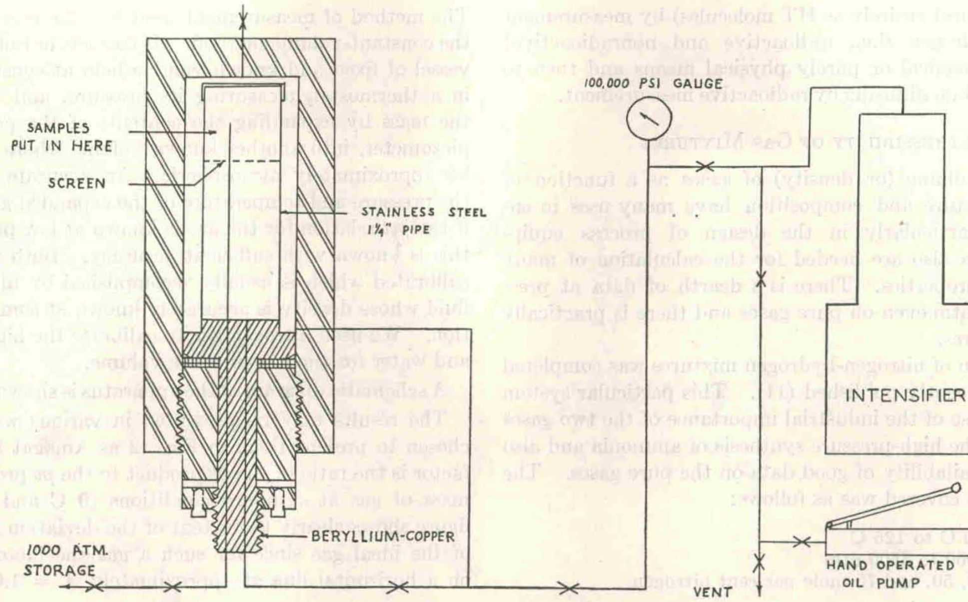

FIG. 10 DIAGRAM OF APPARATUS FOR TESTS OF HYDROGEN EMBRITTLEMENT AT ROOM TEMPERATURE

Only a very few tests have been made with this apparatus so that no results can be reported at this time. All we can say is that it works in the sense that relatively short exposures (a matter of hours) cause a very considerable effect on some samples when pressures of the order of 4000 atm are used.

Experiments on Permeability. The work on permeability is still in the stage of apparatus design and test. We have learned a few things about how not to build it but we have still to develop and prove a suitable design.

It is recognized that it may be exceedingly difficult to obtain reproducible results in these experiments because of the many variables which may affect the rate and because some of them will be difficult to control. For example, there are numerous variables connected with the previous history of the specimen such as its heat-treatment and the amount of cold-working it received which may be important. The nature of the surface may be significant. It is known that there is an "aging" effect which manifests itself in a decreasing rate of permeation with time. There may be flaws in the specimen which will give spurious results. What this means is that we cannot expect results of high precision but this is probably not too important since only gross differences are likely to be of interest and have practical application. It is planned to investigate a number of variables among which are the following:

(*a*) Nature of the gas; various pure gases and also gas mixtures.
(*b*) Pressure.
(*c*) Temperature.
(*d*) Composition of the metal.
(*e*) Previous history of the metal; cold-working, heat-treatment, etc.
(*f*) Condition of the surface.
(*g*) Strained versus unstrained metal.

The apparatus as now envisaged will consist of a tubular specimen surrounded by a jacket which can be evacuated and the whole assembly placed in an electric-tube furnace for temperature control. The rate of permeation will be measured by the rate of rise of pressure in the evacuated jacket.

Some thought has been given to the use of tritium, the radioactive isotope of hydrogen, as a tracer in these experiments.[4] It might be used simply as a sensitive means of determining how much hydrogen has permeated through a metallic membrane. A very sensitive means of measurement would have some advantages since the amount of gas permeating in any short interval of time is very small indeed and the problem of determining rate of permeation is a difficult one especially at the lower temperatures. We also had in mind the use of tritium to shed some light on the mechanism of permeation. Suppose one used tubular specimens and placed hydrogen containing tritium under pressure inside the tube. The amount of hydrogen retained in the specimen and its radial distribution conceivably might be measured by cutting off successive layers of the tube, dissolving each cut in acid and measuring the radioactivity. The radioactivity measurements would be a means of distinguishing the hydrogen that originated from the acid from that which permeated the metal.

In studying the effect of pressure on permeability, one usually increases the pressure on one side of the metal wall and maintains a low pressure on the other side. This results in an increase both in the total pressure and in the pressure gradient. For some purposes it would be desirable to increase the total pressure at constant pressure difference. It would be difficult in this case to measure the rate of permeation by the usual techniques but the use of a radioactive tracer offers a possible method.

When the pressure is not the same on the two sides of the diffusion membrane it will be strained. It might be of interest to measure also the permeability under a condition of no strain. This might be done by using an inert, i.e., nondiffusing gas, such as helium or argon, to equalize the total pressures while still maintaining a partial-pressure difference of hydrogen. In such a case the use of tritium to "tag" the hydrogen molecules in order to measure those which penetrate the wall of the metal, would be most useful.

One difficulty that arises in the use of tritium to trace hydrogen is the fact that these two isotopes are quite different in atomic weight and may have appreciably different rates of diffusion. However, it should not be too difficult to measure the relative permeation rates of hydrogen and tritium (actually the latter

[4] This was first suggested by Dr. S. W. Wan, formerly Research Associate in Chemical Engineering at Yale.

will be present almost entirely as HT molecules) by measurement of the total hydrogen (i.e., radioactive and nonradioactive) permeating by chemical or purely physical means and then to determine the tritium diffusing by radioactive measurement.

Compressibility of Gas Mixtures

Data on the volume (or density) of gases as a function of pressure, temperature and composition have many uses in engineering and particularly in the design of process equipment. Such data also are needed for the calculation of many thermodynamic properties. There is a dearth of data at pressures above 1000 atm even on pure gases and there is practically nothing on mixtures.

An investigation of nitrogen-hydrogen mixtures was completed and the results recently published (11). This particular system was chosen because of the industrial importance of the two gases and their use in the high-pressure synthesis of ammonia and also because of the availability of good data on the pure gases. The range of variables covered was as follows:

Temperature: 30 C to 125 C
Pressure: 1000 to 3500 atm
Composition: 25, 50, and 75 mole per cent nitrogen

The method of measurement used was the one commonly called the constant-volume method. It consists in isolating the gas in a vessel of fixed and known volume held at constant temperature in a thermostat, measuring its pressure, and then determining the mass by expanding the contents of the pressure vessel, or piezometer, into another known volume where the pressure will be approximately atmospheric. An accurate measurement of the pressure and temperature of the expanded gas gives the mass if the *pvt* relation for the gas is known at low pressure. Usually this is known with sufficient accuracy. Both volumes must be calibrated which is usually accomplished by filling them with a fluid whose density is accurately known at some specified condition. We used nitrogen gas to calibrate the high-pressure vessel and water for the low-pressure volume.

A schematic diagram of the apparatus is shown in Fig. 11.

The results may be presented in various ways but we have chosen to present them in Fig. 12 as Amagat factors A. This factor is the ratio of the *pv* product to the *pv* product of the same mass of gas at standard conditions (0 C and 760 mm). The figure shows clearly the extent of the deviation from the concept of the ideal gas since for such a gas the factors would all fall on a horizontal line at approximately $A = 1.072$. At the top

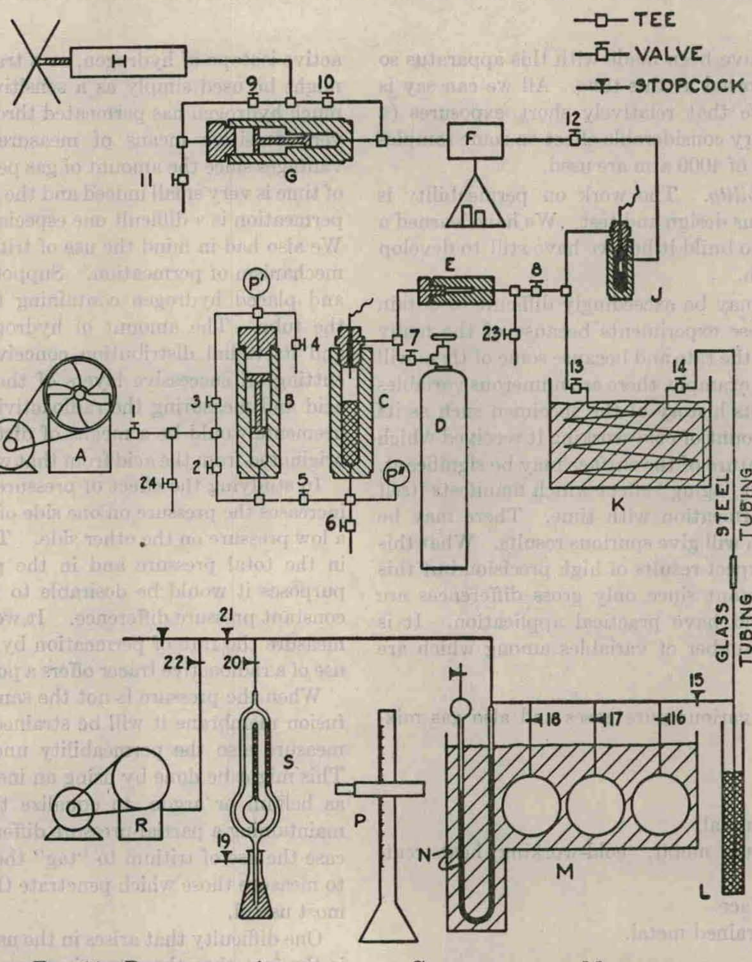

Fig. 11 Diagram of Apparatus for Compressibility Measurement

A	Triplex hydraulic pump	J	Mercury U-tube
B	Aminco intensifier	K	Constant-temperature oil bath
C	Gas-compression chamber	L	Safety tube
D	Gas-storage cylinder	M	Constant-temperature water bath
E	Drier	N	Mercury manometer
F	Dead-weight piston gage	P	Cathetometer
G	Small intensifier for piston gage	R	Vacuum pump
H	Cailletet pump	S	McLeod gage