TECHNOLOGICAL INVESTIGATION FOR PRODUCING UO_2 POWDER FROM ADU BY USING ROTARY FURNACE

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ABSTRACT: Uranium Dioxide Powder UO_2 is main material for producing UO_2 fuel ceramic pellets. The technical characteristics of UO_2 powder directly affect on mechanical and physical characteristics of UO_2 fuel ceramic pellets. Project tiled "Technological investigation for producing UO_2 Powder from ADU by using rotary furnace" with the code number BO/01/03-06 for two years 2001 and 2002, on purpose to step by step perfect the technology and equipments for producing UO_2 powder, that is as nuclear fuel. This UO_2 powder may be good material for producing UO_2 fuel ceramic pellets. The results had been achieved as follows:

1. Study on the perfection of the reduction process U_3O_8 to UO_2 in the gas mixture of $3H_2 + N_2$ in inactive condition.

2. Study, design and production of active device system called rotary furnace for manufacturing UO_2 powder from ADU.

3. Study on 4 steps of technology process: drying, calcination, reduction and stabilization of UO_2 powder in the system of rotary furnace from which obtained UO_2 powder with technical characteristics meeting basic criteria of UO_2 fuel powder.

INTRODUCTION

The final quality of sintered UO_2 fuel ceramic pellets depends on starting UO_2 powder characteristics as well as: specific surface area (SSA), oxygen to uranium ratio (O/U), particle size (PS), bulk density (BD) tap density (TaD) .... In all cases, however, the dependence on the characteristics of the precursor from which the powder is obtained by thermal decomposition [3], these precursor conditions include ammonium diuranate decomposition, wet – cake ADU drying, ADU thermal decomposition and U_3O_8 reduction to UO_2 powder.

The research programme reported here has been set up in a attempt to determine by means of the technological parameters of UO_2 powder production process, which depends on technical characteristics of UO_2 powder. The contents of the paper is as follows:

1. Study on the perfection of the reduction process U_3O_8 to UO_2 in the mixed gas of $3H_8 + N_2$ in inactive condition.

2. Study, design and production of device system described as rotary furnace and is carried out studying in 4 steps of technology for producing UO_2 powder from ADU in the system of rotary furnace.

3. The properties of UO_2 powder measured include specific surface area (SSA) by means of BET;

Particle size distribution by means of photography method; O/U ratio by mean of U^{6+}/U^{4+}. Ratio, bulk density, tap density, compact-ability, and sinter-ability.

EXPERIMENTS AND RESULTS

1. The starting conditions

1.1. Materials

The ammonium diuranate-ADU is input material. It is precipitated from uranyl nitrate solution $UO_2(NO_3)_2.xH_2O$ and ammonium hydroxide NH_4OH. The main technological parameters of the precipitation as follows:

- Purified uranyl nitrate solution with a concentration of 80 to 120g U/l.

- Temperature of precipitation: 65-70^{0}C

- Duration of all process: 24 hrs, in this aging time is 6 hrs.

1.2. Equipments and experimental tools.

Equipments and experimental tools are used in studying on the perfection of the reduction process in inactive condition.

- Dryer with operating of temperature grate is automatic control system, the wrong grate of temperature $\pm$ 2^{0}C

- The maximum temperature of LOBA tube furnace (made in RU German) is 900^{0}C

- The boat for ADU is made of Al_2O_3

* They are used in studying device for latter process:

The rotary furnace with technical specification, it is design and production by research programme.

+ Capacity:	100g to 1000g UO_2 powder/ one time
+ Temperature:	room temperature to max 800^{0}C.
Rotary speed:	0 to 10 revolutions/ min, continuously sloppy control.
Time of operation:	Continuously operation 24 ^{h}/24 ^{h}

Absolutely vapor resistance on the condition or rotary movement and high temperature. Use safe H_2 gas pressure.

2. Experiments and Results:

2.1. Study on the perfection of the reduction process in inactive condition:

The main technological process of UO_2 powder from the ADU can be presented as follows:

ADU $\rightarrow$ Dry $\rightarrow$ Calcination $\rightarrow$ reduce $\rightarrow$ Stabilization $\rightarrow$ UO_2 Powder.

In the last years, the studying of the ADU drying and calcination processes had obtained some results. In this paper results of the studying on the reduction technological process will be reported. Orientating experiments of the reduction are carried out on the influence of temperature and time on the UO_2 powder characteristics.

In table 1. Have been collected, for the nine different batches of UO_2 powder each in the same temperature condition and at different time of the thermal treatment, which related to UO_2 powder characteristics.

In table 2. Reported the results studying on the technological reduction process when treatment in different temperature and at one and only time: 4 hours.

Table 1. The influence of the reduction time on the UO_2 powder characteristics.

No	Reduction time, h	UO_2 powder characteristics			
		SSA, m^2/g	PS ≤30μm, %	O/U	BD g/cc
1	1.0	5,240	86,5	2,07	1,49
2	1.30'	5,251	86,2	2,064	1,49
3	2.0	4,903	85,7	2,082	1,49
4	2.30'	4,891	85,1	2,082	1,49
5	3.0	4,460	84,7	2,111	1,56
6	3.30'	4,530	82,6	2,120	1,56
7	4.0	4,372	80,7	2,112	1,56
8	5.0	4,419	78,1	2,145	1,56
9	6.0	4,195	75,6	2,140	1,67

Table 2. The influence of the reduction temperature on the UO_2 powder characteristics.

No	Reduction temperature ^0C	UO_2 powder characteristics			
		SSA, m^2/g	PS ≤ 30μm, %	O/U	BD, g/cc
1	500	7,87	87,1	2,23	1,27
2	550	7,28	86,5	2,18	1,31
3	600	6,53	86,7	2,15	1,22
4	650	5,17	85,2	2,037	1,29
5	700	4,85	81,5	2,017	1,43
6	750	4,27	77,8	2,018	1,61
7	800	3,96	72,7	2,017	1,63

Discussion

Reduction time and reduction temperature influence to UO_2 powder characteristics.

It is common that the higher reduction temperature and the longer reduction time lead particle size and specific surface area in reported to significantly decrease: SSA = 7,87 m^2/g and PS ≤ 30μm in 87,1% at temperature of 500^0C, but it is SSA= 3,96 m^2/g and PS ≤ 30μm in low 72% at temperature of 800^0C.

The authors carefully study on the uranium dioxide powder production [16], according to two processes taking place in the reduction reaction. the first is chemical

change U_3O_8 to UO_2. The second is a physical change. Physical change can take place both in calcination and in reduction. The physical change are greatly dependent on temperature. Very low temperature in calcination and reduction lead to generation of pyrophoric UO_2 powders. On the other hand high temperature excursions mean uncontrolled physical changes resulting in the powder, which lead to problems in compaction and sintering the powder.

2.2. Study on technology of UO_2 powder production from ADU in rotary furnace:

The rotary furnace is designed and made by this research programme. It is similar to the rotary furnace for producing UO_2 powder at the Nuclear Fuel Complex (NFC) in India. The construction of the rotary furnace is the same Fig.1. The technical specification of the rotary furnace may be seen it the section II.1.2.

The research programme is carried out studying on technology of UO_2 powder production from ADU in this rotary furnace with 4 steps: drying, calcination, reduction and stabilization.

The influence of the thermal treatment time and treatment temperature on the UO_2 powder characteristics have been introduced in table 3.

Table 3. The influence of the thermal treatment parameters on the UO_2 powder characteristics in the rotary furnace.

No	(Z_1), °C	(Z_2), h	(Z_3), °C	(Z_4), h	SSA, m^2/g	PS ≤ 10 μm (%)	O/U	BD, g/cc	GD, g/cc	SD, g/cc
1	550	2	550	3	14,20	58,15	2,25	1,09	4,44	10,15
2	750	8	750	6	5,68	49,81	2,04	1,27	5,33	10,20
3	550	2	750	6	7,15	54,79	2,16	1,15	5,20	10,20
4	750	8	700	3	7,04	54,03	2,11	1,21	5,29	10,33
5	550	8	550	6	6,53	54,03	2,08	1,23	5,25	10,27
6	550	8	750	6	6,72	54,03	2,13	1,25	5,31	10,35
7	750	2	750	8	5,39	54,03	2,15	1,33	5,35	10,41
8	750	8	750	6	5,37	54,03	2,07	1,31	5,34	10,27
9	550	8	750	6	6,92	54,03	2,14	1,26	5,27	10,31
10	650	2	600	3	9,87	54,03	2,17	1,17	5,22	10,29
11	650	2	600	6	7,84	54,03	2,11	1,23	5,29	10,31
12	650	8	700	6	6,14	54,03	2,09	1,22	5,30	10,34
13	700	8	700	4	5,22	51,01	2,08	1,25	5,31	10,37
14	700	2	650	6	6,27	51,01	2,11	1,28	5,34	10,30
15	700	8	650	6	5,62	51,01	2,14	1,31	5,35	10,39
16	600	8	600	6	6,43	53,15	2,07	1,27	5,26	10,28

Note: Z_1 - Calcinnation temperature, ^0C

Z_2 - Calcinnation time, h
Z_3 – Reduction temperature, ^{0}C
Z_4 - Reduction time, h
SSA- Specific Surface Area, g/cc
PS – Particle Size, μm
BD – Bulk Density, g/cc
GD – Green Density, g/cc
SD – Sintered Density, g/cc.

Stabilization process:

Fresh reduced UO_2 powder of medium or high surface area is pyrophoric at room temperature. But formation of a single chemisorbed layer of oxygen at low temperatures or under condition of limited oxygen access prevents pyrophoricity. Stabilization is carried out in rotary furnace similar to that of calcination and reduction except that: there are no heating elements in the equipment. The UO_2 powder to be stabilized is made to simple through a slightly tilted rotating tube, in such a way that the particles are spread out and exposed to limited supply of air + nitrogen. In the condition, UO_2 powder surface is oxide, which forms the U_3O_8 thin film.

It is tenacious and physically protects the inner material from further oxidation.

Study on the stabilization process of UO_2 powder

Three UO_2 powder samples from three experiments are used to obtain in experimental conditions, but have not been stabilized. After stabilization at temperature of 30^{0}C and in duration of 4 hours. At the six UO_2 powder pasts are to keep in duration of sis months. The measuring UO_2 powder characteristics are conducted and the results are presented in table 4.

Table 4. The difference of the stabilized and non-stabilized UO_2 powder characteristics.

No	O/U		SSA (m^2/g)		BD (g/cc)	
	non stabilized UO_2	Stabilized UO_2	non stabilized UO_2	Stabilized UO_2	non stabilized UO_2	Stabilized UO_2
1	+0,17	+0,04	-1,06	-0,23	+0,24	+0,10
2	+0,14	+0,03	-0,99	-0,38	+0,36	+0,04
3	+0,16	+0,03	-0,78	-0,23	+0,35	+0,11

Discussion

After the keeping in 6 months, the characteristics of the all UO_2 powder samples have been changed. But the technique parameters of non stabilized UO_2 powder greatly increased and decrease than stabilized UO_2 powder: O/U ratio and bulk density increased, specific surface area decreased. It is common that, stabilized process of UO_2 powder is very important.

The research programme is carried out studying on technology of UO_2 powder production from ADU in this rotary furnace with 4 steps drying, calcination, reduction and stabilization.

The influence of the thermal treatment time and treatment temperature on the UO_2 powder characteristics

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STUDY ON TECHNOLOGY FOR FABRICATION OF CORUNDUM CERAMICS

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INTRODUCTION

Study on production of advanced ceramics is one of advanced technological aspects. Advanced ceramics is a product of modern material technology. It is widely produced and used in industrial branches all over the world. Corundum ceramics is a ceramics based on alumina (98% of Al_2O_3). The such material production requires materials and additives of high quality (high purity, fine particle size, suitable form), modern equipment for process and characterization and appropriate technology.

The subject "Study on technology for fabrication of corundum ceramics" with the content of study on material and producing corundum grinding ball and crucible have been done for 2 years (2001-2002) at Institute for Technology of Radioactive and Rare Elements.

1. Theory

The materials are classified by the type of chemical bonds between atoms. In ceramics, chemical bonds are ion and covalent. Corundum ceramics is high alumina ceramics which belongs to structural ceramic type. Structure of crystal phase is determined by X-ray diffraction (XRD). Microstructure of ceramics is generally observed by optical microscope or scanning electron microscope (SEM). Density of ceramics that reflects densification of ceramics after sintering is determined by immersion method. The micro-hardness is determined by Vicker method with the loads of several grams to one kilograms. The bend strength is determined by method of 3 points or 4 points. Fracture toughness is determined by the length of cracks at the indentation caused by the load of 10 kg.

The ceramics production consists of steps: starting material treatment, forming, sintering and characterization.

The treatment of starting materials comprises steps of powder mixing and granulation. Sometimes material powders are granulated by handmade method. In the case of commercial alumina powders, granulation is not necessary because they are already granulated and ready for forming. There are some different forming methods: cold die pressing, hot pressing, isostatic pressing, slip casting, tape casting and extrusion.

Corundum ceramics is sintered at high temperature (1550-1600) in the air for 1-2 hours at the temperature increasing speed of 100-120^{0}C/h.

The additives play a very important role in the sintering process because they have many effects: 1/ impel densification and depress grain growth (in the case of MgO), 2/ impel both densification and grain growth (in the case of TiO_2), 3/ depress both densification and grain growth (in the case of Ni_2O_3) and 4/ depress densification

and impel grain growth (in the case of SiO_2). As result of that, the choice of properly additives is really very important.

2. Experiment and result

2.1. Starting materials

- Alumina powder AC-45 , purity ≥98%,
- Alumina powder Al_2O_3 -799, purity ≥98%,
- Alumina powder Alcoa CT-3000SG, purity 99.999%,
- Alumina powder Matroxid KMS-92 having composition of 96%wt Al_2O_3, 2.5%wt CaO, 3.6%wt SiO_2, 1.5%wt MgO,
- Alumina-zirconia ZTA-85 powder having composition of 83.8%wt Al_2O_3, 15.1%wt ZrO_2, 0.8%wt Y_2O_3, 0.1%wt SiO_2,
- Other additives: MgO (PA), SiO_2 (PA), CaO, kaolin, dolomite, polyvinyl alcohol (PVA), polyethylene glycol (PEG), Duramax D-3021.

2.2 Experiment and result

2.2.1 Preparation of ceramic specimens based on single oxide mixture

Aluminas used as a foundation of ceramic were AC-45, Pechiney and Alcoa CT-3000 SG . Additives were MgO, SiO_2, CaO_2. Additives and aluminas were mixed in order to obtain the mixture containing 92-98% Al_2O_3.

Starting materials were moisturized with 10% PVA solution and then mixed in a ball mill. Grinding time was 24 hours. The obtained mixture was granulated in the size of 1mm. Specimens were formed by hydraulic pressing at the press of 1.2T/cm^2 as bars having dimension of 5x5x40 mm. After drying at 80-100^{0}C for 12 hours, these bars were roughly abraded and their density was determined. The bars were sintered at 1600^{0}C. Temperature increasing speed was 100^{0}C/h. Dwell time was 1 hour. Density of sintered bars was measured by immersion method. The method of 4 points was employed for bend strength determination. Properties of these specimen series have not been verified . The following observations were extracted from the obtained data of physico-mechanical characteristics:

- In the same preparing conditions, the smaller granule size powder has, the better densification is.

- The effect of MgO in impelling the densification and of SiO_2 in depressing the densification were clearly observed by comparing density of different ceramics specimens having different MgO and SiO_2 compositions.

- Bend strength of specimens containing CaO was higher than others.

2.2.2 Preparation of ceramics based on commercial alumina

Ceramic specimen series were prepared based on commercial alumina (Alcoa CT-3000 SG, Matroxid KMS-96, KMS-92, and ZTA-85). These aluminas were already granulated. Green specimens were prepared in the same way as the one used for specimens based on single oxides. They were sintered at different temperatures (1550, 1600, 1650^{0}C). Dwell time was 1-3 hours. Physico-mechanical properties of specimens were thoroughly estimated (Data showed in the table).

2.2.3 Preparation of grinding ball and crucible

Grinding ball

Starting materials used for preparing corundum grinding ball comprise alumina having high purity (>98% Al_2O_3), additives for pressing and for sintering. The starting materials were mixed together in a ball mill for 20 hours and moistured with 10% PVA solution. The obtained moisturized compounds was granulated in the size of 1mm. Green compacts were made in a cylinder shape by hydraulic press using steel die at the press of 1.2-1.5T/cm^2 and in a spherical shape by hydro-isostatic using latex die at the press of 1.0-1.2T/cm^2. After drying at 80^{0}C for more than 20 hours, the compacts were sintered at 1550^{0}C. Temperature increasing speed was 100^{0}C/h. Dwell time was 1-2 hours.

Crucible

Hydro-isostatic press: Starting material powder was mixed with the binder of 10% PVA solution and then granulated in the size of 1mm. The obtained compound was pressed in latex die having a polished steel model at press of 1.5T/cm^2. After drying at 100-120^{0}C, the green crucibles were sintered at 1600^{0}C for 2 hours. Temperature increasing speed was not more than 100^{0}C/h.

Characteristics of corundum ceramic specimens

Starting materials	Specimen symbol	Density		Microhardness Hv 1.0	Fracture toughness MPa.m$^{1/2}$	Bend strength MPa
		g/cm^3	% TD			
Alcoa CT-3000 SG	A-11-1	3,93	98,5	1752,0 + 68,0	3,57 + 0,37	308,0 + 38.9
	A-12-1	3,94	98,7	1802,4 + 36,4	3,22 + 0,30	330,0 + 59,5
	A-21-1	3,93	98,5	**1959,0 + 54,9**	**4,02 + 0,41**	270,7 + 17,6
	A-22-1	3,94	98,7	1821,0 + 68,2	3,52 + 0,34	**349,4 + 13,8**
ZTA-85	ZTA-11-1	4,13	98,3	1822.,2 + 39,6	3,16 + 0,25	459,4 + 53,5
	ZTA-12-1	4,15	98,8	1760,2 +92,4	3,54 + 0,26	452,2 + 38,1
	ZTA-21-1	4,15	98,8	**1825,0 + 47,5**	3,30 +0,14	400,2 + 12,2
	ZTA-22-1	4,16	99,1	1786,2 + 36,0	**3,58 + 031**	**46,.1 + 0,5**
Matroxid MKS-96	M96-11-1	3,72	94,4	1314,0 + 60,7	3,66 + 0,39	235,6 + 2,3
	M96-12-1	3,77	95,7	1376,8 +35,0	**4,48 + 0,47**	272,0 + 29,4
	M96-21-1	3,75	95,,2	1351,0 +50,2	3,10 + 0,55	237,5 + 21,4
	M96-22-1	3,79	96,2	1286,0 + 69,9	4,02 + 0,48	255,5 + 50,0
	M96-12-3	3,77	95,7	**1355,4 + 42,2**	3,49 + 0,60	**307,1 + 8,1**
	M96-22-3	3,80	96,4	1279,4 + 87,0	3,02 + 0,17	286,5 + 39,7
	M96-13-2	3,77	95,7	1103,3 + 98,7	3,20 + 0,32	258,5 + 5,7
	M96-23-2	3,80	96,4	975.7 + 128,.0	3,16 + 0,36	273,9 + 6,4
Matroxid MKS-92	M92-11-1	3,67	94,3	1250,0 + 52,1	4,20 + 0,88	290,8 + 75,1
	M92-12-1	3,66	94,1	1200,0 +25,0	3,88 + 0,48	289,4 + 35,9
	M92-21-1	3,68	94,6	1184,4 + 59,4	3,68 + 0,35	**330,6 + 15,4**
	M92-22-1	3,67	94,1	1263., + 129,0	**4,03 + 0,46**	324,1 + 14,4

	M92-12-3	3,67	94,3	1173,6 + 60,0	2,92 + 0,19	262,7 + 22,6
	M92-22-3	3,68	94,6	1178,0 + 56,2	2,93 + 0,09	268,7 + 39,7
	M92-13-2	3,66	94,1	**1294,,2 + 76,7**	3,18 + 0,17	311,3 + 10,4
	M92-23-2	3,66	94,1	1051,0 + 39,4	3,13 + 0,30	293,6 + 10,5

Slip casting

Gypsum moulds were made by mixing plaster with water, whose content was 70-80%wt. Slurry comprises 1000.00g of Alcoa-3000 SG; 250.61g of water; 5.00g of Duramax D-3021. The slurry was mixed in a ball mill for 24 hours and poured into the gypsum mould and remained in the mould for 5-7 minutes. After green crucibles were taken out of the moulds, they were kept at room temperature for about 24 hours to dryness and precalcinated at 1000^0C for subsequent mechanical treatments. For precalcination, dwell time was 30 minutes and temperature increasing speed was not higher than 50^0C/h. The obtained products were polished by abrasive paper and then sintered at 1600^0C. Dwell time was 1 hour. Temperature increasing speed was 120-150^0 C/h.

CONCLUSION

- Sintering speed and sinter-ability decreased in the order of the powders: ZTA-15, Alcoa CT-3000SG, KMS-96, KMS-92 and decreased with decreasing press for forming.
- Densification of ceramic specimens was satisfied (94-98% TD). Data of micro-hardness, fracture strength, bend strength of 4 points all reached standard features of corundum ceramics.
- In specimen A21-1 only phase of α-Al_2O_3 existed while two phases, α-Al_2O_3 and T-ZrO_2 , existed in specimen ZTA11-1. T-ZrO_2 was a strong effective additive in improving physical-mechanical characteristics of corundum ceramics.
- In the cases of M96-22-1 and M92-11-1, it seemed that a liquid phase occurs during sintering. In these specimens, additives is mainly located at the grain boundary, causing the existence of Al-Mg and Al-Ca silicates.
- The simultaneous existence of these phases was confirmed by energy dispersion spectrometry.
- Appropriate conditions for production of these corundum ceramic specimens were successfully found.
- Obtained products: 50 kg of grinding ball, 60 corundum crucibles.

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