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TITLE: SOME NEW TECHNIQUES IN TRITIUM GAS HANDLING AS APPLIED TO METAL HYDRIDE SYNTHESIS

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***SOME NEW TECHNIQUES IN TRITIUM GAS HANDLING AS APPLIED
TO METAL HYDRIDE SYNTHESIS**

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ABSTRACT

A state-of-the-art tritium Hydriding Synthesis System (HSS) was designed and built to replace the existing system within the Tritium Salt Facility (TSF) at the Los Alamos National Laboratory. This new hydriding system utilizes unique fast-cycling 7.9 mole uranium beds (47.5g of T at 100% loading) and novel gas circulating hydriding furnaces. Tritium system components discussed include fast-cycling uranium beds, circulating gas hydriding furnaces, valves, storage volumes, manifolds, gas transfer pumps, and graphic display and control consoles. Many of the tritium handling and processing techniques incorporated into this system are directly applicable to today's fusion fuel loops.

INTRODUCTION

A state-of-the-art tritium Hydriding Synthesis System (HSS) was designed and built to replace the existing system within the Tritium Salt Facility (TSF) at the Los Alamos National Laboratory¹. This facility was initially activated December 5, 1974, to support the Laboratory's weapons program. The TSF consists of a large glovebox system designed for handling highly reactive metal tritides, in particular Li(D,T), connected to a gas purification system (GPS) for maintaining an inert atmosphere. Both the glovebox and the GPS are interfaced with a detritiation system designed to remove tritium from all effluents prior to release of these effluents to the environment. The existing on-line metal hydriding system contained in the 11 m³ glovebox line will be replaced by a new system called the HSS.

The existing two-manifold system consists of manually operated valves with Kel-F seats, a 0.75 liter volume induction-heated hydriding furnace without loop circulation, two standard volumes totaling six liters, a 2.5 mole uranium bed (15g of T at 100% loading), a display panel showing only valve positions, and a 2.7 liter/s oil fore pump used to evacuate the system (Fig. 1).

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The original oil rotary-vane gas transfer pump was eventually replaced by a five liter/s scroll pump² backed by a metal-bellows compressor³.

Fig. 1. Existing two-manifold synthesis line that will be replaced by the HSS.

Improvements incorporated into the new system are: easily replaceable metal-bellows sealed and pneumatically actuated valves⁴ with vespel seats; two 1.4 liter capacity induction-heated hydriding furnaces with loop circulation, three large gas storage volumes totaling 185 liters, two 7.9 mole uranium beds (47.5g of T at 100% loading), and a mobile control and display console showing complete system parameters (Fig. 2). A five liter/sec scroll pump backed by two metal-bellows compressors and an oil-free molecular drag pump are used for gas transfer and system evacuation.

Fig. 2. Hydriding Synthesis System (HSS) during assembly.

By incorporating the HSS into a glovebox of the same geometry as the original box, tritium releases and removal time will be minimized. Unlike the old system, the glovebox for the HSS was divided into two sections to provide for the additional volume necessary for the larger components. The top, inert section contains the manifolds, U-beds, and valves while the bottom

section provides secondary containment for the standard volumes, hydriding furnaces, pumps, rf-capacitors, and furnace switches. An existing mass-spectrometer⁵, designed for analyzing low masses, that was used with the old system will be retained for isotopic gas analyses.

When appropriate, commercially available components are listed and the reasons why they were selected. This is not to say that these are the only components available that can meet these stringent requirements, however, for these applications they are the ones that have been selected at the Tritium Systems Test Assembly (TSTA) and the Tritium Salt Facility (TSF)⁶.

FAST CYCLING URANIUM BEDS

Uranium beds (U-beds) that permit fast-cycling times, rapid ³He removal, large tritium storage capacities, and a small physical size are desired for today's tritium processing systems. These design features are incorporated into the U-beds of the HSS by utilizing a high input power, a high storage capacity of 7.9 moles/bed, and a 6:1 length-to-diameter ratio that permits a small physical size (Fig. 3). Also, single-pass tritium removal efficiency is improved by a series-flow design that increases tritium contact area and residence time (Fig. 4).

Fig. 3. Fast cycling U-bed showing three of the five uranium cavities.

Fig. 4. Schematic of U-bed series-flow design that increases tritium contact area and residence time.

Long cycling times and possible U-bed overheating can result from vacuum jacketing. U-beds that are used exclusively for shipping tritium must have permeation jackets to contain the tritium that permeates the primary wall; however, U-beds that are used in process loops within gloveboxes that are purged or have detritiation systems would not require jackets. Since these U-beds will be used in a glovebox line that has a detritiation system, tritium permeation was not a major concern; however, these beds are fabricated from type 316 stainless steel which has a lower permeation rate to tritium than type 304L. Leak tight integrity and high reliability from these beds are gained by utilizing an all-welded design.

Each bed is made from 316 solid stainless steel bar stock. Five holes are bored lengthwise through the stainless steel, one through the center and four on a 63.5 mm radius bolt circle. This configuration provides 77.2 cm³ per cavity. Uranium hydrides to a density of 3.4 g/cm³; therefore, each cavity is filled with 262.5 grams of uranium⁷. Quartz wool is used to prevent the larger particles from migrating from one cavity to the other. Once filled, the cavities are seal-welded with cross-connecting end plugs that permit a series flow through the bed. Between the five uranium cavities are eight 300 watt resistance heaters, four heaters at each end. Surrounding the all-welded primary containment vessel are three stainless steel heat-shields. Two manually operated metal-bellows sealed valves are used at the inlet and outlet. The uranium hydride is contained by three parallel-connected removable 50 micron sintered stainless steel filters.

Temperature indication and over-temperature protection are provided by redundant temperature controllers connected in series. To provide fast temperature response times shielded chromel/alumel thermocouples are inserted into thermocouple wells between the heater and uranium cavities. These beds can reach a normal operating temperature of 500⁰C in air within

30 minutes with an external surface temperature of 150⁰C. Overpressurization protection is dramatically increased by the high strength inherent with this type of construction. Cooling is by forced or free convection using glovebox helium.

CIRCULATING GAS HYDRIDING FURNACES

Metal hydriding without loop circulation should be avoided with tritium gas containing concentrations of ³He greater than 2%. During hydriding the ³He concentration will increase and blanket the material, eventually stopping the hydriding reaction. Blanketing can be prevented by using pure gas, which requires ³He removal one or two days prior to hydriding, or by circulating the gas through the furnace. Tritium handling becomes much easier if the latter method is employed.

A novel gas circulating design, that will eliminate ³He blanketing during hydriding, is incorporated into the furnaces of the HSS. Samples to be hydrided are placed into a thin iron crucible that fits into the furnace well. The furnace well is grooved and contains a partition that permits gas to pass from the iron crucible to the outside of the partition where it exits the furnace. At temperatures above 600⁰C, tritium readily passes through the walls of the iron crucible completely exposing the sample surface to tritium. Typical hydriding pressures are from 1 to 2 atmospheres.

The HSS contains two induction-heated hydriding furnaces capable of heating 1.4 liters of material to 850⁰C. The heated furnace well, including the rf-coil, are contained by a jacket that is backfilled with nitrogen and maintained at 0.5 atm. pressure during heating. This pressure is easily maintained and prevents the rf coils from arcing. Tritium that permeates through the hot zone during the hydriding operation is contained by the jacket and evacuated at the end of the run. Unlike the U-beds, these furnaces require jacketing because they extend into the lower uncontaminated section of the glovebox. The upper portion of the well and the outside surface of the furnace is cooled by circulating cooling water. Water is not permitted in the upper inert section of the box. Furnace power is supplied by a locally controlled, 50 kw induction heating unit located in the basement of the building.

VALVES

Tritium containment requires valves with leak rates of 10⁻⁹ cm³/s or less. Three valve designs were evaluated by testing seven to nine valves of each design. The three valve types used were: (1) Hoke 0361G4Y (50.8 mm dia. operator); (2) Nupro 4BK-93NC (101.6 mm dia. operator); and (3) Nupro 4BG-93NC (same as (2), except with stellite stems). Stem tips and bellows gaskets of valve types one and two were changed from the standard Kel-F material to polyimide prior to testing. For valve type (3) only the stem tip had to be changed; the bellows

gasket is metal. Our experience with tritium has shown that polyimide is far superior to Kel-F for this application.

The testing sequence included cycling each valve 2500 to 10,000 times using unfiltered room air, followed by envelope and seat leak testing. This test is a severe test of valve reliability because the HSS process gas is filtered.

All of the seven polyimide-stemmed Nupro valves tested passed the final leak test of 10^{-9} cm³/s after cycling each 4,000 to 10,000 times. After 4,000 cycles four out of the eight stellite-stem Nupro valves failed. The remaining four had cross-seat leak rates comparable to the polyimide-stem valves. Visual inspection of the failed valves showed a crystalline appearance of the stellite stems and corresponding galling of the mating stainless steel seats. All of the polyimide-stem Hoke valves failed the final leak test; some were wide-open after the test. As controls on the polyimide modification, two Hoke valves with Kel-F stem tips and body gaskets were tested as received. After 4,000 cycles one was wide-open and the other had a leak rate of 10^{-7} cm³/s. No bellows leaks were detected in any of the valves tested. None of the air operators failed during the tests, although the minimum required opening pressure increased slightly as the Nupro valves wore (Fig. 5).

Fig. 5. Valve test results.

Based on this test, the Nupro 4BK-93NC valves with polyimide plastic stems were selected. After consulting with the factory and showing them the results of the test, they thought the ideal valve to use would be their 4BG type valve reconfigured with polyimide stems and tube extension welded to Cajon SS-4-VCR-3 glands with captured Cajon SS-4-VCR-1 gland nuts. The bellows seal on the 4BG type is metal; therefore this seal would not have to be changed. After these factory modifications were made, the model number of this tritium compatible valve became SS-4BG-U51-VPL-93NC.

Valves of this type were ordered for the HSS with 3-way, 24 vdc miniature solenoid actuator control valves⁹. To insure adequate pressure and volume, helium is supplied to the

valve actuator manifold using a 4.0 liter reservoir. Copper lines supply helium from the solenoid valves to the actuators. Copper was chosen over plastic because plastic would eventually harden and crack due to the radiation exposure and the extreme dryness (<0.5 ppm H₂O) of the glovebox atmosphere. Since helium is used in the inert upper section of the HSS, the actuators are vented directly to the glovebox.

STORAGE VOLUMES

Three cylindrical storage volumes 10, 25, and 150 liters, fabricated from 316 stainless steel, are used in the HSS. These volumes are contained in the lower section of the box and stacked one upon the other to save space. Gas can enter or exit from the ends of each volume by Cajon SS-8-VCR-9 elbows that are welded into the ends.

Pressure and temperature indication is provided for each volume. Pressure transducers with digital outputs are used for absolute pressures¹¹, while compound mechanical gauges provide quick pressure indications. Temperature indication is from Type K (chromel/alumel) thermocouples. Gas temperature accuracy is increased by using thermocouple wells that are welded into the center of each volume to a depth of the volume radius.

MANIFOLDS

Gas paths and component flexibility are improved over the old system by utilizing a three-manifold design. To insure sufficient flow and to minimize manifold volumes, manifolds are fabricated from 6.35 mm diameter 316 stainless steel tubing, connected by welded tube fittings of the zero-clearance, coined-gasket type⁸. By keeping valve spacing to a minimum, manifold volumes are proportionally minimized.

Valve assemblies are fastened to a common mounting plate by using two positioning pins and a wing-nut. The positioning pins are screwed into the mounting plate which was drilled and tapped by a numerical controlled machine to insure accurate pin alignment. Horizontally slotted angle plates fastened to each valve provides accurate valve alignment (Fig. 6). Once the glovebox becomes tritium contaminated this technique permits rapid valve removal and reconnection through the box gloves. If low stack releases are to be maintained by tritium processing facilities, glovebox window removal must be kept to a minimum.

Fig. 6. Valve assembly showing slotted angle plate with positioning pins.

GAS TRANSFER PUMPS

The gas transfer pumps used in the HSS are dry, hermetically sealed, and packless. Two metal-bellows compressors containing no fluorocarbons in the pump chamber are used to transfer gas. For evacuation, these pumps are supplemented by a metal-bellows sealed scroll pump connected in series. Gas will pass through the scroll pump with very little resistance to flow when the scroll pump is off, but filters must be used on these pumps due to the very close tolerances in the orbiting scroll geometry. The advantage of these pumps is that they do not contaminate the system with oil or mercury vapor that can poison the U-beds.

A 7.5 liter/s molecular drag pump¹² will replace the existing oil fore pump. The molecular drag pump has greased lubricated bearings; therefore, is not completely oil-free but is much superior to an oil fore pump for this application.

GRAPHIC DISPLAY AND CONTROL

The HSS is monitored and controlled by a graphic display and control console (Fig. 7). The console contains redundant U-bed temperature controllers; transfer pump controls; and digital temperature and pressure indication for the manifolds, standard volumes, U-beds, and hydriding furnaces. Furnace power controls will be added to the lower section of the console. At the top of the console is a schematic flow diagram with toggle switches representing the valves in the HSS. Each toggle switch has a LED that indicates green or red when the valve is

open or closed. LED indication is from valve stem position micro switches not actuator voltages. System safety is augmented by utilizing locking toggle switches for the process control valves and by utilizing manually-operated valves to control evacuation and supply gas.

Fig. 7. Graphic display and control console.

CONCLUSIONS

System reliability involving uranium beds or other metal hydrides for tritium containment and purification is an important concern in fusion energy programs. U-bed contaminants, such as oil or mercury, should be and can be eliminated from tritium processing streams by the use of oil-free gas compressors and pumps. The most often over-looked design consideration affecting the performance of these systems is the lack of an independent gas circulation path to remove ^3He . If these systems are to provide a high degree of readiness and efficiency, gas circulation must be incorporated into the design.

The design and installation of tritium containment valves are very important to the fusion technology data base. Valves used for tritium containment should be metal-bellows sealed and polyimide-stemmed with cross-seat leak rates of 10^{-9} cm^3/s or less. Maintenance costs, down time, and tritium releases can be minimized by incorporating the positioning-pin technique to mount large arrays of tritium control valves in large tritium processing systems.

The majority of high inventory tritium processing facilities utilize uranium hydride to store tritium. To ensure good valve reliability, efficient filtration is required; however, the performance of these filters will deteriorate in time. Therefore, replaceable filters should be incorporated into the process piping at these facilities to ensure that adequate gas flows can be maintained at design pressures.

ACKNOWLEDGEMENTS

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REFERENCES

1. J. E. Nasise, "Performance and Improvements of the Tritium Handling Facility at the Los Alamos Scientific Laboratory," American Nuclear Society Topical Meeting on Tritium Technology in Fission, Fusion, and Isotopic Applications, Dayton, OH, April 29-May 1, 1980, CONF-800427 (US DOE, Washington, DC, 1980).
2. Scroll pump, Normetex America, Inc., 2 University Plaza, Hackensack, NJ 07601.
3. Metal-bellows compressor, Metal Bellows Corp., 1075 Providence Highway, Sharon, MA 02067.
4. B series valves, Nupro Co., 4800 East 345th St., Willoughby, OH 44049.
5. Mass spectrometer, Nuclide Inc., 642 East College Av., State College, PA 16801.
6. J. L. Anderson, D. O. Coffin, J. E. Nasise, R. H. Sherman, and R. A. Jalbert, "Some Tips on Tritium Technology", American Nuclear Society Second National Meeting on Tritium Technology in Fission, Fusion and Isotopic Applications, Dayton, OH, April 30 to May 2, 1985, CONF-07481896 (US DOE, Washington, DC, 1980).
7. J. J. Katz and E. Rabinowitch, "The Chemistry of Uranium", p.200, McGraw-Hill Book Company, Inc., New York, 1961.
8. VCR fittings, Cajon Co., 9760 Shepard Road, Macedonia, OH 44056.
9. Skinner Valve Division, Honeywell Inc., 100 Edgewood Ave., P.O. Box 1450, New Britain, CT 06050.
10. Hoke Inc., One Tenakill Park, Cresskill, NJ 07626
11. MKS Instruments, Inc., Six Shattuck Road, Andover, MA 01810.
12. Alcatel Vacuum Products, 40 Pond Park Road South Shore Park, Hingham, MA 02043.

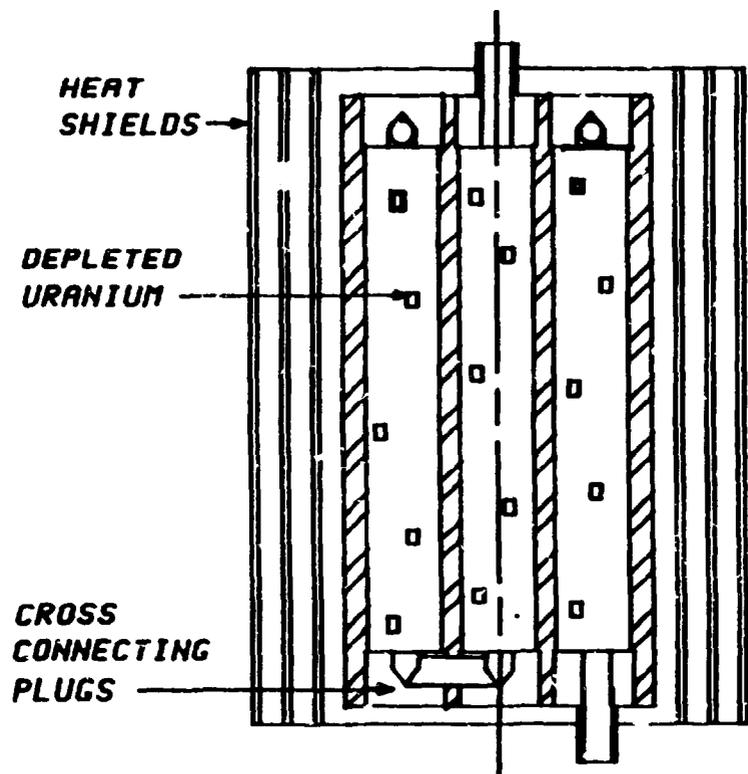


Fig. 3

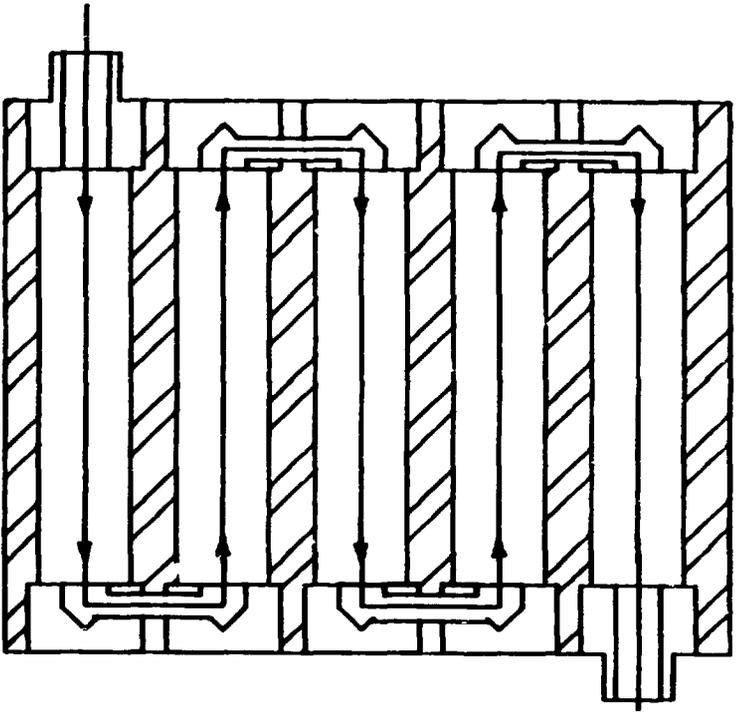
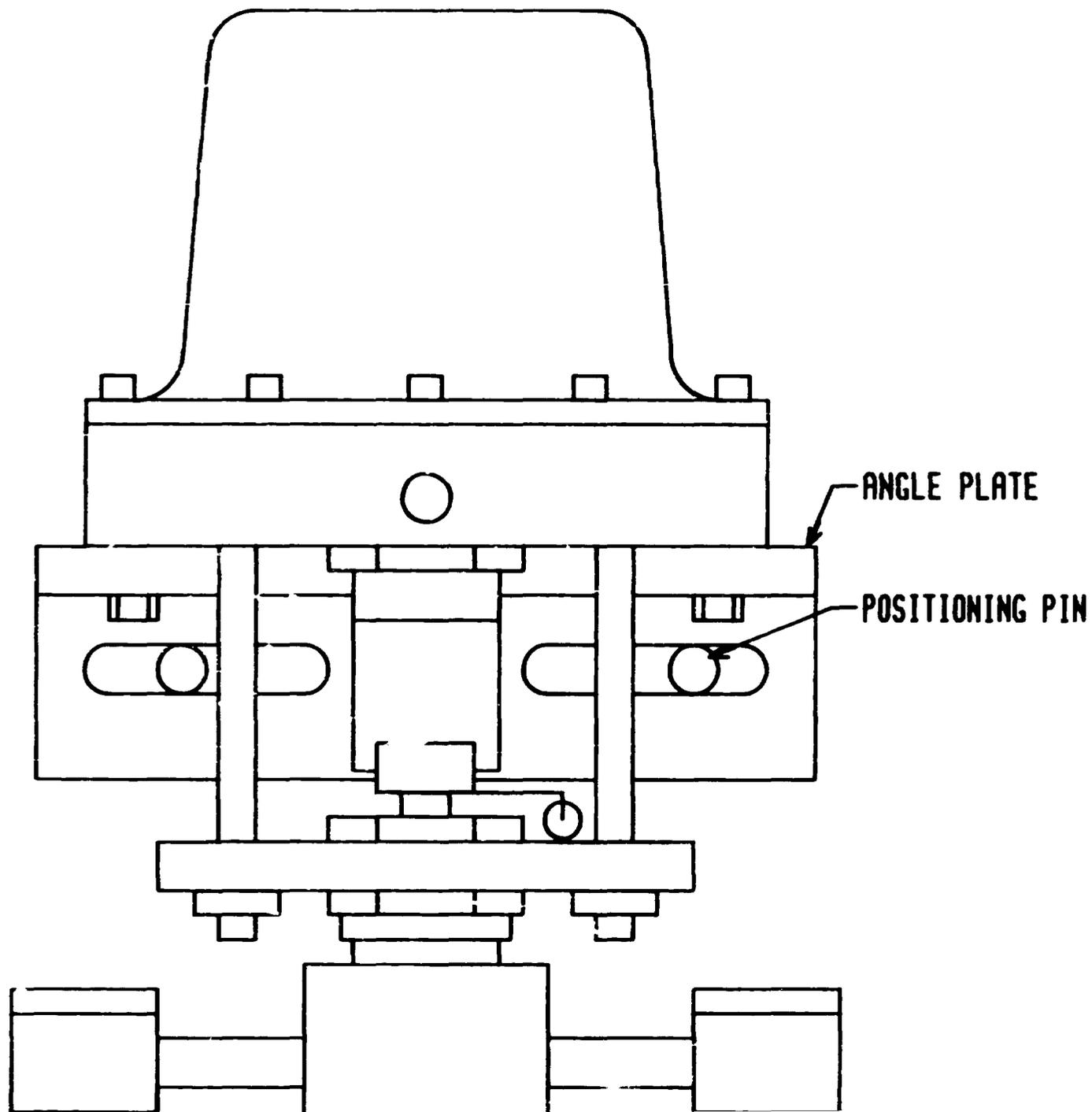


Fig. 4

VALVE TYPE	# OF VALVE TESTED	RESULTS AFTER 4000 CYCLES	RESULTS AFTER 10,000 CYCLES
(#1) Hoke # 0361F4Y Polyimide-stemmed	5	Failed-Leak test	
(#2) Nupro 48K-930K Polymide-stemmed	7	Passed-Leak test	Passed-Leak test
(#3) Nupro 48K-93NC Stellite-stemmed	8	Four failed test <u>Four passed leak test</u>	<u>Four passed leak test</u>
Hoke #036164Y Kel-F stemmed (control-standard)	2	One failed leak test <u>One failed leak test (10^{-7} cm³/s)</u>	

Fig. 5

Fig 6



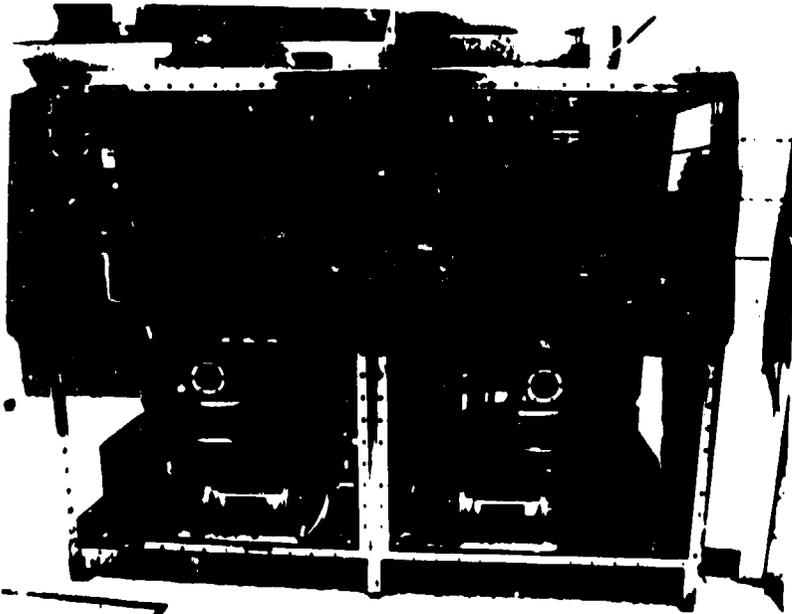


FIG. 2

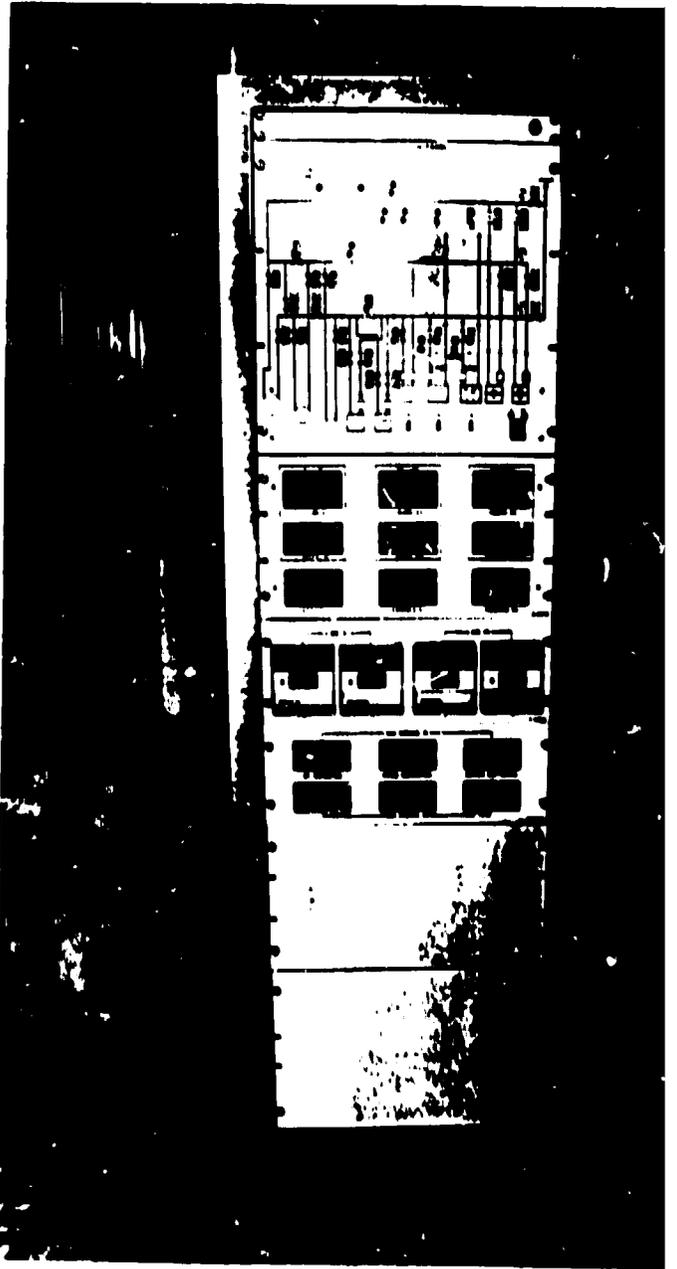


FIG. 7



FIG. 1