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**Y-12 DEVELOPMENT ORGANIZATION
TECHNICAL PROGRESS REPORT**

Part 7 - Lithium and Uranium Chemical Processing (U)

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HIGHLIGHTS

MICROWAVE CONSOLIDATION OF URANIUM POWDER

Microwave energy was used successfully to consolidate small lots of uranium powder. The factors varied were maximum power, hold time, heating rate, gas pressure, batch composition, crucible material, and insulation configuration. The results of these tests indicated that the microwave consolidation process was rugged against all seven factors for their respective parameters. (Page 6)

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LITHIUM AND URANIUM CHEMICAL PROCESSING

MICROWAVE CONSOLIDATION OF URANIUM POWDER

C. L. Calhoun, Jr.

Summary

Microwave energy was used successfully to consolidate small lots of uranium powder. The author conducted a ruggedness test of seven factors to determine the effects of run variations on the results obtained through round-robin testing. The factors varied were maximum power, hold time, heating rate, gas pressure, batch composition, crucible material, and insulation configuration. The results of these tests indicated that the microwave consolidation process was rugged against all seven factors for their respective parameters.

Introduction

Earlier experiments demonstrated that microwave processing can rapidly melt and consolidate uranium powder. The high efficiency of direct coupling to the powder offers potentially significant savings in time and energy. Also, microwave processing should reduce the waste stream associated with conventional processes (insulation liners, heating elements, induction aids, etc.).

Ruggedness testing was used to determine which factors weigh heavily on the final product characteristics. Isolating these factors and optimizing the process on a small scale should reduce the cost and time requirements of the production scale-up effort.

Prior Work

Earlier work at the Y-12 Plant demonstrated strong coupling of microwaves to uranium powder. Several runs demonstrated at least partial consolidation for test batches of ≤ 100 g.

Presentation of Experimental Work

This particular ruggedness test was designed after the method discussed by Grant Wernimont¹ using the experimental outline for studying seven factors at two levels in eight trials. Table 1 shows the design plan. This report lists the selected factors for testing and gives the reasons for their selection and the levels that were used in this experiment.

Table 1. Ruggedness test for microwave consolidation for seven factors in eight trials

Factor	Trial number							
	1	2	3	4	5	6	7	8
Power: A = 1 kW, a = 1.3 kW	A	A	A	A	a	a	a	a
Hold time: B = 10 min, b = 2 min	B	B	b	b	B	B	b	b
Heat rate: C = Profile 1, c = Profile 2	C	c	C	c	C	c	C	c
Pressure: D = <400 μm , d = >800 μm	D	D	d	d	d	d	D	D
Composition: E = Batch 1, e = Batch 2	E	e	E	e	e	E	e	E
Crucible: F = MgO, f = SiO ₂ /Y ₂ O ₃	F	f	f	F	F	f	f	F
Configuration: G = open, g = with lid	G	g	g	G	g	G	G	g

Maximum Power

Power and temperature are related by the equation

$$P_{ab} = C_p km(T_f - T_i)m/t \text{ .}^2 \quad (1)$$

Small changes in the absorbed power or temperature could affect the degree of consolidation. (Levels: 1 kW, 1.3 kW.)

Hold Time at Maximum Temperature

The degree of consolidation may be highly time dependent. (Levels: 10 min, 2 min.)

Heating Rate

The rate of power increase could affect the distribution of power between the sample material and the insulation. Table 2 shows heat profiles. (Levels: Profile 1, Profile 2.)

Table 2. Heating profiles for microwave consolidation

Profile 1		Profile 2	
Power level (W)	Hold time (min)	Power level (W)	Hold time (min)
100	5	100	5
200	5	200	5
300	5	300	5
400	2	400	3
500	2	500	3
600	2	600	2
700	2	700	2
800	2	800	1
900	2	900	1
1000	2	1000	1
1100	2	1100	1
1200	2	1200	1
1300	2	1300	1

Pressure of Chamber

Operation in certain pressure regimes tends to promote corona or plasma formation. This tendency could affect the amount of power absorbed by the sample. (Levels: $<400\text{ }\mu\text{m}$, $>800\text{ }\mu\text{m}$.)

Composition

Variation in the dielectric properties between lots of powder could affect microwave coupling. [Levels: Batch 1 (2-1305), Batch 2 (SDOR Mix 2).]

Crucible

The type and shape of the material of the sample crucible could affect the power distribution within the cavity. [Levels: magnesium oxide (MgO), yttria (Y_2O_3)-coated silica (SiO_2).]

Insulation Configuration

Radiative heat loss could prevent the material from reaching suitable consolidation temperatures. [Levels: open crucible, lid with 1-in. aluminum oxide (Al_2O_3) insulation on top of crucible.]

The success of individual runs was evaluated as a function of pickling performance in a 0.3-*M* nitric acid solution. The rate of dissolution can be directly correlated to sample porosity to reflect the degree of sample consolidation. The individual samples were dried and weighed after pickling for 5 min, 15 min, 1 h, 4 h, and 7 h, respectively. The corresponding dissolution rates were then averaged to interpolate a value for the total time interval.

Table 3 compares the average value of the dissolution rates for the eight test conditions in which the factor was held at the high level and the value obtained when the low level was used. The difference between these two values is compared with a critical difference. Differences in excess of the critical difference are significant at the 95% confidence level. Differences less than the critical value indicate that the procedure is rugged against variations of that factor at the levels used.

Table 3. Average results of the ruggedness test

Factor	Average of capitals (g/min)	Average of lower case (g/min)	Difference (g/min)
A	0.0772	0.0994	-0.0222
B	0.0493	0.1273	-0.0780
C	0.1080	0.0685	0.0395
D	0.1300	0.0466	0.0834
E	0.0408	0.1358	-0.0954
F	0.0404	0.1362	-0.0958
G	0.1056	0.0710	0.0346

The differences between the two results can be defined as d_i . The standard error, s_d , is given by the equation

$$SQR [(2 * d_i^2)/n] , \quad (2)$$

where n is the number of degrees of freedom.

The standard error for these experiments is

$$s_d = 0.1019 \text{ g/min} . \quad (3)$$

The 95% confidence interval is given by

$$\pm (2.13) * (0.1019) = 0.2170 \text{ g/min} . \quad (4)$$

By comparing the confidence interval with the differences of the high- and low-level results, one observes that the process is rugged against all factors tested.

Future Work

Although microwave consolidation proved to be rugged for all seven factors, the effect of these variables outside of their limited parameters remains to be studied. Future work will broaden these parameters and expand the number of factors for evaluation.

Experiments will be initiated to evaluate the accuracy of noncontact temperature measurements. Scale-up experiments will also continue when the new glove box unit is installed.

References

1. Grant Wernimont, "Ruggedness Evaluation of Test Procedures" *ASTM Standardization News*, 13-16, March 1977.
2. P. A. Hass, "Heating of Uranium Oxides in a Microwave Oven," *Am. Ceram. Soc. Bull.*, 58 [9] 873, 1979.

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