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Report No. BMI-532

PROGRESS REPORT

on

ZIRCONIUM PILOT PLANT
RESEARCH AND DEVELOPMENT

to

ATOMIC ENERGY COMMISSION
CONTRACT NO. AT(30-1)-771B

by

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D. M. Rosenbaum, H. F. Ross, and I. E. Campbell

Classification cancelled by authority of

Letter from H. R. Carroll, USAEC

on *12/28/56*

(Signed) *J. Garrett*

(Date) *1/4/57*

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November 20, 1951

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SUMMARY

Installation of a Hilco oil purifier for the vacuum pumps permitted continuous operation of the vacuum pumps in the cyclical-flow pilot plant during the past month. Raising the vaporizing salt temperature gave a means of increasing the iodide flow rate nearly 100 per cent above the design capacity of 120 pounds per hour.

Two single-filament runs, each of four cycles duration, produced some data for a study of the effect of flow rate and deposition pressure on the rate of zirconium deposition. Pressure-drop measurements were used to predict flow rates. A maximum production rate of 0.05 pounds

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per hour per foot occurred at a pressure between 2 and 3 millimeters, using a pressure range between 1 and 8.5 millimeters to study this effect. Based on flow and deposition rates, decomposition efficiencies ranged from 0.9 to 13.7 per cent in this plant as compared with 17 to 28 per cent for the 16-inch bulb. Plots of the data show that these differences cannot be explained by pressure and flow rate alone. The low plant rates might be caused by:

- (1) inadequate separation of iodine from iodide,
- (2) unknown catalytic effects from undesirable air leakage into the system, and
- (3) poor flow patterns in the large diameter bulb.

This latter point has been confirmed this month by the nonuniform deposition on the filaments of a multiple-hairpin array.

Experimental work on this plant was terminated on November 9. Material in the iodine condensers was melted and recovered. These condensers were dismantled and washed thoroughly. The iodide condensers were flushed with water after attempting to remove condensed iodide with no success. The plant has thus been freed of all corrosive and toxic materials and it is ready for further operation or complete dismantling.

Zirconium tetraiodide, containing some free iodine, was produced from zirconium carbide in the internally heated furnace. A rate of 267 pounds of ZrI_4 per hour per square foot was obtained with an operating temperature of 1000 to 1100°C. On the basis of this result,

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the production of zirconium tetraiodide from its carbide at good rates in an internally heated furnace has been demonstrated. Recommendations were made to modify the furnace for better operation on a large scale.

A small pilot plant for measuring sublimation and deposition rates was erected and operated for one week. In this time, it was demonstrated that a vibrating flash plate gives excellent heat transfer for sublimation. Deposition rates obtained were poor. This was attributed to air leaks around the screw-feed shaft and outgassing of the powder. With some modifications, this type of pilot plant seems ideal for obtaining fundamental data on the iodide-flow process at a low research cost.

INTRODUCTION

Previous deposition runs in the cyclical-flow pilot plant had produced low deposition rates of 0.05 pounds per hour per foot or less. Low flow rates were thought to be responsible, as interpreted from 16-inch-bulb experiments. To increase the flow rate in the pilot plant, provisions were made to increase the fin and wall temperatures.

To maintain trouble-free vacuum pumping operation, a Hilco purifier was installed. The plant was then ready for studying the effect of velocity and pressure of zirconium tetraiodide on the decomposition reaction for single and multiple filaments.

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PLANT OPERATION

Deposition Runs

Before interpreting the results of deposition runs made between October 15 and November 8, the method of predicting flow rates will be discussed.

Flow-Rate Measurements

The original design for measuring gas flow in the plant called for a hot-wire anemometer. This device was tested and found to be unreliable because of the changing ambient salt temperature and low flows of gas. Work had been started in the Electronics Division at Battelle to make a Hastings-type thermopile gage. This instrument was insensitive to ambient temperature changes but very sensitive to low flows. There was no opportunity to test this gage because of the termination of the present contract.

To obtain a fairly reliable estimate of the flow rate, pressure-drop data were obtained by measurement of pressures with Dubrovin gages at the vaporizer, deposition bulb, and condenser. Argon gas was pushed into the pressure tap every 15 minutes to prevent back-diffusion and condensation of ZrI_4 . Pressure-drop calculations as a function of flow rate for the process piping between the vaporizer and condenser were made. Provided the condenser pressure was less than 1 mm., the flow of ZrI_4 was computed as $W = 12.3 p_1 - 10$

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where W = the flow of ZrI_4 in pounds per hour and p_1 = upstream or vaporizer pressure in millimeters of Hg.

There is a degree of uncertainty in these calculations, because the viscosity of ZrI_4 gas is not precisely known. Unless the gas is associated, it is felt that the above equation should predict flow rates within ten per cent, because large changes in viscosity produce minor changes in the friction-factor estimate required for pressure-drop calculations.

Multiple-Filament Run

A 42-foot filament was heated for over 8 hours before burn-out, the longest operation attained on a multiple filament in this plant. The gas flow was upward and no metal deposition was observed until the bulb pressure dropped to 2.5 mm. and $K/cm.$ was at a high value of 0.8. The rate for the metal-depositing period was 0.75 pound per hour or 0.017 pound per hour per foot. Since the flow rate was estimated as 52 pounds ZrI_4 per hour, the decomposition efficiency was 9.6 per cent.

Inspection of the filament hairpins showed uniform deposition on all filaments except the two fastened to the electrodes where the burn-out occurred. These two filament segments showed a gradual taper from bottom to top over a 4.5 foot length. This was a sign that the flow pattern in the bulb was not uniform.

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Single-Filament Runs

Two runs of four cycles each were made in which wide variations in flow rates and pressures were achieved. The results are listed in Table 1, together with calculated metal-production rates and efficiencies. Because of the gradual decrease in pressure over a cycle, it was necessary to use integrated average values in computing the pressure data shown in Table 1. A typical plot of the pressure drift of one cycle is given in Figure 1.

The pressure drift was probably caused by the increasing difficulty of vaporizing iodide which had spalled off during the early stage of the heating cycle when material adjacent to the fins was rapidly vaporizing.

On the last cycle of Run D6S, the temperature of the fin tubes was 425°C. This gave sufficiently high heat-transfer rates, thus allowing a throttling of the vaporizer exit valve to maintain constant deposition-bulb pressures at levels of 8.5 and 2 mm. of Hg successively.

The rate of deposition is plotted as a function of bulb pressure and mass velocity in Figure 2. The Reynolds number criterion was not used in this analysis, because the diameter of the wire was not the controlling variable in the set of experiments. Unfortunately, the pressure-velocity variables cannot be separated, using process equipment where the piping constricts the flow. Although the number of experimental points are limited, there appears to be a maximum deposition

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TABLE 1. SUMMARY OF RESULTS OF DEPOSITION RUNS D5S AND D6S

Cycle No.	Direction of Gas Flow	Pressures—mm.		Flow Rate		Estimated Reynolds Number, DG/μ	Zirconium Production Rate lb. hr. ⁻¹ ft. ⁻¹	Decomposition Efficiency, per cent
		Vaporizer	"D" Bulb ^f	ZrI ₄ lb./hr.	Mass Velocity lb. hr. ⁻¹ ft. ⁻² G			
Run D5S								
1a	Down	4.8	2.9	51	23.4	11.3	0.060	9.4
1b	Down	3.8	2.0	37	17.0	10.4	0.0447	9.7
1c	Down	2.0	1.1	20	9.2	5.5	0.0342	13.7
2	Up	5.8	3.2	64	29.4	12	0.0267	3.3
3	Down	3.7	2.1	38	17.4	—	0.0447	9.4
4	Up	d	d	d			0.0304	—
Run D6S								
1	Down	5	3	54	24.8	11.7	0.0459	6.7
2	Up	8.5	4.7	97	44.5	—	0.0322	2.5
3	Down	6.2	3.8	68	31.2	—	0.0412	4.7
4a	Up	e	8.5	197	90.5	36.5	0.0223	0.9
4b	Up	e	2	78	25.8	14.8	0.0356	3.5

d Pressure readings not reliable.

e Valve from vaporizer throttled, flow estimated from "D" bulb pressure.

f Condenser pressure was lower than 1 mm.

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FIGURE 1 TYPICAL PLOT OF PRESSURE AND CONDUCTIVITY DATA FOR A DEPOSITION CYCLE

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FIGURE 2 DEPOSITION RATE VERSUS MASS VELOCITY AND BULB PRESSURE ON SINGLE FILAMENTS

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rate for this plant at bulb pressures between 2 and 3 mm. Pressure is the controlling variable beyond this 2 to 3 mm. range, even though the iodide flow increases. When the system operates at lower pressures, the iodide flow is the predominate variable.

Downflow operation was consistently better than upflow under comparable conditions of flow and pressure. This is a clue that the flow pattern in the large-diameter deposition bulb may not be uniform.

Decomposition efficiencies ranged from 0.9 to 13.7 per cent. These were much lower values than the 17 to 28 per cent efficiencies obtained in the 16-inch-bulb experiments*. Low zirconium production rates and poor efficiencies in the cyclical-flow plant as compared to the 16-inch bulb cannot be explained in terms of pressure or iodide-flow rates (see Figure 2 for comparison of data). Trouble with the plant rates may be caused by (1) poor flow patterns in the larger bulb, (2) inadequate separation of iodine from iodide, or (3) unknown catalytic effects from undesirable air leakage into the system. The project was terminated before these possibilities could be investigated.

Hardness data as well as oxygen and nitrogen analyses on these two runs are listed in Table 2.

The nitrogen-oxygen ratio is close to that of air so that the salt leaks were not prevalent in these runs. NO_2 was not exhausted at the vacuum pumps. This was further proof of the absence
* BMI-521.

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of salt leakage. Since all of the nitrogen is coming from air, the question of leak rate should be discussed on the basis of the crystal

TABLE 2. PHYSICAL AND CHEMICAL MEASUREMENTS OF TWO CRYSTAL BARS PRODUCED IN CYCLICAL FLOW PILOT PLANT

	Run D58	Run D68
Length, feet	12.0	12.42
Final weight, pounds	26	21
Final diameter, inches	1.02	0.90
Average Vickers hardness	247	243
Per cent nitrogen	0.32	0.33
Per cent oxygen	0.07	0.08

bar analyses. The nitrogen content of the bars is at least 60 times higher than specifications. Cold leak rates on the plant showed that the maximum nitrogen pick-up should not be more than 20 times the specifications, with due allowance for low-metal deposition rates. There is a possibility that air leaking into the system, principally from the valves, is chemisorbed on the iodide during standby periods at condensing salt temperatures. A cold leak-rate test would show a lower leak than actually existed. Hot leak tests always showed higher and more erratic leak rates, but this was attributed in the past to argon adsorption. The facts just presented warn that cold leak rates may not be a valid indication

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of true leakage and that low-nitrogen zirconium cannot be produced in a system containing iodide unless the leakage is held to very low values.

Vacuum Pumping System

After the Hilco oil purifier was installed, there was continued trouble-free performance during the two weeks period before the plant was shut down. This was a vast improvement over the previous record when vacuum pumps operated without a purifier. The only difficulty was the high oil-pumping rate of the worn pumps. This was reduced by replacing the brass end bearings. If the purifier was installed with new pumps, the trouble would not exist.

Dismantling and Final Clean-Up of Plant Facilities

Iodine was recovered from all of the iodine condensers. Each condenser was then removed from the plant and washed thoroughly. Attempts were made to recover the remaining iodide in the system by subliming with argon carrier gas. The difficulties involved made recovery of either the iodide or iodine uneconomical. Therefore, the iodide condensers were washed in place.

All of the corrosive and toxic compounds have been removed from the equipment, and the plant is now ready for complete dismantling, if required.

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EXPERIMENTAL PROGRAMIodination of Zirconium Carbide

Two runs were made in the internally heated furnace after covering the split graphite electrode with a quartz tube and adding additional heat to the exit line.

In the first run, which lasted four hours, only about 8 pounds of zirconium tetraiodide were produced. This product condensed as a bright red powder and not a cake, since argon was used as a carrier gas. The low production rate of zirconium tetraiodide resulted from the low flow of iodine used. The furnace operation was satisfactory, so that the next run at higher iodine flows was started immediately.

In the second run, full heat was applied to the iodine boiler. The time of the run was begun when the liquid iodine reached the boiling point. It was terminated after 2.8 hours when the bottom thermocouple registered gas superheating, meaning an empty boiler existed. The progress of iodination within the bed could be followed by about a 50° temperature rise in the reaction zone which moved progressively from the bottom inlet to the top exit. The outside-wall temperature was 1100°C. at the center, but tapered off to 900°C. at the top because of heat losses from the end plate. The results of this run are shown in Table 3. The zirconium conversion was 79 per cent, while that computed for iodine was 73 per cent, since there was an excess of iodine passed through the bed.

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TABLE 3. DETAILS OF THE IODINATION OF
ZIRCONIUM CARBIDE, RUN ZC-7InputZirconium carbide, minus 3/4 plus 1/4-inch lumps
from Bureau of Mines

Weight, pounds	12.1
Analysis	
% zirconium	89.3
% carbon	10.9
Weight as zirconium added, pounds	10.8

Iodine

Weight, pounds	—*
----------------	----

Output

Zirconium tetraiodide

Weight, pounds	69.7
Analysis	
% zirconium	12.3
% total iodine	86.9
% free iodine	22.5
Weight as zirconium converted, pounds	8.55
% conversion of zirconium	79.2

Furnace residue

Weight, pounds	4.25
Analysis	
% carbon	52.1
% zirconium, by difference	47.9
Weight as zirconium remaining, pounds	2.02

Zirconium weight balance, pounds

Input = output in condenser + furnace residue	
10.8 = 8.55 + 2.02	
10.8 = 10.57**	

Iodine flow rates (based on iodine found in the products)

Pounds per hour	21.6
Pounds per hour per square foot	272
Linear velocity, feet per second	0.49

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TABLE 3. Continued

Iodide Production Rate

Pounds per hour	20.4
Pounds per hour per square foot	267

* Measurement could not be made because the liquid level indicator on the I_2 boiler was stuck.

**This inequality represents only a 2 per cent error in the material balance.

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Inspection of the interior of the furnace showed that the quartz sleeve had melted at the lower part of the furnace but the split graphite electrode was in good shape. It is now believed that the original trouble with a corrosive attack of electrode was from water vapor present in the crude iodine used. This iodine was recovered from a melt-down operation in the zirconium pilot plant and contained some hygroscopic iodide.

Although the bottom electrode design worked satisfactorily for the last two runs, a top electrode assembly was to be tested before building a larger unit for the plant.

As a result of the experience gained in operating the furnace according to the original design given in Report BMI-528, page 13, other modifications should be incorporated for improved furnace performance.

1. Heat the iodine inlet line with an internal heater.
2. Slope the iodine inlet line downward to prevent solids from backing too far into the inlet line.
3. Seal the carbon tube to the furnace shell with refractory cement to prevent undue seepage of iodine into the insulation between the carbon tube and the steel wall.

The production rate in the last run of 267 pounds of ZrI_4 per hour per square foot of cross-section is quite high. Therefore, in scaling up the furnace to fit the requirements of the pilot plant, lower flows of iodine could be used and still achieve ample daily production with better iodine-conversion efficiencies. A study of the applied

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kinetics of this reaction would be desirable to predict optimum operating conditions. However, optimum conditions are not absolutely necessary if a design were required immediately, because good production rates can be obtained if the furnace temperature is held at 1000°C. or above and a partial condensing system used to separate the unreacted iodine.

Sublimation and Thermal Decomposition of Zirconium Iodide

In last month's report*, some of the alternate methods of sublimation were discussed. In particular, a small vibrating flash plate coupled with a rotary scraping condenser offered advantages over the present fin-tube heat exchangers for moving the zirconium iodide gas at high flow rates.

The vibrating flash plate could be built for a small pilot plant with a minimum of expense and time. This could be coupled to a deposition unit and the rate of metal formation studied at higher flow rates and lower iodide pressures than was possible in the large pilot plant. There was insufficient time to construct and operate a scraping condenser in the time remaining on the contract, but a tubular condenser could be substituted for the present experimental program.

With these thoughts in mind, the pilot plant shown in Figure 3 was constructed in 3-1/2 weeks and the testing period was carried on for the week of November 5. Orders to terminate the experimental work on November 9 prevented any further testing of this plant.

* BMI-531, page 10.

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FIGURE 3. EQUIPMENT USED IN STUDYING SUBLIMATION AND THERMAL DECOMPOSITION RATES OF ZIRCONIUM IODIDE

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Referring to Figure 3, most of the equipment was built from 304 stainless steel which was available from early experimental work on fin-tube heat exchangers. In places where steel was used, such as flanges and the screw feeder, the parts were nickel-plated.

The screw feeder was constructed from a nickel-plated Russell-Jennings 1-inch car bit with a 3/8-inch shaft sealed by two Victoprene seals. The feed rate of the screw was calibrated as a function of RPM using ZrI_4 powder having a bulk density of 90 lb./cu.ft. The range covered was 2 to 14 pounds of ZrI_4 per hour when the system was at atmospheric pressure and no vibration of the assembly was employed.

The bottom plate of the vaporizer was a 1/2-inch thick stainless steel plate, 12 inches in diameter. A 10-inch diameter center circle was milled to 3/32 inch thickness. At this thickness, a small Syntron vibrator, Model V-15, would cause the ZrI_4 powder to vibrate vigorously at about one-half the maximum amplitude of the vibrator. A thermocouple bead was brazed to the plate, which was heated by two ring strip heaters having a total rating of 1.8 kw. Temperature control was maintained by a Foxboro controller.

The deposition bulb was 6 inches in diameter by 2 feet long. A hairpin, utilizing a total length of 2 feet of 90-mil starting wire, was welded to stainless steel electrodes.

The condenser for these tests was a single fin tube, using air as a coolant. This tube developed a leak after the second run and was replaced by a straight pipe. Dry-ice-acetone traps were placed in

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a series—parallel arrangement with a Megavac and later Kinney VSD 8-811 vacuum pump.

Four runs were made during the week of operation, but in none of the runs could a suitable operating vacuum or feed rate be obtained. The summary of results is presented in Table 4.

TABLE 4. SUMMARY OF DATA OBTAINED IN THE PILOT PLANT
EMPLOYING A VIBRATING FLASH PLATE VAPORIZER

	Run No.			
	DB 1	DB 2	DB 3	DB 4
Temperature of vaporizing plate, °C.	475	480	490	450
Temperature of deposition bulb walls, °C.	450	477	485	442
Screw feeder, RPM.	20	10	10	2.5
Calibrated feed rate, lb. ZrI ₄ /hr. ^a	5	2.5	2.5	0.5
Actual feed rate, lb. ZrI ₄ /hr. ^a	10.1	104	29.7	44
Pressure in deposition bulb, mm.	b	7→	0.60→	1.5→
		29	4.9	3.3
Pressure at vacuum pump, mm.	2.0	0.21→	0.36→	0.69→
		5.0	1.3	3.85
Electrical constant - K./cm.	0.99	0.85	0.95→	1.02→
			1.13	1.08
Time of run, hours	4.1	0.5	1.8	0.33
Average deposition rate, lb. nr. ⁻¹ ft. ⁻¹	0.0127	c	0.0204	0.031
	0.0127	c	0.0204	0.031

- a TAM iodide powder with a bulk density of 90 lb./ft.⁻³.
- b Pressure taps were not ready for operation during this run.
- c Filament burned out before any good deposition data were obtained.

In the first run, the pressure taps on the vaporizer and deposition bulb were not ready for operation. Pressure at the Megavac pump, used for the first run, was high indicating a leak or outgassing of the powder. The run terminated when solid lumps plugged the screw conveyer. The deposition rate was low, but the zirconium deposited

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looked good. Alterations for succeeding runs incorporated a screening operation and outgassing of the powder at 150°C. over night, installation of a large Kinney pump, and pressure measurements at the vaporizer and deposition bulb.

In the last three runs, improvement in the deposition rate occurred but the filament had a black deposit which looked like ZrI_2 . This was possible, since the flow rates were 3 to 10 times higher than desired. In Runs DB 2 and DB 4, the powder was actually blown through the unit. Any powder deposited on the filament would be converted to ZrI_2 .

Calculations were made to see what mesh-size powder would be entrained by the vaporization of 50 pounds per hour of ZrI_4 from a 10-inch diameter flash plate operating at 2 mm. pressure. Dusts under 325 mesh would be entrained. Although no screen analysis was made of the TAM iodide used in the work, it is doubtful whether it contained much powder below 325 mesh.

In the third run, DB 3, the powder actually vaporized and condensed as a solid cake with no loose powder present in the equipment. Under such conditions, the rate was 29.7 pounds of ZrI_4 per hour and the heat-transfer coefficient was 20 BTU. $hr.^{-1} ft.^{-2} ^\circ F.^{-1}$, an excellent value for a vacuum sublimation. The deposition rate was still low in this run. This may be the result of a high partial pressure of non-condensibles, since the system was designed to have very low pressure

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drops, or it is possible that the filament was operating at too low a temperature for such a high flow rate.

The powder feed rate could not be controlled by the speed of the screw. This trouble could be caused by (1) rapid outgassing of the powder as it reached the low pressure region of the screw, thereby blowing the powder through the unit, (2) a leak at the screw shaft which would act as described for outgassing, or (3) vibration of the reservoir, giving additional translational energy to carry more powder along the screw.

It is more likely that the first two points are the cause of the trouble. The G.E. helium leak detector was not working at the time the tests were made, so it was impossible to check for vacuum leaks around the seals of the screw conveyer shaft. The G.E. freon leak-detector system, operating at 5 pounds pressure, showed no leaks at the seal or in any other part of the system. However, a seal that holds pressure does not necessarily hold a vacuum, even though the Victoprene seals were designed for this dual purpose. The outgassing problem is more serious and may give rise to special precautions in preparing and handling of the powder in this straight-flow process.

A summary of the work which was done on the small pilot plant during the week's operation shows:

1. High vaporization rates can be obtained from a vibrating flash plate.
2. Feeding powder to the plate at a constant and controllable rate presented some difficulty. This may be overcome by pumping between

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Victoprene seals, proper outgassing of the powder, using a smaller diameter screw, and sloping the feed screw upward to avoid translation of the powder by vibration.

3. Deposition rates could not be properly studied because of high noncondensable pressures. There is an indication that too high a flow rate favors the deposition of ZrI_2 if the filament temperature is too low.

4. No corrosion of the flash plate was evidenced during the short period the plant was in operation. A thorough study of corrosion must be made, however, since the entire process may depend on the durability of the flash plate.

5. This type of equipment offers a means of rapidly obtaining good data at a low cost on the fundamentals of an iodide-flow process for the production of zirconium metal.

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