STUDIES ON FUEL ELEMENTS FOR POWER REACTORS AT CMN-IPEN

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ABSTRACT

The major part of this paper deals with the production and development of sintered UO_2 pellets for PWR nuclear reactors. performed at CMN-IPEN. The methods of preparation of UO_2 sintered pellets, as well as the tests carried out during processing of the material and the final characterization tests of the sintered pellets are described.

Processes were developed for laboratory and pilot-plant production. Laboratory studies are done to verify the effects of the materials and processing variables on the final product properties. The acquired experience in laboratory production was used to establish stardadization and uniformization of the process to get reproducibility in the pilot-plant. In the pilot-plant production the process was planned to allow for sintered pellets within confident specifications.

Other studies on materials for nuclear fuels, as for example the development on the preparation and characterization of mixtures of oxides (UO_2 - ThO_2) and the preparation and properties of zirconium alloys for fuel cladding are also presented. Coated microspheres were also prepared and characterized for application in HTGR fuel elements. Irradiation tests of these microspheres are being developed.

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1 - INTRODUCTION

The IPEN's Nuclear Metallurgy Center has carried out research for the development of reactor fuels fabrication processes. An area of special importance refers to PWR fuel fabrication particularly after the choice of this reactor type by Brasil.

The main efforts have been concentrated in the methods for making sintered UO_2 pellets. Initially a laboratory for the fabrication of these pellets was installed, in order to develop the techniques adequate for their preparation and the verification of the main parameters influence on the final properties of those sintered pellets[1-4]. Each processing phase is characterized by tests and controls. Testing and controling methods were introduced and conveniently modified when considered necessary for correlating the UO_2 sintered pellets physical properties with the variables of the material being processed and of the process itself. The testing and preparation methods, were developed for UO_2 with natural concentration of U^{235}, but enriched UO_2 was also processed.

In a later phase of the development, a pilot plant for UO_2 fabrication was designed and built [5], aiming the production of UO_2 pellets with characteristics complying with international quality standards. The plant was designed in order to allow for great flexibility in case operational changes would be required during production in and experimental scale. In this way it was possible process standardization concomitantly with easyness to alter stages of processing when working with different initial materials. The assurance for the production of sintered UO_2 pellets with "a priori" determined characteristics are the knowledge and control of the parameters influencing the final properties.

The post-irradiation evaluation (PIE) was also programmed for pellets prepared considering different specification. The results already obtained and new tests now being developed show that the adapted fabrication procedures are acceptable.

The IPEN Nuclear Metallurgy Center know-how in the area of fuel rods assemblying comprises encapsulation of pellets in aluminum tubing[6]. This experience is expected to be extended to fuel rod

made of zirconium alloys. In this respect the development is in a preliminary stage of fundamental properties investigation of these alloys.

Other development involving the study of various concepts of fuel for power reactors were also carried out. In this context it is possible to mention the preparation and characterization of mixed-oxide UO_2 - ThO_2 fuel and of fuel microspheres for HTGR reactor. The microspheres are made in the IPEN Chemistry Engineering Center the pyrobarbon multi-layer coating being deposited at its Nuclear Metallurgy Center. These microspheres are being prepared for post-irradiation evaluation.

This paper presents a succint review of the results obtained in the development of this fabrication and characterization processes adapted for UO_2 pellet production. It is discussed work in progress on the preparation and characterization of other fuel materials. Some results in the area of coating materials are also shown, as well as the potential of the IPEN Nuclear Metallurgy Center installed capacity for future studies.

2 - UO_2 PELLETS

2.1 - FABRICATION OF UO_2 SINTERED PELLETS

The UO_2 pellet fabrication process used is basically UO_2 powder pressing and 1600-1700°C sintering in hydrogen atmosphere.

When working with enriched uranium the starting material is enriched UF_6. Two processes for obtained UO_2 have been used, one designated the ADU (ammonium diuranate) route and the other the so-called AUC (uranyl-ammonium tricarbonate) processing, utilized in Germany[7]. Each of these processes produces powders with different characteristics that will influence the sintered pellets final properties. However both procedures might originate high quality pellets. The process adopted by IPEN for producing UO_2 sintered pellets was that utilizing the UO_3 - ADU route developed in pilot-plant scale by the IPEN Chemical Engineering Center. The processing in the IPEN Nuclear Metallurgy Center starts with ADU air-calcination (700-750°), followed by reduction in H_2 atmosphere. Pellets have also been

fabricated with UO_2 powders produced from AUC[3].

In the pressing of UO_2 - ADU powder a binder is normally used, while for the UO_2 - AUC powders binders are not required. Double--action presses are employed, with green densities of 5 to 6 g/cm^3 for pressures in the 1 to 3 t/cm^2 range. Sintering is carried out at in high temperatures furnaces under H_2 atmosphere, the resulting densities varyng between 94 to 95% of the theoretical value for UO_2 (10,96 $g.cm^3$).

All the parameters influencing pellets final properties are controlled during fabrication. The fabrication process control demands testing and evaluation of the influence of those parameters over the properties of the sintered pellet. The properties of the sintered pellet are also evaluated by means of characterization tests.

As it was mentioned before, the UO_2 powder characteristics are dependent of the preparation method. It has been verifyied that the UO_2 powder characteristics influences significantly the UO_2 pellets pressing and sintering. For this reason tests were developed to allow for a complete characterization of the UO_2 powders used. During fabrication important process variables to be controlled are: compacting pressure, pellet density after pressing, sintering time and temperature. After sintering characterization tests of the sintered pellets are carried out.

2.2 - TESTS PERFORMED

2.2.1 - Powder testing

For characterizing properly the UO_2 powder sinterability, the following physical tests are made: surface area (BET method), average particle size (Fisher method), particle density (the picnometer). granulometric distribution (sieving and sedimentation balance), particle morphology (optical and electron microscopy). The oxygen/metal rat. of the UO_2 powder is also determined.

The values of those UO_2 powder parameters are dependent of the preparation method used and are significant indications of the sinterability and final properties of the UO_2 pellets. There is a simultaneous effect of the various variables and an interrelation

among them in such a way that it is not always possible to individualize the influence of each of them. However, that group of parameter might be used to predict performance differences during processing for UO_2 powders of different origins. For instance, in a comparative study of UO_2 powders originated from AUC and ADU[3], it was shown that the microstructure differences in the UO_2 pellets after sintering is due mainly to morphology differences as shown by scanning electron microscopy. This study suggested, also that the porosity distribution at the powder surfaces could cause differences in the sintering kinetics and microstructure of the sintered pellet. These ideas were recently confirmed by transmission electron microscopy[9]. In this work it was shown a large porosity concentration in the particles of UO_2 originated from AUC, while such porosities are not observed in UO_2 made from DUA. Such surface and bulk porosities differences are factors responsible for different BET surface area values and they can contribute to explain the superior sinterability of UO_2 - AUC relatively to UO_2 - DUA. In another work[10], it is shown the influence of the granulometric distribution under 325 mesh, as determined by a sedimentation technique.

The characteristics of UO_2 powders have also influence their pressing behaviour[9,11]. Other tests also important for the pressing control are carried out: flowability test and determination of its tap density powder (for powder as received, slightly and considerably tapped, respectively). The flowability test informs on the powder die-filling characteristics, while the tap density indicates how much material a certain die cavity can held. The evolution of the three above mentioned tap density values indicates an index related to the ability of the powder particles to accomodate during die-filling and powder pressing. For instances, a powder with an high percentage of fine particles has less flowability, making it difficult the die-cavity powder filling in an automatic press. It was verifyied that the granulometric distribution and particle morphology have fundamental importance in the pressing operation.

2.2.2 - Processing test controls

During processing the UO_2 powder is pressed in cylindrical pellets and sintered. The processing parameters jointly with the UO_2 powder characteristics determine the properties of the sintered pellet.

The pressure values during pressing determine the green density, one of the major parameters in the sintering cycle. For the same conditions, it was shown that for higher green densities, higher will be the density of the sintered pellet. Table I shows the effect of the pressing on the final density of the sintered pellet. The pressure in the pressing stage shall be chosen as a function of the powder characteristics and of the sintering cycle to be utilized. After pressing the pellets must present an adequate degree of physical intergrity, with number of chips and cracks smaller than maximum specified values, always considering the desired characteristics for the final sintered pellet.

The sintering cycle greatly influences the pellet final properties. The normally predetermined parameter desired for the sintered pellet is its density. In the sintering process the density of the sintered pellet is higher for higher sintering times and temperatures. The sintering temperatures normally vary in the range 1500-1750°C, for sintering times between 1,5 and 8 hours depending on the powder characteristics and the pressing conditions. It was recently developed a sintering process in an oxidizing atmosphere, by means of which it was possible to reach high UO_2 density at temperatures as low as 1100°C[12].

During the processing of a certain powder, the four variables discussed above determine the pellets final properties. Considering that the green density is a function of the pressure during pressing, it is possible to consider just the other three variables. Therefore any variation in one of these three independent variables might originate a change in the final properties of the sintered pellets. It is possible to develop tests to check the influence of one more variables over a certain specific property of the sintered pellet, for instance its density. This is a test normally carried out by determining powder behaviour during sintering and designated as sintering test. Figure 1 shows the pellet final density variation as

a function of the pressing pressure, for determined sintering time and temperature.

The dilatometric test gives data on UO_2 sintering kinetics and also relevant information on the pellets sintering cycle[13].

2.2.3 - Sintered pellets tests

After sintering the final properties of the pellets are determined. The majority of such properties are those dependent upon the fabrication process and significant for the pellet in - pile behaviour.

Once verifyied the pellets physical integrity, tests are developed to determine: geometric and immersion densities, dimensional parameters, open and closed porosities, size and distribution of grains and pores (these last two by means of microstructural observations).

The density values associated with the pellet porosity and microstructure are the parameters that characterize the sintering process. For the same value of the final density it is possible to have different microstructure, as it was already discussed, depending on the characteristics of the powder utilized. The interest for the geometry and distribution of porosities increased after the verification of its importance in the in - pile densification phenomenon, observed in power reactors. The densification models now available show the importance of porosity distribution in the control of the phenomenon. For this reason it is necessary to develop a process leading to a pellet with stable porosities. A resintering test is developed with the purpose of checking the porosities structure stability. In this test the pellet is heated up to a temperature higher than the regular sintering temperature and its density variation is measured. Table 1 shows resintering data for pellets processed from powders with different origins.

It is possible to carry out other tests for determining UO_2 pellet properties, as for instance studies relating the porosity influence on UO_2 fracture[15], observing pellet fractures after

compression testing.

A sintered UO_2 pellet to be utilized in a fuel road is normally processed through centerless grinders to attain a uniform final — diameter. All the dimensional tests are made after this operation.

2.3 - FABRICATION IN PILOT-PLANT SCALE

With the experience resulting from laboratory scale production, the construction of a pilot plant was planned for a more substantial pellet production, but envisaging ample flexibility for experimental scale production[5]. This plant was designed to reach specified properties for the UO_2 pellets, using as starting materials various uranium salts or oxides, and in a coherent manner with the objective of getting precise and reproducible data for the different processing phases. Figure 2 presents the pilot-plant lay-out with indications for the various phases of the desired processing.

2.3.1 - Adopted method

The fabrication in the pilot-plant starts with the uranium salt receival. The initial operation is a calcination, in which the uranium salt is transformed in an uranium oxide, U_3O_8. This operation is carried out in a muffled furnace presenting great temperature uniformity in all its internal volume assuring uniformity for the product so obtained. For the development of this operation, the uranium salt is treated in refractory steel trays, assembled in a special mounting.

The obtained U_3O_8 is homogeneized and goes to a sieving operation passing through a 65 mesh sieve, the + 65 mesh fraction being ball--milled, thereafter returning for the sieving operation.

The reduction operation is carried out in crucibles passing through an horizontal semi-continuously tubular furnace, in an hydrogen atmosphere. The furnace has two independently controlled hot-zones, being water cooled in its outlet.

The UO_2 powder is homogeneized being prepared for the pressing operation which, depending upon the final required characteristics, might be developed in a mechanical hydraulic press, using double-action

and a floating die. In case it is needed, a pre-pressing operation may be carried-out in a rotary press, with subsequent breakage and granulation of the pre-pressed pellets.

The green pellets are placed in Mo crucibles and sintered in an hydrogen atmosphere. The furnace has two independently controlled temperature zones, one of them able to reach a maximum temperature of 1750°C in steady-state operational conditions.

After sintering the pellets go through a finishing operation in centerless grinders, in order to obtain the desirable final diameter with great precision.

The pellets not accepted after processing are reoxidized and recirculate starting with the U_3O_8 reduction step.

2.3.2 - Process control

It is desirable to obtain pellets with specific final characteristics. Such final characteristics must comply with determined standards[16]. In order to make this objective possible, tests and controls are developed during the processing leading to standardization and reproducibility. Figure 3 presents a concise operation fluxogram with the tests corresponding to each phase.

The tests carried out in each processing stage are chosen among those previously discussed and presented in this paper, with the addition of two chemical tests, the spectrometric determination of trace contaminants plus humidity analysis. In each stage testing was established, to assure the final product to be complying with the desired specification. Besides it through these tests it is possible to obtain supplementary information allowing a redefinition of parameters in order to avoid undesirable changes of the final product. In the final sintered pellets the following tests are performed: 1 - chemical analysis; 2 - oxygen/metal (O/U) ratio; 3 - residual gases; 4 - microstructures; 5 - density; 6 - dimensional controls; 7 - state of the surface; 8 - density; 9 - resintering tests.

The tests in powder lots are carried out in samples taken out of the lots using the quartering sampling method. In the green pellets a visual check is carried out for all them, while the density

determinations are made by samples taken out of the pellet batches in pre-established time intervals. The same procedure is adopted for sintered pellets that are submited to a 100% visual inspection, while the other tests are carried out by statistical sampling.

Concomitantly with the testing, they are properly registered jointly with the control documentation for each stage of the process, stating the weight accountability relevant for the managing of the operations as well for the inventary of the material being processed. In this way, each lot of sintered UO_2 pellets has all the documents gathered during the various phases of the processing.

2.4 - IRRADIATION TESTS

It is wide recognized the need for fabricating pellets complying with well determined specifications to attend the requirements of utilization in PWR's. The presently adapted IPEN specifications are the results of two decades spent with design, development and testing. However, the definitive confirmation of their engineering reliability may be sustantial only when product performance in-pile becomes available. One way to obtain this type of information is to submit the pellets to irradiation tests, were actual conditions of the fuel utilization are simulated.

With this objective it was established a joint program with the Brazilian Nuclear Energy Commission and the Nuclebrás company, for the irradiation abroad of UO_2 fuel pellets fabricated at the IPEN Nuclear Metallurgy Center. The results obtained up to now are encouraging and they demonstrate the engineering validity of the adapted development line and the IPEN competence to fabricate pellets properly qualifyied.

3 - OTHER RESEARCH IN THE AREA OF POWER REACTOR FUEL

The IPEN Nuclear Metallurgy Center facilities have the capacity to permit research development of other materials utilized in power reactor fuels. The Center has equipment proper for metallic and ceramic materials processing in a comprehensive varicty of methods. The Center has also an ample diversity of apparatures for metallurgical,

mechanical and physical characterization, in order to evaluate basic properties of materials. Consequently a broad research spectrum covered and continues to involve the development of reactor materials processing and characterization, particularly in the area of nuclear fuels.

3.1 - NUCLEAR MIXED OXIDES

Mixed oxides may be used in nuclear fuels. One of these mixtures refers to the composition UO_2 - ThO_2 [17]. One of the reasons for a special interest on this composition is the fact Brasil has large thorium containing ore reserves.

The uranium and thorium oxide powders are mixed mechanically in various proportions. The mixture is pressed and sintered in different conditions. A series of characterization tests of the sintered mixed oxide is carried out afterwards. The studies in this area permited by means of X-ray techniques [18], the evaluation of the solubility degree between the two oxides, developed during fabrication. Using an electron microprobe technique it was also possible to determine the homogeneity level reached on sintering [19]. The electron microprobe tests show an high concentration of heterogeneities for certain fabrication procedures, with regions containing high uranium segregation content. At present a method based on electrical measurements is being developed to determine the degree of solubility between the two oxides, attained after sintering.

3.2 - MICROSPHERES COATED FUEL FOR HIGH TEMPERATURE GAS COOLED REACTORS (HTGR's)

Gas cooled power reactors and particularly the modern generation of HTGR's may utilize with great thermal efficiency coated actinide--oxide-microspheres as fissile material bearing component of their fuel. These microspheres are prepared by sol-gel processes or by hidration methods, these last developed particularly in Germany.

The HTGR's may use pyrocarbon-silicon carbide multi-layered coated microspheres dispersed in a graphite matrix. The graphite matrix

may take various configurations, as for instande rods, spheres or hexagonal-base prisms.

At the IPEN Nuclear Metallurgy Center UO_2 -U_3O_8 microspheres have been produced using gel microspheres fabricated at the IPEN Chemistry Engineering Center or abroad. The UO_2 gel microspheres are sintered in air in the 900 - 1400°C range to produce high-density U_3O_8 particles, or they may be sintered in reducing atmospheres, to produce up to 97% of the theoretical density spheres with diameters in the interval from 70 to 500 mm. Gel microspheres containing co--precipitation U and Th salts may be transformed in solid-solution UO_2 - ThO_2 microspheres by air-sintering at 1300-1600°C.

U_3O_8 microspheres so obtained have been dispersed in aluminum, 304 stainless steel and Zircalloy-II matrixes, producing fuel plates by hot-roll milling and fuel rods by swaging, with considerable potentia for utilization in power reactors.

A comprehensive program for out-of pile dimensional and microstructural characterization of microspheres has been carried out at the IPEN Nuclear Metallurgy Center with CNEN's support, starting in 1979. The Brazilian produced microspheres were object of an extensive analysis of their sintering kinetics under controlled oxygen potentials developed mainly abroad in the University of Illinois Materials Research Laboratory[20], that confirmed the excellent sintering behaviour observed at IPEN.

Preliminary studies of PWR pellets developed during 1979 in the Nuclear Metallurgy made by pressing gel microspheres in pellet configuration, demonstrated that the very high sinterability of the material presents an attractive potential for the development of a new low temperature PWR pellet sintering process. At present both the Brazilian Nuclebras and the German Nukem companies are pursueing this line of development.

The pyrocarbon coating of Brazilian made microspheres was starter in 1971 using a one-inch diameter fluidized-bed graphite tube furnace, enclosed in an argon- filled quartz tubular chamber, heated in a 12 cm long zone by an high-frequency induction furnace. The quartz tubing has

to be cooled on its outside surface by an high flow of nitrogen-gas, in order to make possible coating at up to 1600°C. A mixture of argon--methane was used, the metham decomposition at that temperature originating the pyrocarbon deposited over the surface of the microspheres. The single-layer coating so obtained was reasonably homogeneous, but the above described Brazilian made apparatus made it difficult to obtain reproducible results.

Using a specially german designed fluidized-bad coating furnace, it was possible to obtain in 1978-1979 much better quality pyrocarbon coatings. This furnace is also able to produce TRISO type of coated particles, with two pyrocarbon layer and a third constituted by silicon carbide, originated from operation with triclormethil-silane vapor. The furthering of a microspheres TRISO coating program depends on the definition of the role the HTGR's, in the context of the Brazilian Nuclear Energy Program.

In 1980 it was initiated a study of the irradiation behaviour of actinide-oxide microspheres produced at the IPEN engineering facilities. Initially only dimensional and microstructural changes will be analysed using the now available hot-cell facilities and electron microprobe of the Nuclear Metallurgy Center. High burn-up in pile studies are expected to be carried out in a near future abroad using BISO or TRISO coated microspheres dispersed in graphite rods.

Considering the needs of the PWR pellet development program and the interest to properly quality Brazilian made TRISO coated fuel, it is being planned the implementation of a post irradiation evaluation program at IPEN with necessary facilities in a already buit installation at the Physical Metallurgy, Metallography and Testing area of the IPEN Metallurgy-Center.

3.3 - CANNING MATERIALS

In the Nuclear Metallurgy Center development program, it is planned to fill zircalloy tubing purchased abroad with UO_2 pellets produced at IPEN. The zircaloy tubing must have well determined properties to be qualified as reactor-grade fuel canning. These

properties may be evaluated mainly through dimensional, mechanical and corrosion testing. The available facilities permit the evaluation of most of these properties. At present the test methods for Zircaloy tubing are being developed in the context of their adequation to the Brazilian power reactor program. After the tubing characterization it is intended to develop methods for pellet rod filling and rod closure by welding, in order to obtain properly qualifyied fuel rods.

An example of this canning materials development is the study of Zr-Nb alloys[21]. In the corresponding work, Zr-Nb alloys were prepared in laboratory scale, their Nb contents varying between 2,5 and 5,1 W/O. Different thermomechanical treatment were applied, followed by the corresponding microstructural characterization, mechanical properties determination and oxidation behaviour. The results showed that the properties of the alloys so produced satisfy the pertinent specifications for nuclear applications[22]. The isothermal oxidation of Zr-Nb and Zircaloy-2 with different carbon additions was studied in the temperature interval between 500-800°C[23]. The oxidation kinetics was determined by thermogravimetry and the kinetics curves obtained were related to the compositions of the alloys investigated.

4. Conclusions

1. The IPEN Nuclear Metallurgy Center developed methods for the fabrication of UO_2 sintered pellets. The tests performed evidenced the effect of process variables and materials on the properties of UO_2 sintered pellets.
2. The fabrication in pilot-plant conditions assures the production of UO_2 sintered pellets with specified properties.
3. The production facilities in the pilot-plant have ample flexibility, allowing for an easy adaptation for the fabrication of pellets using UO_2 powders of different origins.
4. The IPEN Nuclear Metallurgy Center has he capacity to develop methods for preparing and characterizing nuclear fuel materials, such as UO_2 - ThO_2 mixed oxides and High Temperature Gas Cooled Reactor's type of microspheres.
5. It is in program the development of characterization methods for Zircaloy tubing to be used in the preparation of fuel rods. In this context, studies were carried out for preparing and characterizing zirconium.alloys.

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TABLE I

UO_2 pellets characteristics, passintered density

ρ_{ps} = density after resintering

Pressing Pressure (t/cm²)	UO_2 (ASU)			UO_2 (AUC-2)			UO_2 (AUC-3)		
	ρs (g/cm³)	ρrs (g/cm³)	Δρ (%)	ρs (g/cm³)	ρrs (g/cm³)	Δρ (%)	ρs (g/cm³)	ρrs (g/cm³)	Δρ (%)
1,4	[illegible]	10,37	5,1	10,07	10,43	3,5	10,12	10,26	1,4
2,0	10,12	10,42	3,0	10,17	10,41	2,9	10,18	10,35	1,7
2,3	[illegible]	10,46	[illegible]	10,19	10,43	2,4	10,28	10,36	1,8

FIG. 1

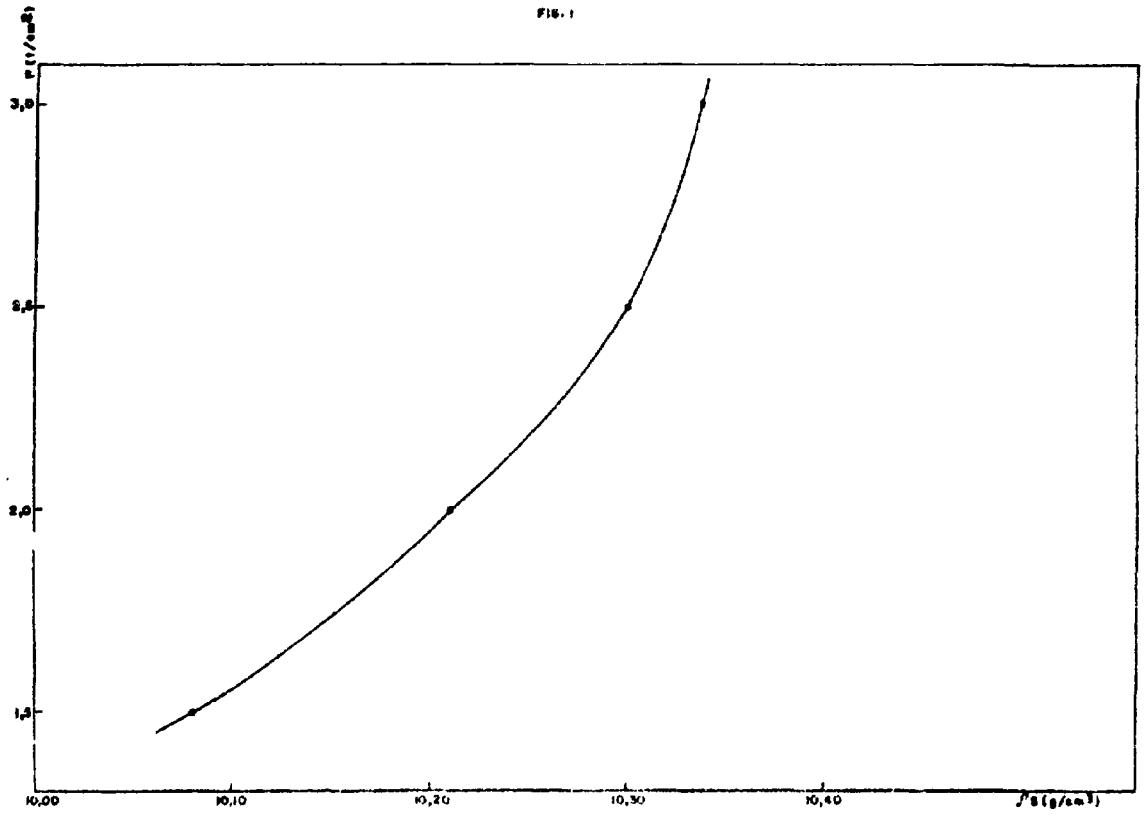
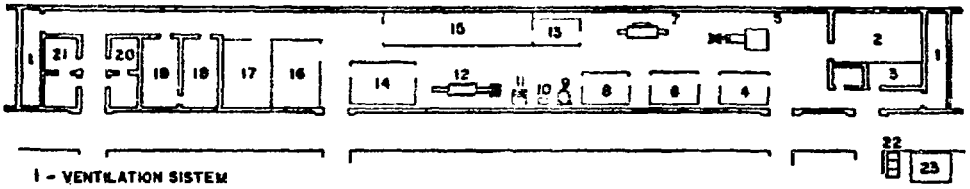


Fig. 1 - Sintered pelle+ UO_2 density as a function of the compacting pressure at 1650°C for 6 hours.



1 - VENTILATION SISTEM
2 - URANIUM SALT STORAGE
3 - ENRICHED UO_2 STORAGE
4 - CALCINING CHARGE PREPARATION
5 - CALCINING FURNACE
6 - SCREENING AND REDUCTION CHARGE PREPARATION
7 - REDUCTION FURNACE
8 - BLENDING OPERATIONS AND MUFFLES
9 - PRE-PRESSING UNIT
10 - GRANULATOR
11 - PELLET PRESSING UNIT
12 - SINTERING FURNACE
13 - DIMENSION CONTROL ROOM
14 - CENTERLESS GRINDING
15 - ROD LOADING UNITS
16 - END PLUGS WELDING
17 - PELLETS STORAGE
18 - PERFOMANCE TEST LABORATORY
19 - SEM AND MICRO PROBE
20 - MEN'S LOCKER ROOM
21 - WOMEN'S LOCKER ROOM
22 - AUXILIARY POWER SUPPLY
23 - BOTTLED GASES STORAGE

Fig. 2 - Pilot plant lay-out (area 1300 sqm)

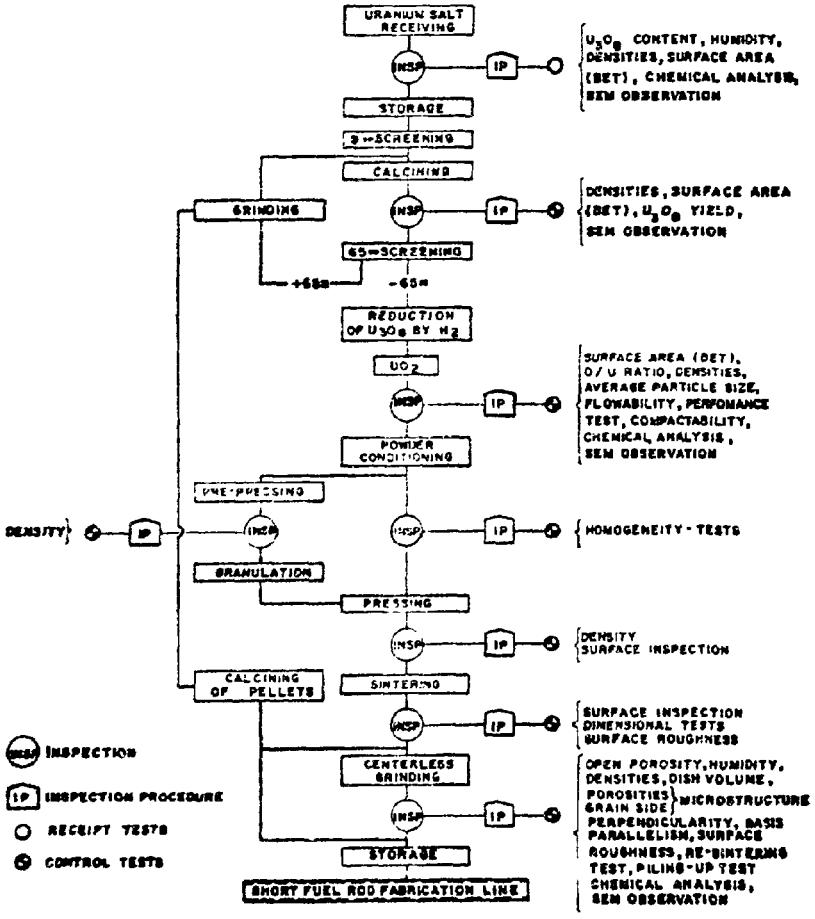


Fig. 3 - Simplified flowsheet of pilot plant operations, with indications of the performed tests.