

IS-190

ANNUAL SUMMARY
RESEARCH REPORT IN ENGINEERING
for
July 1, 1959-June 30, 1960
by
Ames Laboratory Staff

AMES LABORATORY
RESEARCH AND DEVELOPMENT REPORT
U.S.A.E.C.



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IS-190

Chemistry-General (UC-4)
TID 4500, August 1, 1959

UNITED STATE ATOMIC ENERGY COMMISSION
Research and Development Report

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RESEARCH REPORT IN ENGINEERING
for
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by
Ames Laboratory Staff

August 1960

Ames Laboratory
at
Iowa State University of Science and Technology
F. H. Spedding, Director
Contract W-7405 eng-82

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Printed in USA. Price \$ 1.00 . Available from the

Office of Technical Services
U. S. Department of Commerce
Washington 25, D. C.

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ANNUAL SUMMARY RESEARCH REPORT IN ENGINEERING

For the Period July 1959-June 1960

This report is prepared from material
submitted by group leaders of
the Laboratory

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ISC-130	ISC-573
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ISC-220	ISC-836
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July 1, 1959 - June 30, 1960

Under the direction of F. H. Spedding, L. E. Burkhart, G. Burnet,
R. W. Fahien, R. W. Fisher, G. Murphy, E. H. Olson,
M. Smutz and D. R. Wilder.

Chemical Engineering

1. Liquid-liquid Extraction in Pulse Columns (L. Burkhart)

The size and distribution of dispersed phase droplets in a pulse column are important factors in the efficiency of the column and also in the geometric design of the column. Three systems--hexone-acetic acid-water, benzene-water, and n-heptane-water--were used in a 3-inch diameter pulse column to measure droplet size along the lower flooding curve. Average droplet size varied directly with pulse amplitude, pulse frequency, hole diameter, and Reynolds number. Along the lower flooding curve the combined effect of pulse amplitude and pulse frequency was more important than either variable alone in determining the average droplet size.

At operating conditions above the lower flooding points where forced recycle in the column was significant, the average size of the dispersed phase droplets varied directly with hole diameter and inversely

with pulse amplitude and pulse frequency.

All droplet size measurements were made photographically while the column was in steady-state operation.

A method of calculation was proposed to set the physical size of a pulse column, the method being subject to the following assumptions:

(1) the continuous phase preferentially wets the plates, (2) the column is operating in the mixer-settler region, (3) an equal volume of liquid flows through each perforation.

Reproducible discontinuities in the average droplet size curves near the lower flooding points were explained by an analysis of column operation in relation to pulse amplitude, pulse frequency, and dispersed phase hold-up. This was verified by high-speed motion pictures of the pulse column in operation. Groups of very small droplets, usually present in the column, disappeared at operating conditions such that the hold-up of the dispersed phase was of the proper volume to produce an integral number of droplets during one half-cycle of the pulse generator.

2. Performance of a Scheibel Extractor (L. Burkhart)

The droplet behavior and dispersed phase hold-up in Yorkmesh packing was studied with an equilibrated system of methyl isobutyl ketone and water with the ketone phase dispersed. Similar studies were then made on a 3-inch diameter Scheibel column.

The droplet size in a Scheibel column is determined by the droplet size in the mixing sections. Very little growth in the average droplet

size occurs in the packing. At a constant stirrer speed, the behavior of a Scheibel column with respect to droplet size and hold-up closely parallels a packed extraction column.

A sampling technique, developed to obtain the concentration of both phases inside the operating extractor, was used to obtain concentration profiles along the column. For the system, methyl isobutyl ketone-acetic acid-water, approximately 40% of the extraction took place in the packing in the normal operating range of the column.

By estimating the specific transfer surface it was possible to remove the effect of changing transfer area from the $(NTU)_{OE}$ in the packing. This showed that the stirrers produce considerable axial mixing in the packing. The axial mixing reduced the efficiency of the packing, at normal operating stirrer speeds, to approximately 50% of its efficiency at zero stirrer speed.

3. Recovery of Copper and EDTA from Rare-Earth Ion Exchange Wastes

(L. Burkhart)

The eluant solution of copper-EDTA represents a considerable fraction of the cost of separating the rare-earth elements by ion exchange. The presence of large amounts of copper also poses a potential health problem in disposal of large amounts of any industrial waste. Preliminary work was done to see if the copper-EDTA complex could be broken by hydrogenation at moderate temperatures and pressures. The ideal yield of this reaction would be metallic copper and EDTA for recycle

to the process.

Hydrogenation runs were made at 500 psig and at temperatures from 25°C to 150°C. The chelate was successfully broken and metallic copper was obtained. The recovery of EDTA has not yet been determined. The results of this preliminary work appear promising, however.

4. Preparation of Yttrium Fluoride (R. Tischer and G. Burnet)

A report (IS-8), "Preparation of Yttrium Fluoride Using Fluorine", by R. Tischer and G. Burnet was distributed.

Abstract--This report deals with the preparation of a low oxygen content yttrium fluoride using commercial fluorine gas. It was shown that the purity of yttrium fluoride prepared using hydrogen fluoride gas could be improved by treating it with fluorine (1) at a temperature of 600°C under pressures in the range 20-25 psig or, to a lesser degree, (2) at 25°C and 1 atmosphere pressure.

Preparation of yttrium fluoride by direct fluorination of yttrium oxide was found to be a spontaneous process which ceases short of equilibrium under the conditions studied. Fluorination of yttrium chloride resulted in a more complete conversion than that obtained with the oxide, but the degree of conversion was not high enough to be of interest.

Calculations made using the best data available at this time show the reaction of yttrium oxide with fluorine gas is a highly exothermic reaction which would be expected to proceed spon-

taneously to almost complete conversion. A theory is presented which attempts to explain why this did not occur under the conditions imposed.

It was demonstrated that fluorine gas supplied in pressure cylinders can be safely handled in a laboratory hood under the conditions cited above if proper precautions are taken.

5. Effect of Vapor Properties on Entrainment from Bubble Cap Trays

(G. Burnet)

Entrainment from bubble cap trays and similar liquid-vapor contacting apparatus is frequently a serious problem in the processing of radioactive materials. The purpose of this project is to develop an improved correlation for predicting entrainment from bubble cap trays.

All correlations now available have neglected the effect of vapor viscosity and to some extent approximated the effect of vapor density and velocity through the tray. In this investigation the rate of entrainment was studied for five gases with varying properties using 12- and 18-inch tray spacings in an eight-inch-diameter single bubble cap per tray column. It was found that the viscosity of the gas significantly affected entrainment rate in the 18-inch tray spacing but did not have an effect on entrainment where the 12-inch tray spacing was used. This difference was accounted for by noting that frictional transfer of momentum between vapor and droplets occurs in the turbulent flow region for the former tray spacing and in the intermediate flow region for the latter. Two vapor parameters were derived from an idealized mechanism of a

single droplet suspended in the vapor--first in turbulent and then in intermediate flow. The two parameters correlated the entrainment data in an excellent manner.

Future work calls for a repetition of the experiments conducted so far, but at a third tray spacing. This will serve to confirm the data thus far accumulated.

6. A Forced Convection Loop for Circulating Liquid Lead-Bismuth Mixtures

(G. Burnet)

The goal of this project was the design, construction, and operation of a forced convection loop for circulating liquid metals. The loop will be used in the future to study various pyroprocessing operations such as heat transfer, fluid flow, and vapor-liquid equilibria. Various phases of the project are discussed individually below.

(1) Dynamic Corrosion Tests. Previous tests have shown type 430 stainless steel and 2 1/2% Cr - 1% Mo steel to offer the greatest resistance to corrosion by the lead-bismuth eutectic. Further tests have been run with these materials to determine more about the mechanism of corrosion. Several 500-hour runs have demonstrated that the Cr goes into solution very slowly while the Fe from the steel reached saturation within 100 hours. This supports the theory that the corrosion mechanism is controlled by solid state diffusion after an initial short period of surface attack. These tests were run in the temperature range of 550 to 900°C. The effect of various surface treatments on the corrosion of type 430 stainless steel

by the eutectic was investigated. It was found that pickled, nitric acid passivated, or air oxidized surfaces had a negligible effect on corrosion resistance.

When the eutectic was saturated with Fe or C, a negligible effect on corrosion resistance of type 430 stainless steel was noted. When the eutectic was saturated with Cr, a substantial reduction in corrosion was observed. This indicates that the removal of Cr from solid solution is at least partly responsible for the corrosive action.

(2) Thermal Convection Loops. During this report period a type 430 stainless steel loop was operated for a total of 4400 hours, with a hot leg temperature of 520 °C and a cold leg of 410 °C. The loop was used to observe corrosion resistance and to test three different types of flanged joints. Observations resulting from this work confirmed the degree and nature of attack of 430 stainless steel by the eutectic observed in the preceding dynamic corrosion tests.

(3) Forced Convection Loop. The forced convection loop containing a new type of liquid metal pump has been in continuous operation for 3500 hours. The pump is of a diaphragm type and is operating at a rate of 1 gpm. The hot leg temperature is held at 500 °C and the cold leg at 370 °C.

7. Development of a Liquid Metal Cooled Condenser (G. Burnet)

In the distillation of liquid metals, a condenser is required that is capable of condensing metal vapor in a liquid (molten) form. The

liquid thus produced can then be returned to the distillation apparatus as reflux or removed as a product.

The unit designed and tested as a part of this project is cooled by boiling NaK. By controlling the pressure of the NaK in the condenser, the temperature of the condenser surface can be controlled. The unit was shown to give highly reproducible condensing surface temperatures and when tested in a simple distillation apparatus gave the results in the table below:

Table 1.

Results of Runs Made with the Stainless Steel Distillation Unit Using
Pure Bismuth as the Test System

Pressure of system (microns)	Temperature °C		Length of run (hours)	Product Obtained (grams)
	Boiler	Condenser		
0.08	660	482	22.0	2.3
0.10	671	548	22.0	4.3
0.08	671	571	21.5	6.7
0.10	671	593	21.5	7.1

8. Vaporization of Liquid Metals (G. Burnet)

This project is concerned with a study of the mechanism of vaporization of binary liquid metal mixtures. The information gained will be

used in the design of a boiler for use in the vacuum distillation of metals.

The test system now being used is bismuth-antimony. The enrichment factor has been shown to be principally a function of temperature and composition. The factor varies with temperature and has been found to reach a maximum when the liquid metal composition is nearly 50% bismuth and 50% antimony. The factor falls off to near the theoretical value of unity as the composition of the mixture approaches either pure bismuth or pure antimony.

A theory to explain these phenomena has been proposed and is based upon the presence of Sb_4 which is significantly more volatile than the more common species, Sb_2 .

9. Effect of Inhibitors on the Corrosion of Stainless Steel by the Lead-Bismuth Eutectic (G. Burnet)

The effect of ten additives, including vanadium, magnesium, zirconium, and cerium in inhibiting the corrosion of stainless steel by the lead-bismuth eutectic is being investigated. These are added in concentrations of 100 to 300 parts per million. Their effect is being studied in a dynamic corrosion test apparatus. All tests are being conducted at 700°C and the results assessed using X-ray diffraction of the specimen surface, photomicrographs, and mass spectrometer analysis of the eutectic.

Preliminary results indicate the formation of various nitrides that are effective in inhibiting the degree of surface corrosion.

10. Liquid Metal Extraction (E. Olson)

Report IS-3, "Extraction Equipment and Sampling Technique Used in Semi-Continuous Silver-Uranium Extraction Studies", by Edwin H. Olson, Morton Smutz and Clayton W. Watson was distributed.

Abstract--Aqueous solvent extraction methods for reprocessing metallic fuels have two main disadvantages. First, the process involves numerous steps such as dissolution in acid, extraction, conversion to a salt, and reduction to the metallic state. Secondly, relatively large volumes of radioactive solutions are obtained.

This report describes small scale, semi-continuous extraction runs for extracting irradiated neodymium from a molten uranium-neodymium alloy, using droplets of molten silver at 1250° and 1290°C. Two different silver drop sizes and flow rates were used. The graphite extractor, silver metering device, and the extract sampling equipment are described.

The experimental extraction data obtained are presented. These data were correlated reasonably well with equations developed for multistage batch extractions. The actual neodymium concentrations obtained were compared with equilibrium data to determine extraction efficiencies for the various runs. Extraction efficiencies of 15% to 35% were found, with higher efficiencies resulting when large-size drops were used. In these runs, a considerable amount of neodymium was apparently re-

moved by carbide formation with the container wall prior to the extraction with silver.

The objective of current research on this project is to obtain equilibrium data for the system uranium-silver-neodymium at 1250°C, and to perform a simulated two-stage batch counter-current extraction. The data obtained should permit a better evaluation of this method of reprocessing reactor fuels than has been possible previously.

11. Two Phase Disperser (E. Olson)

Although the two phase disperser described in a previous report (ISC-1051) was originally considered for use with liquid metals, it is at present being investigated for use with conventional aqueous-organic liquid-liquid extraction systems.

Three simple extraction systems with widely varying physical properties have been tested to date. Variation in disperser speed, solvent to feed ratio, interface level and extractor throughput have been found to have little effect on the over-all extractor efficiency. The two phase disperser has been found capable of attaining between 1.35 and 1.65 theoretical extraction stages, depending upon the system used. A recent modification of the two phase disperser has been built and will be tested in the near future.

12. Preparation of Yttrium Fluoride (E. Olson)

Report IS-2, "Preparation of Yttrium Fluoride Using Ammonium Bifluoride" by John Walker and Edwin Olson was distributed. Following

is a summary of the results.

The use of ammonium bifluoride as a hydrofluorination agent in the preparation of low oxygen content yttrium fluoride was studied as a possible alternate for anhydrous hydrofluoric acid. The starting compound was yttrium oxide and the two main variables investigated were reaction temperature and mole ratio of ammonium bifluoride to yttrium oxide. Graphite crucibles and an inert gas sparge to remove the oxygen-bearing gases formed in the reaction were employed. The effects of the use of a vacuum, and the effects of particle size on the oxygen content of the product yttrium fluoride were also studied.

In small scale runs, each of the following three sets of conditions resulted in a reasonably low oxygen content yttrium fluoride:

- (1) Nine moles of ammonium bifluoride per mole of yttrium oxide, reacted for 2 hours at 130°C and then heated to 400°C to drive off the excess ammonium bifluoride.
- (2) Seven moles of ammonium bifluoride per mole of yttrium oxide reacted for 2 hours at 230°C and then heated to 400°C to drive off the excess ammonium bifluoride.
- (3) Seven moles of ammonium bifluoride per mole of yttrium oxide reacted at 100° to 115°C for 2 hours under a vacuum to remove water, ammonia and other reaction products followed by raising the temperature to 400°C to remove the excess ammonium bifluoride.

Duplicate runs, scaled up by a factor of 18, were made, employing the conditions given in (1) and (2) above. No adverse affect on the oxygen content of the product fluoride was noted. It was noted that longer times were required to remove the excess ammonium bifluoride. Graphite crucibles were found to be satisfactory.

Yttrium metal prepared from the yttrium fluoride made in the large scale run contained approximately 2500 ppm oxygen.

13. Preparation of Sodium Ethyl Sulfate (E. Olson)

Sodium ethyl sulfate is used in the preparation of various rare-earth ethyl sulfates which are then grown into single crystals for use in studies of electronic structure.

The current process for preparation of sodium ethyl sulfate is as follows:

- (1) Preparation of ethylsulfuric acid by reacting concentrated sulfuric acid with absolute alcohol
- (2) Addition of sodium carbonate to convert the ethylsulfuric acid to sodium ethyl sulfate and neutralize the unreacted sulfuric acid
- (3) Separation of the alcoholic sodium ethyl sulfate solution from the slurry by settling and centrifugation
- (4) Crystallization of hydrated sodium ethyl sulfate from the alcohol solution by freezing.

Because sodium ethyl sulfate and ethylsulfuric acid solutions decompose readily on heating, all operations must be carried out at or below

room temperature.

14. Growth of Single Crystals (E. Olson)

Single crystals of zinc, tin, lead and silver chloride have been grown with a simple, low cost, Bridgman-type crystallizer. The unit consists of two identical furnaces with powerstats to control the temperatures, a sample lowering assembly, a crystallizing crucible assembly, all of which are contained in an angle-iron frame measuring 21 inches wide by 14 inches deep by 25 inches high. In growing single crystals with this unit, a sample of the material is first melted in the crystallizing crucible suspended in the upper furnace. The sample is then lowered at a constant rate into the lower, colder furnace. As the sample enters the colder furnace the molten sample in the tip of the crucible nucleates many small crystals. Generally one nucleus, because of favorable crystallographic orientation, grows faster than the others and crowds out the other nuclei as the sample is lowered. This results in one large, single crystal.

Nuclear Engineering

1. Slurry Program (G. Murphy)

The use of lead particles to study the coring phenomenon in an aqueous slurry containing relatively heavy particles was found unsatisfactory because of oxidation of the lead. The possibility of using other fluids was investigated and experiments were run using the most attractive fluids. Of these, bromobenzene was the most satisfactory, but it proved impossible to obtain coring of lead particles 0.0014 in. in diameter within the limits of variables investigated.

Study of the glass-water system was resumed. A finger pump was installed to reduce surges that adversely affect coring. Work is now being conducted on the improvement of photographic techniques in order that definitive measurements of coring may be made.

2. Engineering Properties of Materials (G. Murphy and R. E. Uhrig)

2.1 High-Temperature Testing Facilities

Development of the high-temperature extensometer using linear variable differential transformers has continued and calibration tests show that a continuously variable multiplication ratio up to 4000 is possible. The instrument has a range of 1/4 in.

Tensile stress-strain diagrams were obtained using the LVDT extensometer on uranium at temperatures up to 600°C. Tensile stress-strain diagrams were obtained on tantalum at temperatures up to 1000°C.

Report IS-48, "A Method of Specimen Corrosion Protection for High Temperature Creep Testing", by J. R. Bohn, R. E. Uhrig and Glenn Murphy was distributed.

Abstract--The determination of mechanical properties of materials at elevated temperatures presents difficulties, particularly when the material to be tested is subject to oxidation. Various methods have been employed to permit the evaluation of high temperature creep properties. The method described in this paper was developed on the basis of modifications of a technique developed for the protection of high temperature fatigue specimens. The method involves encasing the creep specimen in a flexible capsule which is capable of withstanding exposure to the atmosphere for extended periods at temperatures up to 1000°C.

Extensive testing of materials such as uranium and tantalum has provided the basis for claims relative to the effectiveness of this technique.

Report IS-66, "An Autographic Elevated Temperature Creep Testing Facility", by Robert L. Hammel and Robert E. Uhrig was distributed.

Abstract--The problems of obtaining accurate strain data, providing reliable temperature control, and affording oxidation protection arise in the experimental determination of the elevated temperature creep properties of materials. The apparatus described in this paper provides an autographic strain record using

commercially available equipment, reliable temperature control for both constant or programmed cyclic temperatures, and oxidation protection by means of specimen encapsulation.

2.2 Creep of Tantalum

Report IS-26, "Observations on Creep of Tantalum", by Glenn Murphy and R. E. Uhrig was distributed.

Abstract--Data on the creep of tantalum in tension at approximately 800°C and 20 ksi indicate an activation energy for creep of 109,600 cal/gm-mole. The stress parameter was evaluated directly from the test data as 5.65×10^{-4} in.²/lb, and from a plot of the Zener-Holloman parameter against stress as 5.81×10^{-4} in.²/lb.

It was observed that the third stage of creep consisted of an interval in which both the strain and strain rate varied exponentially with time followed by an interval, terminating in failure, in which the creep rate increases rapidly. Special high temperature test equipment was used to provide an argon atmosphere around the test specimens.

Report IS-125, "Creep of Tantalum Under Cyclic Elevated Temperatures", by Robert L. Hammel and Robert E. Uhrig was distributed.

Abstract--An initial study was conducted on the effects of cyclic temperature variations of small amplitudes upon the creep properties of tantalum. The results show a marked weakening of the material when a temperature variation of a few degrees is applied in a sinusoidal manner at a rate of one cycle per hour.

Application of a temperature compensated time parameter for the prediction of the cyclic temperature curves is inadequate to explain the observed increase in creep rate. With vacuum arc-cast tantalum, possessing high initial cold work, relief of the residual cold work strain appears to contribute to the acceleration of the strain rate as evidenced by a reduction in hardness after the cyclic temperature application. Sintered tantalum showed a similar acceleration of the creep rate under cyclic temperatures without a change in hardness. Experimental results indicate that an optimum temperature condition may exist for plastic deformation to occur under cyclic temperatures.

Report IS-138, "A Proposed Model for High Temperature Creep of Tantalum", by Robert E. Uhrig was distributed.

Abstract--A model for the creep of tantalum at elevated temperature has been presented in which the increase in strain rate with strain is related to the reduction of the cross-sectional area accompanying strain. Comparison of experimental data with the proposed theory indicates that the dominant creep mechanism beyond the second stage of creep starts at a point of highest temperature or at a stress concentration, spreads throughout the volume in the immediate vicinity, and eventually results in localized "necking down".

3. Hot Loop Studies (R. Fisher)

3.1 Dynamic Tests

Tests were performed at 800°-1000°C on yttrium, niobium, and tantalum loops, circulating thorium-magnesium and uranium-chromium eutectics. The preparation of very pure niobium metal by the electron beam technique made it possible to contain thorium-magnesium successfully, where previous tests with commercial grade material had failed.

3.2 Development of Electromagnetic Pump

The performance characteristics of the electromagnetic pump can be improved in several ways. Side bars brazed to the outside edges of the pump section are analogous to the end rings in the rotor of an induction motor and provide a low resistance path for eddy current flow in the direction of fluid flow. However, external side bars are not practical as a means of improving the performance characteristics of the linear induction pump. The lack of a metallurgical bond between the tantalum pump duct wall and the inconel sheath of the pump section plus the high resistivity of the inconel sheath would limit the effectiveness of the external side bars. In addition, a low resistivity side bar material capable of operating in the atmosphere without oxidation at high temperature and a brazing material with adequate strength at 1000°C would be required.

Table I. Effect of Side Bars at Constant Voltage

Side Bar	Voltage, Volts	Phase Current, Amperes	Power, Watts	No-flow pressure psi
None	100	9.1	160	0.119
Chrome-Plated Cu	100	9.1	170	0.249
Cu	100	9.2	210	2.330

Data on the chrome-plated Cu side bars illustrate the effect of non-wetting between the copper and mercury. The pressure generated at zero flow is double that without side bars, but wetting provides a 20-fold pressure improvement. This illustrates the value of decreasing the voltage drop and resistance to eddy current flow in the fluid flow direction. With the voltage drop a minimum in this direction the voltage drop across the duct increases, producing larger eddy currents and higher pressure. The use of the wetted copper electrodes increased the pumping efficiency from 0.010% to 0.18% with the 20-fold pressure improvement.

The effect of the side bars can be partially obtained by increasing the cross-sectional area of the pump duct. This decreases the eddy current density and resistance to eddy current flow. The increase in pressure resulting from the increase in pump duct width and cross-sectional area is shown in Table II.

Table II. Effect of Pump Duct Width

Pump Duct Width/ Pump Width	Duct Thickness Inch	Voltage Volts	Current, Amperes	Relative No-Flow Pressure
0.765	0.250	175	8.80	1.00
0.988	0.250	175	8.03	1.29
1.361	0.250	175	7.80	1.83

Thus, the no-flow pressure rises in direct proportion to the increase in pump duct width and cross-sectional area.

The instantaneous flux distribution in the air gap of the pump has been determined. The current drawn by each coil during three phase operation was individually supplied from a single phase source. The instantaneous flux distribution was then determined from the magnitudes of the individual flux distributions, the phasing, current direction in the coils at the instant of time, and other considerations. The instantaneous flux distribution, as shown in Fig. 1, for four successive instants of time is not sinusoidal in nature. This is because each phase winding is one concentrated coil and is not distributed over the stator surface. Between successive instants of time the field travels the distance from one pole face to the next, or two inches. This is equivalent to a velocity of 60 feet per second. The area bounded by the flux distribution curve is nearly constant, indicating that the effective value of flux density remains nearly constant with time as the field travels uniformly at 60 feet per second.

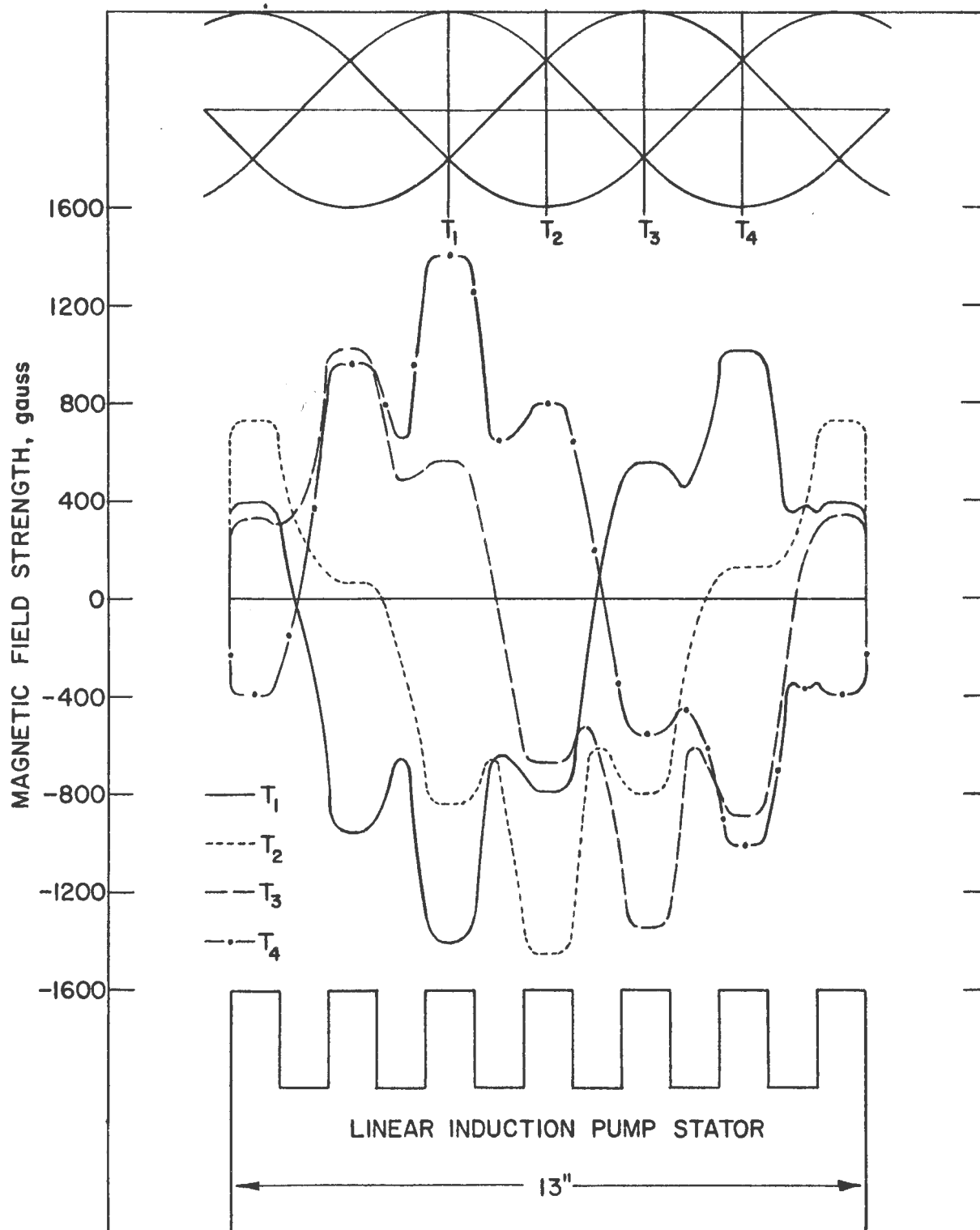


Fig. 1. Instantaneous flux distribution at 100 volts (76 laminations, 0.443-inch air gap)

The value of B^2 , (flux density)², obtained by squaring each ordinate of the instantaneous flux distribution curve is $0.518(10^6)$ gauss². The value of B^2 from the three phase flux curve is $1.041(10^6)$ gauss². Thus, the effective value of B^2 is exactly one-half of that obtained from the three phase flux distribution and the effective value can be determined from three phase measurements without resorting to individual phase measurements and field calculations.

The ideal pressure generation for the 76-lamination pump at 100 volts with a 0.250 inch duct thickness and 0.443 inch air gap is 6.04 psi. The actual pressure produced was 2.33 psi which is 40% of that predicted from ideal theory.

Ceramic Engineering

1. Sintering Mechanism of Uranium Dioxide (D. R. Wilder)

Report IS-154, entitled "Roles of Nb_2O_5 , U_2O_5 , and TiO_2 in the Grain Growth and Sintering of UO_2 ", by James F. Watson and D. R. Wilder was distributed.

Abstract--The sintering characteristics of UO_2 , UO_2 with 0.1% V_2O_5 , UO_2 with 0.4% Nb_2O_5 and UO_2 with 0.1% TiO_2 were studied under hydrogen atmosphere. The UO_2 and the UO_2 with TiO_2 were found to sinter by classic diffusion mechanisms. The non-stoichiometry of the UO_2 and the contribution of excess oxygen from the additives were found to be important factors contributing to the sintering rate.

2. Uranium Glasses (D. R. Wilder)

Report IS-107, entitled "Uranium Glasses. I. Fundamental Considerations", by C. D. Wirkus and D. R. Wilder has been distributed.

Abstract--The principles involved in forming glasses containing uranium are discussed. Materials having low thermal neutron cross sections that could be useful components in a uranium glass are listed and their properties are discussed.

Report IS-158, the second phase of this three part investigation, entitled "Uranium Glasses. II. Uranium Silicate Glasses", by C. D. Wirkus and D. R. Wilder was also distributed.

Abstract--The maximum concentration of uranium dioxide (45 w/o) was determined for silicate glasses. A number of stable glasses were developed. The properties of a typical high-uranium-content silicate glass are discussed.

The third and final phase concerns phosphate glasses and is still under investigation. An area of glassy stability has been found to exist in the $\text{UO}_2\text{-P}_2\text{O}_5\text{-SiO}_2$ ternary. No outstanding properties have been found in the glasses in this system.

3. Protective Coating for Yttrium (D. R. Wilder)

A new technique has been developed for applying a glassy, impervious coating of glass to yttrium. The detrimental oxide films which are formed at the interface during or prior to the firing operation are dissolved by the glass. A glass containing an appreciable amount of cerium oxide has been found most suitable for this task, giving good protection to yttrium in air to about 900°C.

4. Sintering Mechanism of Refractory Oxides (D. R. Wilder)

As annotated bibliography, ISC-288, Supplement II, "Sintering Mechanism as Applied to Refractory Oxides, 1955-1959", by D. R. Wilder was distributed.

APPENDIX I: LIST OF REPORTS FROM THE AMES LABORATORY

1. Reports for Cooperating Laboratories

- ISC-1076 H. A. Wilhelm. A History of Uranium Metal Production in America.
- ISC-1095 Lawrence Burkhart and R. W. Fahien. Pulse Column Design.
- ISC-1115 Ames Laboratory Staff. Physics. Semi-Annual Research Report. July-December, 1958.
- ISC-1116 Ames Laboratory Staff. Chemistry. Semi-Annual Research Report. July-December, 1958.
- ISC-1173 William S. Rawls and L. J. Laslett. The Determination of Photoneutron Thresholds.
- IS-1 C. V. Banks, O. N. Carlson, A. H. Daane, V. A. Fassel, R. W. Fisher, E. Olson, J. E. Powell and F. H. Spedding. Studies on the Preparation, Properties and Analysis of High Purity Yttrium Oxide and Yttrium Metal at the Ames Laboratory.
- IS-2 John Walker and Ed Olson. Preparation of Yttrium Fluoride Using Ammonium Bifluoride.
- IS-3 Edwin H. Olson, M. Smutz and Clayton W. Watson. Extraction Equipment and Sampling Technique Used in Semi-Continuous Silver-Uranium Extraction Studies.
- IS-8 G. Burnet and R. Tischer. Preparation of Yttrium Fluoride Using Fluorine.
- IS-14 Ames Laboratory Staff. Physics. Semi-Annual Summary Research Report. January-June, 1959.
- IS-15 Ames Laboratory Staff. Chemistry. Semi-Annual Summary Research Report. January-June, 1959.
- IS-16 Ames Laboratory Staff. Engineering. Semi-Annual Summary Research Report. January-June, 1959.
- IS-17 Ames Laboratory Staff. Metallurgy. Annual Summary Research Report. July, 1958-June, 1959.
- IS-26 Glenn Murphy and Robert E. Uhrig. Observations on Creep of Tantalum.
- IS-47 O. N. Carlson and A. L. Eustice. Vanadium-Chromium Alloy System.

- IS-48 J. R. Bohn, R. E. Uhrig and Glenn Murphy. A Method of Specimen Corrosion Protection for High Temperature Creep Testing.
- IS-50 R. Kniseley, V. A. Fassel and F. Lentz. Misidentifications in the Arc Spectra of the Rare Earths.
- IS-57 D. H. Dennison, F. H. Spedding and A. H. Daane. The Determination of the Melting Point, the Vapor Pressure, and the Decomposition Temperature of Yttrium Tri-Iodide.
- IS-66 Robert L. Hammel and Robert E. Uhrig. An Autographic Elevated Temperature Creep Testing Facility.
- IS-67 Toshihiro Okada. Note on the Determination of the Magnetoresistance Tensor of a Crystal Having the Symmetry O_h or O .
- IS-107 C. D. Wirkus and D. R. Wilder. Uranium Glass I. Fundamental Considerations.
- IS-115 O. N. Carlson, F. A. Schmidt and R. L. Wells. A Study of the High-Temperature Air-Oxidation of Yttrium Metal.
- IS-125 R. L. Hammel. Creep of Tantalum Under Cyclic Elevated Temperatures.
- IS-138 Robert Uhrig. A Proposed Model for High Temperature Creep of Tantalum.

2. Publications

- Atoji, M. and R. C. Medrud, The Structures of Calcium Dicarbide and Uranium Dicarbide by Neutron Diffraction. *J. Chem. Phys.* 31, 332-337 (1959).
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APPENDIX II: LIST OF SHIPMENTS

<u>Destination</u>	<u>Item</u>
University of Arizona General Stores Building Fifth Street and Mountain Avenue Tucson, Arizona	5 gm ytterbium oxide
Monsanto Chemical Company Mound Laboratory Miamisburg, Ohio	1 lb cerium metal
University of California Los Alamos Scientific Laboratory Los Alamos, New Mexico	2 metal cylinders lanthanum and cerium 50 gm lanthanum metal 50 gm neodymium metal
Michigan State University P. O. 39530 East Lansing, Michigan	20 gm cerium metal
H. B. Nicholas B-11 Research Building Iowa State University Ames, Iowa	10 gm yttrium oxide 10 gm lanthanum oxide 10 gm cerium oxide 10 gm neodymium oxide 10 gm dysprosium oxide 4 gm samarium oxide 2 gm gadolinium oxide 2 gm erbium oxide 2 gm ytterbium oxide 1 gm praseodymium oxide 1 gm holmium oxide 1 gm thulium oxide 1 gm terbium oxide 1 gm lutetium oxide
Mr. Sebastian Amer Box 122, Station A Ames, Iowa	20 gm Er_2O_3
Dr. Milton Burton Department of Chemistry University of Notre Dame Notre Dame, Indiana	10 gm Dy_2O_3 25 gm La_2O_3 25 gm Gd_2O_3 25 gm Y_2O_3 4 gm Lu_2O_3
Mr. Pierre Pelen French Embassy 2535 Belmont Road N. W. Washington, D. C.	samples of uranium metal (natural)

<u>Destination</u>	<u>Item</u>
University of California Receiving Department 2000 Carleton Street Berkeley 4, California	25 gm ytterbium metal 25 gm thulium metal 50 gm samarium metal
Dr. Masao Atoji Oak Ridge National Laboratory Oak Ridge, Tennessee	30 gm lutetium metal 30 gm ytterbium metal 30 gm terbium metal 2 glass vials of thorium carbide-thorium deuteride x-ray powder samples 46 gm lutetium carbide 110 gm thorium carbide 42 gm ytterbium dicarbide 30 gm Lu_2O_3 30 gm Yb_2O_3
Dr. Richard J. Weiss Materials Research Laboratory Ordnance Materials Research Office Watertown 72, Massachusetts	1 pc gadolinium metal appx. 10cm x 5cm x 0.1cm
Iowa State University Room 142 Research Building Ames, Iowa	200 ml 5% rare-earth chloride solutions: YCl_3 , NdCl_3 , GdCl_3 , DyCl_3 and YbCl_3 1500 ml 5% gadolinium chloride solution
J. C. Jamieson R. 21, 5641 S. Ingleside Avenue Chicago 37, Illinois	100 gm cylinder cerium metal
Mr. Warren DeSorbo General Electric Company Schenectady, New York	50 gm crystal bar vanadium
Dr. Glenn A. Crosby University of New Mexico Albuquerque, New Mexico	10 gm Sm_2O_3 10 gm Dy_2O_3
Mr. Dave Drennen Battelle Memorial Institute Columbus 1, Ohio	50 gm crystal bar hafnium metal
Mrs. Mary E. White Massachusetts Institute of Technology Cambridge 39, Massachusetts	1 gm yttrium metal

<u>Destination</u>	<u>Item</u>
Professor Paul A. Beck University of Illinois Urbana, Illinois	100 gm crystal bar wire appx. 1/16" diameter vanadium
Phillips Petroleum Company Atomic Energy Division Idaho Falls, Idaho	18 pc. 1" x 1/2" x 1/8" yttrium metal
Argonne National Laboratory Bailey Road Lemont, Illinois	250 gm distilled yttrium metal
Acct. Prop. Affr. Warehouse 335 Fort Belvoir, Virginia	5 gm praseodymium metal 5 gm samarium metal 5 gm neodymium metal 5 gm dysprosium metal 5 gm terbium metal 5 gm gadolinium metal 5 gm holmium metal
Veterinary Physiology & Pharmacology Room 100, Vet Physiology Ames, Iowa	1200 ml 5% dysprosium chloride solution 0.5 gm gadolinium oxide 0.5 gm dysprosium oxide 0.5 gm ytterbium oxide 0.5 gm yttrium oxide
Brookhaven National Laboratory Warehouse 100 Upton, L. I., New York	3 pc. thulium metal
Mr. W. E. Jardner Atomic Energy Research Establishment Harwell, Didcot Berks, England	5 gm crystal bar vanadium
Dr. Alvin Boltax Nuclear Metals Inc. Concord, Massachusetts	1 - 1" x 0.35" x 0.03" thorium metal