## QUALITY ASSURANCE IN CANDU-TYPE FUEL MANUFACTURING

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### **ABSTRACT**

This paper is concerned with fabrication of UO<sub>2</sub> pellets from UO<sub>2</sub> powder by powder metallurgical methods (pelletizing and sintering). The effect of ejection to molding force ratio on the fired pellets properties was studied. It is observed that pellets cold-pressed at ratio less than 75% is cracked to two parts. The effect of the sintering temperature on the fired pellets properties was studied. It was found that the sintering of UO<sub>2</sub> pellets at 1650 °C leads to production of pellets within the qualification requirements.

The data and information available in the ASTM for each step in UO<sub>2</sub> pellets fabrication process and the technical experience (gained or published) are transformed into a group of logic flow charts (LFC'S). These logic flow charts are collected to form a module of a software to qualify the sintered pellets and also gives a technical assistance according to the ASTM for each step in the fabrication process.

### INTRODUCTION

A nuclear reactor requires a large number of similar reactor components of identical and strict specifications (pellets, cladding tubes, fuel rods, etc.). This situation calls for a very high degree of reproducibility at each production step and for the consistency of raw materials over long periods of time. This reproducibility can be achieved only by technological efforts and specific methods of control on the processes and products (1).

The large numbers of identical items, combined with the requirement for high reproducibility of processes and products and the relatively short time in the reactor, lead to the possibility and necessity of establishing a statistical evaluation of process parameters and product properties. Quality cannot be achieved only by control; quality must be planned, produced and verified within each processes. Quality is not a product of formalized actions; it is a consequence of logical approaches on the basis of scientific and technical understanding of the products and processes under the limitations of operational requirements (2,3).

The basic objectives of the quality documents includes the following: The quality assurance plan (QAP), design drawings, technical specifications, process outline, the quality assurance procedure manual (QAPM), the inspection and test plan, manufacturing and QC instructions and traveller cards (list followers, route cards). Each of these terms is clearly defined in the QA literature on nuclear industry (1-5).

The present work is concerned with fabrication of UO<sub>2</sub> pellets from UO<sub>2</sub> powder by powder metallurgical methods (pelletizing and sintering). Also, A program is designed to qualify each process in the fabrication procedure according to the ASTM (6-13). The program also gives a technical assistance and explanation for the reasons of rejecting the pellets.

#### EXPERIMENTAL WORK

## Green pellets production:-

The aim of pre-pressing is to produce UO<sub>2</sub> slugs from natural UO<sub>2</sub> powder. Press (Komage K-6) is used to press the slugs and the green pellets. A die of 25mm diameter is used to produce the slugs (about 25.5 mm diameter). The considered parameters to be checked and adjusted were: fill position or depth and press settings (die travel, top punch travel and final pellet length). UO<sub>2</sub> powder was pressed at a molding pressure of 490, 610, and 650 kPa. The slugs were crushed and the desired particle fraction was obtained by sieving the crushed slugs. The tested sieve sizes are 10 and 12 mesh size. Zinc stereate was added to the granulates (0.02 wt%) and then blended in a V-blender for 15 min.

The blended  $UO_2$  granules are transferred to the press and fed into the die. A die of 15.25 mm diameter is used to produce the green pellets. Final pressing done at 2.6 MPa. The effect of the filling depth, molding pressure and withdrawal pressure were studied.

### Sintered pellets production:

Initially, the green pellets were sintered in a tubular horizontal furnace (Heraus type, Germany) in hydrogen atmosphere for 2 hours at  $1600^{\circ}$ C,  $1650^{\circ}$ C and  $1700^{\circ}$ C  $\pm$   $20^{\circ}$ C.

Later on, The lab was equipped with a top loading tungsten/ molybdenum furnace (Material Research Furnaces, INC, MRF, USA) with a maximum operating temperature of 2000°C. The heating element is a tungsten mesh of  $5.0^{\circ}$  I.D. x  $10.0^{\circ}$  inch height to yield a  $4.0^{\circ}$  diam. x  $8.0^{\circ}$  height uniformly-heated usable work space. The experience gained in the old setting furnace was utilized in qualifying the new furnace. The operating conditions were; Sintering temperature ( $1650 \pm 20$  °C), sintering gas ( $H_2$  gas), sintering time (2 hours), and heating and cooling rate (5 °C/min)

### **Visual Inspection:**

Pits, inclusions, chipping of the dished and flat ends and chipping of the circumferential surface of the each pellet were visually inspected

### **Density measuring:**

The density was measured both geometrically and by immersion in water. The accepted pellet density should be in the range of  $10.6 \pm 0.15$  g/cm<sup>3</sup>.

#### Microstructure examination:

Sintered pellets for microstructure examinations are taken. A longitudinal cross section on the diameter of the pellet, was prepared and examined. The mean grain size was measured. The accepted mean grain size is in the range of 5-30 µm.

#### O/U ratio

The O/U ratio of the sintered density was determined gravimetrically. For more details, the above measuring techniques are published elsewhere (14).

### **QACAF** - Software

A nuclear reactor fuel requires a very high degree of reproducibility at each production step and consistency of raw materials over long periods of time. A software program is designed to fulfill these requirements and to give a technological assistance and to control the processes and products.

The prepared flow charts have been transformed into a computer software using Borland C<sup>++</sup> (under Windows) with Object Oriented Programming (OOP). Screen displays were designed to facilitate with the user. Nested windows containing successively linked menus that can be accessed by the user appear to be the best interface. Simple indication of preference using a mouse affords a clear interaction. The main features of Quality Assurance in Candu Fuel (QACAF) program are given in Fig. (1). This program is divided into a group of subprograms, each subprogram is dealing with one subject. All the information and data related to qualification of processes used in UO<sub>2</sub> fabrication published in ASTM are transformed into a logic flow charts. The logic flow charts also include the technical experience (gained or published).

The logic flow charts are given in the form of a group of "IF-THEN" rules. The program covers the following topics; lot acceptance, production of sintered UO<sub>2</sub> pellets, Inspection of pellets and the reports related to each process. The program also define if the produced UO<sub>2</sub> pellets are accepted or not and give technical reasons in case of rejection. Fig. (2) gives an example (Qualification of sintered pellets) of the logic flow charts which collected to form the program.

#### RESULTS AND DISCUSSION

Experiments were carried out to study the effect of pressing parameters (filling depth, lower scale and pressing time) on the sintered pellets properties. The time of pressing and ejection time was 4 seconds.

The filling depth was calibrated to get green pellets with 20 mm length. It was found that filling depth with a range of  $4.8 \pm 0.2$  on the filling scale satisfied this requirement.

The effect of the ejection force to the molding force ratio on the sintered pellets properties was studied. It was found that if this ratio is less than 75%, the fired pellets is cracked into two pieces.

It was also found that pressing the pellets at a molding force with a range of 46 - 51 kN resulted in getting sintered pellets satisfying the qualification requirement (diameter not less than 12.15 mm). The sintering temperature experiments indicated

that firing the pellets at 1700 °C resulted in pellets with diameters less than 12.15 mm. Sintering temperature of 1650 °C fulfilling the qualification requirements with respect the pellets diameter. The average grain size of fired pellets at 1700 °C (8.5  $\pm$  0.5) is less than that of 1650 °C (12.4  $\pm$  0.5) but still in the accepted range.

Experiments were carried out to calibrate the top-loading furnace. The sintered pellets were qualified using the designed program. In the first experiment, 85% of sintered pellets were rejected because of the cleanness of the gas. The H<sub>2</sub> gas was cleaned and tried again. For the second run, It was found that 90% of the pellets were rejected and it was recommended to adjust the ejection to mold force. The pellets were pressed at a force of 20/40 KN. In the third experiment, the effective heating zone was calibrated. It was found that the effective heating zone should be 2 inches away from each heater's ends. The pellets laid 1 inch away from heater's ends were unaccepted because of the lower density than 10.45 g/cm<sup>3</sup>. The density was in the range of 10.3 g/cm<sup>3</sup>. In the fourth experiment, the pellets were pressed at ejection / molding force of 37/48 KN and the pellets were qualified using the program. The qualification report of the sintered pellets is shown in Table (1). Consequent runs are being treated in the same manner.

### **CONCLUSIONS**

- Filling depth with a range of  $4.8 \pm 0.2$  on the filling scale satisfied the requirement of 20mm green pellets length.
- The ejection force to the molding force ratio should not be less than 75% otherwise, the fired pellets is cracked into two pieces.
- Pressing the pellets at a molding force with a range of 46 51 kN resulted in getting sintered pellets satisfying the qualification requirement (diameter not less than 12.15 mm).
- Sintering the pellets at 1700 °C resulted in over sintering, while, sintering at a temperature of 1650 °C fulfilling the qualification requirement with respect the pellets diameter.
- In QACAF program, the information required for qualifying each process in UO<sub>2</sub> production in accordance with the ASTM have been transformed into a group of LFC'S and then into a software program. This program also have a technical assistance for less skilled personnel involved in powder characterization or sintered UO2 pellets qualification. This software program can be used on its own as a substitute of the standards of so many processes and techniques used in fuel fabrication process starting from powder receiving, characterization, pellets production and qualification of the production processes and products.

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# **QACAF** Program

# O Lot acceptance

- Characterization
- Sinterability
- O/U ratio
- Surface area

## O Production of sintered UO2 pellets

- Pressing
- Sintering
- Grinding

## O Inspection

- Visual inspection
- Microstructure inspection

## O Reports

## -Lot acceptance reports

- -Lot powder characterization
- -Lot powder surface area report
- -Lot powder O/U report
- -Pressing report
- -Sinterability report
- -Final acceptance report

# -Manufacturing reports

- Slugs manufacturing report
- Green pellets manufacturing report
- Sintered pellets manufacture report

## -Qualification reports

- Qualification of pressing process report
- Qualification of sintering process report
- Qualification of grinding process report

## -Quality control reports

- Quality control of pressing process report
- Quality control of sintering process report
- Quality control of grinding process report

## -Inspection reports

- Inspection of green pellets
- Inspection of sintered pellets
- Inspection of ground pellets

# -Acceptance of sintered and ground pellets report

## O Technical assistance

Pressing

Sintering

Pellets grinding

Microstructure examination

Characterization techniques

Qualification

TABLE (1) THE QUALIFICATION REPORT OF SINTERING PROCESS (EXPERIMENT NO. 4)

Lot N	Lot No. (001)	Con	Container No. (01)	. (01)	No of pel	pellets (040)		Green pellets status: (accepted)	ets status:	(accepted		Purging gas: Ar	s: Ar	
Sinter	ring temp	Sintering temperature (1650 °C)	(1650 °C)		Heating rate (05 °C/min)	(05°C/	_	Cooling ra	Cooling rate (05 °C/min)	_	Sintering time:	ne: (2hrs)	s)	
Hydre	ogen flow	Hydrogen flow rate (2.5 L/min)	5 L/min)		Date: / / 1997		- •	Sintering c	Sintering carried out by:	by:				
Meası	uring car	Measuring carried out by	yc	Ins	Inspection can	carried out by	it by:					Ex	perime	Experiment No. (4)
Pellet	Foreign	cracked	Spalled-	Inner	location	pits	Length	Top	Middle	Bottom	Weight	density	grain	accepted/
Š	color	to two pieces	off	pellet/oute r pellet	in the sintering	chips		diameter	diameter	diameter			size	rejected
-	N <sub>o</sub>	No	No	No		A	15.25	12.30	12.19	12.31	18.810	10.47	A	Accepted
7	οN	No	No	No	1	А	15.25	12.34	12.18	12.35	18.856	10.47	A	Accepted
3	No	No	No	No	1	A	15.24	12.26	12.18	12.26	18.731	10.48	Ą	Accepted
4	No	No	No	No	1	A	15.23	12.27	12.21	12.26	18.808	10.50	A	Accepted
S	Ño	oN	No	No	1	A	15.23	12.27	12.19	12.26	18.778	10.50	Ą	Accepted
9	No	No	No	No	1	A	15.21	12.27	12.18	12.26	18.720	10.49	A	Accepted
7	No	No	No	No	1	A	15.26	12.28	12.17	12.26	18.756	10.48	Α	Accepted
8	No	No	No	No	1	A	15.23	12.28	12.19	12.25	18.724	10.47	A	Accepted
6	No	No	No	No	1	A	15.24	12.29	12.17	12.24	18.688	10.46	A	Accepted
10	No	No	No	oN	1	A	15.25	12.28	12.16	12.25	18.703	10.47	A	Accepted
11	No	No	No	No	2	А	15.23	12.26	12.15	12.25	18.648	10.47	A	Accepted
12	No	No	No	oN	2	A	15.23	12.26	12.18	12.25	18.711	10.48	A	Accepted
13	No	No	No	No	2	Ą	15.22	12.25	12.17	12.25	18.676	10.48	A	Accepted
14	No	No	No	No	2	A	15.24	12.26	12.19	12.26	18.782	10.50	А	Accepted
15	No	No	No	No	2	A	15.24	12.26	12.15	12.26	18.703	10.49	A	Accepted
16	No	No	No	No	2	A	15.23	12.26	12.16	12.26	18.671	10.47	A	Accepted
17	No	No	No	No	2	Α	15.24	12.24	12.17	12.26	18.683	10.47	A	Accepted
18	No	No	No	No	2	A	15.23	12.24	12.18	12.26	18.739	10.50	A	Accepted
19	οN	No	No	No	2	4	15.24	12.23	12.19	12.26	18.777	10.51	A	Accepted

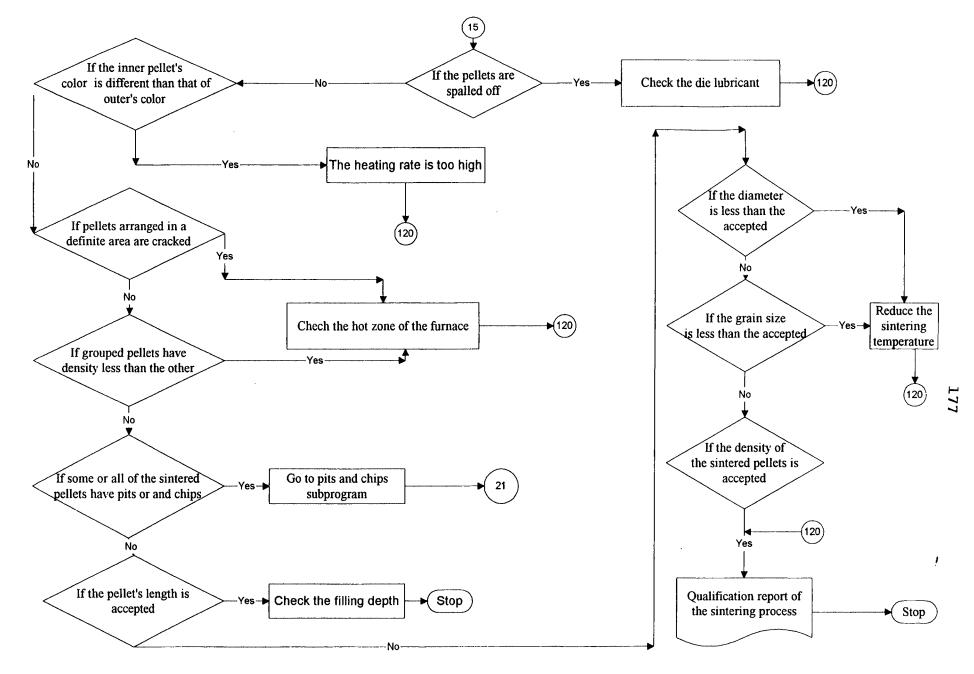


Fig. (2) cont.: The logic flow chart of qualification of sintering process