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European Atomic Energy Community — EURATOM
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Società ANSALDO S.p.A. — Genova

NUCLEAR FUEL WITH BURNABLE POISON

Part 2. Preparation and out-of-pile evaluation of $Gd_2 O_3$ poisoned UO_2 pellets

by

G. ABATE-DAGA, I. AMATO and G. GRAPPIOLO
(FIAT)

Topical Report
January 1, 1969 — May 31, 1970

1970

Contract No. 008-61-12 PNII

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ABSTRACT

Gd_2O_3 microspheres about 500 microns diameter, have been molybdenum coated through metal vapor deposition in fluidized bed. The coating thickness has been maintained in the range 25-30 microns.

The optimum density of galodinia microspheres in order to maintain coating integrity at high temperature is in the range 85-90 % of theoretical value (T.D.). UO_2 pellets poisoned with 1200 ppm of Gd_2O_3 were obtained at about 95 % T.D.

Tests performed at high temperature both under gradient and isothermal conditions showed an acceptable behaviour of molybdenum coated gadolinia microspheres in UO_2 pellets.

KEYWORDS

MOLYBDENUM
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VAPORS
COATING
FLUIDIZATION
SUSPENSIONS
GADOLINIUM
URANIUM OXIDES
PELLETS
POISONING

INDEX

1. INTRODUCTION *

In a previous paper (1) the advantages of the use of nuclear fuel poisoned with gadolinia microspheres have been shown; in the same paper the preparation and characterization of Gd_2O_3-microspheres had been reported. Scope of this paper is a description of the experiments carried out in order to obtain Gd_2O_3 poisoned UO_2 pellets suitable as nuclear fuel.

The technology of fabrication of UO_2 pellets poisoned with Gd_2O_3 microspheres involves the problem of compatibility between the two materials.

Allis Chalmers (2) studied this problem from a phenomenological point of view and concluded that Gd_2O_3 and UO_2 start to react at 1500°C and the reaction is complete at 1705°C for soaking time of two hours. Beals and Handwerk (3,4) studied by X-rays and chemical analyses the system UO_2 - Gd_2O_3 by cosintering of a mixture of components at the temperature of 1700°C and concluded that a face-centered-cubic solid solution exists over the entire compositional range between UO_2 and Gd_2O_3.

On these bases it is obvious that at typical reactor operating conditions it is not possible to maintain the discrete distribution of poison and that it is necessary to find a diffusional barrier between Gd_2O_3 and UO_2. This barrier ought to have the following properties:

1) to be compatible chemically with sintering atmosphere of pellets;

2) to be compatible chemically with UO_2 and Gd_2O_3 both in isothermal and gradient temperature conditions;

3) to have thermal expansion coefficient very close to that of Gd_2O_3 and UO_2;

4) to be low-neutron-absorber;

5) to be stable under irradiation.

These requirements are satisfied only by some of the so called refractory metals such as tungsten, molybdenum, niobium and tantalum.

These latter are to be excluded because the atmosphere of sintering is hydrogen and niobium and tantalum have a strong trend to hydriding (5,6).

Both tungsten and molybdenum satisfy to nuclear requirements also if molybdenum is the lowest neutron absorber. The molybdenum seems to be the most suitable material. Nevertheless his thermal expansion coefficient is somewhat different from that Gd_2O_3 and UO_2.

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* Manuscript received on September 22, 1970

The compatibility between stoichiometric UO_2 and molybdenum has been studied up to 2000°C by Sanderson et others (7) and their results show that no reaction occurs up to 2000°C.

On the contrary no published data are available for the system Gd_2O_3-Mo and at consequence it was necessary to perform some experiences on the compatibility between the two materials.

On these bases, the problem of preparation of UO_2 poisoned with Gd_2O_3 has been affronted in the following way:

a) evaluation of high temperature compatibility between Gd_2O_3 and Mo;

b) optimization of Mo-coating procedure;

c) study of the thermomechanical behaviour of poisoned pellets and of the criteria to avoid cracks in the Mo-coating

d) sintering and characterization of poisoned pellets;

e) high temperature evaluation of poisoned pellets both in isothermal and in gradient temperature conditions.

2. EVALUATION OF HIGH TEMPERATURE Gd_2O_3-Mo COMPATIBILITY

In order to evaluate the compatibility between molybdenum and gadolinia at high temperature, pellets of molybdenum powder with dense gadolinia microspheres have been attained through current powder metallurgy procedures. Molybdenum pellets were hydrogen sintered and a density of about 95% of theoretical value was obtained.

The dense molybdenum pellets gadolinia doped were undergone thermal treatment of2000°C up to 20 hour. No reaction at the interface between molybdenum and gadolinia microsphere is evident (fig.1). In consideration of this observation it has been assumed that the compatibility of the molybdenum with urania and gadolinia is good enough to recommend the molybdenum as reaction barrier between the urania and gadolinia.

3. OPTIMIZATION OF Mo-COATING PROCEDURE

Vapour deposition process in fluidized bed is the best method for coating urania microspheres.The apparatus used for coating the gadolinia microspheres with molybdenum layers is the same which is described elsewhere (8). The molybdenum coating methods availableare the thermal decomposition of the exacarbonyl and the hydrogen reduction of pentachloride.

Molybdenum exacarbonyl thermally decomposes at temperatures in the order of 200°C. As carrier gas it has been used argon. This type of coating gives layers little compact and adherent to the gadolinia.

The coating type based on reduction of molybdenum pentachloride involves the following reaction

$$2\ MoCl_5 + 5\ H_2 \longrightarrow 2\ Mo + 10\ HCl \qquad (1)$$

The process is carried out as follows: hydrogen (and an inert gas carrier) is passed over or through the solid or liquid metal halide housed in a boat and heated in order to obtain the 20 or 30% (by volume) of metal halide in the vapour phase. Then the gas mixture flows through the coating chamber, reacting at the heated surface and depositing an adherent layer of non-volatile reaction products. The spent gases pass on out of the chamber and are either suitably disposed off.

Hydrogen used in the process must have a dew-point of 55°C, so traps of moleculare sieve were used. The carrier gas flow was 2,5 l/min.

The optimum evaporation temperature resulted to be 190°C, because at lower temperatures the chloride condensed in the colder part of the apparatus and obstructed it. For this reason hydrogen and carrier gas (argon) were preheated before being flown through the evaporator.

The optimum reaction temperature resulted to be 900°C.

Fig. 2 shows some polished cross-section of Mo-coated Gd_2O_3 microspheres;

The molybdenum layer density is about 80% of the theoretical value and the total chlorine pick up about 800 ppm.

4. MECHANICAL BEHAVIOUR OF Mo-COATING

Because of the different thermal expansion coefficients of urania, molybdenum and gadolinia (fig.3) (8-19), departing from the deposition temperature, i.e. equilibrium temperature, (null stresses), there is the rising of some stresses; the related study is complicated by the creep phenomena in all the materials involved and by the fact that is difficult to forecast quantitatively the effect of the present porosities.

It has been calculated (20-27) the stress in the Mo-coating by taking up the elastic behaviour of Mo and Gd_2O_3 by considering a simple surface contact between the two materials.

The Laure's equations (28) were applied, as follows:

$$\sigma_r = - \frac{2 \alpha E}{1 - v} \frac{1}{r^3} \int_a^r T r^2 d r + \frac{EC_1}{1 - 2v} - \frac{2 EC_2}{1 + v} \cdot \frac{1}{r^3} \qquad (2)$$

$$\sigma_t = \frac{\alpha E}{1 - v} \frac{1}{r^3} \int_a^r T r^2 d r + \frac{EC_1}{1 - 2v} + \frac{EC_2}{1 + v} \frac{1}{r^3} - \frac{\alpha ET}{1 - v} \qquad (3)$$

where σ_r and σ_t are respectively radial and circumferential stress, a and r are respectively the outer and the inner radius, α the thermal expansion coefficient, E is Young's modulus, v is Poisson's ratio, T is the absolute temperature and C_1 and C_2 are integration constants.

The solution of these equations is performed by assuming the congruence and the equilibrium between Mo and Gd_2O_3.

Since the circumferential stress is predominant on the radial stress, the values of the circumferential stress at different temperatures are shown in fig.4 for microspheres diameter of 300, 500 and 700 μ and Mo-coating thickness of 5, 10 and 25 μ ; the equilibrium temperature has been undertaken once 300°C (Mo deposition from Mo $(CO)_6$)and once 900°C (Mo deposition from Mo Cl_5). It can be seen that at temperatures relatively low, the value of σ_t is higher than the yield point σ_y .

A comparison between the two Mo-deposition processes shows that lower stresses arise in Mo-coating obtained through the hydrogen reduction of chloride; for this reason the chloride reduction has been the process followed for Mo-coating of gadolinia microspheres.

As a consequence of the stress results the calculation of the bursting pressure of the Mo-coating has been performed. The bursting pressure for a thin spherical shell (29) is given by the relation:

$$p = \sigma_o \frac{t}{R} \left(\frac{n}{e}\right)^n \frac{2^{n+1}}{3^n} \qquad (4)$$

where t and R are respectively the thickness and the radius of the shell, e is the basis of natural logarithms, σ_o and n are constant deduced from the diagram $\sigma - \varepsilon$ in the simplified form $\sigma = \sigma_o \varepsilon^n$. The variation of the bursting pressure with the geometrical parameters of the Gd_2O_3 and the Mo-coating is shown in fig.5 for a temeperature of about 1300°C: the main conclusion is that an increase in the shell thickness has as a consequence an increase of the bursting pressure of the molybdenum shell: for this reason a molybdenum shell thickness in the range 25 - 30 microns has been adopted for coating the gadolinia microspheres.

These considerations do not take into account the behaviour of coated microspheres in UO_2. It is very hard to describe the mechanical phenomena which occur on the microspheres during the densification stage of UO_2 :the most realistic assumption is to consider that UO_2 sinters with mull gap on the gadolinia molybdenum coated inclusion. In this case it is necessary to perform the calculation of the pressure exercised by the molybdenum coating on the UO_2 matrix during the cooling from the temperature Ti (mull stress) to the room temperature; for this calculation the microspheres are considered as molybdenum inclusions. In the elastic field, the UO_2 matrix is subjected to a pressure (30,31) given by the following relation:

$$p\,(T) = \frac{12\, G_o\, K\, \Delta T\, \Delta \alpha}{3\,K + 4\,G_o} \qquad (5)$$

where

$$G_o = 0{,}29\, E_{UO_2}\ , \quad K = \frac{E_{Mo}}{1 - 2v_{Mo}}\ , \quad \Delta T = T_i - T \qquad (6)$$

E is the Young's modulus, v the Poisson's ratio and $\Delta \alpha$ the difference between thermal expansion coefficients.

Fig.6 shows the variation of p versus T for two different values of Ti.

The tensile strenght of UO_2 matrix deduced on the basis of bending test (32) is in the range of 10-15 kg $/mm^2$; at consequence during the cooling step there will be the formation of cracks in UO_2: experiments performed in order to examine this behaviour showed that the cracks originated during the cooling of UO_2 sintered pellets concern a radial zone about 200 microns deep; generally the cracks are stopped by porosities or grain boundaries (fig.7).

In all the above speculative remarks, the relationship and the phenomena in the plastic field are not considered.

Unfortunately the calculation of the deformation and the creep behaviour of molybdemum coated gadolinia inclusions is very difficult to perform also in consideration of the poor creep data of the material involved in the analysis. From a qualitative point of view, it is easy to forecast that the best performance of gadolinia microspheres occurs when the microspheres have porosities that may be absorbed by creep in order to compensate the thermal expansion difference between the molybdenum and the gadolinia.

The examination of the influence of the gadolinia porosities on the performance of the poisoned fuel has been carried out through microspheres with density of 81, 84, 89 and 94% of the theoretical value.

The performance tests have been done, through heat treatment in hydrogen atmosphere at 1700°C up to 50 hours, on the UO_2 pellets poisoned with 25 microns molybdenum coated - 500 microns diameter gadolinia microspheres at the above reported densities. The results obtained are summarized in fig.8: it can be seen that the microspheres with poor density undergo a grain growth process with the coalescence of fine porosities in large porosities; it can be seen that the final density of the microspheres is quite different from the initial value; it can be seen that after 10 hours the microspheres with the density of 94% of theoretical value (T.D.) show some cracks in the coating and some reaction between UO_2 and Gd_2O_3 occurs; it is interesting to observe that also after a treatment of 50 hours at 1700°C these microspheres do not show a very extensive reaction (fig.9).

The coatings of all the other microspheres are not injured.

The main conclusions are that microspheres with density in the range 85-90% of theoretical value are the best solution to be adopted in order to produce UO_2 pellets poisoned with molybdemum coating Gd_2O_3 microspheres.

5.SINTERING AND CHARACTERIZATION OF POISONED PELLETS

In order to evaluate the influence of the gadolinia microspheres inclusion on the sintering rate of UO_2 pellets, sintering experiments have been carried out on pressed pellets doped with 600 and 1200 ppm of gadolinia microspheres, 400-500 microns of diameter, 23-30 microns of coating thickness. The urania, ceramic grade powder, used for pellets preparation was supplied by Nukem Wolfgang (Germany).

The powder properties are summarized in Tab.1.

The mixture was pressed into pellets up to green density of 50-55% T.D.

The green pellets were heated in hydrogen to a maximum temperature of 1700° at a rate of 300° C/h. The sintered densities obtained are shown in fig. 10.

Fig. 11 shows an isothermal sintering curve for the same pellets. In both cases no appreciable behaviour differences between pure and poisoned UO_2 has been observed.

The poisoned UO_2 pellets were characterized by the following tests:

1) porosimetry;

2) open and closed porosity;

3) corrosion behaviour;

In fig. 12 it is represented the pore distribution for unpoisoned and poisoned UO_2 and in fig. 13 there are represented the amounts of open and closed porosity, always for unpoisoned and poisoned UO_2. In both cases no appreciable differences exist among the pellets.

The main conclusions are that the sintering mechanism of the urania is not influenced by the gadolinia microsphere additions at the foreseen concentration: so no difference in open-closed porosity distribution and in open porosity diameter has been observed for poisoned and unpoisoned pellets.

Moreover, corrosion tests performed in pressurized water at 160 atm and 340° C for 50 hrs did not show differences in behaviour of poisoned and unpoisoned pellets.

6. EVALUATION OF POISONED UO_2 PELLETS AT HIGH TEMPERATURE

The poisoned UO_2 pellets were evaluated from the compatibility point of view under high temperature gradient conditions and through high temperature long time treatments.

6.1 Thermal gradient experiments

The thermal gradient experiments were carried out with an apparatus described elsewhere (33).

The heating is performed through an electrically heated tungsten rod; the skin of pellets stack is cooled through a water jacket; the controlled atmosphere was obtained through an argon flow.

The following experiments were performed:

i. temperature of the core of pellets stack: 2300°C;
temperature of the skin of pellets stack: 650°C;
for 1 hour and 3 hours (fig. 14);

ii. temperature of the core of pellets stack: 2500°C;
temperature of the skin of pellets stack: 670°C;
for 20 min. and 1 hour (fig. 15);

iii. temperature of the core of pellets stack: 2700°C;
temperature of the skin of pellets stack: 700°C;
for 5 min. and 20 min. (fig. 16);

iv. temperature of the core of pellets stack: 2900°C;
temperature of the skin of pellets stack: 700°C;
for 3 min. (fig. 17);

The results obtained by the performed experiments showed that the behaviour of the gadolinia poisoned pellets is quite satisfactory under high thermal gradient; generally, the Mo-coating gadolinia microspheres were not damaged in all the tests.

6.2. Isothermal experiments

The isothermal experiments have been carried out on poisoned pellets heated through a tungsten induction heated container. The experiments were performed at 2200°C for 10 hours. As it can be seen in fig.18, no damage on the coating layers has been obtained, but generally the porosity is displaced in a particular area of the interface between the gadolinia microsphere and the molybdenum layer. Generally, this area was located where microcracks between the molybdemum coating and the urania are present: a possible explanation of this phenomenon can be given assuming that the microcracks act as discontinuites regions regarding the thermal behaviour and so the porosities migration occur preferentially in this area.

At this temperature for long time or at higher temperature an intergranular diffusion of the molybdenum into the gadolinia can be observed (fig.19) and in this case the process produces cracks in the coating with consequent UO_2 - Gd_2O_3 reaction (fig.20).

7. CONCLUSIONS

The following main conclusions can be drawn from the performed research work:

1) a molybdenum coating on the gadolinia microspheres avoids the UO_2 - Gd_2O_3 reaction;

2) in order to have a coating which maintains its integrity in spite of the different thermal expansion of the molybdenum , gadolinia and urania microspheres with a density in the range 85-90% of the theoretical density and molybdenum coating thickness in the range 25-30 microns must be used;

3) the sintering behaviour of the UO_2 powder is not affected by additions up to 1200 ppm of 500 microns diameter gadolinia microspheres coated with 30 microns of molybdenum;

4) the perfomance of the poisoned pellets under out of pile thermal gradient up to 2900°C is acceptable;

5) isothermal experiments performed at 2200°C for 10 hours showed that the porosity migration occurs preferentially in the area where cracks are present;

6. isothermal test showed that at high temperature (more than 2200°C) an intergramular diffusion of the molybdenum by the coating into the gadolinia occurs and this phenomenon carries to a distruction of the barrier.

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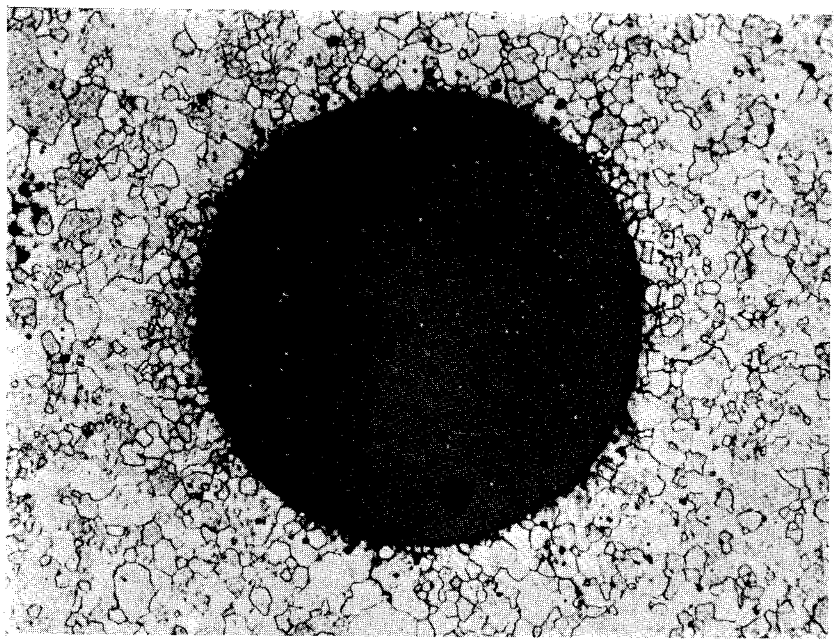
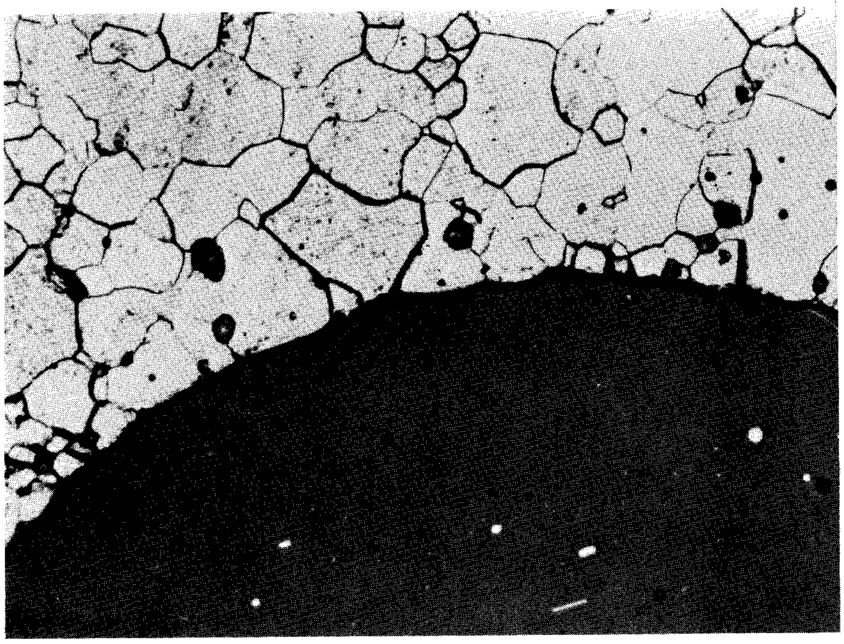
Tab. 1. UO_2 powder characterization

Real density (CCl_4),	g.cm.$^{-3}$	10.39
Tap density,	g.cm^{-3}	2.12
Average particle diameter (Fisher),	μm	2.80
Total surface area (BET),	m^{2}g-1	3.69
External surface area, (Blaine),	m^{2}g^{-1}	0.24
Roughness factor,		15.37
DTA 1st oxidation peak,	°C	205
Temperature difference between 1st and 2nd oxidation peaks,	°C	120
O/U ratio.		2.03



(100 x)



(350 x)

FIG 1 : Compatibility between Gd_2O_3 and Molybdenum (test performed in argon atmosphere a 2000°C for 20 hours, oxalic acid etched)

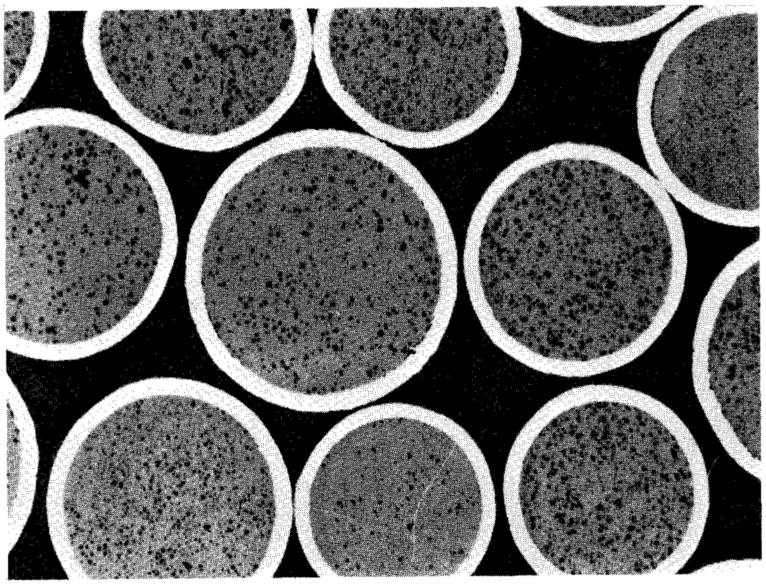


FIG 2 : Polished cross-section of Mo-coated Gd_2O_3 microspheres (100 x)

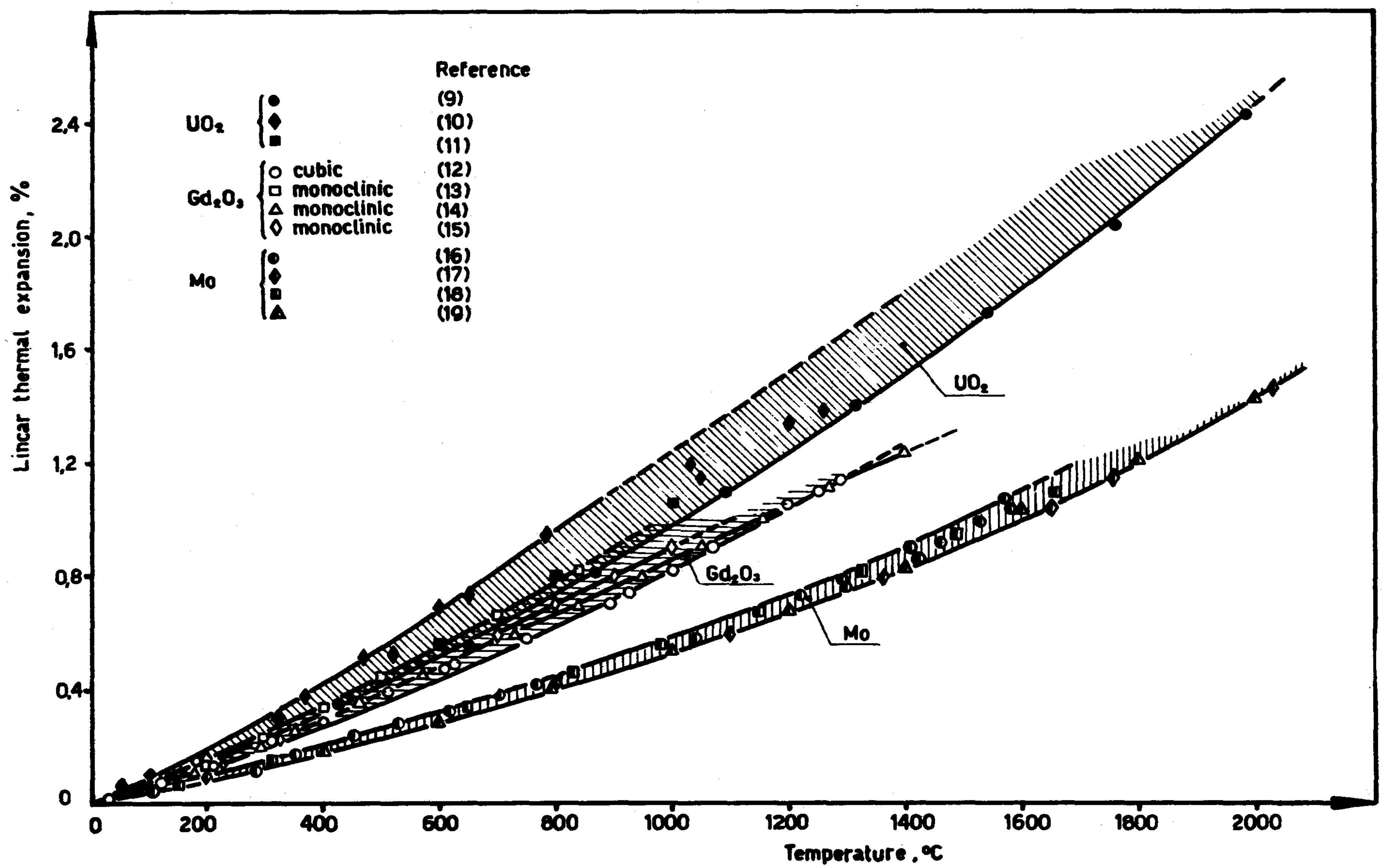


FIG. 3 - Linear thermal expansion of UO_2, Molybdenum and cubic and monoclinic Gd_2O_3

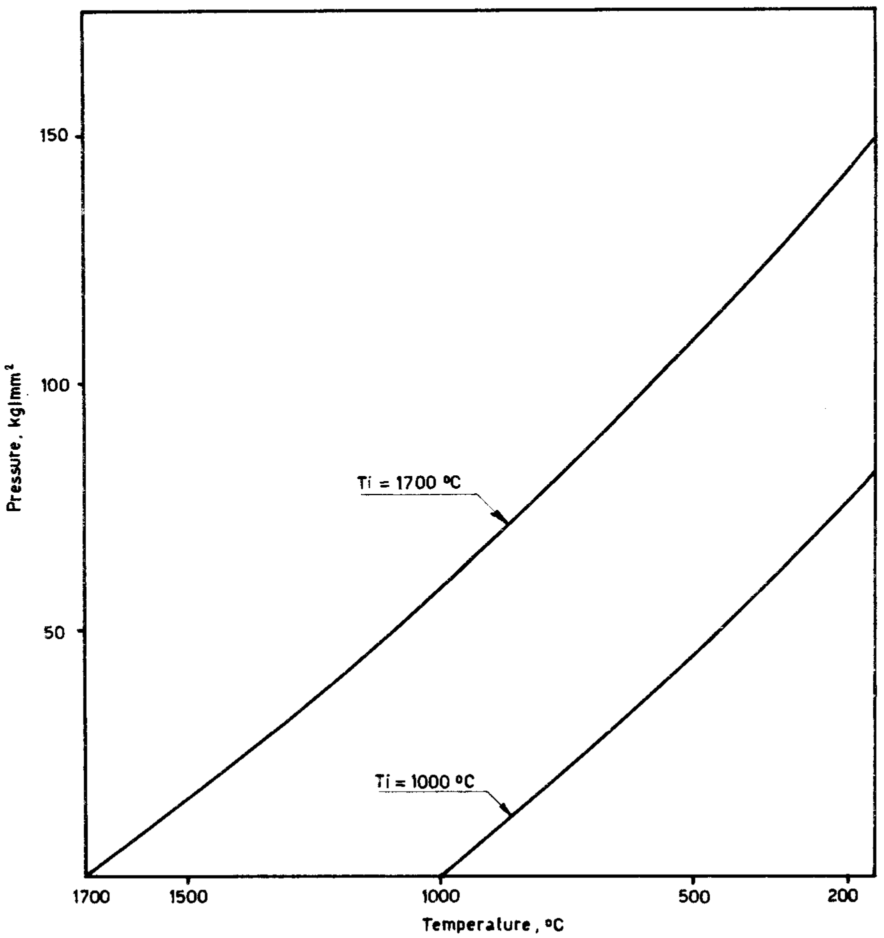


FIG. 6 - Pressure on the UO_2 matrix from the molybdenum coating during the pellets cooling

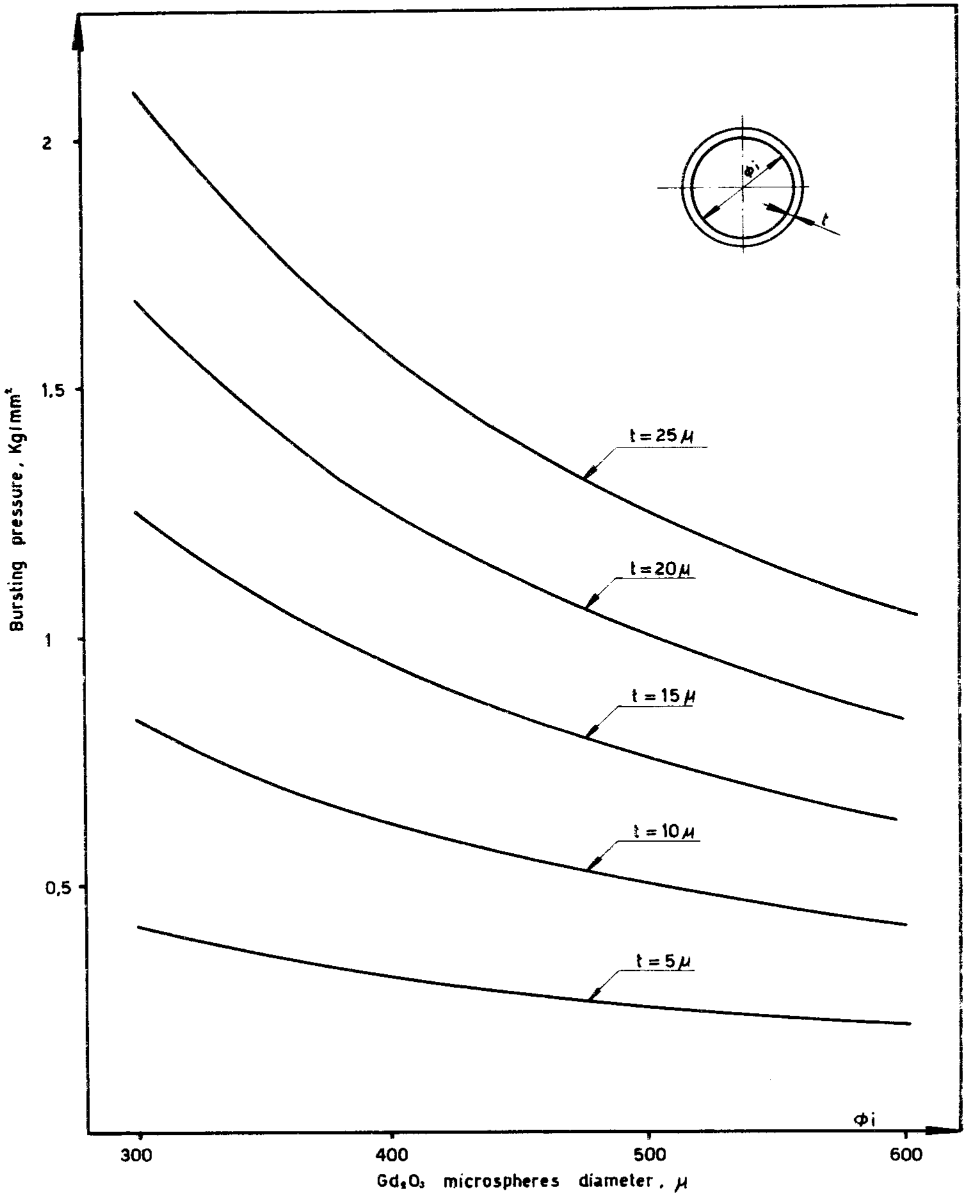


FIG. 5 - Variation of the bursting pressure as a function of Gd_2O_3 microspheres diameter and molybdenum coating thickness

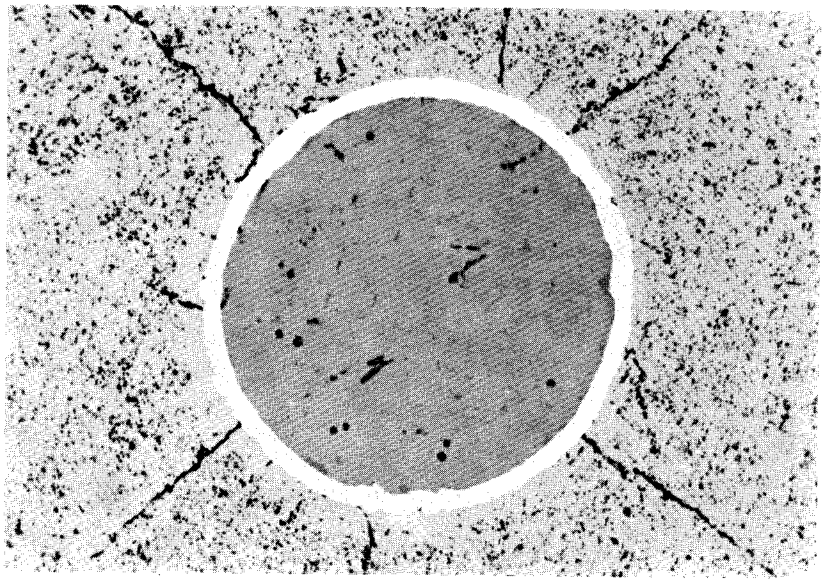


FIG. 7 : Micrographic aspect of UO_2 pellets Gd_2O_3 poisoned (unetched, 120 x)

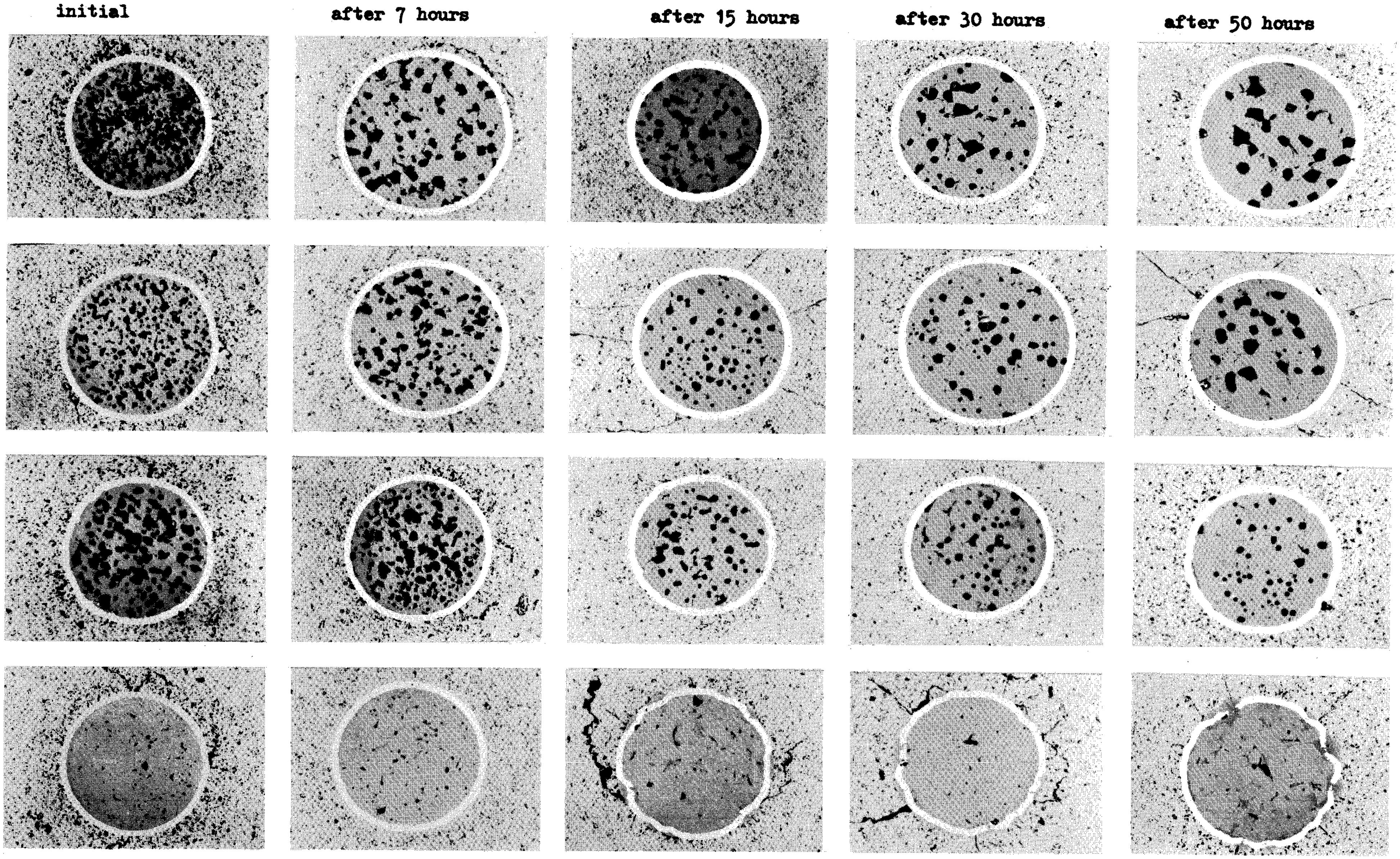


Fig. 8 – Micrographic aspect of molibdenum coated Gd_2O_3 microspheres; initial pellets density: 94–96% T.D.; Gd_2O_3 microspheres densities: (above to below) 81%, 84%, 89%, 94% T.D.; thermal treatment: 1700°C up to 50 hours (unetched, 120 x, reduced to 2/3).

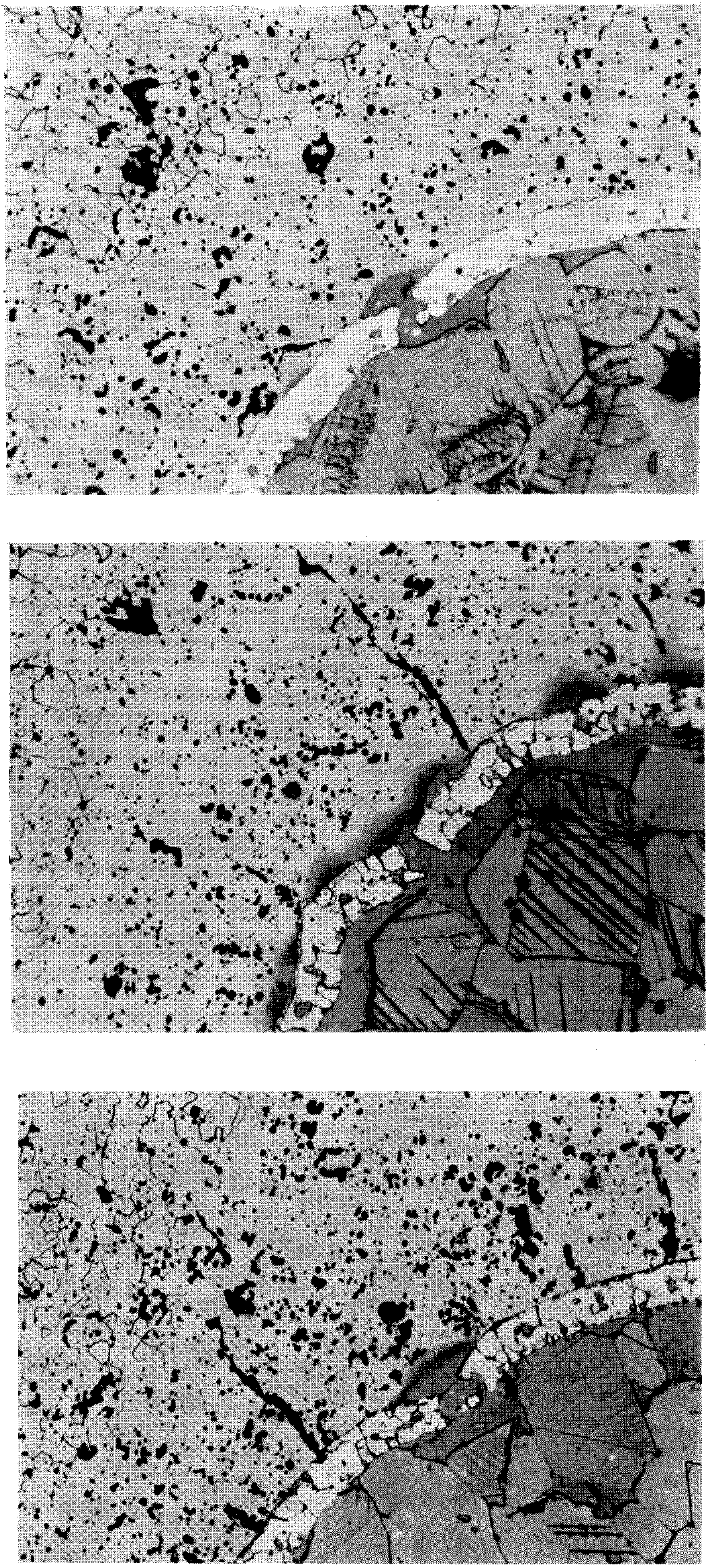


FIG. 9 : Interfacial reaction between UO_2 and Gd_2O_3 (etched, 300 x)

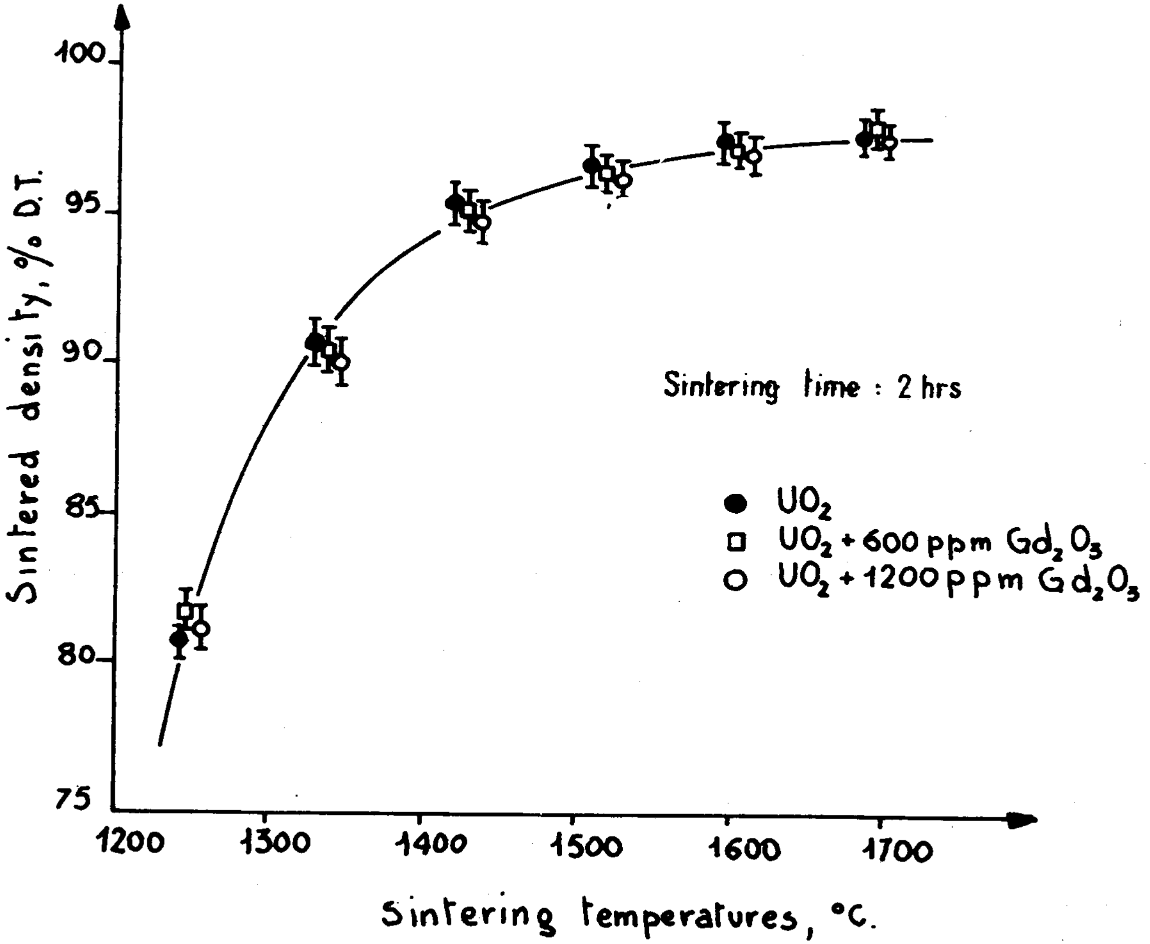


FIG. 10 – Sintered density versus temperature of poisoned and unpoisoned UO_2 pellets (Sintering time: 2 hrs.)

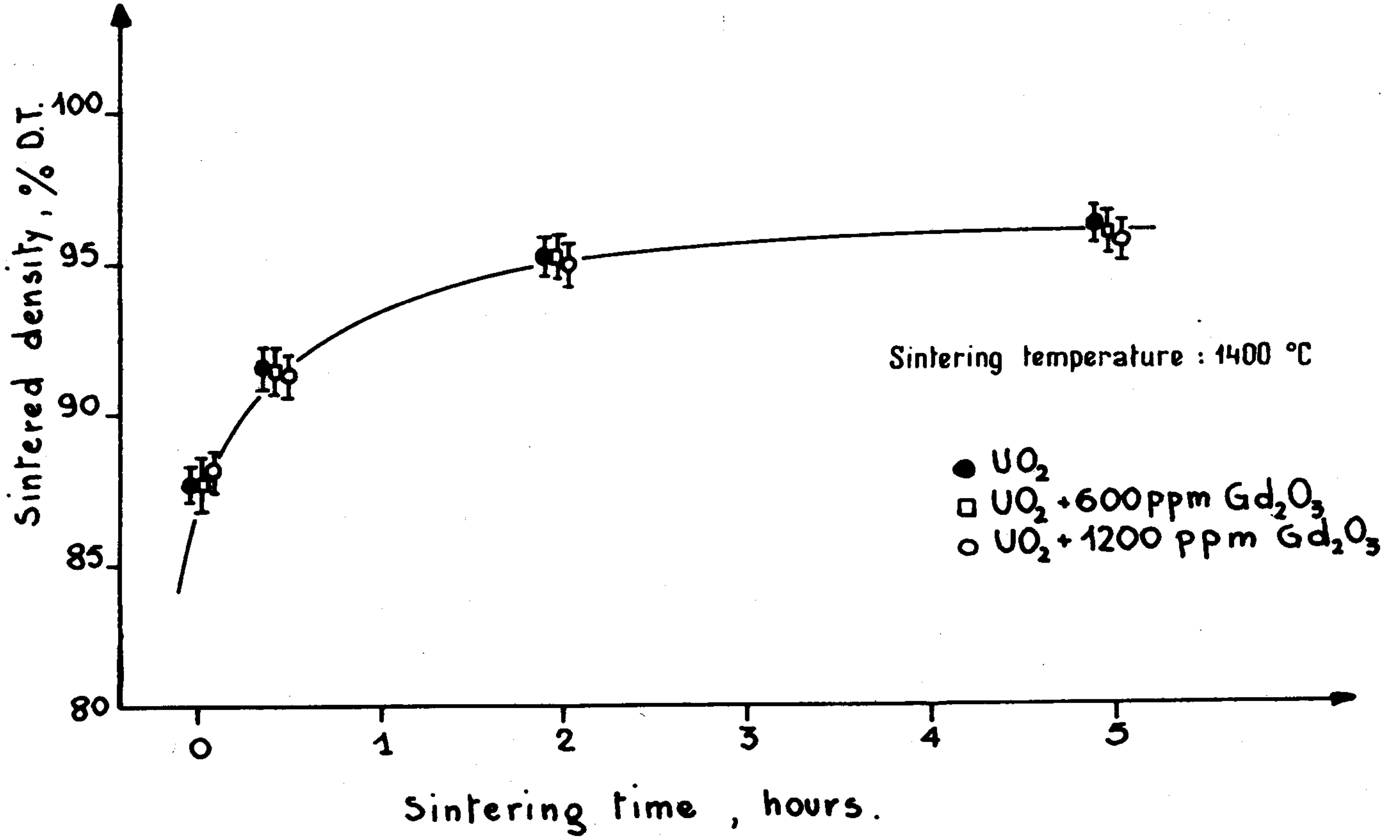


FIG. 11 – Sintered density versus time of poisoned and unpoisoned UO_2 pellets (Sintering temperature: 1400°C)

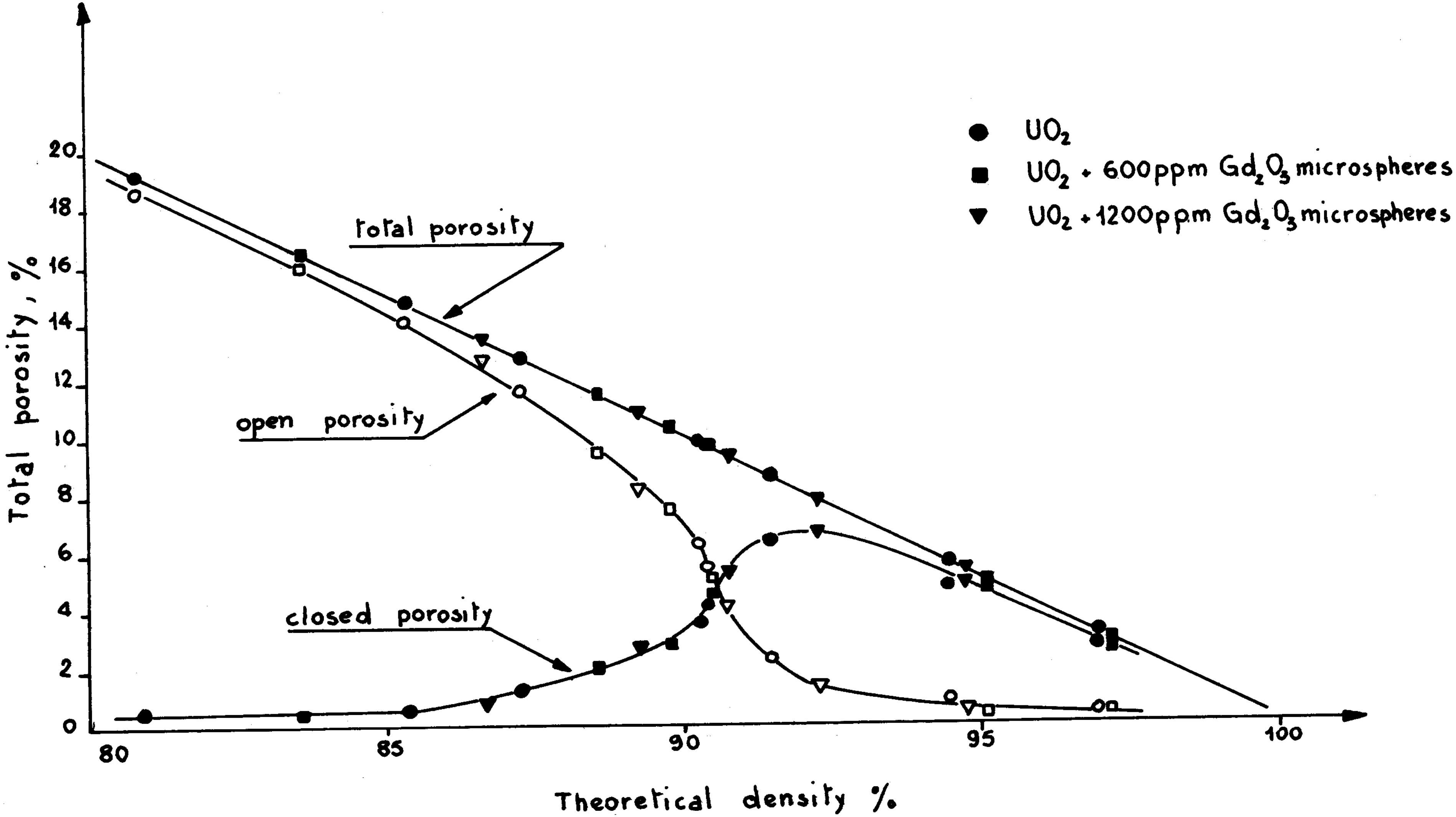


FIG. 13 - Open and closed porosity as a function of density for poisoned and unpoisoned UO_2 pellets

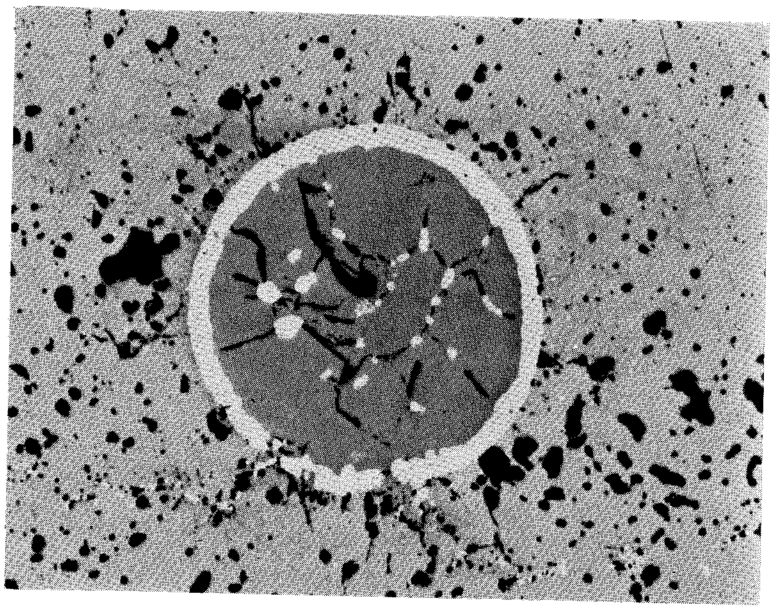


FIG. 19 - Typical microstructure of Mo-coated Gd_2O_3 microspheres after heat treatment. Temperature 2300°C; time 10 hrs (unetched, 100 x)

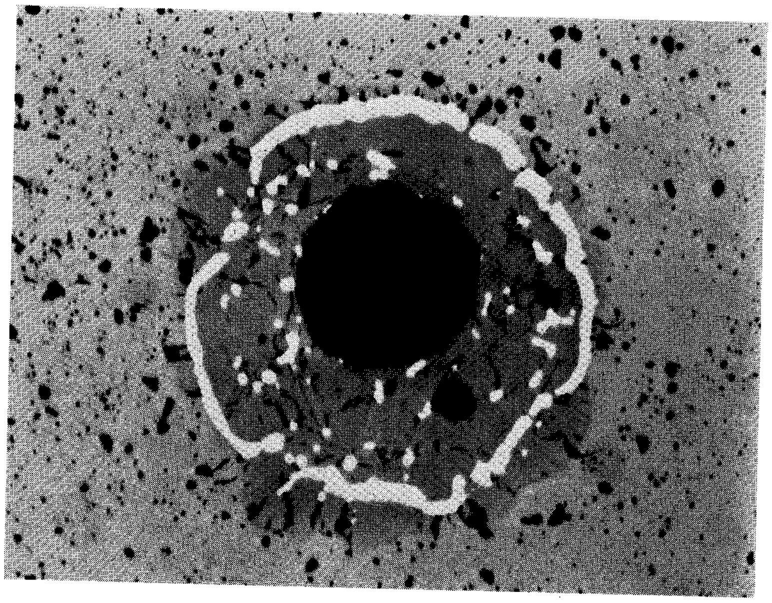


FIG. 20 - Typical microstructure of Mo-coated Gd_2O_3 microspheres after heat treatment. Temperature 2300°C; time 20 hrs (unetched, 100 x)

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Alfred Nobel

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