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# DETAILED SEM - EPMA INVESTIGATION OF HIGH SPECIFIC RADIOACTIVITY PARTICLES (HOT PARTICLES).

K. I. Burin[+], Ts. I. Tsacheva[++], I. G. Mandjoukov[+], B. Mandjoukova[+],

[+]Sofia University, Faculty of Physics, blvd. "J. Boucher" # 5
Sofia - 1126, BULGARIA

[++]Institute of Physical Chemistry ,Bulgarian Academy of Sciences, Sofia - 1040, BULGARIA.

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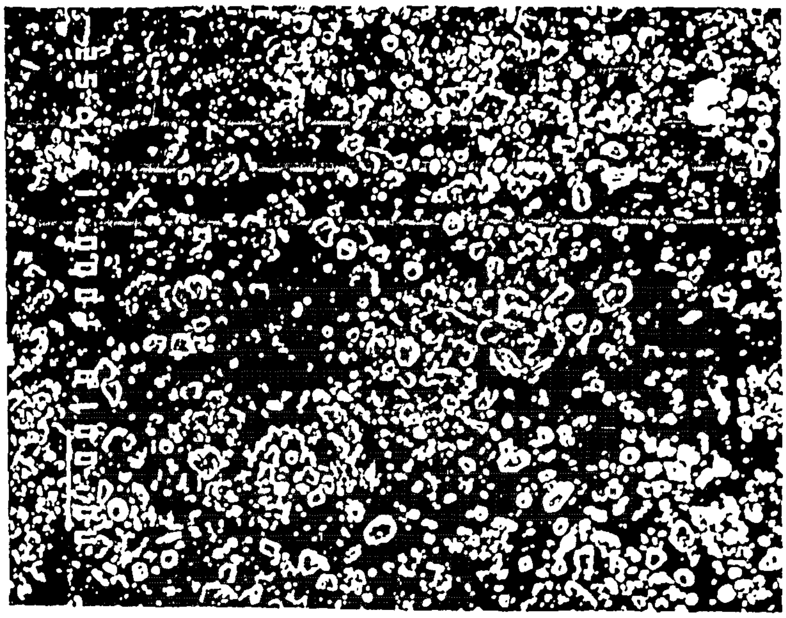
## Introduction.

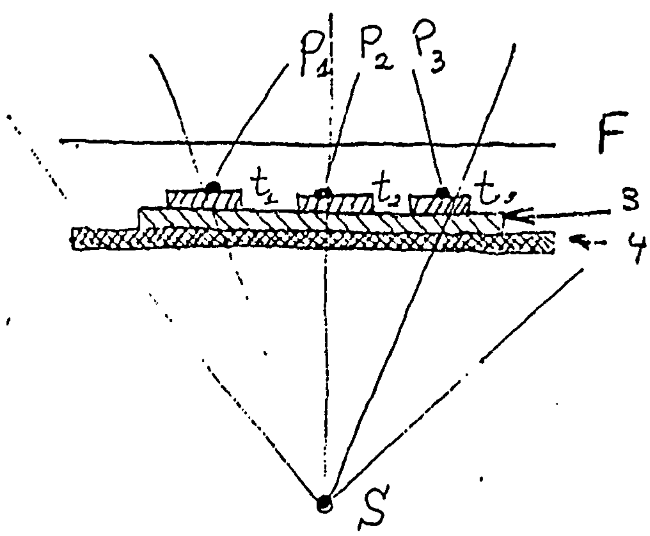
This work follows one published in the "Radiation Protection Dosimetry" journal [1]. There the reader can find details about the way our specimens were prepared and investigated using SEM - EPMA technique. Now we use new methods for more detailed investigation of the reactor fuel-containing particles, spread all over Europe after the Chernobyl accident. Microscopic particles with high specific activities are reported to be present at operating nuclear power plants [2]. For this reason we hope the methods reported here and illustrated with some examples will be of actual interest.

### 1. Easier finding of the particles:

As it is described in [1], we prepare our specimens by applying a piece of adhesive tape on the surface where "point" radioactivity had been detected. Along with the hot particles hundreds of other particles usually stick to the surface of our tape: Fig. 1.



In order to reduce the area upon which the hot particle should be searched for in the scanning electron microscope we proposed and tested the following method: Fig. 2.




P1, p2 etc are the hot particles, collected on pieces of adhesive tape (t1, t2 etc).

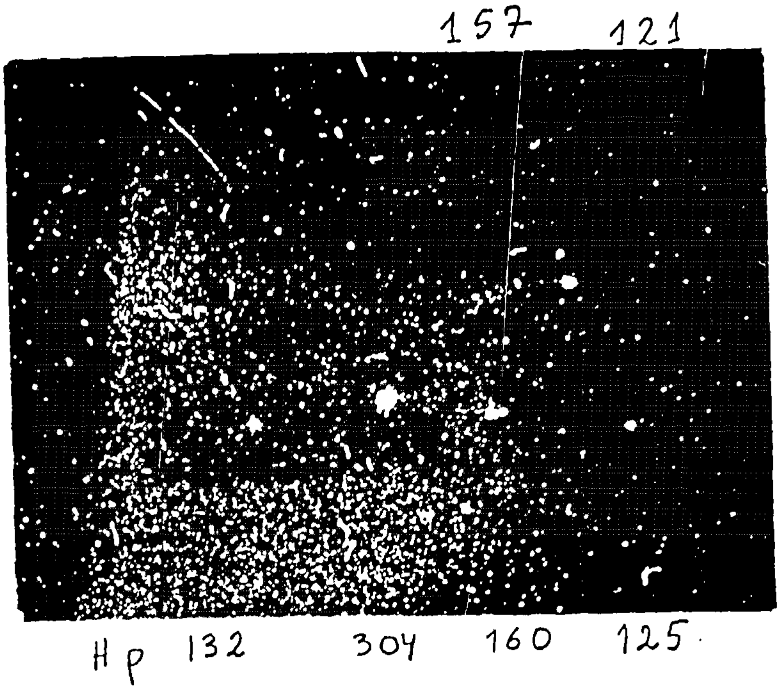
3 is a larger piece of similar tape but with glue on the both sides.

4 is a thin (0.1 mm) Al sheet.

F is a piece of photographic film.

S is a $\beta$-ray source ($^{90}Sr$) with an activity of about $4 \times 10^5$ Bq.

Our experience showed that after two days of irradiation and the proper processing of the film, the Figure # 3 is obtained:




The dots are autoradiographs of the hot particles and the contours of the adhesive tape pieces are visible also. This way we know where to look for our objects.

2. Actual finding of the particles:

No changes were made in comparison with [1] in finding the particles on the pieces of tape. No difficulties were met either. The results will be reported below in the appropriate section.

3. Obtaining of X-ray spectra:

For each particle an X-ray spectrum had been taken from an rectangular area covering the particle completely. The Si(Li) spectrometer was used in order to collect information about all the present elements simultaneously. The mode of high resolution rather than high count rate has been preferred. We did not look for low intensity peaks at this time and between 5 and 10 minutes were sufficient for obtaining of a spectrum. The electron probe current was determined by the acceptable for the Si(Li) spectrometer dead time (not exceeding 20 %), for which no deterioration in the resolution had being observed.

4.Dot maps acquisition:

It is a method of operation of the electron probe microanalyser (EPMA). The electron probe scans a selected area from the specimen, organized as a square matrix of $n^2$ dots. N can be chosen between 16, 32, 64, 128. At every dot the probe hits for a preset time, called "dwelltime" and after that moves to the next one. The Si(Li) spectrometer is in operation all the time. In advance up to about 10 Regions Of Interest (ROI) along the X-ray spectrum are fixed. The software, controlling the operation of the EPMA stores in the memory the gross counts, obtained for each ROI during the dwelltime for each point. Unfortunately background corrections are not provided, which can cause difficulties in some cases.

Further the data collected this way can be displayed as follows:

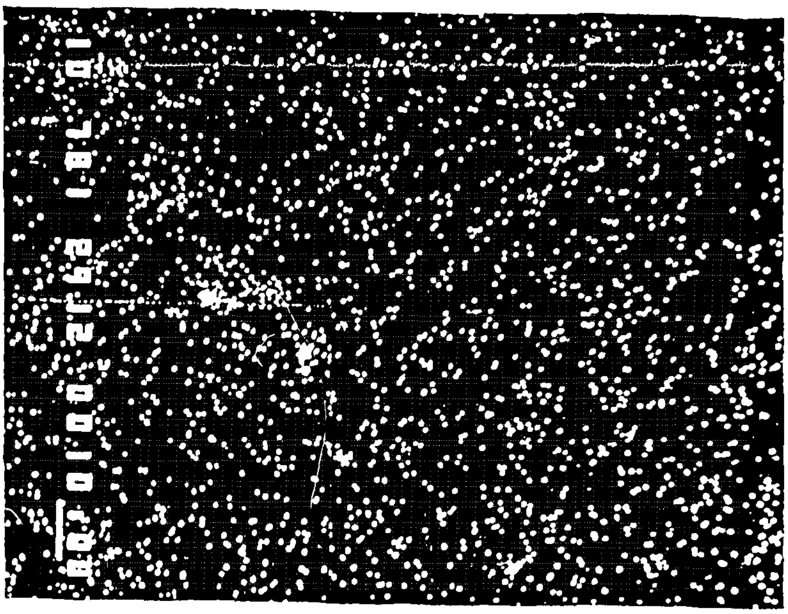
a). For each ROI, which is one to one connected with a given element a map can be recalled on the screen on which each point where the probe had stayed is represented by a square spot with different colour. The colour encodes the number of counts, obtained at the point. It varies from black through blue, green, red, yellow etc, to white, representing the numbers from 0 to 255 divided into 16 steps. Numbers exceeding 255 give white colour. If the maximum number for a map is less than 255,it is taken as the upper value for the 16-grade scale, this way making it more precise. In this way a map of the distribution of a given element, represented by the appropriate ROI in the X-ray spectrum is obtained.

b) Once this map being displayed on the screen, a lot of operations can be made with the data. They include recall of the counts for every dot, overlap of several maps (taken with different ROI), display on a histogram the distribution of the counts for all the dots. (On this histogram operations can be made such as rejection of the counts outside of a selected window. Than a new map can be generated, using the remained distribution of the counts over dots this way enhancing interesting details). Line profiles across the mapped area can be drawn, parts of the map with bigger magnification can be displayed, etc.

The conditions of map acquisition are of particular interest. In order to obtain all the maps for a given particle simultaneously we chose different number of channels in each ROI this way obtaining approximately equal count rate for all the elements. Another problem arises from the bremstrahlung background. It is higher when the electron probe hits the Uranium containing hot particle. For this reason a "phantom" image coinciding in place and shape with the Uranium distribution could appear even for an element not present there.

Results:

Particle # 157 proved to be fragmentated into 5 or 6 pieces as it can be seen on pict. 4 and we did not engage ourselves with it further:



For each one of the remaining particles the x-ray spectrum is presented, a SEM photo in secondary electrons and several dot maps, giving an idea about the distribution of the chemical elements in the particle and around it (see the Appendix).

Conclusions:

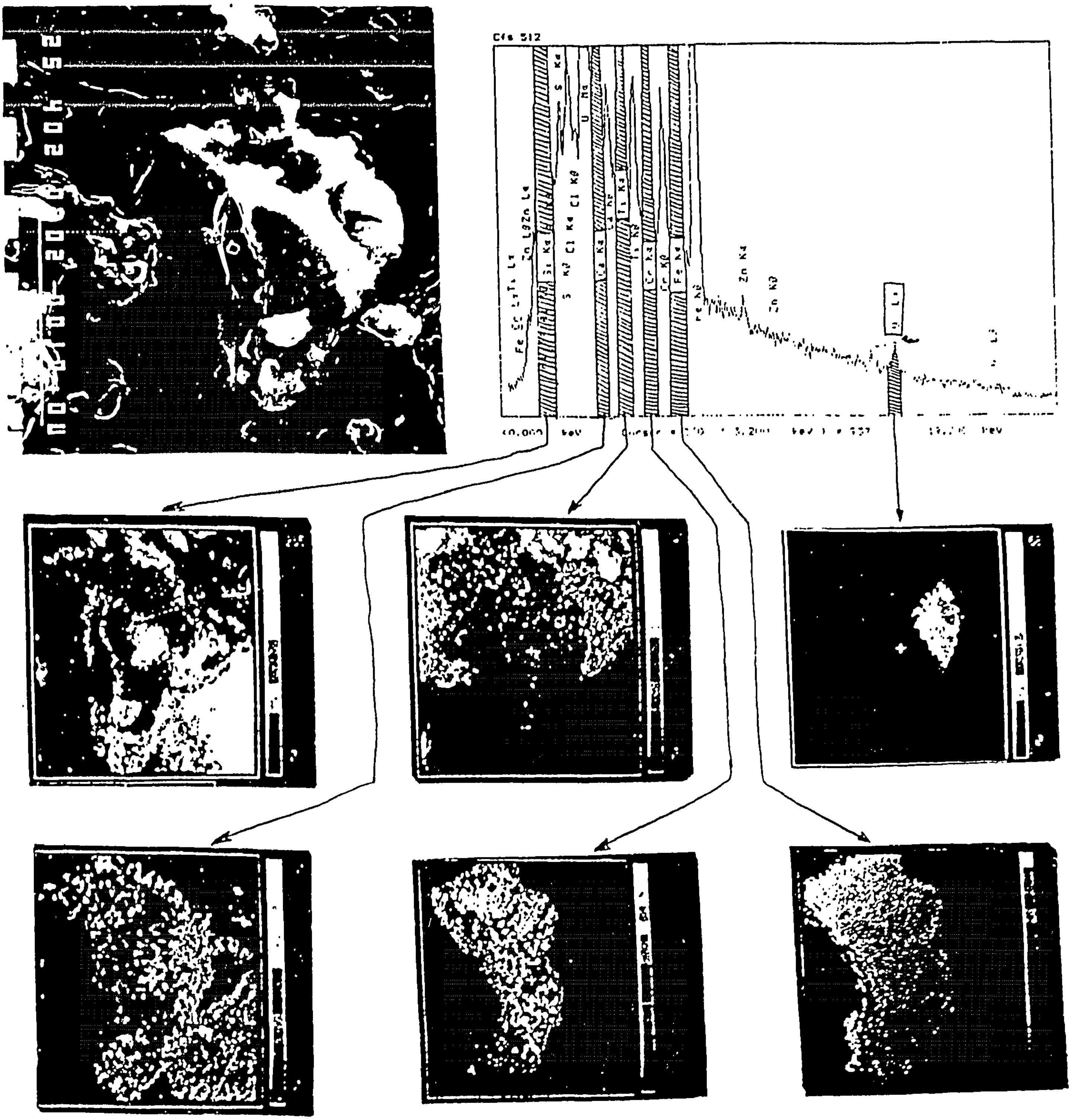
The first conclusion, which can be drawn after the examination of the dot maps is, that this group of particles is not made of pure core fuel, but includes either construction materials, or materials used for the covering of the damaged reactor.

REFERENCES:

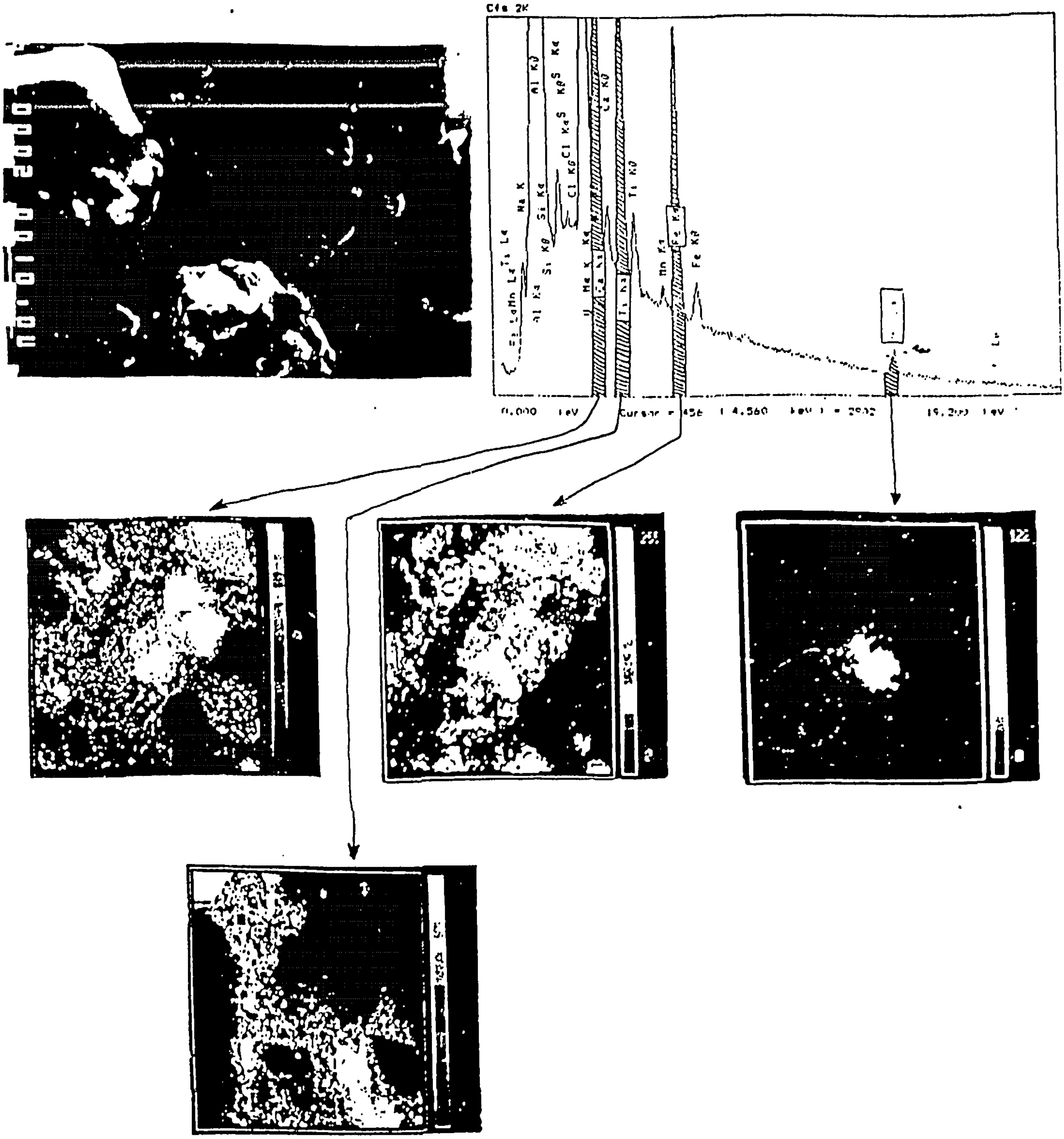
1. I. G. Mandjoukov, K. Burin, B. Mandjoukova, E. I. Vapirev and Ts. Tsacheva: Spectrometry and Visualization of "Standard" Hot Particles from the Chernobyl Accident. Radiation Protection Dosimetry, Vol. 40 # 4 pp 235-244 (1992).

2. lodde, G. M. : Control of Hot Paarticles Challenges GPU Nuclear and the Nuclear Power Industry. Trans. Am. Nucl Soc. (USA). Vol. 60 # 9 pp 517-18 (1989).

Appendix. Particle # 132:



Appendix. Particle # 304:



Appendix. Particle # 160: