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A PROGRAM FOR INVESTIGATION OF METALLIC HYDRIDES AS MODERATORS, REFLECTORS, AND SHIELDS FOR AIRCRAFT REACTORS

by

Paul T. Gilbert, Jr.

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ABSTRACT

The applications of metallic hydrides, especially zirconium hydride, in nuclear technology are reviewed, with particular emphasis upon their use in advanced aircraft reactor designs. Pertinent research currently in progress at other sites is summarized. A two-year program of research is proposed for the purpose of developing and evaluating metallic hydrides having improved properties for use as high-temperature moderators, reflectors and shields, and for supplying fundamental information on the hydrides to be employed in aircraft reactors.

I. INTRODUCTION

A. Advantages of the Metallic Hydrides

Certain metallic hydrides, especially hydrides of titanium and zirconium, have for several years been considered in possible applications as neutron moderators and shields. These hydrides are particularly advantageous because of their high hydrogen content, comparatively low neutron absorption, and thermal stability. Earlier work had indicated that zirconium hydride possessed possibly a better combination of properties than any other substance, for use as a moderator in compact, light-weight reactors operating at high temperatures. Aircraft engines and power generation on a satellite vehicle were the principal applications to receive consideration. Recent intensive work has revealed that hydrides of certain zirconium alloys are admirably suited for use as moderators, moderator-fuel combinations, reflectors and shields in aircraft engines, and these hydrides are being subjected to a rapid engineering evaluation in the field of aircraft nuclear propulsion. The results are so favorable that it is clear that aside from the certainty of their use in aircraft, these hydrides may find more extended application in nuclear technology.

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B. Applications in Nuclear Reactors

Research on hydrides of zirconium and related metals proceeds at an accelerating tempo. Work on hydrides directed to their use in nuclear reactors is currently in progress at General Electric (ANP Department), Wright-Patterson Air Force Base (Wright Air Development Center), Sylvania Electric Products, Battelle Memorial Institute, and, in conjunction with the above, at ORNL and KAPL. Proposals of reactor designs incorporating zirconium hydride as moderator have been made by Coneybear (COO-108), Balent (NAA-AER-Memo-896), and by General Electric as reported in the recent ANP engineering progress reports (APEX-13, APEX-14; a revision of APEX-14, also APEX-15 and a topical report covering metallic hydrides are scheduled to issue soon). Meanwhile, a thorough literature search has been carried out at NAA on the subject of zirconium and hafnium hydrides and deuterides, covering such subjects as applications, thermodynamics, radiation stability, phase relationships, nuclear, physical and chemical properties, fabrication, canning, and alloy systems and cermets. The most recent work on the hydrides of titanium, uranium, etc., has also been examined. Pending completion of this review, reports of the work now in progress at other sites on metallic hydrides have appeared as NAA-SR-Memo-1360 and NAA-SR-Memo-1380.

C. Other Applications

Other applications of zirconium hydride include powder metallurgy and alloy fabrication, bonding ceramics to metals, cladding and surface alloying of uranium, decladding of zircaloy-clad uranium (by hydriding), preparation of zirconium nitride for crucibles, separation of zirconium from uranium by hydriding (as yet unsuccessful), inhibition of corrosion in the uranium fluoride fuels Fulinak and Funazr, activation of control rods, and standardization of analytical procedures for zirconium. Hafnium hydride, in most other respects exceedingly similar to zirconium hydride, has been turned to account by virtue of the large neutron absorption cross section of hafnium, for the preparation of control rods of hafnium-boron alloy (BMI-935). Titanium hydride, in addition to other applications analogous with those of zirconium hydride, is being employed in resistors in connection with a proposed aircraft nuclear power plant (APEX-14, p. 126-7).

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II. CURRENT RESEARCH

A. Massive Hydrides

Limitations upon the use of metallic hydrides resulting from the comparatively poor physical properties of the powder-metallurgical compacts have very recently been removed by the discovery of a method of preparing these hydrides in massive form, having theoretical density. The method and the products obtained are described in APEX-14, p. 138, and NAA-SR-Memo-1380. Massive, metallic hydrides having the formulas ZrH, $YH_{1.5}$, TiH, $(Zr, Y)H_2$, and $(Ti,Y)H_{1.85}$ have thus been obtained. The yttrium alloys are particularly promising; a hydride of 10 per cent yttrium - 90 per cent titanium possesses the highest hydrogen content ($N_H = 8.7 \times 10^{22}$ atoms of H per cm^3) yet found for a solid, massive, metallic material having good physical properties. Moreover, the yttrium-zirconium hydrides exhibit greater thermal stability than would be expected from interpolation, and may be useful in air-cooled moderators at 1100° C. Yttrium hydride itself retains an N_H of 4 up to about 1200° C at 1 atmosphere, and in this respect is perhaps superior to all solid substances. Zirconium-cerium hydrides are reported in APEX-13, p. 164. The neutron cross sections of cerium and yttrium are not much greater than that of zirconium, and the advantages conferred by these alloying constituents outweigh their increased neutron absorption.

Among the properties of these hydrides undergoing measurements are density, thermal expansivity, hardness, elasticity, compressive strength, ductility, tensile strength, thermal conductivity, reactivity with air, compatibility with metals, corrosion by liquids (organic coolants, fluoride fuels, etc.), dissociation pressure, kinetics of equilibration, and radiation stability. The massive hydride ZrH shows twice the tensile strength of zirconium at elevated temperatures, excellent ductility, thermal conductivity slightly exceeding that of zirconium, and excellent stability in air up to 800° C. It appears to be entirely compatible with the liquid coolants and fuels at their respective operating temperatures. Intense pile radiation has not been observed to affect the hydride, but studies are not complete.

B. Cladding

Earlier methods of canning cold-compacted titanium hydride (NEPA-1611) have been supplanted by more successful methods of cladding zirconium hydride, developed by General Electric. One method consists in mass-

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ively hydriding zirconium within a sweater of molybdenum surrounded by stainless steel or an iron-chromium-aluminum alloy; another involves extrusion of a billet sheathed in niobium and stainless steel. Excellent bonds between the hydride and the cladding are obtained, and N_H values as high as 5.7 have been attained with clad, unalloyed zirconium hydride. Preliminary tests of the performance of these clad hydrides in air have been carried to 1000° C with favorable results.

C. Special Applications in Aircraft Reactors

Studies of the preparation and properties of massive zirconium deuteride for use as a neutron reflector have been undertaken at General Electric. Massively hydrided uranium-zirconium alloys containing up to 60 per cent uranium have also been obtained; these are proposed for use as neutron and gamma shields and, at the lower uranium concentrations, as a moderator-fuel material. The latter application is only now beginning to receive attention. It is not certain that a successful aircraft engine can be built employing a uranium-zirconium hydride moderator-fuel element; the requisite power densities are possibly too high.

For use in aircraft reactor shields, zirconium hydride is receiving principal attention. Gadolinium and samarium have been incorporated as neutron absorbers, and heavy metals such as uranium added for gamma shielding. It is expected that the hydride will be cooled by an organic moderator-coolant such as an alkylbenzene, or possibly by a liquid salt. Further, a device in which zirconium hydride activates a control rod following a surge of power, is being built at Wright Air Development Center.

D. Compacts and Liquid Hydrides

Sylvania has been successful in hot-pressing zirconium hydride, but inasmuch as the compacts are inferior in most respects to the massive hydrides, the study of hydride compacts has been discontinued. Similarly, General Electric is no longer concerned with the calcium hydride-beryllium cermets, which had been found useable to 700° C in open air, or with the liquid alkaline earth hydrides, among which a calcium-barium hydride mixture appears to be the best high temperature liquid moderator known, retaining an N_H of 3.3 at 980° C and 1 atmosphere (APEX-13).

E. Alloy Hydrides

Aside from the rare earth alloy hydrides, other observations of hydrides having unexpectedly high hydrogen content or thermal stability include a notation concerning a zirconium-nickel hydride containing 40 per cen more hydrogen than ZrH_2(NEPA-1841), p. 12), a description of a nitrided titanium hydride of markedly reduced dissociation pressure (NEPA-1387), and the statement that small amounts of zirconium may stabilize the higher thorium hydride (ibid.).

At Battelle, hydrides of vanadium (BMI-937, p. 34), chromium (BMI-949, p. 99), and niobium (BMI-964, p. 94) have been prepared containing more hydrogen than hitherto reported for these compounds. Of special interest is the theory of Mallett and Trzeciak (BMI-968, p. 100) that if two metals, one readily forming hydrides and the other not, can form a solid solution having the structure of the first metal, the alloy will hydride as readily as the first metal; by this means exceptionally high hydrogen concentrations should be attainable; the effect of lattice parameters has been considered in BMI-972, p. 109. They have found that a 30 per cent molybdenum - 70 per cent titanium alloy behaves in this manner, absorbing hydrogen to an N_H of 9.8, and containing 20 per cent more hydrogen than titanium hydride under comparable hydriding conditions; they have obtained interesting results also with titanium-vanadium, titanium-niobium and titanium-manganese hydrides. A 20 per cent niobium alloy yielded an N_H of 9.6 (BMI-977, p. 108). X-ray diffraction studies of the phase relationships in these alloys is in progress (BMI-985, p. 114).

At Sylvania, aside from the massive hydriding program, the hydriding of uranium-zirconium alloys has received attention (SEP-163, p. 12; SEP-190, p. 10); certain of these hydrides are ferromagnetic like UH_3, but the results have been somewhat anomalous, and attempts to separate the two metals via the hydrides, by magnetism, elutriation, screening, nitriding or amalgamation, have not succeeded.

F. Dissociation Pressure and Phases

The most recent work on the dissociation pressure and phase diagram of the zirconium-hydrogen system has begun to clarify a situation which was earlier rather confused. The phase diagram shown in BMI-914, p. 66, has been somewhat refined, but certain questions remain unanswered, in particular the status of the gamma phase (Gulbransen and Andrew, J. Electrochem. Soc. 101, 474 (1954) and the metastability reported for epsilon ZrH_2 (BMI-920, p. 73; BMI-937, p. 66). Dissociation pressure measurements are available for the regions of high hydrogen content and high pressure (NAA-SR-1205), low hydrogen content in the alpha and beta phases (BMI-964, p. 55), intermediate hydrogen content at high temperature (current work at Wright Air Development Center), full range of hydrogen content at low temperatures (Gulbransen and Andrew, loc. cit.), alpha phase (Gulbransen and Andrew, J. Metals 7, 136 (1955), and beta, delta and epsilon phases (Edwards, Levesque and Cubicciotti, J. Am. Chem. Soc. 77, 1307 (1955). Some discrepancies and certain unexplored areas remain, but the gaps are rapidly being filled in. Comparable work is being published on the dissociation pressures and phase relations of the hydrides, deuterides and tritides of titanium and uranium and hydrides of the rare earth metals (NAA-SR-Memo-1360; Mattraw, J. Phys. Chem. 59, 93 (1955); Abraham and Flotow, J. Am. Chem. Soc. 77, 1446 (1955)).

With the improved results on dissociation pressures, several papers on the thermodynamics and statistical mechanics of zirconium and other hydrides have appeared. These are summarized in NAA-SR-Memo-1360, and although no unified and entirely satisfactory theory of the phases and thermodynamics of zirconium hydride has yet been presented, such a theory seems to be now within reach. Complete information on partial molal free energies, entropies, activity coefficients, etc., should soon become available. Some current work on electric resistivity, heat capacity and magnetic susceptibility of titanium hydride has also been described in the above memo.

III. OUTLINE OF PROPOSED RESEARCH PROGRAM

A. Search for Promising Hydrides

1. Construction of levitation and hydriding apparatus.
2. Procurement of pure metals, with analyses.
3. Preparation of alloy hydrides.
    a. Cursory dissociation-pressure studies.
    b. Massive hydriding.
    c. High-pressure hydriding.
    d. Density determinations.

4. Theoretical analysis of results in search of generalizations.
5. Directed search for better hydrides.

B. Detailed Evaluation of Selected Hydrides

1. Complete dissociation-pressure survey.
    a. Stoichiometry and high-pressure measurements.
2. Phase studies.
    a X-ray diffraction of quenched samples.
    b. High-temperature X-ray studies.
    c. Metallography.
3. Liquid Phase Studies, if possible.
4. Mechanical properties.
    a. Hardness
    b. Elasticity, rigidity.
    c. Compressive strength.
    c. Tensile properties.
5. Thermal properties.
    a. Expansivity.
    b. Thermal conductivity.
6. Electric conductivity.
7. Effects of contaminants.
    a. Dissociation-pressure studies.
    b. Development of analytical methods.

(C. Reactor Applications; Tentative.)

1. Corrosion of hydrides by
    a. Organic coolants
    b. Fused salts.
    c. Liquid fuels.
2. Stability of clad slugs in air at high temperature.
3. Kinetic studies.
    a. Equilibration rates with gaseous hydrogen.
    b. Diffusion of hydrogen in the solid.
4. Thermal cycling.
    a. Effect on mechanical properties.
    b. Migration of hydrogen.

(D. Special Study of Uranium Alloy Hydrides; Tentative.)

E. Literature Work.

1. Bibliographic File Maintenance.
2. Critical Review.
3. Thermocynamic Calculations.

IV. DISCUSSION OF RESEARCH PROGRAM

A. Coordination of Work

As discussed in Section II, active work directed to the use of zirconium hydride and allied materials in high-temperature nuclear reactors is in progress at several sites. This work is now being extensively coordinated by Wright Air Development Center; the principal effort is being expended by General Electric, which has provided funds for subcontracted work at Battelle. In view of the great promise of the metallic hydrides as reactor materials, a full research effort should be maintained. However, certain aspects of the work are clearly being neglected, and opinions of representatives of several of the interested sites concur that North American Aviation should contribute to the program. This might be accomplished by obtaining a subcontract from General Electric (Dr. Miles Leverett of the ANP Department), or more likely a contract directly from the Reactors Branch of the AEC. In either event, Wright Air Development Center should be privy to our proposal and should exercise the office of coordinating the results of our research. In conferences at WADC, it has been indicated that there is insufficient basic information on the physical chemistry of the hydrides. NAA is in a good position to fill the gaps in the coordinated hydride program.

B. Exploratory Investigation

Firstly, the search for and study of metallic hydrides having greater hydrogen content and/or lower dissociation pressure than those now known, compatible with sufficiently low neutron cross section, is of paramount interest. Other things being equal, the weight of a moderator must vary, as a first approximation, with the inverse cube of the hydrogen density. Objectives include the possibility of designing smaller and lighter reactors, and reactors operating at higher temperatures and power densities.

The search for hydrides of high N_H and high thermal stability would supplement and extend the effort at Battelle and (to a lesser extent) at General Electric. However, we are acquainted with techniques by means of which the work could be greatly speeded and results of improved accuracy obtained. This can be done with the help of electromagnetic levitation. The levitation apparatus will allow rapid preparation of alloys of accurately known composition, uncontaminated by the process of melting, homogeneous, clean and completely recoverable within the precision of weighing. These alloys can then be hydrided directly in situ, preferably under pressure to accelerate the reaction. The purity of the product thus obtained would be such that no subsequent analysis would be required, the hydrogen content being determined directly by weighing.

In this way a rapid survey could be made of various systems, ternary and higher, including non-metallic as well as metallic alloying constituents. Wherever interesting or promising results might appear, the systems in question would be studied in further detail, for the purpose of elucidating the pressure-temperature-composition relationships and the phase diagram. The latter could be tentatively supplemented by means of x-ray diffraction analysis of quenched samples. High-temperature x-ray diffraction might be desirable in the study of systems of special interest, inasmuch as accurate knowledge of phase relationships at temperatures of operation is of basic importance. Unless x-ray diffraction gives sufficiently good density measurements, it might be desirable to build a helium pycnometer according to designs already in use for density determinations in hydride systems (Nottorf, AECD-2984).

Of practical and theoretical interest also is the behavior of dissociation pressure at high hydrogen concentrations approaching stoichiometric compositions, at pressures up to 500 or 1000 psi, such as might prevail in certain types of reactors. The technique of making such measurements has been adequately developed at NAA, where results have been obtained for zirconium hydride of accuracy superior to that achieved in any comparable investigation hitherto reported. With knowledge gained in the cursory study of various systems, and the more detailed study of dissociation pressure and phases in selected systems, generalizations would doubtless come to light, enabling further useful discoveries to be made, perhaps along the lines being explored at Battelle.

C. Electromagnetic Levitation

This method of supporting liquid metals freely in space was reported in 1952 by Okress, Wroughton and their associates (J. Appl. Phys. 23, 545, 1413 (1952)) of Westinghouse; they reported successful levitation of liquid aluminum, silver and copper (Wroughton et al., Electrochemical

Society, New York, Apr. 13, 1953). Application of their methods at NAA had at that time resulted in the levitation of liquid steel, uranium (NAA-SR-Memo-591, 608, and 615), thorium (NAA-SR-Memo-648), and zirconium (Gilbert, American Chemical Society, Cincinnati, Apr. 7, 1955). The method has been shown to be ideally suited to the preparation of small amounts of alloys of reactive metals, uncontaminated by contact with container materials and of accurately controlled composition. Although levitation is too new to have been widely applied as yet, a few recent publications are indicative of increasing interest, especially the work of Polonis and coworkers (<u>Research</u> 7, 10s (1954); J. <u>Metals</u> 6, 1148 (1954)), who have employed levitation-melting apparatus for the preparation of high-purity alloys of titanium and iron.

D. Levitation Apparatus

Fig. 1 shows a suggested apparatus for the preparation of alloys by levitation followed by reaction with hydrogen for the preparation of mixed hydrides. The hydrogen purifying train would be built of copper and stainless steel tubing, to withstand several hundred psi during the high-pressure charging of the alloys with hydrogen. The alloys would be prepared either in vacuum or in a hydrogen atmosphere in the Vycor tube at the right (which may be expected to withstand a limiting pressure of 1000 psi), connected to the copper tubing by wax seals of the type described in NAA-SR-1026. A movable coil of the sort already employed for levitating liquid zirconium would surround the tube closely. The sample would be introduced by opening a soldered connection between a 1/2" and a 1/4" copper tube below the Vycor tube; it would rest upon a graphite mold, slotted to diminish electromagnetic induction, and in turn supported upon a 1/8" copper tube or the like. In operation, the system would be sealed, evacuated, and outgassed while the sample and mold were warmed by the coil; the metallic pieces would then be lifted from the mold with full power in the coil and allowed to melt and mix briefly, after which the liquid mass would be lowered close to the mold and allowed to drop. In some cases a copper mold, so formed as to remove heat readily but not to overload the levitation coil before take-off, might be required, inasmuch as liquid zirconium cast into graphite may adhere strongly. In the subsequent operation, hydrogen would be admitted under pressure and the sample warmed sufficiently by the coil to induce the hydriding reaction. The apparatus would be amenable to the massive hydriding technique as practised by General Electric and Sylvania. It would also be satisfactory for the study of high-pressure hydriding which GE has just begun. With a known volume of gas in the system, the quantity absorbed could be monitored fairly closely by means of the pressure gage. Thus, with pyrometric observation of temperature, a rough idea of the pressure-temperature-composition relationships could be obtained at elevated temperatures and pressures without removing the sample from the apparatus in which the alloy was prepared. The levitation apparatus would also be useful for the convenient addition of non-metallic constituents, such as carbon from a methane atmosphere, sulfur from hydrogen sulfide, nitrogen from ammonia, or oxygen as such.

E. Detailed Evaluation

Following the preliminary survey of materials, a second phase of the investigation would ensue, focussing attention upon the careful evaluation of certain properties of those hydrides shown to be the most promising. Some of the desired investigations mentioned at the WADC conference include the determination of the mechanical moduli (elasticity, rigidity, etc.) of the massive hydrides; better measurements of thermal expansivity; electric resistivity; kinetics of equilibration of the hydride with ambient hydrogen; and diffusion of hydrogen within the hydride. To be sure, other sites may be better equipped to conduct certain of these measurements, especially by the time the exploratory phase of our investigation is completed. Corrosion of the hydrides by liquid fluoride fuels, fused salts, and organic coolants must be investigated for certain applications. It is likely that no further work on radiation stability should be undertaken at NAA, inasmuch as appropriate tests are being carried out at ORNL, and the results seem very favorable. Methods of fabrication, cladding, and testing of stability in air at high temperatures are being adequately handled at other sites. Measurements of density, thermal conductivity, and mechanical strength, and tests of the effect of thermal cycling upon massive hydrides can probably be carried out equally well here or at GE or WADC.

F. Fuel-Moderator Elements

The special problem of the fabrication and testing of metallic hydrides containing uranium for use as moderator-fuel elements will depend upon the tentative work now in progress at WADC, GE and Sylvania, and it would seem premature to set up a research proposal on this item pending the accumulation of further results. If and when it becomes desirable, uranium alloy hydrides can be subjected to the program of research described above. The principal problem at the moment seems to consist in finding a uranium-containing hydride with high N_H and good thermal stability; results have not been too encouraging.

G. Contaminants and Analytical Development

Other topics which NAA would be in a good position to pursue include the effect of contaminants (e.g., oxygen) deliberately or accidentally introduced into a hydride, especially upon the dissociation pressure; and the development of improved analytical methods. It is true that appreciable progress has been made very recently in the determination of hydrogen and oxygen in metallic hydrides, and General Electric employs an apparently very satis-

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factory method for hydrogen, as yet unpublished (NAA-SR-Memo-1380). There is a good probability that the method for determining oxygen in titanium and similar materials, proposed in the forthcoming General Chemistry progress report, may prove to be the most satisfactory hitherto devised, and development of the method seems advisable.

H. Theoretical and Bibliographic Work

It should not be forgotten that NAA has the most extensive file of information on zirconium hydride in existance, and that a critical review and compendium of this information would be of considerable value to workers in the field. Several individuals at WADC have urged that time be allotted for completion and maintenance of this compendium. A particular aspect of this work is concerned with evaluation of the thermodynamic properties of the hydrides on the basis of published data; much more information is latent in these data than has been extracted. Further, the effort to carry out complete thermodynamic calculations would reveal weak spots and gaps, which could then be filled in with the greatest economy of experimental effort. As the program progressed, similar evaluations might be required for hydrides other than of the pure metals.

V. TENTATIVE SUMMARY OF COSTS FOR TWO-YEAR PROGRAM

Fiscal year 1956:		
Survey and evaluation of hydride systems, 2 man-years		$50,000
Frequency converter and other equipment		10,000
Bibliographic and theoretical work, 1-man year		25,000
Total, 1956	$85,000	
Fiscal year 1957:		
Detailed evaluation of hydrides, 2 man-yrs.		$50,000
Equipment		10,000
Reactor applications and uranium alloys, 1 man-yr.		25,000
Total, 1957	$85,000	
Total two-year program		$170,000

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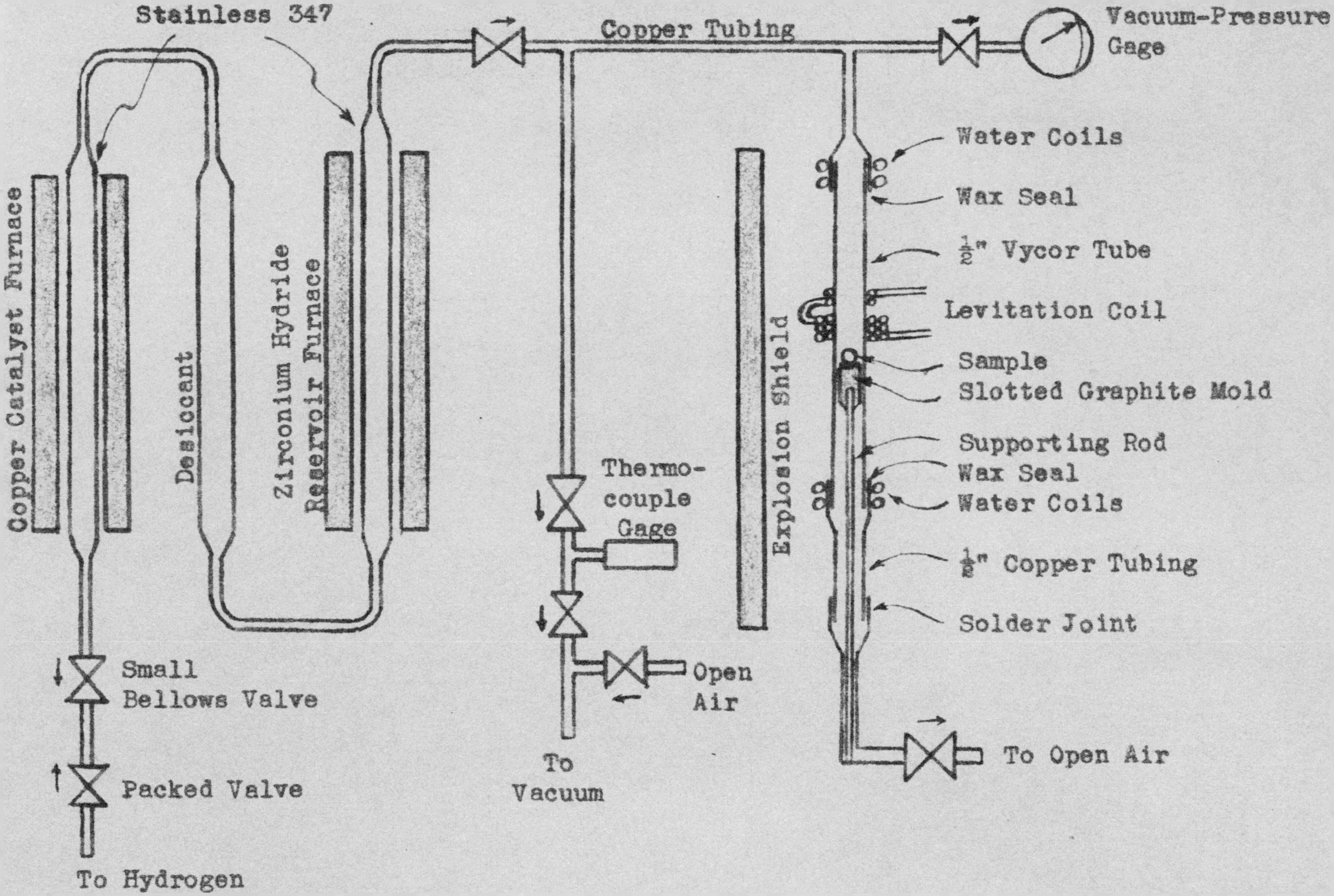

Fig. 1. Apparatus for Preparation and Hydriding of Alloys.