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Development of an Improved "Bubble Through" Saturator

(Title Unclassified)

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1. Introduction

One of the important process parameters in NbC vapor plating operations is the concentration of the salt in the plating stream. Since the melting point of the salt is about 205°C and its vapor pressure at that temperature is 230 mm of mercury, the molten salt can be conveniently evaporated at temperatures above its melting point.

Several types of vaporization schemes have been utilized in ROVER and NERVA fuel element coating. These have in common the passage of a carrier gas through a chamber containing molten niobium pentachloride. In some instances (IASL and WAFF) the temperature of the salt has been controlled along with the flow rate of carrier gas. These systems are empirically calibrated to achieve reproducible salt vaporization rates from run to run. At Oak Ridge the vaporizer has been operated at the boiling point of the salt with salt vaporization controlled by heat input in a carefully insulated system.

In all these systems an excess of carrier gas, usually helium, is employed. That is, the theoretical pickup of salt which is predicted by Dalton's law is not achieved. This can be explained in terms of inadequate mixing between carrier gas and salt, as all the systems in use at present pass carrier gas over the molten salt. The opportunity for carrier gas stratification in such systems is great and delivery rates are difficult

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to control precisely.

Literature published by an NbCl_5 supplier (Stauffer) shows schematically a system in which carrier gas is sparged into molten salt. This literature implies that theoretical pickup of salt can be achieved in such systems but gives no information on maximum delivery capacity as a function of absolute capacity. Accordingly, a series of experiments was undertaken to determine if theoretical salt pickup could be achieved, and to establish design parameters for a system capable of delivering precise quantities of salt at rates on the scale of production operations.

The results of laboratory-scale tests, and tests of a developmental saturator which was later constructed, are presented. Possible design parameters are discussed for a unit which could be used in routine production.

2. Laboratory Tests

Figure 1 shows schematically the laboratory apparatus which was employed to test the concept of sparging carrier gas into molten salt. A 100 ml graduated cylinder (A) was used to contain the salt, and helium was introduced through a gas dispersion tube (B) immersed in molten salt. Auxiliary heating was furnished to the exit line (C) while the remainder of the unit was placed in a silicone oil bath. Stirring and automatic temperature control insured temperature uniformity. During the course of the run, the salt level was measured at intervals and the salt loss was deter-

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Figure 1

Apparatus for Laboratory Saturator Tests

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mined. This data could be compared to the calculated salt loss based on Dalton's law, since the helium flow rate was carefully controlled. An example of such a calculation follows.

Basis: Barometric pressure = 745 mm Hg
Salt vapor pressure at 210°C = 275 mm Hg
Specific volume of salt (STP) = 83 ml/g
Helium flow = 150 ml/min
Salt flow in 5 minutes = 5.5 g

According to Dalton's law, the mole fraction of salt in the exit stream should be $\frac{275}{745} = 0.369$. As measured, the mole fraction of salt in the exit stream was 0.38, therefore, the calculated efficiency is essentially 100%.

Figure 2 is a plot of efficiency as a function of cumulative salt removal from the laboratory apparatus, representing data from a test on November 20, 1963. Within the limits of experimental error, the desired theoretical uptake of salt was achieved and maintained until only a small amount of salt was left in the cylinder.

Furthermore, an indication of scaleup possibilities was obtained from this test. A salt pickup of 1.1 grams per minute had been accomplished from a reservoir containing less than 100 grams of salt. Hence, a salt depletion rate of over 1 percent per minute might serve as a basis for scaleup, provided the temperature could be satisfactorily maintained in a larger system.

In fuel element production it is necessary to provide salt to the coating operation at a constant rate for 390 minutes. Consequently, the

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Figure 2
Laboratory Saturator Test - November 20, 1963
Efficiency vs. Cumulative Salt Removal

salt depletion rate is limited a priori to a maximum of 0.25 percent per minute. In block coating operations, salt must be supplied for 300 minutes, leading to a maximum of 0.33 percent per minute. Thus, both production requirements represent a de-rating of the laboratory test. On this basis, design was undertaken for a saturator which could be employed in block coating operations to prove the advantages of the new saturator concept.

3. Developmental Saturator

3.1 Design and Use

The block coating saturator was designed to incorporate a number of improvements, including more precise temperature control, improved loading procedure, visual access to the salt, and controlled preheating of carrier gas. Figure 3 is a schematic diagram of the unit. Temperature uniformity is obtained by circulating heated silicone fluid between the inner and outer cylinders. The unit can be loaded without being moved, since its exit port is adaptable to the salt charging drum. Visual access to the salt is provided by twin sight ports, one below the salt level and one in the vapor space above the salt level. Each sight port is comprised of two windows which are sealed by a teflon-viton elastomer gasket combination. Carrier gas is introduced through a tube which is preheated by silicone fluid over several feet prior to its introduction at the bottom of the inner cylinder. Thirty holes 1/16 inch in

Figure 3
Schematic of Developmental Saturator

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diameter are provided in the sparger.

In practice, after the saturator is loaded with salt, the system is brought to temperature by means of a 1000 watt resistance heater in the heater well and two 750 watt immersion heaters in an external 1.5 gal. reservoir which heat the circulating silicone fluid. About 3 hours is required for the system to reach operating temperature. The power is then reduced and control is maintained with a simple control unit.

3.2 Calibration

Several calibration runs were performed in which the saturator rested on a platform scale as its effluent was allowed to escape. Periodic weighing of the saturator without interrupting the run permitted construction of a plot showing the constancy of salt loss rate as a function of time. The data are presented in Figure 4. The slight data scatter which is exhibited may be due largely to limitations in the precision of the weighing operation.

It has been shown in all tests up to a delivery rate of 5 pounds per hour that the efficiency, as described in Part 2, is indistinguishable from 100 percent. Thus, if temperature is closely controlled ($\pm 2^\circ\text{C}$) and carrier gas flow is accurately known, the salt delivery rate can easily be predicted to within ± 5 percent from basic physical principles.

3.3 Checkout

The developmental saturator has been tested at WAFF in two coating runs. The first test at WAFF involved employment of the saturator

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Figure 4
Saturator Weight vs. Time During a Calibration Run

in a developmental fuel element coating run on June 2, 1964. Although the saturator had been designed for block coating to deliver only 10 pounds of salt in five hours, it was successfully employed to deliver 19.5 pounds of salt in 5 1/2 hours. This coating run was terminated at 5 1/2 hours because of the salt level's reduction to a point where salt delivery suddenly dropped below the desired rate.

In the second test at WAFF, the saturator was employed in a block coating run on July 2 in which it delivered 8 3/4 pounds of salt in five hours. The 12 percent discrepancy between desired and measured saturator weight loss is largely due to an error in the reading of the calibration curve for carrier gas flow which was committed prior to the run. This error resulted in carrier gas flow being maintained at 2.0 liters per minute instead of the desired 2.2 liters per minute. The 10 percent error accounts for most of the discrepancy. In this light, predictable and measured salt weight loss are within 2-3 percent of each other. In both checkout runs, the saturator performed according to plan and without incident.

4.0 Production Saturator - Design Suggestions

The developmental saturator described and discussed in Part 3 has demonstrated the advantages of sparging carrier gas into molten salt, providing visual access, and maintaining close temperature uniformity

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and control. For production operations, however, a larger, more rugged design would be desirable. The following changes should be considered if design and construction of production units based on these principles is undertaken.

A. A pot furnace could be employed to heat the saturator instead of a circulating fluid system. Variable control can be provided to counter temperature differences inside the furnace. Moreover, the constant agitation provided by sparging insures temperature uniformity in the salt. From an operational standpoint elimination of fluids will eliminate the need for moving parts and obviate the criticality problem which could arise from the use of hydrogenous fluids. The saturator design would also be simplified.

B. Glass ports at right angles merit consideration. One of these could be placed in the top of the saturator for observation, and the other could be used to introduce a light beam.

C. Direct heating of the salt would better be accomplished by a heater which is entirely covered by salt even when the salt is molten and depleted. Initial heating could be accomplished more efficiently in this way without danger of pressure buildup due to hot spots caused by the heating element being partially exposed.

D. The saturator could be designed to hold an entire drum of salt. The unit could then furnish salt for three fuel element batches

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or approximately nine block coating runs without any disturbance to the system. Contamination by air could also be reduced.

E. The sparger could be designed to distribute carrier gas over as large an area of the saturator bottom as possible. Cooling of the salt in the vicinity of gas introduction, caused by removal of heat of vaporization, would thus be minimized and the total delivery capacity at 100 percent efficiency could be increased.

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