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QUARTERLY PROGRESS REPORT, COMPATIBILITY OF COLUMBIUM WITH HYDROGEN ENVIRONMENTS

(1 JULY 1965 TO 30 SEPTEMBER 1965)

by

R. J. Walter and J. A. Ytterhus

Oct 1965

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Air Force Materials Laboratory
Research and Technology Division
Air Force Systems Command
Wright-Patterson AFB, Ohio 45433

Prepared Under Contract AF33(615)-2854

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Research Department of Rocketdyne
A Division of North American Aviation, Inc.
Canoga Park, California

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FOREWORD

This progress report was prepared by the Rocketdyne Research Department under G.O. 8724 in compliance with Part I, Para. B2 of Contract AF33(615)-2854 and covers the period 1 July to 30 September 1965. The work was sponsored by the AFSC Research and Technology Division, Wright-Patterson Air Force Base, Ohio, with Lt. L. D. Blackburn acting as Project Engineer.

ABSTRACT

The first-quarter results for a program to study the compatibility of columbium with hydrogen environments are presented. An environmental tensile testing machine for testing of columbium at high temperatures and high hydrogen pressures has been ordered. The apparatus necessary for the measurement of hydrogen solubility and rates of absorption and desorption is nearly completed. Auxiliary devices have been designed and built. The hydrogen/water vapor experimental apparatus has been completed and checked out for a hydrogen/water vapor ratio of 1.

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INTRODUCTION

Columbium and columbium alloys have certain characteristics which make them quite attractive for use in such applications as advanced, regeneratively cooled rocket engine thrust chambers and ramjets which involve high heat fluxes and high temperatures. However, it has been established that under certain conditions, columbium and its alloys are susceptible to embrittlement and deterioration in the presence of hydrogen and hydrogen/water vapor mixtures. Because these environments will exist in the applications indicated above, it is necessary to evaluate the seriousness of this problem under the appropriate conditions.

There are published data showing that columbium is susceptible to hydrogen embrittlement at least below a temperature of 200 F. Recent work (Ref. 1) indicates that, under some conditions, embrittlement of columbium by hydrogen can occur at temperatures as high as 800 F. A program in progress at Rocketdyne indicates that a columbium alloy, B-66, also suffers loss of ductility from hydrogen at temperatures up to 800 F. There are only very limited data on the solubility and rate of hydrogen absorption into columbium at high hydrogen pressures. There are no data of the effect on the mechanical properties of columbium when exposed to hydrogen/water vapor mixtures.

The objective of this experimental program is to study three primary areas relevant to this problem. These are: (1) the effect of hydrogen at high temperatures and pressures on mechanical properties, (2) the solubility and rates of absorption and desorption of hydrogen at high temperatures and high hydrogen pressures, and (3) the effect of hydrogen/water vapor mixtures on the mechanical properties. These studies are being conducted on both columbium and a columbium alloy, and will allow the accumulation of necessary information for the proper assessment of compatibility under actual rocket engine and ramjet environments.

EXPERIMENTAL PROGRAM

MATERIALS

The materials used in this program are electron-beam melted columbium and B-66 (Cb-5V-5Mo-1Zr). The procurement status of the materials is shown in Table 1. The chemical composition and fabrication history of the 0.030-inch-thick B-66 sheet are listed in Table 2. Chemistry and fabrication data are not as yet available for the other materials.

METHODS

Compatibility With Hydrogen/Water Vapor Environments

The program is designed to determine the compatibility of columbium and the B-66 columbium alloy with hydrogen/steam environments under conditions simulating, except for pressure, those to which materials would be exposed in hydrogen/oxygen engines. Thus, the mechanical properties of the metals and the oxide formation and hydrogen and oxygen absorption into the metals will be determined in hydrogen/steam atmospheres. Two mixture ratios of H_2/H_2O, 1/3 and 1, corresponding to the two limits currently used in hydrogen/oxygen engine operation, will be employed in these tests. The mechanical tests will be conducted in a Marshall tube furnace with a 60,000-pound Baldwin tensile testing machine.

These tests will be performed using approximately the same test sequence previously used (Ref. 2) to ascertain the compatibility of columbium and tantalum with hydrogen at 1 atmosphere of pressure. The specimen will be heated to the test temperature in purified argon and the hydrogen/water vapor introduced after the temperature is stabilized. Following introduction of the hydrogen/water vapor atmosphere, a stress equal to 50 percent of the room-temperature yield strength of the specimens is applied. This stress will be maintained for 30 minutes,

TABLE 1

TEST SPECIMEN PROCUREMENT STATUS

Material	Supplier	Form	Present Status		
			Scheduled Shipment, 1965	Received At Rocketdyne	Test Specimen Preparation
B-66 Alloy Electron-Beam Melted	Westinghouse	0.030-inch sheet	---	Yes	Complete
	Westinghouse	1/2-inch-diameter rod	10-14	No	
	Westinghouse	3/4-inch-diameter rod	10-14	No	
Pure Columbium Electron-Beam Melted	Fansteel	0.030 sheet		Yes	In Process
	Fansteel	1/2-inch-diameter rod		Yes	In Process
	Fansteel	3/4-inch-diameter rod		Yes	In Process

TABLE 2

CHEMICAL COMPOSITION AND FABRICATION HISTORY

(B-66, 0.030-inch-thick sheet)

Element	Chemical Analysis
Mo	5.03 percent
V	4.76 percent
Zr	0.89 percent
O	165 ppm
N	74 ppm
C	80 ppm
Cb	Remainder

Fabrication History, steps

1. Double electron-beam melted and vacuum-arc remelted into 8-inch-diameter ingot
2. Extruded to 3- by 6-inch bar
3. Annealed at 1650 C
4. Forged to 1-1/2- by 9- by 18-1/2-inch slab
5. Annealed at 1550 C
6. Forged to 1- by 12- by 21-inch plate
7. Vacuum annealed at 1375 C
8. Warm rolled to 0.135-inch-thick sheet
9. Vacuum annealed at 1375 C
10. Warm rolled to 0.050-inch-thick sheet
11. Vacuum annealed at 1375 C
12. Cold rolled to 0.030-inch-thick sheet
13. Stress relieved at 985 C for 1 hour

and the specimen then tested to failure at a strain rate of 0.0004 in./min. This test sequence was designed to simulate the conditions which are judged to result in maximum gas reaction and absorption that would occur in a regeneratively cooled rocket engine nozzle operation.

The mechanical tests will be performed at 400 F and up to the highest temperature at which brittle failure occurs or the temperatures at which the materials disintegrate as the result of corrosion. The tests will be limited to a temperature of 1500 F, because of the expected rapid rate of oxidation above this temperature, and also because this temperature is considerably higher than the maximum temperature at which hydrogen at 1-atmosphere pressure would affect the mechanical properties. A lower temperature limit of 400 F is imposed by the difficulty of controlling the furnace temperature below this limit and the possibility of condensing the water vapor in cooler regions of the furnace.

The fractured specimens will be thoroughly analyzed for hydrogen and oxygen pickup and metallographically examined to locate the cause of fracture, the degree of corrosion, and the presence of oxide and hydride particles in the metals. The change of weight during corrosion would not be particularly meaningful because the grip contacts would probably prevent significant corrosion outside the reduced section.

Sheet specimens, stress-relieved but not recrystallized, will be used in this program. It is realized that corrosion may be dependent upon grain size. However, stress-relieved sheet is more representative of probable usage, and the effect of grain size would be reserved for a later program. The specimens will be 4 inches long with the dimensions of the reduced section being 1-1/8 by 1/4 by 0.030 inches. Each specimen is machined so that the longitudinal axis of the specimen corresponds to the rolling direction of the sheet.

Solubility and Rates of Absorption and Desorption of Hydrogen

The parameters to be measured are the solubility, rate of absorption, and rate of desorption of hydrogen in columbium and B-66 columbium alloy. Equilibrium solubility of hydrogen in these metals will be determined over the temperature range of 600 to 1900 F at pressures of 800 and 1500 psi.

The procedure to be employed will involve exposing a cylindrical specimen to a fixed temperature and hydrogen pressure for a sufficient time to achieve equilibrium solubility. The hydrogen will then be driven off, combined with oxygen, collected as water, and weighed. A high-pressure vessel inserted into a tube furnace will serve as the specimen chamber. The chamber will be lined with gold to minimize errors caused by diffusion and/or absorption loss of hydrogen. It will have a hydrogen inlet equipped with a sensitive pressure gage for maintaining a fixed hydrogen pressure and an outlet port leading to the hydrogen analyzer apparatus. Calibration will be accomplished by inserting a tungsten cylinder of the same dimensions as the columbium specimen and subjecting it to the same temperature and hydrogen pressure to be used in the test. The high-pressure source will be valved off, and the valve leading to the hydrogen analyzer apparatus will be opened slowly. The temperature will be raised to 1900 F and the vessel flushed with inert gas. Because tungsten is essentially impervious to hydrogen under these conditions, calibration will determine the quantity of hydrogen present in the void volume in the vessel.

For the test of hydrogen solubility in columbium, a columbium specimen will be inserted into the high-pressure vessel, and exactly the same procedure as described above will be used. The additional hydrogen collected will be approximately the amount absorbed by the columbium.

A correction must be applied to account for the difference in thermal expansion of the tungsten and the columbium specimen. A separate experiment will be performed to measure the thermal expansion of the hydrided columbium.

The rate of hydrogen absorption will be measured over the temperature range of 600 to 1900 F at pressures of 800 and 1500 psi. The absorption rate will be determined by admitting measured quantities of hydrogen at frequent time intervals to a columbium specimen at a constant temperature and essentially constant hydrogen pressure. A high-pressure vessel at room temperature will serve as a hydrogen reservoir and will be connected by a valved tube to another high-pressure vessel serving as the specimen container. This second vessel will be gold lined and inserted in a tube furnace. The system consisting of both vessels, each with its own sensitive pressure gage, will first be evacuated, and then the specimen container brought up to the desired temperature. The vessels will be separated by closing the valve between them and hydrogen admitted to the reservoir at a predetermined pressure. At time t_0, the valve separating the vessels will be opened until the pressure in both vessels is the same (P_0) and then quickly closed. The absorption of hydrogen by columbium will be allowed to proceed for a limited time, during which the pressure in the specimen-containing vessel will decrease slightly to P_0'. The pressure in the reservoir will be brought up to a level above P_0 such that when the valve separating the vessels is opened, the equilibrium pressure will again be P_0. This procedure will be repeated until equilibrium is attained, as indicated by no pressure drop with time in the specimen-containing vessel. By knowing the volume of the reservoir and the reservoir pressure before and after each opening of the valve separating the vessels, the quantity of hydrogen admitted each time may be calculated. The data will then yield the rate of hydrogen absorption at a given temperature and over a small pressure range from P_0 to P_0'. By using a specimen size which is small relative to the volume of the specimen-containing vessel, the expansion of columbium caused by hydrogen absorption makes a negligible contribution to the pressure measurements and may be ignored.

The rate of hydrogen desorption will be measured over the temperature range of 600 to 1900 F at a pressure of 1 atmosphere. A columbium specimen in a gold-lined pressure vessel will be exposed to hydrogen at a given temperature and pressure. The vessel and a flowmeter are isolated from one another by a valve equipped with a bypass. When equilibrium is attained, the bypass is opened, and when the pressure has decreased to nearly 1 atmosphere, the hydrogen gas flow is directed to the flowmeter. Frequent readings of the total hydrogen gas evolved vs time will yield desorption rates at 1 atmosphere of hydrogen pressure. Desorption will be allowed to continue until no further hydrogen gas is evolved from the specimens.

Mechanical Testing at High Pressures

Mechanical tests will be performed to determine the ductile-to-brittle transition behavior of columbium and the B-66 columbium alloy in high-pressure hydrogen. Pressures of 800 and 1500 psi will be used. The tests will be performed at 900 F and up to the highest temperature at which embrittlement is detected. The actual test temperatures will be selected for the purpose of determining as efficiently as possible the temperature range over which the transition from ductile-to-brittle behavior occurs. The strain rate to be used will be approximately the same as the slow strain rate (0.002 in./min Instron cross-head speed) used for tests on hydrogen-charged specimens tested in air, to have direct correlation of data.

It is important the equilibrium hydrogen content be obtained so that the hydrogen content present in the specimen during the test can be predicted from the equilibrium solubility measurements that will be made in another part of the proposed program. Therefore, prior to testing, the specimens will be thermally cycled in hydrogen and held at

a temperature and pressure for a period of time which the absorption measurements would predict to be adequate for obtaining equilibrium hydrogen contents. When the specimen temperature reaches approximately 800 to 900 F, the vessel will be pressurized with hydrogen to the pressure to be used during the tensile tests. Exposing the specimens to high-pressure hydrogen at temperatures below the test temperature may increase the hydrogen content in the specimen. However, because hydrogen evolution has been found to occur readily above 900 to 1000 F, the final hydrogen content should correspond to the equilibrium solubility for the test conditions.

Determination of Reversibility. Reversibility of the hydrogen embrittlement mechanism will be tested by following the same procedure for tensile testing in high-pressure hydrogen, with the exception that when 50 percent of the yield point is reached, this stress will be held for 1/2 hour. The stress will then be removed, the specimen outgassed in helium at about 1500 F, and tensile tested at room temperature. The temperature and pressure conditions of exposure will be such as to effect embrittlement if the specimen were tensile stressed to failure. If exposure to hydrogen at high pressures permanently damages the material, this test would certainly indicate the damage. Following failure, the specimens would be examined as discussed below for specimens tested at high pressures.

Posttest Examination. Examination after testing includes optical microscopy on some of the specimens that failed in a brittle manner. Interstitial chemical analysis will be performed to determine the hydrogen content and the pickup of other interstitials during testing. The actual hydrogen concentration present during the tensile test will not be present when the specimen is removed from the vessel because hydrogen evolution would occur immediately when the pressure is reduced. On the other hand, if the apparatus is allowed to cool to room temperature in a hydrogen atmosphere, the material would become completely hydrided. However, the minimum hydrogen content present during tensile testing can be

estimated by replacing the hydrogen gas with an inert gas immediately after tensile testing and removing the specimen from the vessel before appreciable hydrogen evolution can occur.

Tensile Specimen Design. The tensile specimens will be cylindrical and of the button-head type with reduced section dimensions 1/4 inch in diameter and 1-1/8 inches long.

Test Apparatus. Tensile tests will be performed on specimens in a pressure vessel. One end of the specimens will be attached firmly to the vessel, while the other will be attached to a pull bar which passes through a sliding seal. The vessel will be pressurized with hydrogen, and the tensile specimens will be heated by a tungsten heater.

It is necessary that contaminants in the hydrogen gas be maintained at as low a level as possible so that impurity pickup during the tensile testing does not affect the results of the tests. Also, surface contamination can slow down the rate of hydrogen absorption appreciably. Hydrogen purification will be accomplished by passing the hydrogen through an Engelhard De-oxo unit and then through a molecular sieve trap at boiling nitrogen temperature. The De-oxo unit will remove most of the oxygen impurities present, and the Linde sieve at boiling nitrogen temperature is a standard method of reducing all gaseous impurities down to a total of a few parts per billion (Ref. 3).

Sketches of the apparatus and facility both developed and built with company funds to be used for mechanical testing are shown in Fig. 1 and 2. The test rig consists of three parts. The upper portion is a water-cooled pressure vessel which contains the tensile specimen and heater. The tensile specimen is held firmly to the top of the vessel and is stressed by means of a pull rod extending through the vessel at the bottom.

The lower part of the apparatus contains a load cell which measures the load on the tensile specimens. The pull rod is unrestricted between the specimen and the load cell, and thus the load cell measures the exact

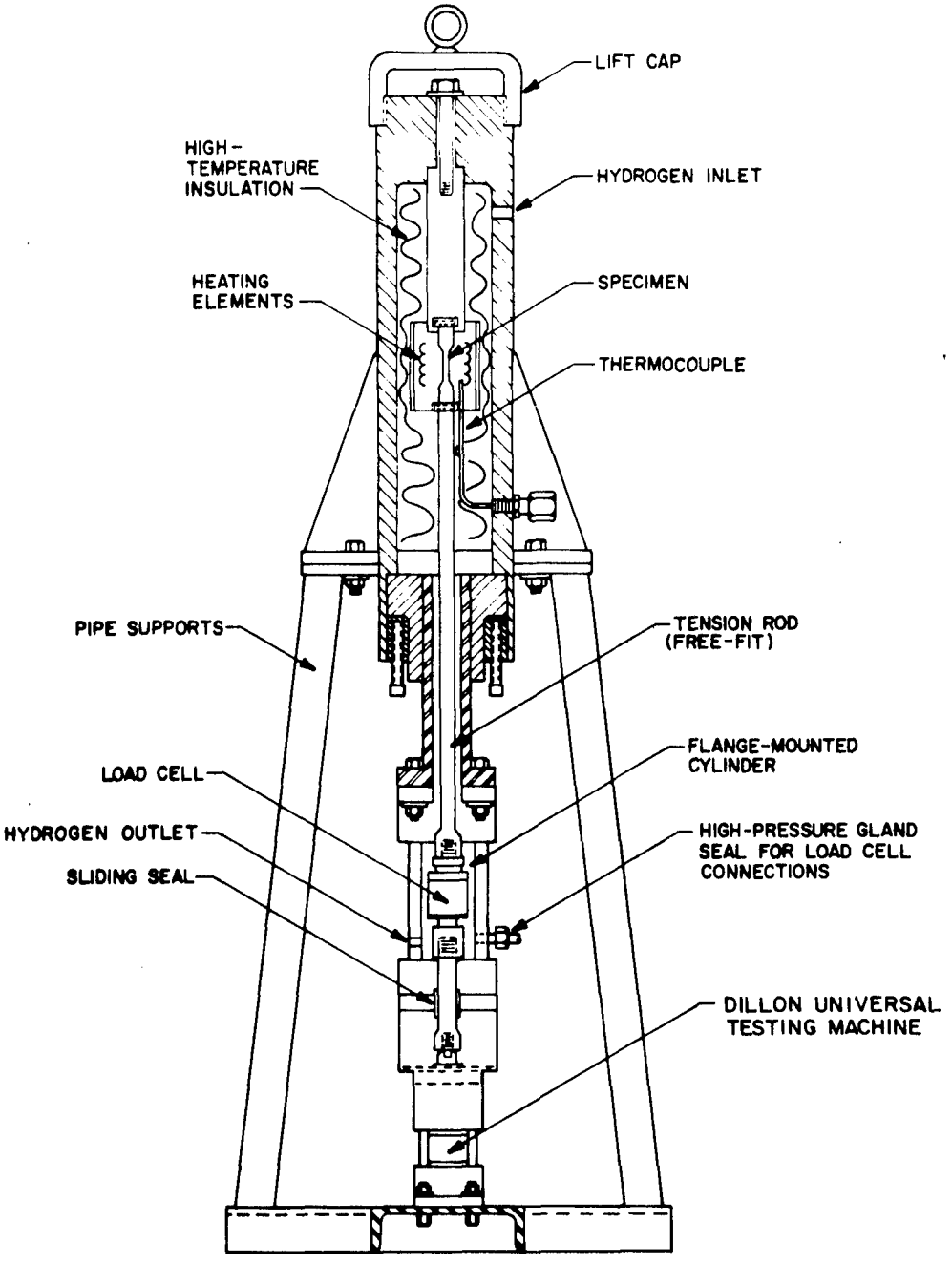

Figure 1. Apparatus for Tensile Testing in High-Pressure Hydrogen

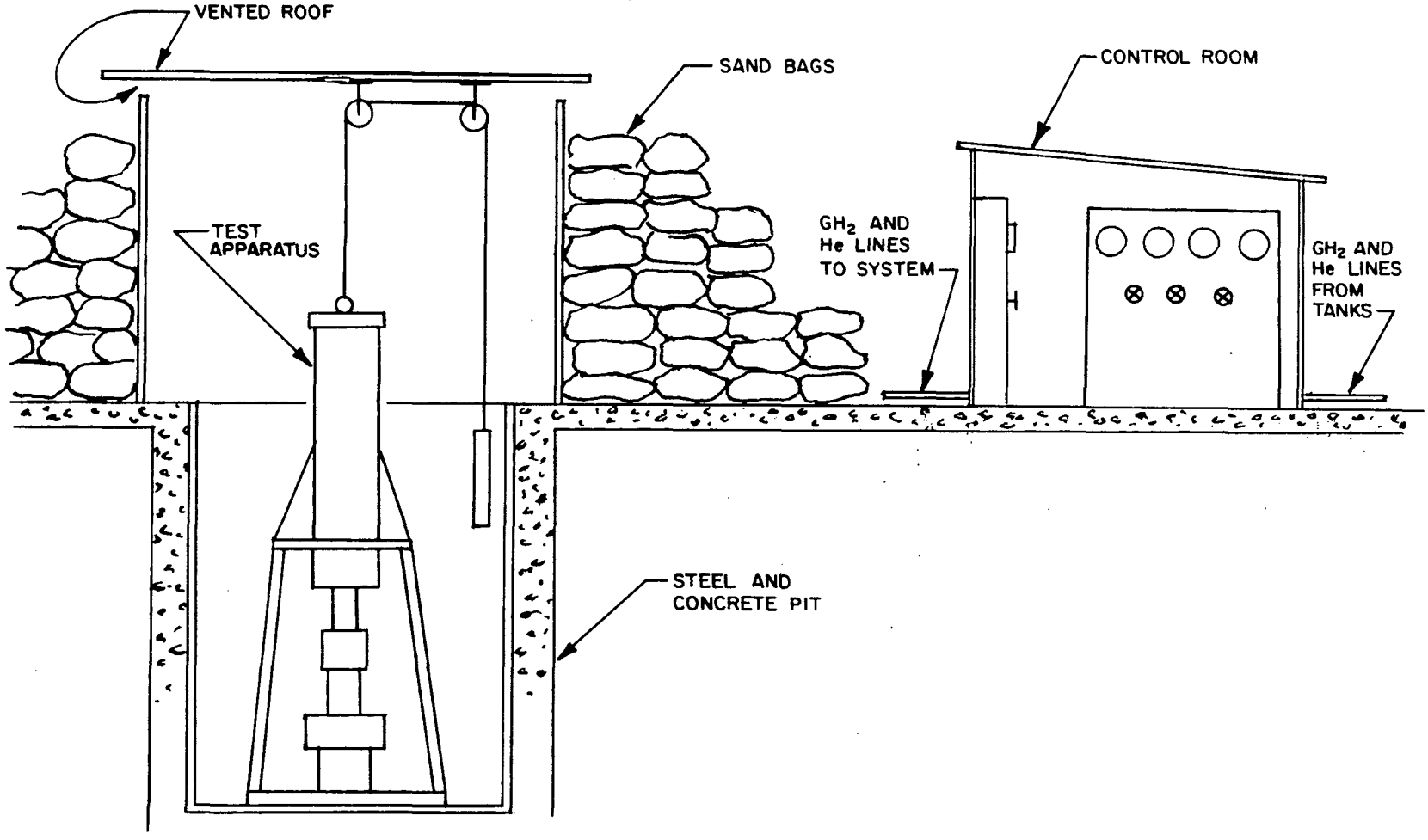

Figure 2. High-Pressure Gaseous Hydrogen Tensile Testing Facility

force exerted on the specimen. The load cell is in a cool-end extension of the pressure vessel, and the gas pressure surrounding the load cell is the same as the pressure in contact with the tensile specimen.

A connecting rod extends from the load cell through a sliding seal and is connected to a screw-driven loading device capable of exerting either a positive or a negative tensile force through the connecting rod. Compression capabilities are necessary because the difference in pressure between the inside and outside of the vessel would exert a force on the connecting rod, tending to stress the specimen in tension.

A completely integrated and fully automatic electrical system will be used to control and record the temperature and loading cycles selected for the mechanical tensile tests. The loading cycle will be predetermined and programmed on an electronic curve follower which will feed a signal to a proportional band controller-recorder. The controller will also receive the load-cell output signal and control the load on the specimen by activating an eddy current hysteresis-type clutch which connects the screw-drive loading mechanism to a continuously running electric motor. The eddy current clutch will engage only enough to follow the programmed loading cycle, resulting in a very smooth and continuous application of load to the specimen. The complete loading cycle will be recorded for each test as a permanent record. The test temperature of the specimens will be controlled using a single set-point proportional controller-recorder and a suitable thermocouple located near the test specimen. The test temperature will be recorded for each test as a function of time.

RESULTS

Compatibility With Hydrogen/Water Vapor Environments

An apparatus, similar to that used by Battles (Ref. 4) for generating hydrogen/water vapor mixtures has been assembled. In addition, apparatus for analyzing the hydrogen/water vapor mixture ratio has been added to

the gas system. A method for analyzing the gas mixture is deemed necesary to ascertain that water vapor is not condensed in the cooler regions of the tensile furnace, and also to assure that saturation is achieved in the constant-temperature bath.

The hydrogen/water vapor testing system is shown in Fig. 3 and 4. Hydrogen used in these tests is purified by a catalytic De-oxo unit converting oxygen to water vapor. The water is collected in a desiccant and all impurities are reduced to less than 1 ppm total by collection in a molecular sieve at the temperature of boiling nitrogen.

The purified hydrogen flowrate is measured by a flowmeter, and is then bubbled into a presaturator containing an internally controlled heater and two external heaters. The hydrogen/water vapor mixture is then sent through the saturator which consists of two bottles containing water and having frit entry ports and a third bottle for mist collection. These bottles are immersed in a precisely controlled constant-temperature bath. All the tubing transporting water vapor is heated with heating tapes. The generated hydrogen/water vapor mixture enters the tensile furnace at the top, then flows through the furnace and out the bottom.

The gas is analyzed after leaving the furnace to ascertain that a proper hydrogen/water vapor ratio is present in the furnace. Gas analysis consists of collection of water by a glass bottle which is weighed before and after to measure the water content. The water-free hydrogen is then passed through a flowmeter and the exact volume measured by water displacement. Calculations of the hydrogen volume include vapor pressure and temperature compensation. A manometer measures the pressure drop across the gas analyzer.

Water is prevented from condensing in the tensile furnace by pumping hot Dow Corning 200 fluid (230 to 250 F) through the pull rods and through the cooling coils protecting the O-rings from overheating at very high furnace temperatures.

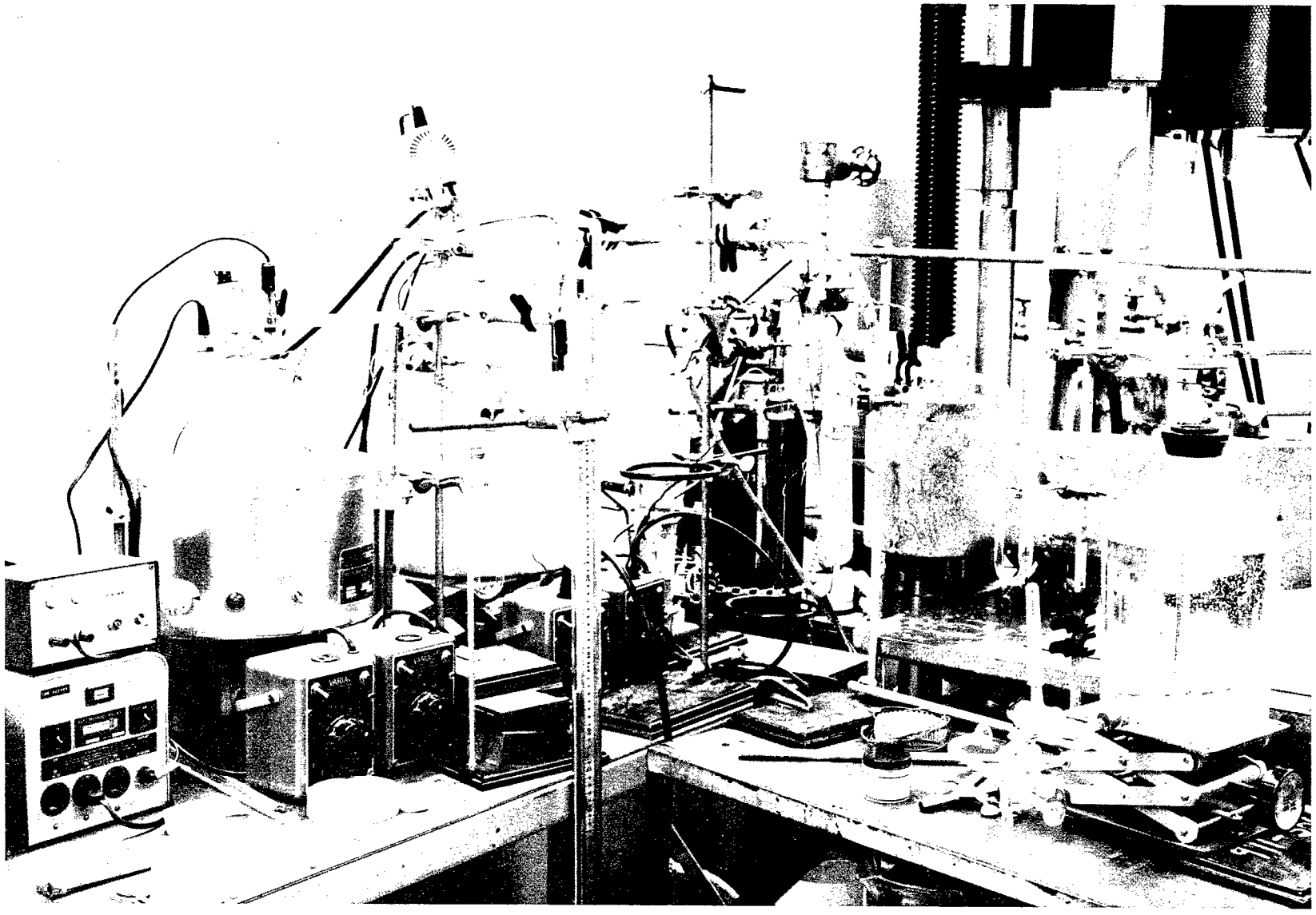

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Figure 3. Hydrogen/Water Vapor-Generating and Gas-Analyses System

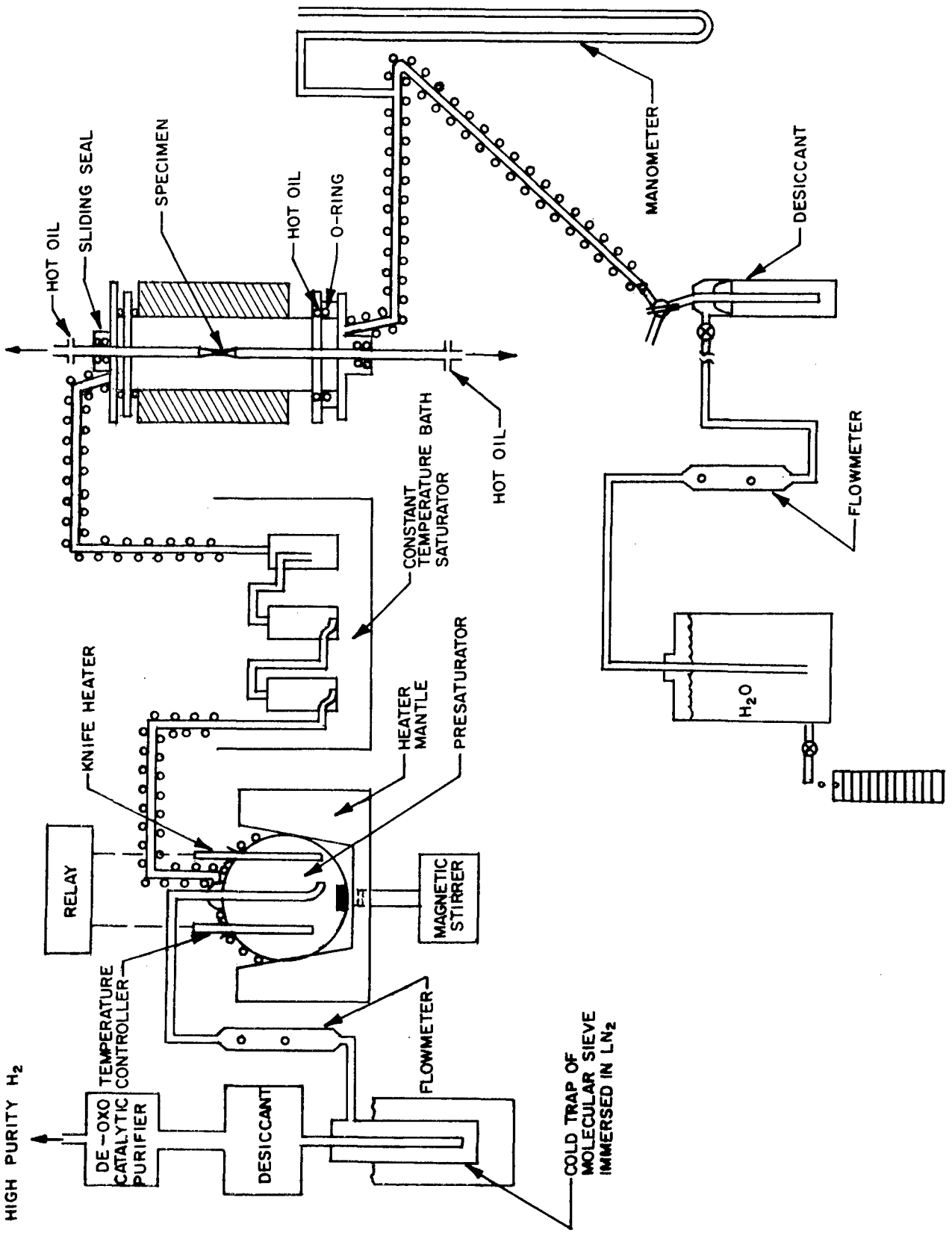

Figure 4. Schematic Representation of Hydrogen/Water Vapor Testing System

To maintain a pure hydrogen/water vapor atmosphere, all the Buna-N furnace O-rings have been replaced by Viton A O-rings. The ball and socket joints in the hydrogen/water vapor apparatus are lubricated with Dow Corning silicone vacuum grease which has a vapor pressure of 5.8×10^{-6} mm Hg (7.62×10^{-9} atmospheres) at 100 C. All the joints in the saturator are O-ring type containing Viton A O-rings. The temperature controller and knife heater are held in the presaturator with neoprene stoppers which have a vapor pressure of 1.1×10^{-4} mm Hg (1.45×10^{-7} atmospheres) at 100 C.

Analysis of the gases from the hydrogen/water vapor generating apparatus before and after entering the tensile furnace indicate that the H_2/H_2O ratio varies between 0.984 and 1.055 for hydrogen flowrates between 233 and 363 cc/min. Therefore there is no indication of water condensation in the cool ends of the furnace over the 400 to 1000 F temperature range studied. These tests were made while the constant-temperature bath was stirred by a 1-inch magnetic stirrer which proved to be inadequate. A propeller-type stirrer has been added, and the H_2/H_2O ratio from two analyses was 1.000 and 1.006 for hydrogen flowrates of 443 and 462 cc/min.

The pressure drop across the gas analysis apparatus is about 8 mm Hg pressure at the flowrates used during the analysis. Since the analysis apparatus will not be attached to the outlet gas stream during mechanical tests, this pressure drop must be compensated for by lowering the temperature to lower the water vapor pressure by 4 mm Hg so that the same H_2/H_2O ratio of 1 is maintained. The H_2/H_2O ratio of 1 was obtained at 82 C with a corresponding water vapor pressure of 384.9 mm Hg. Reducing the water pressure by 4 mm Hg from 384.9 to 380.9, yields the predicted water vapor pressure for obtaining a H_2/H_2O ratio of 1 with the analysis apparatus disconnected from the system. The temperature measurements were not made with a precision thermometer. However, the fact that the required H_2/H_2O ratio of 1 has actually been obtained with the temperatures which were calculated to yield the proper H_2/H_2O vapor mixture indicates that gas saturation with water vapor is indeed obtained in the gas generating apparatus. Calibration has been

restricted to obtaining the H_2/H_2O ratio of 1. Further calibrating for the tests with an H_2/H_2O ratio of 1/3 will be made after all the mechanical tests with the H_2/H_2O ratio of 1 are completed.

Because of the large density difference between hydrogen and water vapor, hydrogen may preferentially accumulate at the upper region of the furnace. If segregation occurs, it would be expected that a higher than normal water content would be evolved from the furnace until steady state was reached. To test this, an analysis was made 15 minutes after the H_2/H_2O vapor mixture entered the tensile furnace to replace argon at 400 F. The flowrate was such as to replace the gas in the furnace tube only seven times. The results, however, did not indicate above-normal water content which would suggest that gas segregation is negligible. Furthermore, it is thought that the mid-point in the furnace, where the specimen is located, would have close to an average H_2/H_2O vapor-gas mixture even if gas segregation occurs.

Performance of the mechanical tests has been temporarily delayed because one of the $MoSi_2$ tensile furnace heating elements burned out.

Solubility and Rates of Absorption and Desorption of Hydrogen

To obtain exact data on the solubility of hydrogen in columbium and B-66, the total expansion of hydrided columbium under given conditions of temperature and pressure must be known. These data are not available from the literature; therefore, a device has been designed and constructed to measure thermal expansion over the temperature and pressure ranges of interest. The device consists of a small chamber with quartz windows which will be attached to the specimen-containing pressure vessel by a short, straight length of tubing. The specimen, in the form of a 2-inch-long cylinder, will be placed in the gold-lined vessel in a vertical tube furnace. A quartz rod resting on top of the specimen will extend through the tube and into the chamber. A fiduciary mark on the quartz rod will be sighted through the window, and its movement observed with a precision

cathetometer. The procedure will involve four measurements, as follows. The background expansion will be determined by measuring the marker movement using a long quartz rod resting on the bottom of the high-pressure vessel. Then a columbium specimen will be put in the vessel with a suitably shortened quartz rod and the expansion measured under 1 atmosphere of inert gas. The difference of these two curves, corrected for the removed segment of quartz rod, will represent the thermal expansion of the metal. These values will be compared with literature data on the thermal expansion of pure columbium. Next, the same measurement will be performed in helium at a pressure of 1500 psi to determine if there is any effect with the increased pressure. Calculations considering the compressibility at these pressures indicate the effect will be negligibly small. Finally, the measurements will be made in hydrogen at pressures of 800 and 1500 psi. These expansion data, compared with the solubility data described earlier, will yield exact values of the solubility of hydrogen in columbium and B-66 under the prescribed conditions of temperature and pressure.

The desorption rate experiments require a flowmeter capable of measuring fairly small quantities of gas at very low flowrates. A commercially available device with the proper specifications could not be located; therefore, a flowmeter capable of the required precision has been designed and built. It operates on the principle of liquid displacement, and involves a long, slender glass tube filled with a saturated sodium chloride solution. The hydrogen gas to be measured first passes through a coil in a constant-temperature bath and then into the top of the measuring tube. A balance tube which can be easily moved in a vertical direction is connected by a flexible hose to the bottom of the measuring tube. A manometer at the gas entrance port is continuously monitored and maintained at 1 atmosphere by moving the balance tube as the hydrogen gas displaces the liquid in the measuring tube. Automatic readout is provided by a series of closely spaced electrical contacts in the measuring tube. As the liquid level drops, these pairs of contacts are successively broken. Each pair constitutes a branch of a parallel circuit in which a fixed voltage is imposed. The current in the circuit, then, decreases stepwise with decreasing liquid level, and

the voltage drop across a fixed series resistor in the circuit also decreases. This voltage is continuously recorded on a high-speed strip chart recorder, giving the voltage, and therefore, the liquid level vs time. Knowledge of the exact volume of the tube between each pair of contacts will give the rate of gas flow. This apparatus is currently being calibrated.

Mechanical Testing at High Pressure

The mechanical testing apparatus has been ordered and delivery is expected on 10 January 1966. Design of the testing facility depicted in Fig. 2 is nearing completion.

FUTURE WORK

MATERIALS

The remainder of the materials for this program will be received and fabrication of the specimens will be completed during the next quarter.

EXPERIMENTAL

Compatibility With Hydrogen/Water Vapor Environments

The mechanical testing for both H_2/H_2O ratios will be performed during the next period. In addition, most of the posttest examination such as metallographic examination and vacuum fusion analysis for oxygen and hydrogen will be performed during the next quarter.

Solubility and Rates of Absorption and Desorption of Hydrogen

During the following quarter, the thermal expansion of hydrided columbium and B-66 under the pressure and temperature conditions applicable to this experiment will be measured. Columbium and B-66 specimens will be prepared and solubility determinations initiated. Calibration of the low flowrate flowmeter will be completed and the experimental apparatus for measurements of hydrogen absorption rates will be assembled and tested.

Mechanical Testing at High Pressures

Delivery of the testing apparatus is not expected until the third quarter. Construction of the test barricade and control shelter for the instruments and valves will be started during the next quarter.

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	Air Force Materials Laboratory Wright-Patterson AFB, Ohio 45433

13. ABSTRACT

The first-quarter results for a program to study the compatibility of columbium with hydrogen environments are presented. An environmental tensile testing machine for testing of columbium at high temperatures and high hydrogen pressures has been ordered. The apparatus necessary for the measurement of hydrogen solubility and rates of absorption and desorption is nearly completed. Auxiliary devices have been designed and built. The hydrogen/water vapor experimental apparatus has been completed and checked out for a hydrogen/water vapor ratio of 1.

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Unclassified
Security Classification

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Columbium Hydrogen Embrittlement Hydrogen Solubility Mechanical Properties						

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DEPARTMENT OF THE AIR FORCE
AIR FORCE RESEARCH LABORATORY
WRIGHT-PATTERSON AIR FORCE BASE OHIO 45433

12 December 2005

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