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IRRADIATION DEVICES FOR FUSION REACTOR MATERIALS

RESULTS OBTAINED FROM IRRADIATED LITHIUM ALUMINATE AT

THE OSIRIS REACTOR

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**IRRADIATION DEVICES FOR FUSION REACTOR MATERIALS**

**RESULTS OBTAINED FROM IRRADIATED LITHIUM ALUMINATE  
AT THE OSIRIS REACTOR**

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Studies about controlled fusion reactor of the Tokamak type require the examination of the radiation effects on the behaviour of various potential materials.

Thus, in the first part of this paper, are presented the devices adapted to these materials studies and used in the OSIRIS reactor.

In a second part, is described an experiment of irradiation ceramics used as candidates for breeding material and are given the first results.

**I - THE OSIRIS REACTOR AND THE COLIBRI DEVICE**

**I,1 - The OSIRIS reactor :**

The OSIRIS pool reactor, of open core type, generates, for a nominal power of 70 MW, high neutron fluxes :

- $4.5 \cdot 10^{18}$  n/m<sup>2</sup> s in fast flux
- $3.5 \cdot 10^{18}$  n/m<sup>2</sup> s in thermal flux

with a maximal gamma heating of 8 W/g.

The fuel, manufactured in France, is a "Caramel" type fuel, low enriched (6 to 8%), made of  $UO_2$  plates, clad in zircaloy.

The present configuration of the reactor core allows five experimental in core positions, several positions in the beryllium reflector and many peripheral positions in the pool (figure 1). The neutronic characteristics of these various positions give a large range of fluxes.

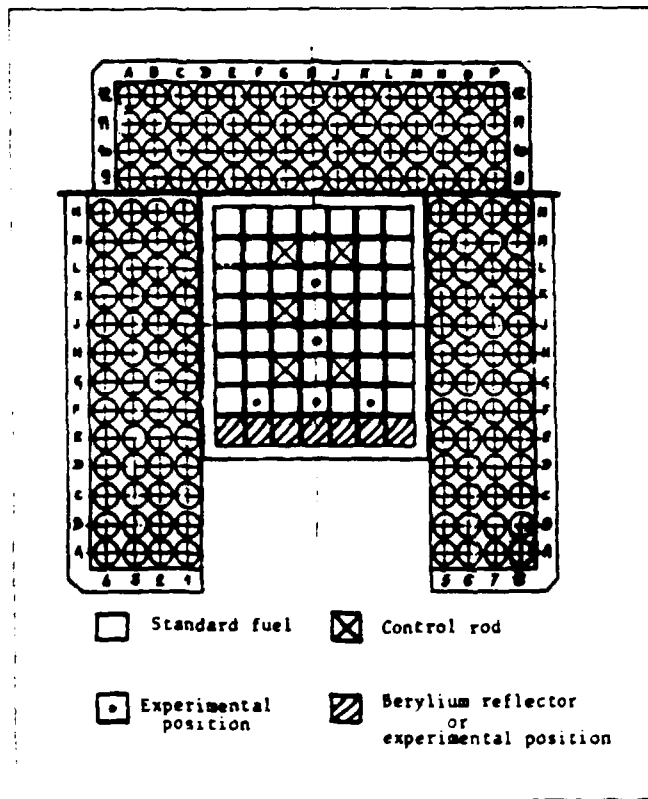


Figure 1 : Irradiation positions in the OSIRIS reactor

The irradiations, most frequently, concern the studies about the structure materials and the various nuclear fuel, the activation analysis, the production of radioisotopes, the silicium doping....

But taking into account the capabilities and characteristics of the OSIRIS reactor, it is easy to comply with irradiation requests for the research program on controlled fusion reactors.

In this program, the irradiations essentially concern the structure materials, protection ceramics and breeding materials and require a high flux, obtained in the core of the OSIRIS reactor. In order to answer to these problems, since a long time, we have been disposing of the COLIBRI device which is the basis of particular devices developed for the fusion program.

1,2 - The COLIBRI device (figure 2) :

More than hundred COLIBRI rig have been irradiated, during times exceeding 12.000 hours in some case. In its in flux part, the COLIBRI device consists of two stainless steel containers placed one inside the other. The gap between these two volumes corresponds to a gas jet whose thermal conductivity can be varied during irradiation by adjusting the proportions of an helium and nitrogen mixture / 1 /.

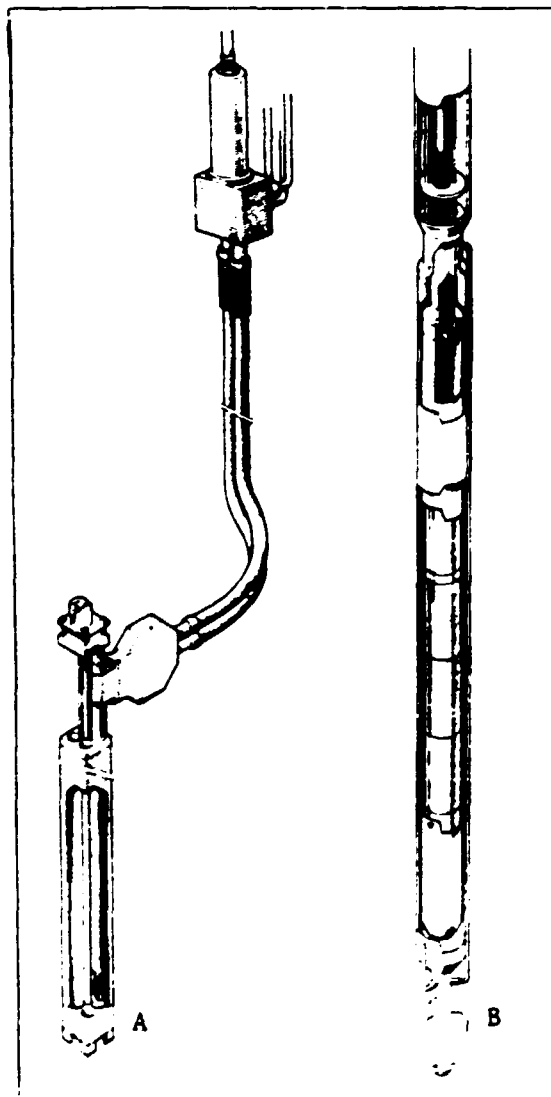


Figure 2 : COLIBRI device  
A - In pile installation  
B - Useful in flux part

Irradiations can be done from 250 to 1200°C, and a single device is able to work at two temperature levels. More over, when it is necessary for the good operating or keeping of temperature, electrical heaters can be set on the sample-holder in the inside of the device, or drowned in the stainless steel of the COLIBRI inner pipe.

The in core positions allow to put one to four devices, with a useful diameter of 67 to 25 mm.

## II - DEVICES DEVELOPPED FOR THE FUSION PROGRAM

### II,1 - Study of the fatigue crack growth :

A COLIBRI type with electrical heaters, allows to study the fatigue crack growth on a CT18 specimen, whose thickness is only 5 mm in order to reduce the transverse thermal gradient.

The sample-holder, reloadable in hot cells, holds two specimens ; one is put in alternative tensile stress and the other, free, used as a check sample. These two samples are set in the same flux and temperature conditions (250 to 450°C). The temperature keeping to  $\pm 5^\circ\text{C}$  is assumed by electrical heaters and the homogeneity obtained by an immersion in a sodium-potassium alloy (NaK).

As shown on the figure 3 :

- on the applied specimen, the alternative tensile stress is obtained by a  $10^4$  N jack with a frequency of one cycle per minute,
- the continuous measurement of the notch opening (compliance method) is carried out at the lip level by a probe system connected on a LVDT (Linear Variable Differential Transformer) sensor.

This measurement method is doubled by a pneumatic system based on the loss of leaktightness of pressurized holes bored in a perpendicular plan of the crack. This system gives a direct measurement of the length of the crack ; though it is discontinuous.

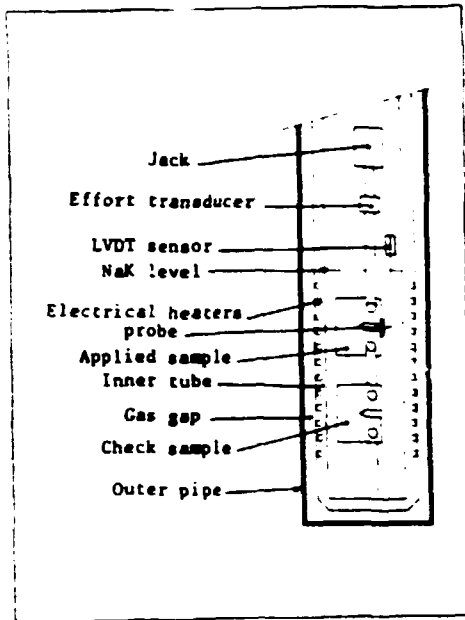


Figure 3 : Study of the crack growth  
Diagram of the in flux  
part of device

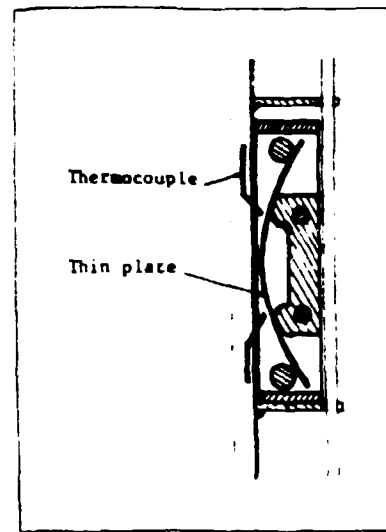


Figure 4 : Study of the stress  
relaxation  
Sample in the bending  
system

## II,2 - Study of relaxation :

A COLIBRI device, equipped with electricals heaters, and a sample-holder permit the study of stress relaxation of materials. The samples are thin plates ; the stress is obtained by bending the plate and maintaining it in position during the irradiation with the system shown on the figure 4 (three points flexion). A measurement of the residual deformation in hot laboratory gives the relaxation under irradiation.

The sample-holder, with a useful diameter of 60 mm, can hold twenty samples on five different levels. Each level has at least two flux integrators and four thermocouples.

To keep the temperature of samples at  $350^{\circ}\text{C} \pm 5^{\circ}\text{C}$ , the whole sample-holder is immersed in NaK and the temperature regulation is ensured by the COLIBRI electricals heaters whose position and power are adapted to this type of loading. Besides, this device can be reloaded in hot cells with new samples.

II,3 - Study of creep under traction :

Issued from the COLIBRI device with electricals heaters, this rig permits the study under irradiation of axial creep of materials subjected to a constant traction effort. It is a hot cells unloading and reloading device which allows to do a samples metrology in hot laboratory between the irradiation cycles.

The sample-holder receives two trains of three tubular test-pieces with a maximum diameter of 12 mm subjected to two different efforts up to 100 daN.

As shown on the figure 5, the test-pieces are put under stress by pressurised bellows, an automatic system regulates the pressure to ensure a constant stress. The homogenisation of the test-pieces temperature is ensured by the NaK in which they are immersed ; the temperature is roughly reached with the gas gap and adjusted at  $\pm 5^{\circ}\text{C}$  with the electrical heaters.

A new sample-holders allowing a more important effort on one train of three test-pieces is now studied.

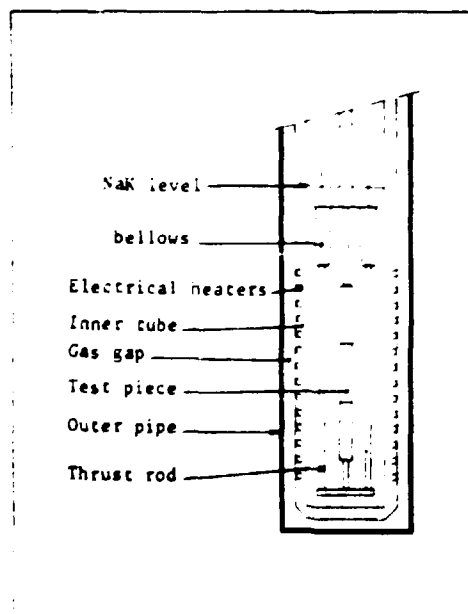


Figure 5 : Study of creep under traction  
Diagram of the in flux part of device

II,4 - Irradiation of insulating ceramics :

A COLIBRI device of useful diameter of 30 mm allows the neutron irradiation of insulating ceramics in the aim to check the evolution of their mechanical and dielectrical properties. This rig, partially shown figure 6, permits to irradiated a high number of samples (200) of various geometry (parallelepiped, cylinder, disk) and from various nature in a high purity helium environment. The temperature, only regulated by gas gap, is  $500^{\circ}\text{C} \pm 10^{\circ}\text{C}$ . The sample-holder is made of graphite barrels with a double wall. So, the samples are protected from any contact with the metallic parts of the device. Besides, thermocouples and flux integrators are placed in graphite crossbars. The samples, made of high purity and low activable products, can be easily carried and handled in a glove-box after irradiation. This device can be used again after having received a new sample-holder.

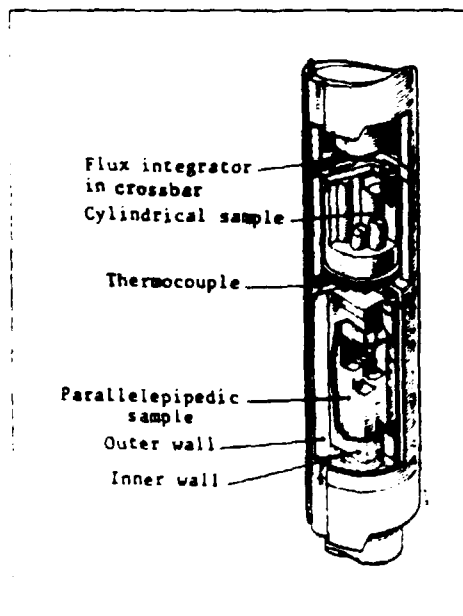


Figure 6 : Insulating ceramics irradiation detail of the sample-holder



II,5 - Irradiation of lithium containing ceramics :

Many studies are also made on breeding materials, especially lithium containing ceramics. The study of their behaviour after irradiation can be done in a COLIBRI rig of a diameter smaller or equal to 25 mm, specially studied for tritium confinement. Particularly, the whole in pile part has a double controlled containment, and the circuit aimed to be tritiated is completely welded and has no organic joint. To avoid any tritium accumulation in the device, which would favour its diffusion outside, all the circuits are swept at regular intervals. The monitoring of the experiment is made from a special glove-box, with a powerful ventilation.

The samples, clad or unclad, are cylinders or disks of various dimensions, placed in barrels (figure 7). The temperature from 400°C up to 800°C  $\pm$  10°C are regulated by gas gap. The samples environment is high purity helium. This device can admit a tritium production of 500 Ci.

It is in particular with this rig that the ALICE 01 experiment on lithium aluminate has been realized.

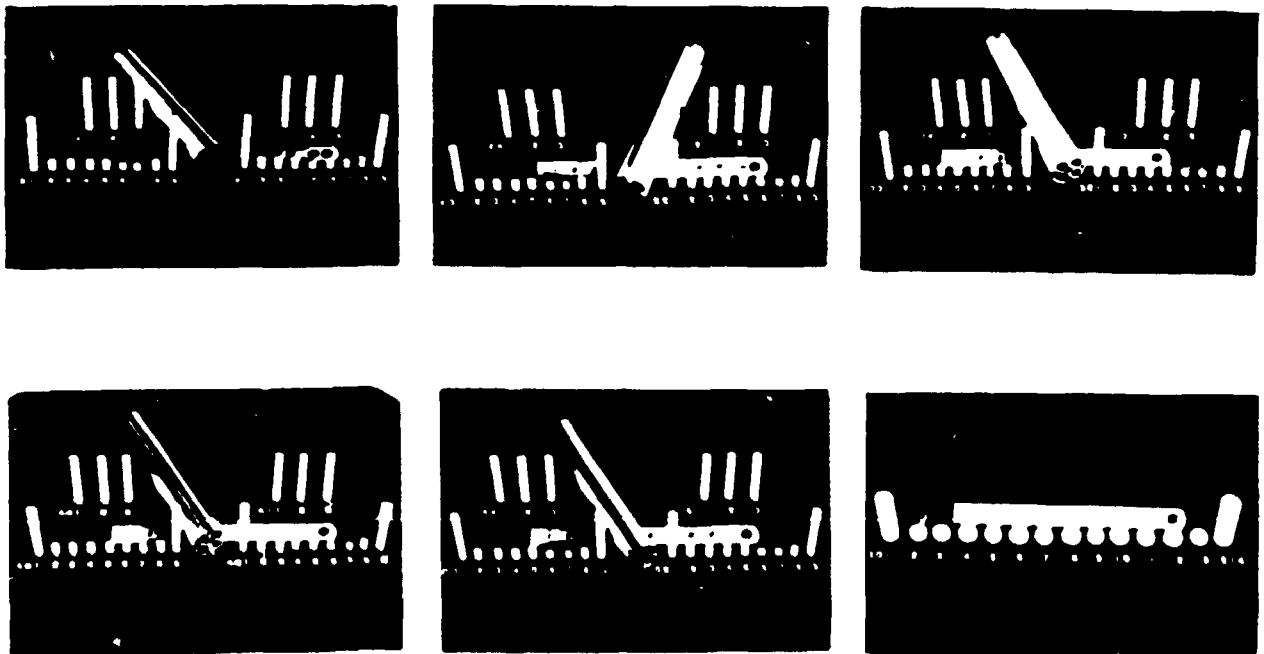


Figure 7 : Specimens before irradiation with their corresponding barrels

### III - ALICE 01 EXPERIMENT

#### III,1 - Specimens characteristics :

The specimens have been machined out of compacts of porous  $\gamma$   $\text{LiAlO}_2$  corresponding to 6 different groups of very homogeneous textures, listed in Table I.

The specimen dimensions have been chosen to meet the requirements of the post irradiation tests, given in the figure 8 and the Table II.

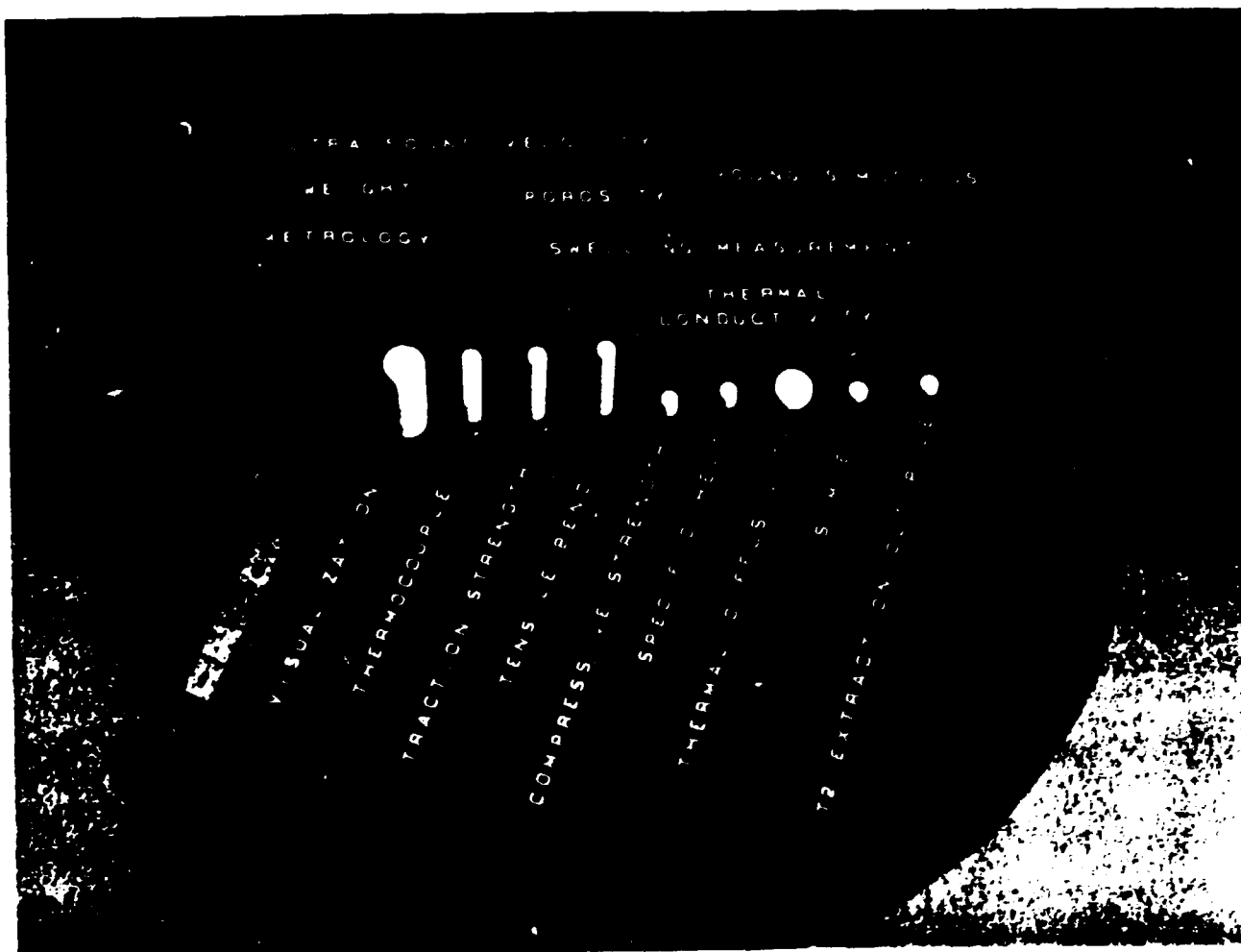


Figure 8 : Porous  $\gamma$   $\text{LiAlO}_2$  samples of different geometries for post irradiation examinations

Table I  
Textural characteristics of the six specimen groups

Textural group	Pore radius $\mu\text{m}$	Porosity	Grain diameter $\mu\text{m}$	Specific surface area $\text{m}^2\cdot\text{g}^{-1}$
1	0.048	0.29	0.35	5.7
2	0.043	0.16	0.68	2.7
3	0.055	0.26	0.47	5.3
4	0.40	0.285	3	0.7
5	0.45	0.24	0.4	5.8
6	1.55	0.23	13	0.2

Table II

Post irradiation examinations (P.I.E.) planned for ALICE 01

The weight  
The metrology  
The ultra sound velocity

} the porosity

} Young's modulus

Mechanical tests

Thermal tests

Scanning Electronic Microscopy (S.E.M.)

X rays

Residual  $T_2$  and  $^4\text{He}$  extraction

Burn up measurement by  $^6\text{Li}/^7\text{Li}$  mass spectrometry determination.

Mechanical characteristics of the different groups are shown on figures 9 and 10. Young's modulus is given as a function of porosity and ultimate compressive strength is represented in the porosity-grain diameter diagram / 2 /.

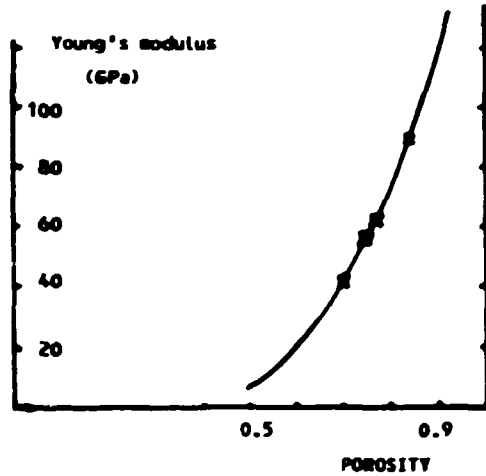


Figure 9 : Young's modulus values for the different textural groups before irradiation

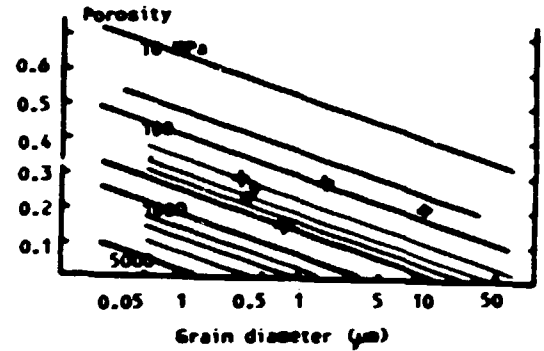


Figure 10 : Ultimate compressive strength values for the different textural groups before irradiation

Non cladded specimens (132) were inserted in six levels of the rig.

Figure 7 shows the specimens and their respective barrels. Five barrels (figure 11) have four housings for specimen insertion. Dimensions are 5.25 mm in diameter , 91 mm in height ; one barrel, not shown on the photograph, has a simple housing 10.25 mm in diameter, 91 mm in height. Barrels 5.25 mm in diameter have 4 stainless steel centering wires 0.05 mm in diameter for centering the 4.99 mm diameter specimens. Because of the sensitivity to air moisture of lithium aluminate / 3 / , all specimens have been annealed 1 hour at 500°C before to be dry weighed ; the sound velocity and the dimensions of the specimens have been measured and the samples were conditioned in tight flasks before insertion in the barrels. The installation of the barrels in the rig took 32 hours. The whole rig was then degassed 42 hours at room temperature and 40 hours at 200°C.

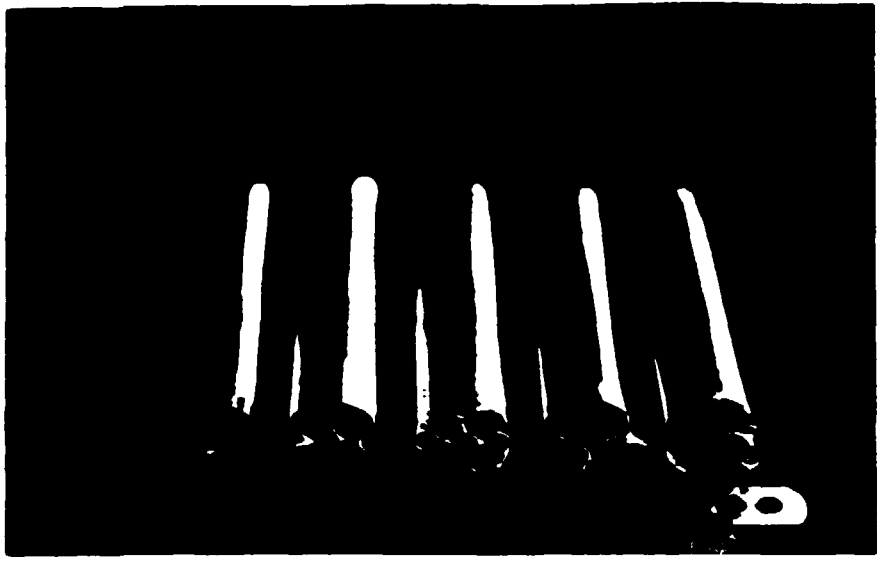


Figure 11 : Five of the six barrels used in ALICE 01 experiment

III,2 - Characteristics of the irradiation :

Figure 12 shows the disturbed neutron energy spectrum corresponding to the ALICE 01 experiment.

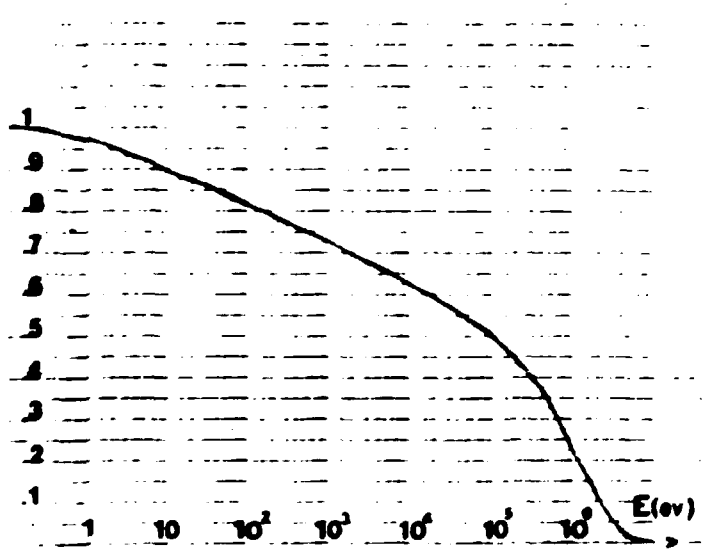


Figure 12 : Disturbed neutron energy spectrum for ALICE 01 experiment

Table III gives the fluence values along the rig. At the maximum flux level, the thermal power released by the neutronic reaction on  ${}^6\text{Li}$  was about  $80 \text{ W/cm}^3$ , the  $\gamma$  power was  $8 \text{ W/g}$  of  $\text{Li AlO}_2$ , therefore was the total power dissipated in the specimens of about  $96 \text{ W/cm}^3$ . Mean temperature chosen for the samples was about  $400^\circ\text{C}$  in the upper part and  $600^\circ\text{C}$  in the lower part of the rig. Their exact values are given on the Table III. Except during two reactor shut-downs, the temperatures measured at each level, remained very stable, as well core than surface temperatures. So that it may be concluded that thermal conductivity did not change much. This has to be confirmed by thermal post irradiation tests.

Two reactor shut-downs occurred, 15 minutes and 33 hours long ; and temperature drops as high as  $400^\circ\text{C}$  per minute affected the  $600^\circ\text{C}$  samples.

Irradiation started october 16, 1984, and ended november 13, 1984, corresponding to 25.7 Full Power Days.

Table III gives also the amount of tritium generated calculated for each barrel, corresponding to about 340 Ci generated from the global 82 g of natural lithium  $\gamma$   $\text{Li AlO}_2$ . The burn up, was of about 16% of the  ${}^6\text{Li}$ .

During the irradiation, the gas atmosphere around the samples was changed two or three times a day, by helium sweeping. The experimental conditions of tritium extraction were optimized.

More than half of the generated tritium would have been removed, as checked in the stack, and confirmed by first post irradiation measurements of residual tritium.

### III,3 - Specimens after irradiation :

The COLIBRI device was dismantled in the "Laboratoire d'Etudes des Combustibles Irradiés" (LECI) at Saclay, and the first post irradiation tests realized also in the LECI (weighing at 0.13 relative hygrometry, metrology, sound velocity, first residual  $\text{T}_2$  and  ${}^4\text{He}$  extractions).

During the dismantling, the lower levels were submitted to mechanical shocks, specially the levels 5 and 6. Conjugated thermal and mechanical shocks seem to be responsables for the breakage of several samples of the levels 5 and 6. The figure 13/A shows a bent and broken sample of level 5 and group 1.

Table III

Barrel number	Core temperature °C	Surface temperature °C	Tritium generated Ci	Samples weight g	Fast fluence $10^{24}$ n/m <sup>2</sup>	Disturbed Thermal fluence $10^{23}$ n/m <sup>2</sup>
1	410	340	32	14	1.78 2.19 2.55	4.58 6.06 7.44
2	390	320	57	13	2.74 3.22 3.63	10.17 12.00 13.54
3	370	250	71	14	3.70 4.10 4.44	12.56 13.76 14.80
4	535	415	71	14	4.59 4.70 4.66	12.81 13.24 13.37
5	610	520	67	13	4.64 4.30 3.84	14.70 13.90 12.09
6	580	-	41	13.5	3.67 3.10 2.47	10.51 8.74 6.75

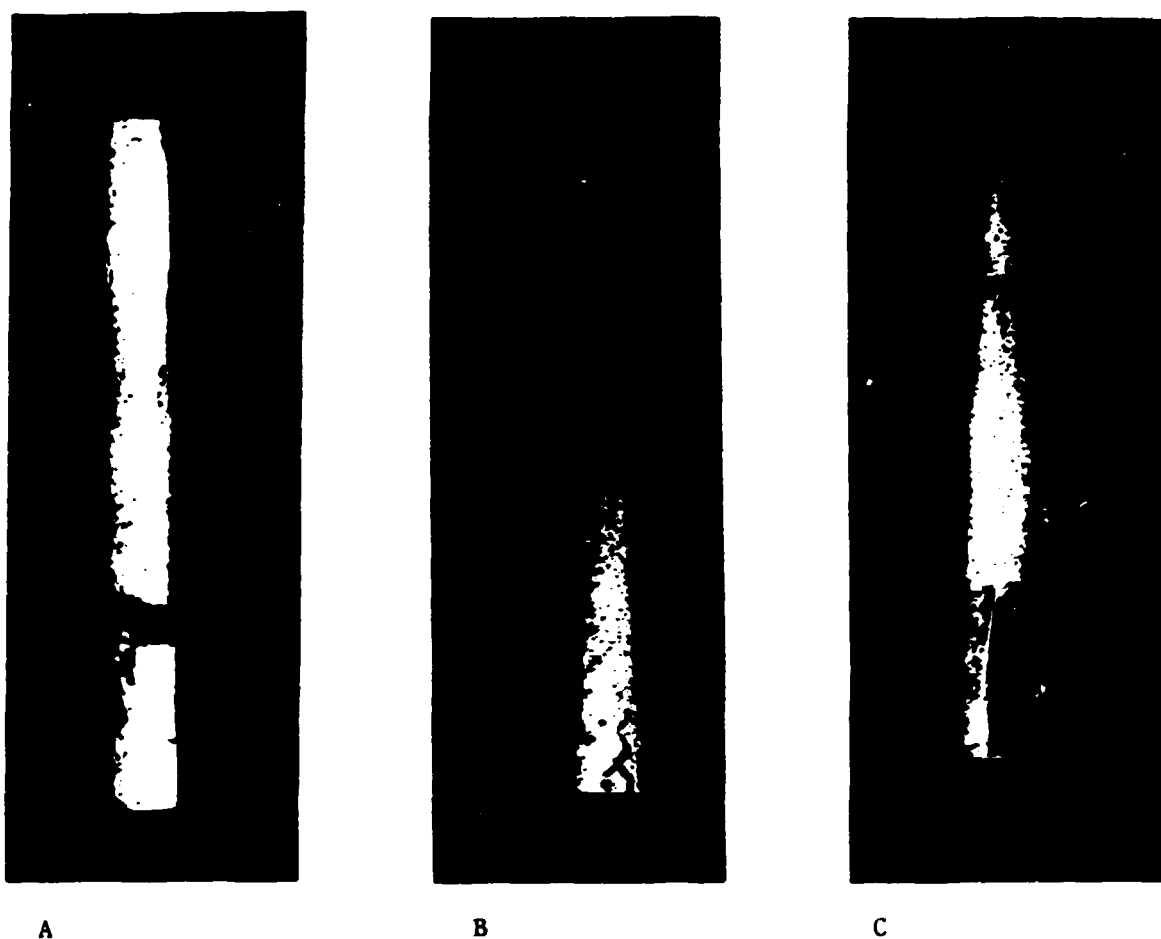


Figure 14 : A - B - C

Samples after irradiation

- A level 5, group 1
- B level 2, group 4
- C level 4, group 3

But, the other samples kept generally their integrity ; figures 13 B and C show two examples of samples of the level 2 and 4, respectively of textural groups 4 and 3.

In particular, samples of high density of the level 4, wich however withstood the maximum flux at the higher temperature, kept their integrity and show no length variations, even if they presented a little decrease of their Young's modulus or a loss of weight.



Transit times of 6  $\mu$ s through a 30 mm long sample, increased of 0.1 to 0.2  $\mu$ s at 400°C and of 0.15 (group 2) and  $\approx$  0.3  $\mu$ s (groups 3 and 5).

Weight losses, negligible for all specimen groups at 400°C, would correspond to more than the loss of  $T_2O + 2 He$  at level 4, at 600°C.

However, first post irradiation extraction runs showed some residual tritium and helium 4, and besides less helium than tritium even on a 400°C irradiated sample.

Any way, samples of group 5  $\gamma LiAlO_2$ , exhibited both the best kinetic of tritium release in "in pile" runs / 4 /, and a good irradiation behaviour, so that this type of aluminate could represent a valuable candidate for a fusion reactor blanket material.

#### IV - CONCLUSIONS

Within the framework of the European Communities Fusion Technology Program, other more classical irradiation experiments are also carried out in the OSIRIS reactor. At the present time, the utilization of the described devices is underway in the OSIRIS reactor as well as devices adaptations allowing still better to comply with the request of the fusion program.

Concerning the ALICE 01 experiment, other post irradiation examinations must be completed. However, this irradiation experiment allowed yet to prove the good behaviour of high density  $\gamma Li AlO_2$ .

An experiment ALICE 02 is prepared, with the best textural  $\gamma LiAlO_2$  specimens, including both natural and enriched lithium samples, and operated at about 600 and 700°C.

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