves, etc.
- Range, 0-40,000 PPM (1-5 VDC)
- Sensors typically maintained calibration ± 2500 ppm for over one month in lab tests

H2 LEAK DETECTION FOR PURGED AREAS

Requirement:

- Measure H2 leakage in purged areas such as LH2 Tail Service Mast, Umbilical Disconnect cavities, etc.

Met by:

- Sample tubes to Remote Sensor Panels containing air and sample flow controllers, mixing chamber, Rexnord catalytic combustion sensor, sample transport pump, and health check instrumentation
- Integral Zero, Span, and Test Gas mods in work
- 10ea. 4-sensor panels completed, awaiting acceptance test and installation

HYDROGEN FIRE DETECTION

Requirement:

- Detect hydrogen fires in gaseous / liquid hydrogen storage and loading facilities, at Launch Pad, etc.

Met by:

- Approximately 60 UV Flame Detectors monitoring flanges, valves, umbilical disconnects, etc., and the astronauts' Emergency Egress path

HAZARDOUS GAS DETECTION SYSTEM (HGDS)

Requirement:

- Measure concentrations of H_2, He, O_2, N_2, Ar in the Orbiter Aft, Payload Bay, Midbody, the External Tank Intertank area, and the Tail Service Mast Hood

Met by:

- Computer controlled quadrupole mass spectrometer in the Mobile Launch Platform with sample tubes through umbilicals into the Flight Vehicle

AREAS SAMPLED

- ET Intertank (LH2, LO2 tanks)
- Payload Bay (payload fluids)
- LH2 Tail Service Mast (MPS plumbing)
- Orbiter Midbody (Fuel Cells)
- Orbiter Aft Fuselage (MPS/SSME)

HGDS ACCURACY AND PRECISION (H2)

Theoretical Concentration	HGDS Reading (Average)	Error (Average)	Standard Deviation	Number of Samples
4040 ppm H2	3971 ppm	-69 ppm	29 ppm	6
1060 ppm H2	1037 ppm	-23 ppm	25 ppm	8
491 ppm H2	511 ppm	+20ppm	22 ppm	25
250 ppm H2	258 ppm	+ 8 ppm	13 ppm	25
100 ppm H2	96 ppm	- 4 ppm	23 ppm	25
50 ppm H2	55 ppm	+ 5 ppm	22 ppm	25
24 ppm H2	27 ppm	+ 3 ppm	11 ppm	25

HGDS ACCURACY AND PRECISION (O_2)

Theoretical Concentration	HGDS Reading (Average)	Error (Average)	Standard Deviation	Number of Samples
3640 ppm O_2	3696 ppm	+56 ppm	13 ppm	6
1060 ppm O_2	1056 ppm	- 4 ppm	23 ppm	8
505 ppm O_2	502 ppm	- 3 ppm	8 ppm	16
255 ppm O_2	249 ppm	- 6 ppm	9 ppm	19
96 ppm O_2	113 ppm	+17 ppm	3 ppm	19
49 ppm O_2	49 ppm	0 ppm	2 ppm	16
26 ppm O_2	31 ppm	+ 5 ppm	3 ppm	20

BACKUP HGDS

Requirement:

- Provide redundancy for Prime HGDS

Met by:

- Totally redundant (including AC power) Sampling System, Mass Spectrometer and Control and Data System. Standard Perkin-Elmer MGA-1200 industrial mass spectrometer (magnetic sector, fixed collectors, ion pumped high vacuum system)

HYDROGEN UMBILICAL MASS SPECTROMETER (HUMS)

Requirement:

- Accurately sample purged areas surrounding Ground-to-Flight Vehicle hydrogen umbilical disconnects
- Samples same purged areas as H2 leak detector panels
- Provides wider dynamic range (0.1 to 100%)
- Measures all significant gases present (H2, He, N2, O2, Ar)

Met by:

- Turbomolecular pumped, multiple collector magnetic sector mass spectrometer (Modified Perkin-Elmer MGA-1200)
- Early prototypes used for Discovery FRF, and STS-35 / STS-38 H2 leak investigation
- Operational unit installed 2/92 for Endeavor FRF to replace prototype

H2 LEAK AND FIRE DETECTION

- H2 Leak Detection for N2/He purged areas
- Cryogenic H2 Leak Dynamics
- UV / IR H2 Flame Detection
- Hydrogen Flame Simulator / Calibrator
- Multispectral Television (MTV)
- Schlieren Imaging of H2 Leaks

HYDROGEN LEAK DETECTION FOR N2/HE PURGED AREAS

Objective:

Develop small, light, rugged, reliable H2 point sensors

- Must be insensitive to O2 concentration

Florida Institute of Technology (FIT) Study

- Survey / report current technology development projects
- Select candidates for further development
- Eventual test / evaluation at FIT / KSC

CRYOGENIC HYDROGEN LEAK DYNAMICS

Objective:

Determine real-world behavior of cryogenic hydrogen leaks

Literature survey, preliminary experiments in work

Preliminary Results:

- Plume is <u>NOT</u> where the hydrogen is

Future Plans:

- Map velocities fields and composition
- Controlled field tests
- Involve University of Central Florida cryogenic / flow dynamnicists

ULTRAVIOLET / INFRARED H2 FLAME DETECTION

Objective:

Develop flame detectors immune to false alarms from H2 Flare Stack

- Flare Stack on LC39 A/B produces H2 Flame 60 to 300 feet in length
- Current sensors pointed away and hooded
- Intermittent false alarms due to reflection and scattering
- Commercial UV/IR Sensors not optimized for H2
- Plan to use UV plus IR plus signal processing
- In-house development in work

HYDROGEN FLAME SIMULATOR / CALIBRATOR

Objective:

Develop optical calibration device to replace H2 flame

- Mimic H2 fire emission spectra and characteristics
- Preliminary design complete
- Hydrogen flame spectral recording in work
- UV bulbs, filters in work (200-240 nM)
- Potential portable field test unit

MULTISPECTRAL IMAGING OF HYDROGEN FIRES

Requirement:

Launch Pad-qualified broadcast quality camera which unambiguously identifies hydrogen fires by false color image over normal TV picture

- Working prototype with boresighted visible and IR (2-5 Micron) Pb Se detector completed
- Pb Se detectors 1x64 scanned array
- Peltier cooling
- Field tested on LC39 H2 Flare Stack
- Production prototype, field test FY93
- Operational implementation FY94

LEAK LOCATION / EVALUATION

- Schlieren Imaging of H2 Leaks
- Ultrasonic Leak Location
- Ultrasonic Imaging of Leaks
- Millimeter Wave Imaging of H2 Leaks
- Plume (Condensed H2O) Image Processing

HAND-HELD SCHLIEREN IMAGING OF H2 LEAKS

- Compact, lightweight, self-contained
- Allows visualization of gas leaks (H2, He, Freon, etc.)
- Useful for finding leaks in complicated piping systems
- Based on 130-yr-old Foucault test for optical mirror figure
- Images changes in index of refraction as variation of light intensity
- Prototype completed and tested
- Supported 2 inch Replenish Valve test at LETF

ULTRASONIC HYDROGEN LEAK LOCATION

Objective:

Determine location of known H2 leakage in complex areas such as Orbiter Aft Fuselage

- Leaks typically emit acoustic energy around 40 kHz
- Focus via parabolic reflector to microphone
- Pan and tilt to determine leak location
- Triangulate with 2 or more units
- Used successfully on STS-38 H2 leak testing

ULTRASONIC IMAGING OF HYDROGEN LEAKS

- Advanced version of Ultrasonic Leak Locator
- Uses 8x8 array of microphones "Ultrasonic CCD Array"
- Displays "image" of acoustic energy on CRT
- Can "illuminate" laminar leak with active source
- Can see leak plumes rising from behind pipes, etc.

MILLIMETER WAVE IMAGING OF H2 LEAKS

Objective:

Image leaks hidden under foam insulation

- Insulation transparent at millimeter wave lengths
- Remote thermometer / radiometer for <u>very</u> cold (20 K) LH2 lines
- Just starting 3-year project

PLUME IMAGE PROCESSING

Objective:

Remotely <u>measure</u> leak <u>rate</u> from Orbiter-to-External Tank 17 inch Disconnect

- Image condensed water vapor cloud (Plume) associated with cryogenic hydrogen leaks
- Assumes adequate information (size, motions, etc.) encoded in plume dynamics to derive leak rate
- Image Processing System in work
- Initial phase of 5 year project

HAZARDOUS GAS DETECTION (MULTIGAS SENSORS)

- Gas analysis by Optical Spectrometry
- Miniature Ruggedized Mass Spectrometer
- Advanced Hazardous Gas Detection Systems

GAS ANALYSIS BY OPTICAL SPECTROMETRY

Objective:

Replace mass spectrometer with multi-spectral optical techniques

Advantages

- No high vacuum system
- Small, lightweight, rugged, inexpensive, simple, and reliable
- Combine emission, absorption (especially laser induced), fluorescence and Raman Spectrometry
- Raman H2 detection demonstrated, O2/N2 will be added
- Project in early conceptual stage

MINIATURE RUGGEDIZED MASS SPECTROMETER (MRMS)

Objective:

Develop a small, rugged multigas sensor for drag-on instrumentation, ascent, re-entry, etc.

- Time-of-Flight mass spectrometer potentially able to meet above requirements

- Current electronics and computer technology enable required sensitivity, speed, and miniaturization

- Two prototype instruments fabricated, performance and vibration testing (to 18 G) in work

ADVANCED HGDS
(HGDS II)

Objective:

Replace 1970's technology HGDS and Back-Up HGDS with a system supportable well into the 21st century

- US Navy submarine-based (militarized) mass spectrometer with turbomolecular-pumped high vacuum system

- Fault tolerant sample system

- Control / data acquisition including expert system for self-validation, troubleshooting, and operator work load reduction

- Prototype in work. Key components (sample system, mass spectrometer) tested on HUMS

ADVANCED HAZARDOUS GAS DETECTION (AHGDS)

Progress:

Mass Spectrometer

- Mass spectrometer selected, modified U.S. Navy CAMS I
- Expanded dynamic range by turbomolecular pumping
- Improved sensitivity for helium leak testing achieved using reverse flow technique

Turbomolecular Pump

- Vibration testing complete
- Long term testing in progress
- Survived every Shuttle launch since STS-26R with no failures

ADVANCED HAZARDOUS GAS DETECTION (CONTINUED)

Progress (Continued):

Sample Delivery System

- Preliminary Design complete, component testing in work
- Key elements used successfully on HUMS

Data Acquisition and Control System

- 68040 processor running 0S9 operating system
- Expert system on separate processor, advisory only except troubleshooting
- Key elements used successfully on HUMS

System provides full redundancy of all active components

H2 LEAK DETECTION
AT NASA-MSFC

WILLIAM T. POWERS
NASA MARSHALL SPACE FLIGHT CENTER

WILLIAM NIEBERDING

HYDROGEN SENSOR TECHNOLOGY AT NASA LEWIS

<u>OBJECTIVE</u>: TO DEVELOP AND TEST POINT CONTACT HYDROGEN SENSORS OPERABLE OVER A WIDE TEMPERATURE RANGE AND ABLE TO DETECT LEAKS FROM POSSIBLY CRYOGENIC HYDROGEN SOURCES.

I. ESTABLISHMENT OF EXPANDED CALIBRATION FACILITY

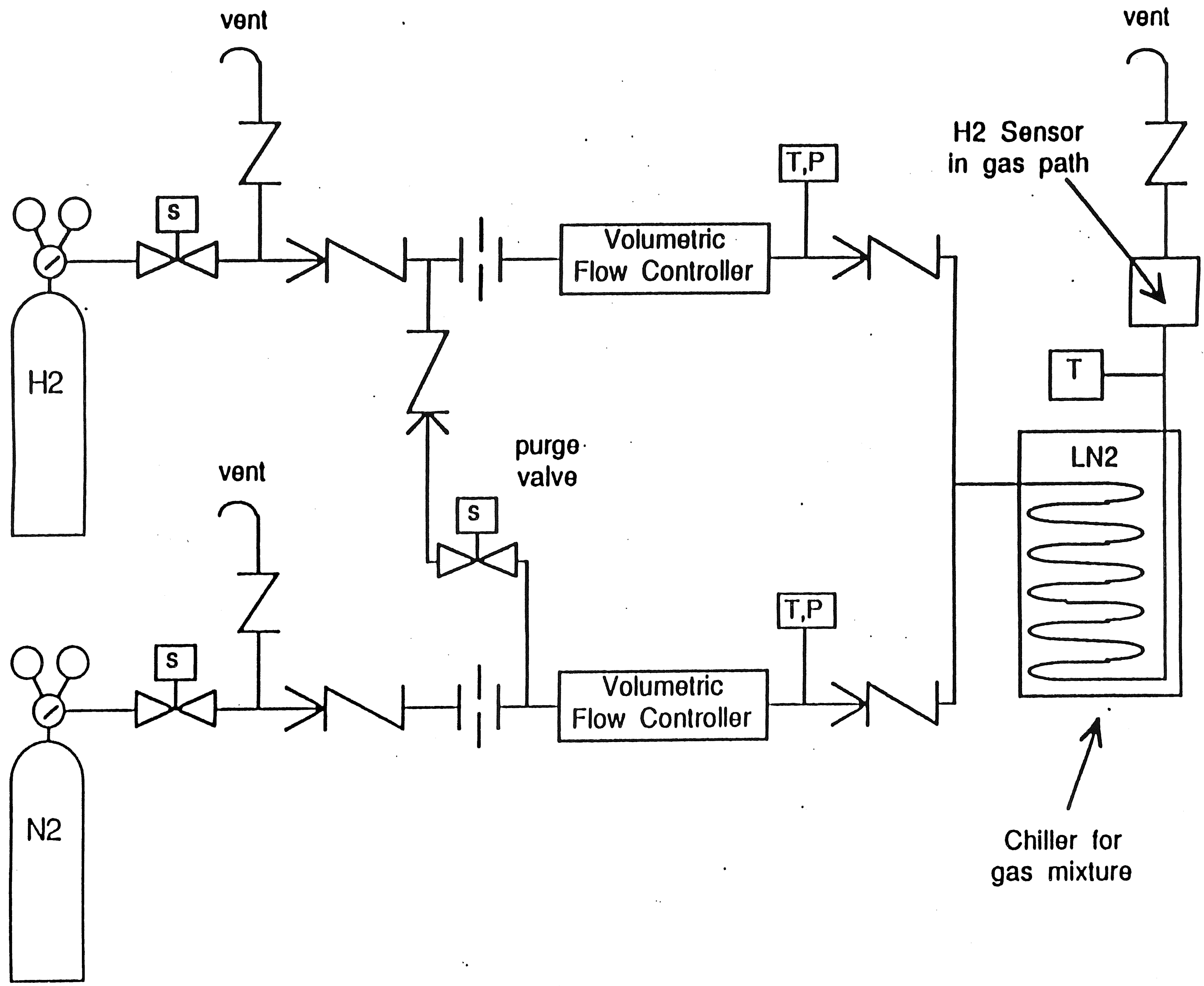
- COMPUTER CONTROLLED FLOW AND DATA TAKING
- MASS SPECTROMETER

II. CHARACTERIZATION OF HYDROGEN DETECTION SYSTEMS

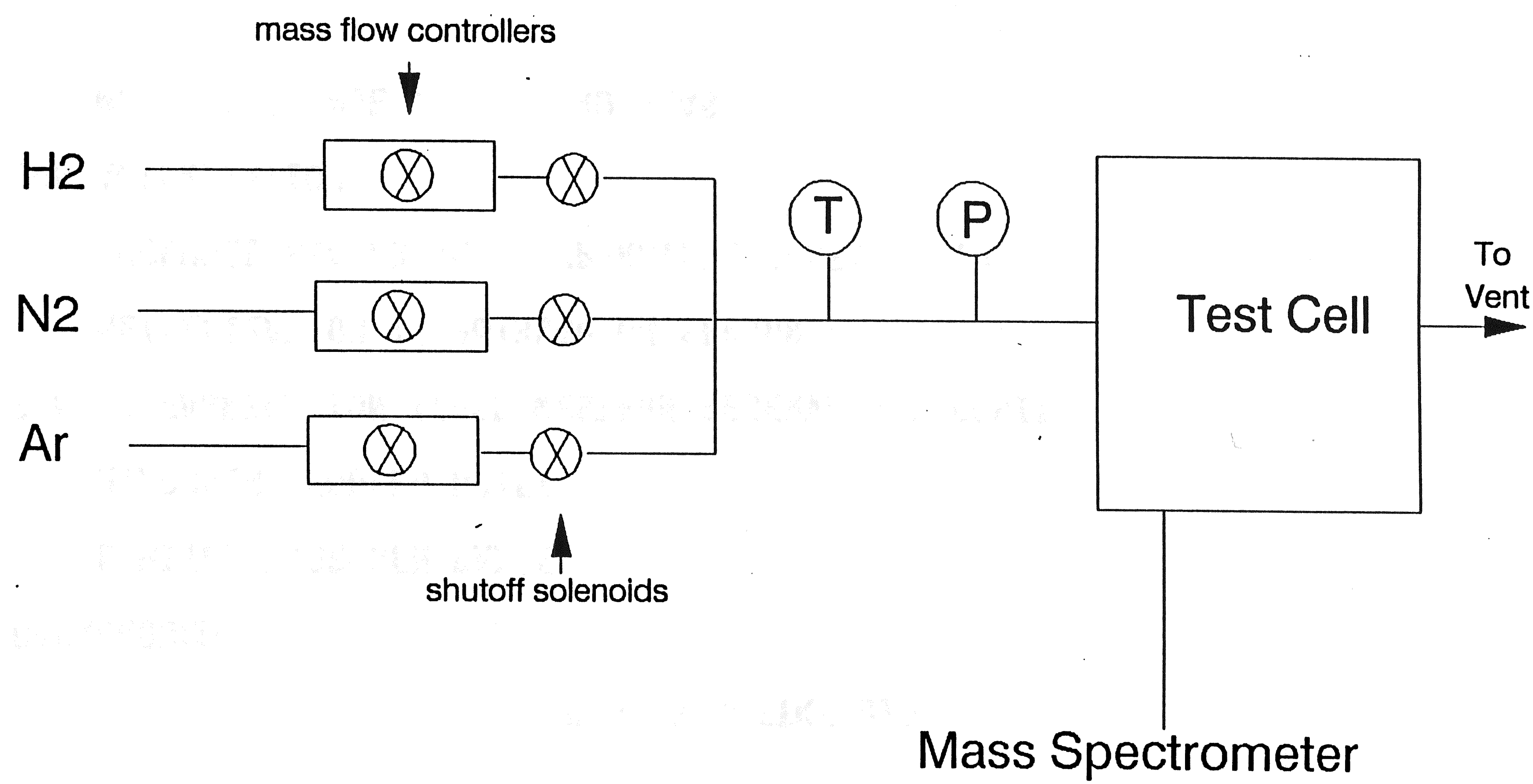
THREE COMPONENTS

- PdAg SCHOTTKY DIODES
- PdCr AS A HYDROGEN SENSITIVE ALLOY
- SiC AS A SEMICONDUCTOR FOR MOS DEVICES

CURRENT SYSTEM



EXPANDED SYSTEM



PdAg SCHOTTKY DIODES

BACKGROUND

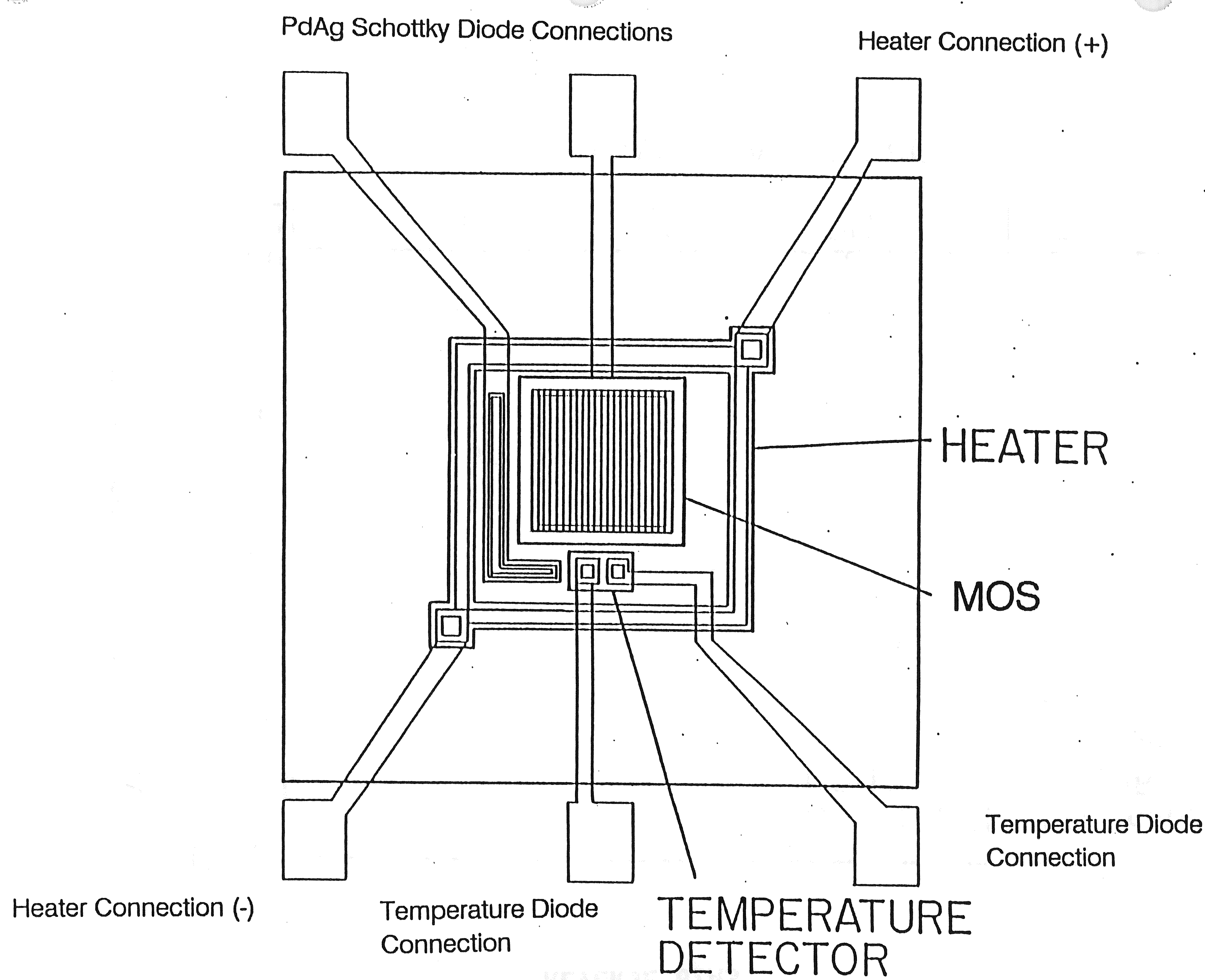
- LIMITED HYDROGEN RANGE
- HYDROGEN INDUCED DRIFT

SENSOR FABRICATION (CASE WESTERN RESERVE UNIVERSITY)

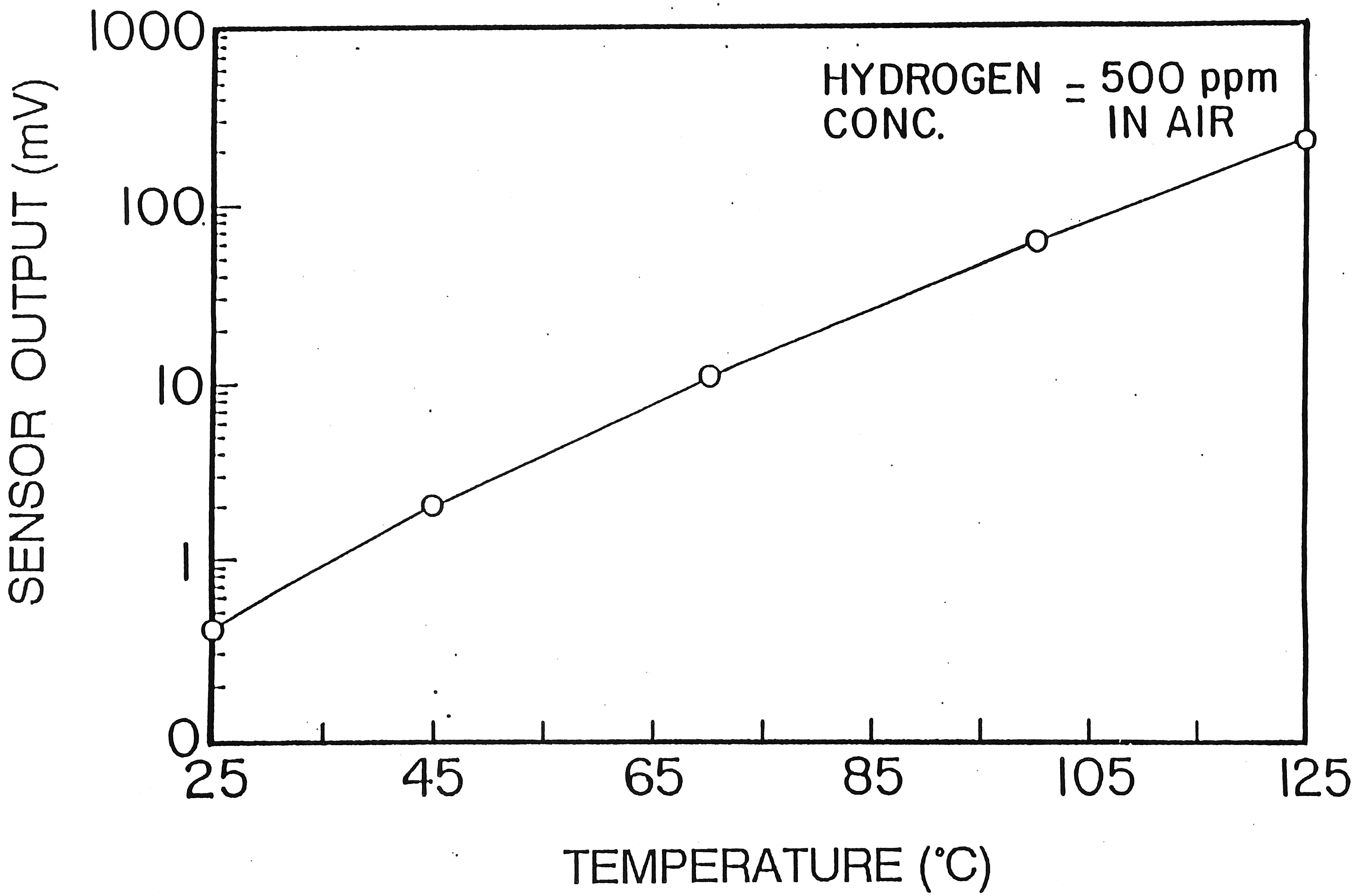
- METALLIZATION IS Pd13%Ag ON SENSOR
- INCLUDES HEATER AND TEMPERATURE SENSING DIODE

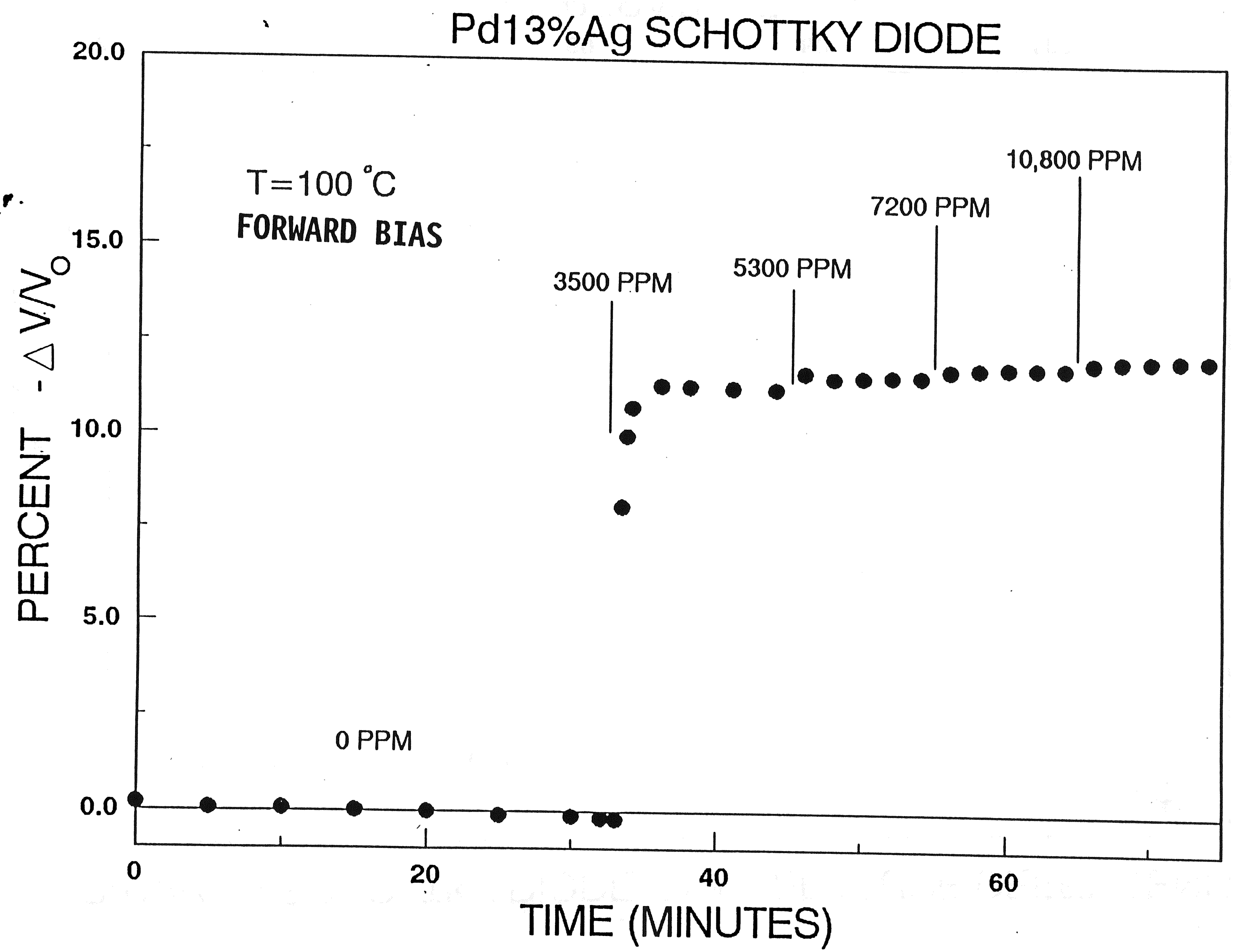
CHARACTERIZATION

- MEASURE CHANGE IN FORWARD BIAS

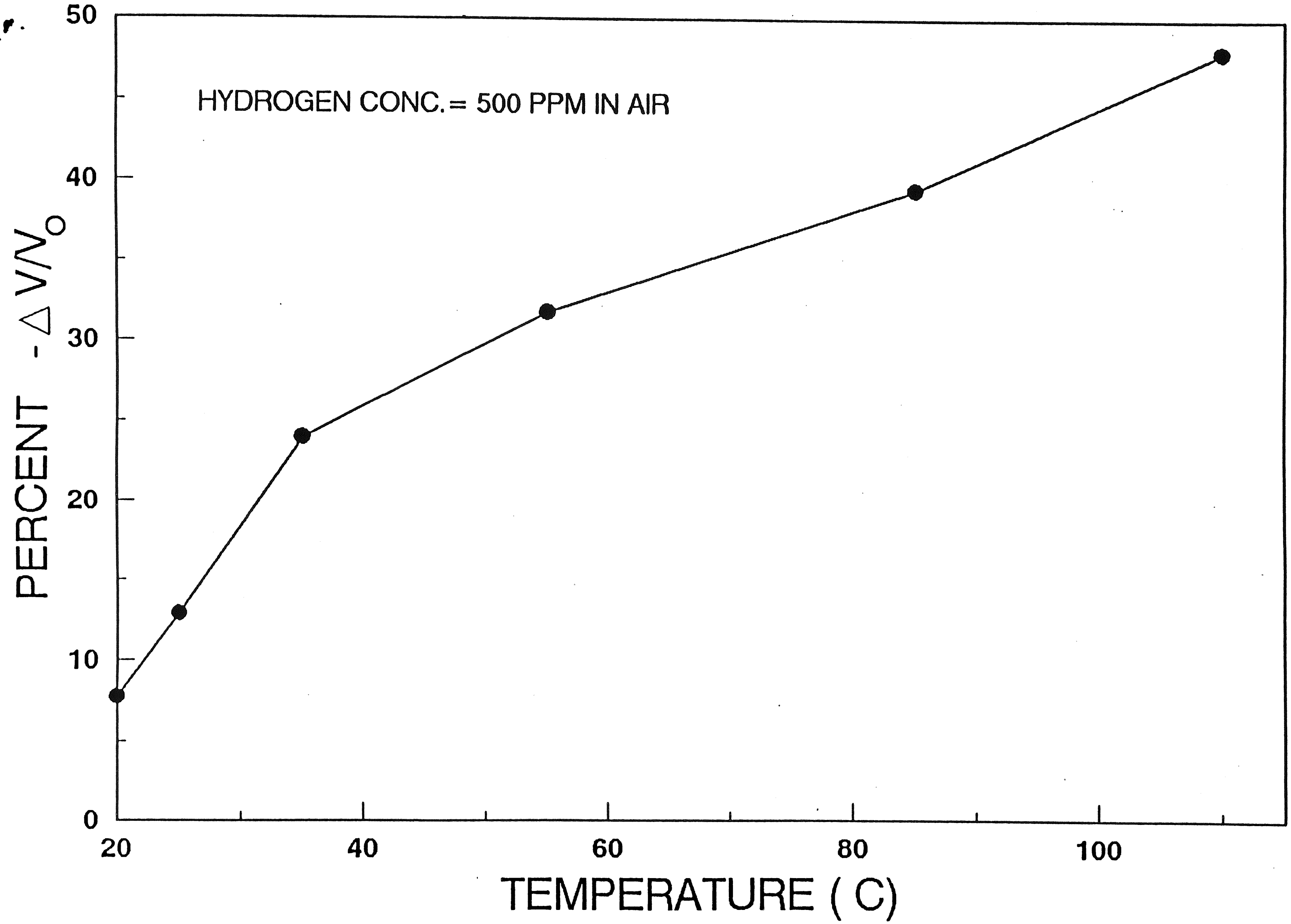

PdAg Schottky Diode Connections
Heater Connection (+)
HEATER
MOS
Temperature Diode Connection
Heater Connection (-)
Temperature Diode Connection
TEMPERATURE DETECTOR

PD13%AG SCHOTTKY DIODE
REVERSE BIAS




Pd13%Ag SCHOTTKY DIODE
T=100 °C
FORWARD BIAS
3500 PPM
5300 PPM
7200 PPM
10,800 PPM
0 PPM
PERCENT -ΔV/V_O
20.0
15.0
10.0
5.0
0.0
0
20
40
60
TIME (MINUTES)

Pd13%Ag SCHOTTKY DIODE: TEMPERATURE DEPENDENCE



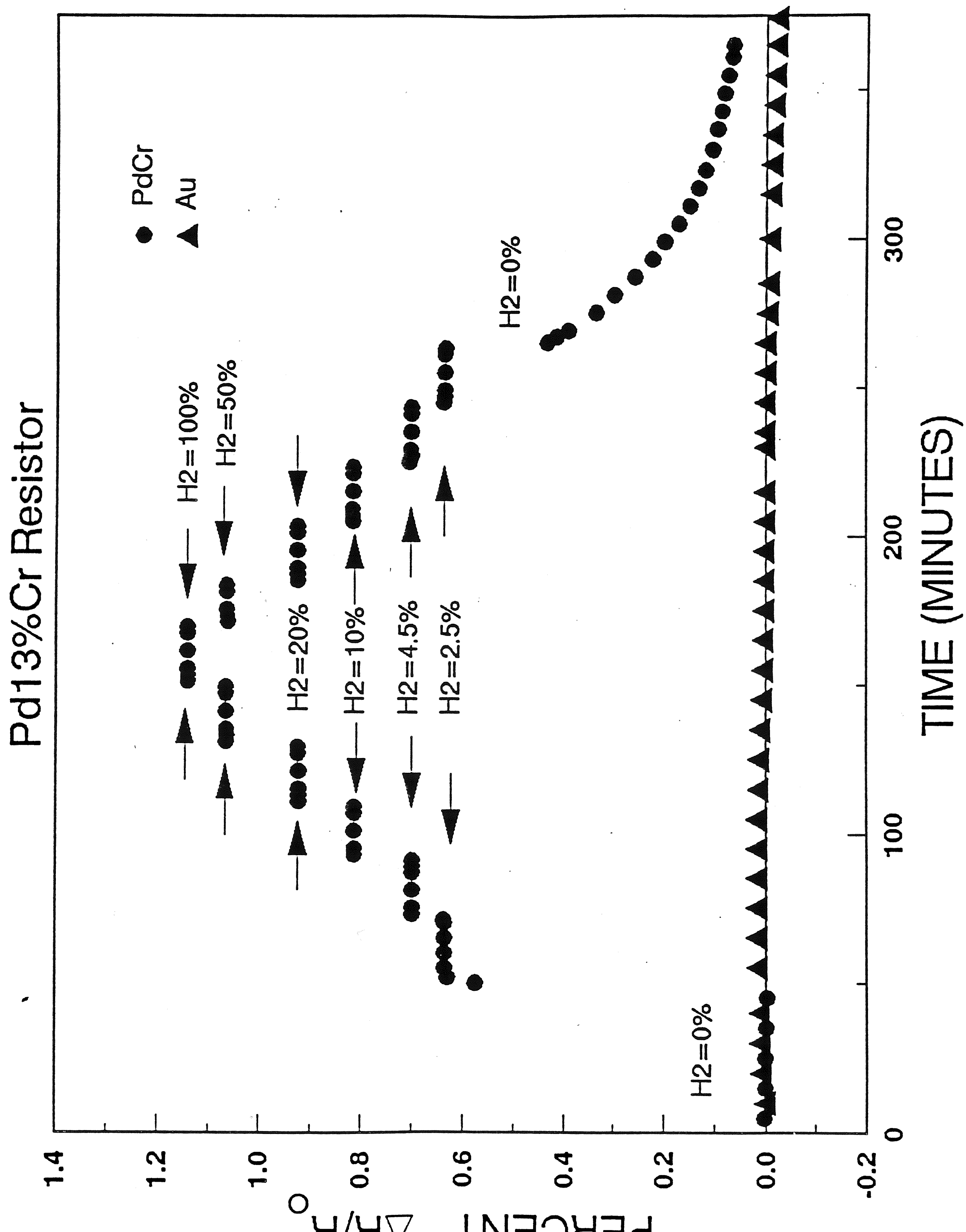
CHARACTERIZATION OF PdCr

BACKGROUND

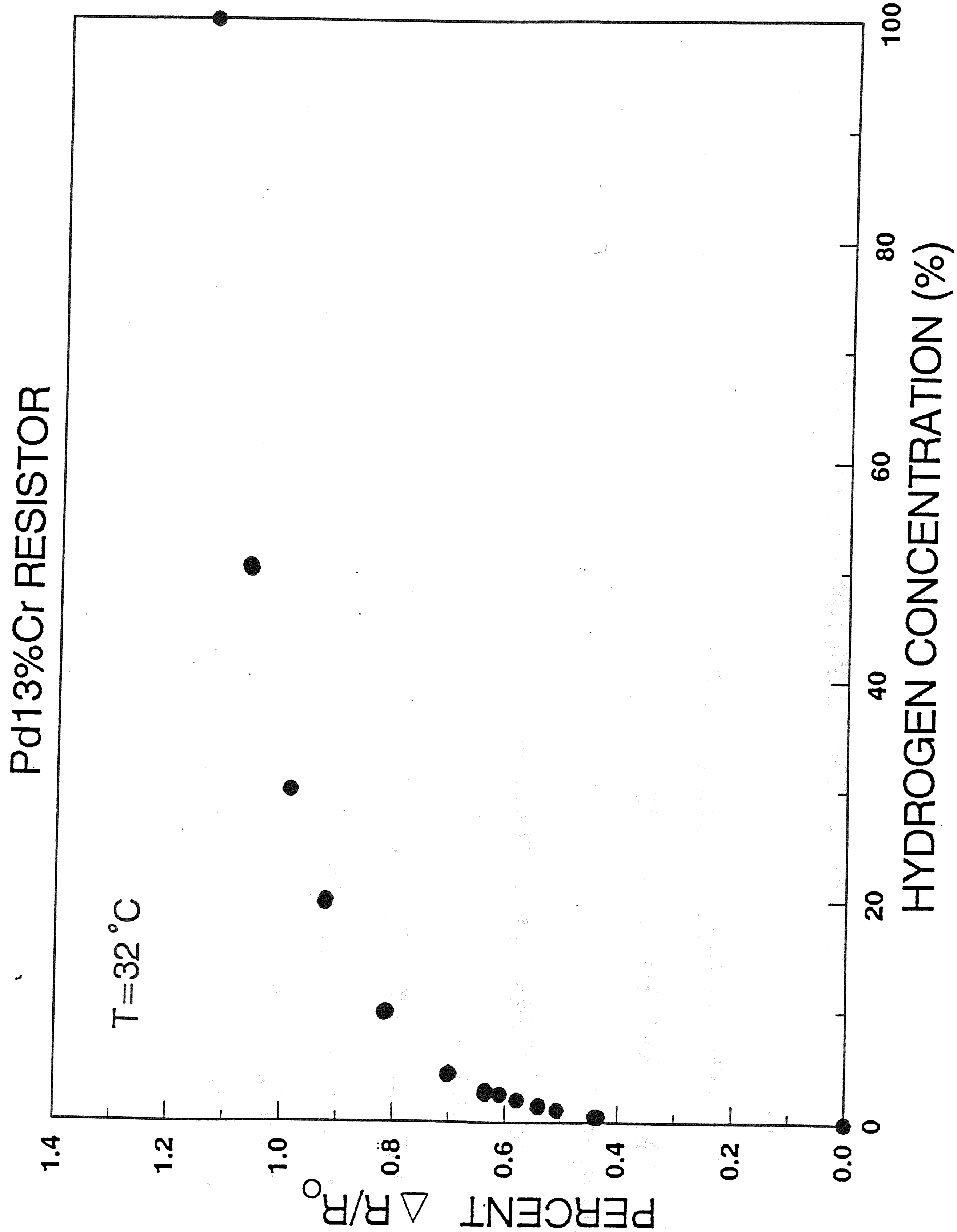
- RECENTLY DEVELOPED STRAIN GAGE MATERIAL (Pd13%Cr)
- ELECTRICALLY AND MECHANICALLY STABLE OVER WIDE TEMPERATURE RANGE

CHARACTERIZATION

- RESISTANCE CHANGE WITH H_2 IN FLOWING N_2 OF Pd13%Cr AND OF Au REFERENCE ON Al_2O_3



Pd13%Cr Resistor
PdCr
Au
H2=0%
H2=100%
H2=50%
H2=20%
H2=10%
H2=4.5%
H2=2.5%
H2=0%
PERCENT ΔR/R$_o$
TIME (MINUTES)
1.4
1.2
1.0
0.8
0.6
0.4
0.2
0.0
-0.2
0
100
200
300



Pd13%Cr RESISTOR
T=32 °C
PERCENT △ R/Ro
HYDROGEN CONCENTRATION (%)
0.0
0.2
0.4
0.6
0.8
1.0
1.2
1.4
0
20
40
60
80
100

SiC AS A SEMICONDUCTOR FOR MOS DEVICES

BACKGROUND

- HIGH TEMPERATURE ELECTRONICS PROGRAM
- FUNCTIONAL TO T> 600 °C

ADVANTAGES

- HIGH TEMPERATURE OPERATION
- THERMAL SHOCK
- THERMAL CONDUCTIVITY
- LESS LEAKAGE CURRENT

CHARACTERIZATION

- PdCr OR PdAg AS MOS METALLIZATION

PLANS

- PdAg SCHOTTKY DIODES
  - FORWARD BIAS AND REVERSE CURRENT IN H_2
  - LONG TERM HYSTERESIS
  - TEMPERATURE SENSITIVITY
  - CRYOGENIC CAPABILITY
- CHARACTERIZATION OF PdCr
  - RESISTANCE CHANGE WITH H_2 IN FLOWING N_2
    - PdCr AND Au ON AL_2O_3 AND SAPPHIRE
  - FREQUENCY CHANGE OF THICKNESS MONITOR
    - PdCr ON QUARTZ WHERE H_2 ADSORPTION CHANGES FREQUENCY
- SiC AS A SEMICONDUCTOR FOR MOS DEVICES
  - EXAMINE BEHAVIOR OF Pd-SiO_2-SiC SCHOTTKY DIODE
    - EFFECT OF H_2 ON V, I-V AND C-V CURVES
    - EXAMINE BEHAVIOR OF Pd-SiC MOSFET

Recent Advances in Solid State Hydrogen Sensors

R.C. Hughes and W. K. Schubert

Sandia National Labs,
Albuquerque, NM, 87185

Collaborators:

Device Fabrication: **Joe Pierce, Ned Godshall**
Jose Rodriguez, **Sarah Everist, Barb Wampler**

UHV Studes: **Bob Bastasz, Walt Ellis** (LANL)
Tony Ricco, **Bob Rye**

Device Testing: Mark Jenkins

Metallizations: Ric Buss

Microsensors for the Space Program

Hydrogen Sensors

1. Shuttle fueling and burn
2. Orbital Transfer Vehicle
3. Advanced Launch Vehicle
4. National Aerospace Plane
5. Fuel Cells

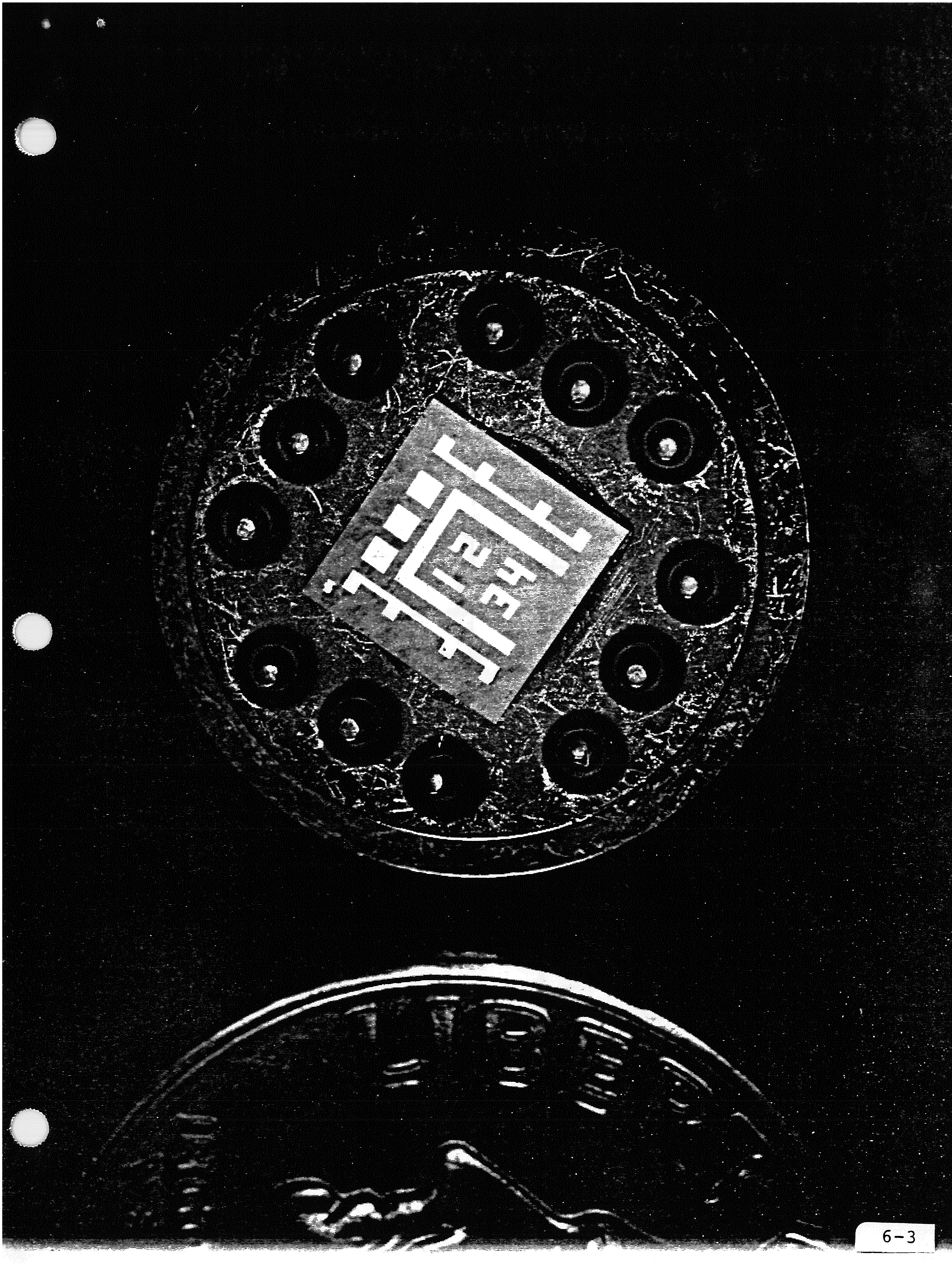
Sensors for the Life Support Tech Program

see JPL Meeting, 11/6/91
Peggy Evanich, Program Manager

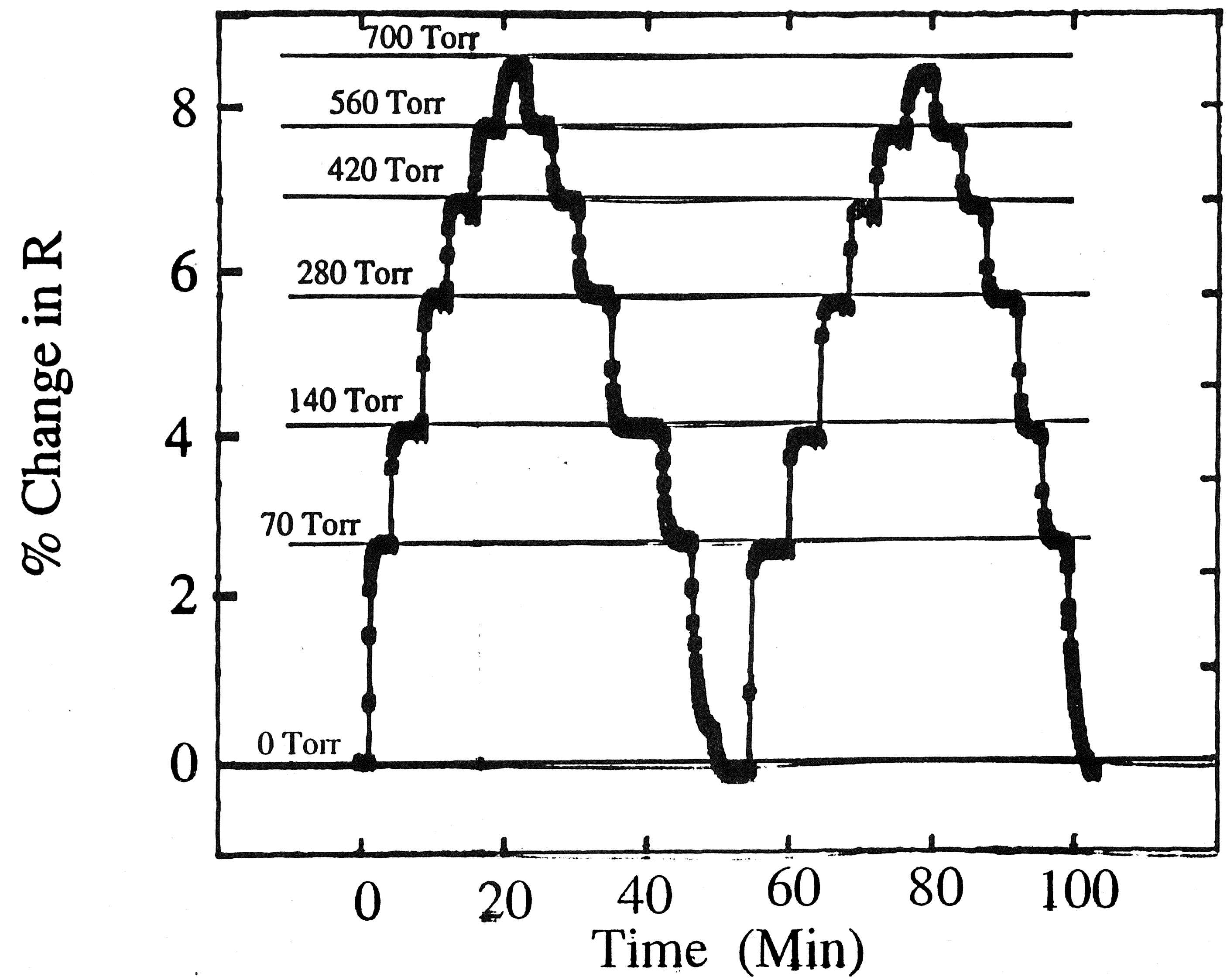
Physical Sensors

Radiation-RADFET array could give spectrum

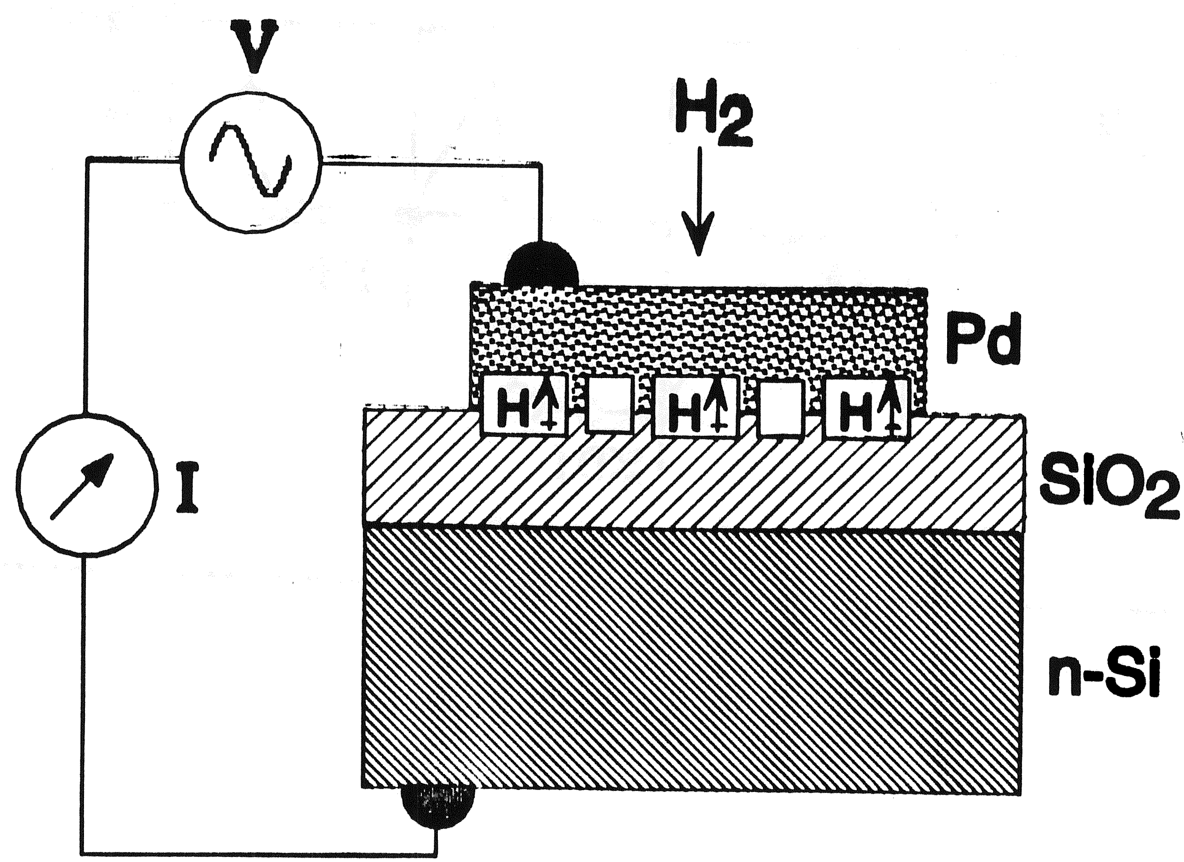
Viscosity- acoustic wave devices have no moving parts, gravity not needed

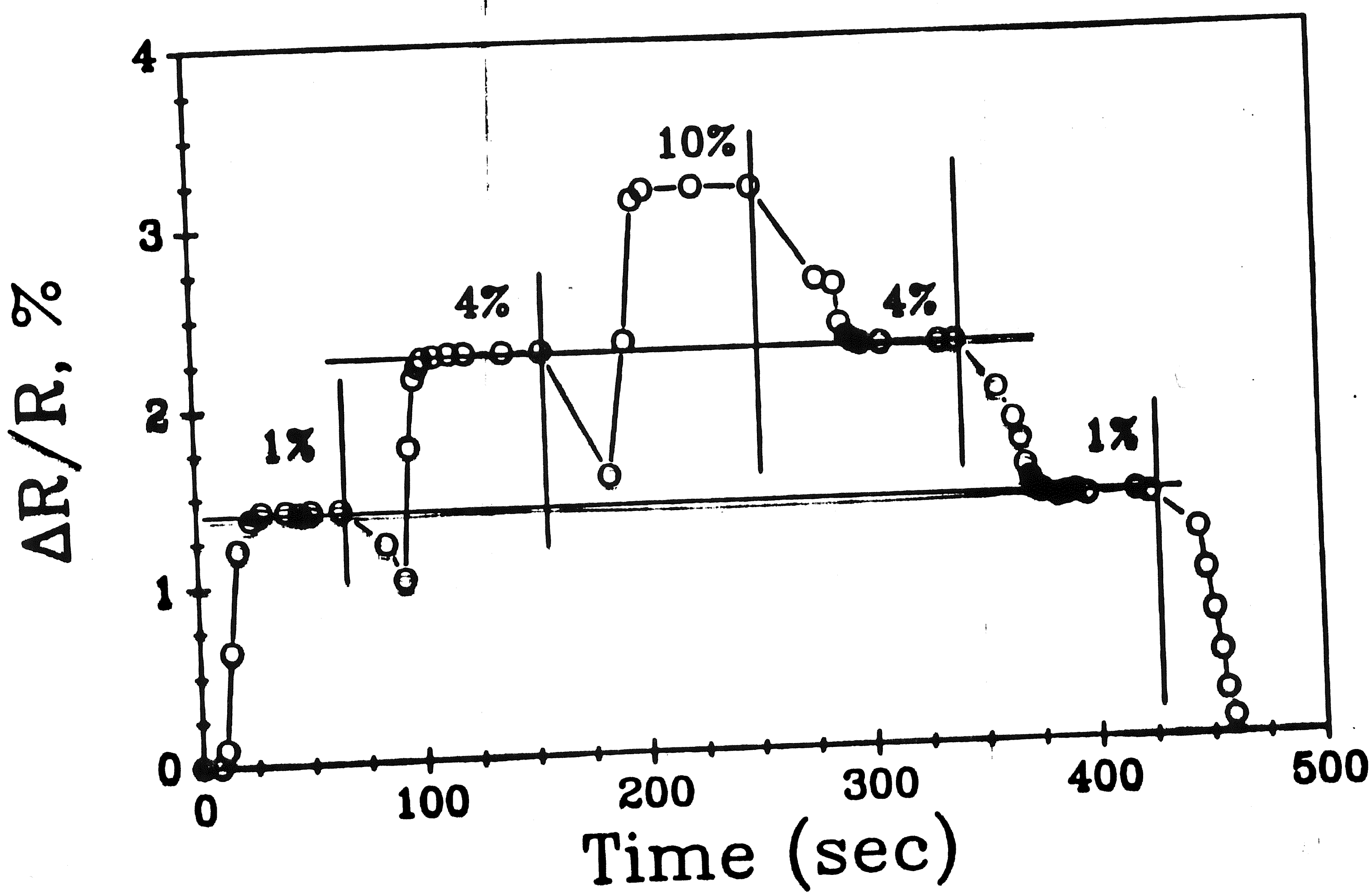


PdNi Chemi-resistor Response to H_2 at 25 C



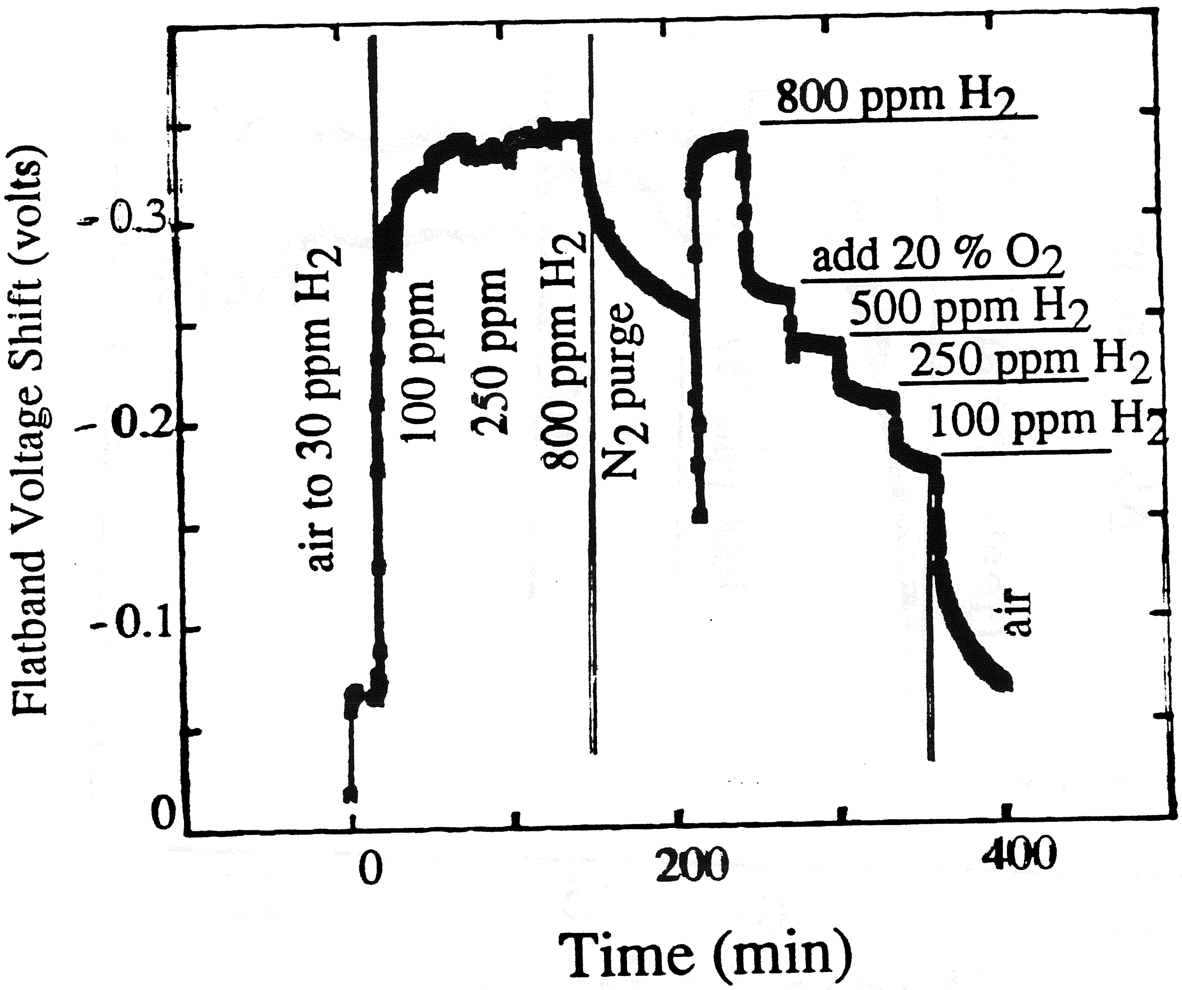
MIS Hydrogen Sensor



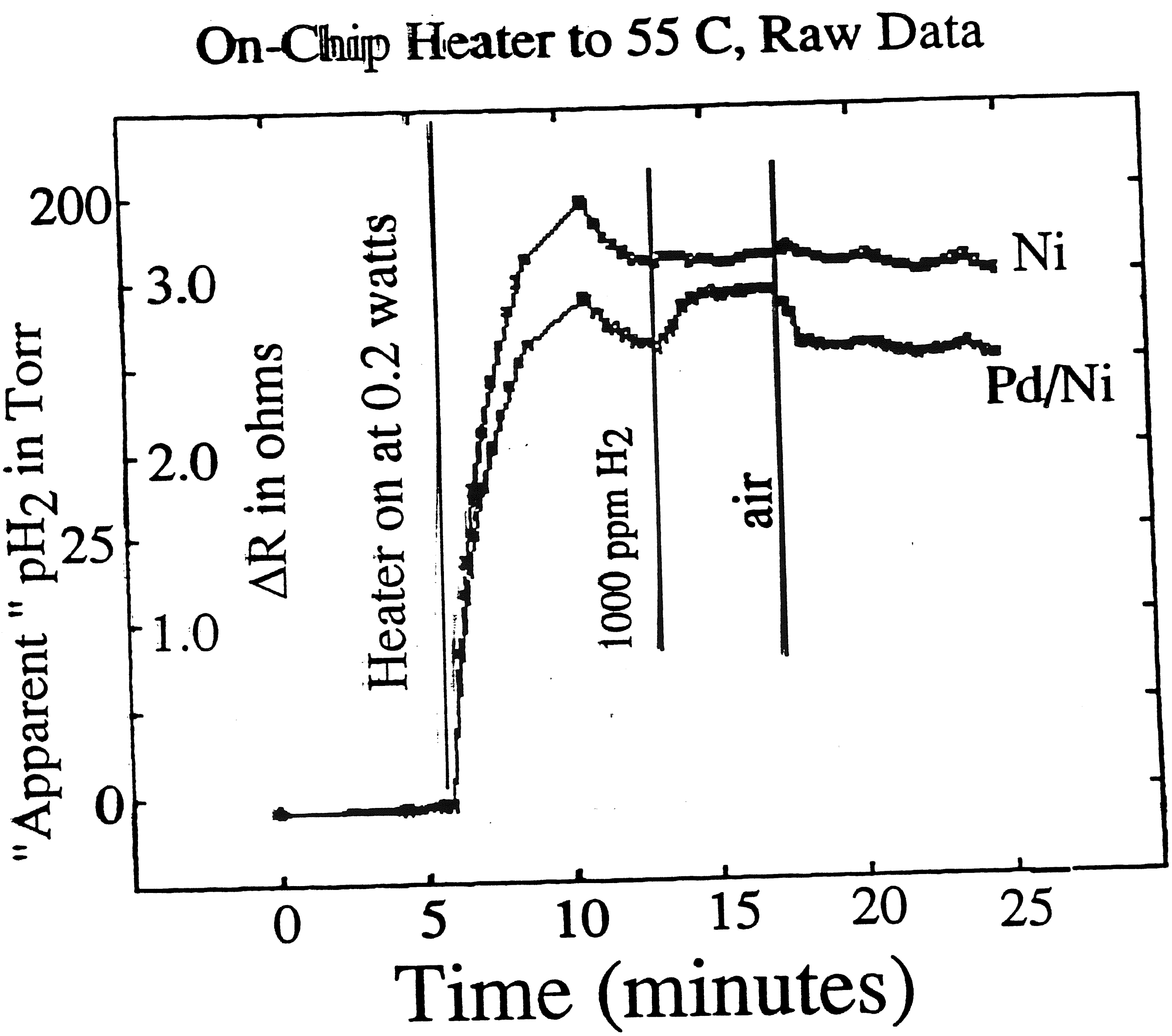


ΔR/R, %
Time (sec)
0
1
2
3
4
0
100
200
300
400
500
1%
4%
10%
4%
1%

PdNi MOS Capacitor at 100 C, H_2 Response

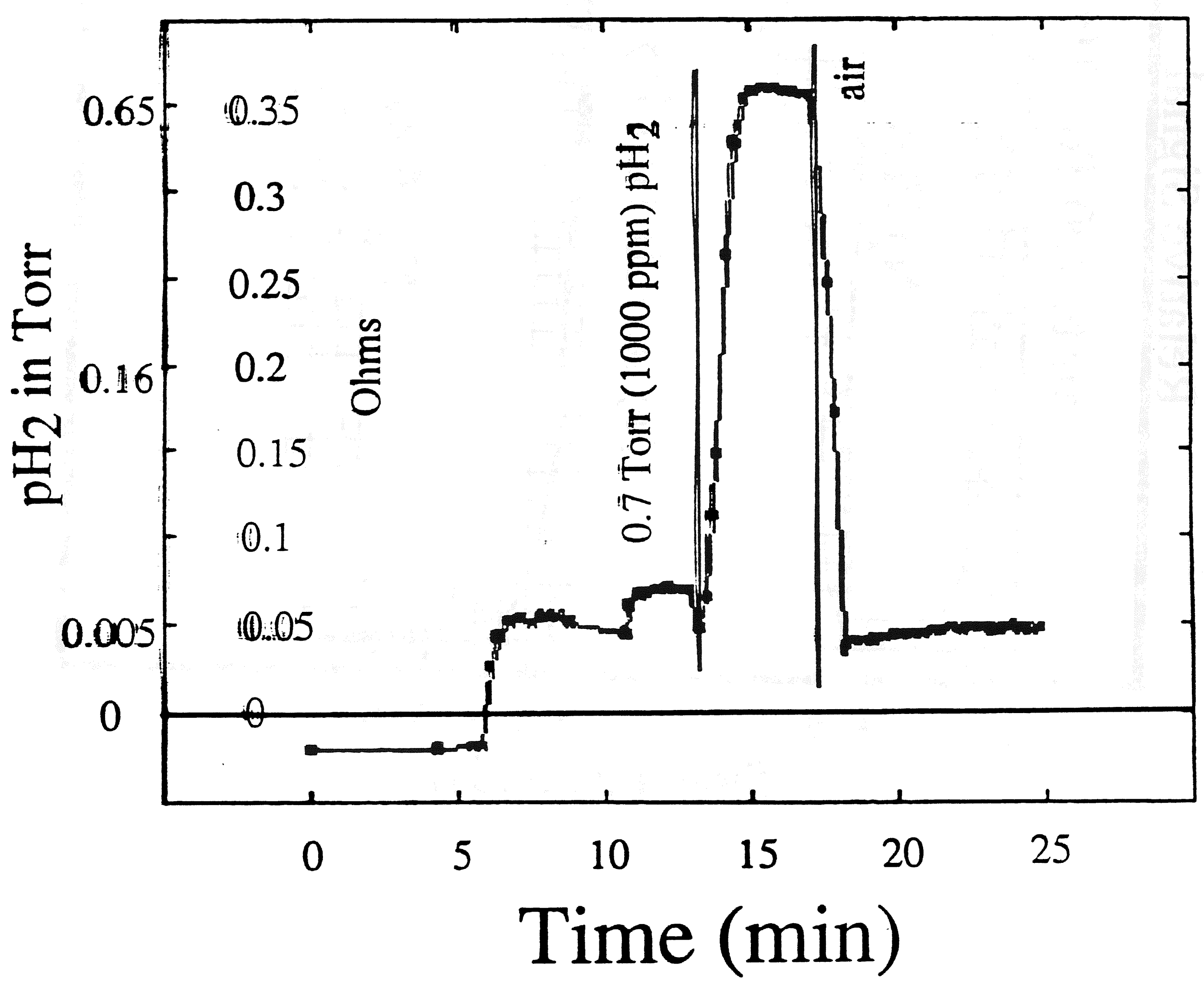


file wrs1, 1/30/92

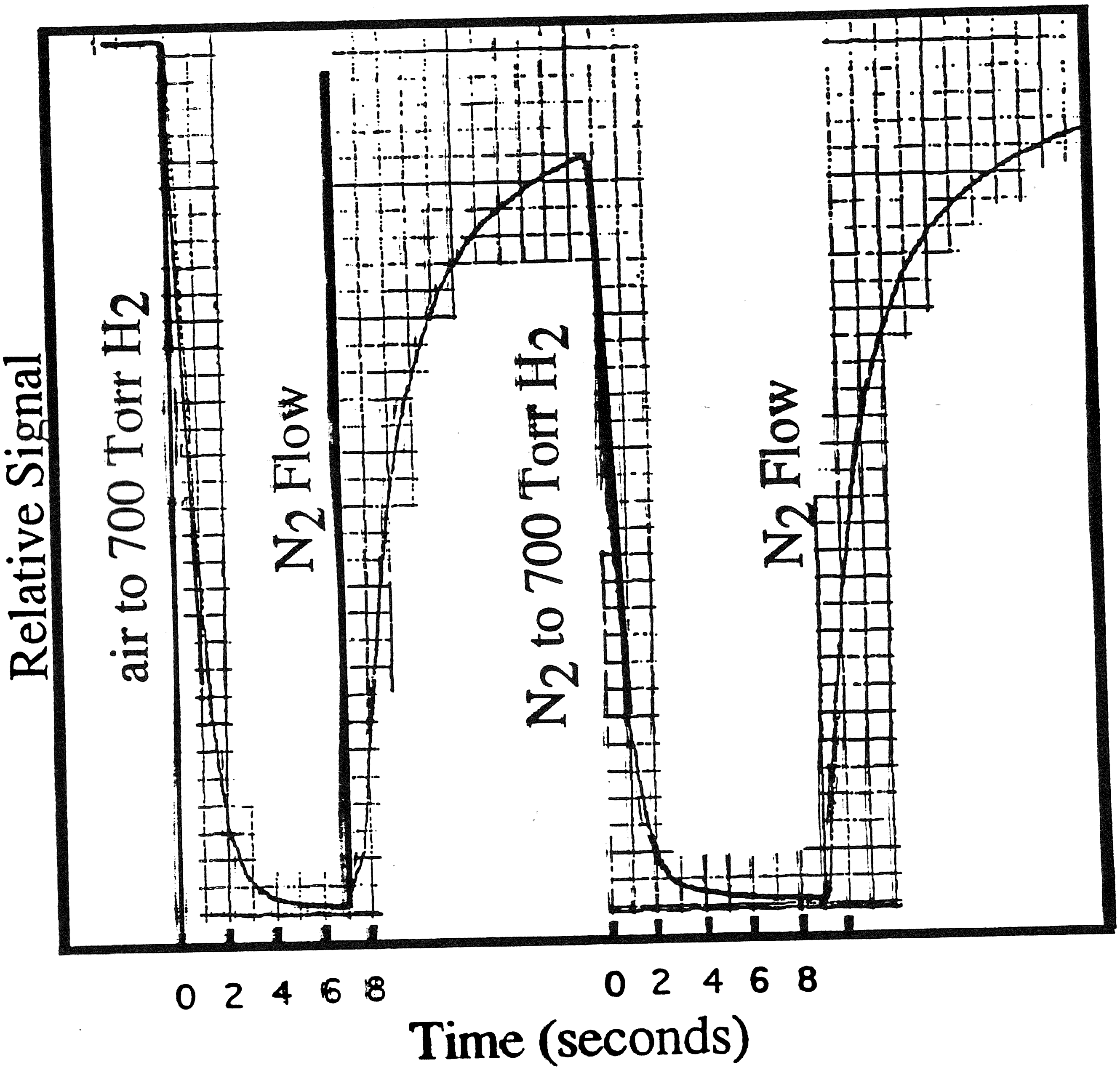


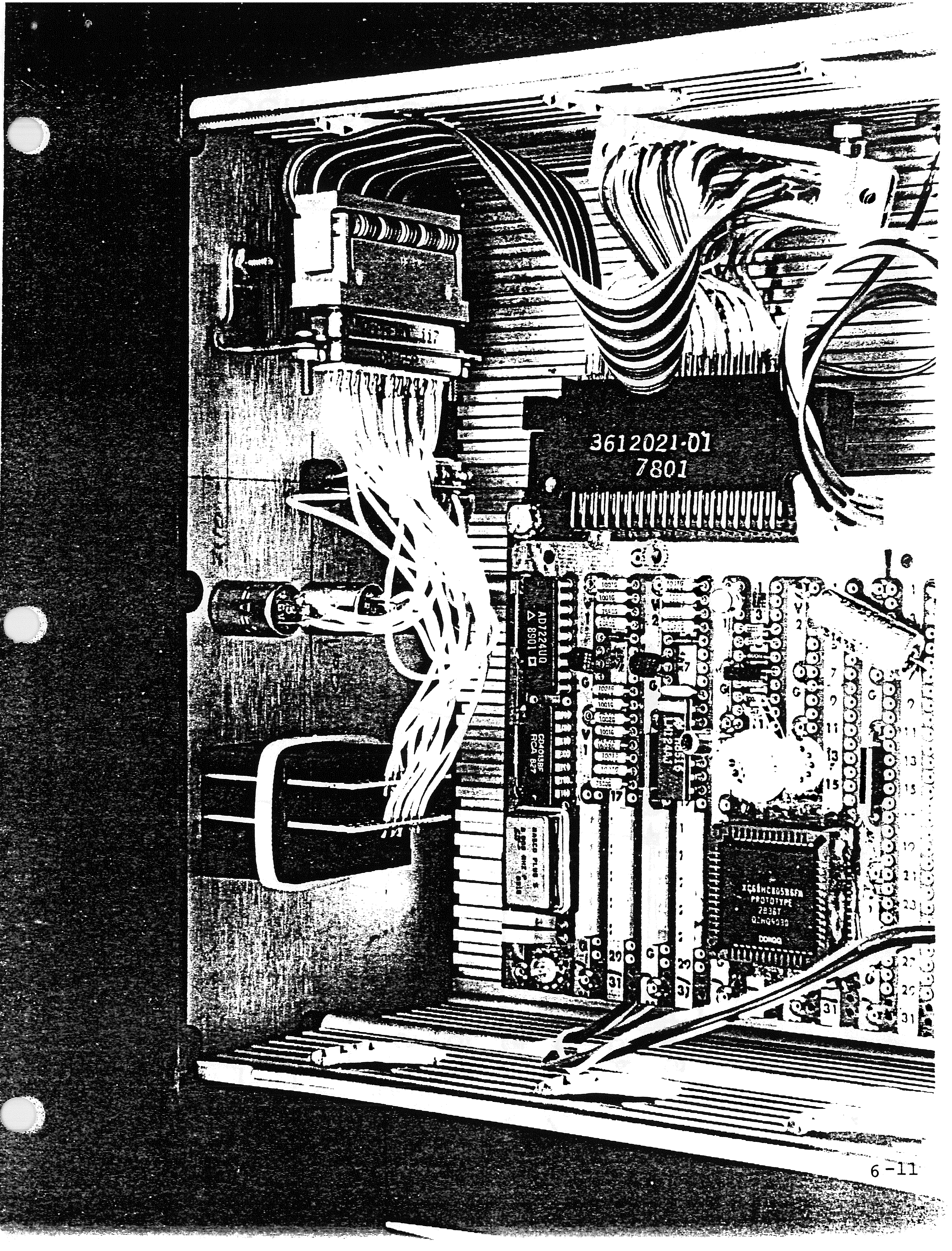
file wrs2, 1/21/92

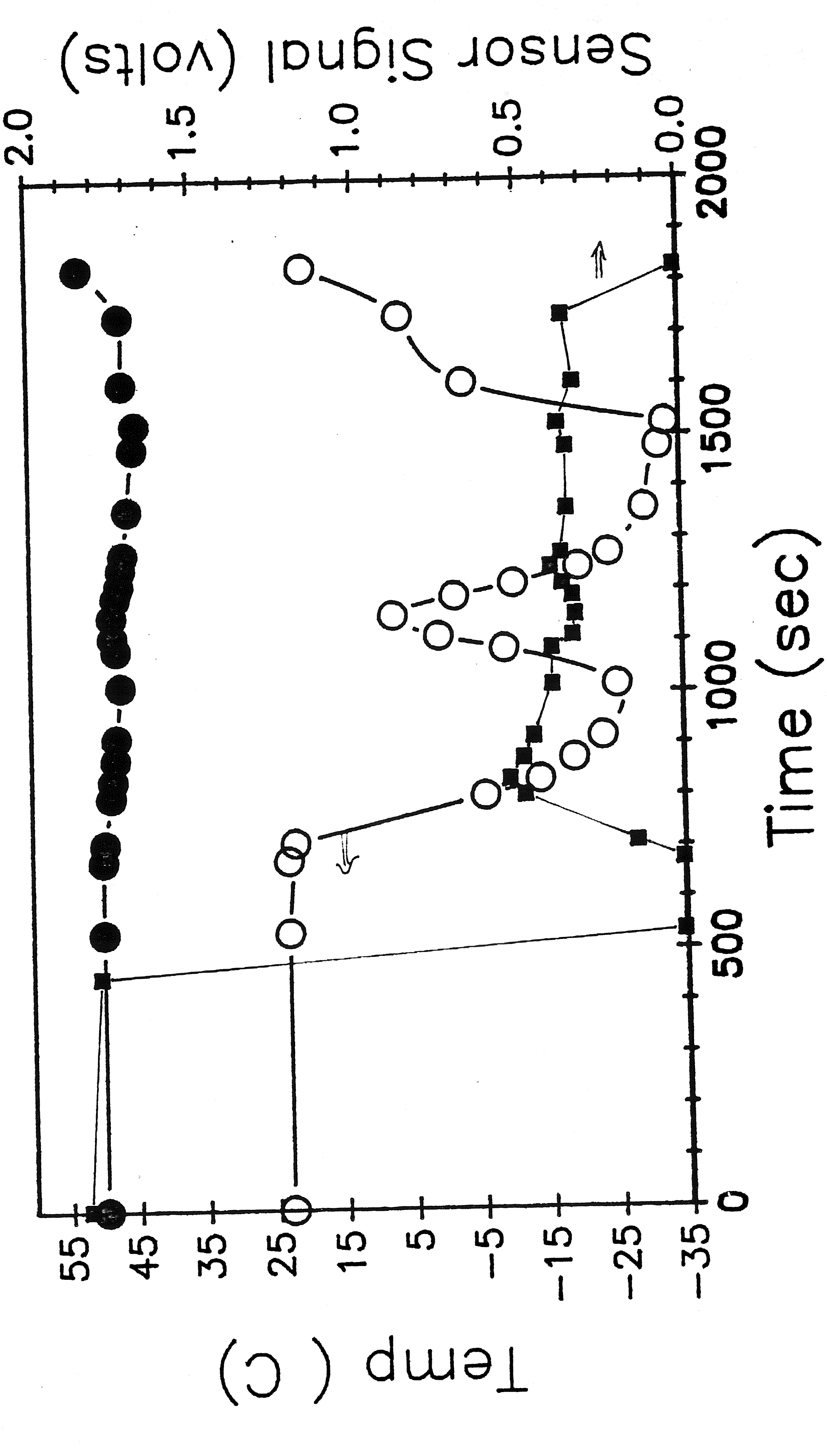
Corrected Pd/Ni Signal for Temperature



Sensor Response at 25 C to Pure H_2




3612021-01
7801
AD7224UQ
8901



Sensor Signal (volts)
2.0
1.5
1.0
0.5
0.0
Temp (C)
55
45
35
25
15
5
−5
−15
−25
−35
Time (sec)
0
500
1000
1500
2000

SANDIA ROBUST WIDE-RANGE HYDROGEN SENSOR MODULE

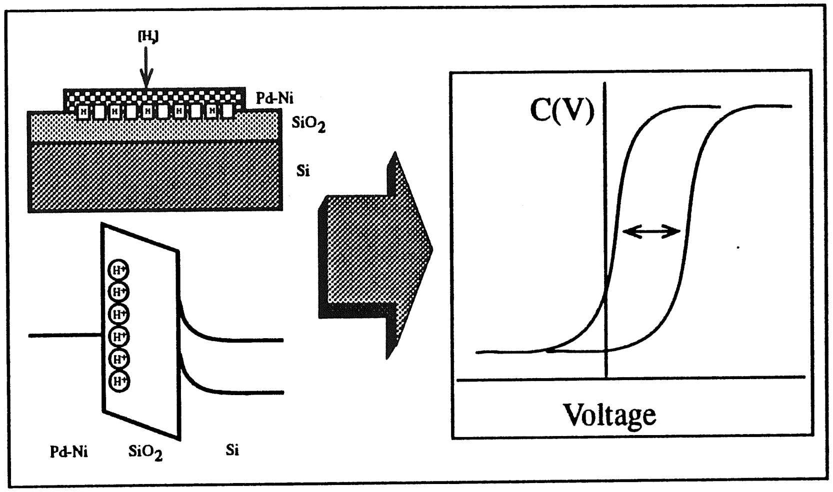
PAUL McWHORTER, JOSE RODRIGUEZ, BOB HUGHES

OUTLINE

BACKGROUND
R&D MODULE
ROBUST MODULE
INTEGRATED MODULE

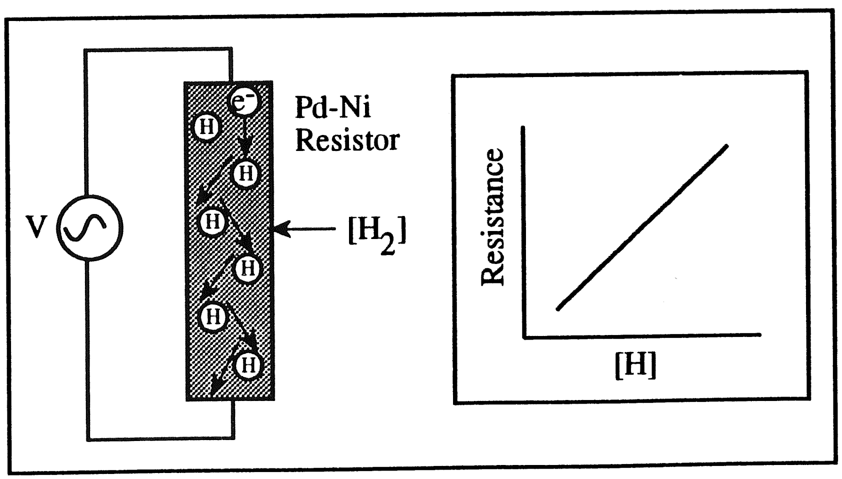
MECHANISMS TO DETECT HYDROGEN ARE WELL ESTABLISHED

Δ Capacitance



HYDROGEN IONS TRAPPED IN THE DIELECTRIC SHIFT THE CAPACITOR'S THRESHOLD VOLTAGE.

Δ Resistance

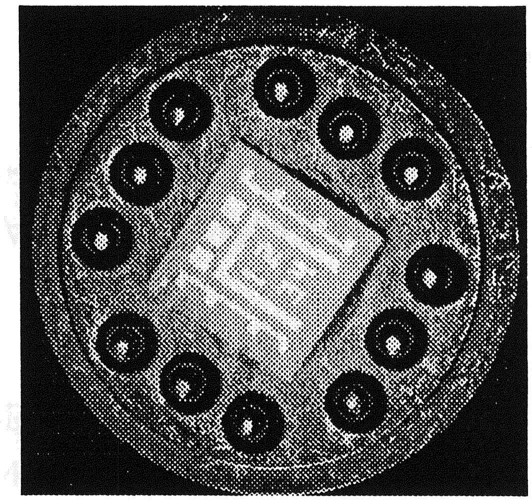


HYDROGEN ABSORBED BY THE PdNi RESISTOR INCREASES THE RESISTANCE

McWHORTER/92

WIDE RANGE R&D MODULE DEMONSTRATES FEASIBILITY

R&D MODULE IMPACT:



DEMONSTRATES FEASIBILITY

USED TO INVESTIGATE MECHANISMS

USED TO DEVELOP MICROELECTONICS PROCESSES

INEXPENSIVE TO FABRICATE (30 STEPS, NO PHOTOLITHOGRAPHY)

R&D MODULE LIMITATIONS:

NOT PRACTICAL IN MOST REAL WORLD ENVIRONMENTS

DIFFICULT TO INSTRUMENT AC CAPACITANCE MEASUREMENTS

FABRICATION PROCESS IS NOT MANUFACTURABLE

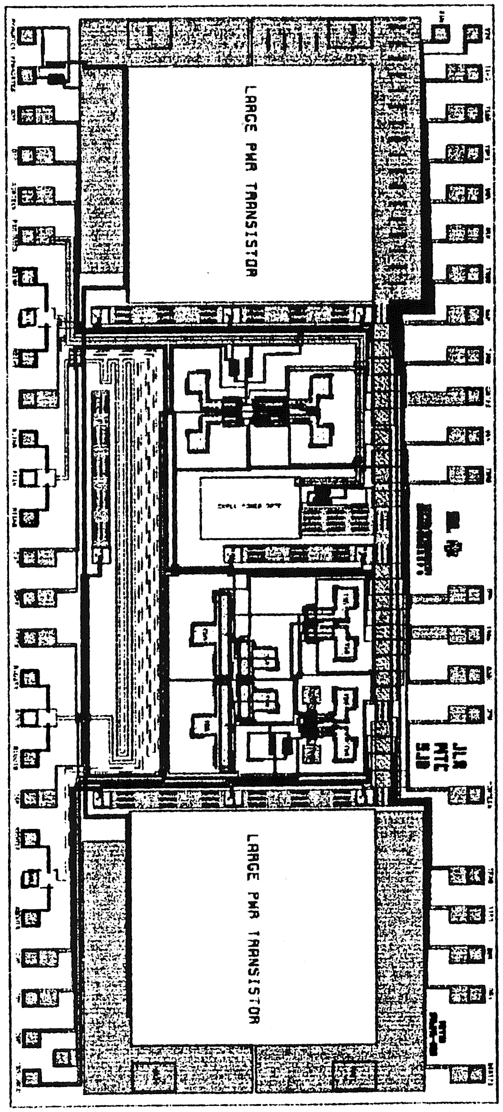
LONG TERM RELIABILITY UNKNOWN

MEASUREMENTS SENSITITVE TO LIGHT, INTERFERENCE, ELECTRICAL NOISE

McWHORTER/92

ROBUST WIDE-RANGE SENSOR MODULE

DESIGNED FOR THE RIGORS OF "REAL WORLD" APPLICATIONS



FEATURES

- PdNi GATE TRANSISTORS FOR DETECTING LOW CONCENTRATIONS OF H2
- PdNi RESISTORS FOR DETECTING HIGH CONCENTRATIONS OF H2
- POWER TRANSISTORS FOR ON BOARD HEATING
- DIODES FOR ON BOARD TEMPERATURE MONITORING

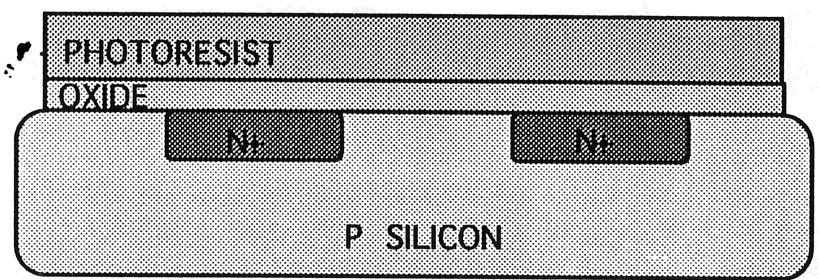
CUSTOMER MPACT

- RELIABLE ON BOARD TEMPERATURE CONTROL
- SIMPLE INSTRUMENTATION OF MEASUREMENTS
- MANUFACTURABLE
- RELIABLE
- PRACTICAL

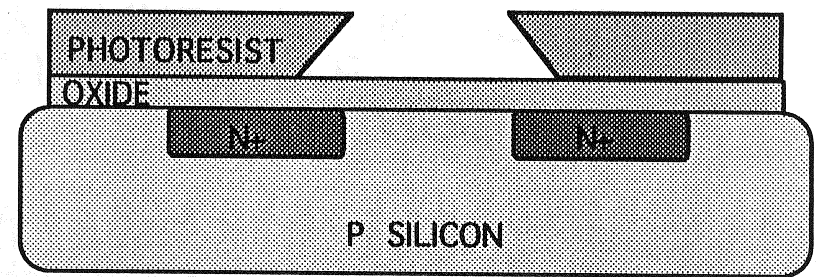
McWHORTER/92

METAL LIFT-OFF APPROACH
IC GRADE PATTERNING WITH R&D FLEXIBILITY

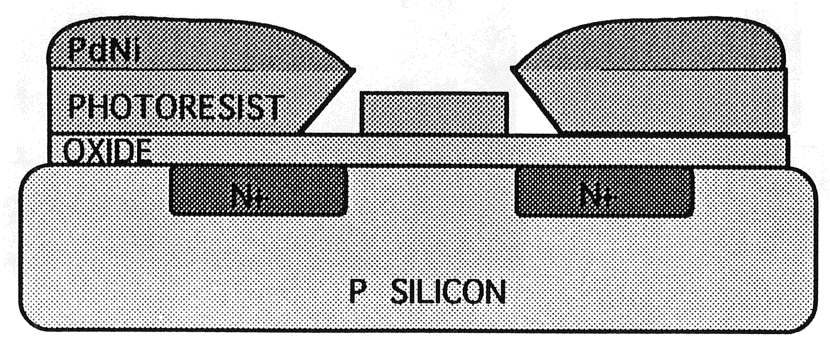
DEPOSIT PHOTORESIST



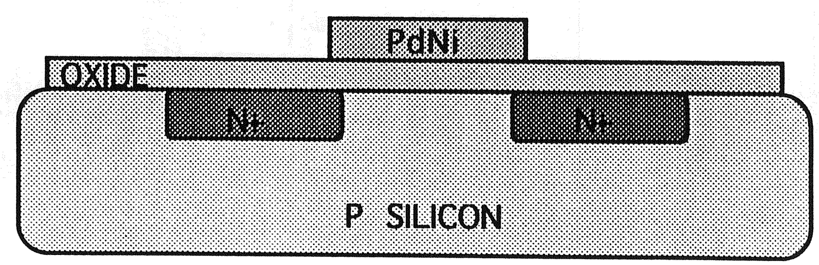
OVEREXPOSE, DEVELOP



DEPOSIT METAL



REMOVE PHOTORESIST



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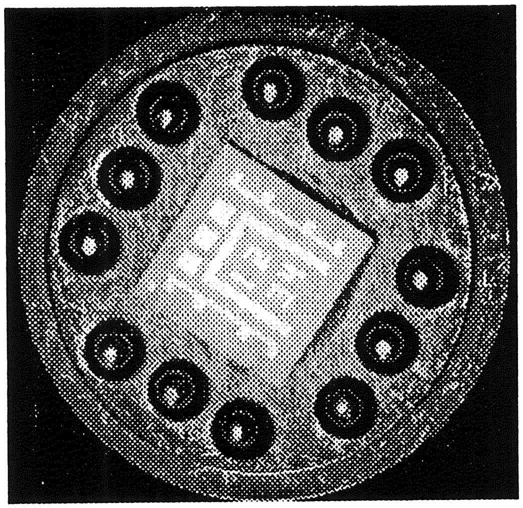
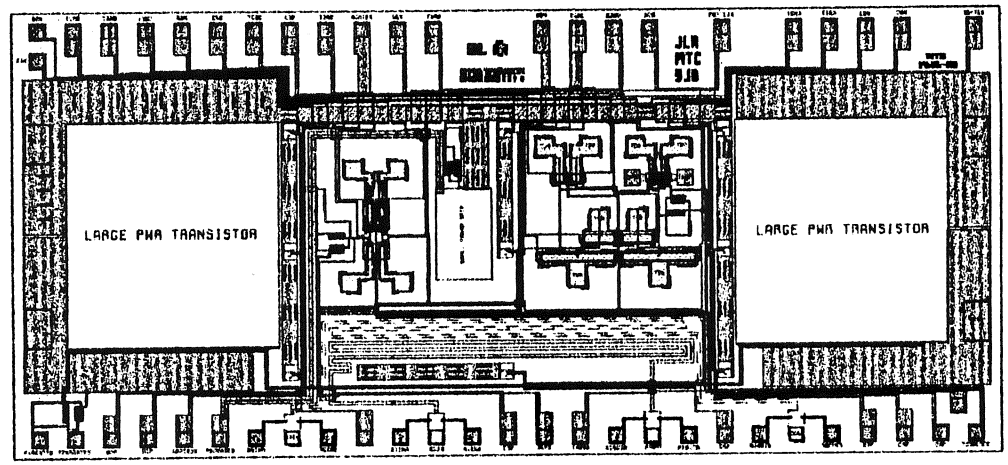
ROBUST AND R&D MODULES ALLOW EFFICIENT R-D-A



R&D MODULE

LOW COST (20K/LOT)
FAST TURNAROUND (1-2 WEEK)
30 STEPS TO FAB

NOT MANUFACTURABLE
NOT PRACTICAL OUTSIDE LAB



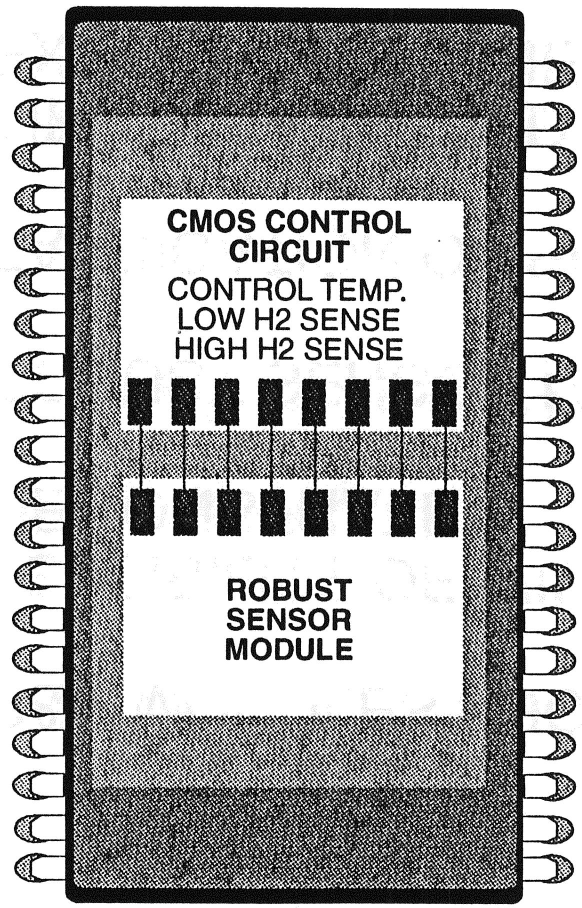
ROBUST MODULE

MANUFACTURABLE
EASY TO INSTRUMENT MEASUREMENT
PRACTICAL
RELIABLE

EXPENSIVE (100K/LOT)
SLOW TURNAROUND (3 MONTH)
300 STEPS TO FAB

McWHOR... '92

SINGLE CHIP HYDROGEN DETECTION
CMOS CONTROL CIRCUIT DESIGN UNDERWAY



FEATURES

- SENSE AND CONTROL OF SENSOR TEMP.
- LOW H_2 DETECTION
- HIGH H_2 DETECTION
- DIGITAL OUTPUT
- LIMITED SELF CALIBRATION CAPABILITY
- MONOLITHIC MODULE IF NEEDED

INITIAL DESIGN OF CONTROL CIRCUIT BEING IMPLEMENTED BY BREADBOARDING COMMERCIAL PARTS. SINGLE CHIP VERSION WILL BEGIN WHEN A SPONSOR IS IDENTIFIED.

McWHORTER/92

SUMMARY

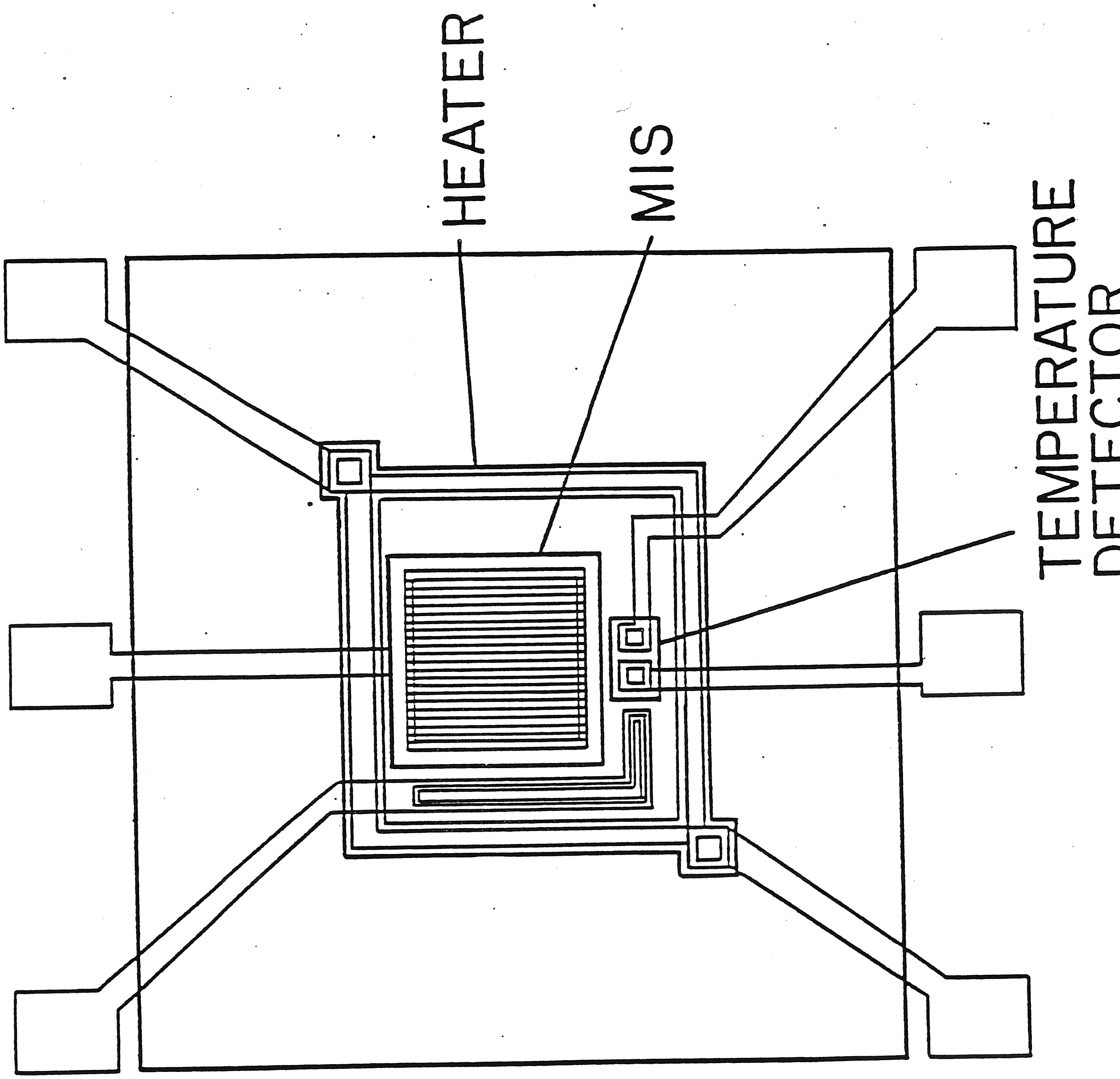
- R&D MODULES SUCCESSFULLY FABRICATED
- FEASIBILITY OF WIDE RANGE HYDROGEN DETECTION DEMONSTRATED
- ROBUST SENSOR MODULE DESIGN COMPLETE
- FABRICATION OF ROBUST MODULE UNDERWAY
- FIRST COMPLETED ROBUST MODULES EXPECTED IN JUNE
- DESIGN OF CMOS CONTROL CIRCUIT UNDERWAY

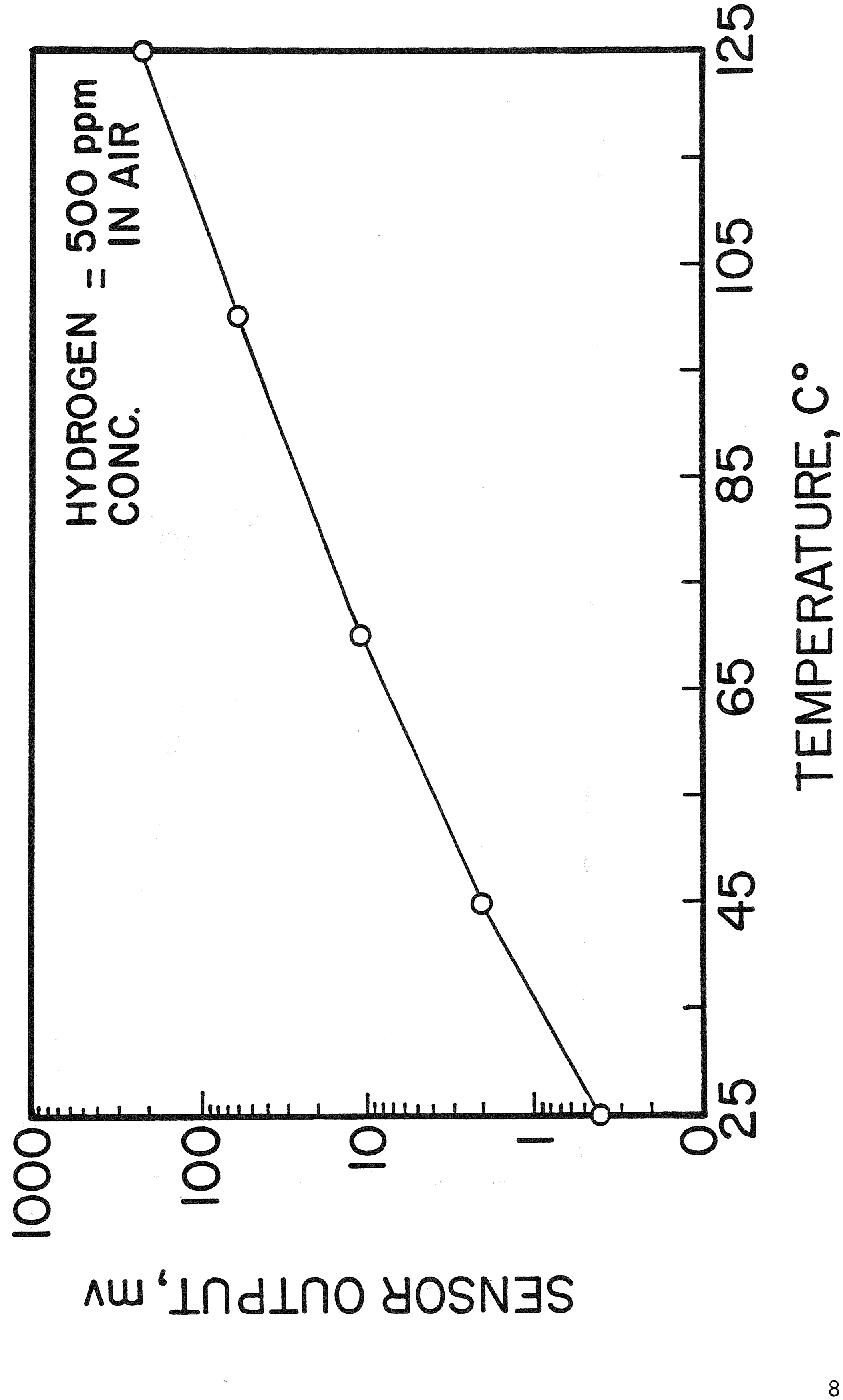
McWHORTER/92

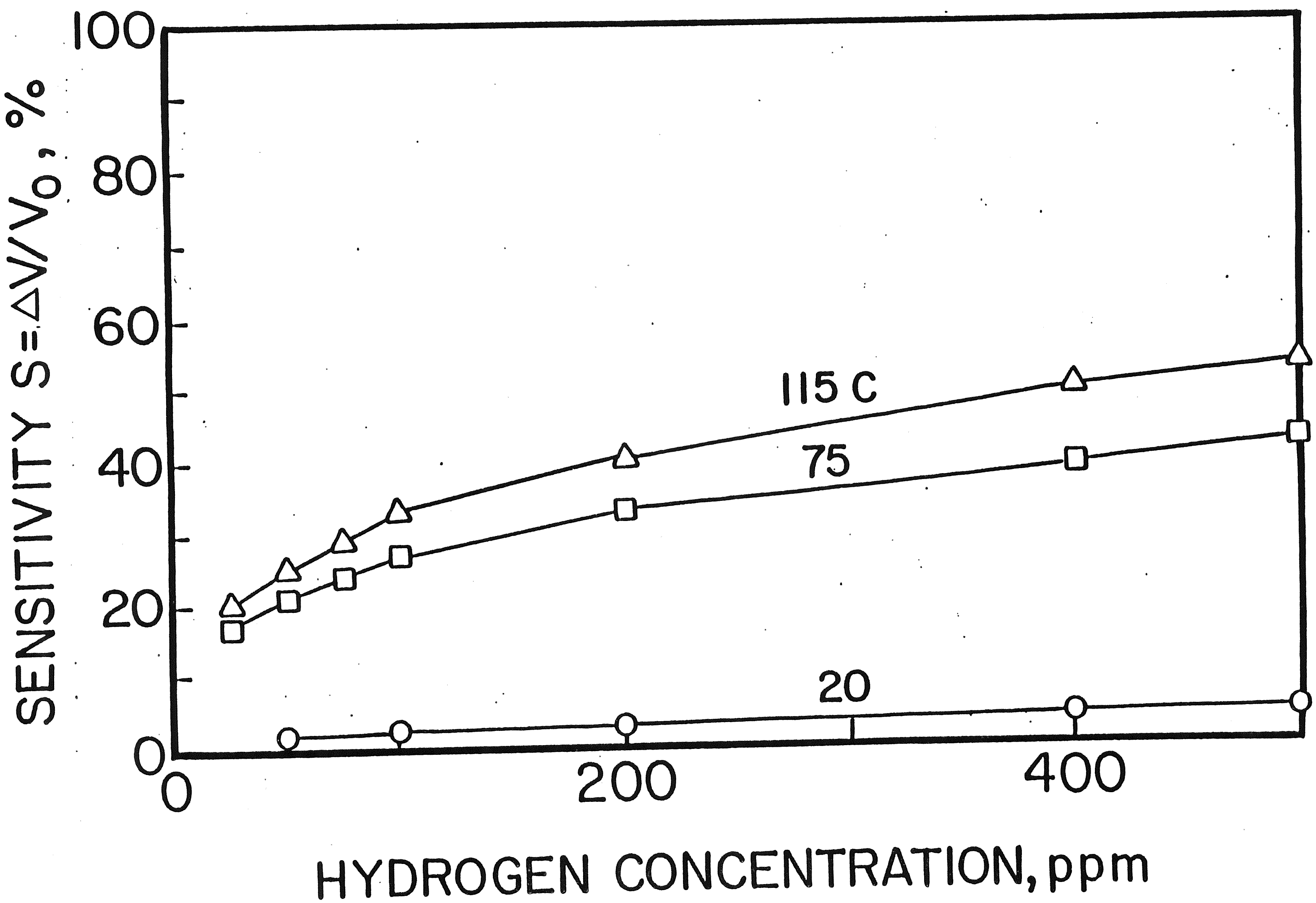
MICROFABRICATED POINT SOURCE HYDROGEN SENSOR

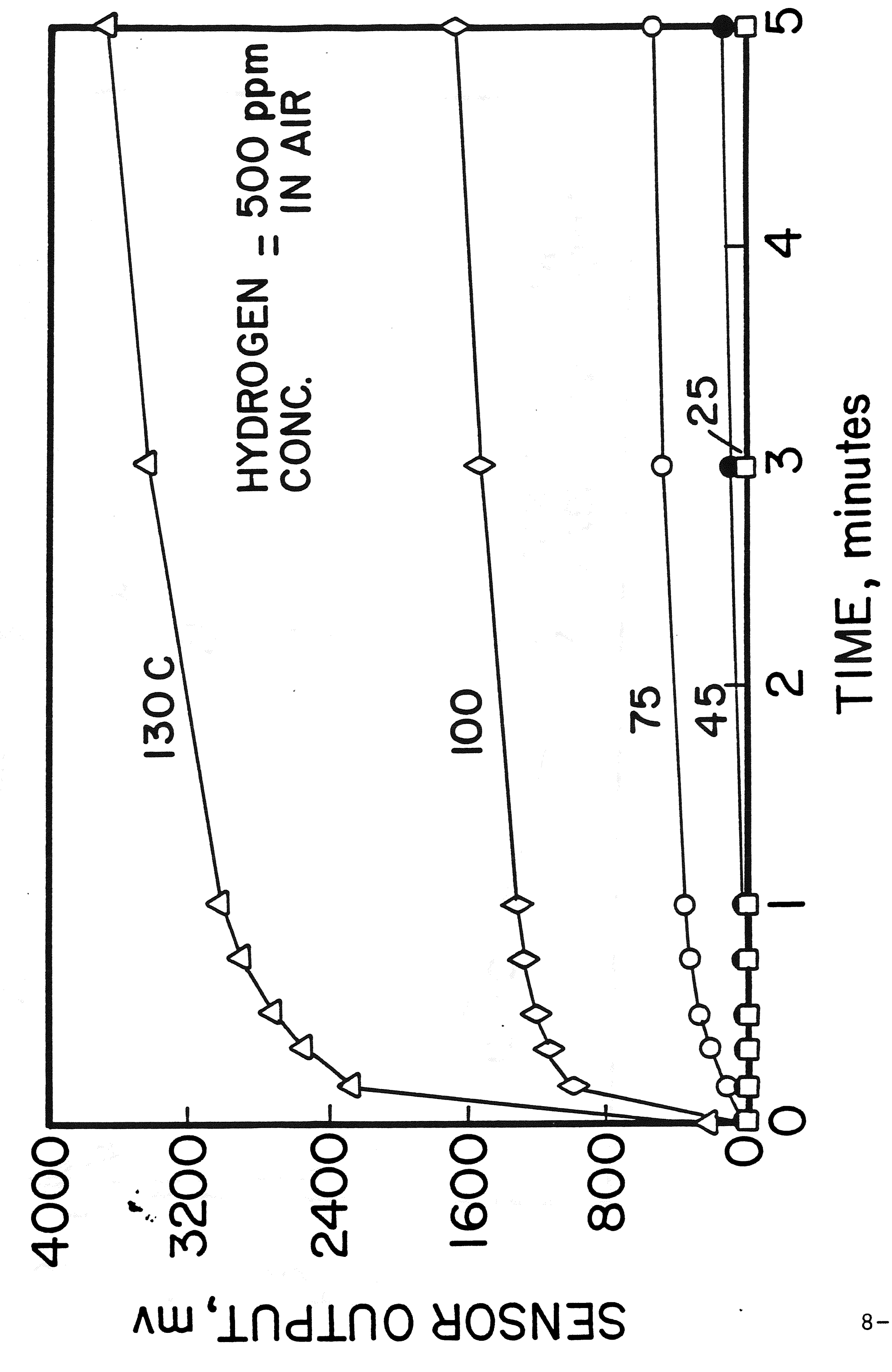
DR. ROBERT A. POWERS

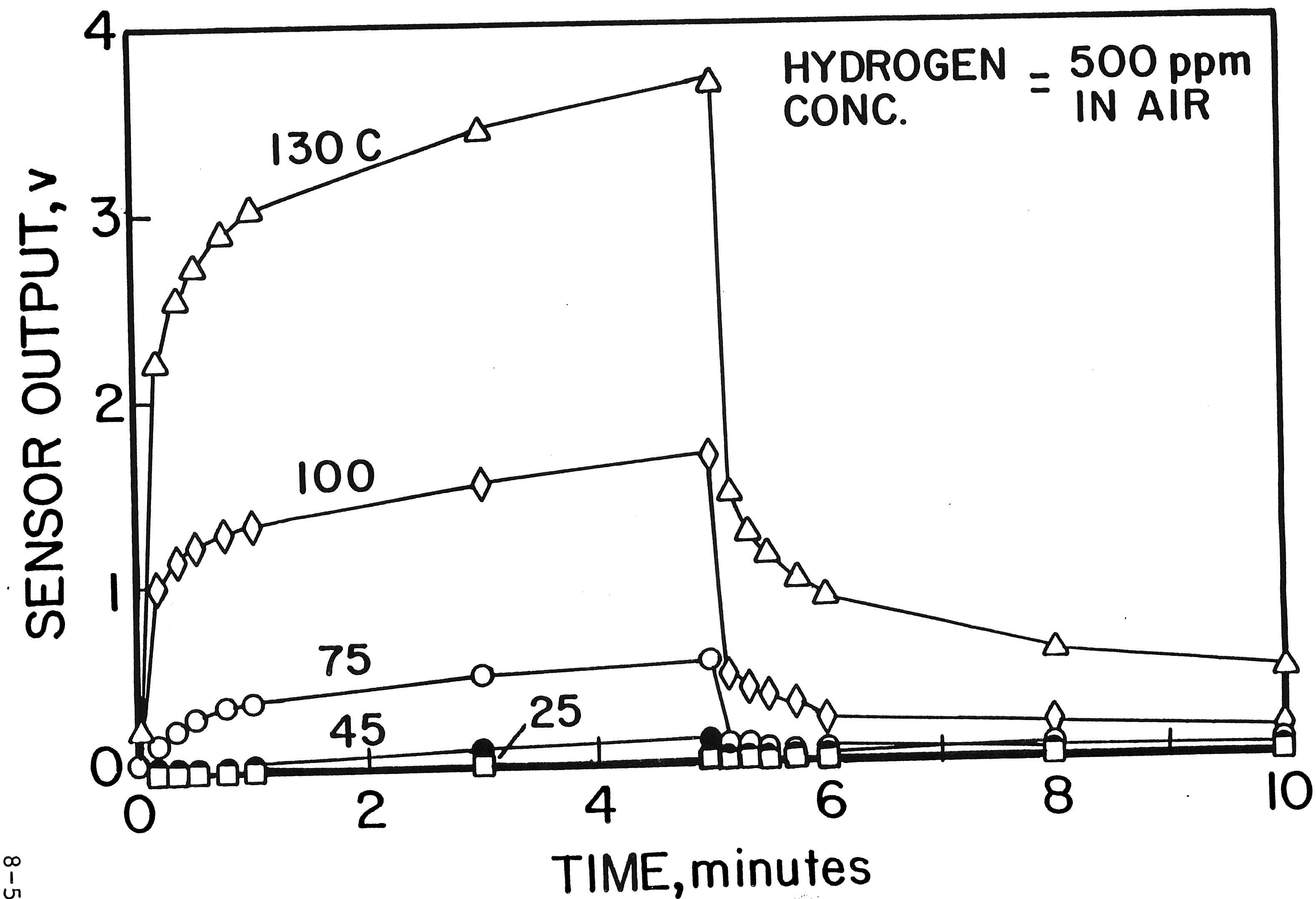
EDISON SENSOR TECHNOLOGY CENTER
CASE WESTERN RESERVE UNIVERSITY
CLEVELAND, OHIO

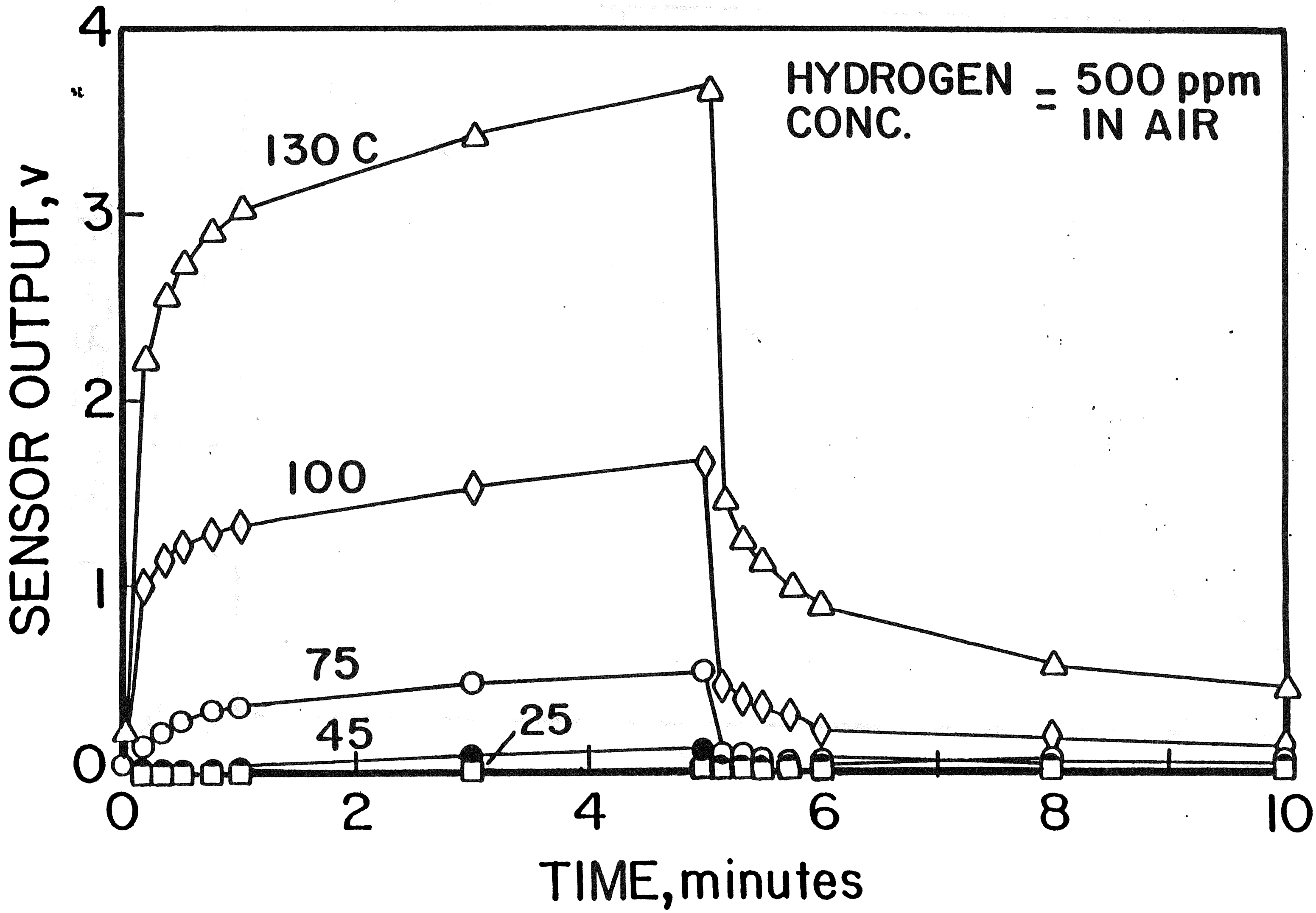

HEATER
MIS
TEMPERATURE DETECTOR

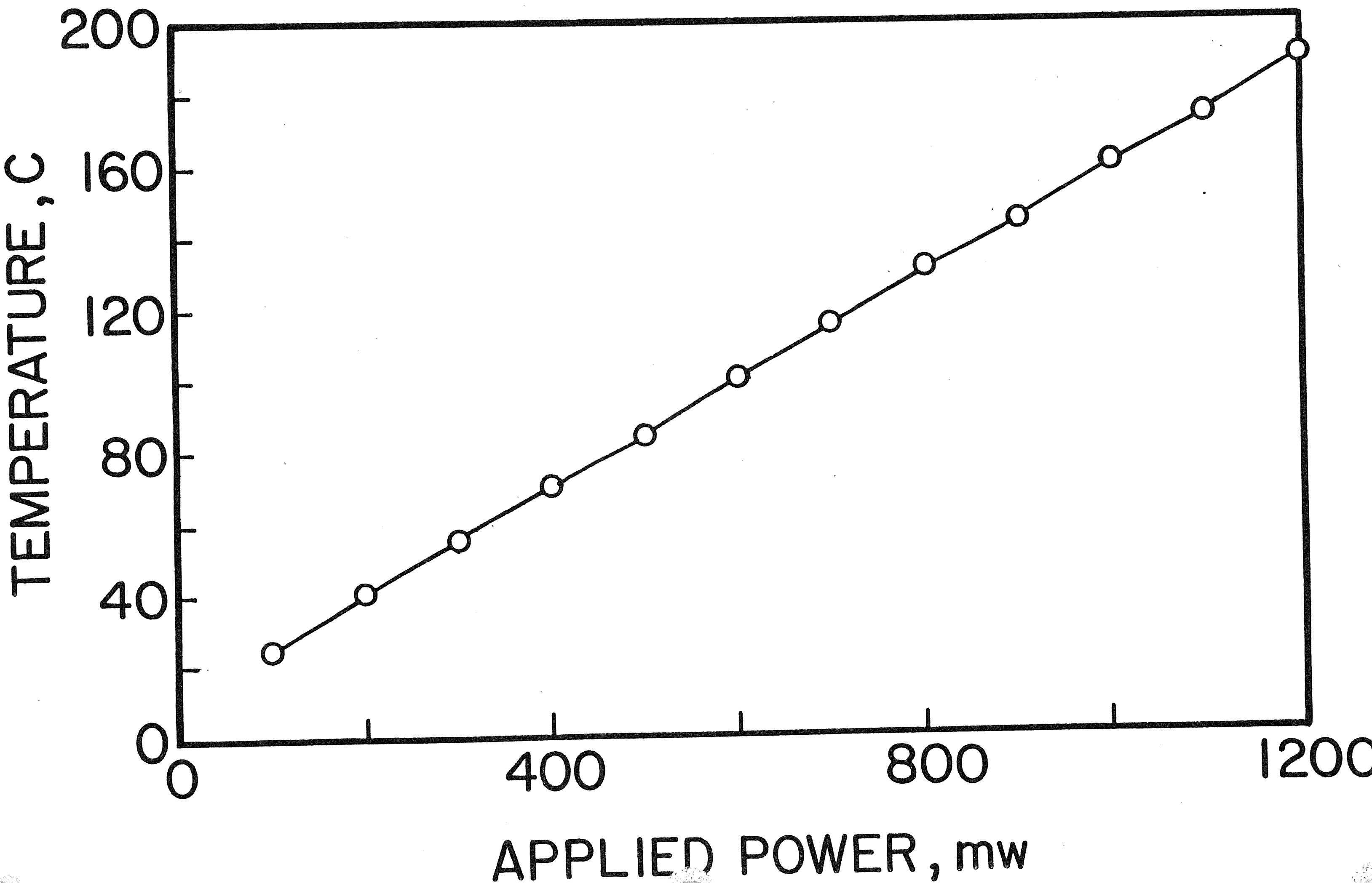

HYDROGEN CONC. = 500 ppm IN AIR
SENSOR OUTPUT, mv
1000
100
10
1
0
25
45
65
85
105
125
TEMPERATURE, C°


SENSITIVITY S=ΔV/V0, %
100
80
60
40
20
0
0
200
400
HYDROGEN CONCENTRATION, ppm
115 C
75
20


SENSOR OUTPUT, mv
4000
3200
2400
1600
800
0
TIME, minutes
0
1
2
3
4
5
130 C
100
75
45
25
HYDROGEN CONC. = 500 ppm IN AIR


HYDROGEN CONC. = 500 ppm IN AIR
130 C
100
75
45
25
SENSOR OUTPUT, v
TIME, minutes
0
1
2
3
4
0
2
4
6
8
10


HYDROGEN CONC. = 500 ppm IN AIR
130 C
100
75
45
25
SENSOR OUTPUT, v
TIME, minutes
0
1
2
3
4
0
2
4
6
8
10


TEMPERATURE, C
200
160
120
80
40
0
0
400
800
1200
APPLIED POWER, mw

QUALIFICATIONS OF METAL-INSULATOR-SEMICONDUCTOR SENSORS FOR H2 LEAK DETECTION

STEVE PYKE
MICROMONITORS, INC.

A HYDROGEN MICROSENSOR CONCEPT

H. Thurman Henderson

Kartalia Chair in Electrical Engineering

Sachin Dhole, PhD Student

UNIVERSITY OF CINCINNATI

Loc. #30, Rhodes Hall

Cincinnati, Ohio 45221

(513) 556-4774

In Affiliation With

UC/SPACE ENGINEERING CENTER

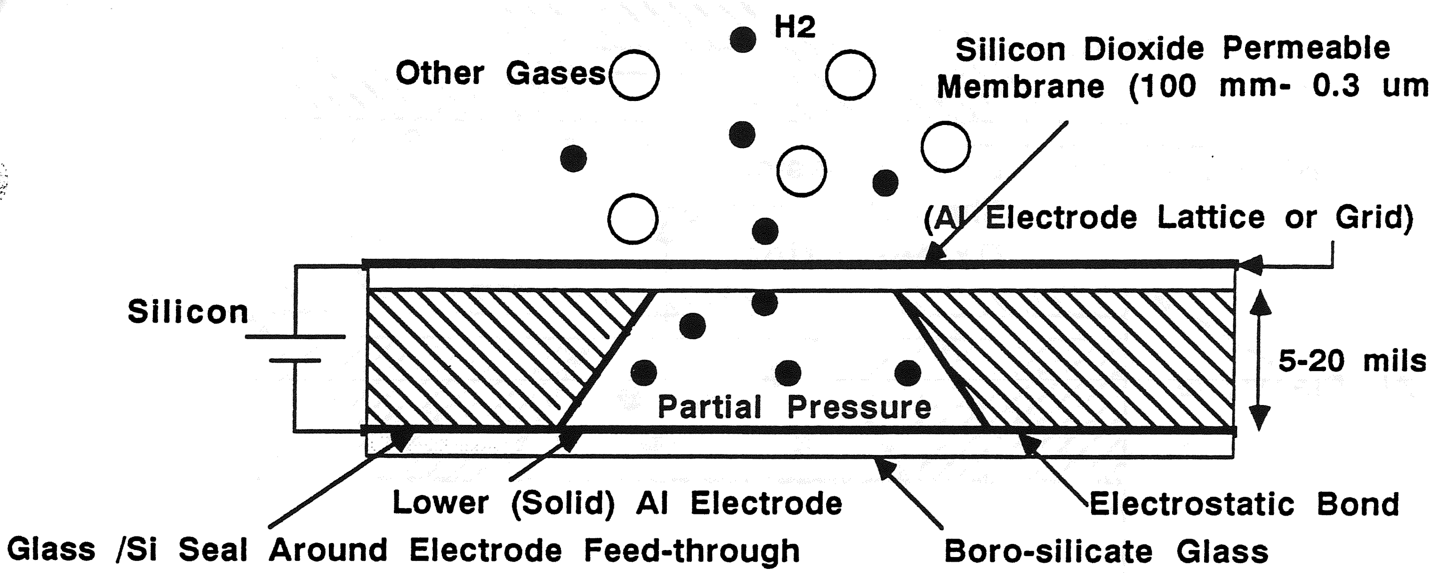
FOR HEALTH MONITORING

AND

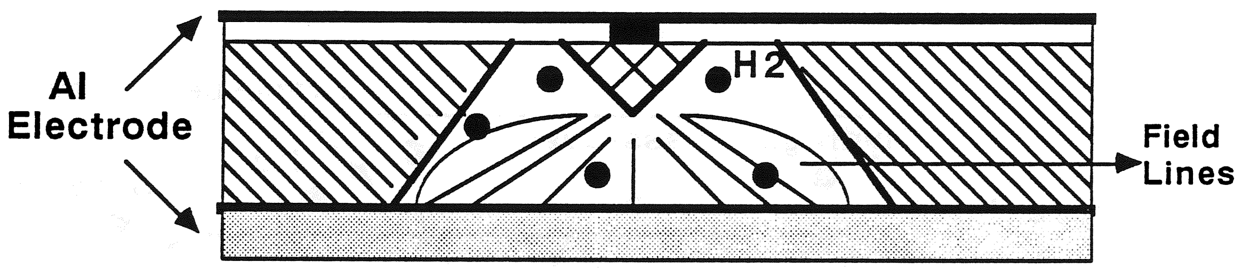
CENTER FOR MICROELECTRONIC SENSORS AND MICROSTRUCTURES

AGENDA

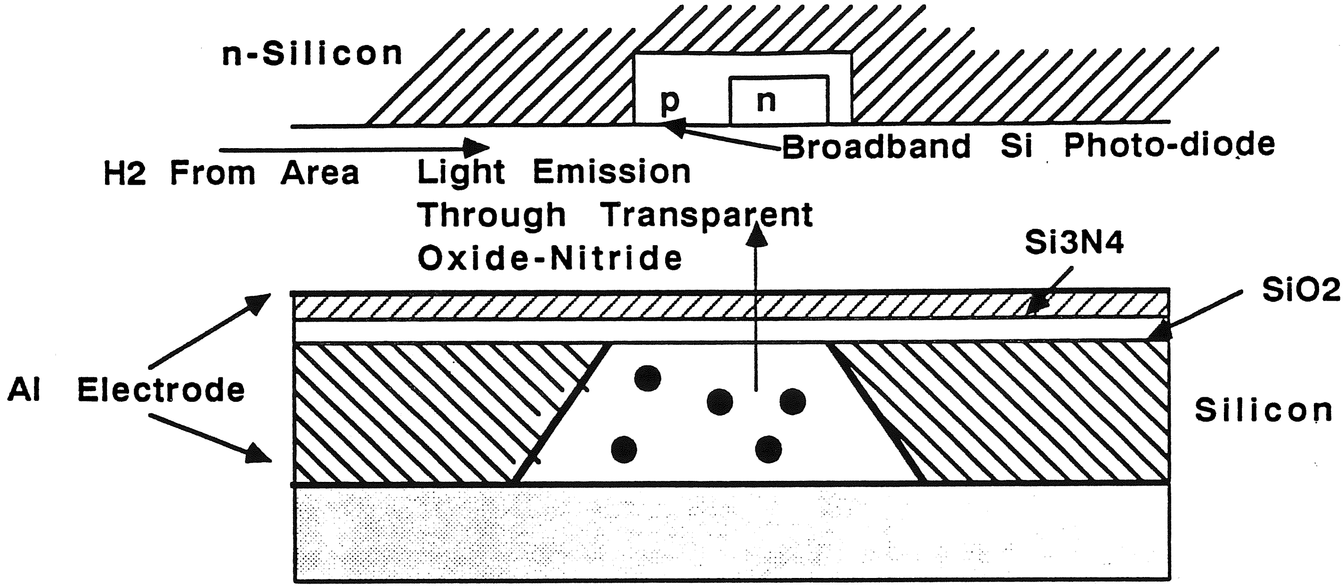
- o Background
- o Historical and Present Methods
- o The New MEMS Technology
- o Proposed Micromachined Silicon Based Hydrogen Sensors
  - o The Permeable Membrane
  - o A Micromachined Hydrogen Gas Tube in a Chip
  - o Deadout Instrumentation
  - o Microspectroscopy/Source
  - o The System
  - o MIR Sensitivity Enhancement



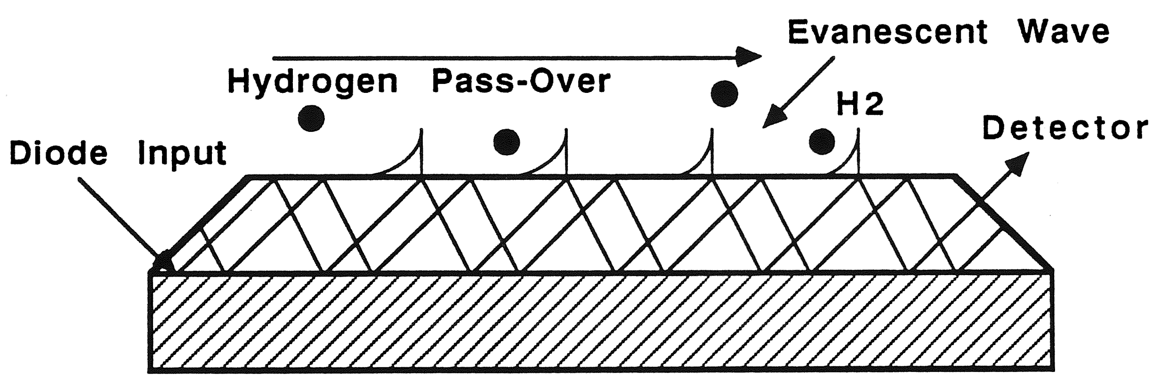
Structure of the Micro-machined H2 Detector Tube



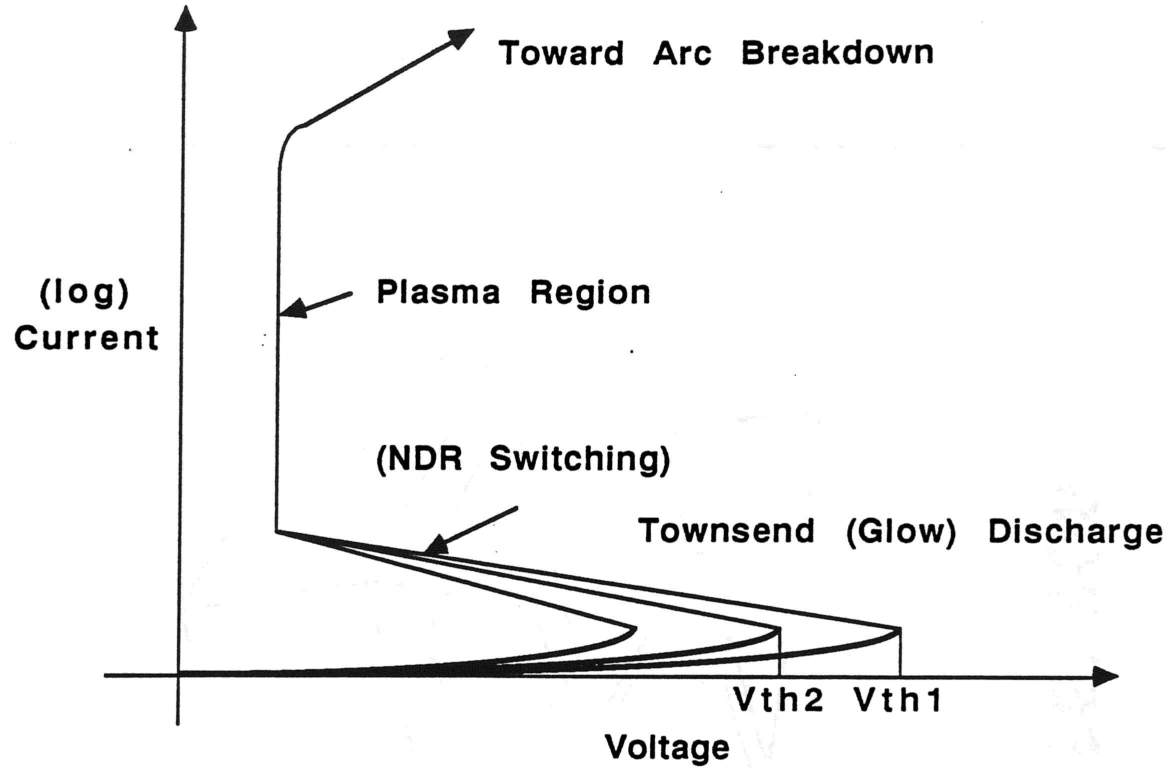
Enhanced electric Field for Switching Threshold Current



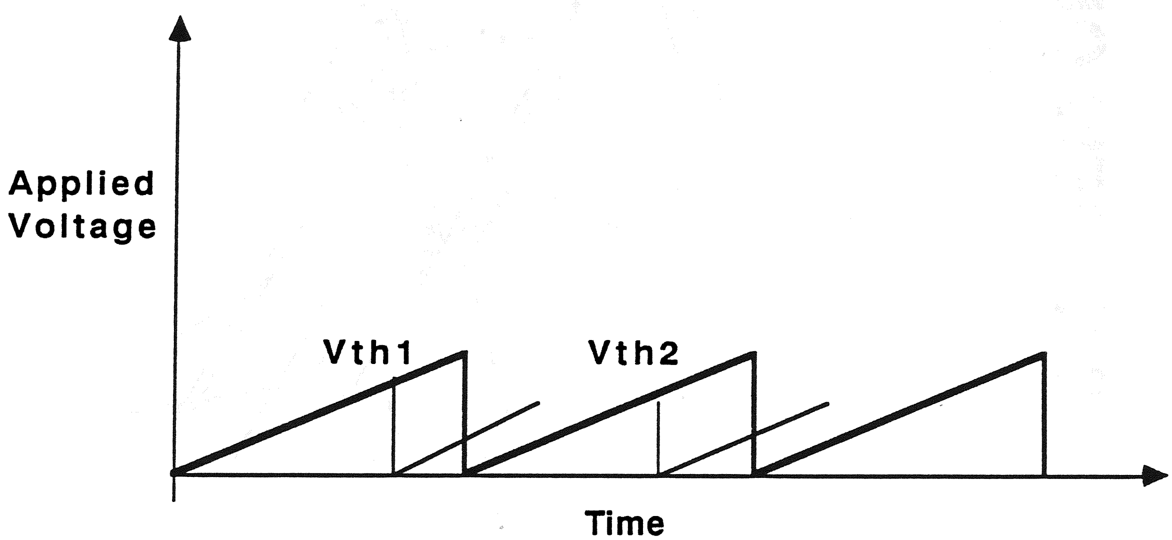
Gaseous Light Emitting Diode with Precise Lines
for Hydrogen Absorption
(All Integrated into a Single Chip)



Silicon Micromachined Wave-guide for Multiple Internal Reflection (MIR) to Increase Absorption Path
(Enhanced Absorption of Hydrogen Lines through ATR)

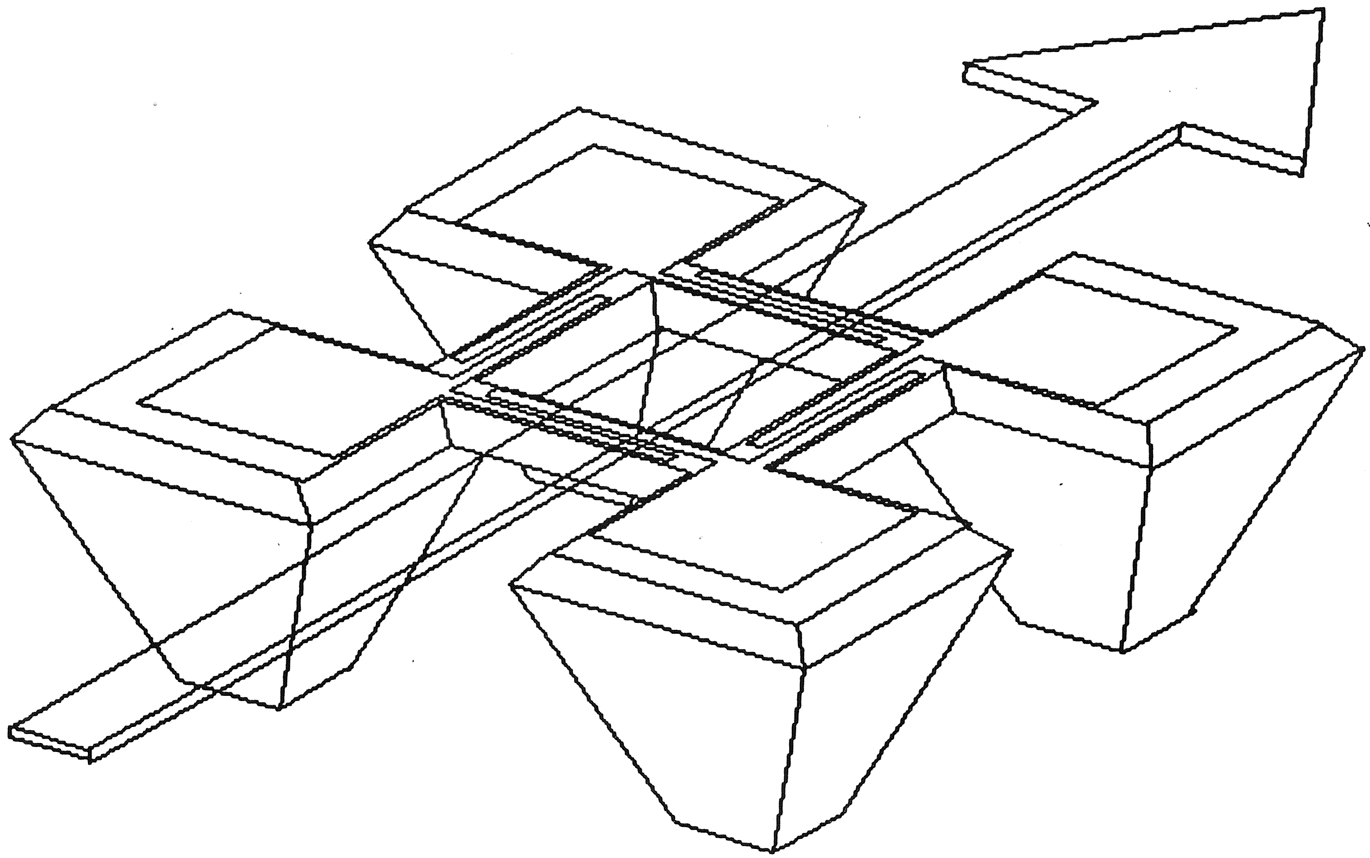


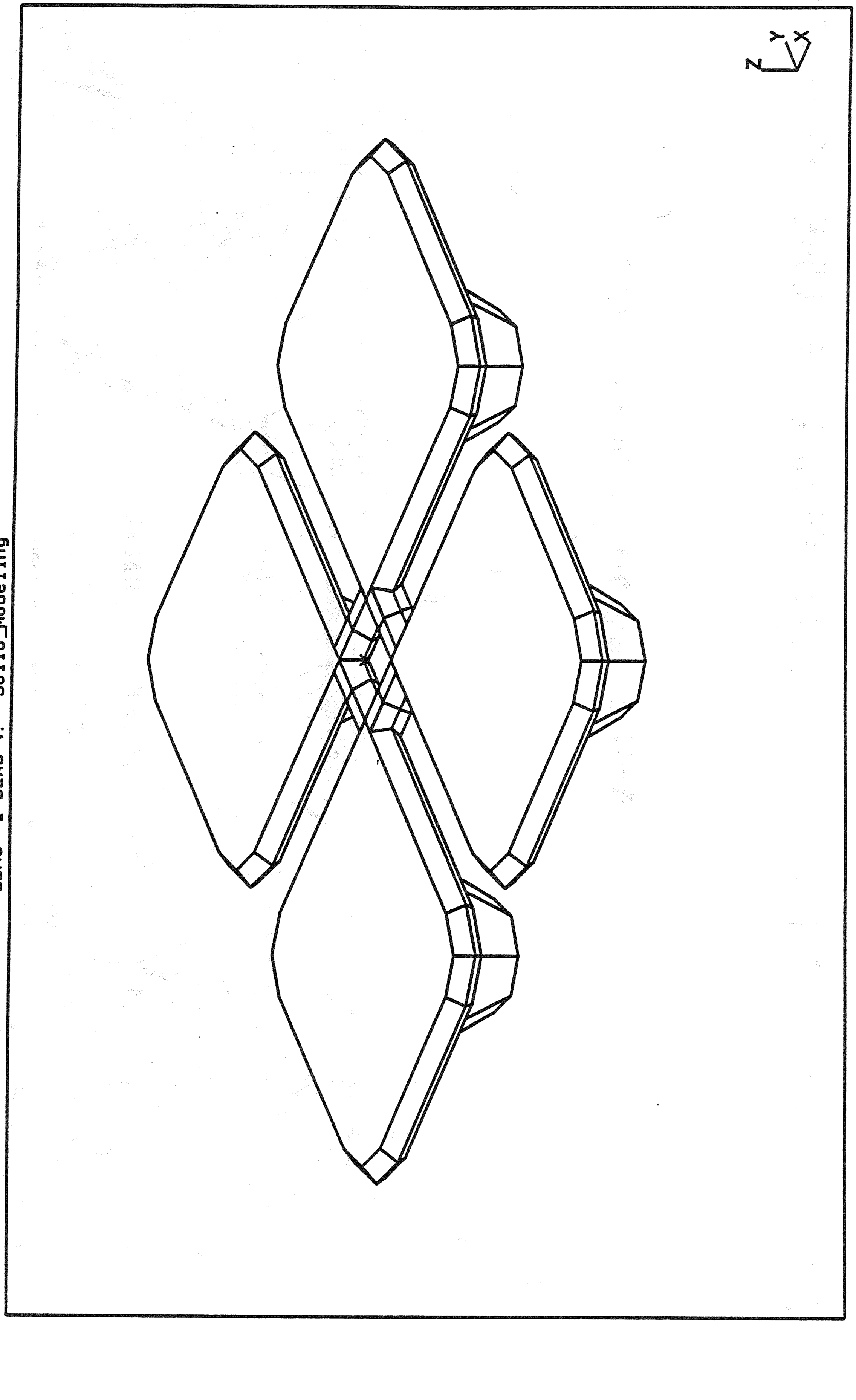
Typical Arc Discharge Curve for the H2 Arc in the Tube



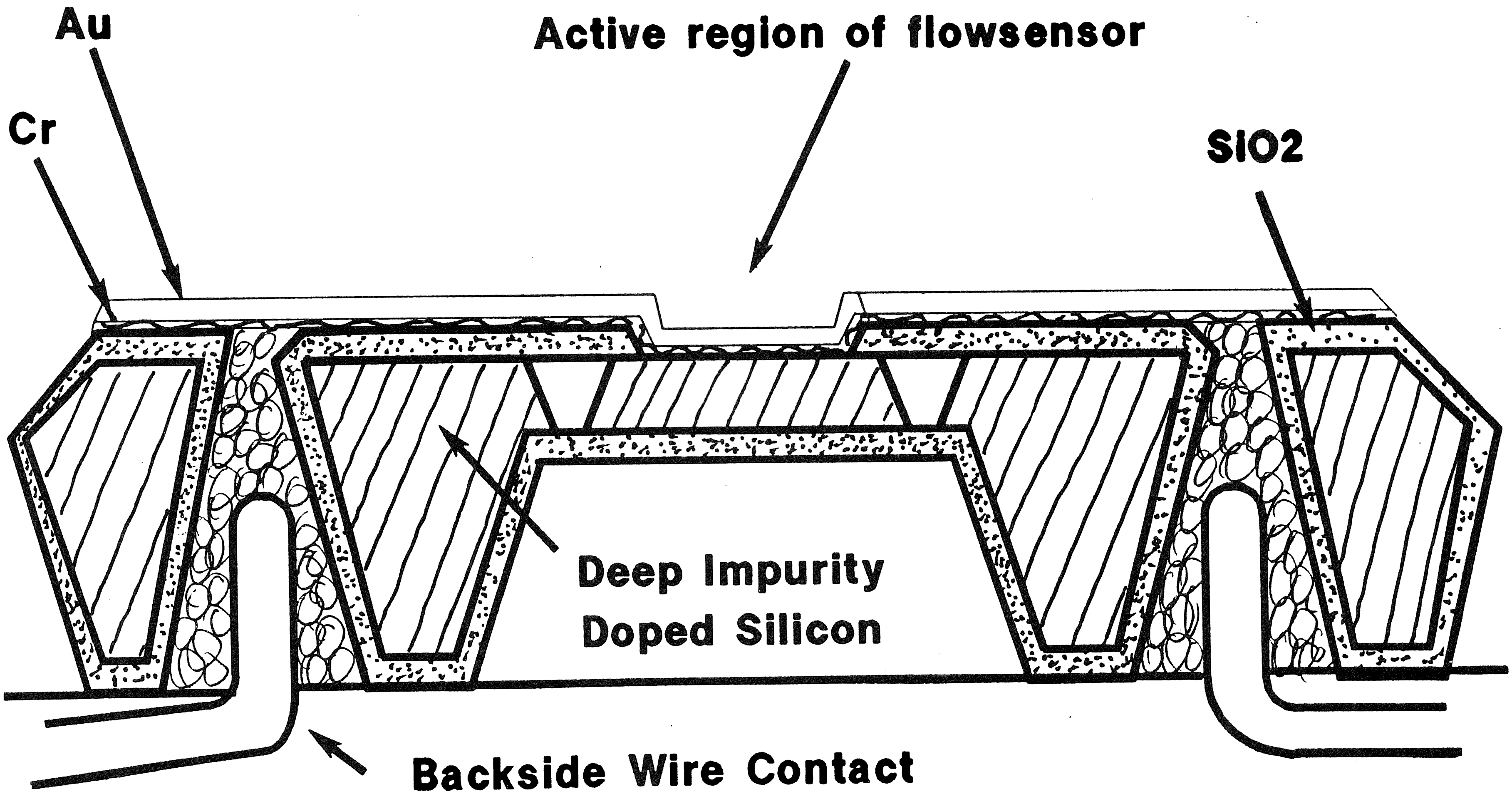
Applied Sweep Voltage to Determine the Partial Pressure of H2 in the Tube

3-D VIEW OF FLOW SENSOR
(Ideal Device)




SDRC I-DEAS V: Solid_Modeling
X
Y
Z

BACKSIDE CONTACT, HIGH TEMPERATURE VERSION



SSME Leak Detection Using Sequential Image Processing

Outline:

I. Program Overview and Feasibility Study - A. Shohadaee, R. Crawford

II. Leak Detection Algorithm - M. Smith

III. Testing and Verification - J.A. Malone

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TULLAHOMA, TN 37388
PS-0022

Program Objectives

Phase I Feasibility study to determine applicability of image processing methods to rocket engine leak detection.

Phase II Development, implementation and testing of algorithms for leak detection on digital image processing equipment.

Phase III Specification and development of systems for application to actual real-time test-stand and launch-pad data.

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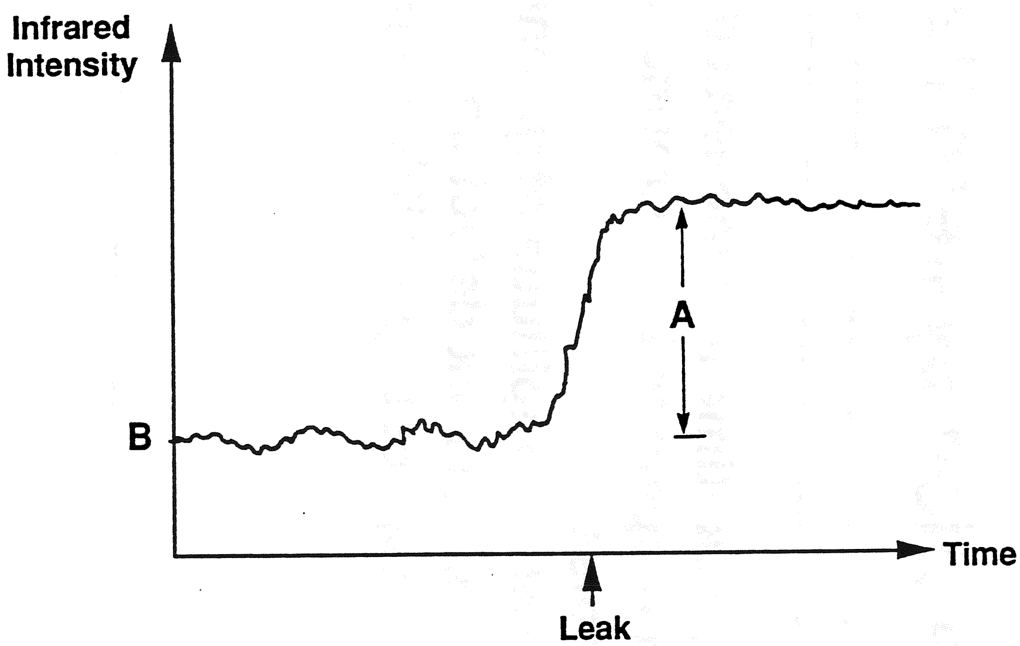
SSME Leak Detection

TEMPORAL RESPONSE STUDY

J.A. Malone
L.M. Smith



Intensity at Single Point in Image

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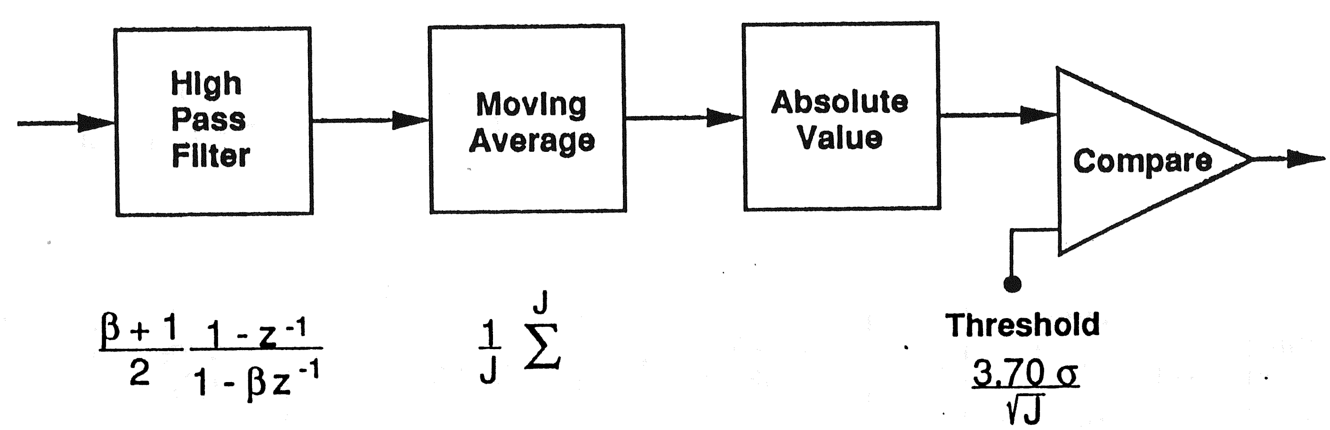
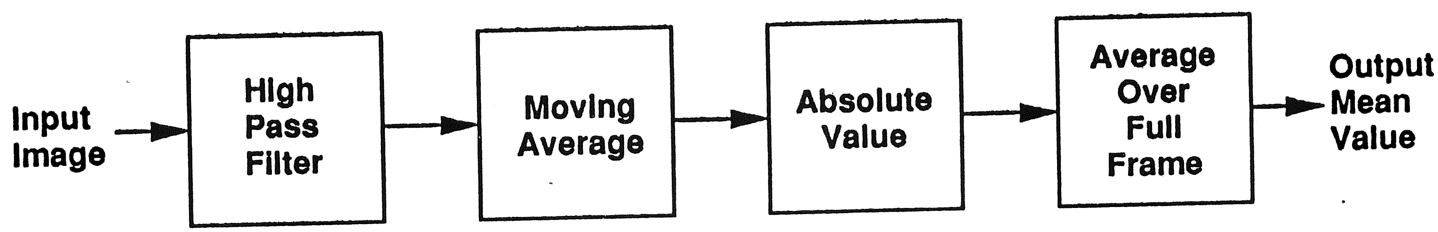
Leak Detection System Requirements

- Provide fast, real-time leak/no-leak decision
- Robust with respect to background intensity levels and signal noise
- Capable of hot (+ amplitude step) and cold (- amplitude step) leak detection
- Robust with respect to possible step amplitudes

Leak/Step Detection System



Leak/Step Detection System



$$\frac{\beta+1}{2}\,\frac{1-z^{-1}}{1-\beta z^{-1}} \qquad \frac{1}{J}\sum^{J}$$

Signal-to-Noise Ratio

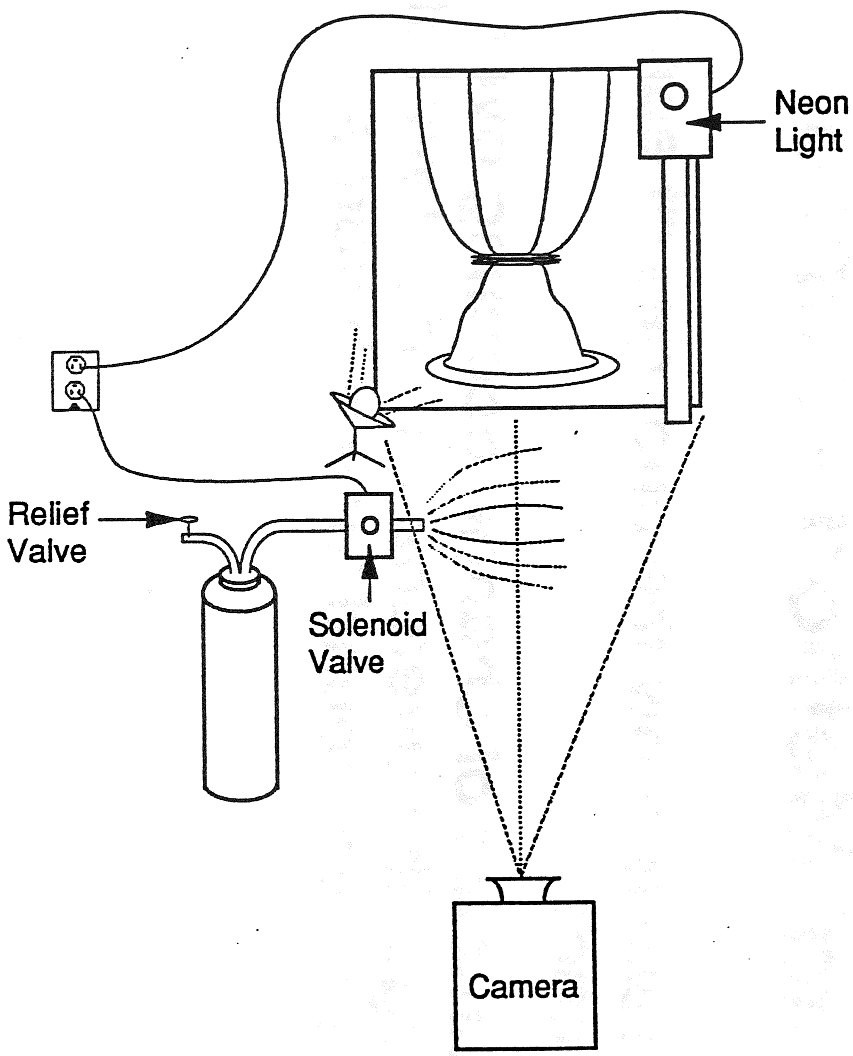
$$\frac{S}{N} = \frac{|A|}{2\sigma}\left(\frac{1+\beta}{1-\beta}\right)\left(\frac{1-\beta^{J}}{\sqrt{J}}\right)$$

can be maximized for given β by:

$$J = -\frac{1.2564}{\ln\beta}$$

Experimental Study

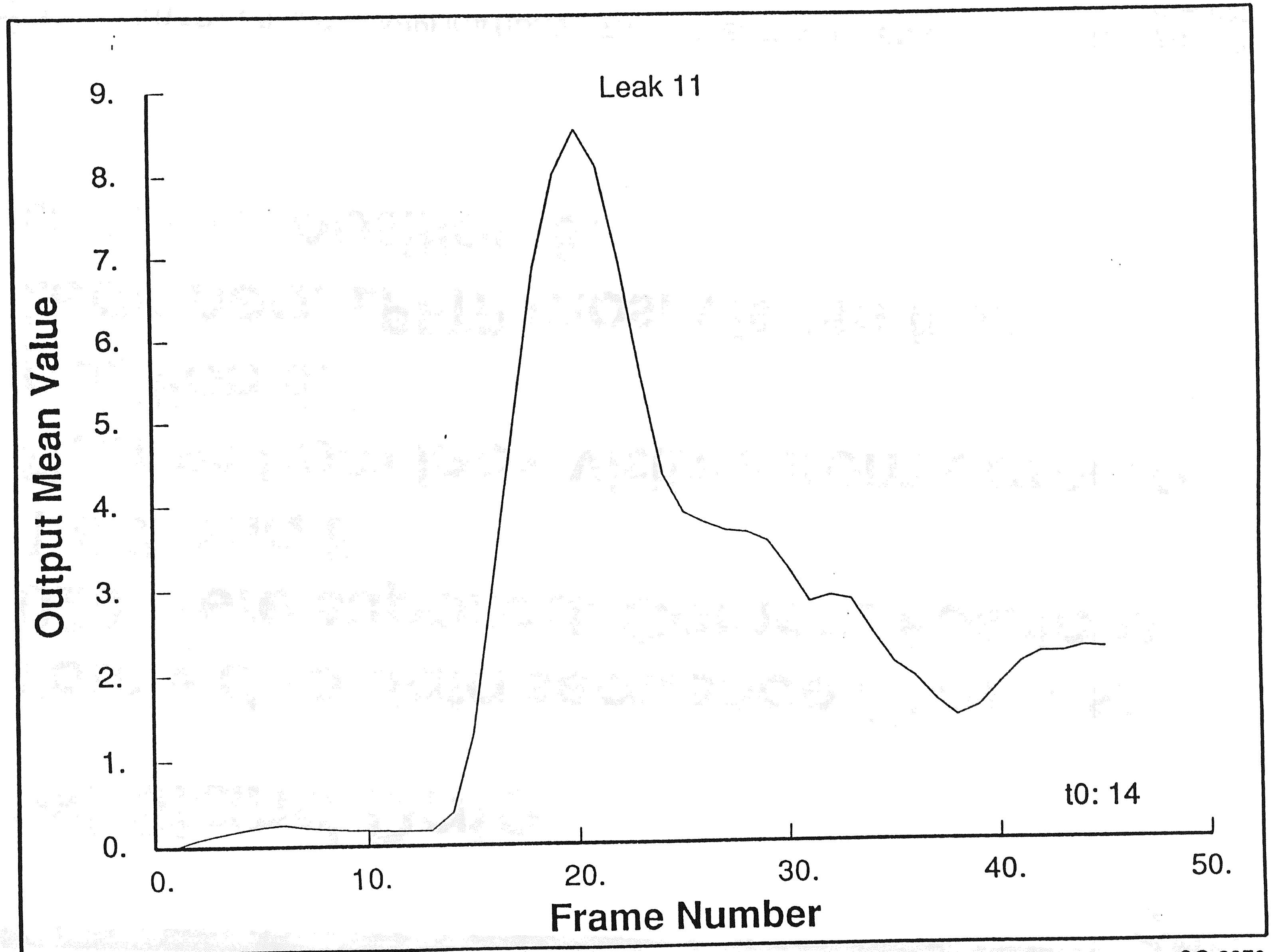
- Create a sequence of images containing a gas leak.
- Record & process full frame images using PC based image processor.
- Provide a method for determining the frame in which the leak was initiated.



IL-1221

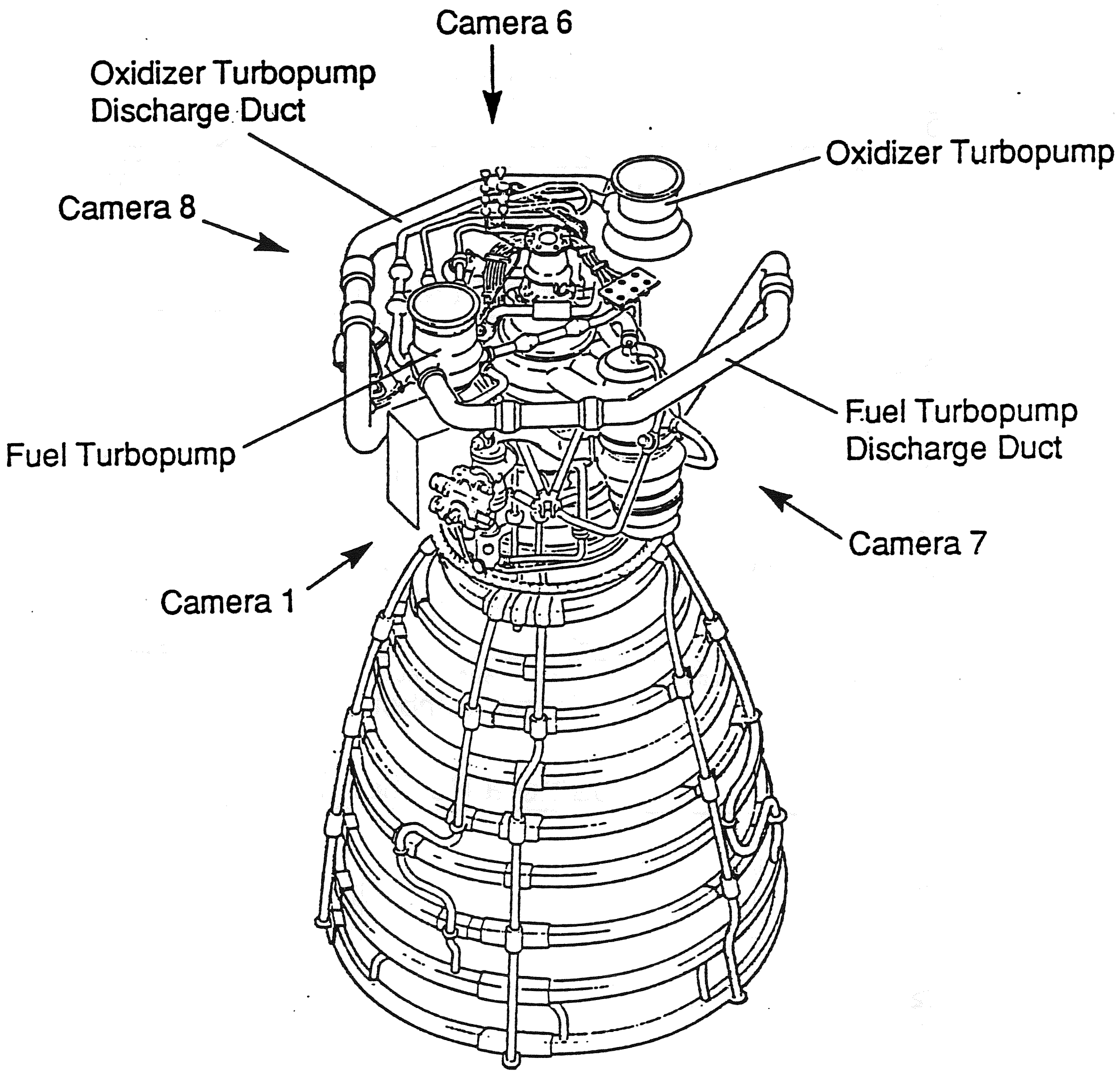
Experimental Method - cont'd

- The previous results show the changes at each point in the image.
- To show the amount of change occuring over the entire image, the average intensity level of the entire image was found for each frame.


Leak 11
Output Mean Value
9.
8.
7.
6.
5.
4.
3.
2.
1.
0.
0.
10.
20.
30.
40.
50.
Frame Number
t0: 14
CG-2270

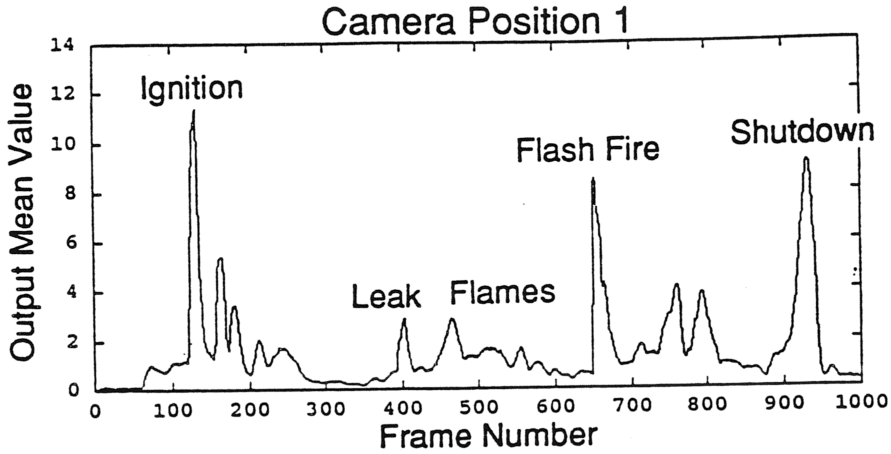
Test Stand Data

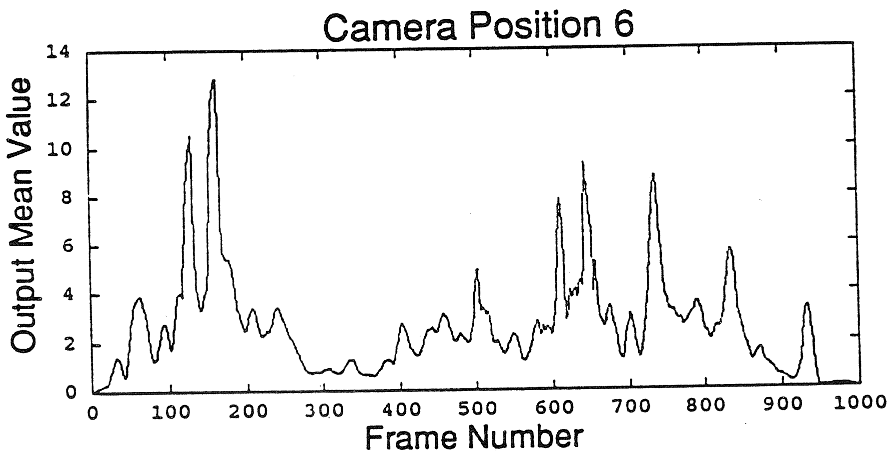
- Four video data sequence from Test 020 were supplied: Camera Positions 1,6,7 and 8.
- Flames from leak visible from camera position 6.
- Leak near LPFTP most visible from camera position 8.

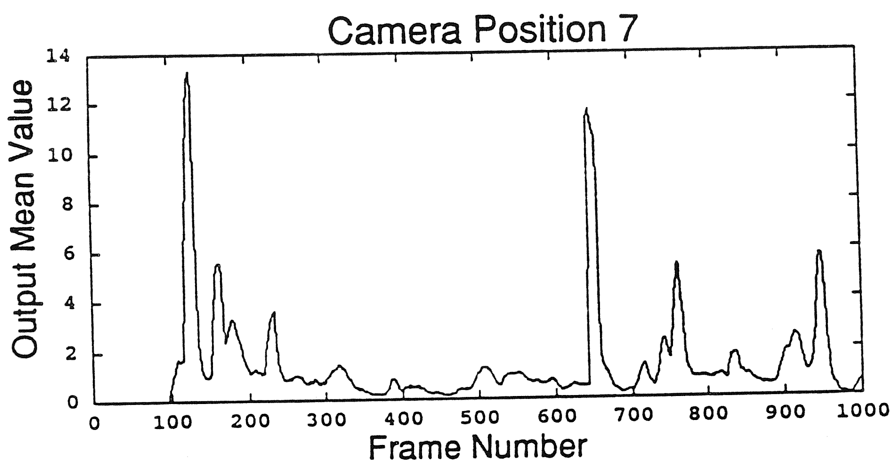


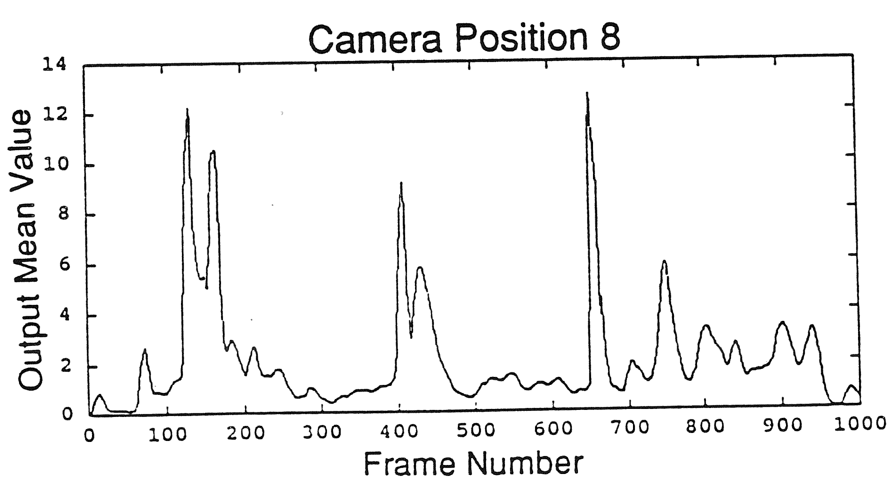
Camera 6
Oxidizer Turbopump
Discharge Duct
Oxidizer Turbopump
Camera 8
Fuel Turbopump
Fuel Turbopump
Discharge Duct
Camera 7
Camera 1

IL-1297


Camera Position 1
Ignition
Leak
Flames
Flash Fire
Shutdown
Output Mean Value
Frame Number
0
2
4
6
8
10
12
14
0
100
200
300
400
500
600
700
800
900
1000


Camera Position 6
Output Mean Value
Frame Number
0
2
4
6
8
10
12
14
0
100
200
300
400
500
600
700
800
900
1000


Camera Position 7
Output Mean Value
Frame Number
0
2
4
6
8
10
12
14
0
100
200
300
400
500
600
700
800
900
1000


Camera Position 8
Output Mean Value
Frame Number
0
2
4
6
8
10
12
14
0
100
200
300
400
500
600
700
800
900
1000

CONCLUSIONS

- Developed leak detection algorithm and implemented it on PC based image processor.
- Tested algorithm on experimental gas leak image sequence.
- Processed actual test stand data.
- System is effective and ready for real-time development and implementation.

Phase III - Future Work

- System specification for real-time implementation.
- Application to actual test-stand and launch-pad data.
- Investigation of other approaches - Raman scattering, infrared vs. visible, multi-channel image processing.

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TULLAHOMA, TN 37388

Program Focus is the Application of H_2 Microsensors

Phase 1:

1. Define the Requirements and Conceptual Design for a Distributed Array of Leak Detection Sensors on ETO-Type Engines.

2. Design an Experimental Breadboard System.

Phase 2:

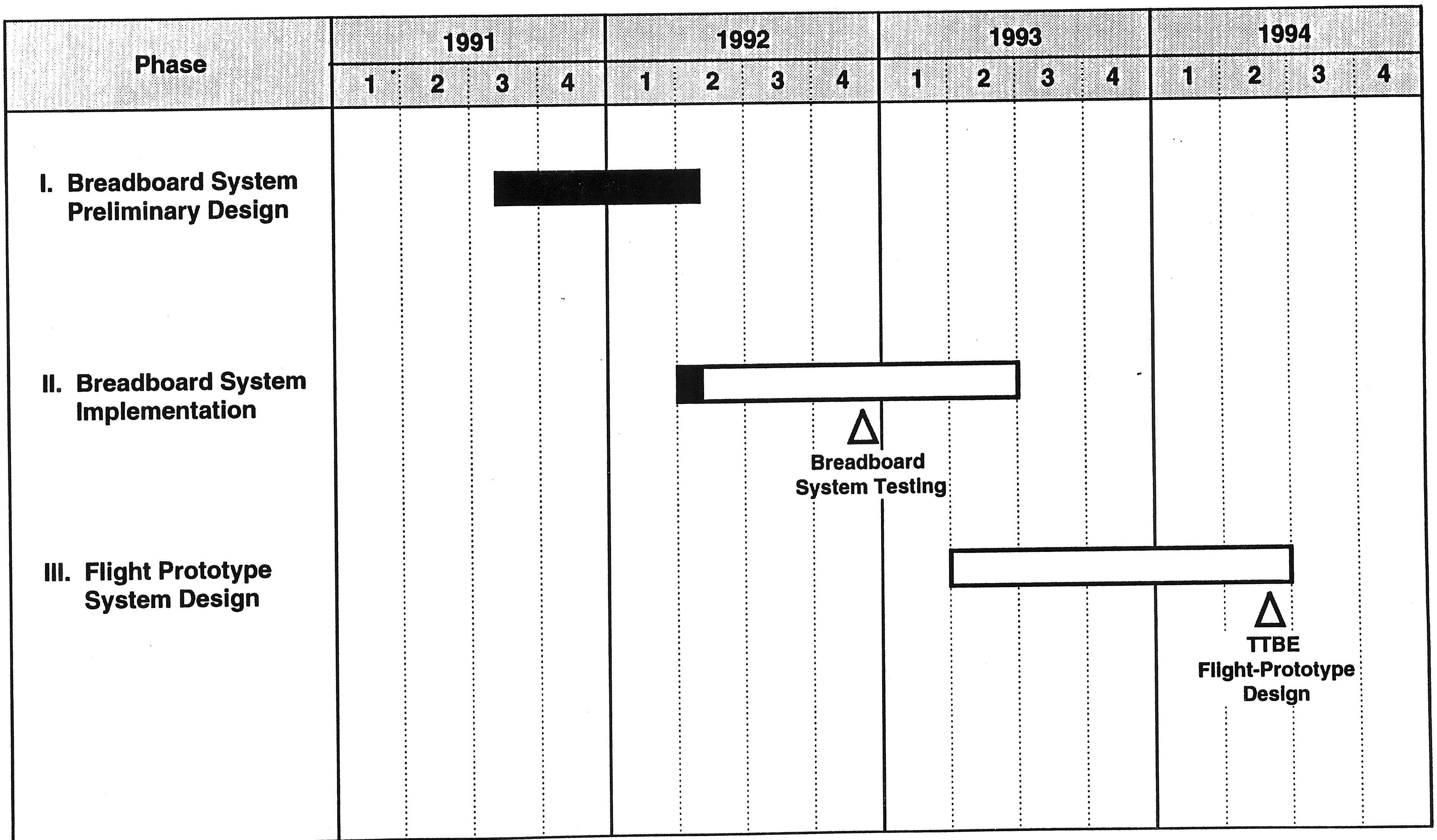
3. Assemble Experimental Breadboard System and Conduct Experiments Simulating Leaks in a Pump-Fed Liquid Rocket Engine.

Phase 3:

4. Design and Fabricate Prototype Leak Detection Sensors Incorporating Integrated Circuit Technology.

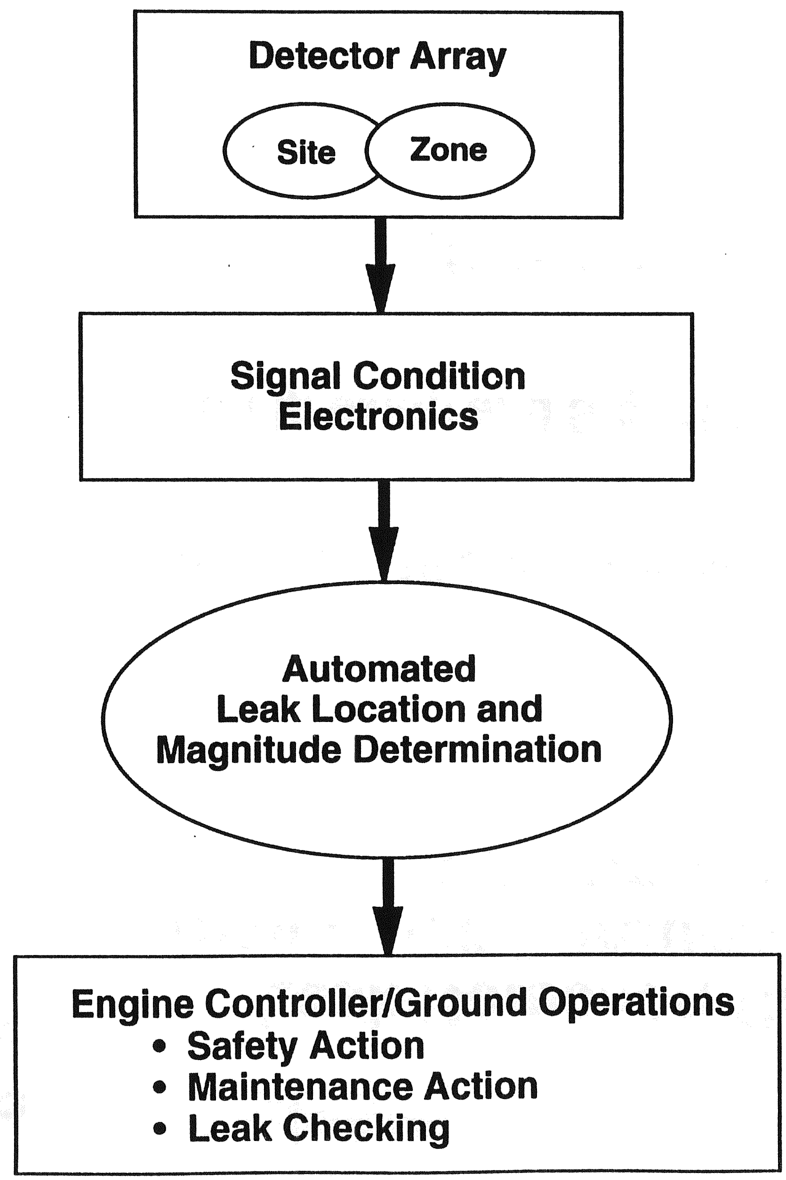
5. Develop a Plan to Test the Prototype System in Field Experiments at NASA/MSFC TTBE.

Breadboard System Development is Underway



14.26.08

Distributed Sensor Leak Detection Process



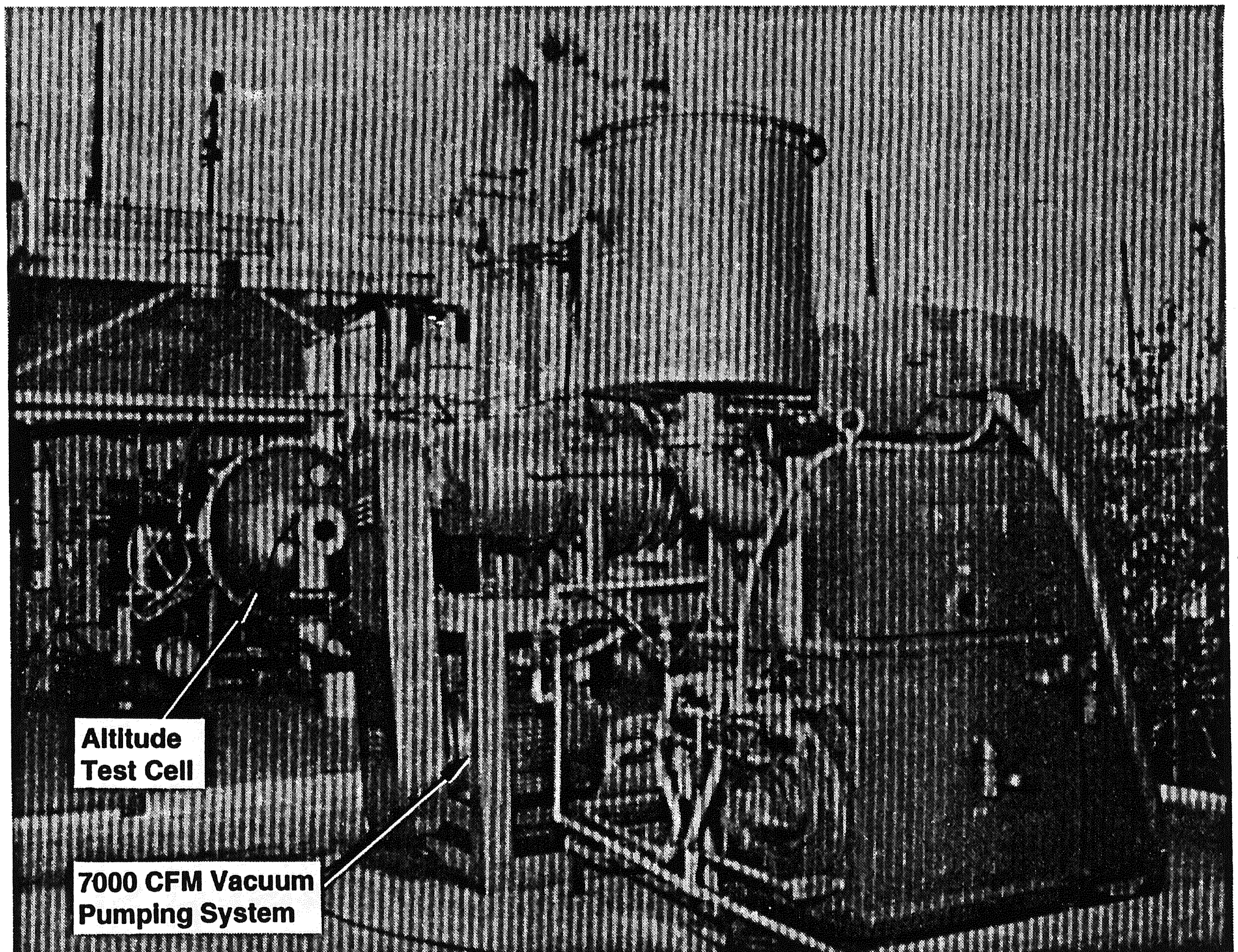
- Silicon Based H_2 Microsensors
- Multiplexed Data Acquisition and Signal Filtering
- Knowledge Based Processing
- Leak "Imaging"

14.26.09

Leak Detection Component Evaluation Through Breadboard System Testing

- Sensors (16 Sensor Array) Zone and Site Types
- Multiplexing and Signal Conditioning Electronics
- Knowledge Based Processing Software

Breadboard System Testing Will be Conducted at Sea Level and 50,000 ft Altitude Conditions



Aerojet Leak Detection Facility

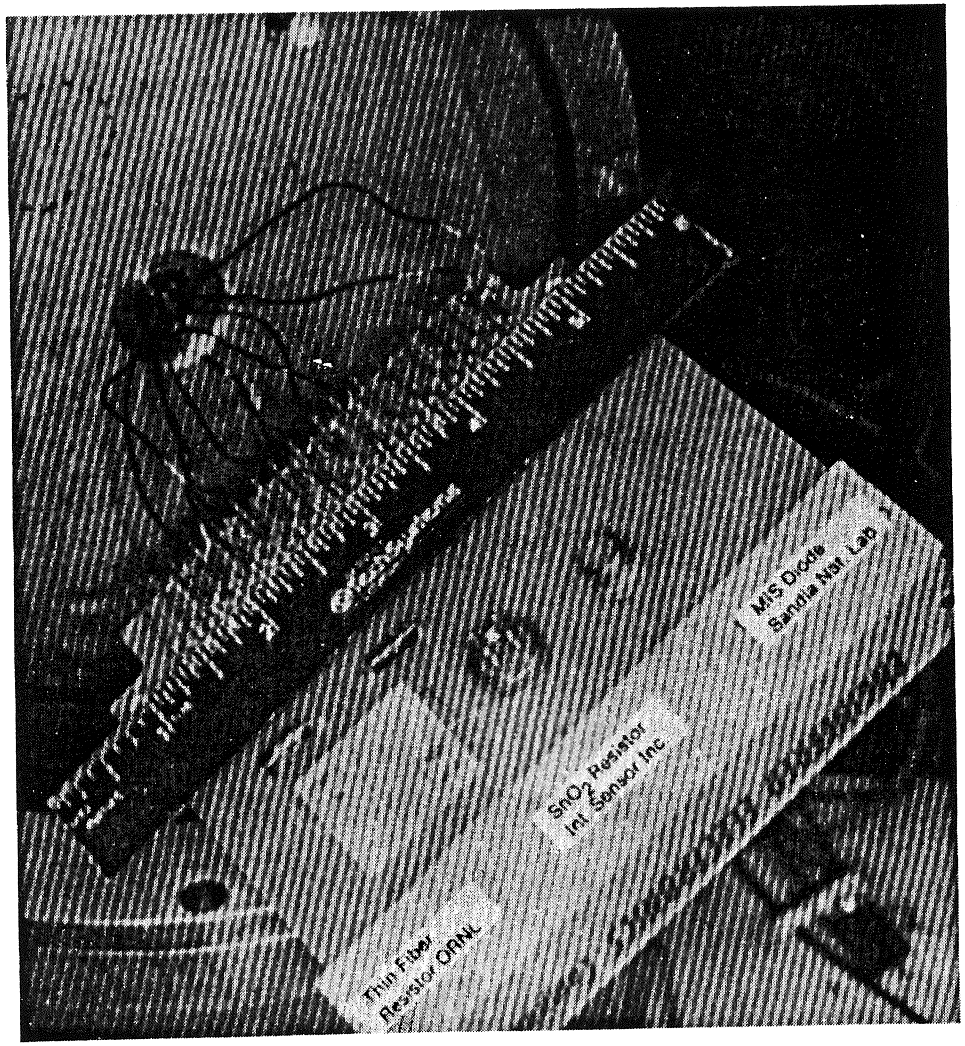
Flight-Prototype System is Based on Breadboard Scale-Up

- 144 Sensors at 72 Locations on Engine
- Flight-Type Hardware
- Real-Time Software Potential
- Engine Controller Interface

Background

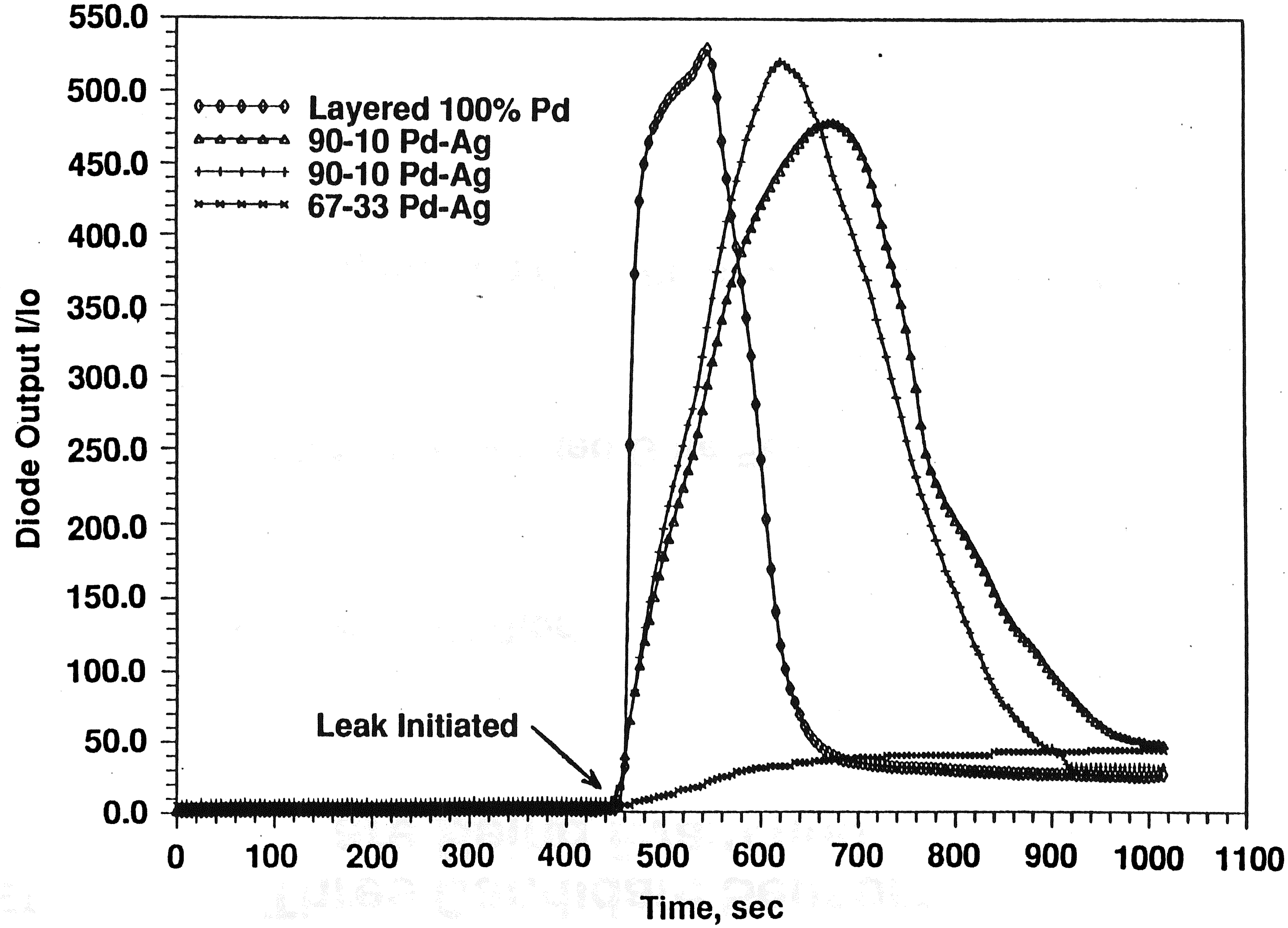
- Leak Detection Strategies Studied for Space-Based OTV, 1987-1988 (NAS 3-23772)
  - Matrixed H_2 Sensors Offer Near Term, Cost Effective Approach
  - No Commercial Sensors Available for Space ETO Environments
- Sensor Evaluation and Characterization Testing, 1989-1991 (Aerojet IR&D)
  - Pd/Ag MIS Diodes, Pd Resistors, Tin Oxide Sensors
  - Temperature, Gas Composition, and Pressure Effects

Pd/Ag MIS Diodes Have Demonstrated Suitable Characteristics



Sensors Mounted for Head-to-Head Testing

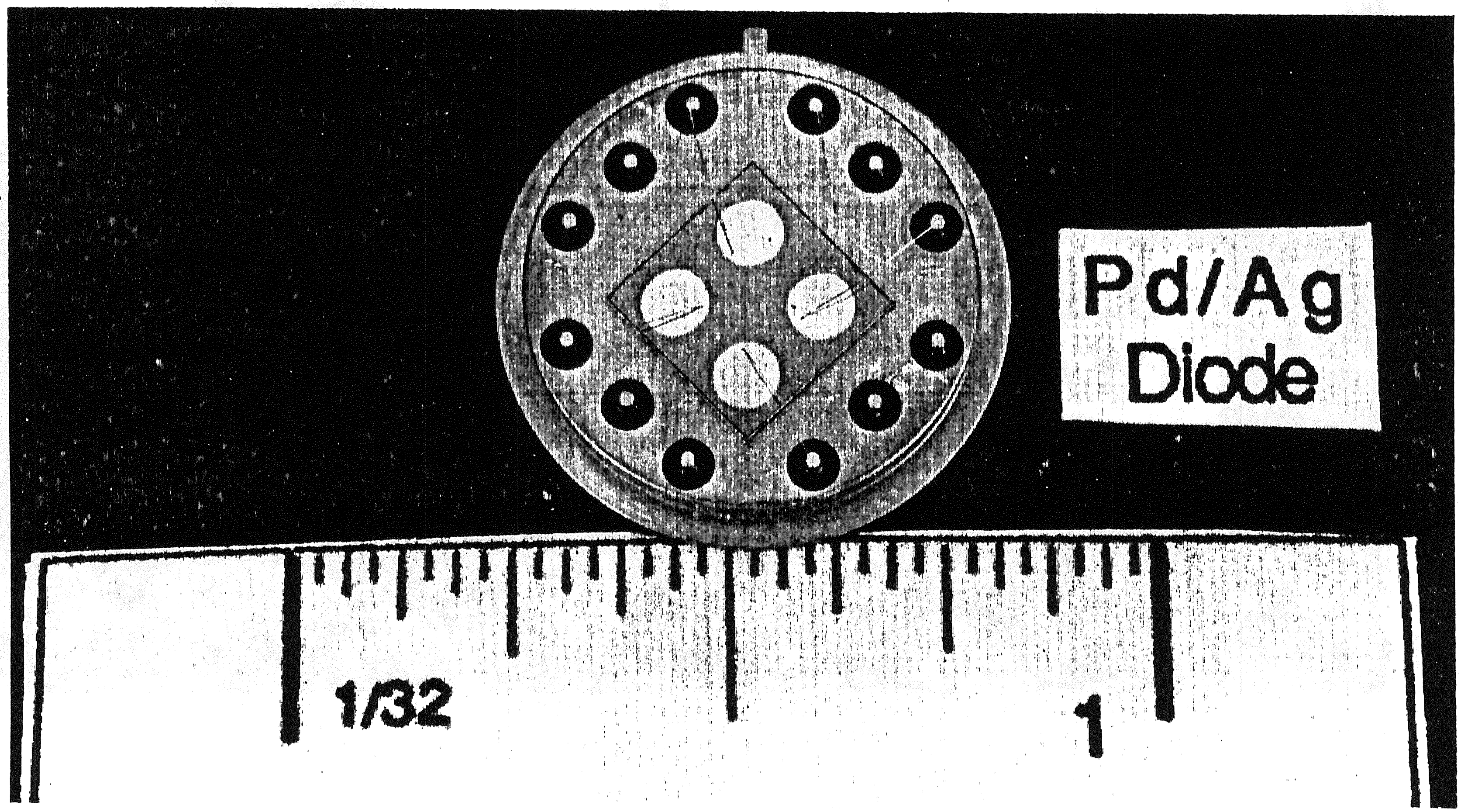
Gate Composition Influences Detection Element Speed



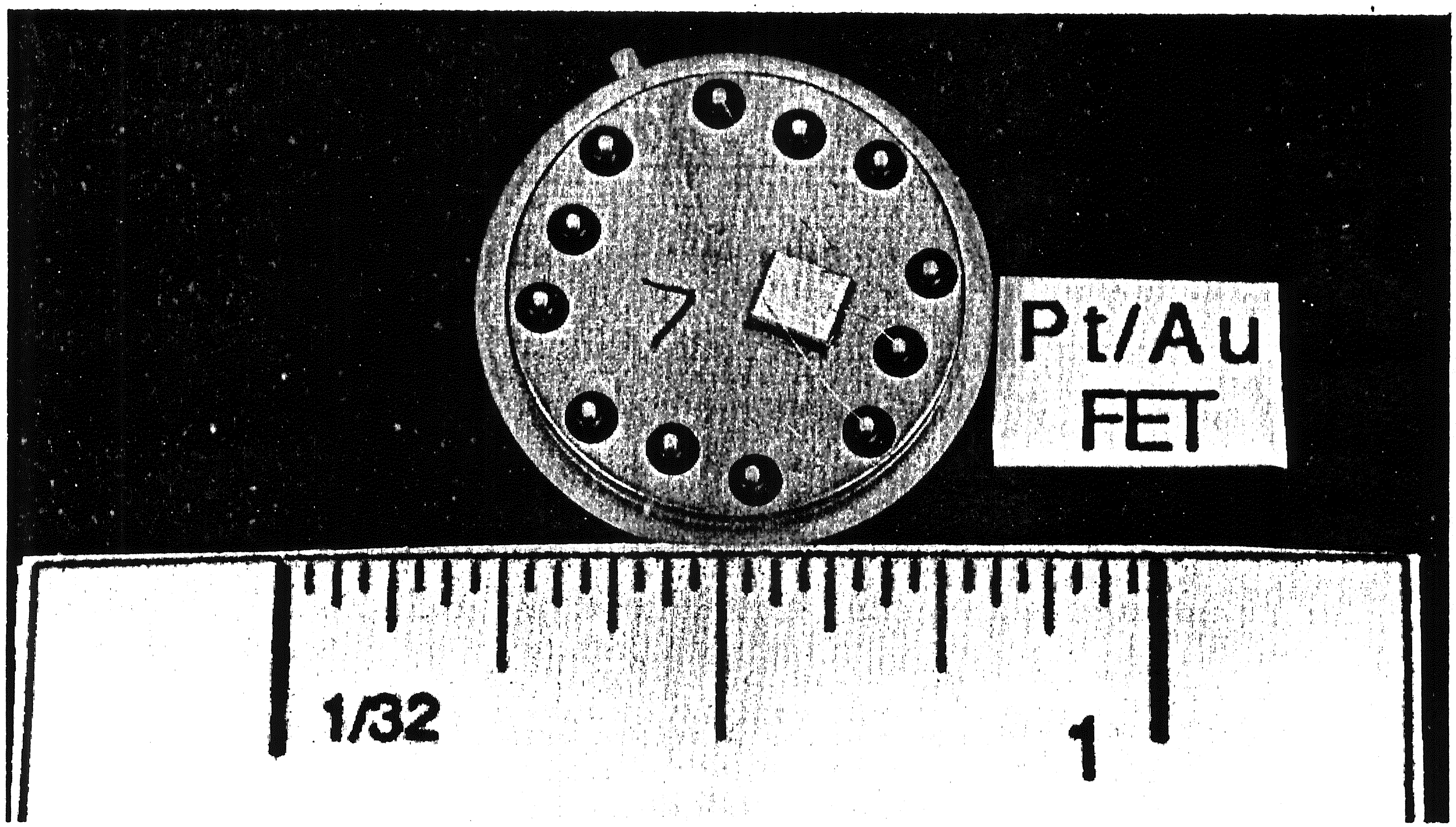
Three Candidate Sensors are Being Evaluated

- Pd/Ag MIS Diode
- Pt/Au Suspended Gate FET
- Temperature Conditioned Pd/Ag MIS Diode

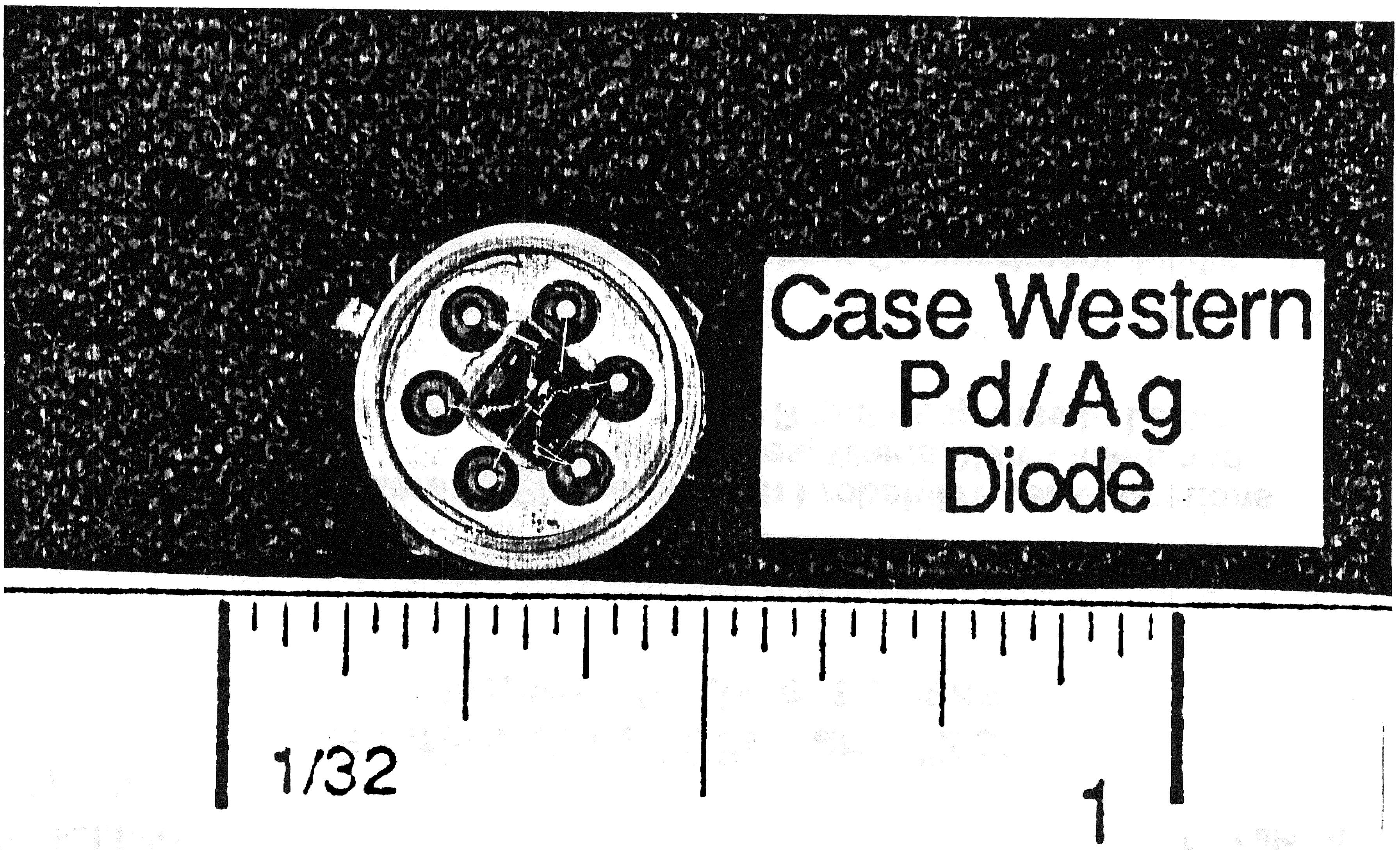
Palladium MIS Diode Hydrogen Sensor



Platinum FET Hydrogen Sensor



Heated Palladium MIS Diode Hydrogen Sensor



System Uses "Site" and "Zone" Sensors to Detect Leaks

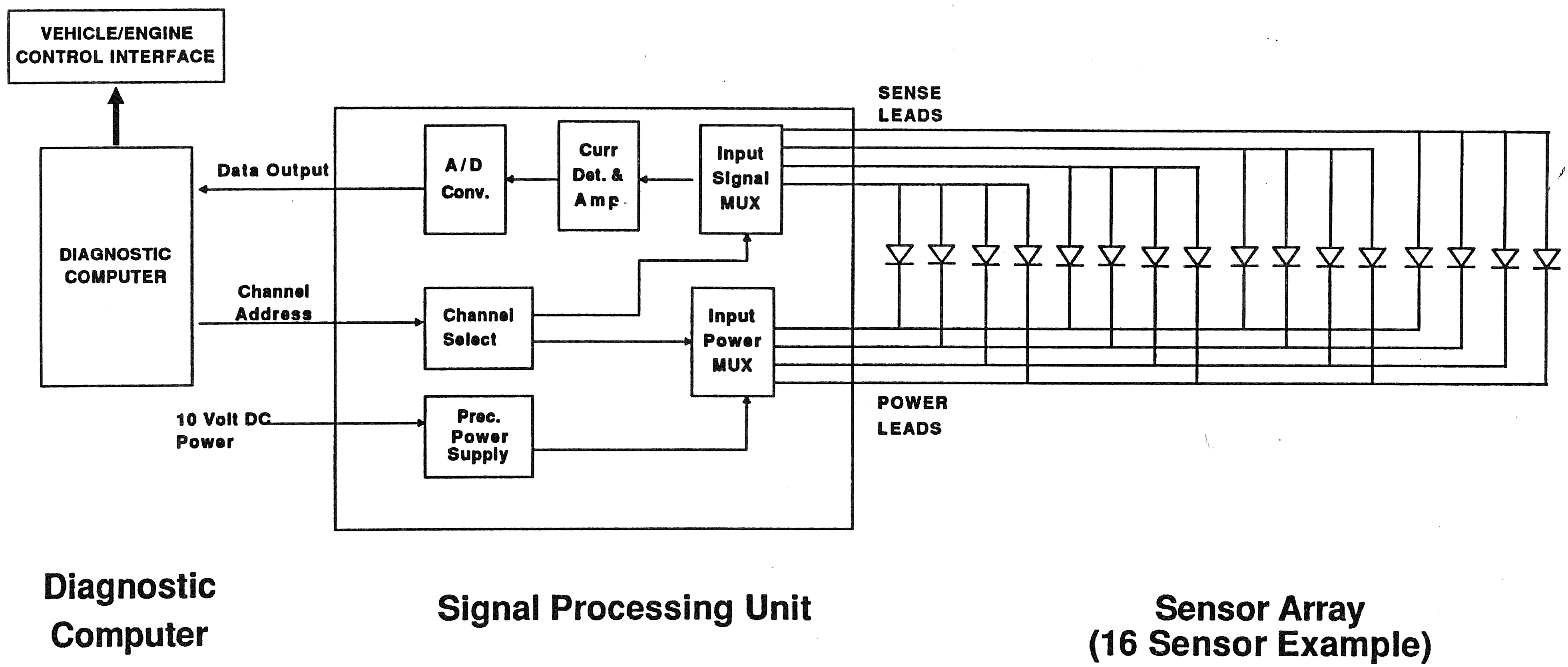
"Site" Sensors: Placed on High Probability Leak Locations Such as Flanges, Welds, Valve Inlets and Outlets. Gives Rapid Response to Leaks

"Zone" Sensors: Placed Throughout Compartment. Leaks are "Imaged" by Processing Concentration vs. Time from "Zone" Sensors.

Sensor Requirements

	Site Sensor	Zone Sensor
Response Time	$<$ 10 Seconds	
Recover Time	$<$ 5 Minutes	$\leq$ 1 Minutes
Senitivity	$10^2 - 10^3$ ppm	$\leq 10^2$ ppm
Saturation Limit	$>$ 1%	$>$ 4%
Operating Pressure	50 - 800 Torr	
Temperature	Must be Able to Withstand Proximity to Cryoganic Gas Regions	
Background Oxygen Concentrations	0-20%	

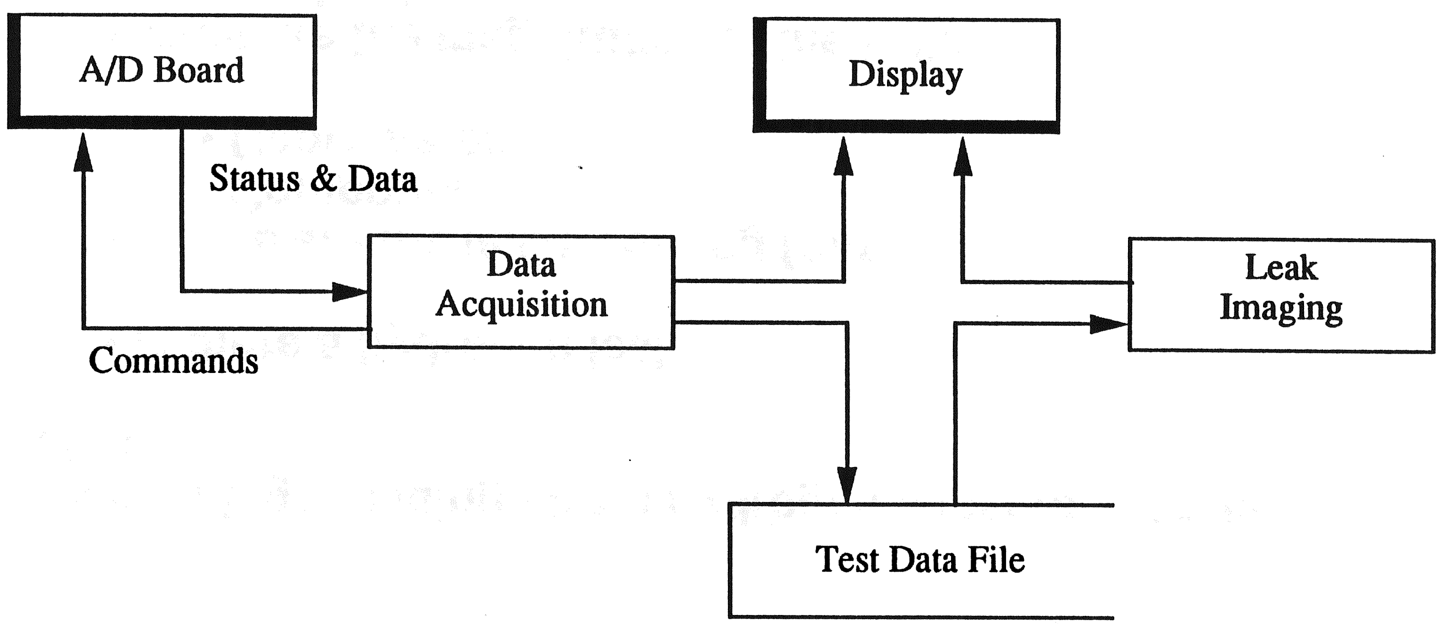
Signal Multiplexing, Conditioning and Conversion Process



GenCorp Aerojet

Propulsion Division

Top-Level System Dataflow

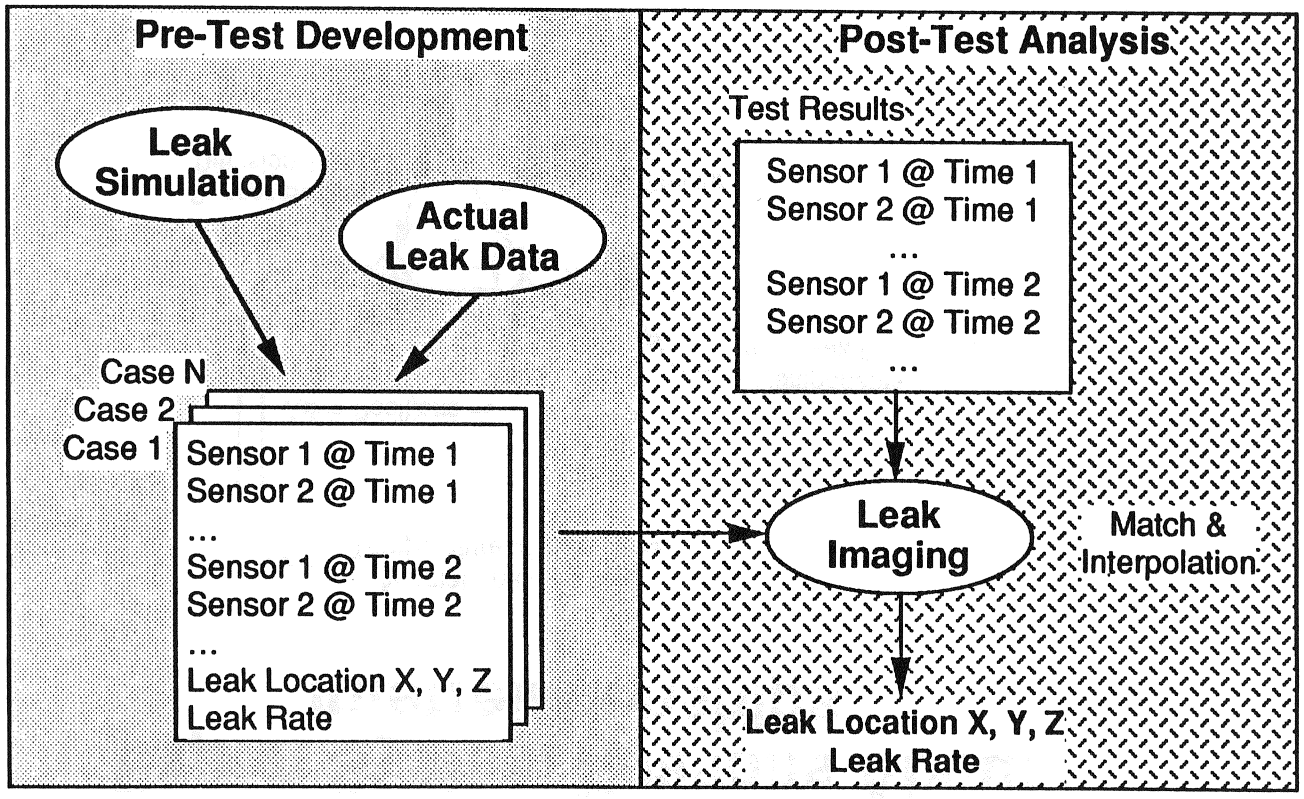


Knowledge-Based Software Interprets Sensor Readings

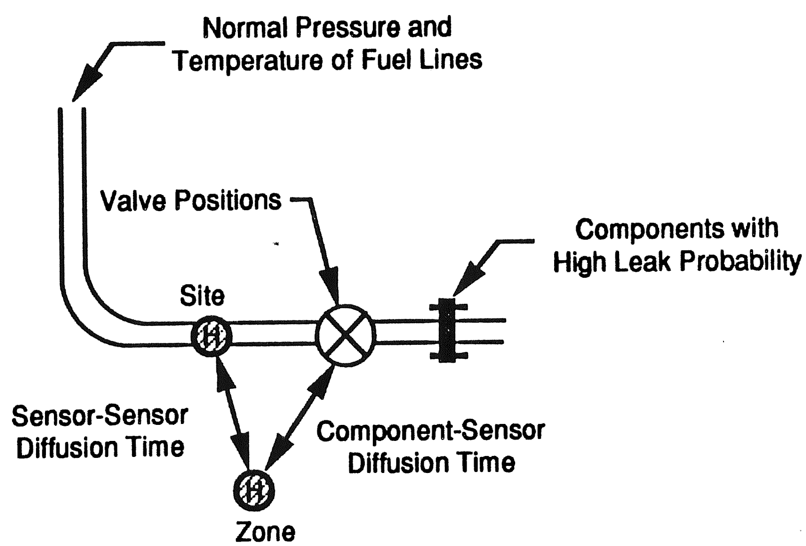
Given voltage readings from hydrogen sensors over time, determine:

- Is there a hydrogen leak?
- Where are the leaks coming from?
  - Component
  - Coordinates
- What are the magnitudes of the leaks?
  - Leak rate
  - Hydrogen concentrations
- What does the "hydrogen cloud" look like?
  - Visual feedback to test engineers

Case-Based Leak Imaging Provides Rapid Leak Identification



Expert System Reasons About Interrelationships



Example:

IF There is No Way to Explain the Relationship Between Two High Concentration Regions
THEN Hypothesize Multiple Leaks

IF a Concentration Region is Asymmetric WRT Diffusion Model
THEN Hypothesize Convection Flow

Solution Methodology

- Neural Network used to get "Ballpark" Location and Rate for Each Leak
    - System will be Trained Initially on Simulations
    - Learn Over Time as Real Cases Become Available

- Expert System Refines and Buttresses with Model-Based Reasoning
    1. ID Regions of High Concentration
    2. Determine Number and Regions of Leak(s)
    3. If Asymmetric, Estimate Convection Flow Magnitude & Direction
    4. Isolate Source of Each Leak to a Component
    5. Estimate Rate or Magnitude of Each Leak

Knowledge-Based Detection System Will Be Implemented With NASA Software

- Macintosh IIci, Color Monitor
- CLIPS Expert System Shell (NASA Johnson)
- NETS Neural Network Package (NASA Johnson)
- Scientific Visualization Package (Commercial Software)
- THINK C (Symantec)

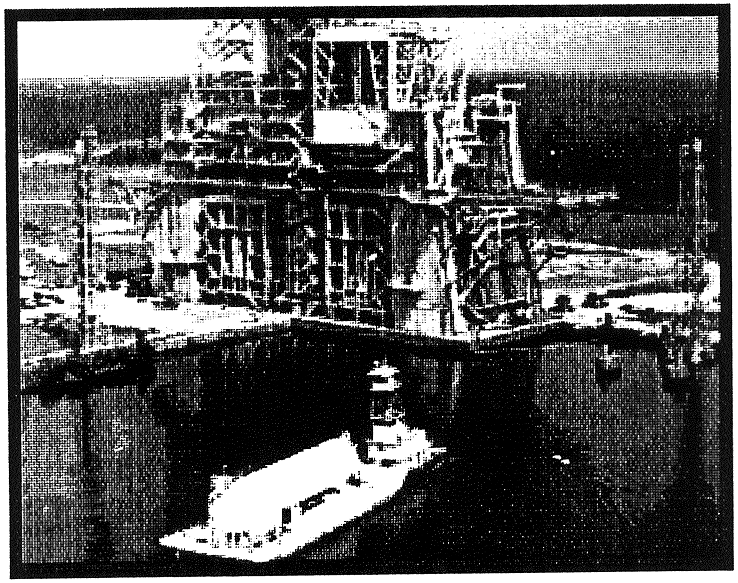
Technology Needs/Recommendations

- Continue Microsensor Technology Research
- Continue Leak Detetion System Development
- Concurrent Development of Fully Integrated Sensors
- System Implementation and Field Testing

LEAK DETECTION AT STENNIS SPACE CENTER

JAY D. HUNT

STENNIS SPACE CENTER
HYDROGEN DETECTION REQUIREMENTS
BACKGROUND



FACILITY

* WORLDS LARGEST HYDROGEN CONSUMER

* LARGE AND COMPLEX FACILITY
  - MANY POTENTIAL LEAK LOCATIONS

* NOW HANDLING 10 MILLION LBS / YR. H_2
  - SSME TEST SUPPORT

* FUTURE CONSUMPTION UP TO 20 MILLION LBS / YR. H_2
  - SSME, NLS, NASP

* DIVERSE REQUIREMENTS
  - BARGES / TRUCKS / TRANSFER LINES / STORAGE TANKS / SSME HARDWARE

LEAK MANAGEMENT SYSTEM NEEDS

* FAST RESPONSE
  - < 1 SECOND

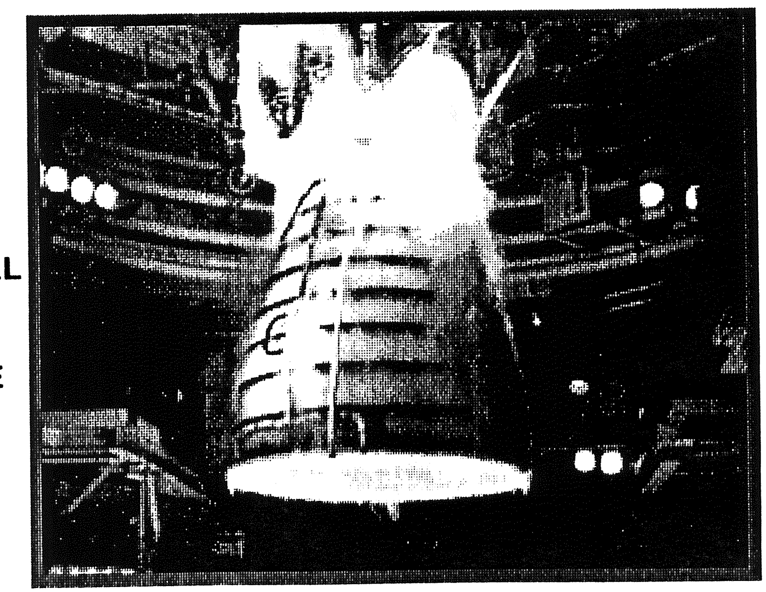
* WIDE OPERATING TEMPERATURE RANGE

* IMMUNITY FROM NOISE AND ENVIRONMENTAL INTERFERENCES

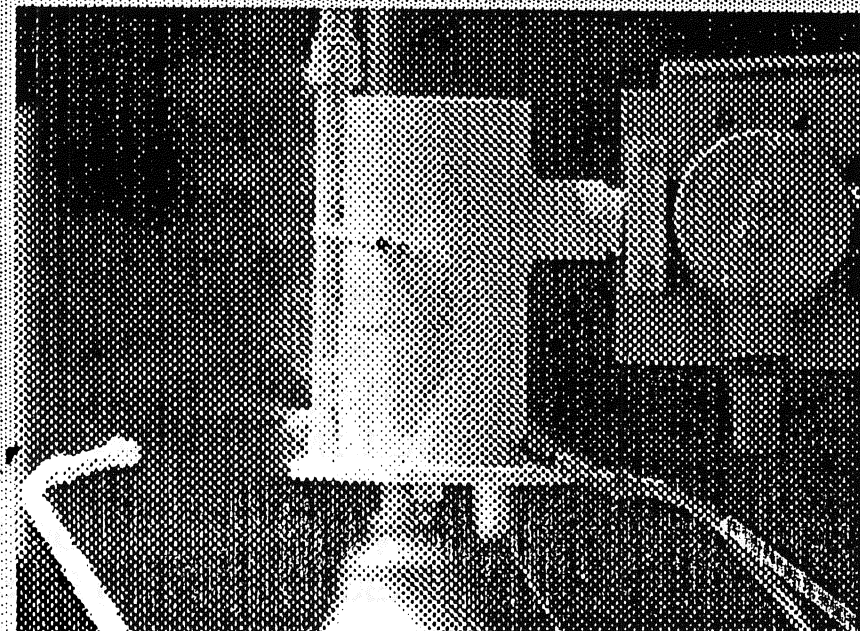
* LOW UNIT COST FOR WIDE AREA COVERAGE

* LOW OVERHEAD COSTS FOR REDUCED OPERATION EXPENSE

* ENHANCED AUTOMATION FOR IMPROVED MONITORING SYSTEM EFFECTIVENESS



DEVELOPMENT OF HYDROGEN DETECTION SYSTEMS AT SSC

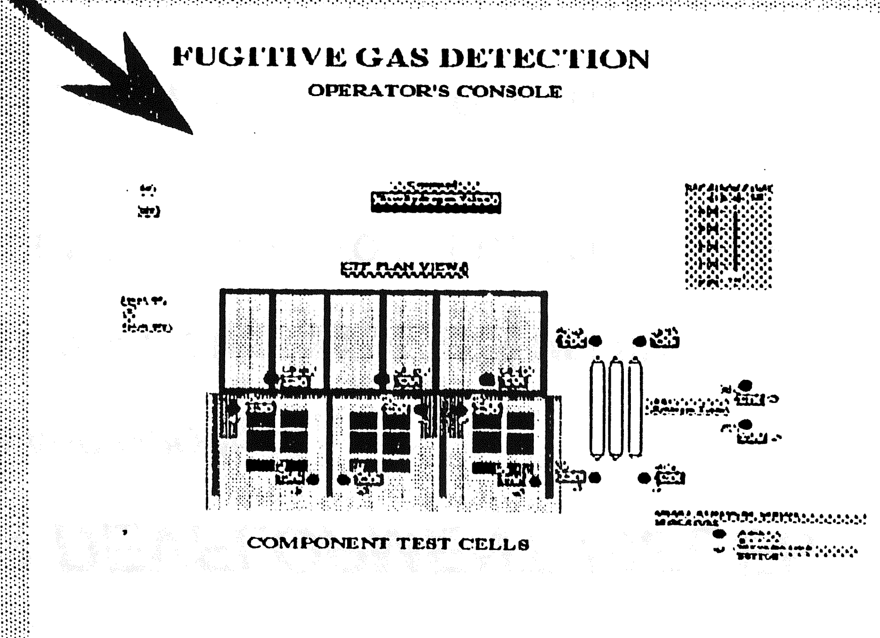


SMART HYDROGEN SENSOR (SHS)

- POINT OF LEAK SENSOR
- MICROPROCESSOR
- FASTER
- RUGGED AND RELIABLE
- WIDE OPERATING RANGE
- COMPLETED IN FY90
- INTEGRATED WITH FGDS FY91

FUGITIVE GAS DETECTION SYSTEM (FGDS)

- WIDE AREA MONITORING
- GRAPHICAL DISPLAY AND
- USER INTERFACE
- DIGITAL SENSOR NETWORK
- BETTER OPERATOR RESPONSE
- COMPLETED FOR CTF FY91
- OPERATOR TRAINING IN FY92



SSC HYDROGEN SENSOR DEVELOPMENT ISSUES

- WORLDS LARGEST CONSUMER OF HYDROGEN AND GROWING
- UNDEPENDABLE HYDROGEN DETECTION SYSTEMS = HIGH COST SAFETY MEASURES
- MORE COVERAGE OF VALVES, JOINTS, AND FITTINGS NEEDED TO REDUCE LOST PROPELLANT COSTS
- REDUCING OPERATIONS COSTS REQUIRES MORE COVERAGE, MONITORING AUTOMATION, AND GREATER SYSTEM DEPENDABILITY
- SSC'S NEAR TERM DEVELOPMENT INITIATIVE WILL BENEFIT OTHER NASA CENTERS AND PROGRAMS WITH LEAK MONITORING REQUIREMENTS
- SSC REQUIREMENTS DICTATE SYSTEM SPECIFICATIONS FOR:
  - ENHANCED AND AUTOMATED MONITORING CAPABILITY (FGDS)
  - WIDE SENSITIVITY AND TEMPERATURE RANGE
  - IMMUNITY FROM NOISE AND INTERFERENCES
  - LOW UNIT COST
  - HIGH RELIABILITY AND LOW OVERHEAD COSTS
  - HIGH SPEED RESPONSE

SSC SOLUTION

PROVIDE FOR NEAR TERM DEVELOPMENT OF HYDROGEN MICROSENSOR THROUGH CO-SPONSORSHIP OF SANDIA'S WRAM RESEARCH PROGRAM

OPTICAL LEAK DETECTION OF H_2

ROBERT N. HINDY
ROCKETDYNE DIVISION
ROCKWELL INTERNATIONAL FA60

OPTICAL NON INTRUSIVE LEAK DETECTION FOR THE SPACE TRANSPORTATION SYSTEM

Robert N. Hindy
(818) 718-4847

April 29, 1992

Rocketdyne Division
Rockwell, International

Rockwell International
Rocketdyne Division

AGENDA

Introduction - objective

Review of issues and requirements

Optical leak imaging: overview

Review of supporting technologies

Applications

Summary and Conclusions

Recommendations

OBJECTIVE

- Provide higher reliability system to improve safety
- Reduce Turnaround Time
    - Reduce Overall Cost

BENEFITS OF OPTICAL LEAK IMAGING TO SHUTTLE OPERATIONS

Current Shuttle Leak Detection Operation	Optical Leak Imaging
• Routine leak detection on existing shuttle program • Manpower intensive - (20,000 hrs/flight) • Not automated (less reliable, not repeatable) • Limited capability	• <2000 hours --non-intrusive, cryogenic • Computerized--more reliable • Flexibility
• Anomalous leak problem • trouble shooting → program delays, $	• Discriminates (H_2, O_2, N_2,) • Ground/On board • Rapid, Precise

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SHUTTLE LEAK DETECTION: SPECIAL ISSUES

- Large number of joints over a large area
  - Over 100 orbiter joints and 450 engine joints in Boattail alone
- Many ducts covered with insulating foam
  - Diverts leaks and disguises leak sources and magnitudes
- Current techniques inconclusive
  - Difficult to identify leak source
  - Difficult to determine leak severity
- Intricate, convoluted duct geometry
  - Complicates physical access to potential leak sources
- Launch commit criterion

SHUTTLE LEAK DETECTION DESIRES/NEEDS FOR REDUCED TURNAROUND/COST & ENHANCED RELIABILITY

GROUND

- Inspection, diagnostics and repair
  - Leak location to the joint level
  - Coarse determination of leak flow rates
  - Resolve down to hundreds of scims during cryogenic loading
  - Resolve under 0.5 scim prior to cryogenic loading
  - ~ minutes monitoring time

FLIGHT

- Monitoring Operational safety response:
  - Leak location to the major component level
  - Detection and identification of leaking propellant gas
  - Determination of total leak rate
  - ~1 second response time

KEY SELECTION CRITERIA FOR OPTICAL TECHNOLOGIES

- For all Shuttle applications:
  - Resistance to background effects
  - Adequate leak resolution and sensitivity
  - Calibration and operational requirements
  - Adequate detection speed
  - No potential hazard or interference with vehicle
- For propellant leak detection:
  - Ability to detect and distinguish H2 and O2
- For flight:
  - Flight suitability
  - Durability, weight

APPLICABILITY OF OPTICAL IMAGING TECHNIQUES

	Discrimination From Background Effects	Sensitivity to Gases	Leak Resolution	Speed	Possible hazard	Flight Traceability
IN-FLIGHT*						
Absorption	+	-	++	+	+	+
Fluoresence	+	-	+	+	+	+
Interferometric Methods	-	+	+	+	+	-
Refractive Methods	-	+	+	+	+	-
Rayleigh Scattering	-	+	+	+	+	+
Raman Scattering	+	($H_2O_2N_2$)	+	+	+	+
Condensed H2O,CO2 Visualization	-	+	-	+	+	+
BETWEEN-FLIGHT						
Propellant sensitive Paint	+	H2	+	+	-	+
Absorption	+	(SF_6,N_2O)	+	+	+	+
Soap solution	+	+	+	-	+	-

+: FAVORABLE
-: UNFAVORABLE

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AUTOMATED OPTICAL LEAK IMAGING

- General approach
  - Image components with camera or scanner
  - Use optical methods to make leaking gas visible in image
  - Use automated processing to identify, quantify leaks in image
- Advantages
  - Remote detection of leaks at their sources
  - Reduction of inspection time potentially orders of magnitude
  - High sensitivity to low leak rates

ADVANCED TECHNOLOGIES ARE AVAILABLE AND MATURING RAPIDLY

BASIC TECHNOLOGIES

- **Fiber Optics**--Aerospace/medical developments, imaging, smart skins
- **Lasers**--compact, diodes, wavelength control, imaging/surveillance
- **Electronics** -- computers, size, image processing

SYSTEMS APPLICATIONS

- **Condition Monitoring**
- **Diagnostics**--Raman combustion, PLIF, shock tunnel
- **Ground Based Leak Detection** (USAL, SSME TTB, ELV)
  - SF6 Gas, Optical Imaging Demo, ELV (Production)
- **Flight Leak Detection** (SSME TTB)

  Reducing size of laser systems (demo) Raman

OPTICAL LEAK IMAGING

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TEN YEARS OF OPTICAL LEAK DETECTION SYSTEM DEVELOPMENT AT ROCKETDYNE

1980

- Holographic leak detection lab demonstrations: IR&D (Patent)
- Ground-based automated leak imaging systems: USAFAL contract

1983

  - Differential absorption (patent)
  - Schlieren
  - Speckle interferometry
  - Quasiheterodyne holographic interferometry

- Propellant leak detection feasibility demo: IR&D

1985

  - Absorption-based oxygen leak detection in lab

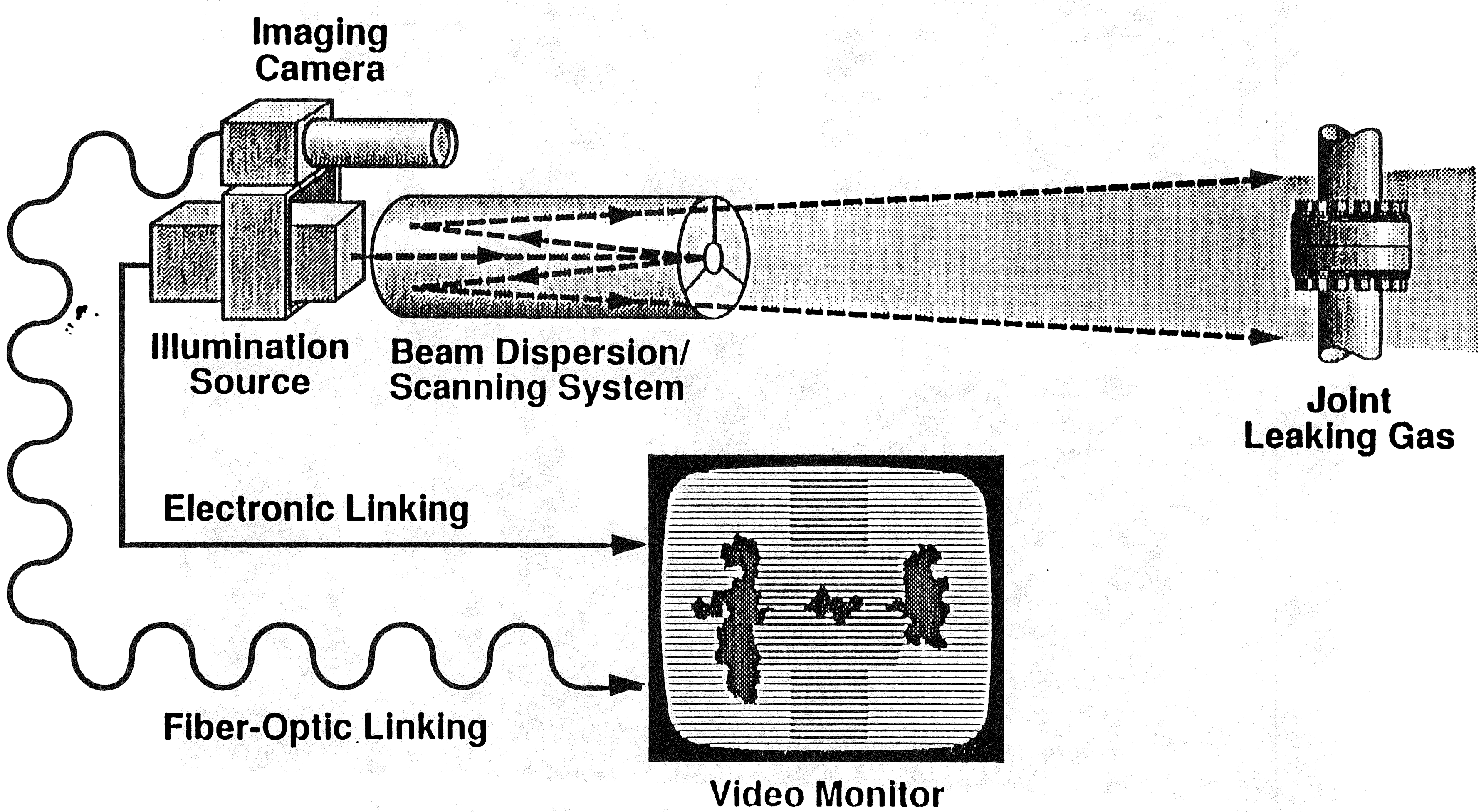
- Ground based engine leak detection task (MSFC, SSME TTBE)

1988 - CURRENT

  - Leak imaging using IR absorption technique
  - Focus on qualification of pressurant gas

- In-flight engine leak detection task (MSFC, SSME TTBE)
  - Raman-based detection of propellants
  - Detection of hydrogen leaks in lab test

Optical Leak Detection



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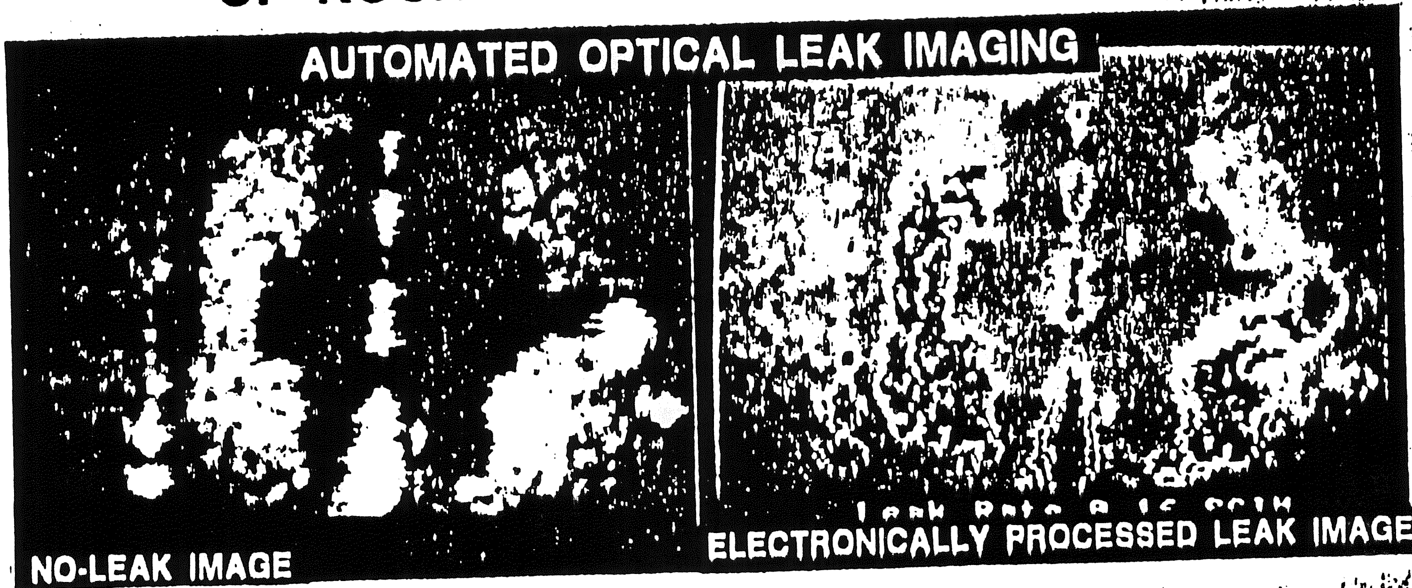
AUTOMATED OPTICAL LEAK INSPECTION OF ROCKET ENGINE COMPONENTS



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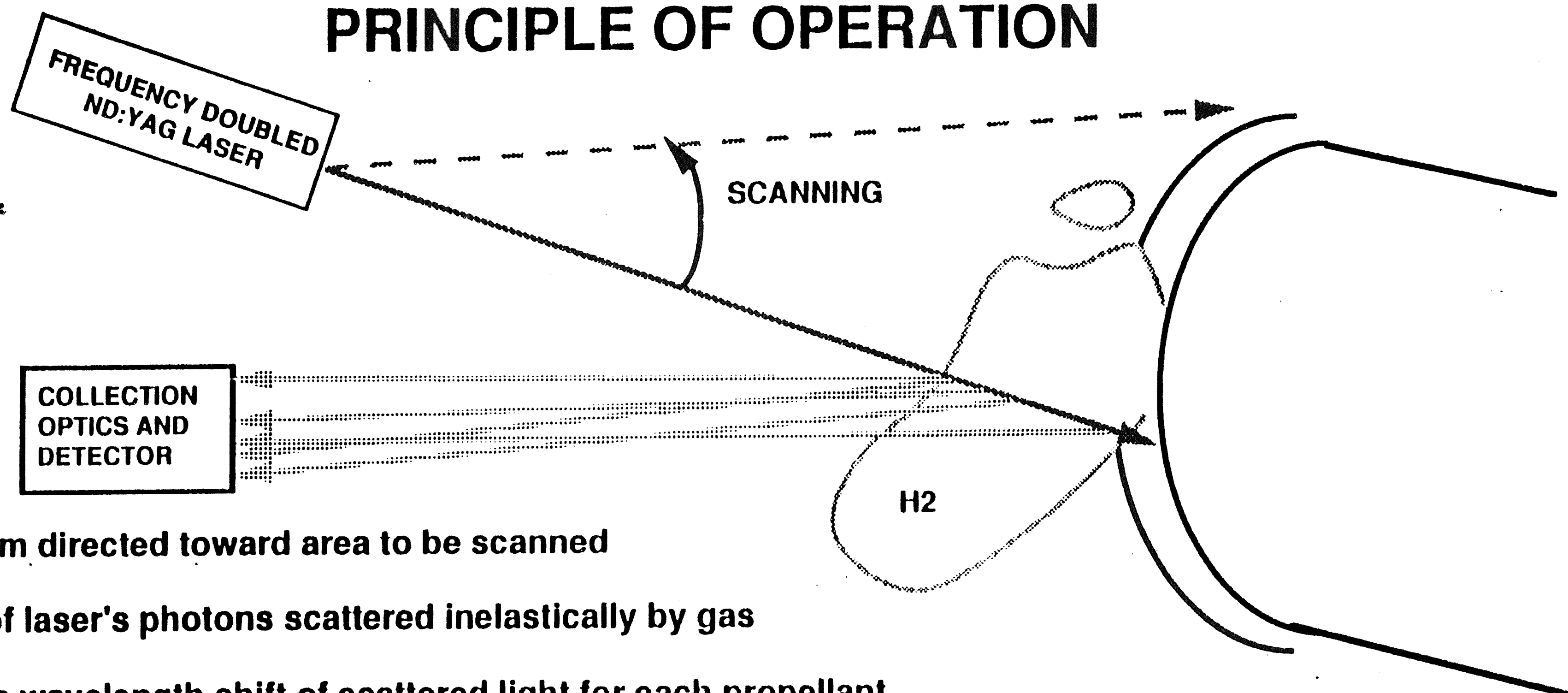
SC87C-4-1679

IN - FLIGHT LEAK DETECTION

TASK 17

RAMAN -BASED OPTICAL LEAK DETECTION: PRINCIPLE OF OPERATION



- Laser beam directed toward area to be scanned
- Fraction of laser's photons scattered inelastically by gas
    - <u>Unique wavelength shift</u> of scattered light for each propellant
- Scattered <u>light filtered</u>, collected and detected optically
    - Intensity proportional to leaking gas concentration
- Test area <u>scanned</u> to create <u>image</u> of leaking gas
- Measurements integrated to determine gas quantities and leak rates

RAMAN BOAT TAIL PROPELLANT LEAK DETECTION APPROACH

- 532 mm visible laser source remotely located illuminates potential leaking joints through a system of fiber optics and scanners

- Raman scatter from H_2 @ 683 mm H2O 660 nm, N2 608 nm, O2 578 nm is coupled through a series of scanners and fiberoptics back to a high sensitivity detector

- Entire system is controlled by microprocessor and cycles through all potential leaking sites

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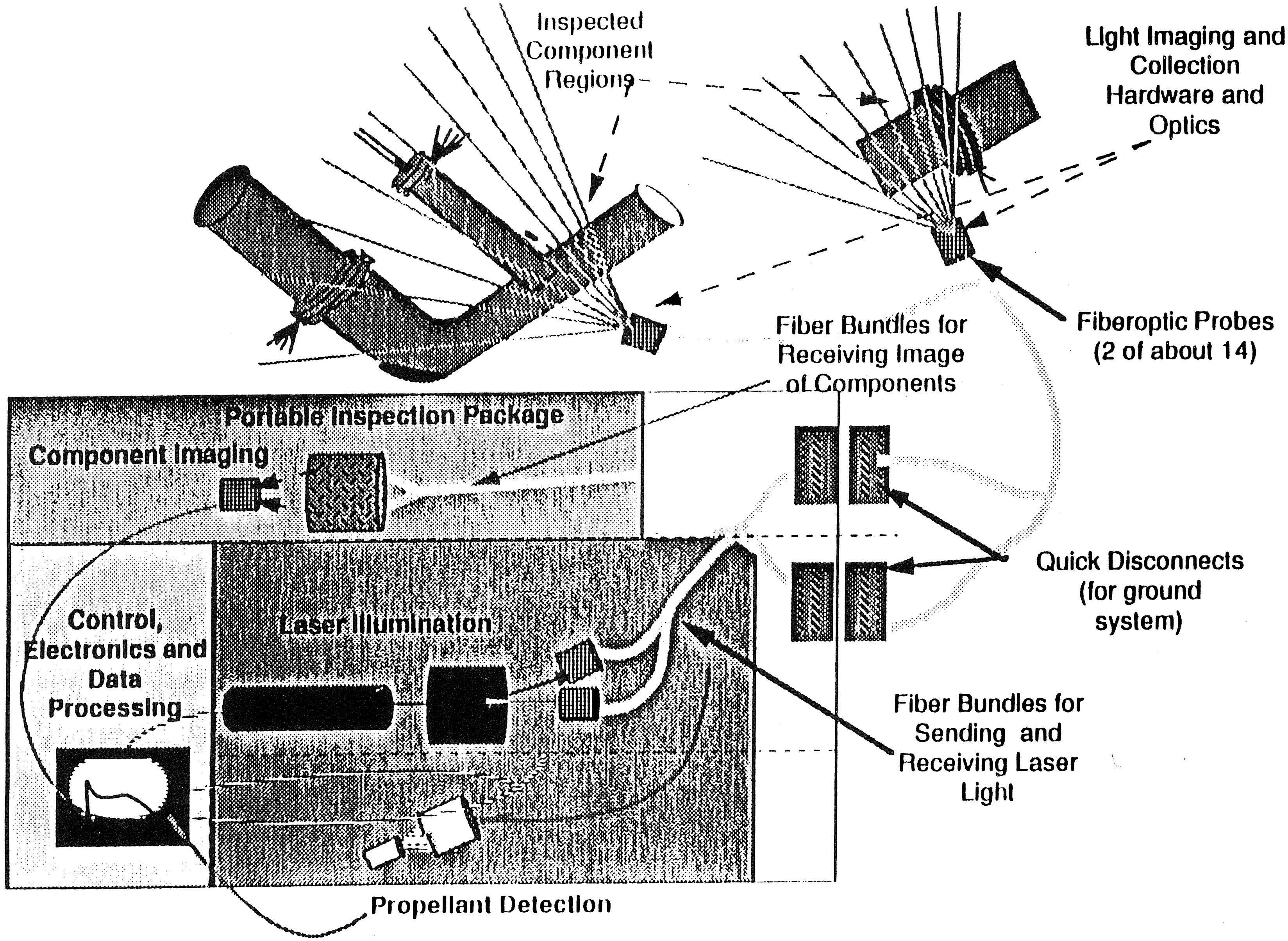
BASELINE SYSTEM



IN - FLIGHT LEAK DETECTION

THEORY OF SCANNING TECHNOLOGY

- A LASER BEAM SCANS ACROSS THE FIELD OF VIEW OF AN OPTICAL SYSTEM
- THE OPTICAL SYSTEM ACCEPTS LIGHT FROM THE REGION THE LASER BEAM PROBES AT ANY GIVEN TIME
- PRESENCE OF H_2 OR O_2 IS REVEALED AS BACKSCATTERED, RAMAN - SHIFTED LIGHT ON A PIXEL BY PIXEL BASIS
- A REAL - TIME, SHOT - NOISE LIMITED IMAGE OF THE LEAK IS CONSTRUCTED

IN - FLIGHT LEAK DETECTION

PROGRESS AS OF END FISCAL 91

- THE CONCEPT HAS BEEN DEMONSTRATED FOR A CONTINUOUS WAVE LASER
- SHOT NOISE LIMITED PERFORMANCE HAS BEEN EXPERIMENTALLY VERIFIED AS A SCALING LAW
- SCALING LAW PREDICTS PERFORMANCE FOR ANY SET OF DESIGN PARAMETERS

OTHER NASA EFFORTS WHICH MAY EMPLOY THE SAME TECHNOLOGY:

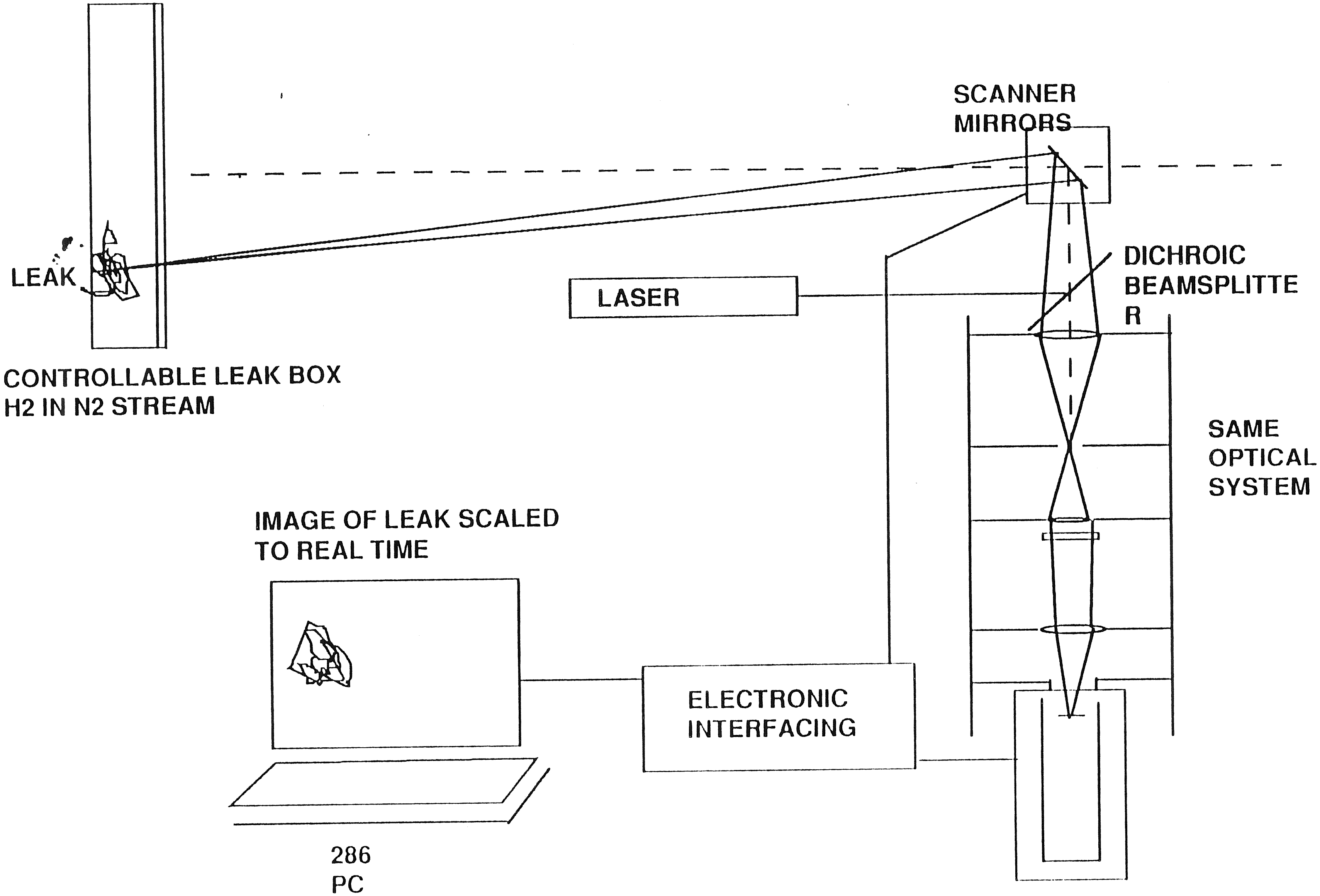
- NASP

IN - FLIGHT LEAK DETECTION

CURRENT YEAR TASK OBJECTIVES

- CONVERT TO A PULSED LIGHT SOURCE FOR S/N IMPROVEMENT
- CONSTRUCT A SCANNING IMAGING DEMONSTRATOR TO IMAGE H_2 LEAKS
- DEVELOP SOFTWARE TO ACCUMULATE AND SCALE THE IMAGERY TO REAL TIME
- START CONVERSION OF SCANNING TECHNOLOGY TO MORE SOPHISTICATED MICROCHANNEL PLATE TECHNOLOGY
- QUICKLY TEST A NEW INTENSIFIED CAMERA (PRINCETON INSTRUMENTS), REPORT RESULTS TO CUSTOMER

IN - FLIGHT LEAK DETECTION
PHASE III IMAGE DEMONSTRATOR



IN - FLIGHT LEAK DETECTION

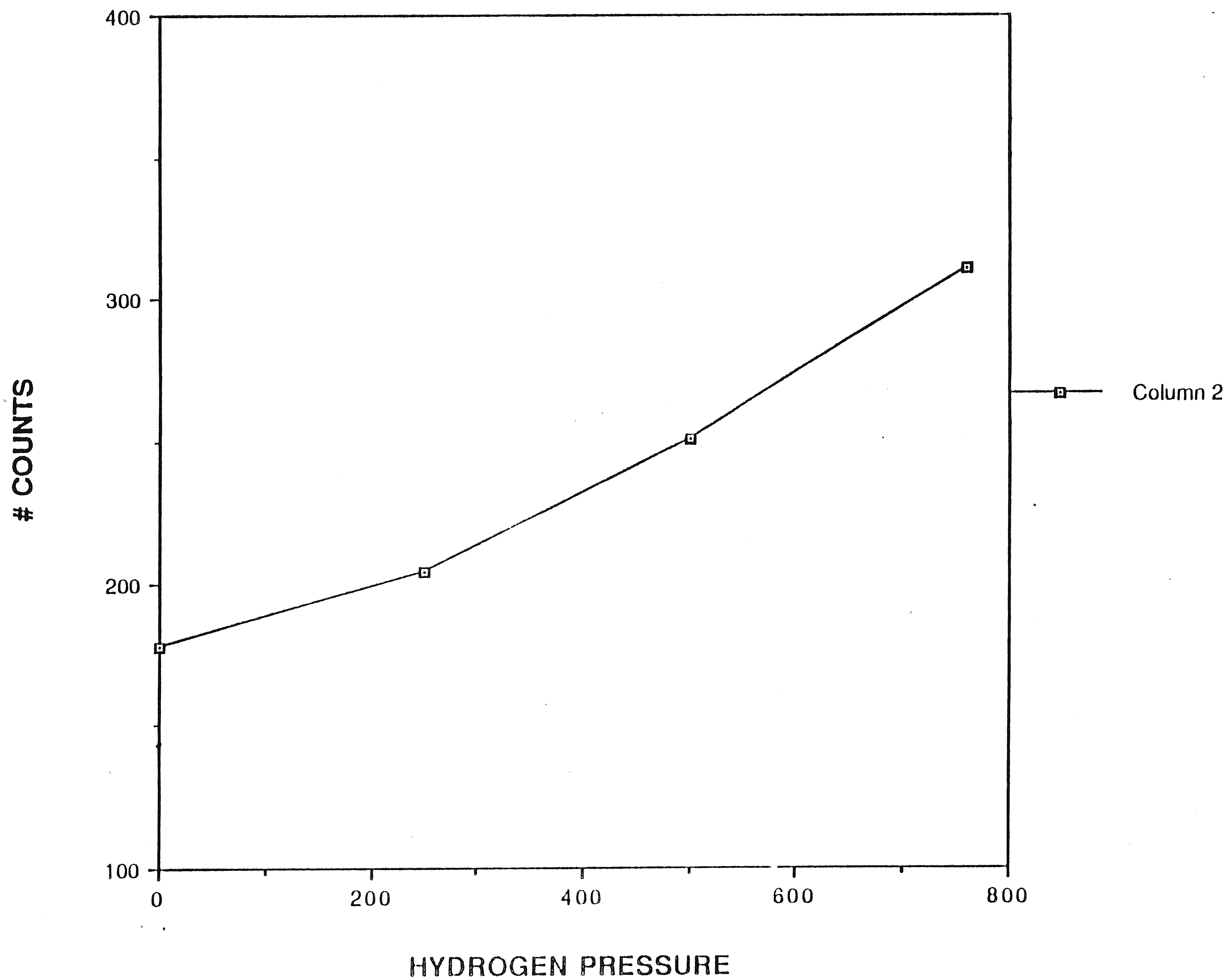
CURRENT YEAR WORK ACCOMPLISHED: DETECTED RAMAN SIGNAL USING PULSED LASER, TESTED PRINCETON INSTRUMENTS CAMERA

WORK TO BE PERFORMED: PERFORM MORE TESTS WITH PULSED LASER, START WRITING SOFTWARE, START BUILDING IMAGING SYSTEM

KEY TECHNICAL CHALLENGES:

- INTEGRATE ALL THE HARDWARE, AVOIDING PROBLEMS SUCH AS GROUND LOOPS, SAFETY FAILURES, ETC.
- SOFTWARE IS THE CRITICAL INTEGRATION ELEMENT, ESP. IMAGING PROCESSING SOFTWARE

COUNTS VS. H2 PRESSURE
1ST RUN 4/3/92 1000 PULSES   10 microJ



IN - FLIGHT LEAK DETECTION

FUTURE PLANS:

- DEVELOP A USEFUL INSTRUMENT: COMPACT, LIGHTWEIGHT, ETC. EASY TO USE AND CONFIGURE
- INTEGRATE INTO TTB IN 1993
- CUSTOMIZE IMAGE PROCESSING TO THE SSME ENVIRONMENT, QUANTUM NOISE ENVIRONMENT, AND HUMAN PERCEPTION INTERFACE

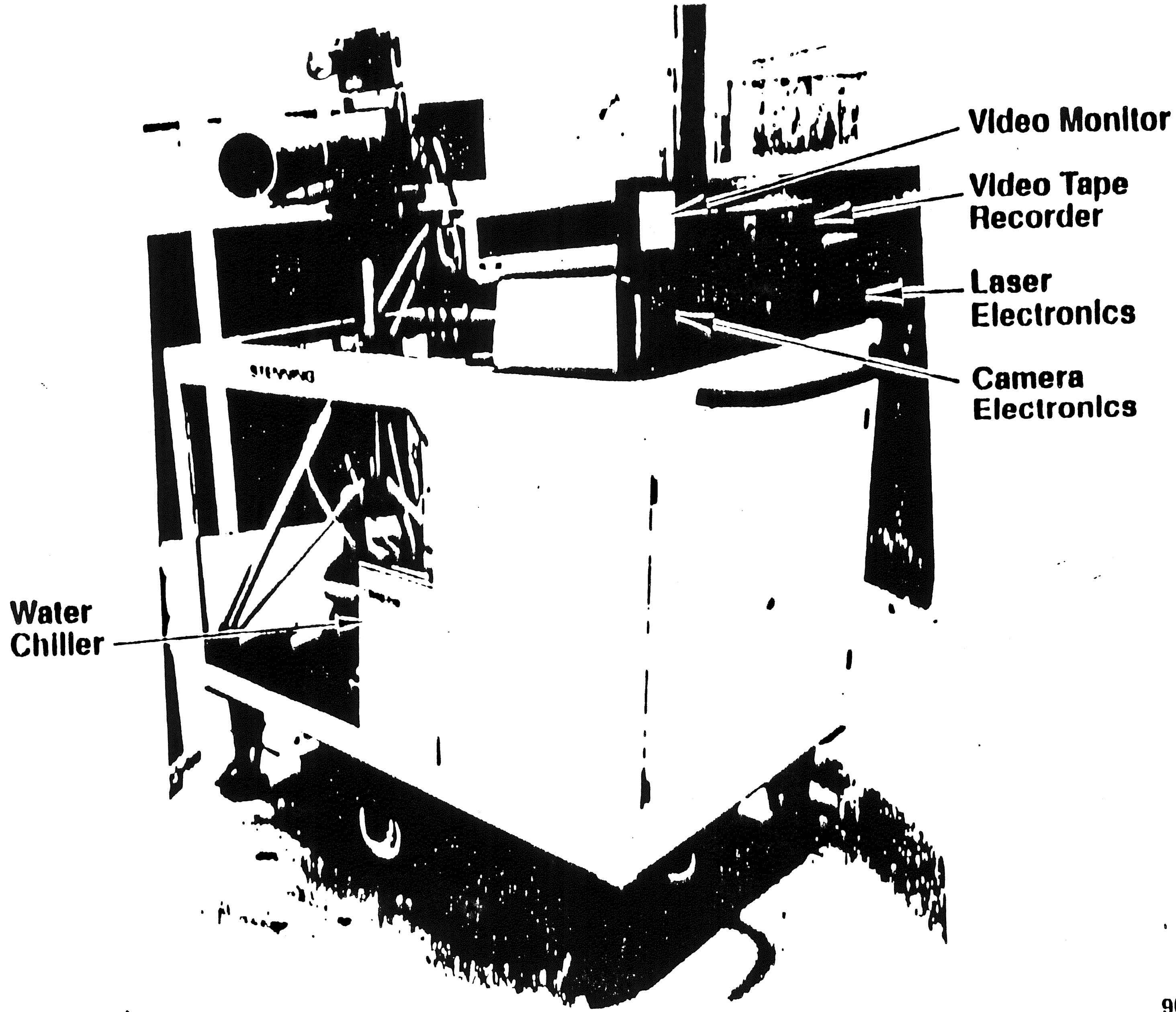
TTB TASK #18

GROUND-BASED LEAK DETECTION

FACTORY APPLICATIONS OF OPTICAL LEAK IMAGING

- SSME heat exchanger (training fixture)
  - Leak detected with sniffer but could not be located
  - Optical system located leak source
- SSME mockup video
  - Multiple leaks imaged and located optically
- Peacekeeper
  - Leaks located in fabrication fixture
- Delta RS27 (production engine) nozzle--
  - Leak detected by mass spec & water, but could not be localized
  - Leak localized optically to two-tube region

Cart-Mounted Leak Detection System



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Rockwelldyne Division

90c-4-1498
M083

TTB GROUND-BASED LEAK DETECTION

PRIOR YEARS WORK:

PHASE I:

- COMPLETED A COMPREHENSIVE STUDY OF SEVEN ALTERNATIVE TRACER GASES.
- CONFIGURED OPTICAL LEAK DETECTION SYSTEM FOR MANUFACTURING ENVIRONMENT.
- SUCCESSFULLY IMAGED LEAKS ON SSME TOOLING NOZZLE.
- PROVED THAT HIGHLY DILUTE TRACER GAS CONCENTRATIONS (< 2 %) WERE EFFECTIVE FOR LEAK IMAGING.

PHASE II:

- EXPLORED IMPROVEMENTS IN OPTICAL LEAK DETECTION SYSTEM UTILITY / PORTABILITY THROUGH THE USE OF A PASSIVE (NON-LASER) ILLUMINATION TECHNIQUE.
- COMPLETED NITROUS OXIDE TRACER GAS STUDY.
- DEMONSTATED AMBIENT INFRARED LEAK DETECTION CAPABILITY WITH NITROUS OXIDE TRACER GAS AND AN UNMODIFIED IMAGING SYSTEM.
- NITROUS OXIDE APPROVED FOR USE ON ALL SSME SUBSYSTEMS.

TTB GROUND-BASED LEAK DETECTION

NITROUS OXIDE LEAK IMAGING SYSTEM DEMO AND EVALUATION

- IN COOPERATION WITH THE SSME POWERHEADS AND DUCTS DEPARTMENT AN SSME NOZZLE WAS SET ASIDE TO USE FOR NITROUS OXIDE LEAK TESTING AND EVALUATION

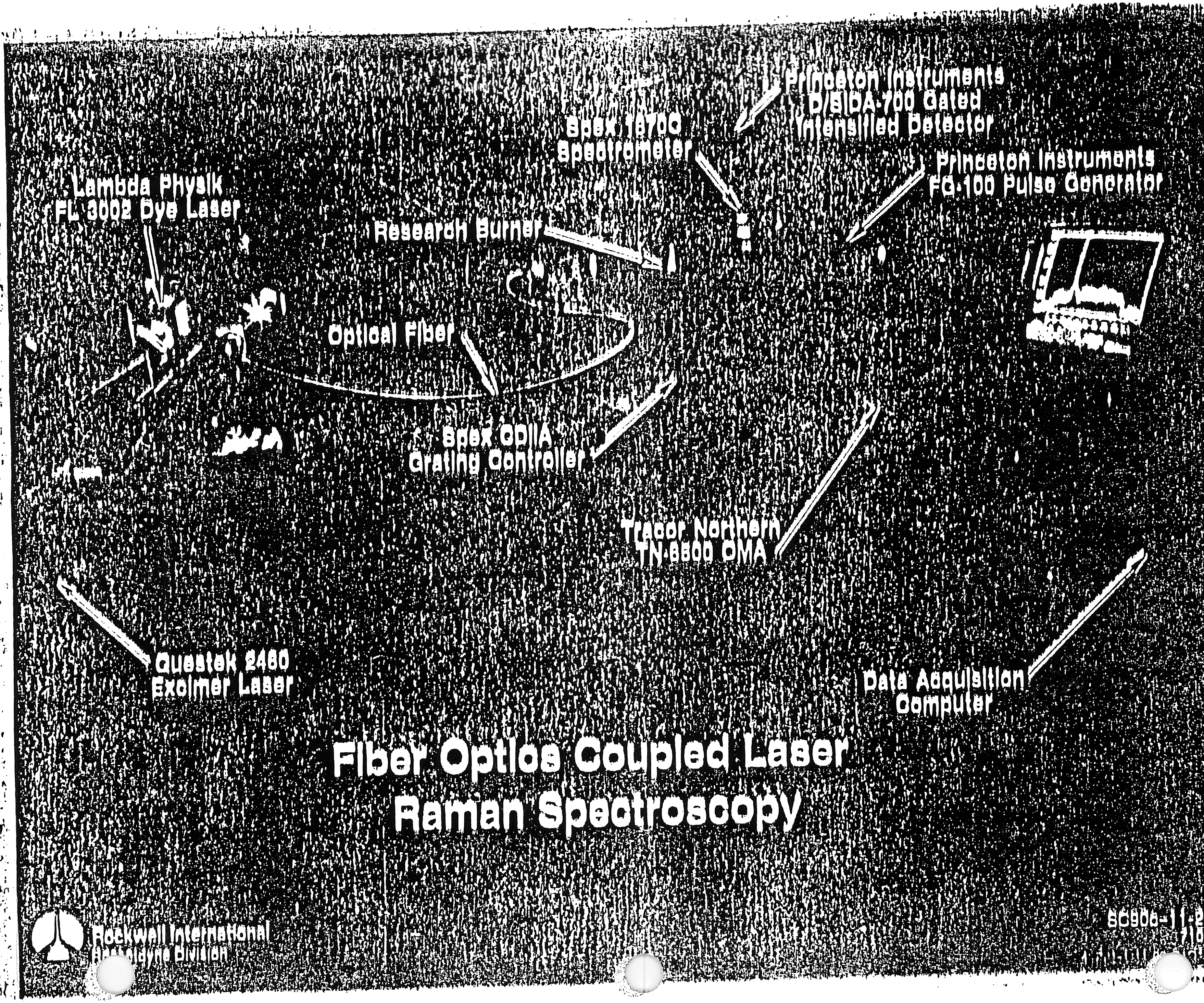
- AN AMBER ENGINEERING, STOCK 128X128 FOCAL-PLANE-ARRAY IR CAMERA WAS OBTAINED FROM THE MANUFACTURER AS A MARKETING DEMONSTRATION BY THEM AND WAS OPERATED BY THEIR SALES ENGINEER DURING THE N2O LEAK TESTING

- WITH NO APPLICATION-SPECIFIC SOFTWARE OR HARDWARE ON THE IR CAMERA SYSTEM , UNCALIBRATED LEAKS WERE IMAGED ON THE NOZZLE WITH THE HELP OF IR LIGHTING AND AIR-FLOW VARIATIONS, AS WELL AS CAMERA GAIN AND OFFSET ADJUSTMENTS

- LEAK-RATE CAPABILITY EVALUATION TESTING OF THE AMBER 4128 IR CAMERA WAS PERFORMED AT THE MANUFACTURER'S TEST LAB. NITROUS OXIDE CALIBRATED LEAK RATES OF LESS THAN 0.25 SCIM WERE OBSERVED

SUPPORTING TECHNOLOGY FOR LEAK DETECTION

- Fiberoptic RAMAN Combustion Monitoring
- Fiberoptics for Harsh Environment--NASA LEWIS Video
- Multipoint Multiparamete Combustion Diagnostics--NASA LEWIS
- NASP Diagnostics
- In-flight H2 Detection
- Hypersonic Tunnel Combustion Diagnostics
- Imbedded Flame Sensors



Princeton Instruments
D/SIDA-700 Gated
Intensified Detector
Spex 1870C
Spectrometer
Princeton Instruments
FG-100 Pulse Generator
Lambda Physik
FL 3002 Dye Laser
Research Burner
Optical Fiber
Spex CDIIA
Grating Controller
Tracor Northern
TN-6500 OMA
Questek 2460
Excimer Laser
Data Acquisition
Computer
Fiber Optics Coupled Laser
Raman Spectroscopy
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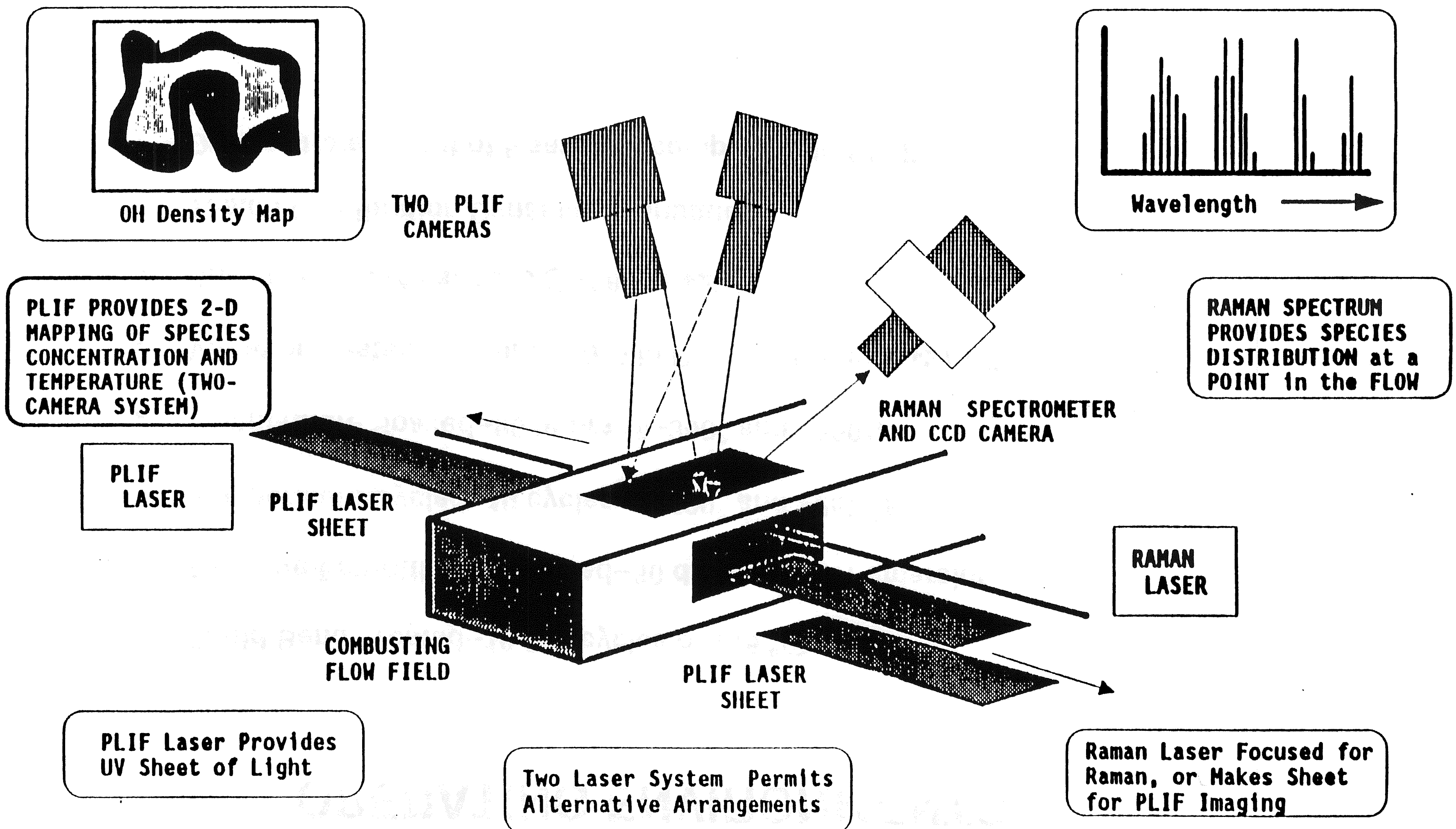
SC906-11-2

SIMULTANEOUS PLIF AND RAMAN SPECTROSCOPY for MULTIPOINT, MULTIPARAMETER DIAGNOSTICS

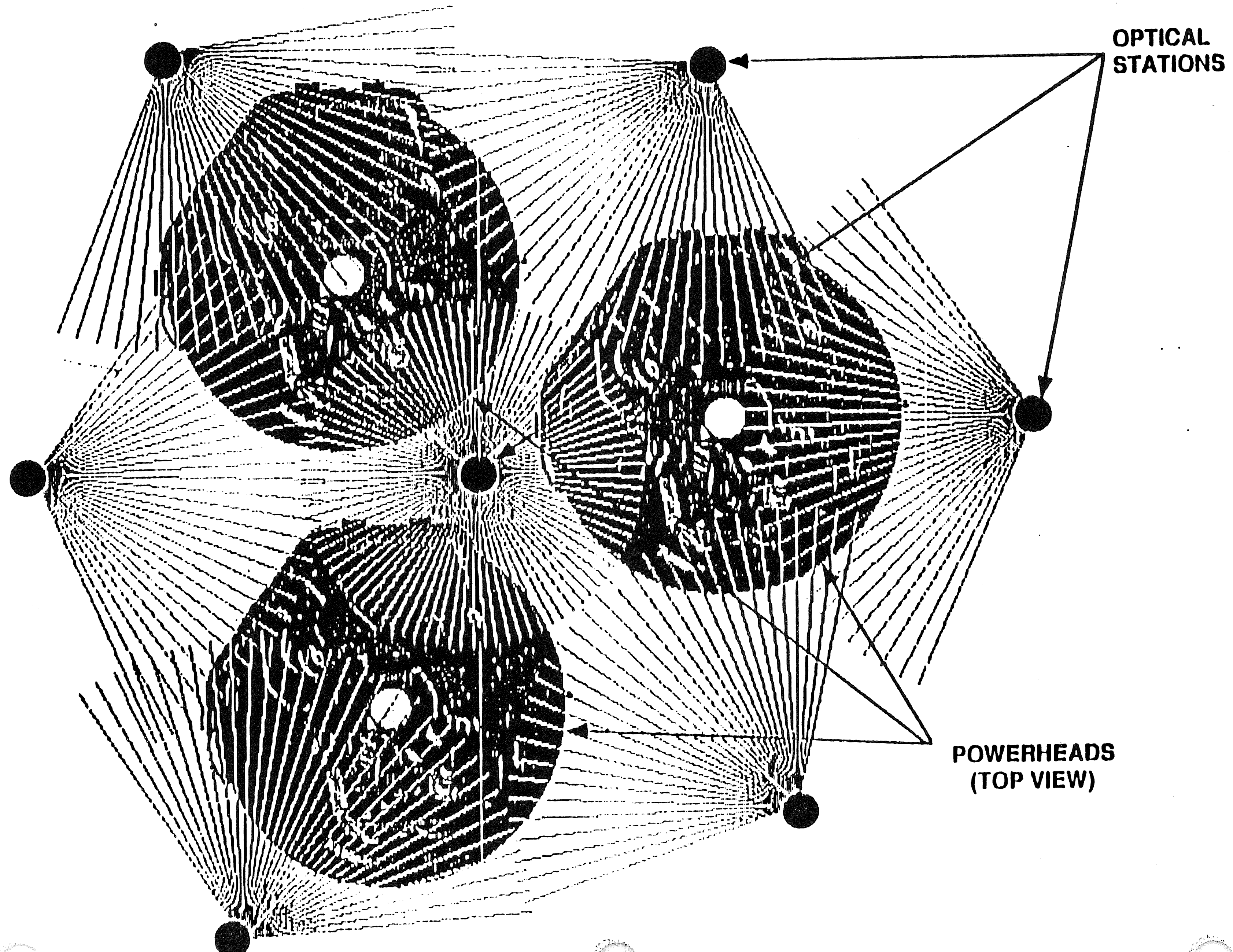


FIBER OPTICS FOR EXTREME OPERATING ENVIRONMENTS

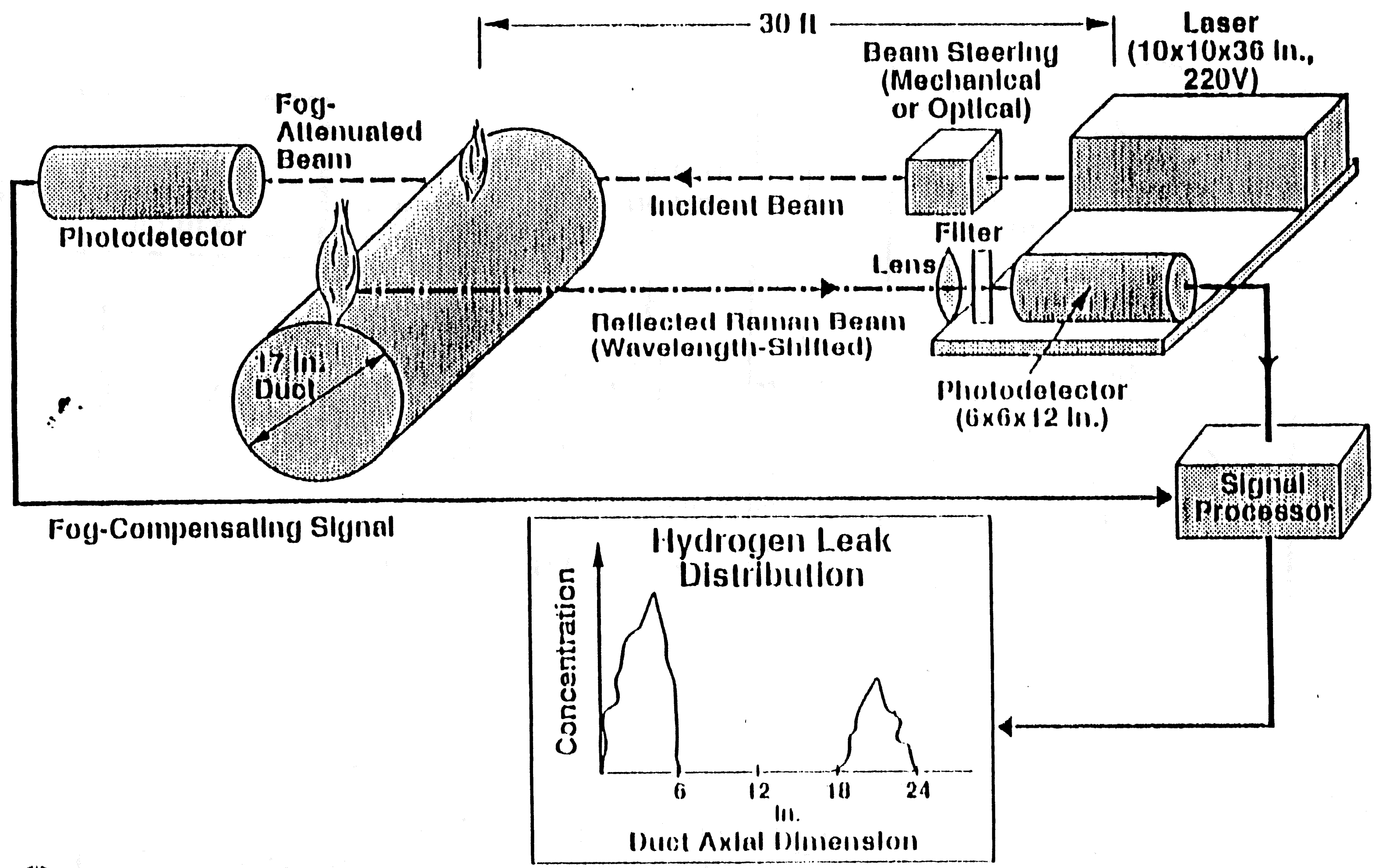
- "Cold-Bend" Tested--10,000 cycles of $\pm 45^{\circ}$ @ -300°F
- Moisture Embrittlement Tested--30 days of water emersion
- Temperature Cycled--10 cycles of -300° and $+257^{\circ}$F
- Temperature Soaked--96 hours @ -300° and $+500^{\circ}$F
- Vibration Tested--1 hour @ 40 G in R.T., -280°F, and $+275^{\circ}$F
- Shock Tested--48 shocks @ -280 to $+275^{\circ}$F
- Tested for Vibration/Dynamic Attenuation
- Demonstrated 3 out of 5 samples compatible to SSME

APPLICATION
LEAK DETECTION

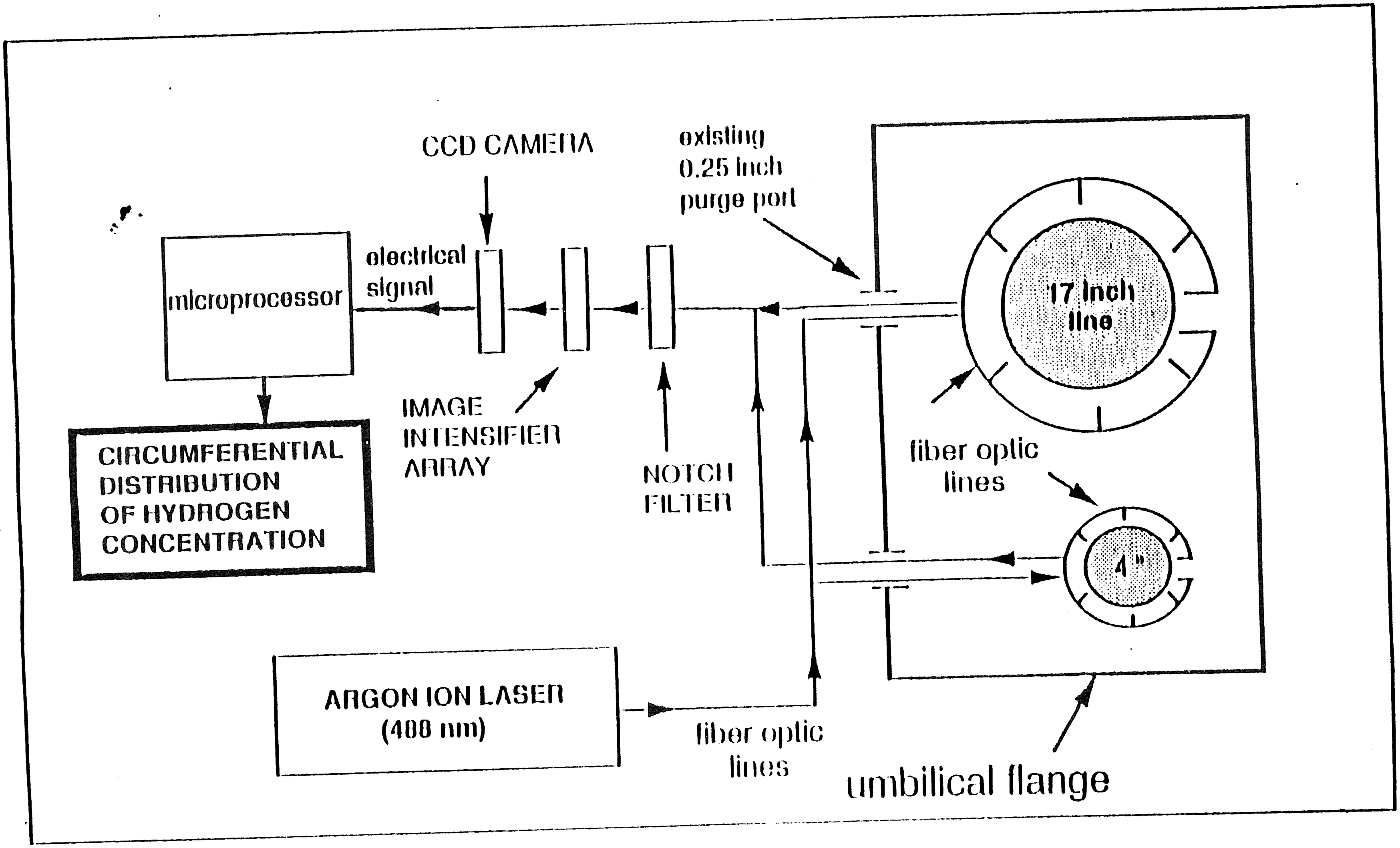
POTENTIAL SYSTEM CONFIGURATION : BOAT TAIL OPTICAL LEAK DETECTION



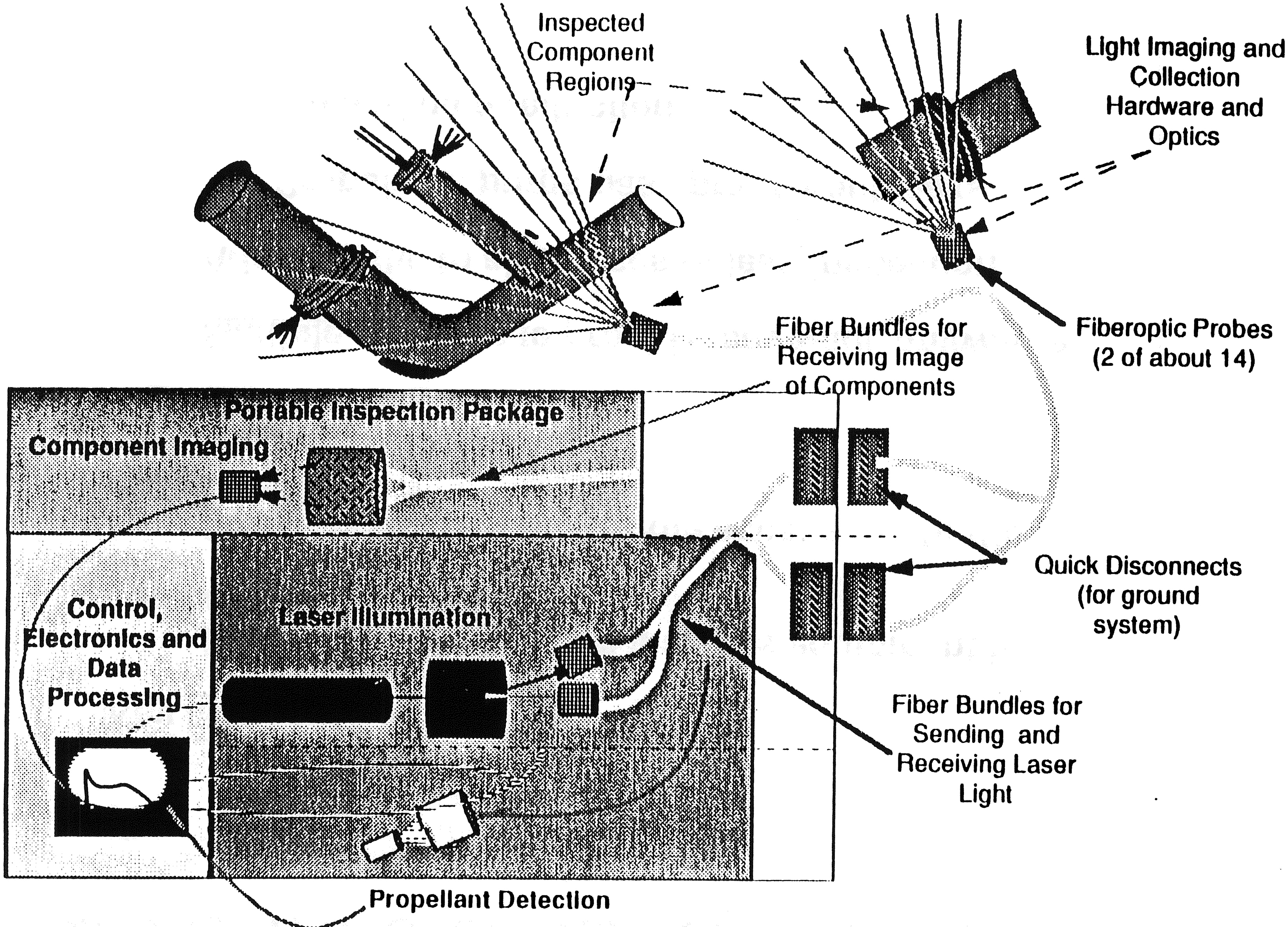
Conceptual Optical Leak Detection System For 17-In. Duct Simulator



BLOCK DIAGRAM OF LASER RAMAN REMOTE HYDROGEN DETECTION SYSTEM



BASELINE SYSTEM



RECOMMENDED SYSTEM APPROACH: MODULAR SYSTEM USING FIBEROPTIC PROBES

Key elements

- laser, imaging and data analysis equipment in compact modular package
- fiberoptic probes for remote access to Shuttle components

Design philosophy

- Multiple modules to provide coverage anywhere
- Adaptability to all classes of leak inspection
  - Propellant, purge gas, pressurant leaks
- Automated data reduction

SYSTEM ISSUES FOR SHUTTLE APPLICATION

- Determine configurational requirements for adequate coverage
  - Effect of optical obstructions
  - Number of discrete optical stations
  - Application of fiber optics
  - Location of system components
- Identify and evaluate mission-suitable components
  - Lasers & detectors
  - Fiberoptics; mounting and coupling hardware
  - Scanning and signal processing instrumentation
  - Laser, electrical safety
- Optimize system sensitivity vs weight, speed and spatial resolution
- Define and develop automated inspection capability
  - Scanning and image construction
  - Leak detection and location
  - Assessment of hazard and required response
- Integrate into a ruggedized system package

SUMMARY

- Optical leak imaging essential for rapid leak inspection
- Rocketdyne is commited to optical leak imaging system development
    - 10 years of IR&D and contract development
    - Optical leak imaging systems demonstrated and in use
- Technology programs ongoing for flight and ground leak detection development and implementation
    - Technology available at component level
    - Fiber optics, lasers, detectors, electronics
- Baseline system conceptualized

CONCLUSIONS

OPTICAL LEAK IMAGING CAN ENHANCE RELIABILITY, SAFETY, AVAILABILITY

- TECHNOLOGY MATURITY DEMONSTRATED
- RAPID, AUTOMATIC, NON INTRUSIVE, LARGE AREA

IMPLEMENTATION OF THIS OPTICAL LEAK DETECTOR CAN DRAMATICALLY REDUCE SHUTTLE TURNAROUND TIME AND COSTS

RECOMMENDATIONS

- INITIATE ACCELERATED PROGRAM TO DEVELOP OPTICAL LEAK INSPECTION SYSTEM
  - *GOAL*: GENERIC APPLICATION TO ALL CLASSES OF SHUTTLE LEAK DETECTION

NATIONAL LAUNCH SYSTEM

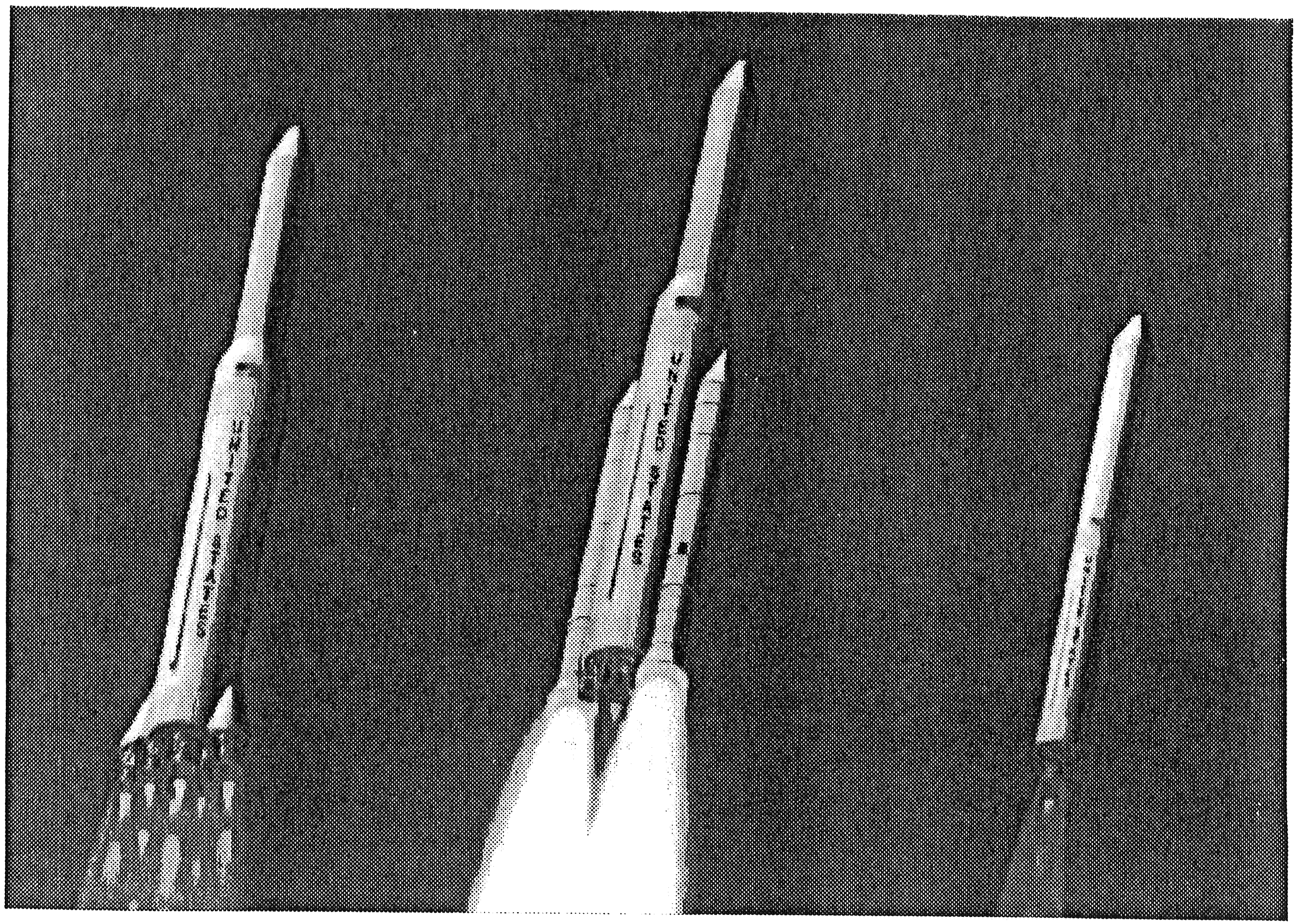
HYDROGEN LEAK DETECTION TECHNOLOGY
TECHNICAL INTERFACE MEETING
KENNEDY SPACE CENTER
APRIL 29-30, 1992

HAZARDOUS GAS DETECTION SYSTEM DESIGN REQUIREMENTS
FOR
NATIONAL LAUNCH SYSTEM (NLS)

PRINCIPAL INVESTIGATOR
JACQUELINE GUERRERO
(310)922-4285

Rockwell International
Space Systems Division

NLS Family of Vehicles



NLS-2
(50K P/L)

NLS-1
(HLLV)

NLS-3
(20K P/L)

NLS Requirements Are Derived From Administration, Congressional, Advisory Committee Guidance

DEVELOP A LAUNCH SYSTEM

- PROVIDE A RANGE OF PAYLOAD CAPABILITIES
- IS SIGNIFICANTLY MORE OPERABLE THAN EXISTING VEHICLES (MEASURED BY RELIABILITY, LAUNCH ON SCHEDULE, PAYLOAD EXCHANGE CAPABILITY, SURGE, FAULT TOLERANCE)
- → PROVIDE A <u>SIGNIFICANT REDUCTION IN PER-FLIGHT COSTS</u>
- → CAN BE DEVELOPED AT <u>AFFORDABLE COST AND SCHEDULE</u>
- → IS EVOLUTIONARY – <u>CAN/WILL ACCEPT NEW TECHNOLOGIES</u>
- IS MAN-RATEABLE
- SUPPORT THE COMMERCIAL LAUNCH VEHICLE INDUSTRY BY PROVIDING TECHNOLOGY, SYSTEMS, VEHICLES, AND/OR INFRASTRUCTURE

Valuable "Lessons Learned" from Space Shuttle Program Must be Considered for NLS Program

NATIONAL LAUNCH SYSTEM

- SIGNIFICANT EXPERIENCE GAINED IN THE AREA OF HYDROGEN LEAK DETECTION PROVIDES INSIGHT AS TO WHAT CONDITIONS & REQUIREMENTS MUST BE ADDRESSED
  - IMPORTANCE OF QUANTIFICATION OF INDIVIDUAL LEAK SOURCES
  - POTENTIAL FOR "SMART CRYO" LEAK(S) WHICH ONLY APPEAR AT CRYOGENIC CONDITIONS
  - POTENTIAL FOR MULTIPLE LEAK SOURCES

Rockwell In-House Study is Defining NLS Hazardous Gas Detection System Design Options

NATIONAL LAUNCH SYSTEM

- **WILL SEEK OUT NEW TECHNOLOGY TO ADDRESS TOP LEVEL REQUIREMENTS**
  - EXPENDABLE
  - MAN-RATEABLE

- **WILL USE KEY SELECTION CRITERIA TO DETERMINE SUITABLE APPLICATION OF TECHNOLOGY TO SUPPORT NLS FIRST FLIGHT IN YEAR 2002**
  - GAS DISCRIMINATION
  - SYSTEM COMPLEXITY
  - OPERATING ENVIRONMENT
  - VIBRATION(MISSION SUITABLE COMPONENTS)
  - SYSTEM ACCURACY & SENSITIVITY
  - SYSTEM WEIGHT & SIZE
  - COSTS (RECURRING & NONRECURRING)
  - TECHNOLOGY MATURITY
  - SAFETY
  - MAINTAINABILITY/RELIABILITY
  - EASE OF INSTALLATION

Rockwell Study Objective - Design a System Which Best Fulfills Operational Requirements

NATIONAL LAUNCH SYSTEM

1.) REMOTE DETECTION AT THE SOURCE (LOCATION & QUANTIFICATION)

2.) SIMULTANEOUS REMOTE SENSING OF <u>HYDROGEN</u>, OXYGEN, HELIUM , ARGON

3.) REAL-TIME MONITORING ON THE GROUND AND IN FLIGHT

4.) OPERATIONAL IN CRYOGENIC ENVIRONMENT

5.) NON-INTRUSIVE, NON-DESTRUCTIVE

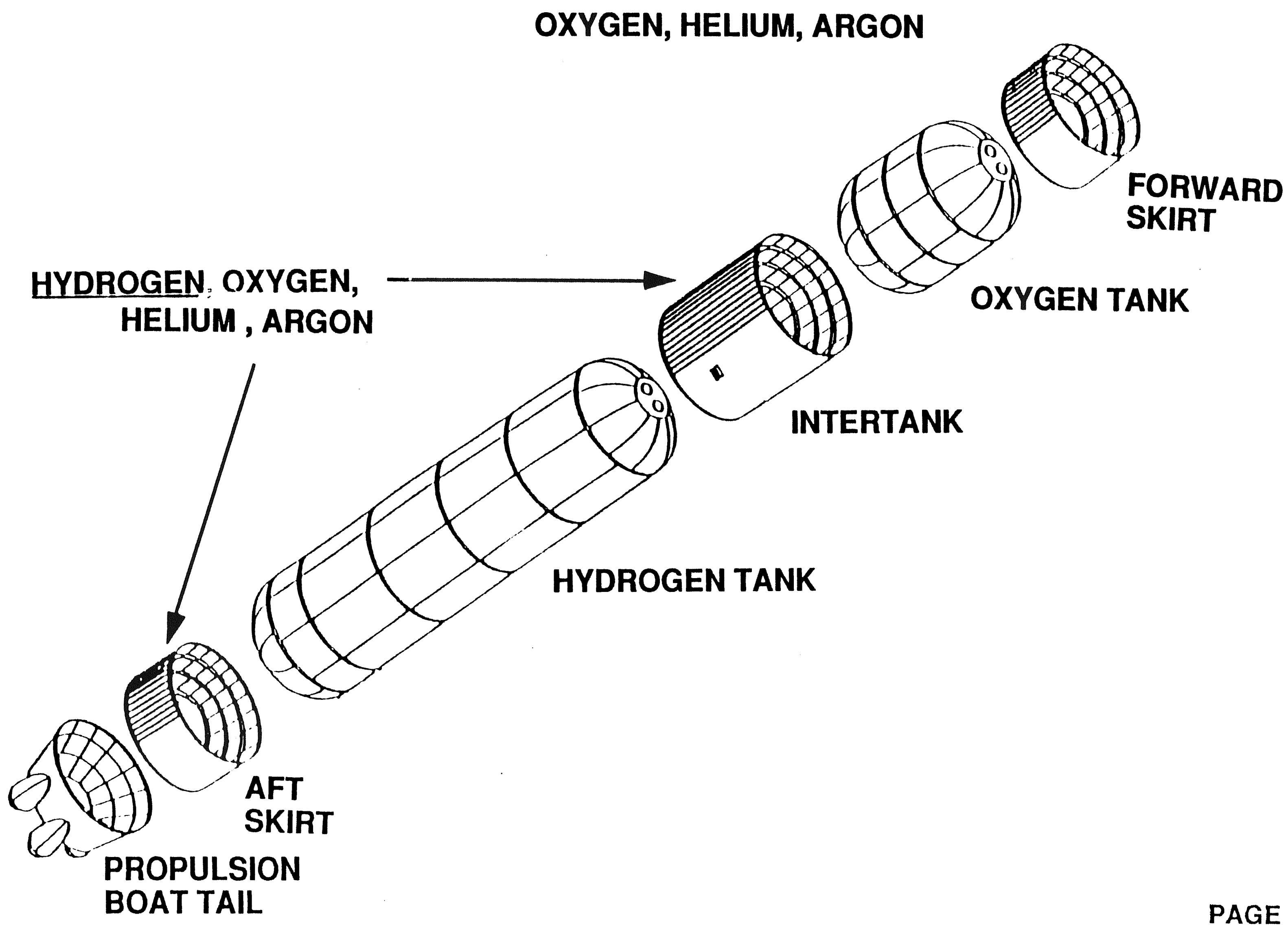
NLS Requirements are Similar to Space Shuttle's

	SHUTTLE	NLS
VEHICLE DEFINITION		
Expendable	☐	☒
Man-Rateable	☒	☒
VEHICLE CONFIGURATION		
Cryogenic LO2 Propellant	☒	☒
Cryogenic LH2 Propellant	☒	☒
Helium Pressurization	☒	☒
Nitrogen Purged Compartments	☒	☒
Enclosed Boat Tail	☒	☒
Open Boat Tail	☐	☒
HAZARDOUS GAS DETECTION SYSTEM OPERATION		
Monitoring on Ground	☒	☒
Monitoring during Ascent	☒	☒
GAS SPECIES DETECTION		
Oxygen	☒	☒
Hydrogen	☒	☒
Helium	☒	☒ *
Argon	☒	☒ *

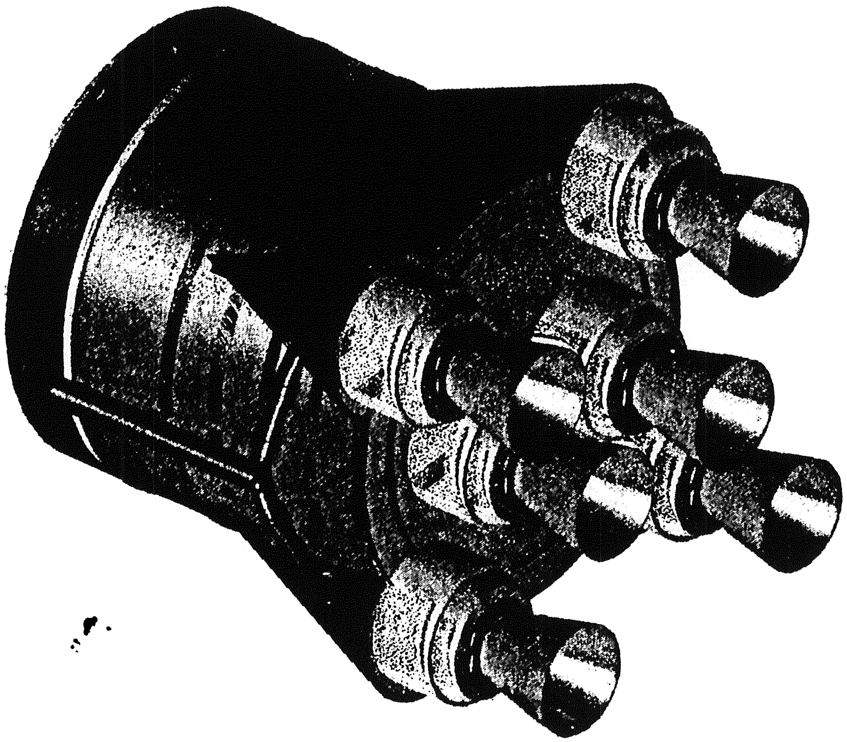
NO DECISION ON BOAT TAIL CONFIG. YET REACHED (Enclosed Boat Tail / Open Boat Tail, NLS)

****NOT REQUIRED DURING ASCENT***

Hydrogen Leak Detection Required in 2 of 3 NLS Compartments

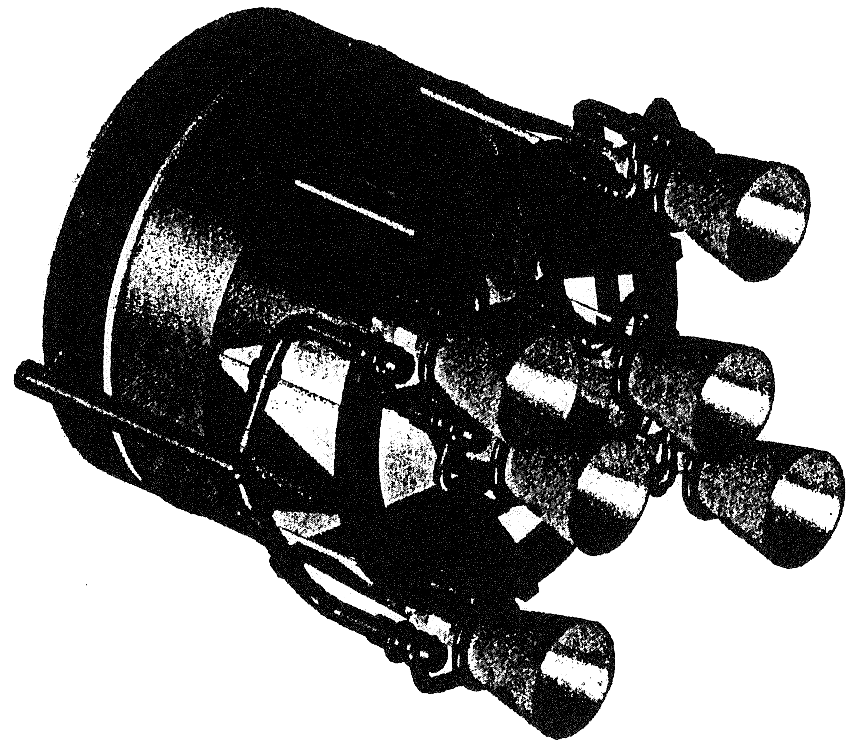


Enclosed vs. Open Boat Tail May be Greatest Challenge in HGDS Definition



ENCLOSED

VS.



OPEN

ENCLOSED

- FORCES GREATER RELIABILITY & ACCURACY REQUIREMENTS ON A HGDS AND PURGE SYSTEM TO ASSURE SAFETY PRIOR TO AND DURING FLIGHT
- CREATES A CONTROL VOLUME WHICH CAN POTENTIALLY EASE INTERPRETATION OF DATA
- LEAK DETECTION FOCUSES ON BOTH VEHICLE HEALTH MONITORING & FLAMMABILITY CONCERNS

OPEN

- MINIMIZES CONCERN OF DETONATION/COMBUSTION OF HAZARDOUS GAS BUILDUP
- DATA SUSCEPTIBLE TO EXTERNAL FACTORS (WIND, HUMIDITY, TEMPERATURE)
- LEAK DETECTION FOCUSES ON VEHICLE HEALTH MONITORING

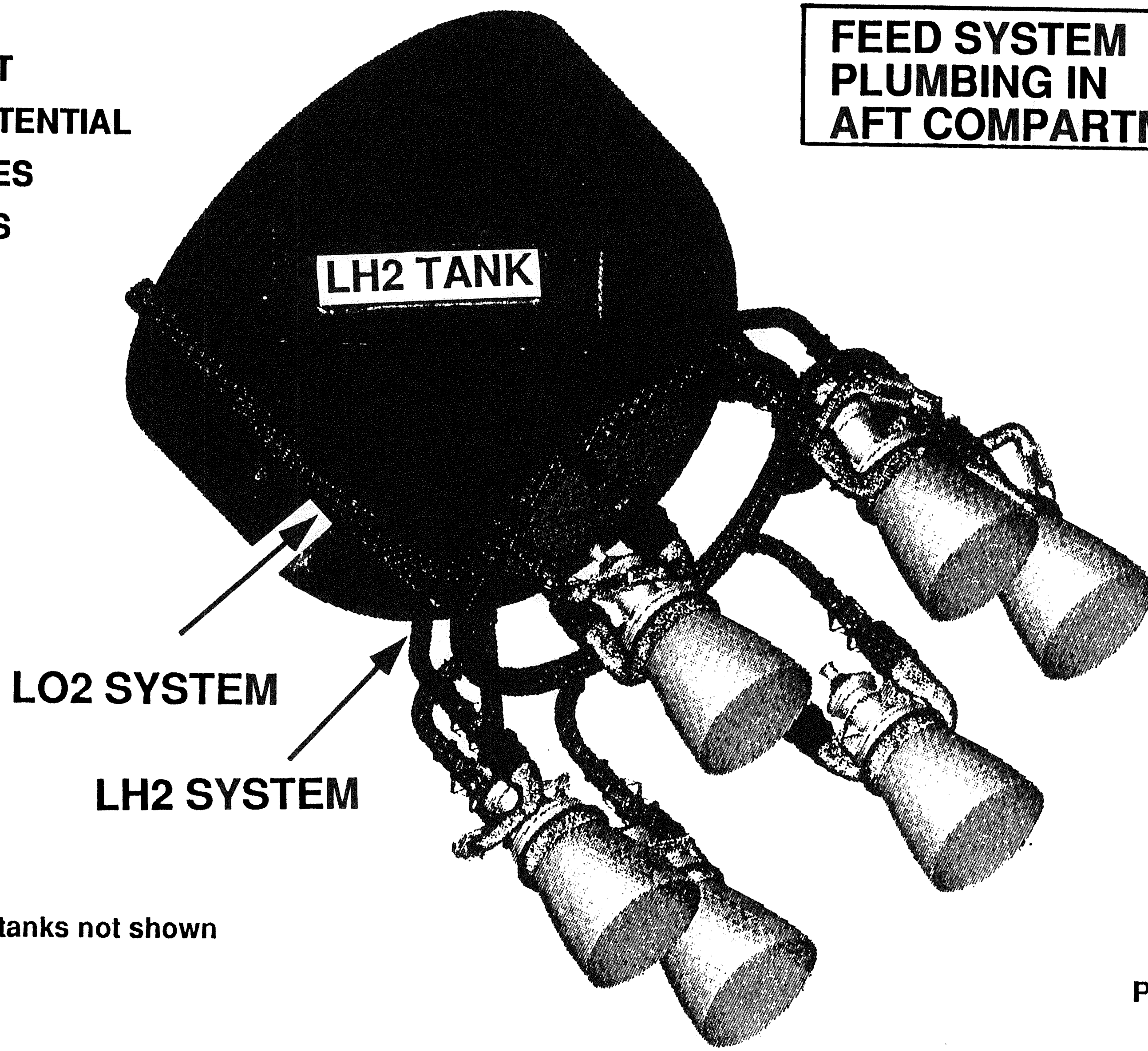
Propulsion Feed System Complexity May Reduce Specific Technologies Applicability

NATIONAL LAUNCH SYSTEM

ISSUES

- LINE OF SIGHT
- MULTIPLE POTENTIAL LEAK SOURCES
- AREA ACCESS
- LARGE AREA

FEED SYSTEM PLUMBING IN AFT COMPARTMENT



Note:helium tanks not shown

Demonstration Testing is Next Step Towards Final Selection

NATIONAL LAUNCH SYSTEM

- PRELIMINARY CONCEPT STUDY PHASE TO BE COMPLETED JUNE 1992
  - TECHNOLOGY/SYSTEM OPTIONS FOR NLS WILL BE DEFINED

- ROCKWELL INVESTIGATING POTENTIAL IN-HOUSE DEMONSTRATION TESTING OF SELECTED TECHNOLOGY AS NEXT PHASE OF STUDY TO EVALUATE TECHNOLOGY CAPABILITY
  - LOOKING TO "LEAK DETECTION INDUSTRY" TO PROVIDE INFORMATION AND TECHNOLOGY
  - IF PROOF-OF-CONCEPT SUCCESSFUL, FURTHER TESTING WILL BE NEEDED TO:
    - IDENTIFY MISSION-SUITABLE COMPONENTS
    - OPTIMIZE SYSTEM (SENSITIVITY, WEIGHT, SPEED ,ETC.)
    - DEFINE & DEVELOP AUTOMATED INSPECTION CAPABILITY
    - INTEGRATE TECHNOLOGY INTO A ROBUST SYSTEM PACKAGE

ROCKWELL IS WORKING TOWARDS ACHIEVING NLS PROGRAM GOALS BY UTILIZING ADVANCED LEAK DETECTION TECHNOLOGY

HYDROGEN DETECTION at AEDC

A Presentation of low Pressure Time Response Tests of Cayalytic Sensors

Benjamin W. Hartsfield

April 29, 1992

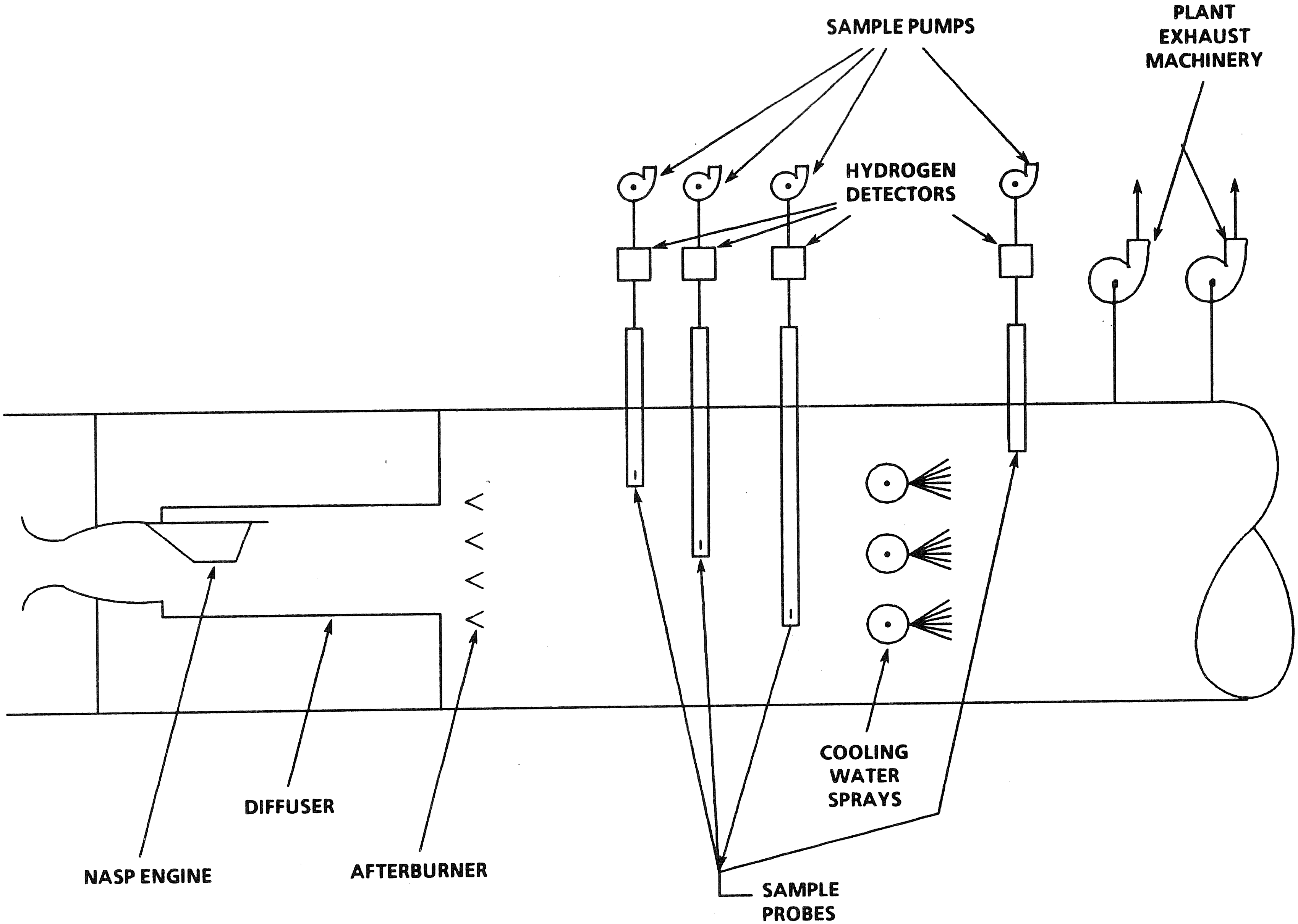


FIGURE 1. C-1 HYDROGEN SENSOR INSTALLATION

BWH 2-19-91

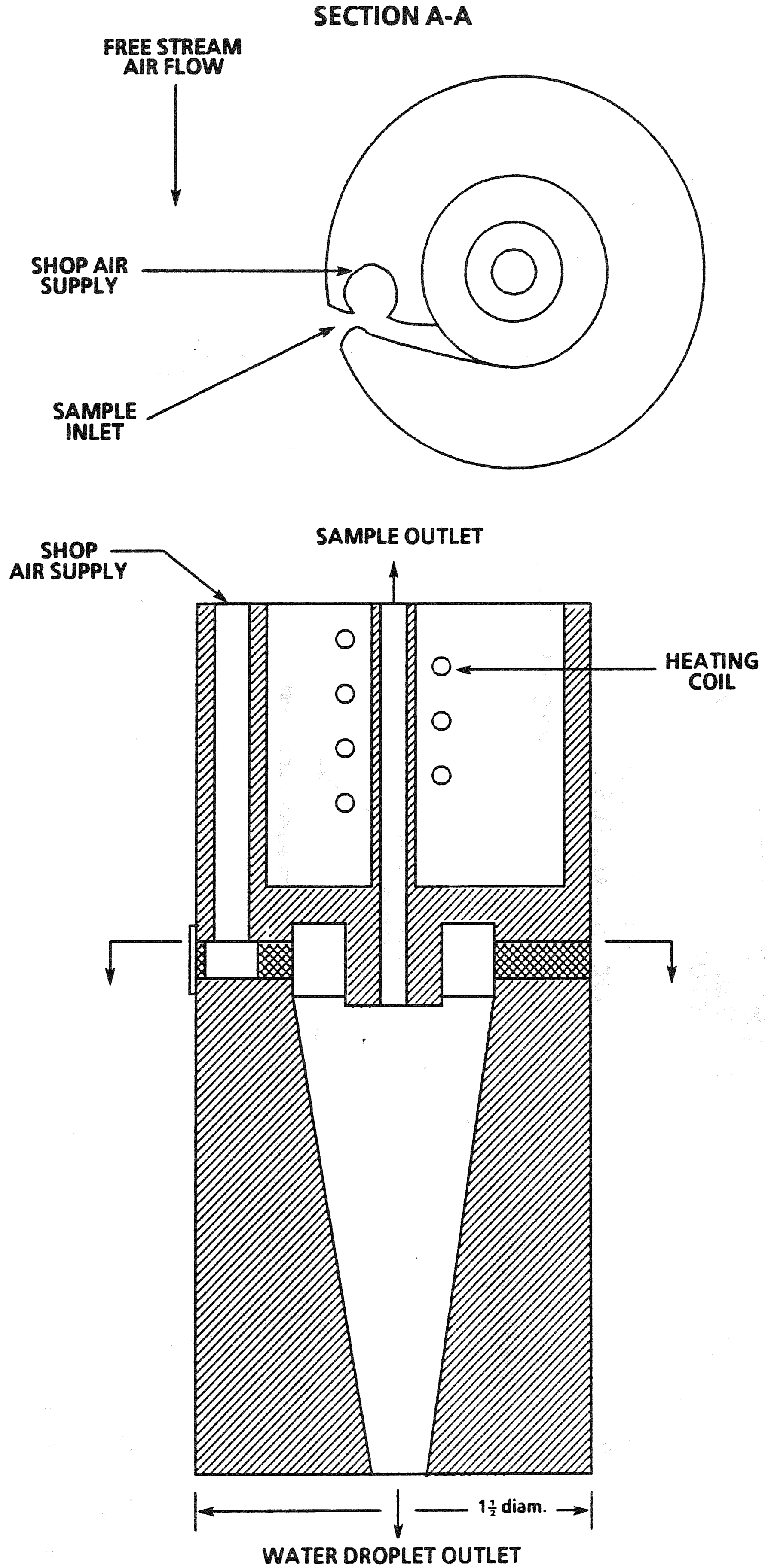


FIGURE 2. **WATER DROPLET SEPARATING SAMPLE PROBE**

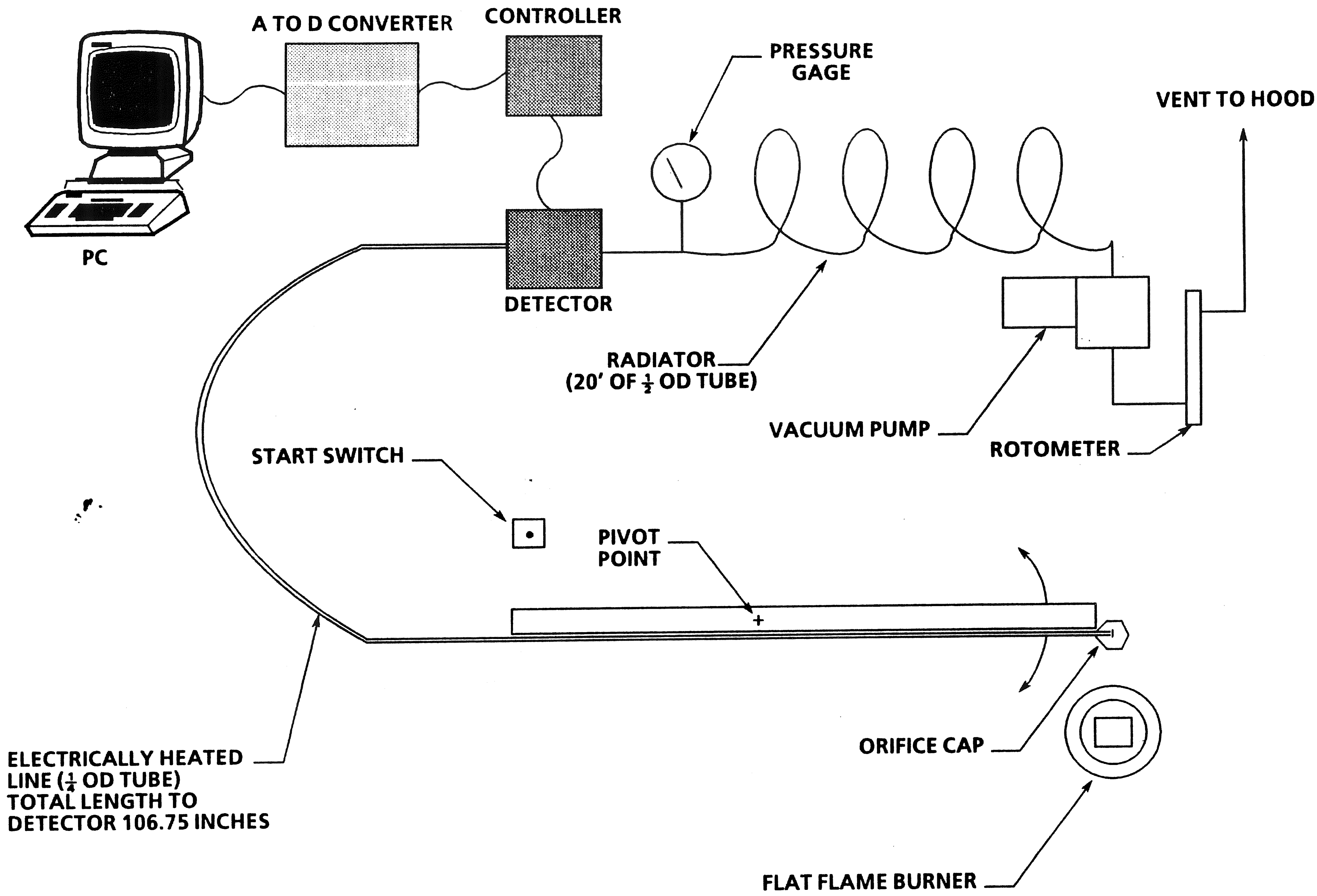


FIGURE 3. HYDROGEN DETECTOR TEST SETUP

MANUFACTURER /MODEL NO.	DETECTOR TYPE	MODIFICATIONS	PRESS PSIA	TEMP °F	% LOW EXPLOSIVE LIMIT	RISE TIME, sec.	DECAY TIME, sec.
Detector Electronics Corp./U8700	Catalytic	Bread Board Controller	11.2	75.	100.	2.1	2.6
General Monitor/SC100	Catalytic	As Received	14.3	75.	75.	5.1	7.6
M.S.A./Part #487811	Catalytic	As Received	10.9	78.	40.	3.3	3.8
M.S.A./Part #487811 *	Catalytic	Flame Arrestor Removed – Respan	11.4	77.	73.	3.1	4.4
M.S.A. Part #487811	Catalytic	Flame Arrestor Removed – Respan	11.0	250.	104.	2.7	3.8
M.S.A. Part #487811	Catalytic	Flame Arrestor Removed – Respan	10.9	300.	102.	2.4	3.6
M.S.A. Part #487811	Catalytic	Bread Board Controller with Flame Arrestor	11.5	77.	104.	3.3	3.9
Matheson Model 3100	Solid State	As Received	14.3	75.	103.	1.5	7.7

FIGURE 4. SENSOR COMPARISONS

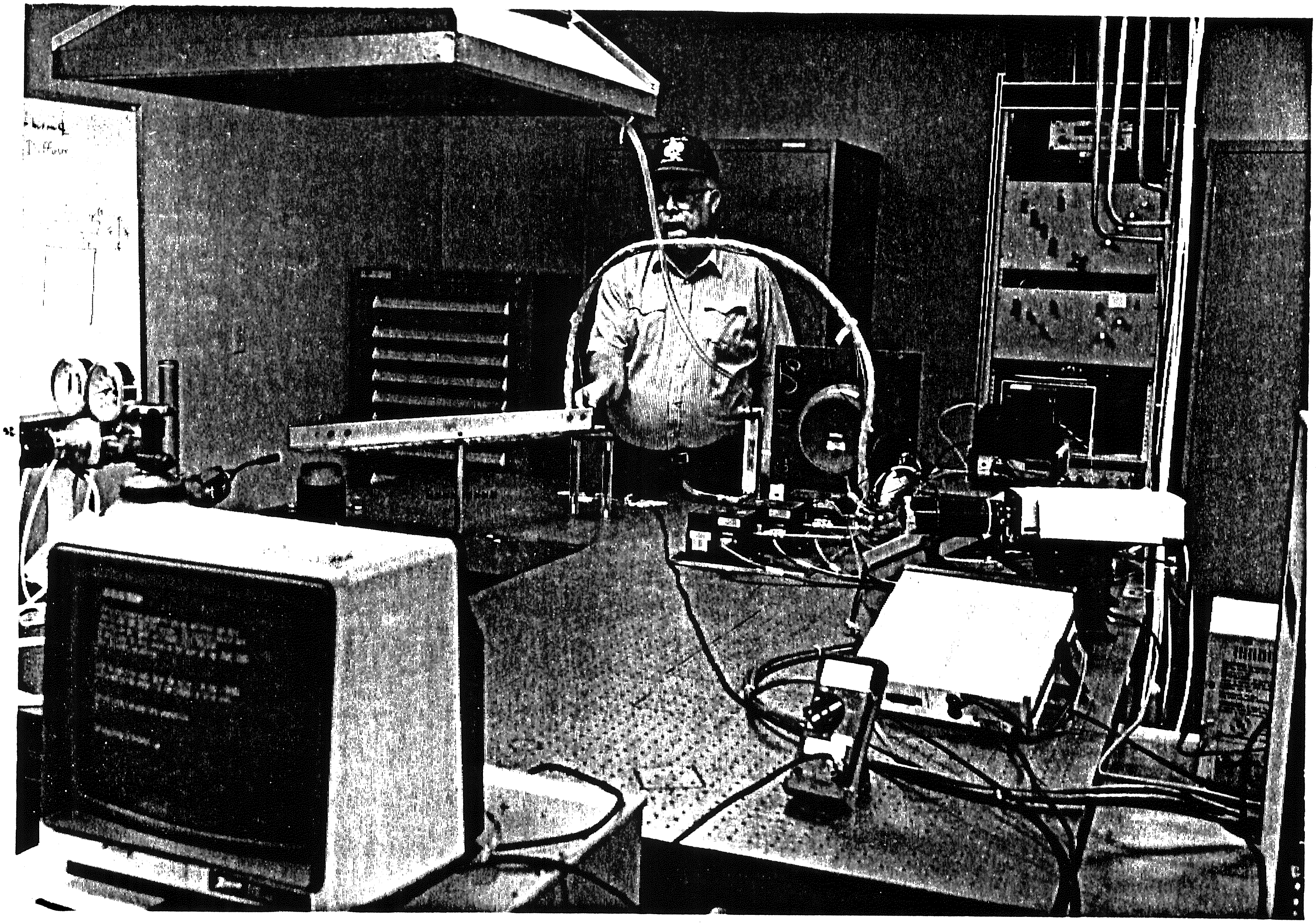
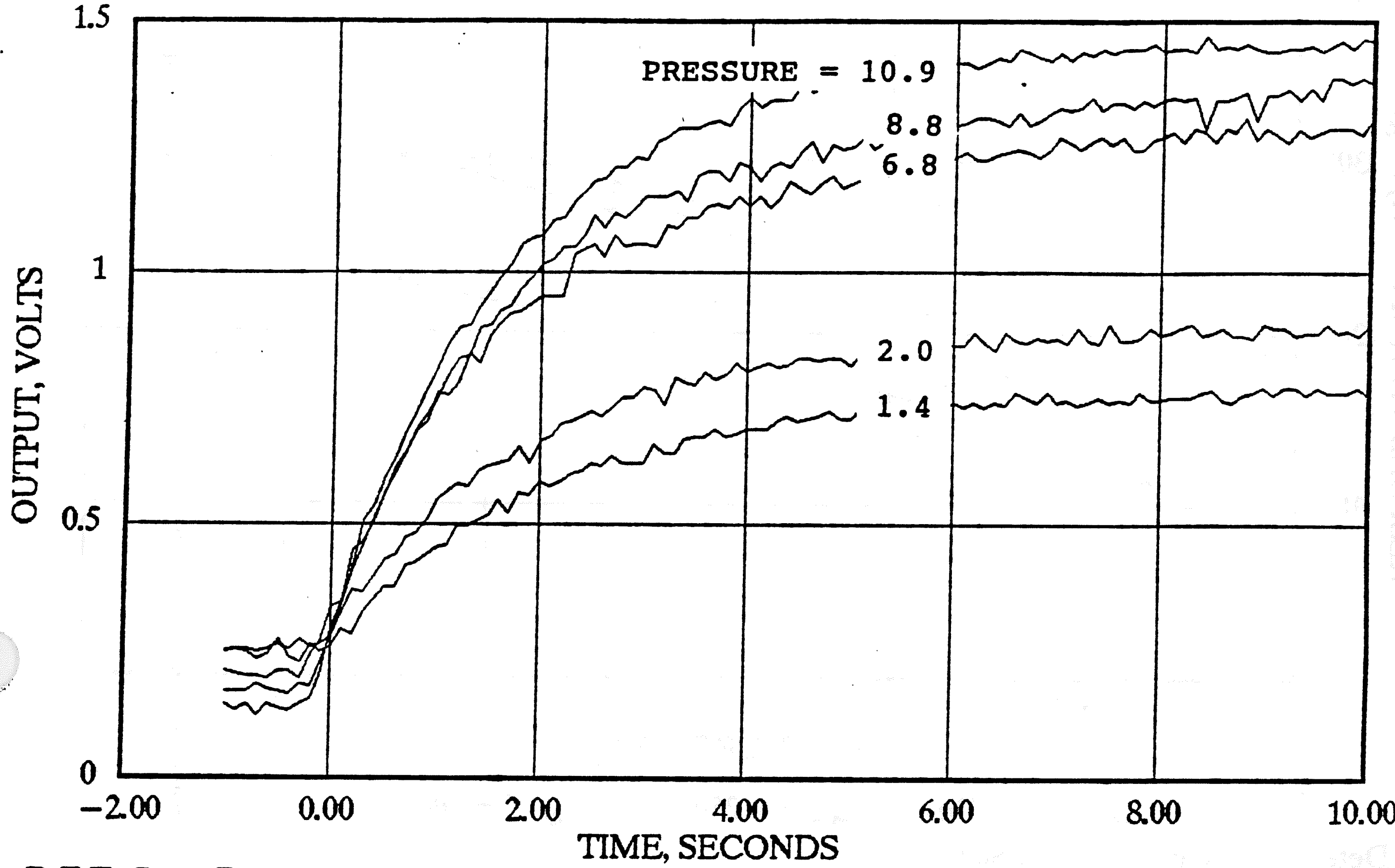


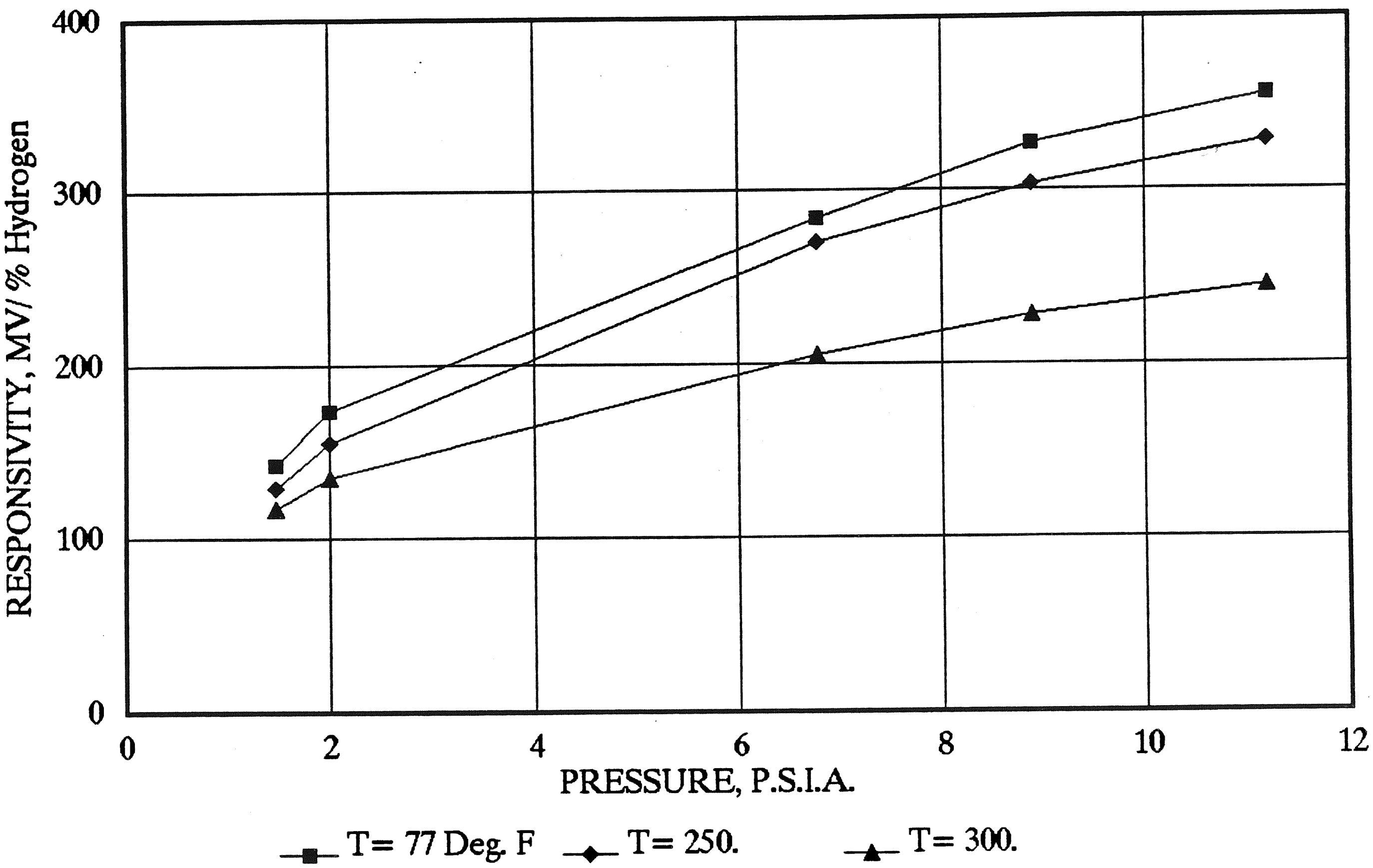
FIGURE 5. HYDROGEN SENSOR TEST SETUP

Figure 6 Catalytic Sensor Characteristics

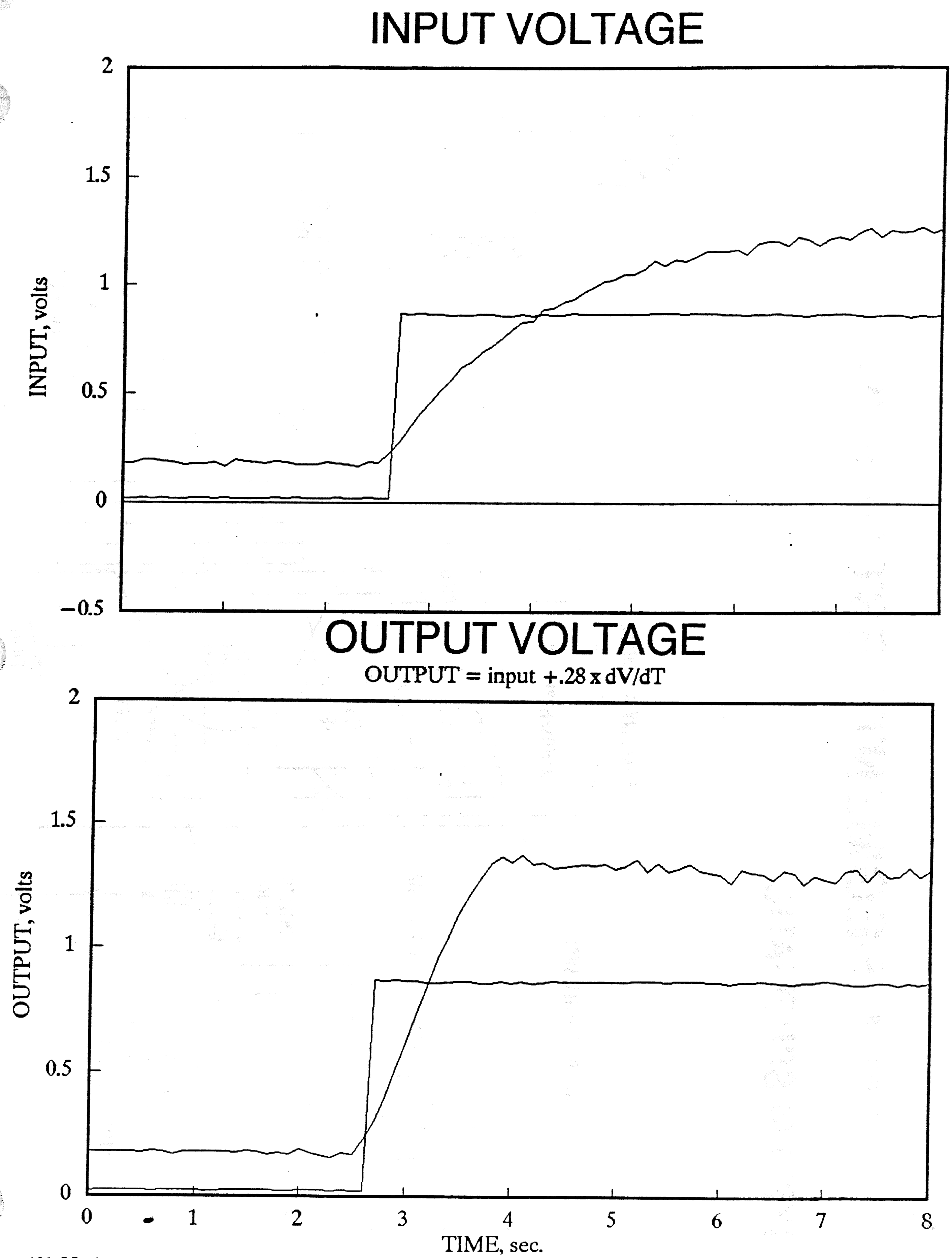


D.E.T. Corp. Detector
4.1 % Hydrogen by Volume

FIGURE 7. RESPONSIVITY



Detector Electronics Corp. Sensor
4.1 % Hydrogen by Volume

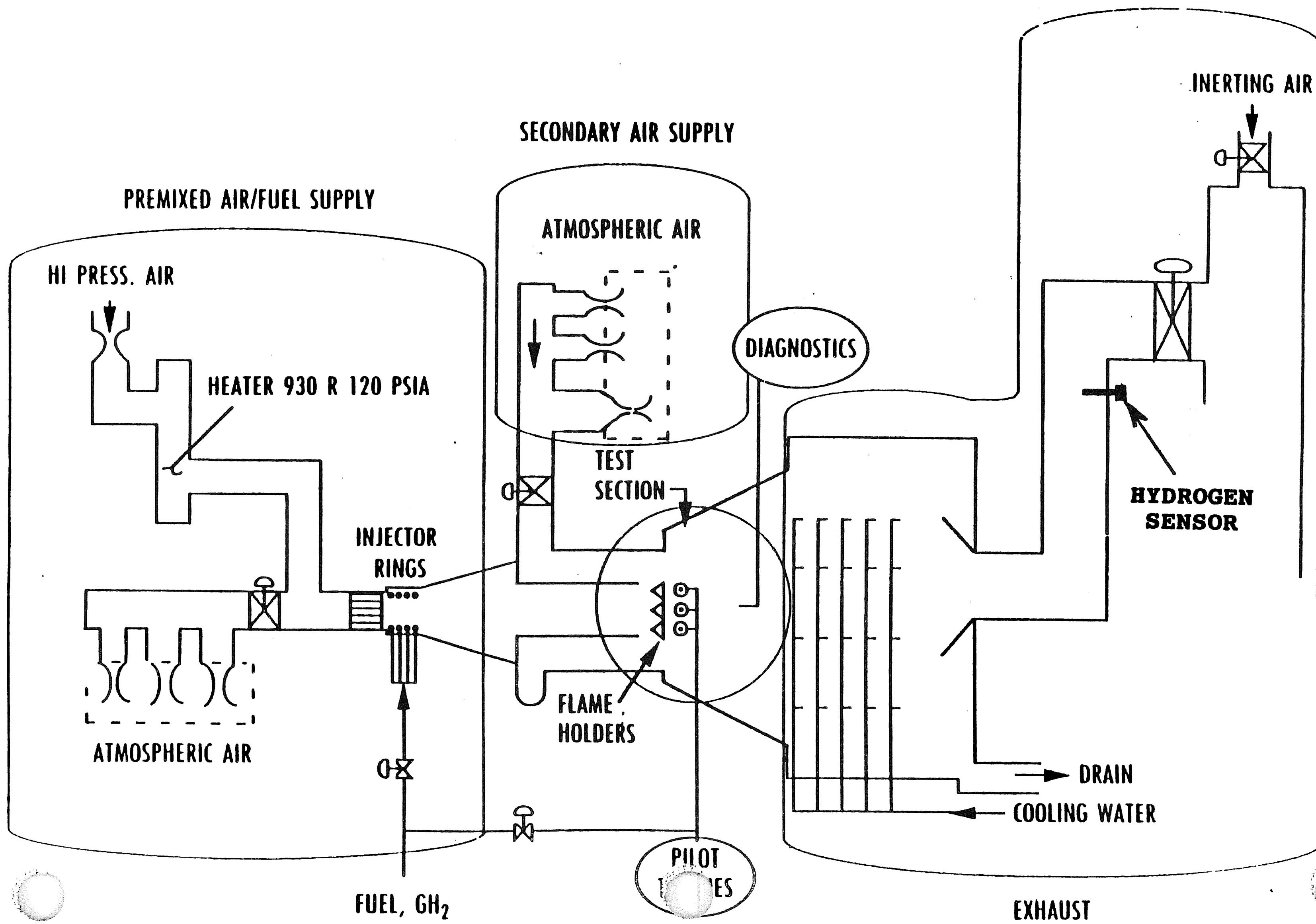


4% Hydrogen
OUTPUT, volts

FIGURE 8. EFFECT OF PREDICTIVE ALGORITHM

FIGURE 9. **SEGMENT TEST PLANS**

• SYSTEM SCHEMATIC



Leak Detection for Launch Vehicle Applications

NAS 8-39029

Hydrogen Leak Detection Technology
Technical Interchange Meeting

Kennedy Space Center

April 29-30,1992

Leak Detection for Launch Vehicle Applications

NAS 8-39029

Sponsor:	NASA Marshall Space Flight Center
NASA Monitor:	W.T. Powers, EB 22
Program Manager:	Darby B. Makel
Project Engineer:	Roy H. Sakabu
Participating Organizations:	Micromonitors NASA LeRC Case Western Reserve University

Remote Detection & Imaging of Hydrogen Concentration by Raman Lidar

Dr. I. Stuart McDermid

Robert Martin

JPL

April 30, 1992

PURPOSE

- Present a concept for an imaging Raman Lidar system for remote detection of hydrogen leakage
- Show the system parameters available for application optimization
- Indicate feasibility

GENERAL REQUIREMENTS

- Detect the location of hydrogen leaks in a target volume
- Quantify the potential explosive hazard by determining the volume of H_2 with concentration over 4%
- Measurement uncertainty, confidence level, operations period, and response time as specified

APPROACH

- Laser radiation source
  - Ultraviolet, pulsed laser
  - Homogenized, expanded beam
- Detect Raman scattering by hydrogen
  - Filter blocking of reflected and Rayleigh scattered radiation
- Multi-angle video imaging
  - CCD or photodiode array
  - Detector photon integration and page RAM storage
  - Surveillance and UV camera images summed for display
  - Computer image processing
  - VCR recording

ADVANTAGES

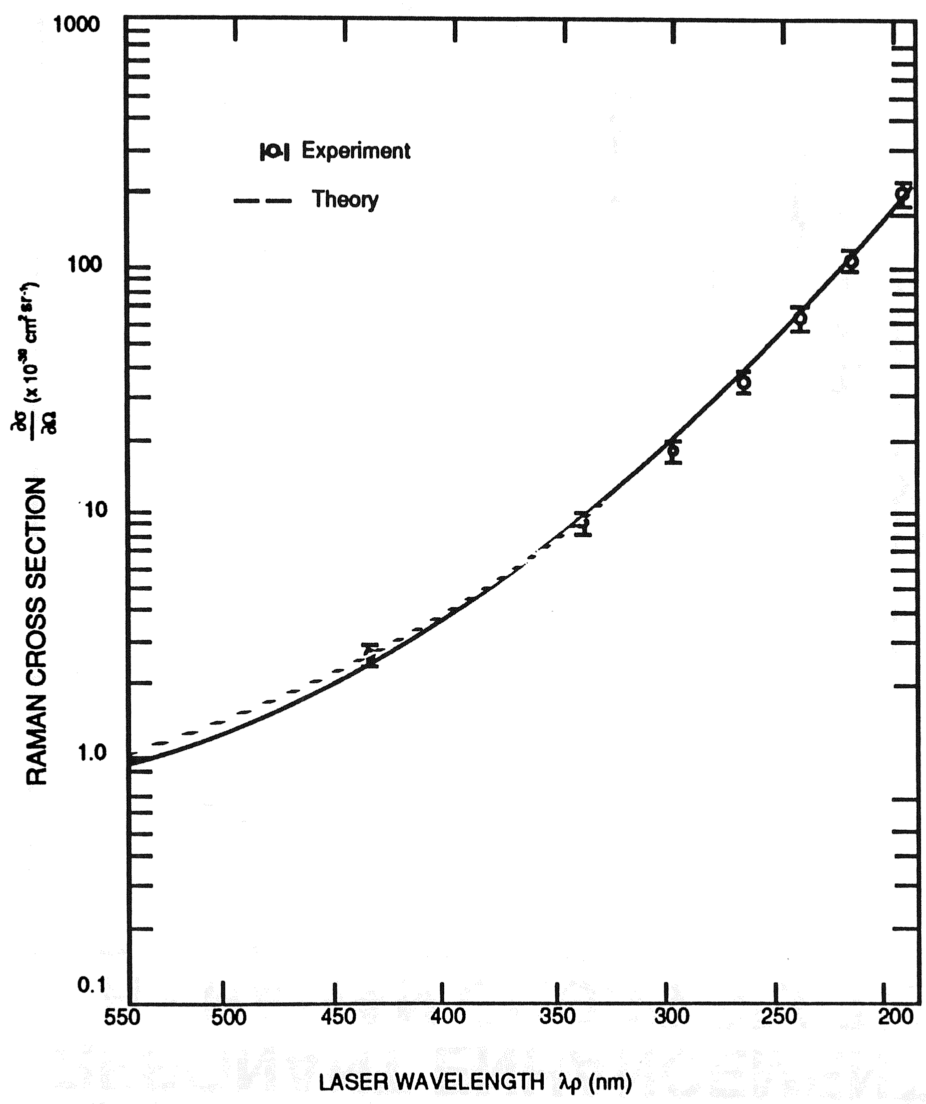
- Lidar is mature and well tested
- Species specific and sensitive
- Solar blind
- Positional data from multi-angle imaging rather than pointing and ranging
- Noninterference with Working Operation
  - Eye safe
  - No ignition hazard
- Large Area (Volume) Coverage
  - Divergent beam projection
  - Total coverage each pulse
- Rapid Response, continuous operation

LIDAR EQUATION

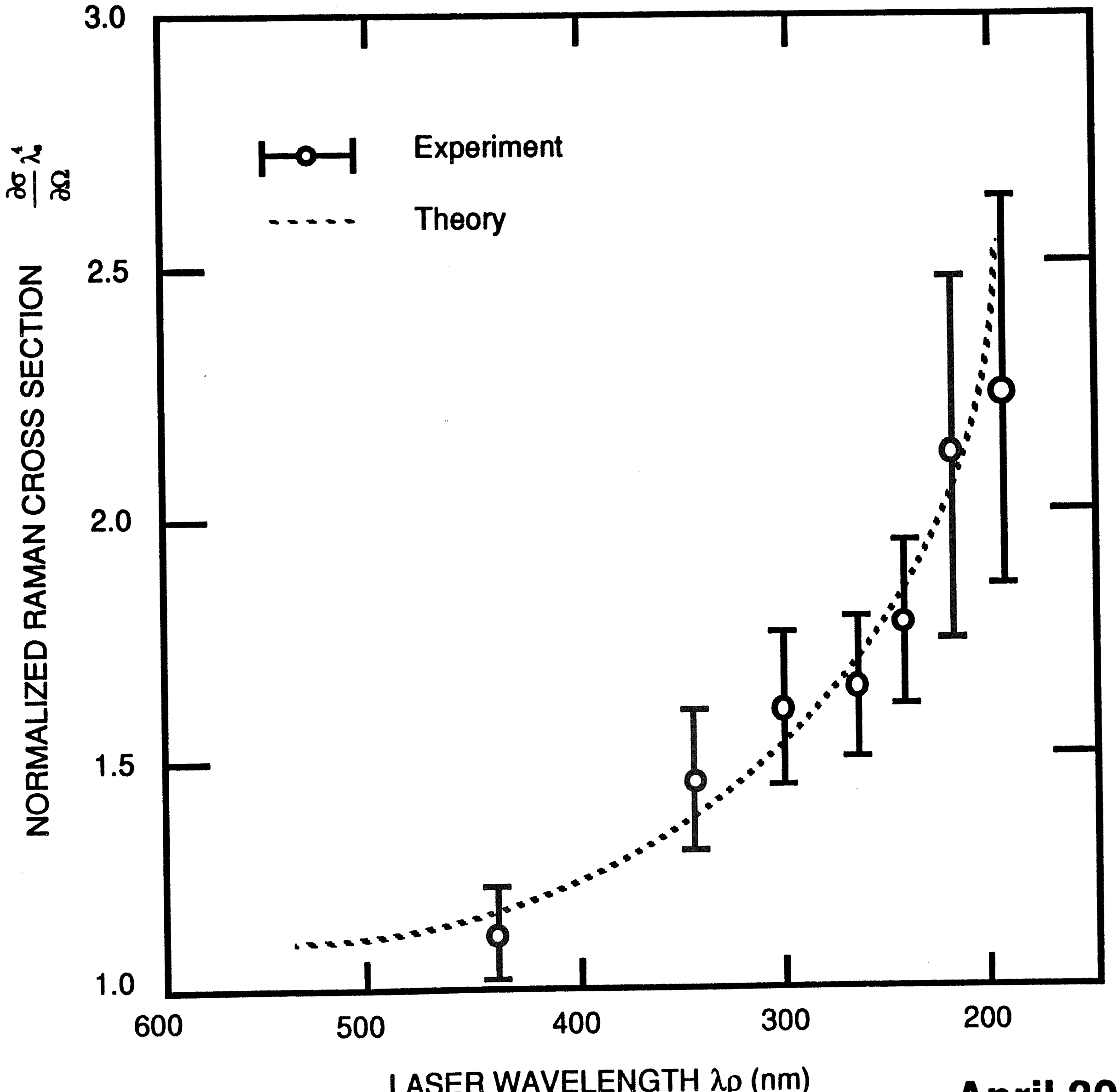
$$\mathbf{E}(\lambda,\mathbf{R}) = \mathbf{E}\ \xi(\lambda)\ \mathbf{T}(\mathbf{R})^2\ \xi(\mathbf{R})\ \beta(\lambda,\mathbf{R})\ \Delta\mathbf{R}\ \frac{\mathbf{A_o}}{\mathbf{R}^2}$$

Modified by the Raman shifted wavelength, and the backscatter coefficient replaced by the Raman scattering cross-section

ABSOLUTE VIBRATIONAL RAMAN SCATTERING CROSS SECTION FOR H2



RESONANT ENHANCEMENT OF H2 RAMAN CROSS SECTION



ATMOSPHERIC TRANSMISSION

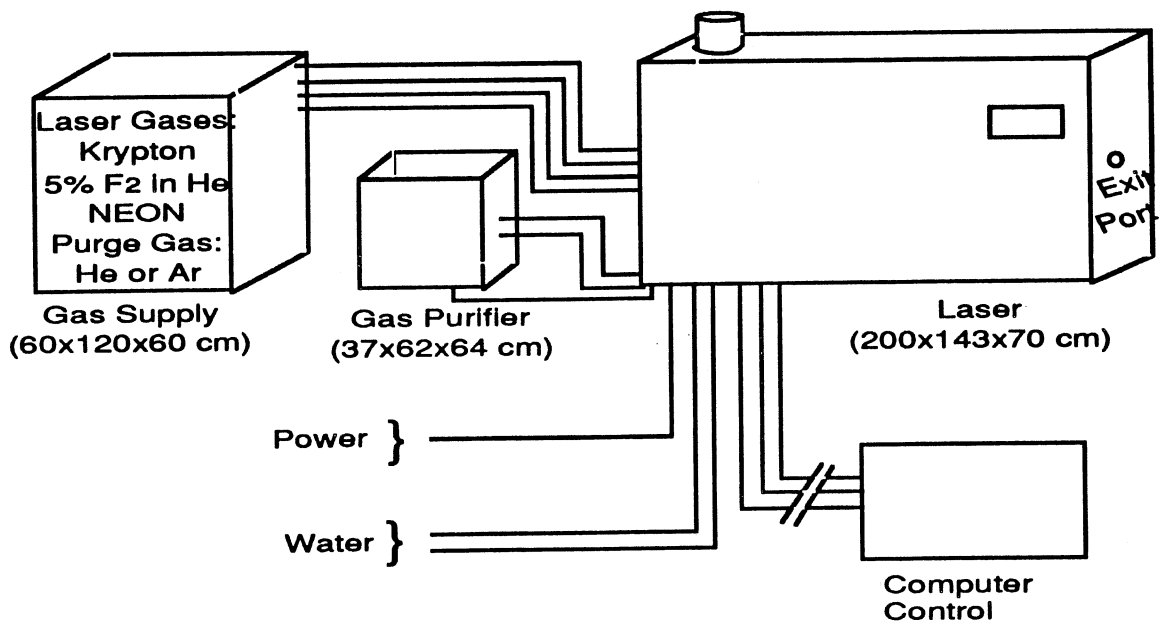
$$\alpha(\lambda) = \alpha_R(\lambda) + \alpha_A(\lambda) + \alpha_{O3}(\lambda)$$

λ (nm)	α (cm-1)	T(%) 15m	T(%) 25m	T(%) 50m	T(%) 100m
248.0	2.25x10-5	96.7	94.5	89.4	79.9
276.5	1.36x10-5	98.0	96.7	93.4	87.3
308.0	5.59x10-6	99.2	98.2	97.2	94.6
353.2	4.03x10-6	99.4	99.0	98.0	96.1

LASER SYSTEM

(Example)

- 100 W stabilized power at 400mJ and 250 Hz
- >1000 hours preventative maintenance interval
- Guaranteed service free operation for > 10^9 laser pulses



LASER ILLUMINATION OF TARGET VOLUME

- Homogenizing rod for uniformity
  - *Di Benedettoet all*, 1991
- Beam expansion to illuminate target volume
  - Energy density 10^3 below eye-safe exposure level

TARGET RADIANCE

$$E_i = E_L \, T_L \, (A_i / A_T)$$

$$E_{Ph} = [E_i \ \lambda] \, / \, 1.987 \times 10^{-16}$$

$$\beta_i = \sigma_M \, [M] \, V$$

(assume volume element contains 100% H2 at atmospheric pressure)

$$J_i = \beta_i \times E_{Ph}$$

RECEIVER & IMAGING SYSTEM

- Collection optics: 50 mm to 150 mm aperture
- Spectral filters: Fabry-Perot notch filters with FWHM of 2 nm and transmission of 0.01% 10 to 15 nm off center wavelength, 0.0001% available. Peak transmission 30-40%
- Filter wheel for H_2, N_2 and neutral density
- Transmission efficiency ~25%
- Cooled photodiode detector array or CCD depending on desired dynamic range and integration time. 100x100 to 256x256 element arrays
- Electron read noise ~4000 photons (diode array)
- Image page RAM storage with computer control and processing and VCR recording

SENSITIVITY CALCULATION

$$S_i = J_i \, T_R \, (A_o / R^2) \, \xi(F) \, I$$

The number of photons received in the integration time by a pixel in the imaging detector from the test volume element.

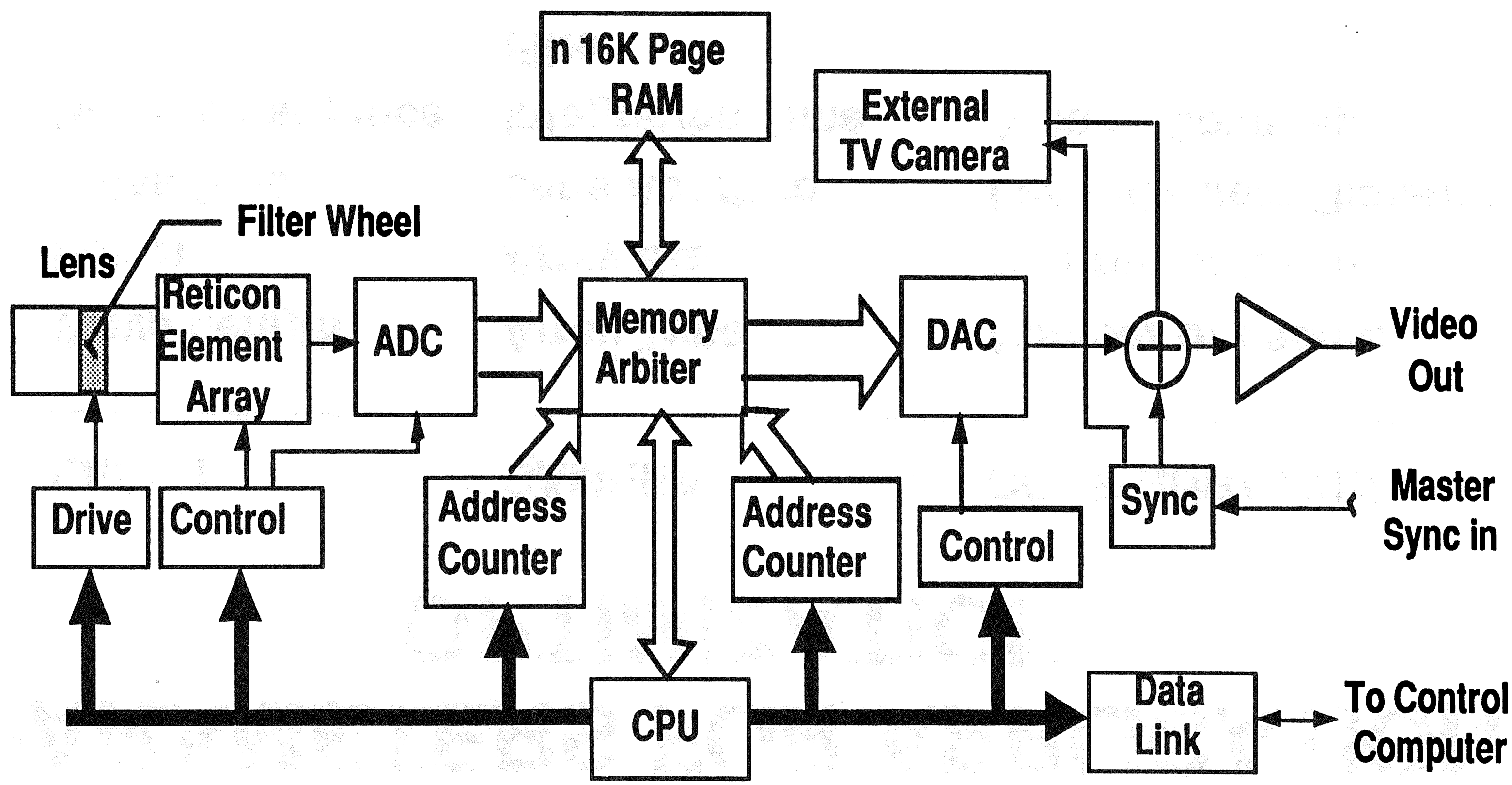
LEAK RATE CONSIDERATION

- Raman lidar technique provides hydrogen concentration
- Leak rate derived from sequences of measurements and diffusion/transport model
- 1 scim $\equiv 7.0 \times 10^{18}$ molecules per second
- 2000 scim is sufficient to totally fill 550 cm^3 each second (4.4 5x5x5 cm element volumes)

CALIBRATION

- Periodic lab calibrations of camera elements: >6 months
- Preventive maintenance on external optical surfaces prior to major events
- Pre and post test calibrations of system using N2 notch filter
- Fixed concentration of H_2 in a quartz flask observed in real time as desired

CAMERA BLOCK DIAGRAM

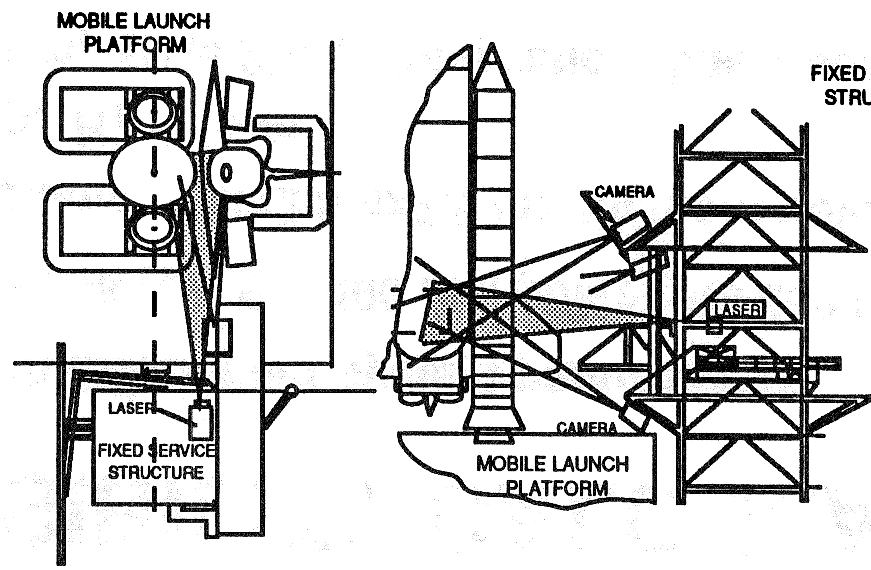


PARAMETERS FOR APPLICATION OPTIMIZATION

LASER	CAMERA	CONFIGURATION
Wave Length	Array Type	Number of Lasers
Power	Array Size	Number of Cameras
Pulse Rate	Lens Aperture	Laser/Camera Distance
Beam Divergence	Integration Time	Area of Coverage
	Filter	

SHUTTLE 17-INCH DISCONNECT APPLICATION

Laser System Area Illumination, projected divergent beam



FEASIBILITY CALCULATIONS

- Shuttle 17-inch disconnect system parameters:
  - Laser: λ = 248 nm, E = 400 mJ, pulse rate 250 Hz
  - Target volume: 5mx2.5mx2.5 m; Element volume 5x5x5 cm
  - R_L = 25 m, R_C = 15 m
  - A_o = 50 mm, $\xi(\lambda)$= 25%, Integration time 1 second, 100x50 array
- Calculated values
  - T_L = 94.5%, T_R = 98.0%, J_i = 1.45×10^7 photons per pulses per sr, S_i (248) = 4000 photons per second per second per pixel
- Signal-to-noise ratio of approximately unity at 100% H_2 concentration of a single element volume
- Parameter options available:
  - A_o x 9
  - Integration time x 10
  - Target volume x 2
  - Intensifier x 10^6

RAMAN LIDAR MEASUREMENTS

SAMUEL H. MELFI
NASA GODDARD SPACE FLIGHT CENTER

Remote Laser-based Detection of H_2 by Laser-excited Amplified Spontaneous Emission

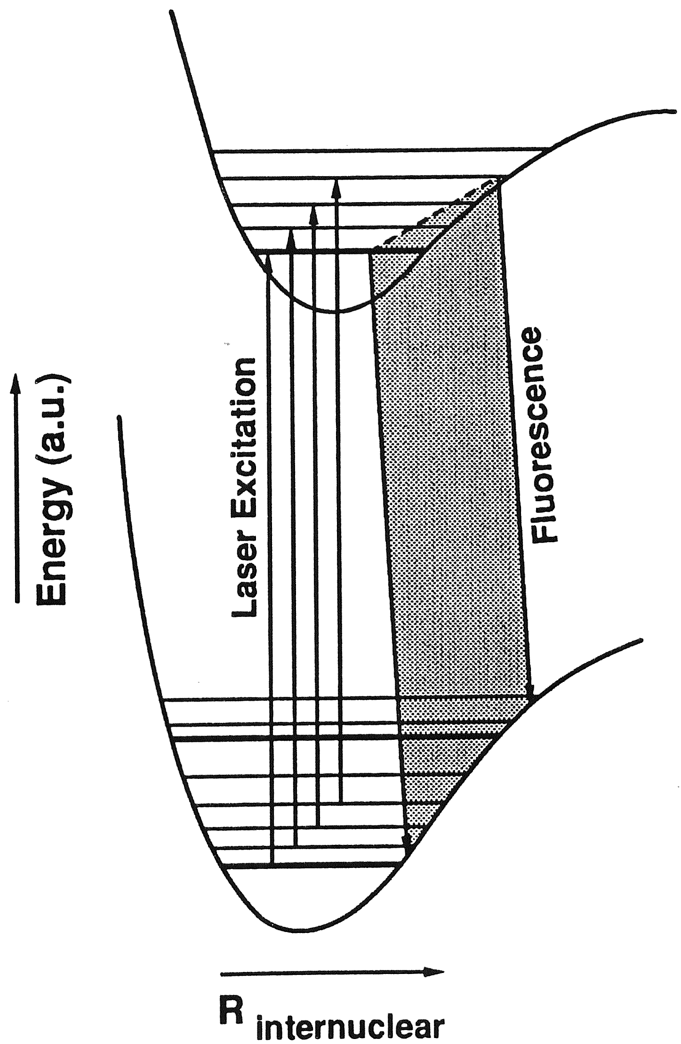
Andy Sappey
CLS-2
Los Alamos National Lab

NASA KSC presentation
April 30, 1992

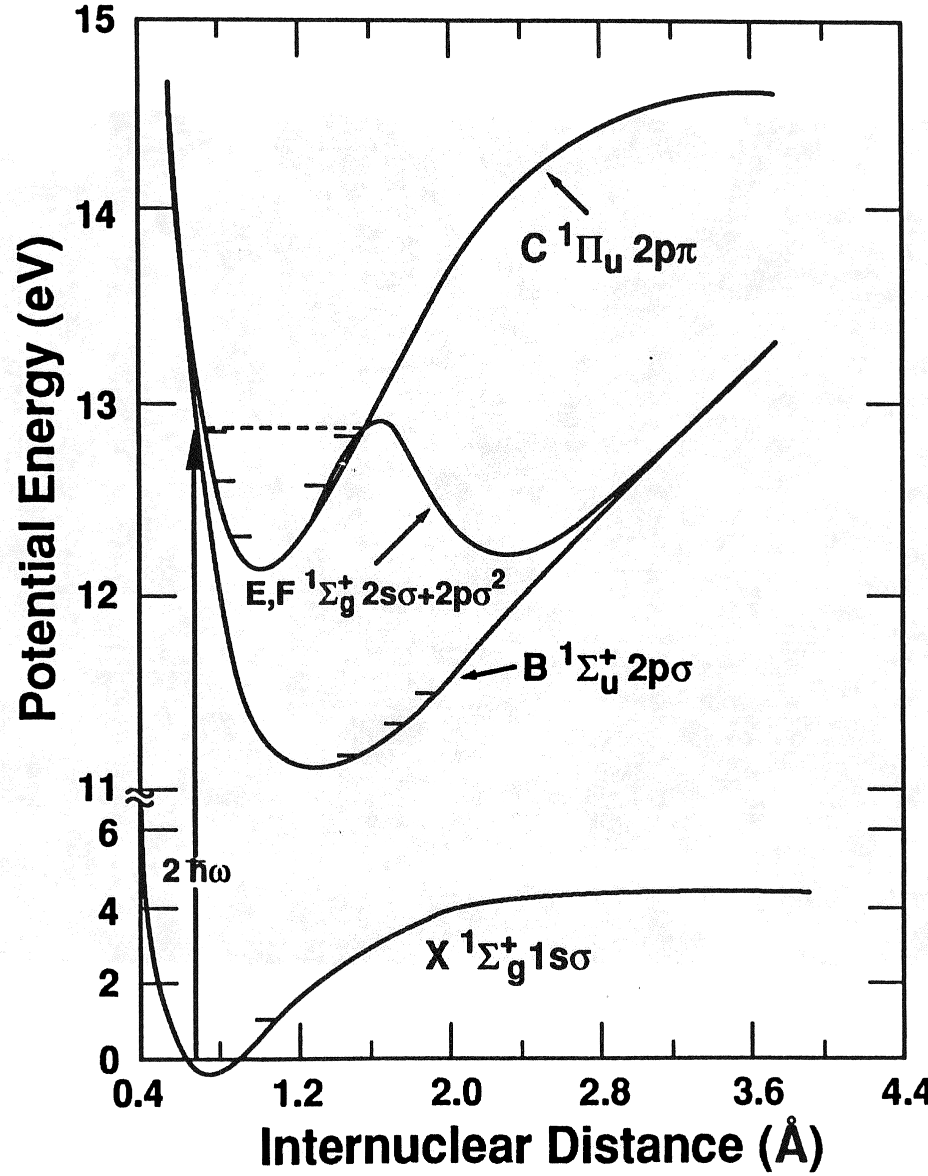
Los Alamos
CLS-92-592

Laser Induced Fluorescence Excitation Scan

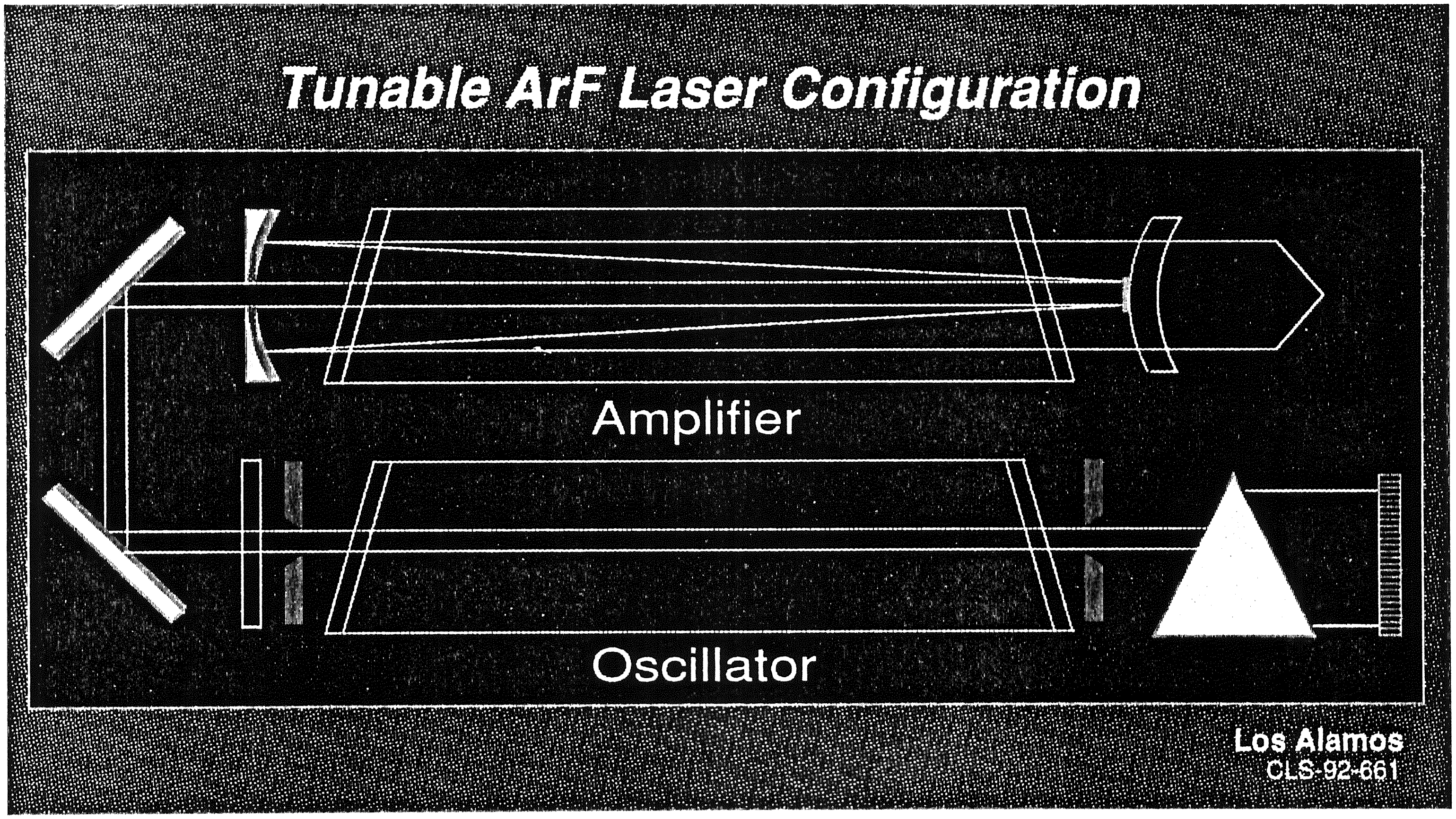
Scanning the laser excites different rotational levels of the ground vibronic state ↔ temperature, number density



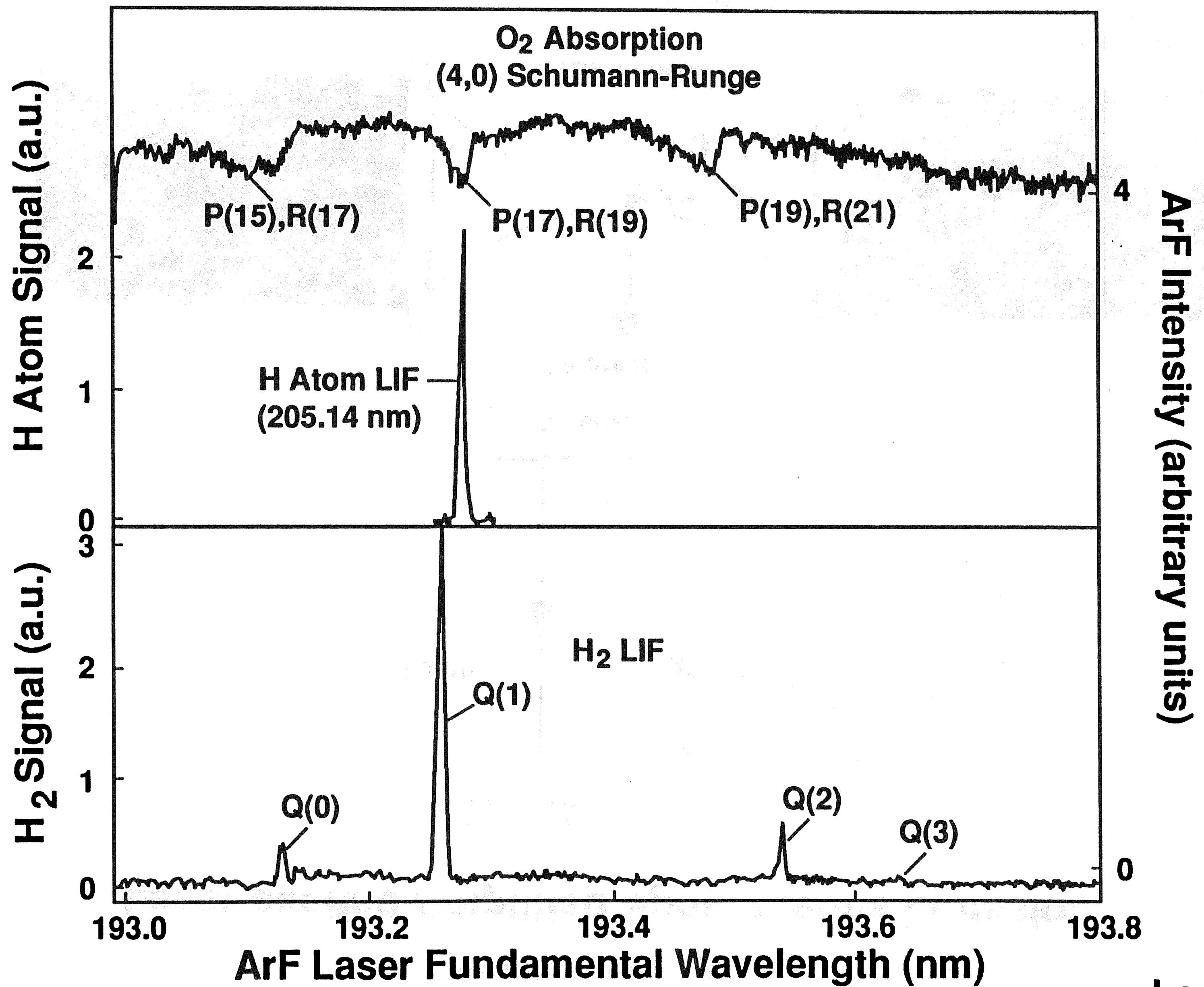
H_2 Potential Energy Surfaces



Los Alamos
CLS-91-1848

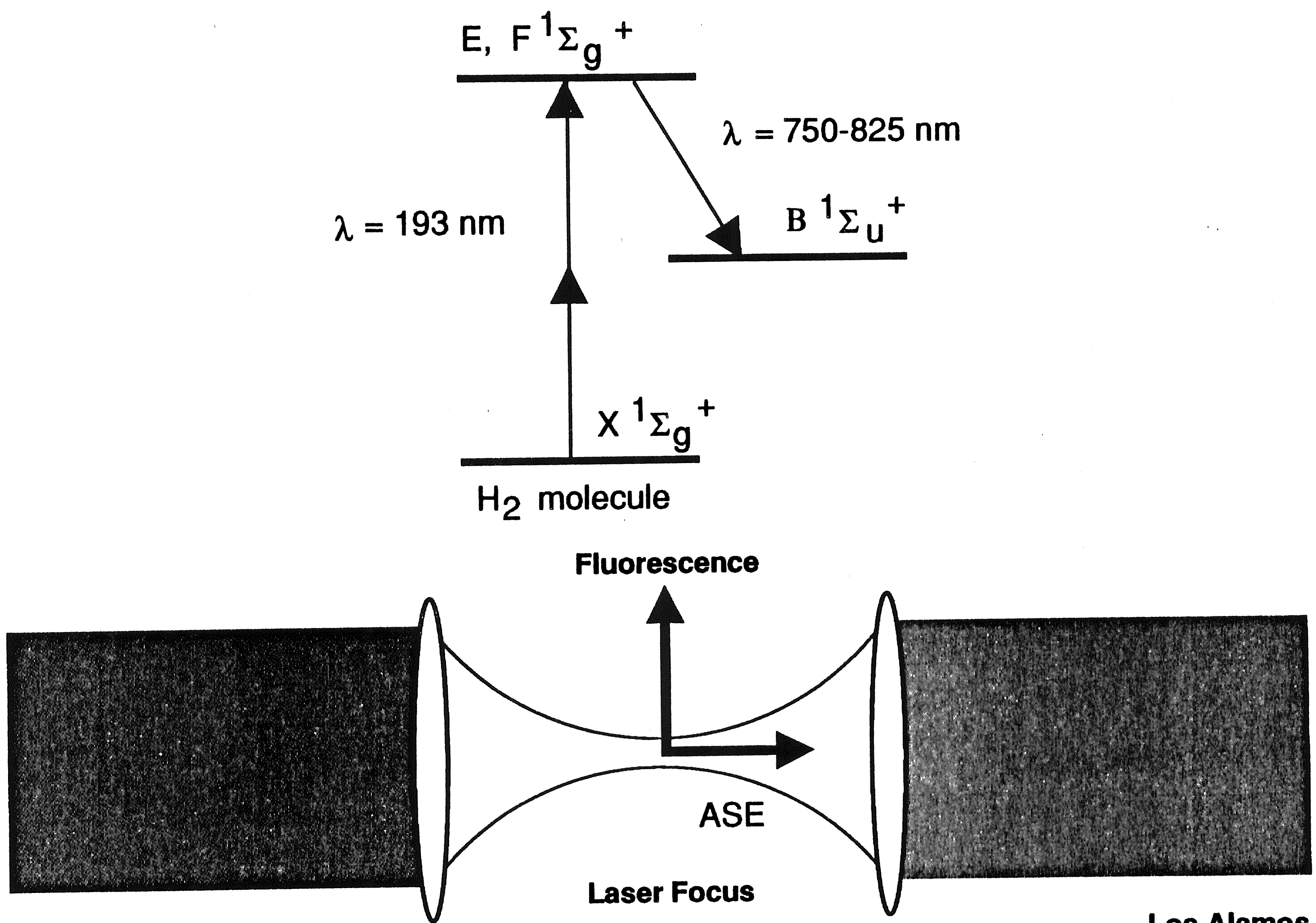

Tunable ArF Laser Configuration
Amplifier
Oscillator
Los Alamos
CLS-92-661

ArF Excimer Spectra of H_2, H, and O_2



Los Alamos
CLS-91-1849

Laser-excited Amplified Spontaneous Emission



Los Alamos
CLS-92-594

Advantages

Resonant → very sensitive $\sim 10^{10}\,cm^{-3}$ (ppm)

Highly collimated signal → slow optics collection

Good spatial resolution

Quenching largely defeated due to rapid SE

Disadvantages

Effects due to atmospheric pressure quenching unknown

Oxygen absorption (purge beam path) affects laser propagation over long distances

Must focus laser to promote two-photon transition (however 2-photon cross section is quite large)

Los Alamos
CLS-92-593

US NAVY NEEDS FOR HYDROGEN MEASUREMENT

Dr. Jeffrey R. Wyatt
Naval Research Laboratory
Washington, DC

SOURCES OF HYDROGEN

SUBMARINES

- BATTERY CHARGING
- OXYGEN GENERATION BY WATER ELECTROLYSIS
  - Internal detection
  - External detection
  - Note: Submarines produce about 7000 cu ft of Hydrogen/day

FACTS OF LIFE (For Life Support)

One Person	Ft^3/hr	Lbs/day	Lbs/90 days	Tons/submarine Crew
Oxygen	1	1.8	160	12
Carbon Dioxide	1	2.5	250	19

Oxygen Sources

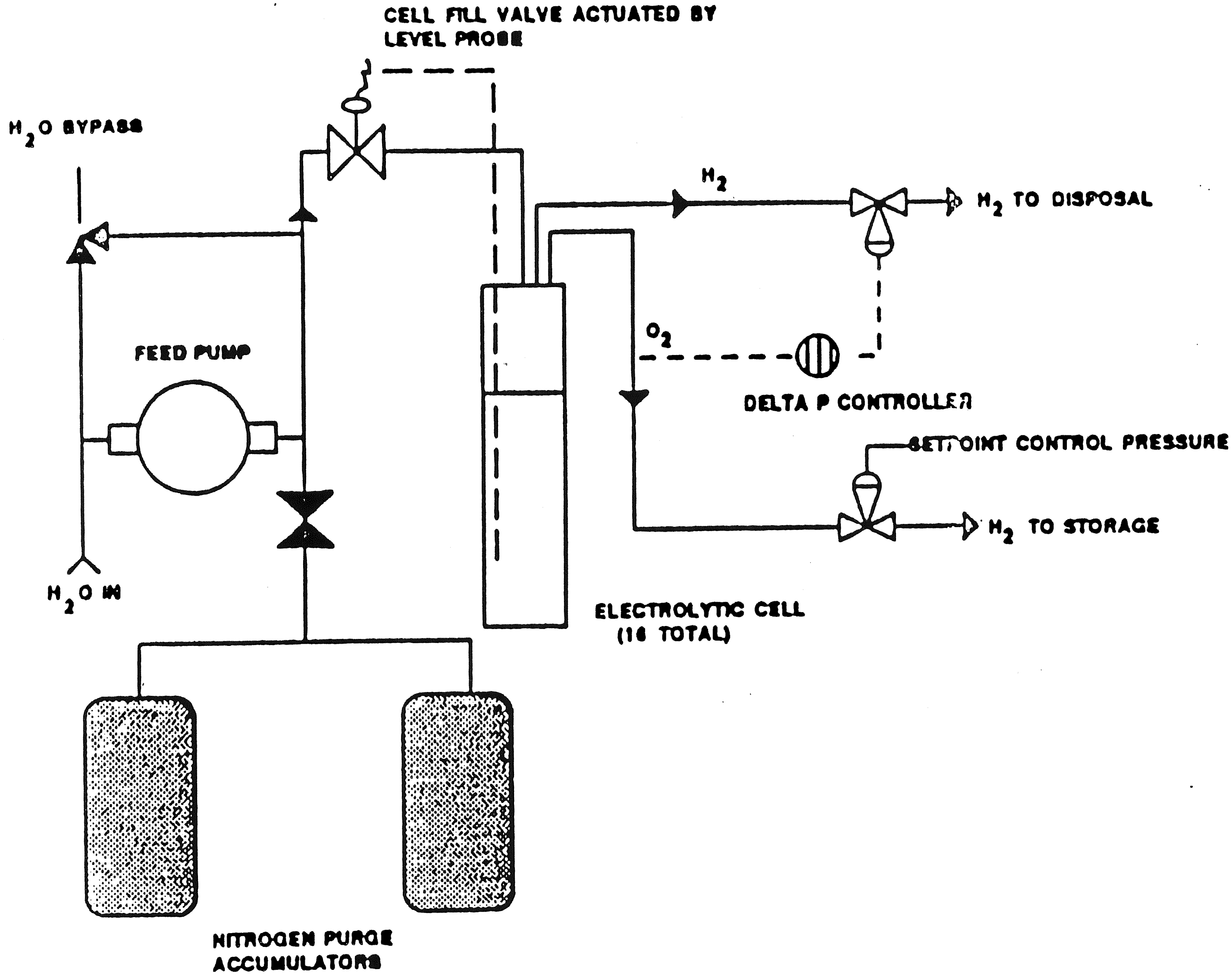
Oxygen Candles: 2.5 lbs for each lb of oxygen produced

Water Electrolysis: 500W/person

Carbon Dioxide Removal

To maintain a 0.5% CO_2 level in the atmosphere requires processing 200 ft^3 of air/hour/person. This requires 200W/person for a MEA scrubber or 1.4 lbs of LiOH for each lb of CO_2.

SIMPLIFIED SCHEMATIC OF OXYGEN ELECTROLYZER



SOURCES OF HYDROGEN

ALL SHIPS

- CATHODIC PROTECTION OF SHIPS

$$Mg \rightarrow MgO + Mg(OH)_2 + H_2$$

$$Fe_xO_y \rightarrow Fe + O_2$$

- THUS ALL SHIPS PRODUCE TRACE AMOUNTS OF HYDROGEN IN SEA WATER WHICH POTENTIALLY COULD BE DETECTED

CURRENT METHODS OF DETECTION

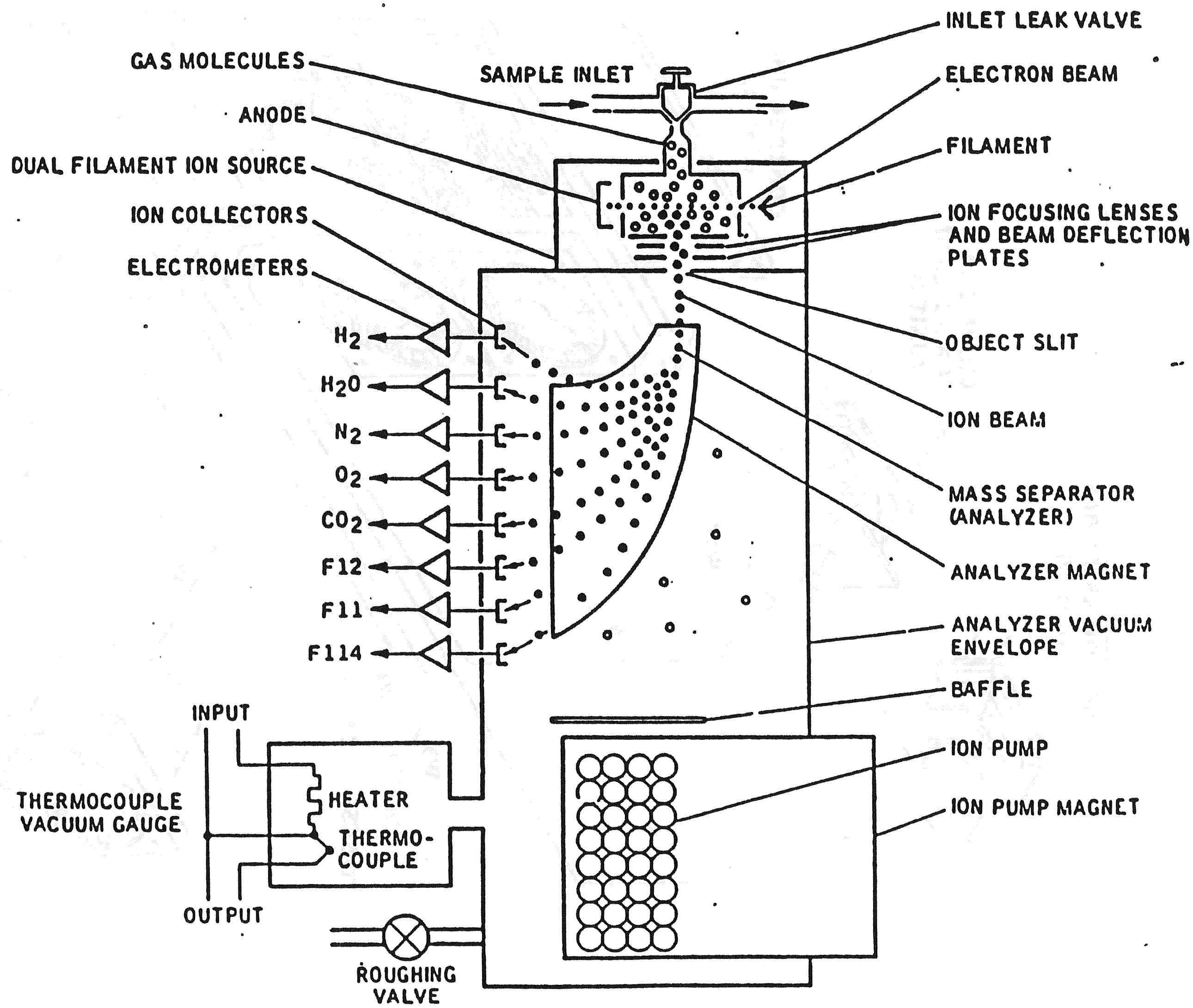
- SUBMARINE ATMOSPHERE -- Central Atmosphere Monitoring System (CAMS-I and CAMS-II) magnetic mass spectrometers, monitor from 0.1% to 10%.

    - Specific, minor humidity interference at low levels
    - Expensive $200k-$350k
    - No calibration required, two year MTBF

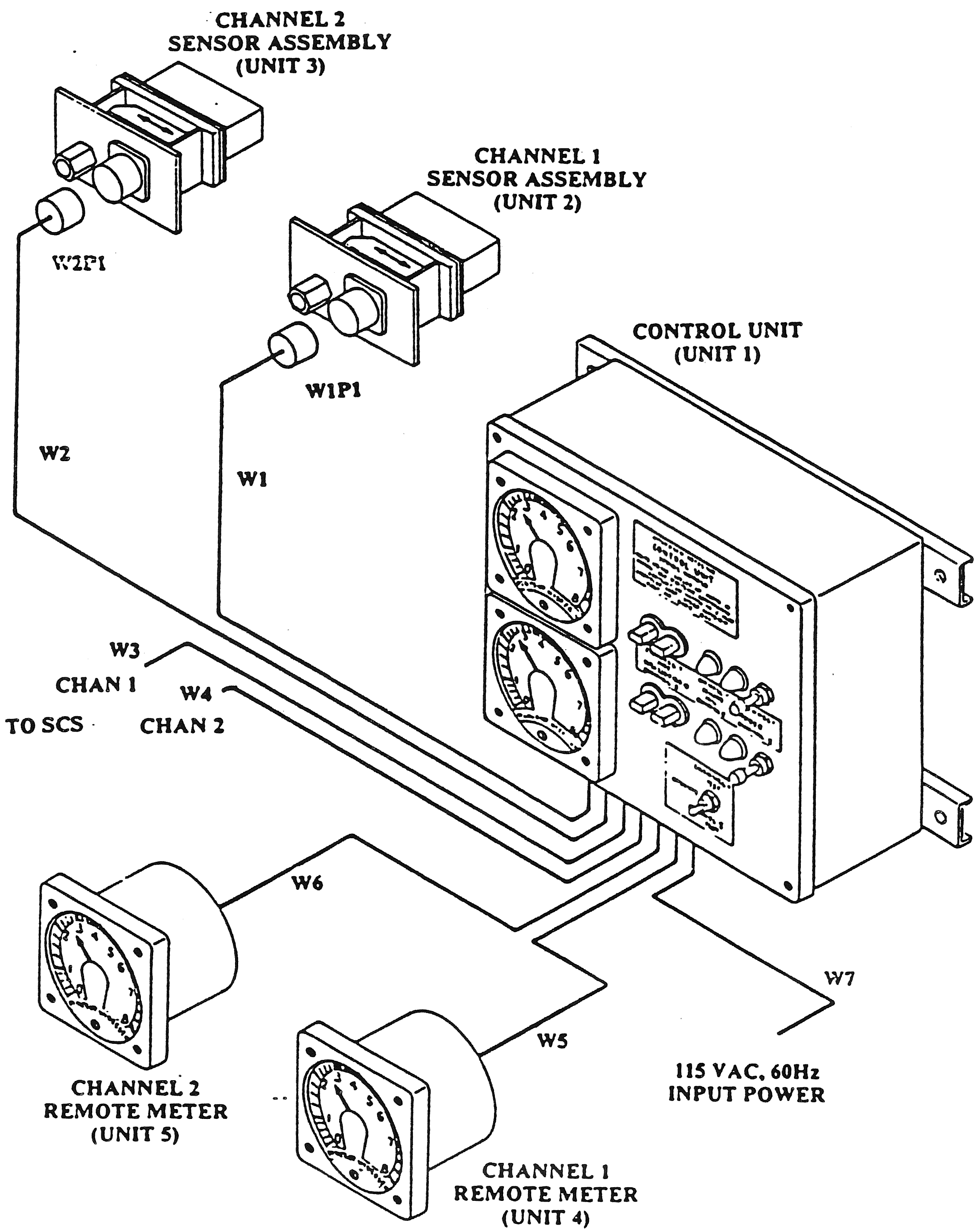
- BATTERY WELL -- Thermoconductivity

    - Simple, proven 1950's technology
    - Requires calibration due to drift
    - Operates in a flowing environment

SCHEMATIC OF CENTRAL ATMOSPHERE MONITORING SYSTEM, MARK-I (CAMS-I)



SCHEMATIC OF BATTERY WELL HYDROGEN DETECTOR



CURRENT METHODS OF DETECTION (Cont)

- **OXYGEN GENERATOR -- Catalytic Oxidation**
  - Simple, proven technology
  - Requires calibration due to drift, daily zero and biweekly span
  - Action levels vary from 0.5 to 3%
  - May be affected by replacement refrigerants

- **IN SEA WATER -- Cannot discuss**
  - Trace level detection needed

NEEDS

1. A small simple hydrogen in air sensor with the following characteristics:

   - Span the range 0.1-10%
   - Seldom requires calibration
   - Small enough so that many potential leak points could be monitored
   - Intrinsically safe

2. A sensor to measure oxygen in 3000 psi hydrogen

3. A sensor to measure hydrogen in 3000 psi oxygen

4. Trace hydrogen in sea water, order of magnitude: less than one ul hydrogen in one liter of water

PRESENTATION OF NEW HYDROGEN PIPING DESIGN GUIDE

CGA PHAMPLET G5.4

MICHAEL A. DEAKTER

The past two days we have crossed the gamut of current technology with regard to detection of hydrogen. You are the government - you have some control over your facilities and your personnel that handle and are exposed to hydrogen systems.

Now think of industry - what is happening out there where hydrogen is used everyday - tens of thousands of cubic feet per hour; at any given hour some 50 liquid hydrogen trucks are crossing the highways and interstates hauling hydrogen to thousands of users every day, every hour.

Hydrogen:

INCREASED APPLICATIONS:

Globally we are continuing to see new uses and an increasing demand for industrial gases. Hydrogen, one of the five major bulk industrial gases is following in that increased demand, these increasing customers all have new hydrogen applications which are unique and challenging.

Many new facilities and laboratories are requiring hydrogen as a source gas. There varied needs and applications are diverse. Their demand and volume usage vary as much as the differing applications. What was thought to be a very special and exotic cryogenic liquid in the 1950's during Americas early space program has become a standard industrial gas with hundreds of uses in the 1990's. Liquid hydrogen is now shipped all around the globe. As the increased uses and needs for hydrogen develop so must our awareness and required knowledge for safety concerning hydrogen and the equipment associated with its uses. Some traditional industrial applications of hydrogen use are hydrogenation of cooking oils, shielding of power turbins for windage and winding losses, a clean burning fuel gas for aerospace to annealing furnaces and large chemical refineries. The Micro electronic and computer chips manufacturer usage of H2 is up 800% in the past 10 years. That is about 300 new LH2 tanks, each 1,500 to 20,000 gallons stationary storage vessels setting in customers parking lots. The large scale industrial and aerospace uses for hydrogen have been around now for more than 40 years. These applications and their particular uses of hydrogen are well documented in the aerospace industry. The safety practices associated with each particular hydrogen application traditionally came from you the aerospace user as a service from the industrial gas company or equipment manufacturer. Unfortunately you learned as you grew. As you developed applications for hydrogen you also developed safe procedures. Many times however, incidents resulting in leaks, the very type we discuss at this Technical Meeting, are what cause the procedures and safe practices to be reviewed or developed. As the need grows and applications for hydrogen increases so does the need for safer and more efficient ways to bring that safety to the public in ways they can apply it.

Safety and safe practices regarding hydrogen comes first and foremost from within. Your working knowledge and familiarity with hydrogen safety is critical as we continue this decade of increased usage and greater personnel exposure.

Five years ago the Compressed Gas Association (CGA) Hydrogen community was virtually non active. There was only a skeleton committee which updated the hydrogen commodity specification when required to do so. Now the CGA hydrogen committee has grown to over 30 committee members representing industry that actively devotes time to areas relating to handling and safe practices regarding hydrogen. Three years ago the hydrogen committee set out to accomplish a specific task. To develop a standard which describes the specifications and general principles recommended for a hydrogen piping system at a users facility. We know that commercial producers and large industrial chemical refineries using hydrogen have in house existing hydrogen safety specifications and practices. The bulk hydrogen producer along with a fun group from Washington called the DOT insure the safe transportation and labeling of hydrogen. But what happens to it once it is delivered to the customer? Especially the small or unfamiliar hydrogen user; the user with no hydrogen detectors of any kind. Does the responsibility end? Do the safe practices and procedures we employ fade away when we are not involved? Of course they don't. Safety is everyone's concern.

This is why the hydrogen piping standard for user locations has been written; through CGA and its company's input we have incorporated those concepts and safe procedures which have been proven and keep our employees safe while exposed to hydrogen systems as part of our daily work place.

Two typical types of hydrogen piping systems are installed.

One type is the complex distribution and varied use points for hydrogen to be piped through a micro-electronic facility. Now many large electronic facilities require multiple hydrogen outlets throughout a building and throughout the facility, Potentiality exposing hundreds of employees in confined clean room facilities to hydrogen. Usually the hydrogen is supplied from a bulk liquid storage tank set in the parking lot.

The other type of install is single use; the addition of a bulk hydrogen storage tank and distribution line at existing or newly constructed industrial gas plant or at a government facility.

These two examples are diverse. On one hand the end user with multiple use points and multiple applications. The other, at an industrial gas production or aerospace test facility with only one line and one use point per system. The parallel, the equal, the thing these two applications and diverse facilities have and must have in common is SAFETY. Safe practices and safe operation. As diverse as any two facilities can be, the precautions, standards, and level of safety should always be as high as possible. All to often we here of a project or engineer that requires a hydrogen source (either liquid or high pressure) and has to design and install or have installed by a typical mechanical contractor his own hydrogen lines. What standard does he go to? Where does the engineer or contractor look for guidance...

Lets look at what standards were available:

The hydrogen storage vessel is constructed to rigid ASME and/or DOT specifications. Special considerations are made for temperature and pressure conditions. The hydrogen storage tank location, whether from a liquid source or a high pressure source or combination of both, is covered by NFPA gaseous 5ØA and NFPA liquid 5ØB standards. All the possible safety considerations are reviewed and discussed and these standards and codes are available now and have scheduled reprints for 1993. But wait.... what about the pipeline from the storage tank to the location of use? What integrity to prevent leaks has the delivery system and its hardware been designed to? Well up to now that's were the safety - as far as the written form was dropped. This new CGA hydrogen piping standard, soon to be published, is for the end user, such as a facility engineer in a micro electronic plant or a production engineer at a chemical refinery. You the aerospace user/designer and facility manager need to use consistency to design new piping for hydrogen lines.

This type of hydrogen piping standard is designed as a guide and a reference tool for all hydrogen users and applications. I assure you it will minimize and reduce your leaks and your potential hazards.

Let me briefly remind some of you, and hopefully educate some of you, to those properties of hydrogen which make it so unique and how this piping standard deals with hydrogen leak tight integrity. The following information paraphrases the information which the new document covers.

"Hydrogen is a flammable gas. It is colorless, odorless, tasteless and nontoxic. It is the lightest gas know, having a specific gravity of 0.0696 (air = 1.0). Hydrogen diffuses rapidly in air and through porous materials. At atmospheric pressure the flammable range is from approximately 4 percent to 74 percent by volume of hydrogen in air. Hydrogen-air mixtures are extremely easy to ignite requiring only Ø.Ø4 millijoules (mJ) ignition energy compared to Ø.25 mJ for hydrocarbons. When cooled to its boiling point of -423 degrees F (-252.8 degrees C) hydrogen becomes a transparent liquid only one-fourteenth as heavy as water. All other gases except helium become solids at the temperature of liquid hydrogen. Due to its small molecular size, hydrogen diffuses rapidly through porous materials. It also dissolves in and diffuses through metals slowly at ambient temperatures. It may leak out of a system which is gas-tight for air or other common gases at equivalent pressure. Hydrogen can also leak through the inside passages of multiple-strand wire conductors. Hydrogen is nontoxic, but it can act as an asphyxiant by displacing the oxygen in the air. Remember, the flammability hazard of hydrogen is present at much lower concentrations than those causing asphyxiation. Liquid hydrogen, and also the cold gas evolving from the liquid, can produce severe frost burns similar to thermal burns upon contact with the skin and other tissues. Liquid hydrogen flowing through uninsulated lines causes the liquefaction of air on the outer surfaces and oxygen-rich liquid air will drip from the line." In some cases it may cause $\emptyset_2$ enrichment of the immediate area. "Hydrogen is non-corrosive and considered non-reactive with most common metals, but hydrogen service will generally reduce the mechanical properties of certain metals by the complex process of embrittlement which can occur in three ways: A) Environmental hydrogen embrittlement, B) Internal hydrogen embrittlement, C) Hydrogen reaction embrittlement." It is important to know and understand these embrittlement concerns when designing and specifying a system. The next section discusses piping system criteria and vent or exhaust piping considerations. "Hydrogen piping systems should be designed in accordance with the principles of ASME B31.3, other applicable codes and regulations, and the special requirements for hydrogen service. Single wall piping is used for gaseous hydrogen service and for liquid hydrogen service when thermal insulation is not required. Vacuum insulated piping is recommended for liquid hydrogen service. All electrical equipment and electrical grounding apparatus shall comply with NFPA 7Ø, National Electrical Code (7). The piping system shall be bonded and grounded to dissipate static electricity. Material specifications and thickness requirements for piping and tubing shall conform to the Code. Piping, tubing, valves and fittings for operating temperatures below -20 degrees F (-28.9 degrees C) shall be fabricated from materials listed in the Code for the operating temperature, or shall meet the impact test requirements when tested at the minimum operating temperature to which the system will be subjected in service. Austenitic (300 series) stainless steels meeting the temperature limits of the Code may be used for liquid and gaseous hydrogen product piping, tubing, valves and fittings.

Carbon steel is usually limited to warm gas service, but may be used for gaseous hydrogen piping, tubing, valves and fittings within temperature limits of the Code. Carbon steels with higher strength levels, high carbon and low alloy content are more susceptible to hydrogen embrittlement and their use should be thoroughly reviewed. Pipe, valves, fittings of gray, ductile, or malleable cast iron shall not be used in hydrogen service. Low melting point materials - aluminum, copper, brass and bronze - have reduced strength at elevated temperatures. Their use should be in accordance with Article 317 and Appendix G of the Code. Plastic piping and tubing shall be avoided in hydrogen piping systems, expect under controlled conditions for low flow and low pressure laboratory applications, and when used in accordance with the Code. Some of the major system components are:

pressure relief devices; hydrogen storage tanks, and the containers of transport vehicles, are protected by pressure relief devices as specified by the ASME Code (8) or DOT regulations (9). The storage system devices do not protect the downstream gaseous or liquid hydrogen piping systems which need to have their own pressure relief devices. Pressure relief devices shall be provided in hydrogen piping systems to relieve pressures that may exceed the maximum allowable working pressure of the system. The discharge from pressure relief devices shall be piped to a properly designed and functioning hydrogen vent stack discharging outdoors in a location and direction that avoids impingement of escaping gas (or liquid) on adjacent equipment, structures, or personnel. The venting hydrogen should generally be directed upward and the piping designed so that moisture cannot collect and freeze to interfere with proper function of the device. Vents shall not discharge where hydrogen can accumulate, such as below eaves of buildings. Stainless steel and non-ferrous metals are preferred for vent piping to minimize the possibility of ignition due to impingement of corrosion particles against the piping. The vent piping shall be sized to handle the full discharge capacity of the pressure relief devices. Isolation valves with lock-out capabilities are used to isolate portions of the piping system in emergencies and for routine maintenance." These may be a simple lock and chain, isolation is an important safety factor.

"Emergency Isolation Valves (EIV) are normally located on product lines to provide a readily accessible manual or an automatic means of stopping hydrogen flow in the event of an emergency. EIV's should have metal-to-metal seat or a metal-to-metal back-up of a soft-seated valve for positive shutoff. Requirements for remote shut off valves on liquid hydrogen tanks are given in CFR 29 1910.103 (13) and NFPA 50B (4). Excess flow valves for gaseous hydrogen service are self-actuated devices that shut off flow when the built-in sensing mechanism detects that a pre-set maximum flow rate has been reached. EFV selection must be based upon the specific requirements of the equipment or facility to be protected. Check valves prevent reverse flow, which can cause contamination." Often check valve failure is the cause for serious damage when the valve failed to hold back the hydrogen. "Regardless of the type of instrumentation, valves, gauges, regulators and other accessories each shall be suitable for hydrogen service and for the pressures and temperatures which will be encountered. Regulators should be non-venting types or have the vents piped to a safe location. The use of safety glass and blowout plugs on pressure gauges is recommended. In-line restricting devices may be used to reduce flow to gauges. Now here is what we state for basic hydrogen detectors. Hydrogen gas detectors may be installed to sound an alarm or activate shutdown on detection of gas leakage. Sensors should be installed at locations most likely to have an accumulation of hydrogen in the event of a leak. Sensors should be installed at ceiling height. The detectors should be set to alarm at 1% (25% of the lower flammable limit) and to shut down at 2% hydrogen concentration. Typically, detectors are not required for outdoor systems.

Gasket and Sealing Materials:

"Graphite compounds maintain integrity at elevated temperatures and may be used for flange and valve packing gaskets for gaseous and liquid hydrogen.

The use of elastomers and plastics should be limited in gasketing, packing or other sealing elements where their failure due to fire could cause hydrogen leakage. Suitable compound materials should be selected giving consideration to hydrogen compatibility and intended service temperature."

Some Other Additional Requirements For Associated Hardware:

"Cabinets or enclosures containing hydrogen control equipment shall be ventilated or purged with inert gas to prevent the accumulation of hydrogen/air mixtures.

Flexible hoses shall have electrical continuity through the length of hose, or shall have a bonding strap along the length of hose, and be connected at each end to ensure electrical continuity.

Sections of piping or equipment connected by an insulated gasket joint or flexible hoses must have electrical continuity to the system ground. A bonding strap may be used if required."

Regarding Electrical Equipment:

"As hydrogen systems may pose a fire or explosion risk, electrical systems must be in accordance with NFPA 50A, NFPA 50B and NFPA 70, Articles 500 and 501. The type of electrical equipment which may be installed is dependent upon the level of hazard classification of the Code."

The Cleaning Section Covers in General:

"Piping systems should be cleaned before being places in hydrogen service. The procedure selected must be suitable for the type of contaminant and must provide the level of cleanliness required by the application.

Internal parts of components and assemblies should be cleaned prior to assembly. Subassemblies should be cleaned after completion of all welding, machining, hydro testing and other mechanical work. Precautions should be taken to avoid contamination after cleaning.

Cleaning may be accomplished by one or more of several procedures. More detailed cleaning information may be found in CGA G-4.1, which covers oxygen cleaning.

Mechanical cleaning may be accomplished by wire brushing, grinding, blast cleaning or tumbling. These methods remove contaminants such as scale, slag and weld splatter which are not removed by steam, water or chemicals.

Chemical cleaning procedures use solvents, acids, caustic cleaners or detergents to remove oxides, dirt, oil, grease and other organic materials. Selection and use of a cleaning agent is based on the type of contaminant, compatibility with materials and compliance with the governing agency requirements for environmental impact and toxicity.

subjected to a visual inspection of accessible surfaces and a wipe test of inaccessible surfaces.

A careful visual inspection should be performed on surfaces under white light or strong daylight conditions to detect the presence of excessive contaminants. The surface should be recleaned if excessive amounts of the following contaminants are detected:

a. Moisture.

b. Loose corrosion, scale or weld spatter.

c. Dirt, filings, chips or particles.

d. Organic matter, such as grease, oil, paint, markers, crayon and ink.

The following contaminants are not considered objectionable and may remain in the piping.

a. Tightly adherent scale or weld spatter.

b. Moderate rust film.

c. Stains or discoloration.

d. Certain adherent films used for rust prevention as approved by owner.

Wipe tests are an aid to visual inspections and are used to detect contaminants on surfaces which are not accessible for visual inspection."

Installation:

General. "All piping systems should be designed and installed in accordance with ASME B31.3 and local, state and national codes. Consideration must be given to thermal expansion and contraction of the piping when installing pipe anchors and supports. Piping should be routed as far away as practical from other lines and process equipment containing fluids which are hazardous in a hydrogen environment.

Piping joints shall be suitable for hydrogen service at the operating pressures and temperatures, and selection shall consider tightness, mechanical and fire safety acceptability.

Joints in piping and tubing shall preferably be welded for leak tightness and fire safety. Joints may also be threaded, flanged, brazed or made with a suitable mechanical fitting. Soft solder joints are not permitted.

Threaded joints are acceptable on hydrogen piping but should be minimized to reduce potential for leakage.

Flanged joints on hydrogen piping shall have a metal-to-metal seal, or a gasket of material such as graphite, which is difficult to ignite, or be suitably protected from a fire safety standpoint.

Brazed connections must use a brazing alloy with melting point above 1000 degrees F (538 degrees C).

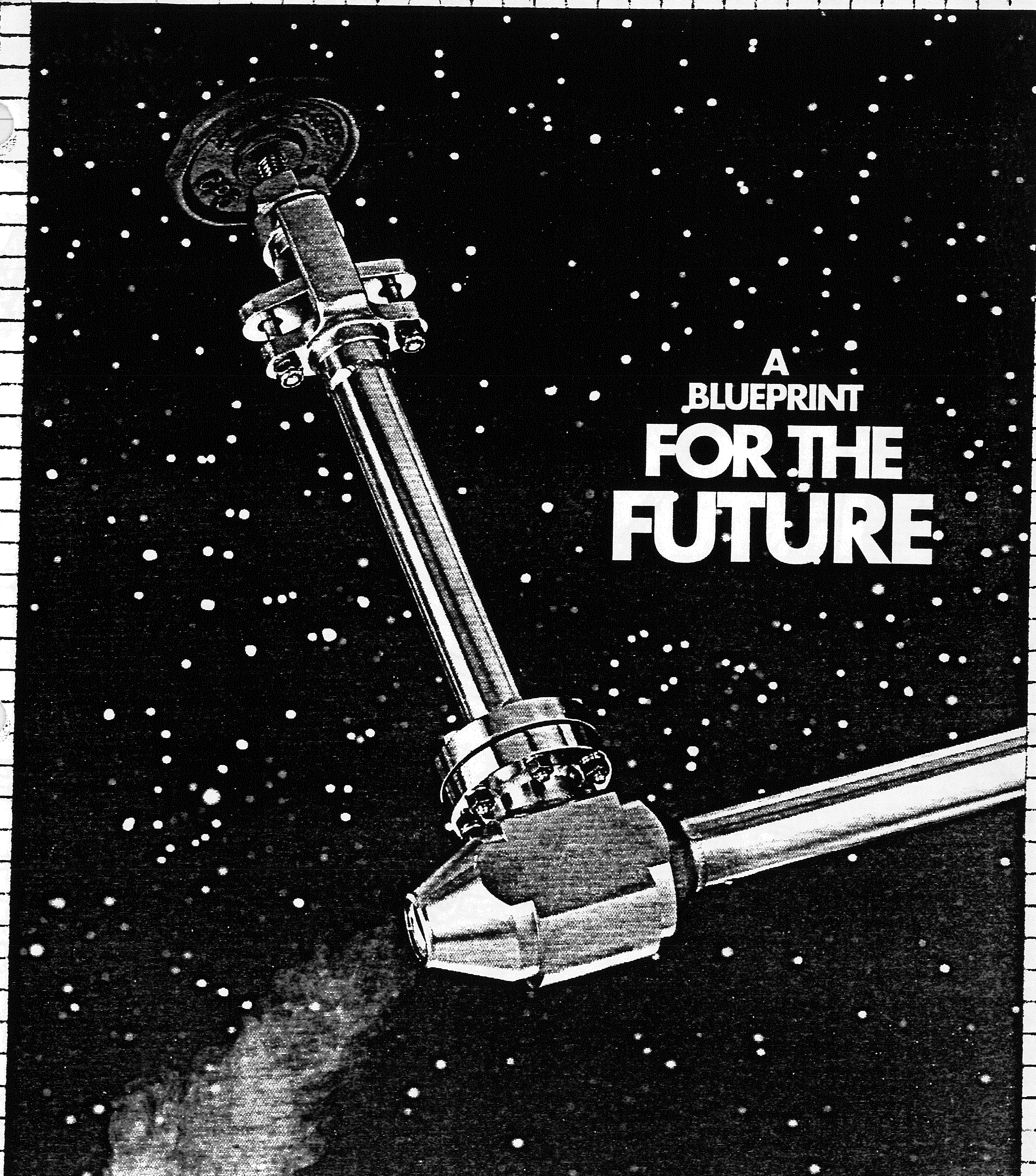
Mechanical fittings including flared, flareless compression, union, proprietary and special joints may be used within the guidelines and limitation of Articles 315 and 318 of ASME B31.3. In selecting and applying a fitting, the designer shall consider the possible adverse effects on the joint due to operating conditions such as high pressure, cyclic loading, vibration, shock and thermal expansion and contraction. The fitting manufacturer's recommendation should be obtained for the particular application."

There is even a typical gaseous hydrogen piping system drawing shown at the end of the printed document. It details all typical components and piping configurations for a safe system. There is a section on above-ground and underground Hydrogen system installation, and all the mechanical concerns to minimize leaks.

This new publication also deals with Start-up and Field Maintenance of new and existing Hydrogen piping systems, as well as sections on inspection and testing.

Most of all, this new documentation contains information, IMPORTANT INFORMATION, for YOU, the Hydrogen user. By establishing a base line document, we disperse the pertinent safety information to the user. But, by everyone's utilizing and employing a document such as this, we will minimize the potential for, and reduce the risk of leaks in Hydrogen systems. Maybe that will take some pressure off some of you H2 detectors out there.

Thank you for your time on behalf of the Compressed Gas Association and myself, Mike Deakter, of Consolidated Precision Corporation.



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Manufacturing Hardware and Servicing the Industrial Gas Industry

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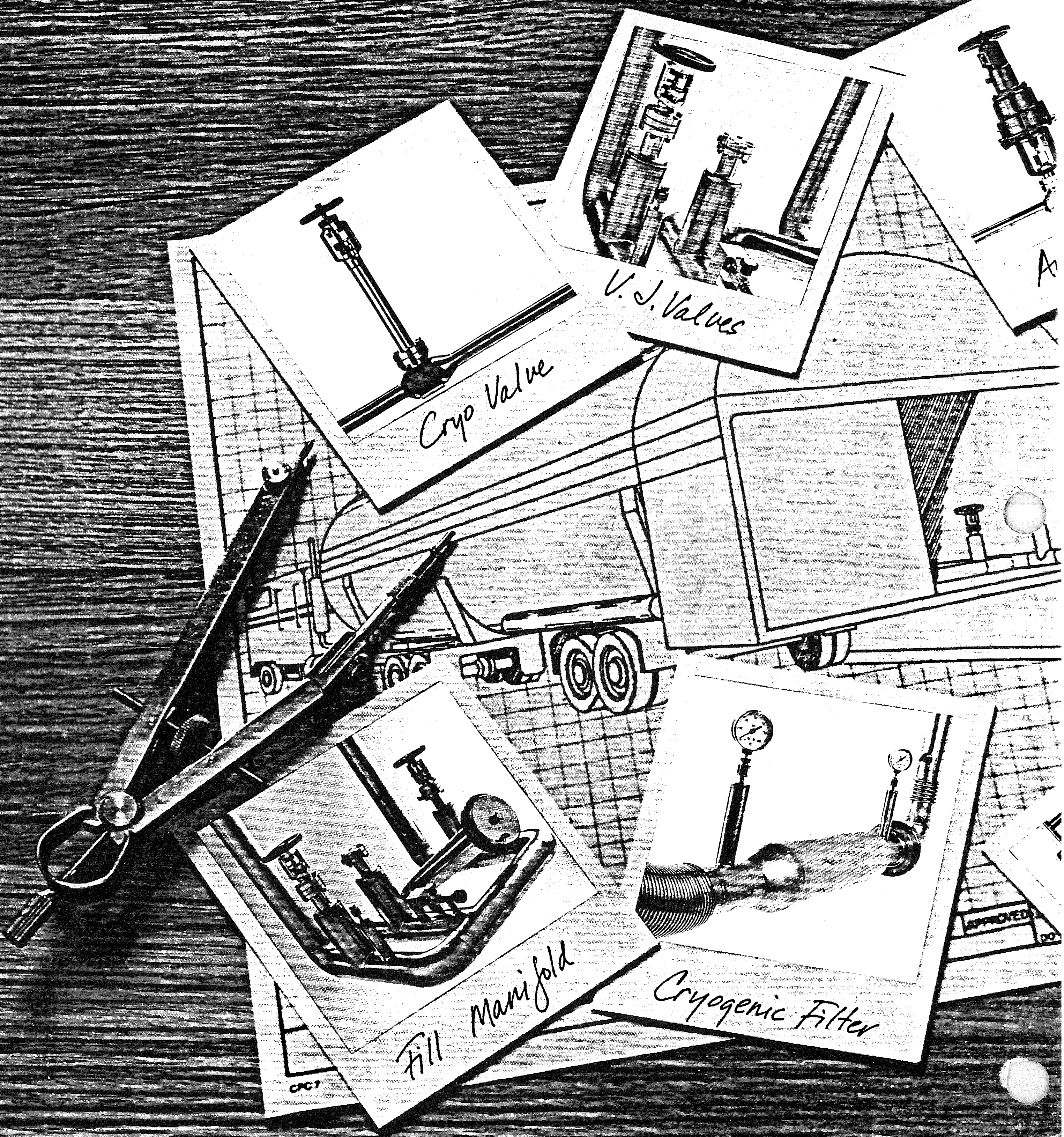

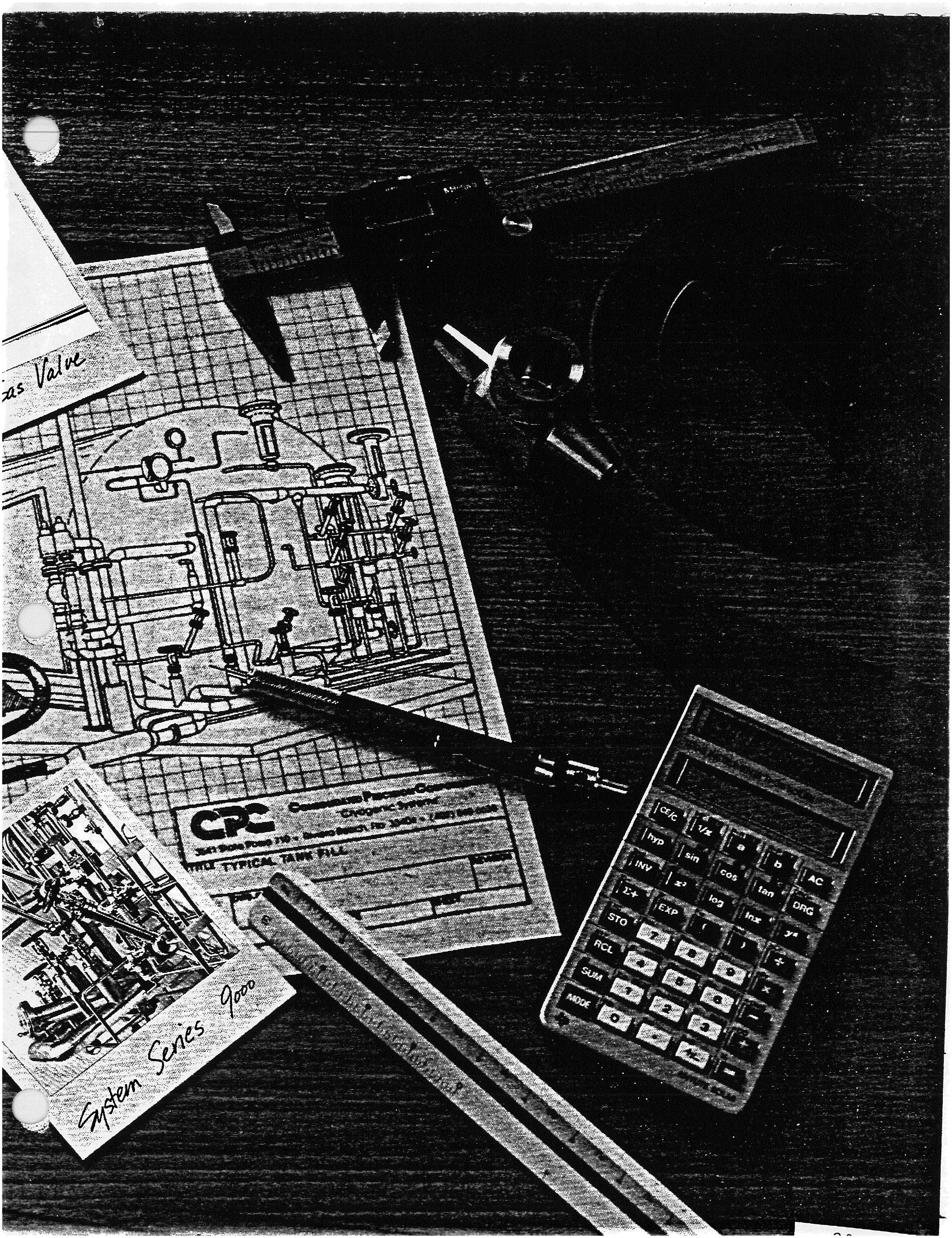
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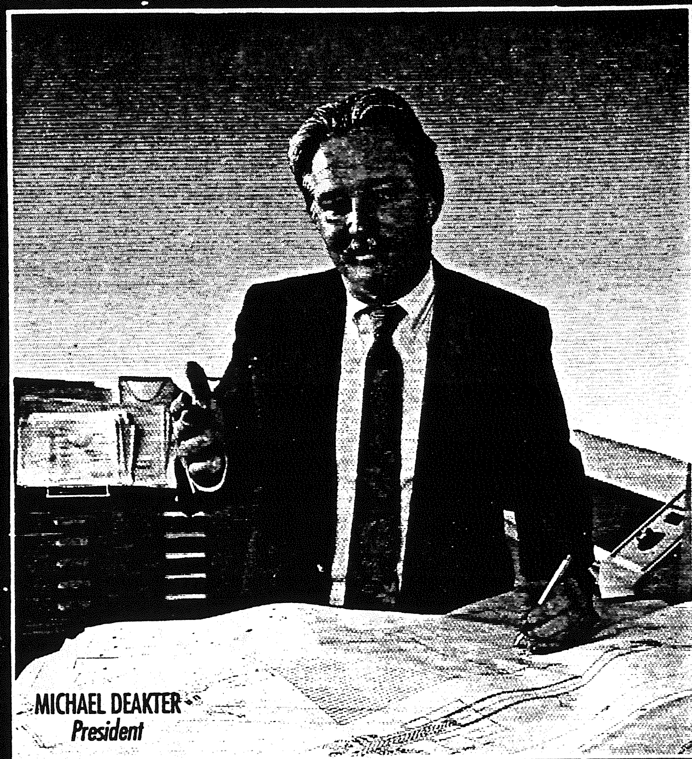

MICHAEL DEAKTER
President

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We design with safety and performance in mind, while satisfying our customer's requirements.

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Based on an evolution of accepted practices, we have contributed to many technological advances throughout the industrial gas industry.

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All design considerations are subjected to rigorous qualification tests, prior to incorporation into our products.

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20-21

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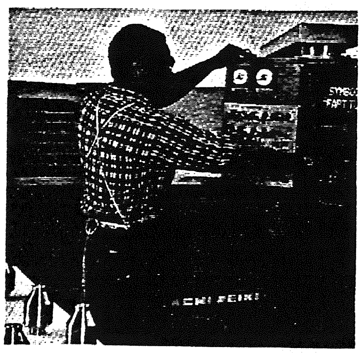
Consolidated Precision Corporation - Technical Welding Service



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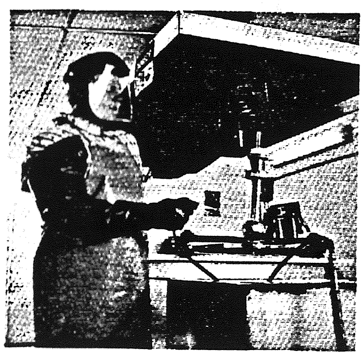
Through the continual updating and expanding of our machining capabilities with the most current technologies, we can assure timely deliveries to our customers.



ELECTRO POLISHING

We have designed our Electro Polishing System to accommodate our complete product line. By maintaining our own in-house system, we control the solution exposure to 316L stainless steel materials exclusively.

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The constantly monitored Deionized Water System at CPC-TWS is an integral extension of our Electro Polishing System. We remove all residues that are generated from the Electro Polishing processes.

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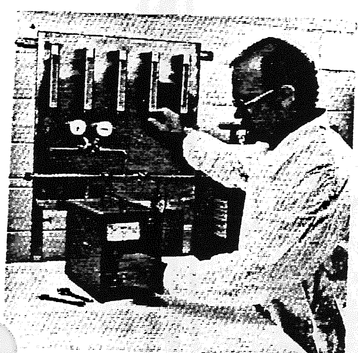
Qualification of component parts and assemblies with the use of leak-detection equipment has always been part of our Quality Control capabilities. For field applications, we also maintain portable leak-detection equipment.

CPC

We put it all together for you!

INSPECTION

From the receipt of raw materials to final system acceptance, our quality control inspectors continually monitor the product throughout our manufacturing process. Utilizing the latest techniques and equipment, our team assures compliance with design specifications.



TESTING

Our standard procedure is to perform a series of tests throughout our manufacturing and installation processes. Our acceptance test procedures and test results are available to our customers when required.

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A Hydrogen Selective Gas Sensor from Thick Oriented Film of MoS_2, WS_2 and TiS_2

KONRAD COLBOW

We have developed and applied for patents on a new gas sensor with higher sensitivity and selectivity to hydrogen gas, utilizing a new technique and a new process. The sensor operates at relatively low temperature (about 120°C or lower), which reduces the power consumption and possibility of gas/air mixture ignition. This can be compared to the present commercial gas sensors (e.g. Figaro of Japan) which operate at temperatures between 300–400°C.

A hydrogen gas selective sensor is fabricated using the semiconducting metal oxides in the form of highly oriented and partially crystalline thick films. The oxides are prepared from their corresponding dichalogenide (normally sulfide) compounds.

In an experimental arrangement, the sensor is prepared starting with a substrate supporting a thick film of highly oriented single layers of the layer compounds MoS_2, WS_2 and TiS_2. The films are deposited between two gold electrical contacts about 1 mm apart. A catalyst such as Pt, Pd or Ru in solution is deposited and dispersed into the thick film. The above film is then sintered in air to convert the oriented sulfide to the corresponding oxides which is sensitive to hydrogen. The sensors' response to hydrogen gas is found to be linear in the range of 30 ppm to about 10^4 ppm (1%) with less than 1 minute in response time at a temperature of 120°C. At room temperature linearity is found in the range 100 ppm to 5 x 10^4 ppm (5%), but the response time is longer. At concentrations of 1% to 5% the response to hydrogen is also linear, but has a different slope than that for concentrations less than 1% hydrogen.

The sensitivity depends not only on the concentration of hydrogen gas but it also on the atmosphere in which hydrogen is sensed. Under equal operating conditions the highest sensitivity for hydrogen is observed in He gas, next in N_2, and the lowest in air.

2 – Preparation of the Films, The House of Cards Structure

The thick oriented films are prepared by the methods of exfoliation: Lithium metal is intercalated between the layers of metal sulfide powder. When immersed in water, the resulting reaction produces hydrogen gas, which expands and separates the powder into single layers in a suspension of pH 12 due to the LiOH formed. At this pH, the single layers are charged negatively due to the OH^- ions on the surface, so the layers repel each other and remain suspended in water for several days. The concentration of the suspension at this stage is about 1g/100ml. The pH of the suspension is lowered immediately after the exfoliation to the pH of the corresponding point of zero charge (e.g. in the case of the MoS_2 to pH=2). Then the material will flocculate in a form in which the basal plans are positive but the edges are negative so that the edges are attracted to the basal plans and form a new structure resembling the house of cards structure. The corresponding pH for WS_2 is found at pH 1 and for TiS_2 is found at pH 2.5. When after decanting the liquor the pH is raised to 3–4 using distilled water, the layers will redisperse, forming a suspension which is stable against flocculation for several weeks. When deposited on a polar material substrate, the layers tend to orient themselves to the plane of the substrate and a highly oriented thick film will form with a thickness depending on the concentration of single layers material in the suspension. With a concentration of about 2 mg/ml the thickness is about 1 micron. The deposited films are dried at room temperature and then sintered in air at 300–350°C.

In conjunction with certain catalysts, the house of cards structure was also found to be an excellent medium for hydrogen storage, being quite competitive in performance with conventional metal hydrides such as $LaNi_5H_6$.

3 – Description of the Drawings

Figure 1. Cross–sectional view of the gas sensor.

Figure 2. Response time of the sensor in air versus operating temperature at a hydrogen concentration of 275 ppm. The response time at 120°C is about 1 minute. At higher temperatures it is less than a minute and at room temperature it is several minutes (not shown in the graph).

Figure 3. Sensitivity of a Mo based gas sensor versus concentration of hydrogen at 120°C in air. The sensor shows linearity in the range of 30 ppm to 10^4 ppm (1%) hydrogen.

Figure 4. Sensitivity of the sensor at different concentrations of hydrogen at room temperature. The sensitivity is linear in the the range of 100 ppm to 5×10^4 ppm (5%).

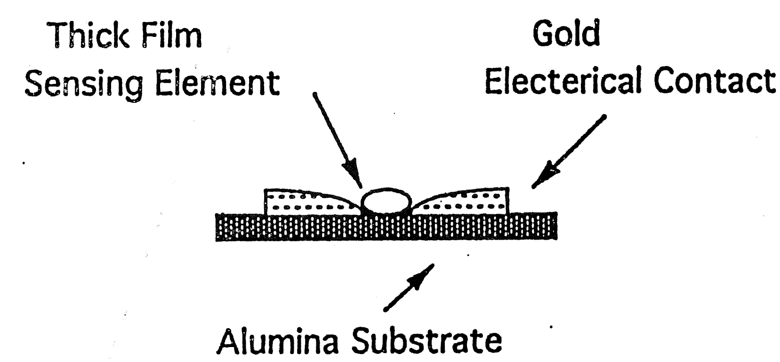
Figure 5. Bar diagram of the response time of the sensor at room temperature in atmospheres He, N_2, and air. The hydrogen concentration tested was 4%. Figure indicates that when the concentration of surface oxygen is reduced by replacing the air with He molecules, the response time is faster and the sensitivity to hydrogen is higher as is shown in figure 7. However the sensor is irreversible in that it must be exposed to oxygen to return to the high resistance base line.

Figure 6. Sensitivity of the sensor at high concentrations of hydrogen at room temperature in N_2 atmosphere.

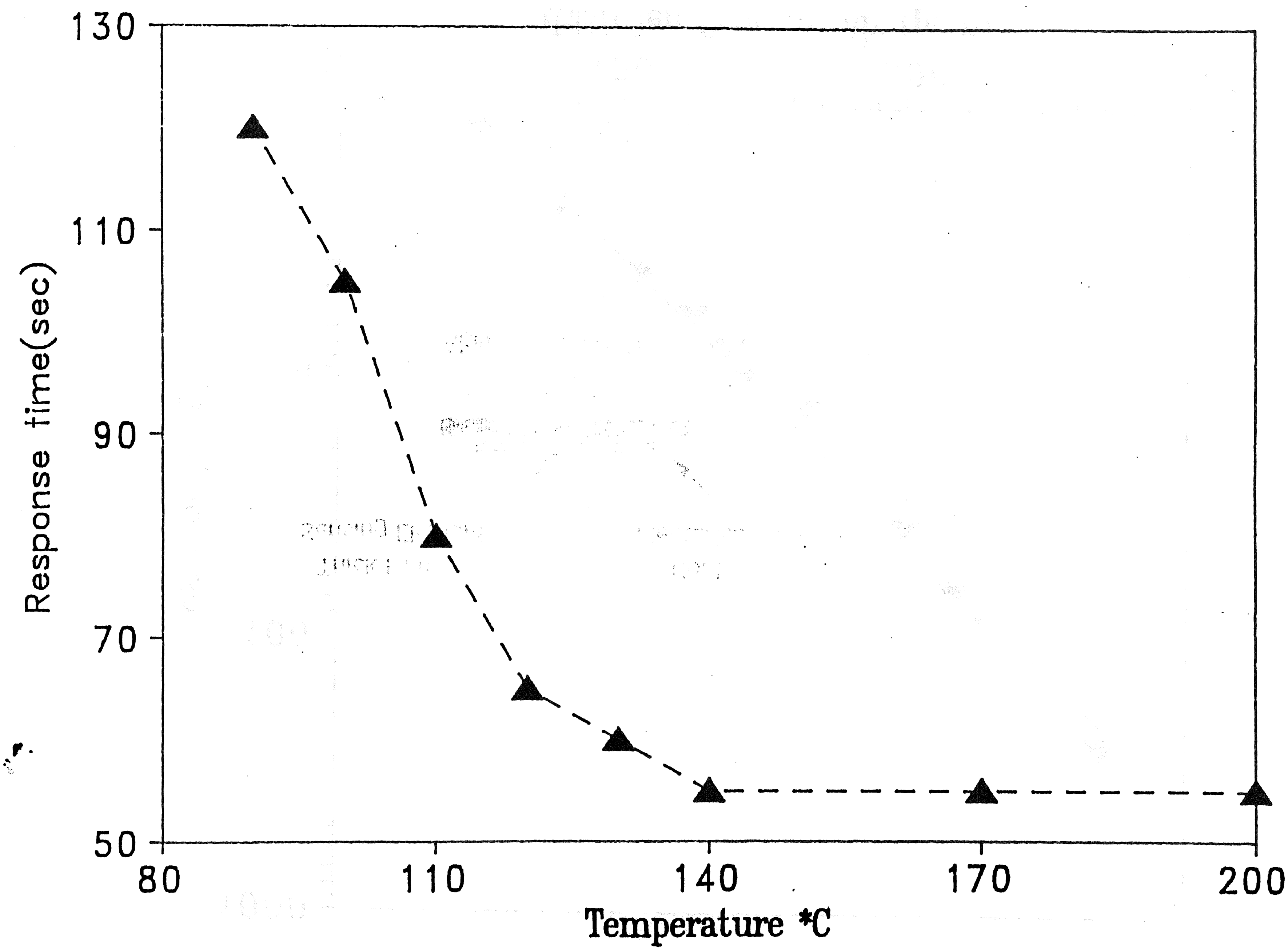
Figure 7. Sensitivity of the sensor for high concentrations of hydrogen at room temperature in the He atmosphere.

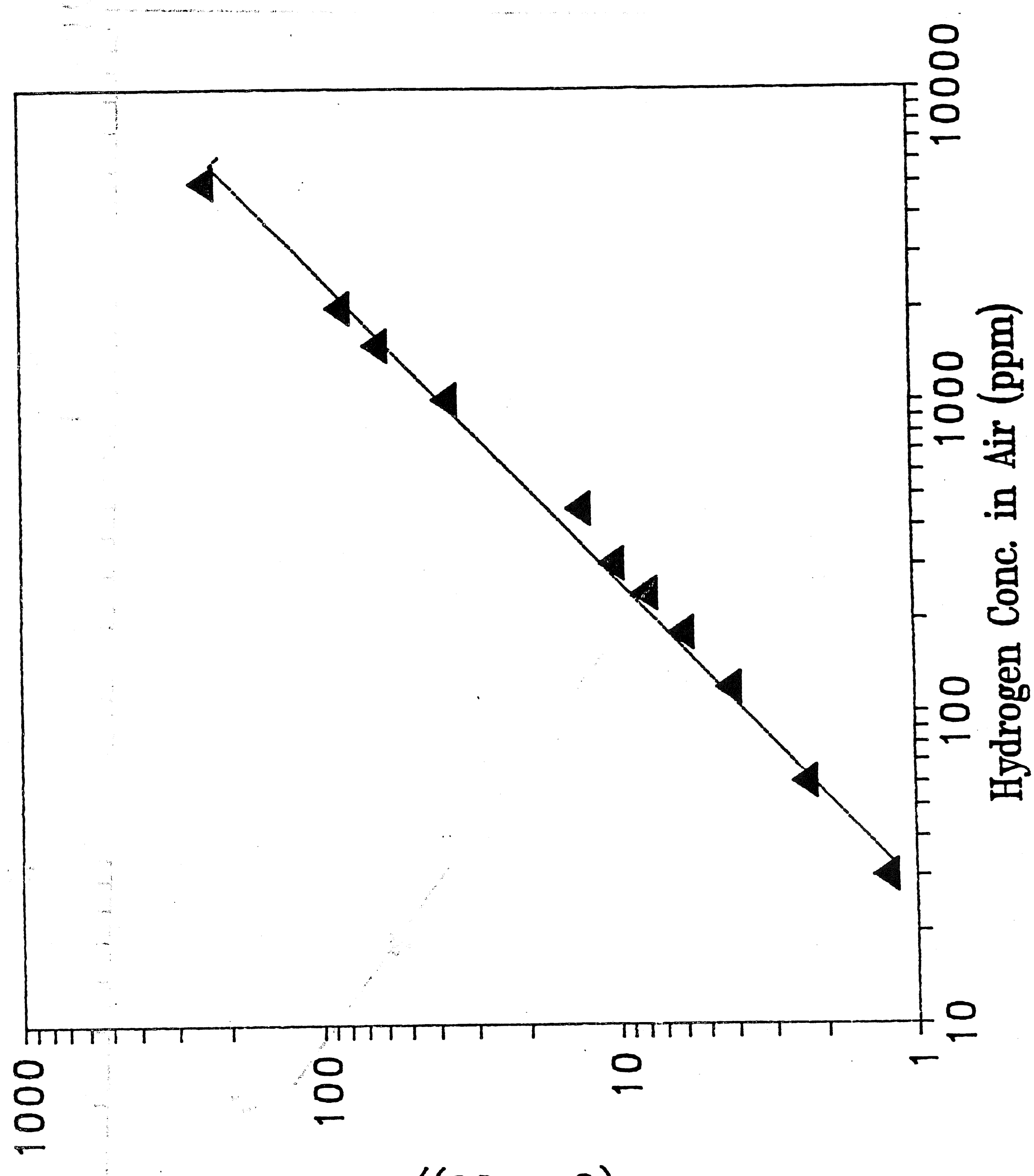
Figure 8. Hydrogen storage desorption curves for the "house of cards structure" compound MoS_2-$Pt_{0.1}H_x$.

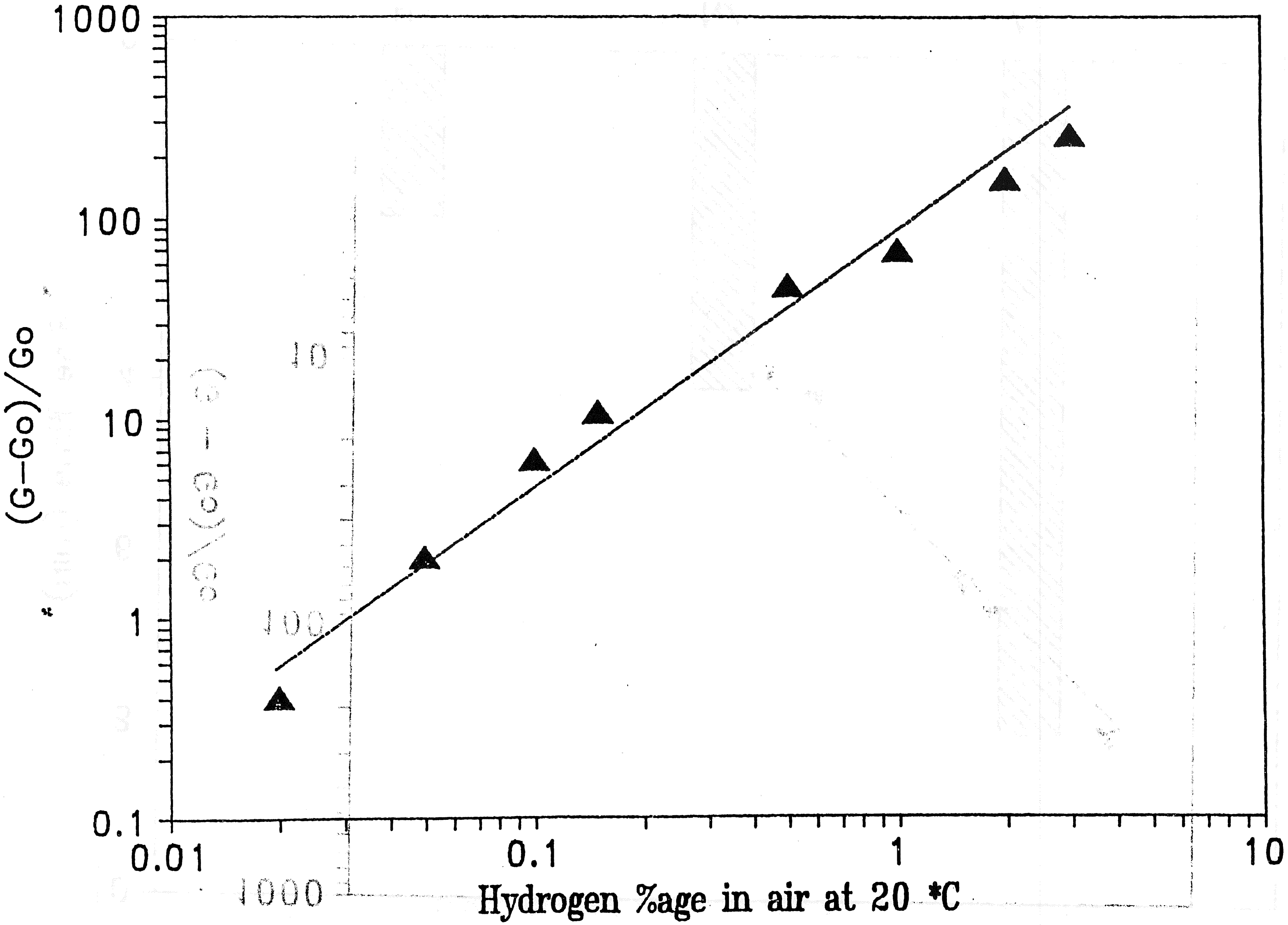
Fig 1



Thick Film
Sensing Element
Gold
Electerical Contact
Alumina Substrate


130
110
90
70
50
Response time(sec)
80
110
140
170
200
Temperature *C


(G − Go)/Go
1000
100
10
1
10
100
1000
10000
Hydrogen Conc. in Air (ppm)



(G–Go)/Go
1000
100
10
1
0.1
0.01
0.1
1
10
Hydrogen %age in air at 20 *C

Fig5

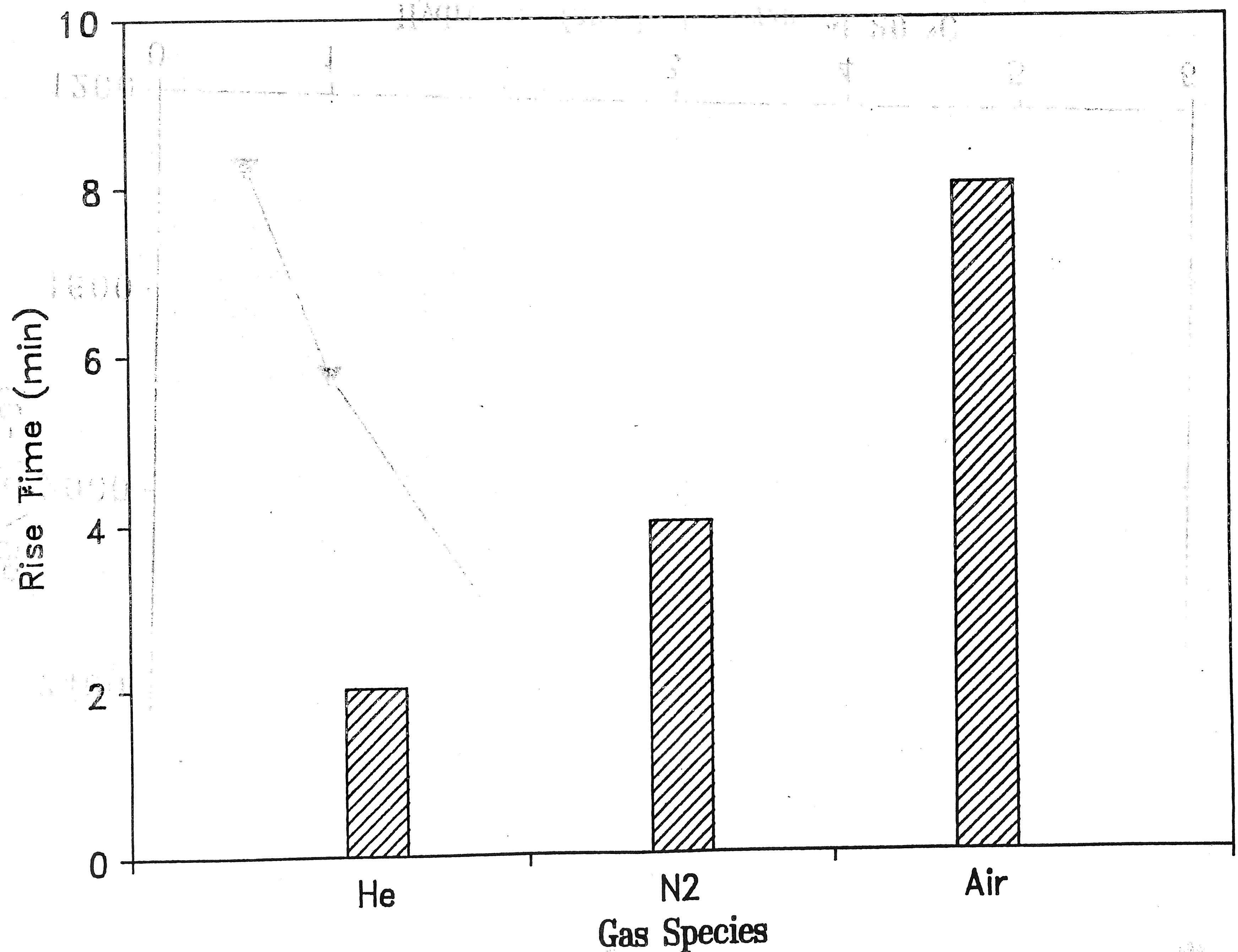
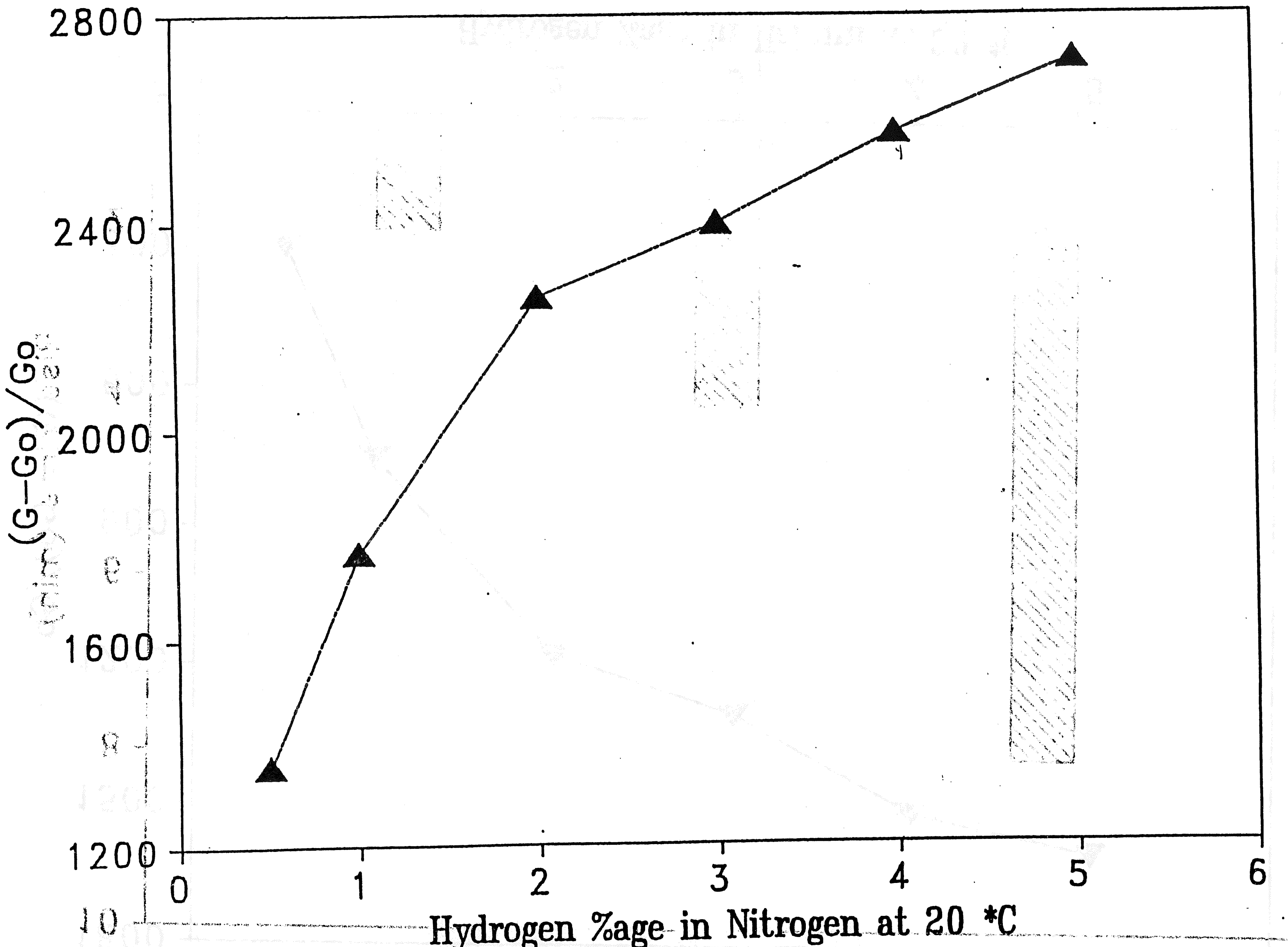
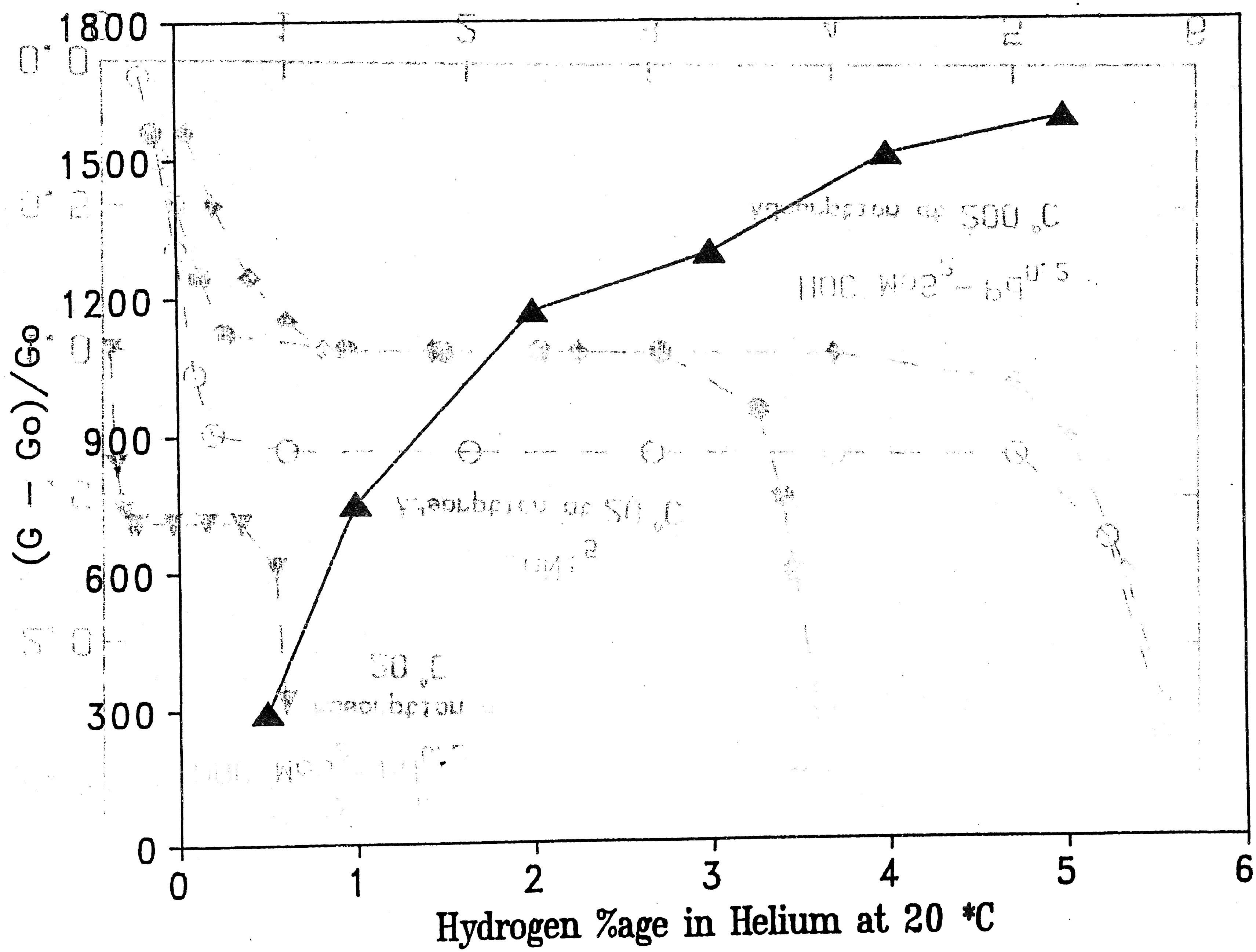


Fig 6

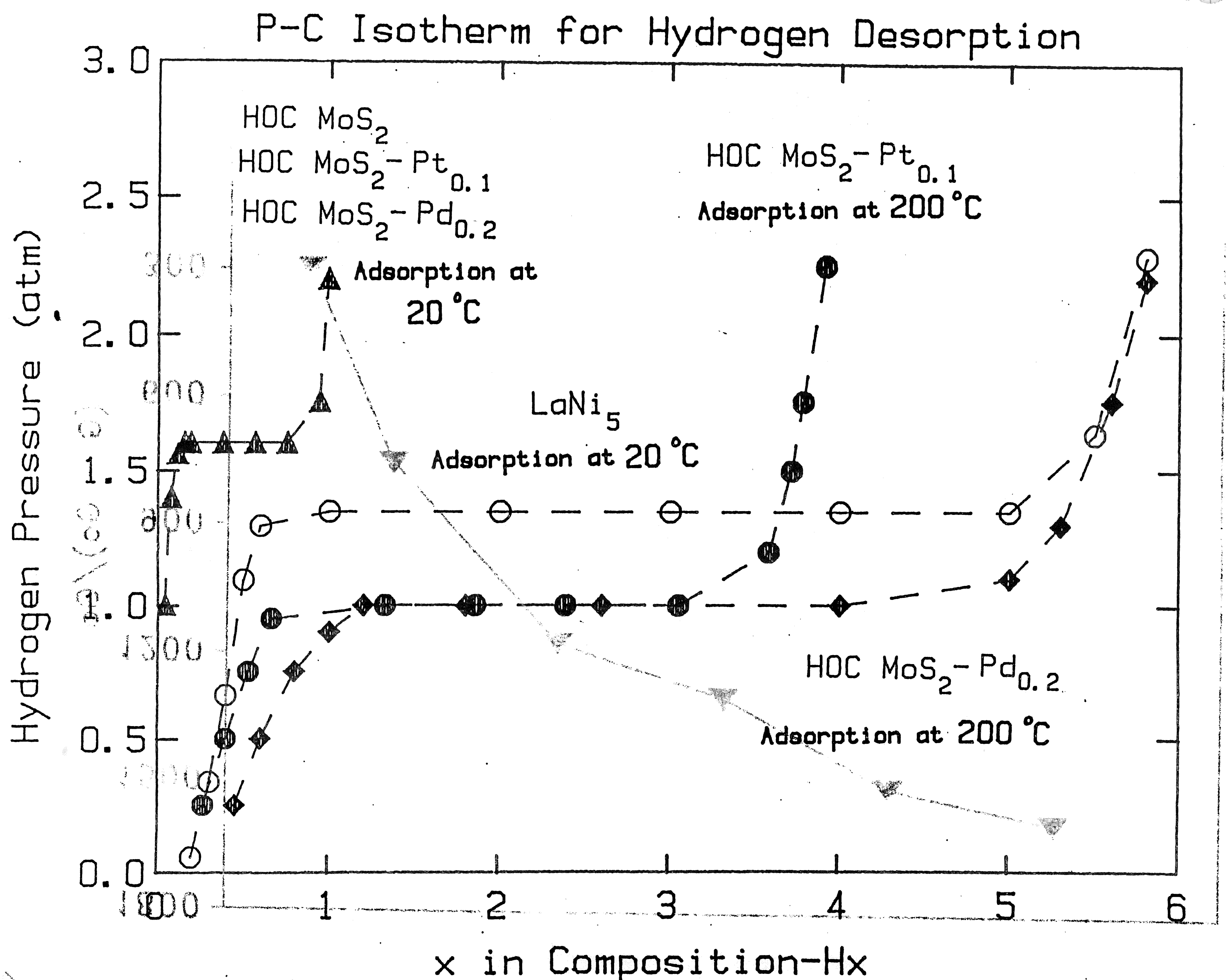


Responsiveness at max Sn

Fig 7



(G − Go)/Go
1800
1500
1200
900
600
300
0
0
1
2
3
4
5
6
Hydrogen %age in Helium at 20 *C


P-C Isotherm for Hydrogen Desorption
HOC MoS2
HOC MoS2-Pt0.1
HOC MoS2-Pd0.2
Adsorption at 20 °C
HOC MoS2-Pt0.1
Adsorption at 200 °C
LaNi5
Adsorption at 20 °C
HOC MoS2-Pd0.2
Adsorption at 200 °C
3.0
2.5
2.0
1.5
1.0
0.5
0.0
Hydrogen Pressure (atm)
0
1
2
3
4
5
6
x in Composition-Hx

H2LD TIM
KSC 1992 ATTENDEES

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