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IN-CELL CONTACT MICRO RADIOGRAPHY

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A. DRAGO and W. HUBER

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Joint Nuclear Research Centre
Petten Establishment - Netherlands

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ABSTRACT

Contact micro radiography is a powerful means to examine HTGR monolayer and coated particle fuels non-destructively. A successful effort has been made at Petten to perform contact micro radiography in post-irradiation analysis.

KEYWORDS

RADIOGRAPHY
FUEL COATED PARTICLES
MICROSCOPY
LAYERS
SPENT FUEL ELEMENTS

C o n t e n t s

F i g u r e s

IN-CELL CONTACT MICRO RADIOGRAPHY

I. Introduction

In the frame of pre- and post-irradiation examinations of HTGR fuels at Petten, equipment was needed for radiographing monolayer compacts and coated particles. At present two techniques are being used for radiographing coated fuel particles. These are contact micro radiography and point projection X-ray microscopy. Resolution of the order of 1 micron is expected and attained using either technique.

In comparison with the point projection X-ray microscopy the contact micro radiography costs less and is less complex too. These advantages were decisive in our choosing to develop this technique for both out-of-cell and in-cell equipment.

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II. Principle of contact microradiography

In general usage, microradiography is frequently taken to mean the method used by Heycock and Nevill (1) in which a thin specimen in close proximity to a photographic emulsion is irradiated with X-rays. This technique is named 'contact-radiography'. The film exposed and processed with this technique may then be magnified and photographed by transmitted light. Magnification of up to x 500 may be used. The magnification limit is imposed by film graininess.

There are two ways to produce contact microradiography, one with low voltage X-rays, and another that uses high-voltage X-rays excited at 150-200 kV (2). In this way the photographic image is produced by photo-electrons originating from the X-ray bombardment of the specimens. In our case the low-voltage technique was prefered. There are two factors that limit the resolution obtainable with a contact microradiograph of an unirradiated material: the geometrical unsharpness due to finite size of the X-ray source and the grain size of the emulsion. When the sample to be examined is a radioactive material, another important factor to take into consideration is the blackening of the film due to radioactive emanations from the sample.

The grain size of the emulsion represents the ultimate resolution obtainable, other factors however, may prevent this being attained. There is a large variety of X-ray films which differ in speed, contrast and grain size. A number of photographic detectors were tested, but the optimum for our purposes was Kodak High Resolution film (Estar Thick Base). Resolving power exceeds 2000 lines per millimeter.

Since a radiograph is a shadow picture, the image produced is affected by the relative positions of the specimen and film, by the size of the source focal spot and by the distance from focal spot to specimen.

In our case, the specimen to film minimum distance is 0,5 mm, the focal spot is 0,4 x 0,8 mm and the distance from focal spot to specimen is about 30 cm.

III. Description of the in-cell contact micro-radiography

The hot cell which is foreseen for this kind of analysis is the α, β, γ tight 'H' cell. This lead cell also contains the microsampling device with handling instruments for coated particles, a periscope and a dimensional measuring device, which at the same time can be used together with a collomator for gamma scanning of fuels.

The in-cell contact micro-radiography equipment has a horizontal beam so that the object has to be put upright in front of the film (fig. 1, 2).

The in-cell apparatus employs an identical AEG X-ray tube to that used for the out-of-cell contact micro radiographic equipment, in which one of the four Be windows has been reduced from 1 mm to 0,4 mm in thickness. The effective focal spot is 0,4 x 0,8 mm when the X-ray tube is installed at a 6° angle of inclination towards the direction of radiation. The admissable power of the tube is 1200 Watts.

The fully stabilized X-ray equipment Iso Debeyeflex 1000, is used.

The output voltage of the instrument can be continuously varied from only 5 kV up to 60 kV. The output current is continuously variable from 3 - 60 mA.

At 5 kV the X-ray tube can be operated at 20 mA.

Two α-tight pipes were installed in the cell, one houses the X-ray tube and the other contains the rabbit system for film exposure. Both pipes terminate in very thin plastic windows against which are located the thin X-ray tube window and the face of the film capsule.

The sleeve which surrounds the X-ray tube is connected to the α-box by a latex booting to allow enough movement for alignment; the other pipe, in which the film is exposed, being rigidly fixed to the α-box. A helium-filled pipe can be interposed in the path between the X-ray tube and the sample to reduce absorption of the soft X-rays.

The specimen holder locates the HTGR monolayer fuels at their circumference and supports them in front of and parallel to the film. The film is located in a plastic capsule the cover of which has a black paper window; a sketch of the arrangement shown in fig. 3. In use, these capsules are inserted into the rotating outer half of the lead shielded shutter system providing access to a pneumatic rabbit (fig. 4) which conveys the capsule holder to its operating position. After radiographic exposure the capsule is withdrawn and the exposed film extracted. After development the film is examined under a microscope.

The transport of the film capsule through the rabbit system takes about 1 second.

IV. General consideration and experimental results on irradiated material

In the post irradiation contact micro-radiographic technique, a limitation in the exposure time and in the clearness of the image is imposed by the level of radioactivity of the dample. Teh presence of this supplementary field of radiation will generate a competitive process of blackening of the film. The α-radiation can be forgotten when deciding on what precautions to take into consideration, because α-particles will be stopped by the window of the film capsule. As far as the gamma radiation is concerned, both the level of intensity and the scattering phenomena that can take place in materials of high atomic number lying near the film capsule. The energy level of the gamma radiation will be generally much higher than that selected for the X-ray, hence the density of ionization in the emulsion by gamma radiation will be lower than for X-rays of the same intensity. All these considerations were taken into account during the realization of the in-cell device,and a final optimization of the film package was made by tests performed with a 5 mC Sr^{90} - Y^{90} source.

The first experimental results have been obtained from irradiated monolayer comoacts. The monolayer compact consists of triplex coated particles bonded together by 'matrix' graphite into an annular monolayer of about 22 mm OD, 12 mm ID and 2 mm thick. A partial view of a radiographic image of a monolayer obtained in the cold laboratory with an equivalent device is presented in fig. 5. It is possible to distinguish, in the figure, delamination phenomena occurring both between the outer pyrocarbon coating and the overcoating and inside the overcoating together with the regions of low density (dark region) present in the matrix.

In fig. 6 is presented a radiographic image realised with the in-cell device on one of the particles contained in an irradiated monolayer. The monolayer was one of a series irradiated at 900°C in the HFR at Petten to a fast neutron dose of $1,1 \times 10^{21}$ n/cm^2 NDE. The operating arrangement was that presented in fig. 3 with an applied voltage of 40 kV and an electron cuurent of 30 mA. The exposure time was 90 sec.

In fig. 7 a partial view of a radiographic image is presented which was obtained from an irradiated graphite sample of 3mm thickness. The operating conditions were the same as mentioned above. In the picture it is possible to observe the region of low density, the inclusion of high density material and the existence of cracks.

The radiographic examination of coated particles presents two advantages: the possibility to study the progression and the evolution of a kernel coating interaction; to reduce considerably the number of the metallographic examinations by an opportune selection of the samples.

A c k n o w l e d g e m e n t

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Mr. Keller for his design and development work,
Mr. Agne and his workshop for the realization of the device,
Mr. Oudaert and Mr. Puschek for the mounting and testing of the device in the hot cell.

L i t e r a t u r e

(1) C.T. Heycock and F.H. Neville, J. Chem.Soc. 1898, 73, 714.

(2) J.J. Trillat, Metallurgical Reviews 1956, 1,3.

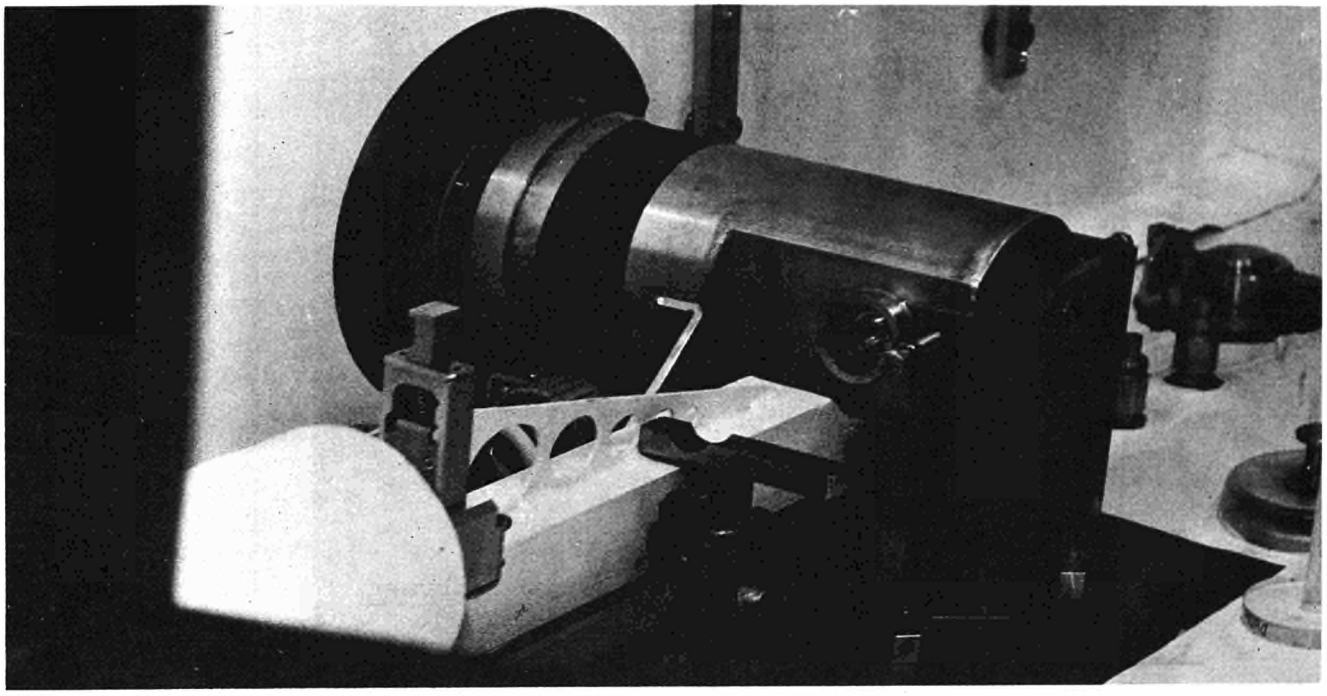

Fig. 1 X-ray tube holder with X-ray window and adjustable support

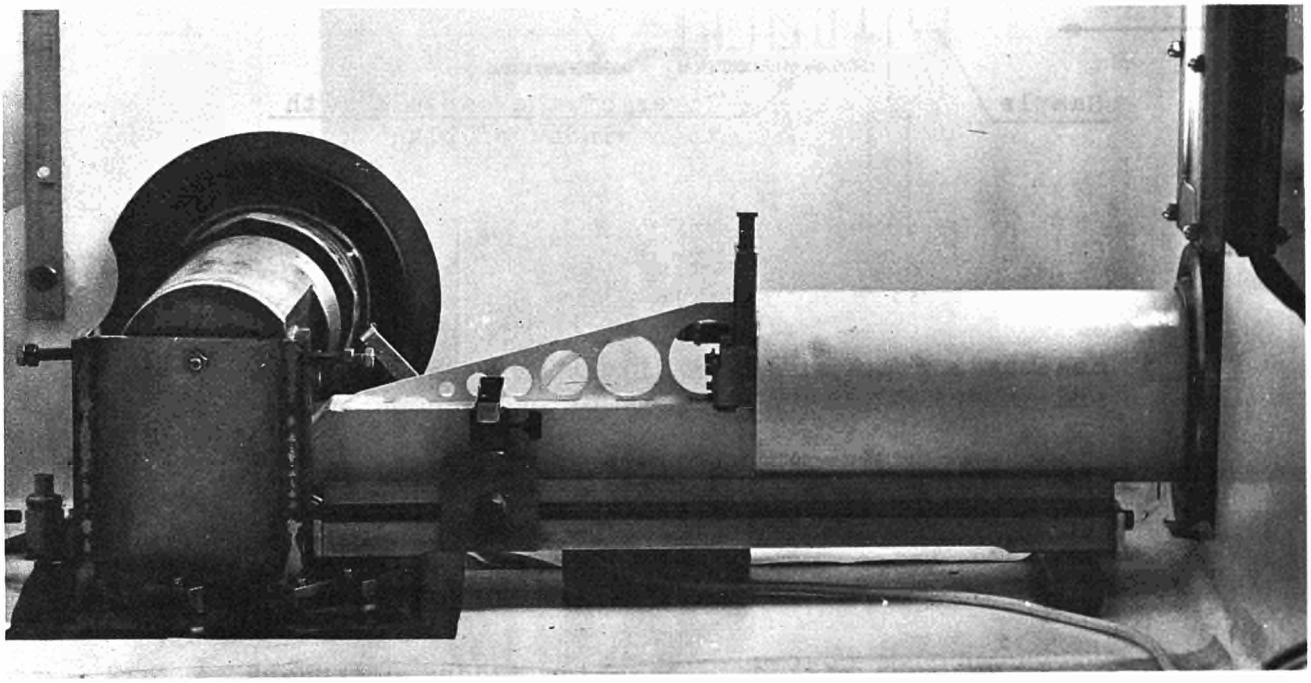

Fig. 2 Film supporting tube with sample holder

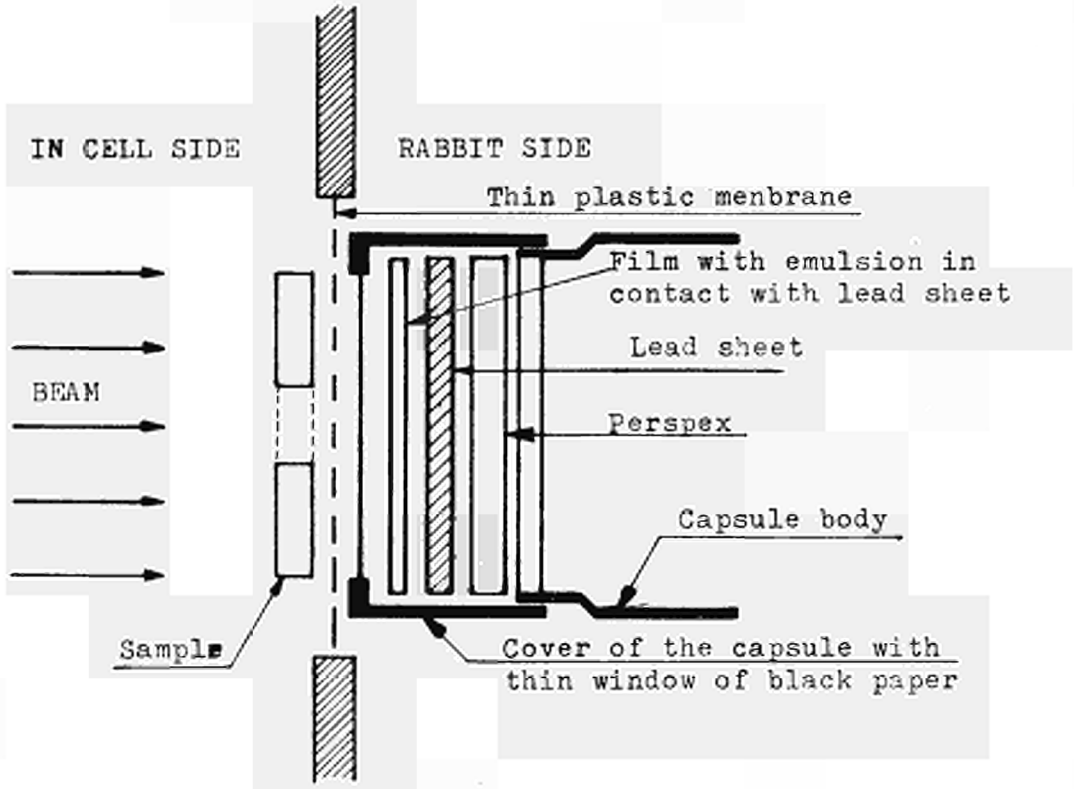

Fig. 3 Schematic representation of the film capsule and the relative arrangement of each component

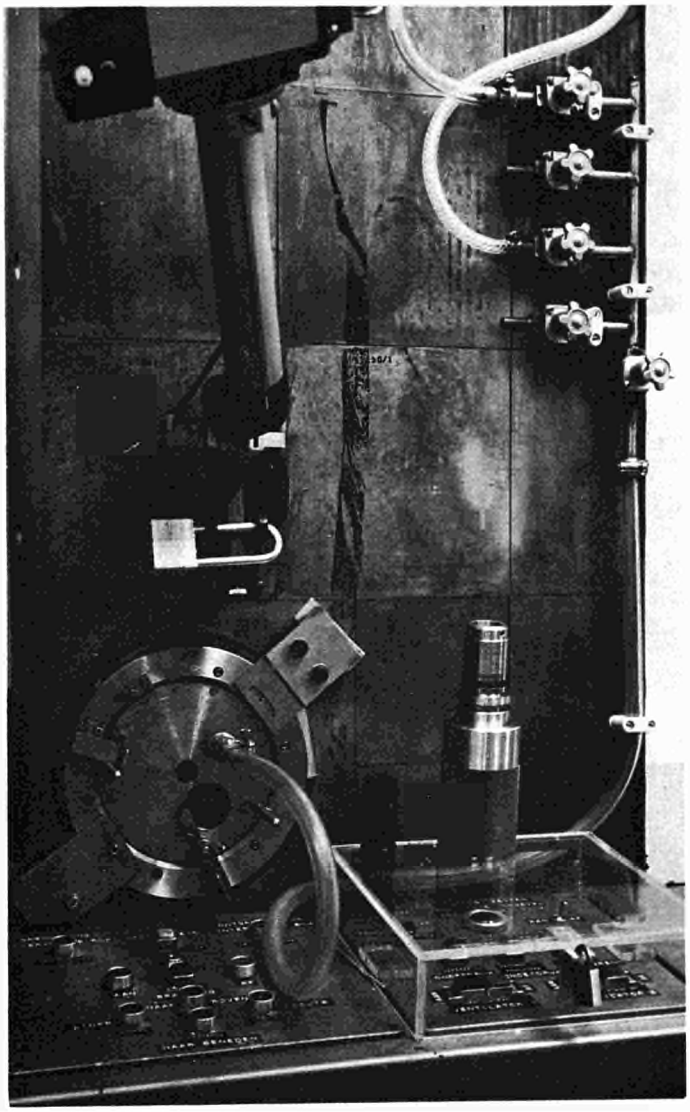

Fig. 4 Pneumatic rabbit entrance with film capsule and enter stainless steel plug

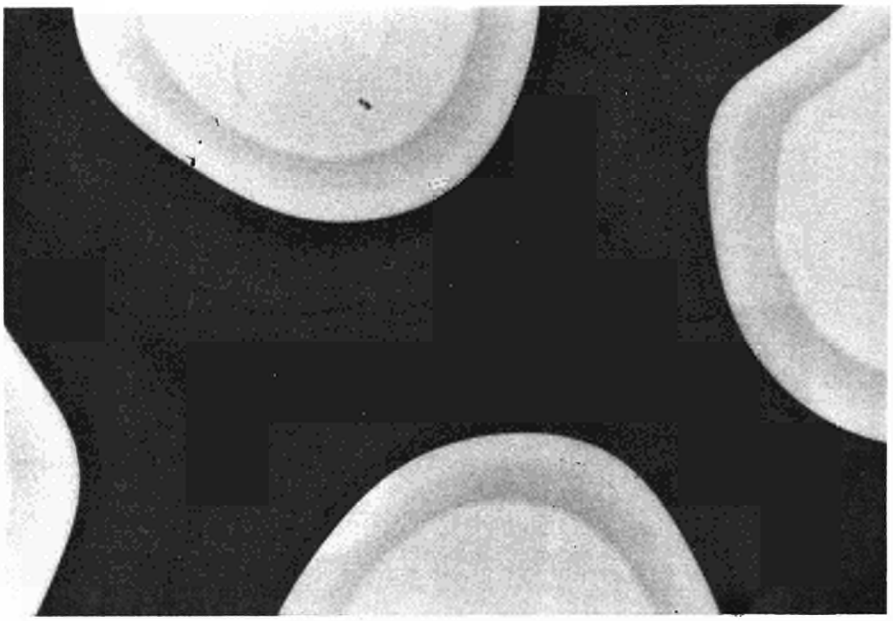

Fig. 5 Partial view of a radiographic image of a unirradiated monolayer compact realized in the cold laboratory

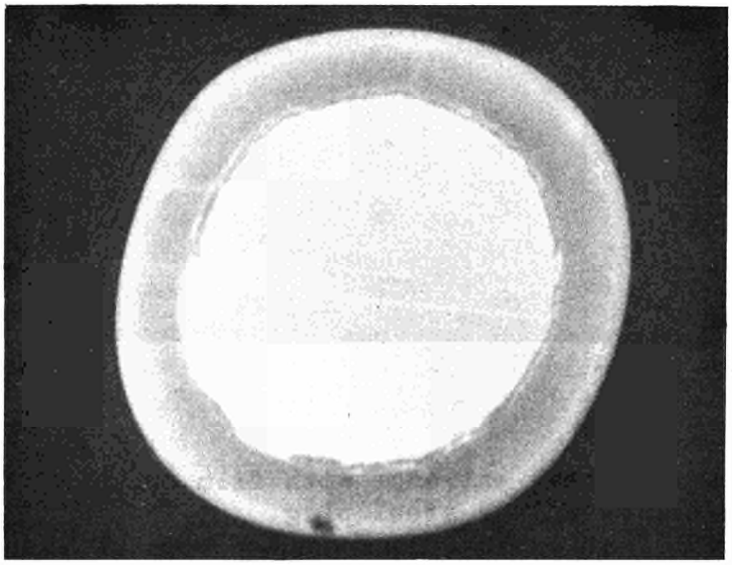

Fig. 6 Radiographic image realized with the in-cell device of a particles contained into an irradiated monolayer compact.

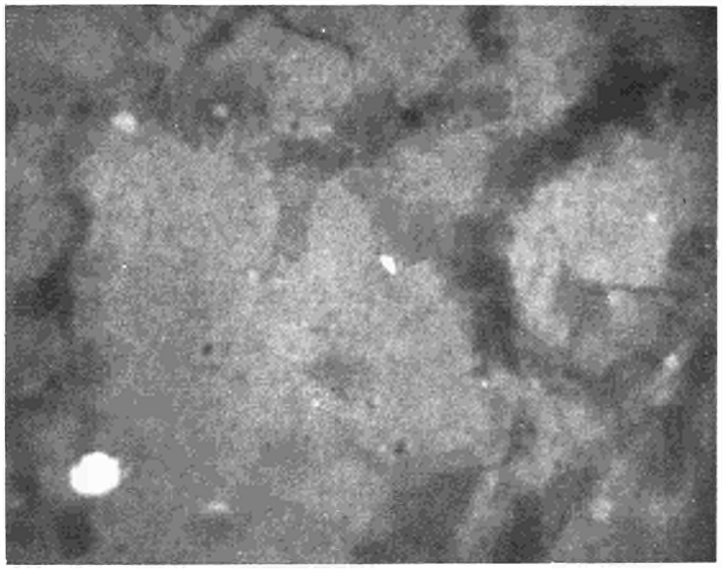

Fig. 7 Partial view of a radiographic image of an irradiated 3 mm thick graphite sample

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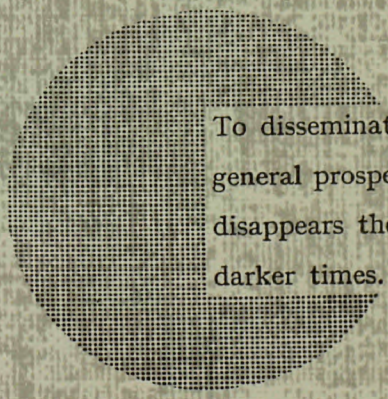

To disseminate knowledge is to disseminate prosperity — I mean general prosperity and not individual riches — and with prosperity disappears the greater part of the evil which is our heritage from darker times.

Alfred Nobel

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