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### Abstract

Since 1973, when a laboratory conceived for the safe manipulation of a few hundred grams of plutonium was built, the CNEA (Argentinean Atomic Energy Commission) has been involved in the small-scale development of MOX fuel technology. The plutonium laboratory consists in a glove box facility ( $\alpha$  Facility) featuring the necessary equipment to prepare MOX fuel rods for experimental irradiations and to carry out studies on preparative processes development and chemical and physical characterization. The irradiation of the first prototypes of (U,Pu) $O_2$  fuels fabricated in Argentina began in 1986. These experiments were carried out in the HFR (High Flux Reactor)- Petten, Holland. The rods were prepared and controlled in the CNEA's  $\alpha$  Facility. The post-irradiation examinations (PIE) were performed in the KFK (Kernforschungszentrum Karlsruhe), Germany and the JRC (Joint Research Center), Petten. In the period 1991-1995, the development of new laboratory methods of co-conversion of uranium and plutonium were carried out: reverse strike co-precipitation of ADU-Pu(OH) $_4$  and direct denitration using microwaves. The reverse strike process produced pellets with a high sintered density, excellent microhomogeneity and good solubility in nitric acid. Liquid wastes showed a very low content of actinides and the process is easy to operate in a glove box environment. The microwave direct denitration was optimized with uranium alone and the conditions to obtain high density pellets, with a good microstructure, without using a milling step, have been developed. At present, new experiments are being carried out to improve the reverse strike co-precipitation process and direct microwave denitration. A new glove box is being installed at the plutonium laboratory, this glove box has process equipment designed to recover scrap from previous fabrication campaigns, and to co-convert mixed U-Pu solutions by direct microwave denitration.

## 1. INTRODUCTION

A program for the small-scale development of MOX fuel technology at the CNEA-Argentina was initiated soon after commissioning of a plutonium laboratory ( $\alpha$  Facility) in 1973. The aim of this program was to study MOX fuel preparation processes; to produce prototype MOX fuel rods for experimental irradiations and to master safety issues related to plutonium handling.

Six rods containing (U,Pu) $O_2$  MOX fuel were fabricated and controlled in the CNEA's  $\alpha$  Facility [1], and the irradiation of these prototypes began in 1986 [2] in the HFR- Petten, Holland.

The first rod has been used for destructive pre-irradiation analysis. The second one was a pathfinder to adjust systems in the HFR [3]. Two additional rods including iodine doped pellets, were intended to simulate 15.000 MWd/t(M). The remaining two rods were irradiated until 15.000 MWd/t(M). One of them underwent a final ramp with the aim of verifying fabrication process and studying the behavior under power transients. The postirradiation examinations were performed in the Kernforschungszentrum Karlsruhe, Germany and in the JRC, Petten.

In the period 1991-1995, two processes of co-conversion of uranium and plutonium have been studied under IAEA's Research Contract N° 6742: reverse strike co-precipitation of ADU-Pu(OH) $_4$  and direct denitration using microwaves.

The selection of these processes was based in their potential advantages: relative simplicity of operation, feasibility to be adapted to glove box work, possibility of producing high density pellets with good microhomogeneity and nitric acid solubility.

At present, new experiments are being carried out to improve the reverse strike co-precipitation process and direct microwave denitration. A new glove box is being installed at the plutonium laboratory, this glove box has process equipment designed to recover scrap from previous fabrication campaigns, and to co-convert mixed U-Pu solutions by direct microwave denitration.

## 2. EXPERIMENTS AND RESULTS

### 2. 1. Irradiation test

The rods were originally designed for the MZFR (MehrZweckForschungsReactor) Karlsruhe, Germany. However, due do the decommissioning of that reactor, the experiments were performed in the HFR, Petten. Some rod dimensions (i.e. fuel length) and the plugs geometry have been modified to meet the specifications of HFR.

Three types of fuel rods were fabricated. Table I shows their main characteristics.

TABLE I. FUEL RODS CHARACTERISTICS (U/PU MIXED OXIDE)

(a) Fuel rod type	A.1	A.3	A.4
Length	24.1	17.9	17.9
Pellets number	21	16	16
Compensating pellets number	1	-	-
Doped pellets number	-	1	3
Doping material	-	I	CsI / Mo
Doping material (mg)	-	3	6.604±0.017 / 1.373±0.036
Simulated burnup (MWd/ton (M))	-	13761±4933	14800±390
Filling gas	He	He	He
Filling pressure (atmosphere)	1.15	1.15	1.15
<b>(b) Pellets</b>			
Density	10.52±0.04 g/cm <sup>3</sup>		
Pellet height	1.12±0.01 cm		
Pellet diameter	1.040±0.001 cm		
Pu <sub>fiss</sub> /U+Pu <sub>met</sub>	0.53%		
Enrichment (U235+Pu)	1.25%		
O/M relation	2.00		
Dishing volume	25±5 mm <sup>3</sup>		
<b>(c) Cladding</b>			
Material	Zry-4		
Inner diameter	1.17 cm		
Thickness	0.06 cm		

The first irradiation was with the A.1.4 rod to adjust systems in the HFR reactor instruments. The experiment verified the response of the HFR reactor control systems with our MOX rod.

The duration of the irradiation was about 100 h. It included a final tamp test. The maximum power level reached was 439 W/cm. [4]

A second experiment started with the irradiation of two fuel rods both containing iodine compounds as a dopant to simulate the effect of extending burnup [2].

Fuel rods A.3 and A.4 have been employed for this second experiment. The irradiation, carried out during 15 days consisted in a steady state condition including two powers cycling between 120 and 290 W/cm and a final ramp to reach 400 W/cm [5]. This final power level lead to a "hoop stress" of about 170 MPa which is likely to induce microcracks in the inner surface of the cladding, representing an incipient defect produced by stress corrosion cracking without reaching the failure threshold of the tubing.

CsI and Mo were mixed with UO<sub>2</sub> (rod A.3), the mixture was introduced in central holes drilled in three pellets and hand pressed. Pure iodine was introduced in one pellet (rod A.4) with the aim of comparing its effect on the cladding with that of combined iodine (CsI in rod A.3).

Behavior of rods A.3 and A.4 was previously described [6-8]:

- there were no failures in the cladding
- microcracks in the inner surface of the cladding were detected during PIE.

The third irradiation experiment (BU15) was carried out with fuel rods A.1.2 and A.1.3, both similar to the pathfinder rod. The power history for this irradiation test was proposed upon calculations made with the BACO (BARra COmbustible) code. The target of the irradiation was an average burnup of 15000 MWd/t (M) (more or less twice the final burnup for Atucha I fuel), which was reached in two main steps:

- (a) Up to a burnup of 8100 MWd/t (M) the rods were irradiated in different locations of the HFR core.
- (b) Up to a burnup of 15000 MWd/t (M) both rods were irradiated in the Pulse Side Facility (PSF) of the reactor.

During the steady state irradiation both rods were assembled together through a threaded coupling and after termination of the bulk irradiation phase both fuel rods were disassembled in the Petten hot cells.

After the steady rate irradiation at an average power level of 230 W/cm one of the rods (A.1.3) was submitted to a power ramp which was divided in two parts:

- (1) a short period of pre-irradiation power level in the PSF position which will be used for the ramp test in order to determine the experiment power versus PSF position characteristics and
- (2) a ramp test starting with a ramp rate of 50 W cm<sup>-1</sup> min<sup>-1</sup> from nearly zero power to maximum 420 W/cm linear fuel rod power and followed by a 6 h steady-state holding at this maximum power.

Both rods behaved during the stationary phase as it was expected:

- no rod failures were detected
- no fabrication defects were evident

During EOL (End of Life) power ramp, A.1.3 behaved as follows:

- a maximum power of 390 W/cm was reached due to the low quantity of remaining fissile material in the fuel rod,
- the power ramp had to be finished earlier than planned due to an increase in activity in the coolant circuit,
- visual inspection of the fuel rod revealed the existence of a small circular hole in the cladding [9].

Post-irradiation examinations of the BU15 experiment included: visual inspection, Eddy current check, neutron radiography, gamma scanning, dimensional control and microscopic examinations including  $\alpha$  and  $\beta$  autoradiography, pore and grain size, evaluation and identification of the failure in pin A.1.3. The main featuring of PIE and a comparison between the results of the PIE and the BACO code simulation have been presented previously [10,11]. The conclusions can be summarized as it follows:

- All the rods behaved as expected, including the defective rod.
- The maximum hoop stress and cladding radial contact pressure appeared in the axial section corresponding with the failure.

- There was a good agreement between experimental results and BACO code predictions, especially with respect to the relationship between the failure position and characteristics and the mechanical demands predicted.
- The presence of microcracks inside the cladding in the doped rods, the coincidence between the predicted and measured pellet cladding gap values, the temperature calculated, the microstructure observed and the grain size distribution indicated a good BACO code evaluation.
- Post irradiation examinations confirmed the calculated value of hoop stress ( $\sigma_v = 207$  MPa). This value indicated that PCI (Pellet Cladding Interaction) - SCC (Stress Corrosion Cracking) was a mechanism likely to produce the failure.

## 2. 2 Co-conversion methods

Two co-conversion methods were selected: reverse strike co-precipitation and direct denitration using microwaves. They usually include several process steps shown in Fig. 1.

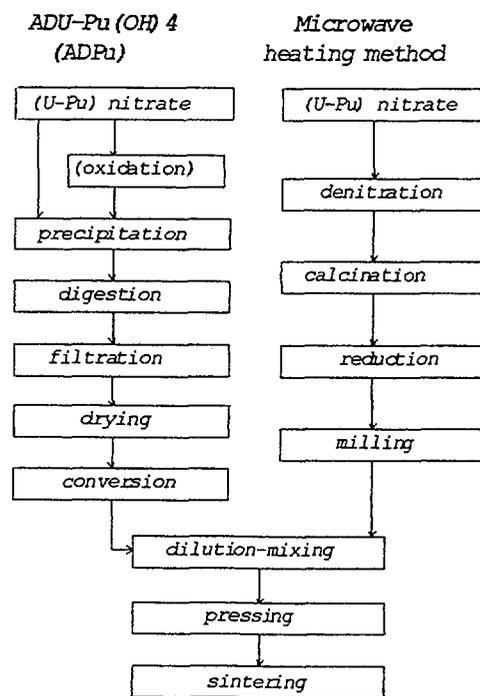


FIG. 1. Diagram of co-conversion methods

### 2. 2. 1. Reverse strike co-precipitation method

In this process, a nitric actinide solution and ammonia gas are continuously fed into a reactor which is thermostated at the chosen temperature and having an initial volume of  $\text{NH}_4\text{OH}$  solution at pH between 4 and 6. The pH is kept constant during all the process while the nitric actinide solution and the ammonia (gas) are injected in the reactor with continuous stirring to prevent preferential precipitation and assure a good precipitate filterability.

When the reaction is finished, the stirring of the slurry continues for one more hour, always at the same temperature. Afterwards the slurry is filtered and dried. The precipitation apparatus is shown in Fig. 2.

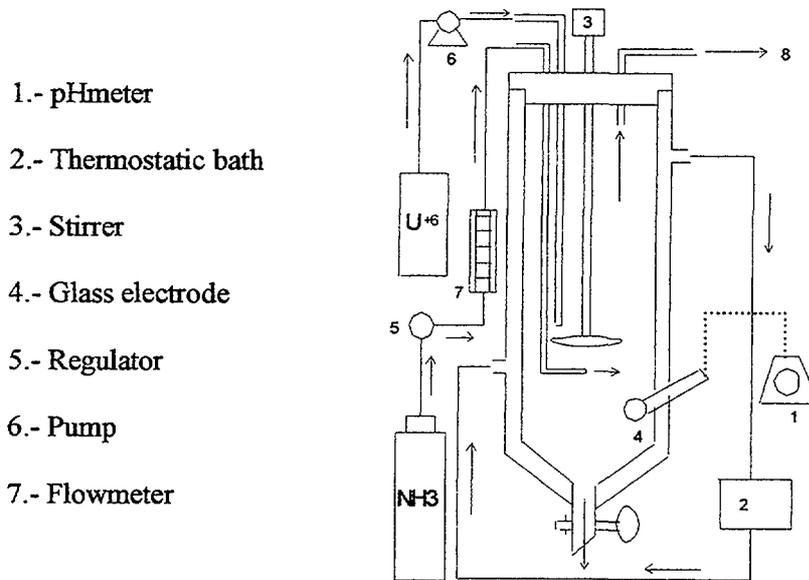


FIG. 2. Precipitation apparatus

The reverse strike process was first studied with uranium alone in the same conditions of the co-precipitation with plutonium.

Two parameters were specially considered in this step:

- Temperature (20, 40 and 60 °C)
- Actinide concentration in the uranyl-nitrate feeding solution (100, 200 and 300 g[U]/l)

The ADU (ammonium diuranate) obtained was converted to UO<sub>2</sub> (uranium dioxide) in two steps:

- decomposition to UO<sub>3</sub> in N<sub>2</sub> atmosphere at 400 °C and
- reduction of UO<sub>3</sub> to UO<sub>2</sub> in N<sub>2</sub>/H<sub>2</sub>(8%) atmosphere at about 650 °C during one hour.

The following analytical determinations have been carried out on the powders prepared under each condition of precipitation: O/M (oxygen/metal ratio), SSA (specific surface area), particle size distribution, flowability and tap density ( $\delta_{tap}$ ).

Several milling, pressing and sintering conditions were studied.

Milling was carried out in a ball-mill with stainless steel jar and Cr-steel milling balls. Tests with different charge ratios (ratio between balls weight and powder weight), different ball size distributions and different milling times were performed.

In the pressing, two different kinds of lubrication were used:

- Internal lubrication: zinc stearate was added in different percentages in some batches
- External lubrication: a vegetable oil was used to lubricate the press die without adding zinc stearate.

Finally, the sintering process was studied choosing two heating profiles shown in Fig. 3.

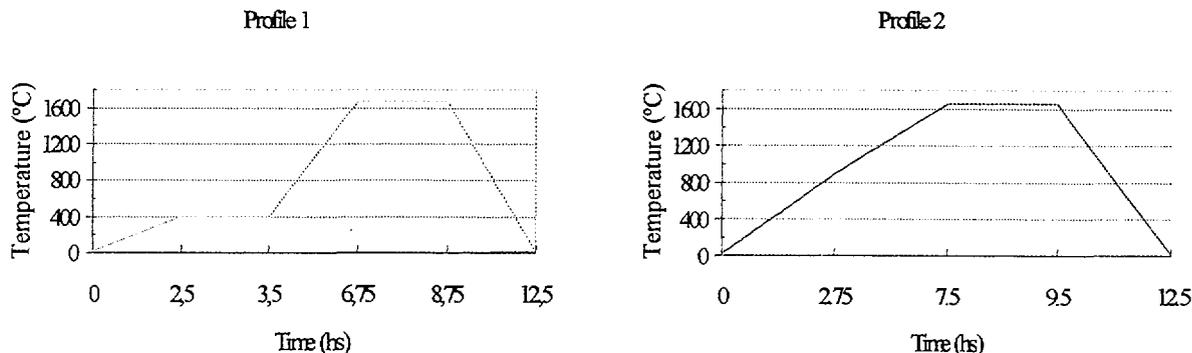


FIG. 3. Time-temperature schedule of the sintering cycles

The results of this step can be summarized as it follows:

- At 20 °C the precipitate was gelatinous and difficult to filtrate
- UO<sub>2</sub> powders obtained from solutions precipitated at 60 °C (with 200 g/l and 300 g/l of actinides in initial solution concentration) showed best sinterability.
- Low content of actinides in liquid wastes (lower than 3 µg/l) and easy operativity.
- UO<sub>2</sub> powders were free flowing, making suitable the automatic pressing.
- The different heating profiles used for sintering showed an important influence on the final density of the sintered pellets.

Typical characteristics of ADU and UO<sub>2</sub> powders obtained from nitric solutions containing 200 and 300 g[U]/l which were precipitated at 60 °C (Types 200/60 and 300/60 respectively) are shown in Table II, and typical results obtained from the pressing and sintering tests on milled and unmilled UO<sub>2</sub> are shown in Table III.

TABLE II. CHARACTERISTICS OF BATCHES OBTAINED FROM ADU PRECIPITATED

Type	ADU		UO <sub>2</sub>							
	$\phi^a$ (µm)	SSA (m <sup>2</sup> /g)	Unmilled				Milled			
			$\phi^a$ (µm)	SSA (m <sup>2</sup> /g)	O/M	$\delta_{tap}$	$\phi^a$ (µm)	SSA (m <sup>2</sup> /g)	O/M	$\delta_{tap}$
200/60	13.5	2.44	8.3	3.4	2.08	2.49	3.0	4.08	2.11	3.39
300/60	12	2.07	8.3	3.4	2.06	2.54	3.0	4.11	2.04	3.46

<sup>a</sup> Average particle diameter

TABLE III. PRESSING AND SINTERING TESTS OF UO<sub>2</sub> POWDERS (VIA ADU<sup>a</sup>)

		Compacting pressure <sup>b</sup> (MPa)	Green density g/cm <sup>3</sup>	Sintered density	
				G/cm <sup>3</sup>	% Theoretical
Unmilled	Profile 1	500	6.54	9.85	89.9
		600	6.71	9.95	90.8
	Profile 2	500	6.54	10.26	93.6
		600	6.71	10.33	94.3
Milled	Profile 1	500	6.53	10.40	94.9
		600	6.74	10.42	95.1
	Profile 2	500	6.53	10.55	96.3
		600	6.74	10.57	96.4

<sup>a</sup> 300/60 Type

<sup>b</sup> external lubrication

The ceramographic analysis of the sintered pellets revealed a homogeneous distribution of pores with average values of grain size between 6 and 7  $\mu\text{m}$ .

The equipment for mixed reverse strike coprecipitation with  $\text{Pu}^{+4}$  was placed inside a glove box. Based on the results obtained in previous steps (with uranium alone), tests with a mixed (U,Pu) solution were carried out.

The conditions were:

-	Solution volume (ml)	474
-	$\text{Pu}^{+4}$ contents (g/l)	57.03
-	$\text{U}^{+6}$ contents (g/l)	228.12
-	Ratio (U/Pu+U)x100	20
-	pH of precipitation	4.5 - 5.5
-	Thermostatic bath temperature ( $^{\circ}\text{C}$ )	60

These tests indicated:

- Low content of uranium and plutonium ( $< 3 \mu\text{g/l}$ , N.D.) in liquid wastes.
- Homogeneous green-yellowish color in the obtained product
- Excellent filterability of the precipitate.

Several tests of calcination-reduction were performed in order to reduce de SSA of the powders and to stabilize towards oxidation in air. At 700  $^{\circ}\text{C}$  during 3 hours the characteristics of the (U,Pu) $\text{O}_2$  were: SSA 5.74  $\text{m}^2/\text{g}$  -  $\delta_{\text{tap}}$  1.46  $\text{g}/\text{cm}^3$ .

The pressing of these powders was difficult due to their low tap density. They had to be pressed between 110-170 MPa and sintered using the profile 2 (see Fig 3). Table IV shows the obtained results (average).

TABLE IV. PRESSING AND SINTERING TEST OF (U<sub>0.8</sub>,Pu<sub>0.2</sub>)O<sub>2</sub> POWDERS

Green density ( $\text{g}/\text{cm}^3$ )	Sintered density ( $\text{g}/\text{cm}^3$ )	% Theoretical Density
4.8	10.63	96.2

The sintered densities were very high and it can be adjusted to agree with the specified values for each reactor.

Ceramographic analysis showed:

- inhomogeneous pore distribution with a higher concentration of pores in some zones of the sintered pellets,
- homogeneous grain size distribution, and
- absence of zones with  $\text{PuO}_2$  islands.

Nitric acid solubility tests were performed in order to verify the solubility of unirradiated sintered pellet. Pellets were attacked with boiling nitric acid 7 M during 6 hours using a reflux condenser. They were totally dissolved at the end of 3 hours' attack.

The result was  $S\% = 99.7\%$ , where the percentage solubility (S%) is defined as:

$$S = P \times (P+N)^{-1} \times 100$$

where

- P is the mass of plutonium found by analysis in the dissolution mixture,  
 N is the mass of plutonium found in the undissolved solid retained by filtration (with blue ribbon paper).

### 2. 2. 2. Direct denitration using microwaves

The direct denitration using microwaves heating method was already used in other countries for mixed oxides preparation [12, 13]. As in the case of coprecipitation, a preliminary set of tests with uranium was carried out.

The direct denitration of a solution consists in the evaporation of the nitric acid and the subsequent thermal decomposition of the solid formed.

The solutions used in different essays were prepared in batches of 2 liters with an uranium concentration of 280 g[U]/l in nitric acid 1 M. These levels of concentration and acidity were chosen in order to avoid polymerization and precipitation effects on plutonium [14].

The denitration was carried out in standard kitchen microwave ovens having the same power level but featuring different chamber capacity and geometric inlet of microwaves in the denitration chamber. It was necessary to make adaptations to prevent  $\text{NO}_3\text{H}$  vapors and nitrogen oxides (NOX) corrosion on the structure and electronic circuits of the oven (see Fig. 4).

1. Denitration chamber
2. Pyrex glass vessel
3. Vacuum gauge
4. Refrigerator
5. Condenser
6. Scrubber
7. Vacuum pump
8. Microwave power unit
9. Microwave guide

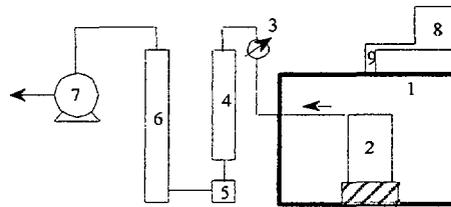


FIG. 4 Denitration device

The solutions were placed into Pyrex glass vessel. The orange product attained, easy to remove from its container, sponge-like, was crumbled with a hand mortar.

Different parameters of each step were studied:

- Based on results obtained in previous works [15], ammonium nitrate was added to the initial solution of one of the batches in order to study the influence of this salt on the final product quality.
- Different reduction and sintering profiles were tried.
- The influence of jar rotation speed, time and charge ratios (see Sec. 2. 2. 1.) was determinate in the milling process.
- Internal lubrication and external lubrication were performed in the pressing step.

The produced powders were inspected for:

- Particle size distribution by means X-ray monitored sedimentation technique.
- SSA by adsorption of helium method.
- O/M ratio by spectrophotometric technique.
- Tap density
- Morphology using a scanner electronic microscope (SEM).
- Composition by X-ray diffraction.

The following conclusions can be drawn:

- Significant improvements on the final pellet density were not observed with the addition of ammonium nitrate in the starting nitric solution.
- The obtained product from the denitration is a mixture of  $\text{UO}_3$ ,  $\text{U}_3\text{O}_8$  and  $\text{UO}_2(\text{NO}_3)_2 \cdot x\text{H}_2\text{O}$ .
- The low content of actinides in the liquid wastes generated during the process implies a recovery of about 99.996 % of the original actinide mass (uranium concentration in the distilled liquids less than 19  $\mu\text{g}/\text{ml}$ ).
- It is possible to control the SSA of  $\text{UO}_2$  powders with different calcination-reduction cycles but this parameter has almost no influence on the sintered density of the pellets.
- The milling process reduce the average size of the particles and increases de SSA, the tap density and the green density. The final result is an important increase in the sintered density.
- The presence of a internal lubricant does not affect the pellet densities if the heating rate, and the dewaxing time and temperature are properly chosen for the lubricant that is used.
- Lower heating rates above 900 °C allow a better densification of the ceramic material during the sintering process. The best conditions can be obtained by dilatometry studies [16].

It was found that the powders prepared in microwave oven 1 reached higher green and ntered densities for the same pressing conditions than those produced in microwave oven 2 (see Fig. 5). Then, further studies were carried out to explain this phenomenon.

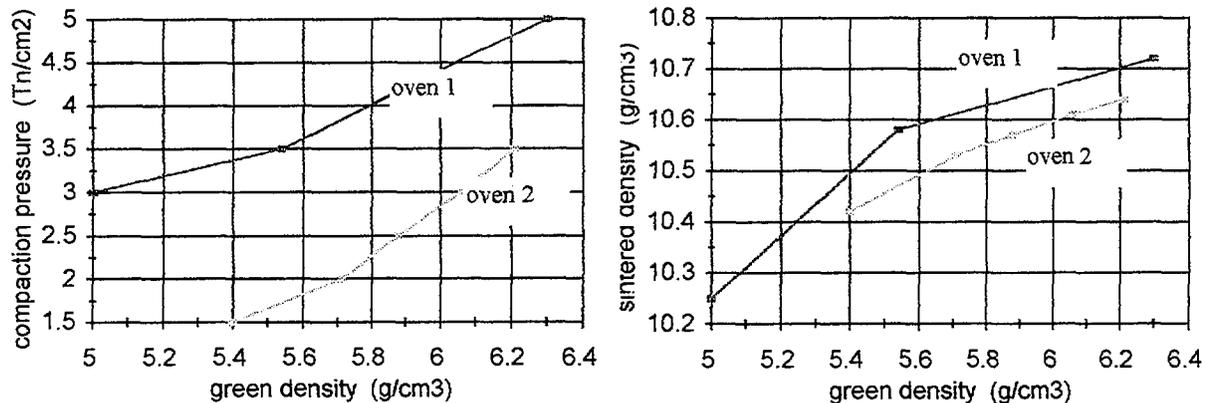


FIG. 5 Pressing a sintering test of  $\text{UO}_2$  powders obtained from different microwave ovens (1  $\text{Tn}/\text{cm}^2 = 100 \text{ MPa}$ )

Several tests under different pressures within the closed reactor vessel have been carried out. The results of these tests are shown in Table V.

TABLE V. CHARACTERISTICS OF UNMILLED  $\text{UO}_2$  POWDERS OBTAINED UNDER DIFFERENT PRESSURE CONDITIONS.

Pressure (mbar)	972 (A)	632 (B)	143 (C)
O/M	2.02	2.05	2.06
$\phi$ ( $\mu\text{m}$ )	0.5	0.45	3.7
SSA ( $\text{m}^2/\text{g}$ )	2.10	2.29	1.90
Tap density ( $\text{g}/\text{cm}^3$ )	2.4	2.35	2.24
Bulk density of denitrated product ( $\text{g}/\text{cm}^3$ )	0.57	0.54	0.40

A reduction in the pressure within the reactor vessel causes a decrease on the bulk density of denitrated product and larger average particle size are attained.

The pressing and sintering results for these powders (without a milling process) are shown in Fig. 6. They indicate that the sintered density of the pellets can be controlled by adjusting the pressure in the reactor vessel during denitration. Higher pressures lead to smaller particle average size and consequently to higher sintered density. A patent for this procedure is being produced in Argentina.

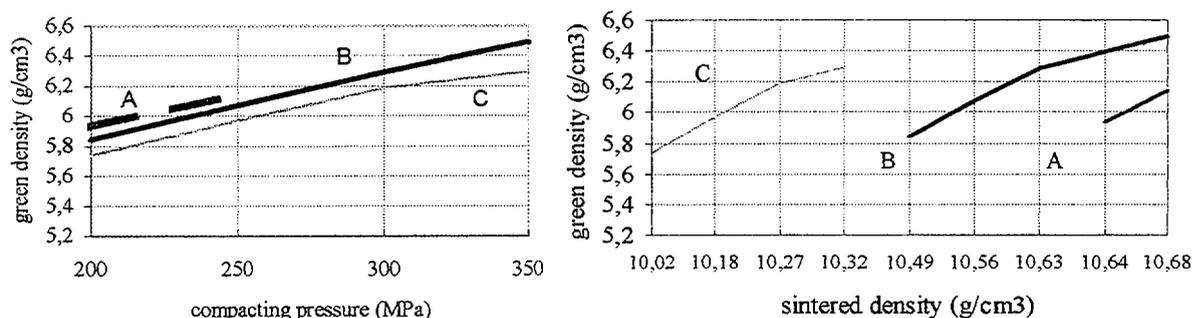


FIG.6 Pressing and sintering test of  $UO_2$  powders obtained from different denitration conditions

At present, a new glove box is being built. The equipment has been designed to recover scraps and to co-convert mixed U-Pu solutions by direct microwave denitration.

### 3. CONCLUSIONS

The continuous operation during more than 20 years of a small laboratory for the development of MOX fuel technology in Argentina produced the following achievements:

- The fabrication and control of MOX fuel rods for an experimental irradiation and a comparison between the experimental results of the postirradiation examinations and the BACO code calculations, producing an upgrade in the code.
- The development of co-conversion processes to produce MOX with significant improvements in the microwave direct denitration method.
- The training of the personnel in safe handling of a few grams of plutonium.
- The development of chemical and physical characterization techniques for MOX fuels.

At present, the research and development continue with the installation of a new glove box. This glove box is equipped to carry out experiments to prepare MOX fuel by direct microwave denitration.

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