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Fuel Coatings Quality Control Radiographic Examination Technique

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HFR TECHNICAL MEMORANDUM

Fuel Coatings Quality Control
Radiographic Examination Technique

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EXECUTIVE SUMMARY

In order to develop HTGR technology in Europe it is necessary to master the HTR fuel manufacturing with a high quality fuel, for this reason the HTR-F project is devoted to fuel technology development. The global objective of the HTR-F is the coated fuel particle qualification at high burn-up with a high reliability.

Characterisation of the particles is a key point to reach a high quality fuel; the task 4 of the HTR-F project is specially dedicated to specify and to test a method, based on X-Rays microradiography, for coatings quality control. The scope of the work presented in this report was to assess the radiographic examination as a method for quality control of the coating of HTR fuel particles.

X-ray evaluations of simulated particles for HTR fuel have been performed at the JRC-IE. Afterwards the coating thickness have been calculated from the analysis of the digitised radiographs by using the software ImagePro Plus to perform:

a) Direct dimensional measurements
b) Density profile analysis

In general, the obtained results for the coating thickness are in good agreement with the specification, showing slightly smaller values than those reported in the report "Coating of particles" (HTR-F Deliverable 16).

The task is concluded with a full study of the errors associated with the measurement technique. By means of dimensional measurements the thickness can be determined with an accuracy of 5 μm. Using the density line profile analysis the coating thickness is calculated with an accuracy of 7 μm.

INDEX

INTRODUCTION

In order to develop HTGR technology in Europe it is necessary to master the HTR fuel manufacturing with a high quality fuel, for this reason the HTR-F project is devoted to fuel technology development. The global objective of the HTR-F is the coated fuel particle qualification at high burn-up with a high reliability.

Based on the experience studies are devoted on fuel kernel fabrication, coating of particle and compact or pebble manufacturing. Another important objective of the work is quality control of such particles.

Characterisation of the particles is a key point to reach a high quality fuel; the task 4 of the HTR-F project is specially dedicated to specify and to test a method, based on X-Rays microradiography, for coatings quality control. A review of different types of control that can be applied to particle fabrication is foreseen in Task 4 of the HTR-F1 project.

The JRC-IE is in charge of assessing the radiographic examination technique for coating QC, in particular to determine the size of the coating layer. This report presents the experimental results of the coating thickness analysis on simulated particles for HTR fuel and constitutes the deliverable number 17.

MATERIALS

In August 2001, approximately 5000 samples of coated particles were received at the JRC/IE coming from CEA/Grenoble.

The Japanese company TOSOH manufactured the particle kernel. The material used as substrate is Zirconia (ZrO_2 on 94.5%) partially stabilised with Yttrium oxide (Y_2O_3 on 5%), plus some impurities. The density of the kernels is about 6 g/cm^3 and the mean diameter of about 650 μm.

The kernel is coated with different layers, namely a Buffer layer and/or an Inner-PyC layer.

The received particles are divided in three different groups as a function of their coating thickness:

ZP 12 (07 & 08): These are samples with only buffer PyC coating. The target thickness is 95 μm.
ZD 13 (07 & 08) are samples with only Inner PyC coating and a target thickness of 40 μm.
ZD 14 (07 & 08) consists of realized buffer and Inner PyC continuous in the same conditions of coating and target thickness like ZP 12 and ZD 13, thus the thickness is 135 μm.

EQUIPMENT

A radiographic examination technique of coated particles has been developed in the past at JRC/IE in view of their integrity evaluation. Such X-ray technique is being used to determine the size of the coating layer. The measurement conditions are given in the Table 1.

Table 1. X-ray characteristics

Apparatus	Andrex MX 4, microfocus
Film Focus distance	400 mm
Voltage	140 kV
Intensity	0.25 mA
Time exposure	40 seconds
Film	Agfa D2/PB

First trial with X-ray was to fix the particles in a single layer and then performing the radiograph. The result is shown in the Figure 1. As it can be seen there exists clearer areas, which correspond with the particle's kernel. However the particle end is not clearly defined and the determination of the coating thickness it is not straightforward. To be able to measure the coating the particles need to be in contact.

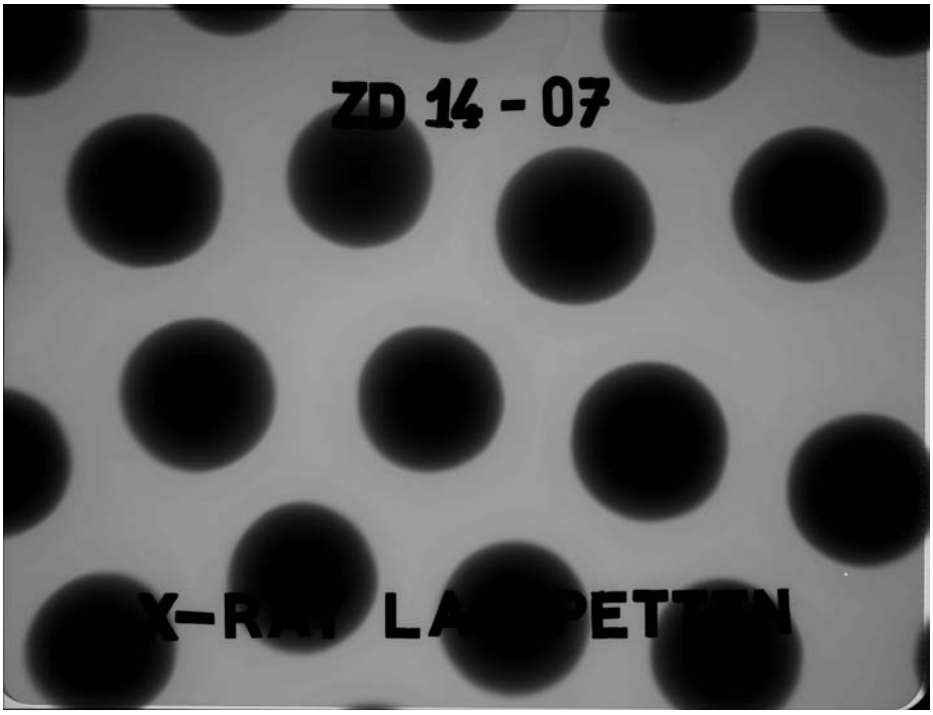


Figure 1. Radiograph without carried

For this purpose an "ad-hoc" particle carrier, which allows fixing the particles in contact between themselves and includes a calibration wire, has been designed, see Figure 2. By mean of this carrier the examination results and their statistical analysis (control based on image analysis system) will provide a more accurate coatings layers metrology.

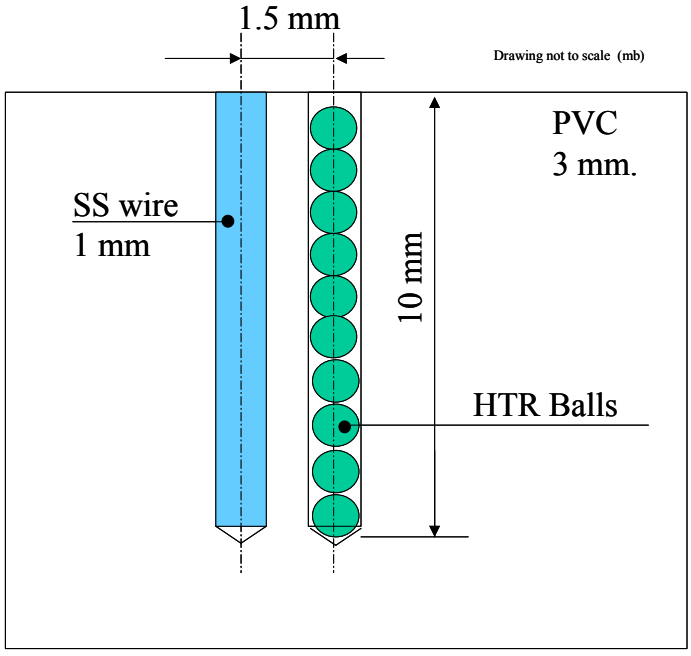


Figure 2. Design of the particle carrier for X-ray examination

The carriers in horizontal position are shown in Figure 3, the support is made from a plastic transparent to the X-rays. The carrier is located vertically in the X-ray device so that the particles are kept in contact by gravity.

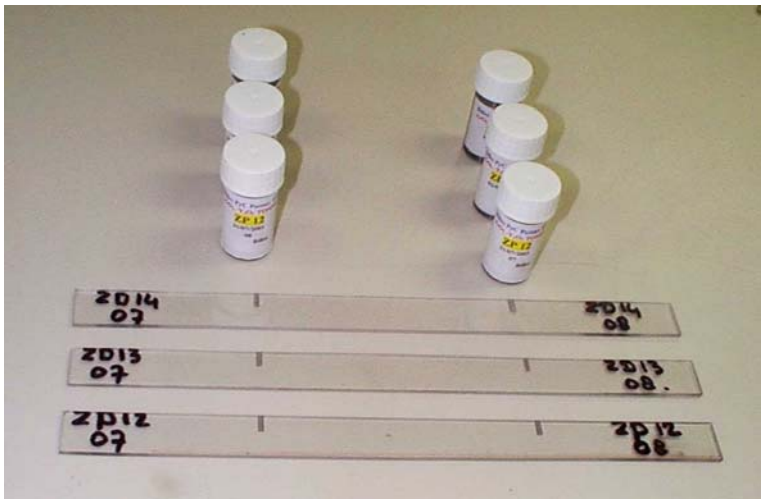


Figure 3. View of the carriers

The particles' external diameter has been also determined by using a profile projector (type Mitutoyo PJ 3000), where the image is as shown in the Figure 4. The procedure consists on taking a sampling of 10 particles for each group and measuring them four times.

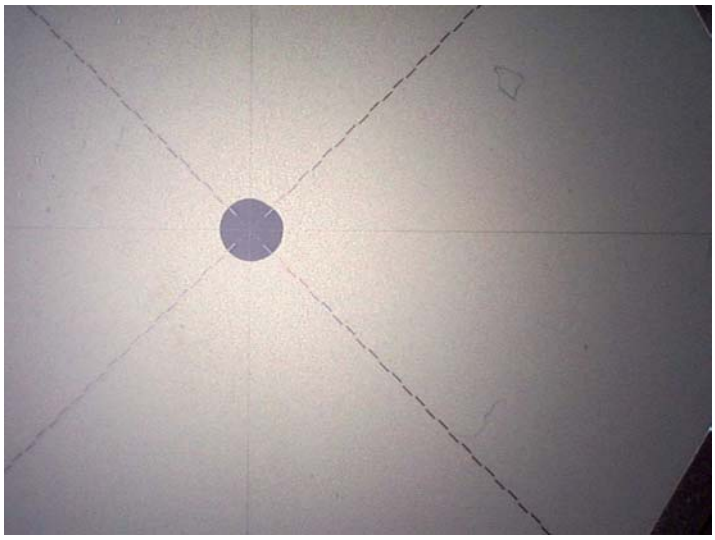


Figure 4. Particle view with the profile projector

IMAGE ANALYSIS

X-ray film was digitised at the X-ray Laboratory using a Kodak Lumiscan 85 Film Digitiser. The digitisation process divides the image into a grid of very small regions called pixels. By convention, pixels are referenced from the upper-left position of the bitmap that is considered as the "0,0 position". The digitised image is then converted into bits of information into 12-bit grey scale format. It means that intensity values are represented with 12 bit integers and provides 4096 levels of greys from 0 to 4095. The image is saved as a TIF file (Tag Image File Format), which can be handled using the Image-pro Plus 5 program. This program provides a full range of utilities for image processing such dimensional measurement, count and measure objects, and density line profile that have been using to determine the coating thickness.

Dimensional measurements: Measurements operations are performed in terms of image pixel position (i.e. the length of a line is calculated by the number of pixels along the line). The spatial scale is calibrated before taking measurements, and then the dimension is measured from centre pixel to centre pixel.

Count and measure objects: For counting purposes, objects are identified by their intensity in monochrome images, the Image-pro analyses the image, calculates the intensity distinguishing objects from background and establishes the intensity range based upon the objects that are found. Once the objects have been defined several measurement options are available, in this analysis the following have been used:

Diameter (mean): Reports the average length of the diameters measured at two-degree intervals joining two outline points and passing through the centroid of the object. This serves to determine the kernel's diameter.

Max/Min Radius: Reports the maximum/minimum distance between each object's centroid pixel position and its perimeter.

Density profile

Line Profile analysis plots the optical intensity values along a given line. A profile plot shows the pixel positions of the line along the X-axis, and on the Y-axis, measures the index value for each position along the line. In a line profile, the X-axis represents the spatial scale (if it has been defined, otherwise it is the distance in pixels), and the Y-axis corresponds to the intensity values (calculated from the intensity calibration). The intensity is calibrated before taking a density profile, by defining the black (indicating that no light is present) and incident or white (indicating that no material is present) levels. The optical density scale reflects an exponential decay of light within a transmitting material, so that the optical density curve is inversely related to the intensity scale:

- Small intensity values reflect large density values
- Large intensity values reflect small density values, i.e. the darker the pixel the denser the material.

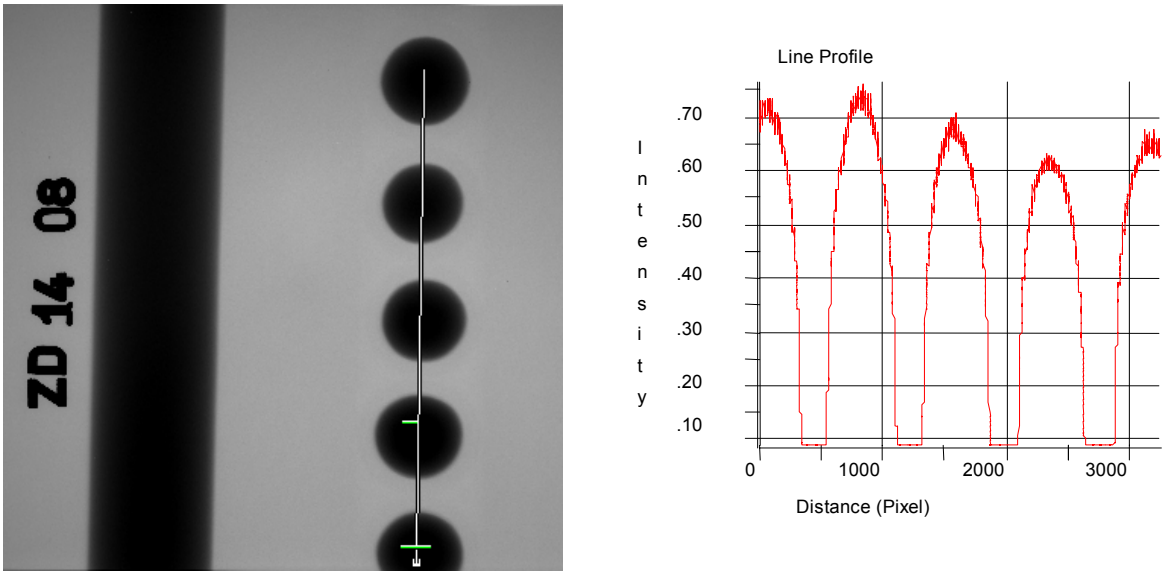


Figure 5. Example of a density profile along the line passing through an entire row of particles (ZD 14-08)

The example shown in the Figure 5 gives an idea of the results from a line profile analysis. The image has been calibrated to define the empty area (the background) as the zero intensity value. The densest materials, in this case the "kernels", which are darker in X-ray graph, and therefore report higher pixel intensity values and decay from the centre to the surface (as effect of the X-ray transmission) in the line profile plot. From kernel to kernel there is the coating as the particles are in contact between themselves, and this is reported by the lower intensities.

RESULTS

Direct measurements

It is known that from the X-ray image the clearest area is the "kernel", from that the kernel's diameter $[d_k]_i$ and kernel's centre can be easily determined. If one is sure that the particles are in contact, the centre-to-centre distance will then constitute the particle's diameter $[D]_i$. And consequently the coating thickness $[S_c]_i$ will be the semi-difference between $[D]_i$ and $[d_k]_i$. This can be seen in the Figure 6.

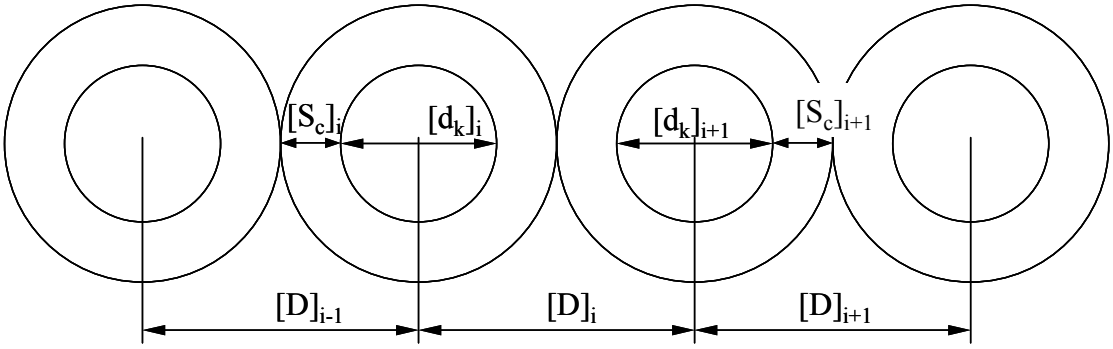


Figure 6. Definition of kernel and particle's diameter and coating thickness

The procedure to analyse the radiographs of the particles loaded in the sample carrier, using the Image-pro is the following:

1. Calibrate the spatial scale using the 1 mm diameter wire.
2. Create the circles that best fit the kernels and obtain their radios.
3. Measure the centre-to-centre distance between two kernels.

The Figure 7 is an example X-ray graph treated for the dimensional measurement. The defined kernel's best-fit circle is marked in yellow as well as the wire length (L1 used to determine the calibration error). The red lines are the distance from centre to centre, that is $[D]_i$. Note that the image contrast has been inverted in order to best draw the kernel.

From the Figure 7 two facts can be observed: First, kernel's diameters are approximately the same but particles from the left image are smaller than these from the right (the length and diameter of the hole where particles were inserted is identical for both carriers, see Figure 2), this depends on coating thickness. Second, kernels are not completely round and thus coating thickness is not uniform. The last induces some errors in determining the thickness, thus statistics have to be applied.

It has also to be remarked that when placing the ZD 13-08 particles some pressure was applied and particles at the bottom collapsed, see Figure 7 left image at the bottom.

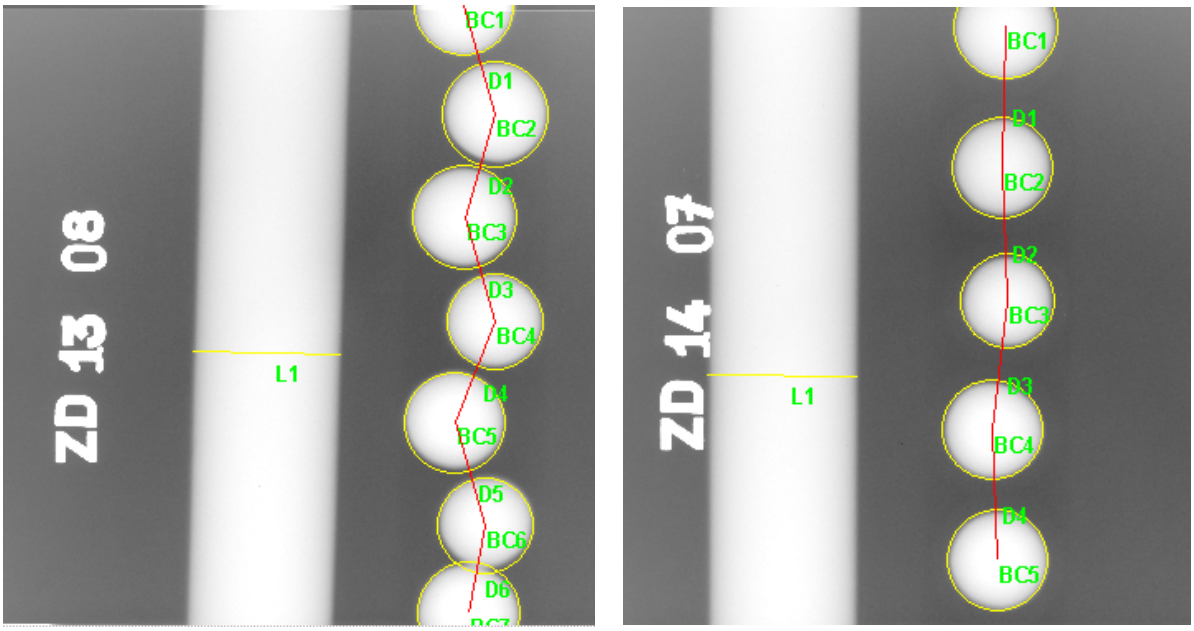


Figure 7. Definition of features for dimensional measurements (ZD 13-08 and ZD 14 07)

4. Then the "corrected" particle diameter Ďi will be:

$$\overline{D}_i = \frac{[D_{i-1}]+[D_i]}{2}$$

5. And the coating thickness $[Sc]_i$ from the Figure 6 for one particle is

$$[S_c]_i = \frac{\overline{D}_i}{2} - \frac{[d_k]_i}{2}$$

6. Therefore the relation between coating thickness and particles' diameter is:

$$RC = \frac{\sum_i^n \frac{[S_c]_i}{\overline{D}_i}}{n}$$

7. And then only remains to obtain the coating thickness for a given batch, S_c, using:

$$S_C = RC \cdot D_m$$

D_m is the measured particles' external diameter, calculated using the profile projector, and reported in the Table 2. The resultant external diameter is in good agreement with the specifications. The tolerance in the diameter is given by the standard deviation of the four measurements for the 10 particles sampled from each batch.

Table 2. Particle diameters from the profile projector

	D_m mm	Tolerance ±
ZP 12-07	0,859	0,031
ZP 12-08	0,878	0,020
ZD 13-07	0,765	0,018
ZD 13-08	0,751	0,011
ZD 14-07	0,958	0,023
ZD 14-08	0,962	0,028

Steps 1 to 5 have been repeated at least twice for each of the six batches. The Table 3 summarises the obtained results for the kernel particle's diameters. The values in the table are the average per row and batch.

Table 3. Kernel diameters from direct dimensional measurements

	d_k average in μm
ZP 12-07	657,4
ZP 12-08	683,1
ZD 13-07	687,1
ZD 13-08	667,3
ZD 14-07	658,9
ZD 14-08	660,0

The results from steps 6 and 7 are shown in the Table 4. As explained in the step 7, in calculating the coating thickness, S_c, the particle's external diameter measured with the profile projector, D_m, has been used. In that way, the measurement uncertainty from the spatial calibration in the analysis of the x-ray graph is minimised.

Table 4. Relation coating and coating thickness from direct dimensional measurements

	RC	D_m μm	S_c μm
ZP 12-07	0,101	859,0	86,93
ZP 12-08	0,103	878,1	90,47
ZD 13-07	0,043	764,8	33,09
ZD 13-08	0,047	751,3	35,51
ZD 14-07	0,138	957,9	132,42
ZD 14-08	0,143	961,8	137,56

The particle diameter (D_x) measured in the radiograph, can be then obtained using the coating thickness and the kernel diameter as follows:

$$D_x = d_k + 2 \cdot S_c$$

The Table 5 reports the average D_x for the six different groups.

Table 5. Calculated particle diameters from the X-ray, direct measurements

	D_x μm
ZP 12-07	838,4
ZP 12-08	868,6
ZD 13-07	766,9
ZD 13-08	745,7
ZD 14-07	939,4
ZD 14-08	954,5

The following figure shows the comparison between the external diameters obtained with the projector and calculated from the X-ray image; one can notice a good agreement between the two measurements.

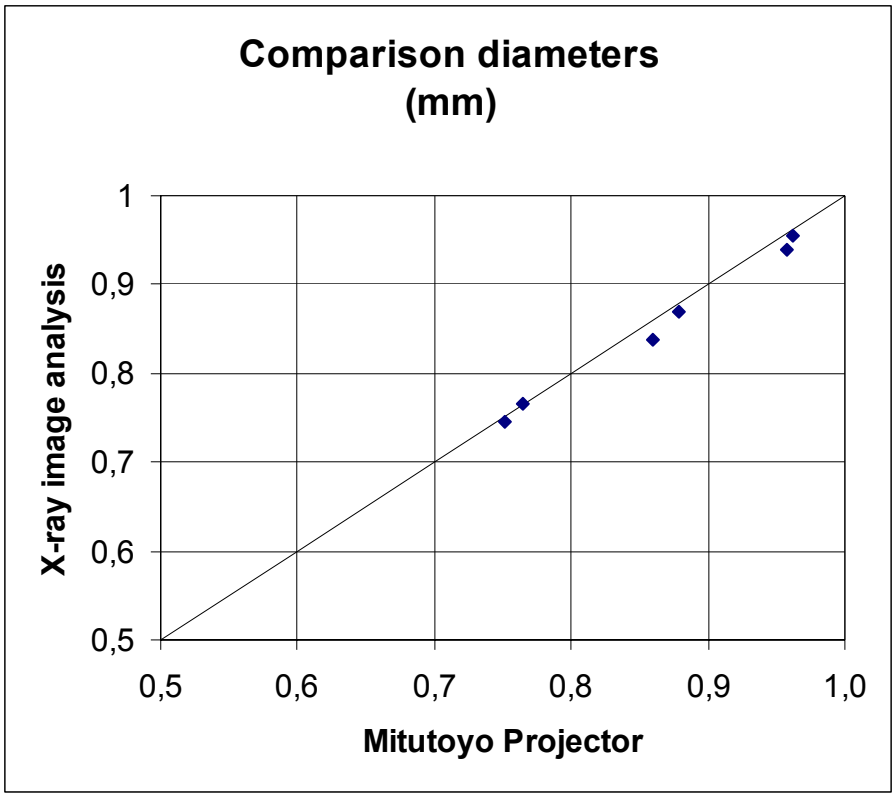


Figure 8. Diameter measured with the profile projector versus calculated from the X-ray

Density profile analysis

Analyses of the optical density have been carried out for each particle of the six different X-ray graphs.

The first step is to calibrate the spatial and the intensity scales. In this case the spatial scale has not been used, and measurements are directly expressed in pixels. Lately pixels are converted into millimetres using the measurements on the calibration wire.

Regarding with the intensity calibration, for each radiograph it has to be established the intensity values representing the "black" and "incident" levels in the image. In our pictures the black level (no rays are passed) is chosen from the identification pieces placed at the left of the X-ray, see for example the Figure 5; and the incident level (the area where there is no material present) is selected from the middle of the background.

After calibration is performed, then objects are automatically identified using the option *Count/Size* of the Image Pro program. Counting the dark objects within our radiograph is particularly helpful to define the "kernels", i.e. their perimeters, radius and roundness, as it has been seen they are not perfectly spherical. The obtained kernels are also used as baselines to draw the line for obtain the density profiles, as it is shown in Figure 9.

In addition the distance from kernel centre to kernel centre has been drawn in order to locate the density profile along the line where particles are in contact, see Figure 9 right image.

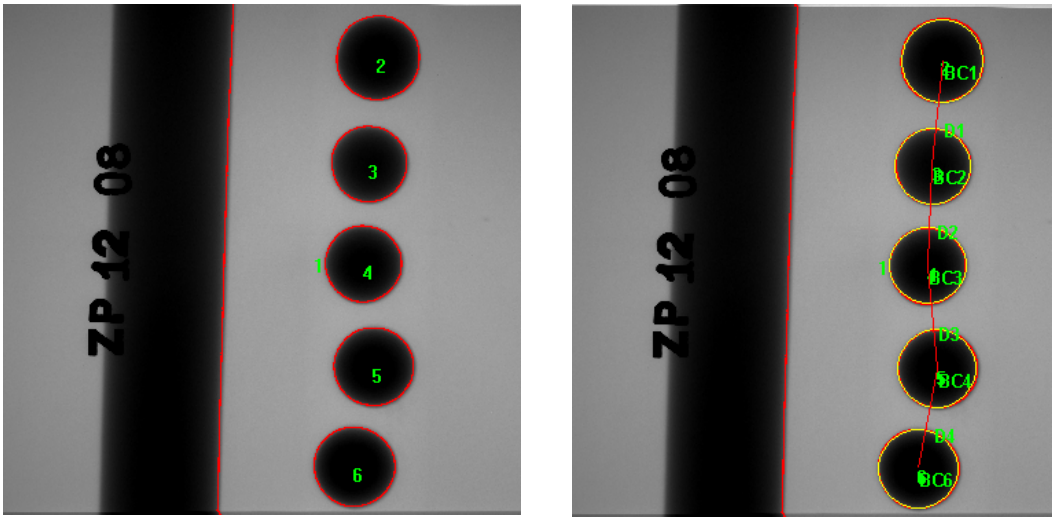


Figure 9. Example of Count/size output from the Image Pro analysis (ZP 12-08)

Then the density profile analyses have been performed on the six X-ray graphs, taking the line profiles between centre-kernel to centre-kernel. A line profile plot is shown in Figure 10, as it can be seen, the higher intensities correspond to the kernels and the lower shelf for intensity (that is the distance between the two kernels) corresponds to the coating material.

Thus the coating thickness $[S_c]_i$, will be the half of the lower shelf dimension. For the Figure 10:

$$[S_c]_{1-2} = \frac{lenght\ lower\ shelf}{2} = 79.35\ pixels$$

Which can be converted to millimetres using the measurement of the calibration wire, for the radiograph under analysis:

$$[S_c]_{1-2} = 0.08797\ mm$$

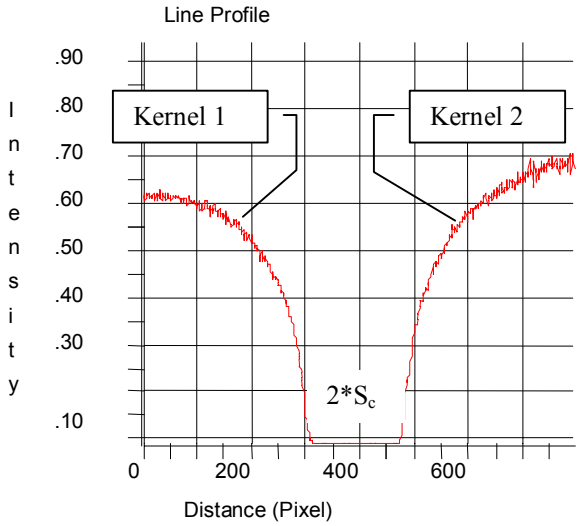


Figure 10. Example of density line profile plot (ZP 12-08, distance between BC1 and BC2)

The Figure 11 to Figure 16 shows the coating thickness determination using line profiles, for the particles of the six batches. The ratio micrometer to pixel (specific for each radiograph) is also reported in the figures.

All line profiles have been obtained according with the procedure explained above. Only a remark has to be done, regarding with the 14-07 set (Figure 15) the intensity calibration has been done on the basis of the grey-level instead of the standard optical density curve. That mean that a filter is applied and this explains the straightness (no oscillations) and sharpness of the line profiles of the Figure 15.

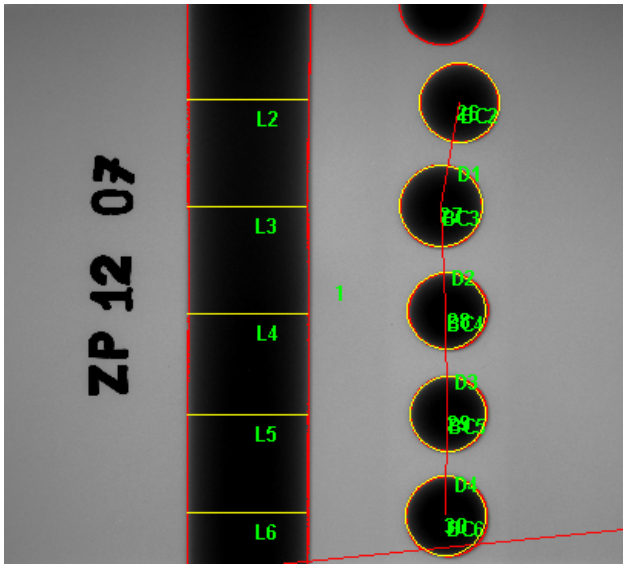
The coating thickness for the different groups (ZP 12-07 to ZD 14-08) can be obtained by:

- Averaging the $[S_c]_i$ measured for each particle of the group. The results are shown in the Table 6

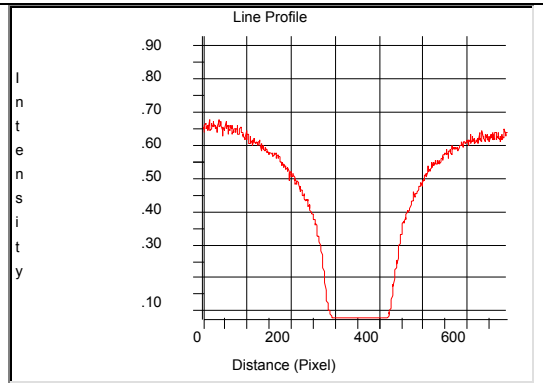
Table 6. Averaged coating thickness from the density profile measurements

	S_c µm
ZP 12-07	87,74
ZP 12-08	86,85
ZD 13-07	30,48
ZD 13-08	30,54
ZD 14-07	122,99
ZD 14-08	132,19

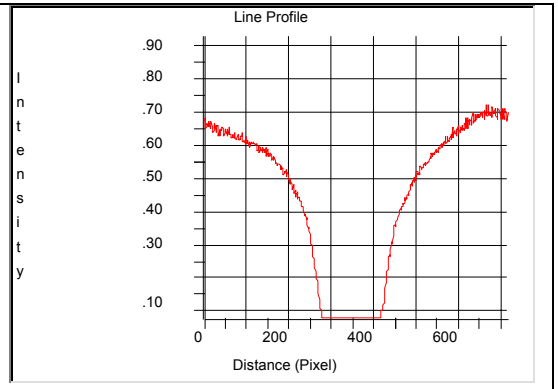
- Or obtain the RC (relation coating) as in the manual measurements, and multiply by the particle diameter measured with the projector. The Table 7 summarises the values for RC and S_c.



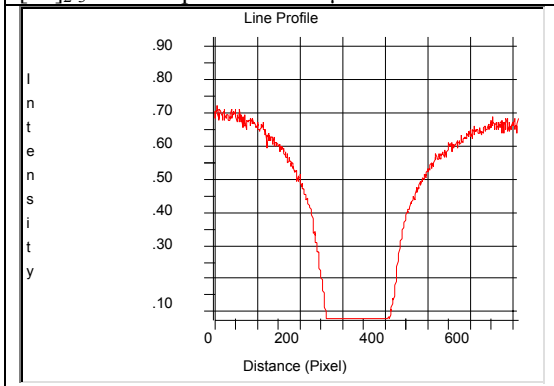
µm/pixel ≈ 1.27



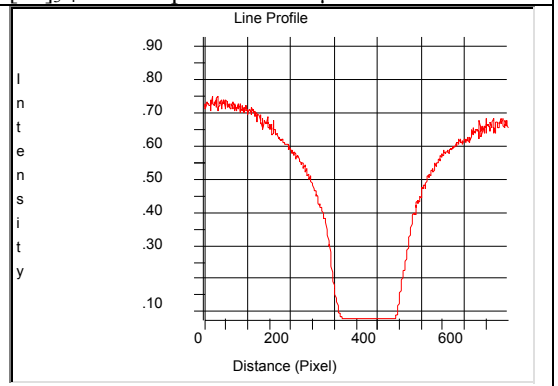
$[Sc]_{2\text{-}3}$ = 69.54 pixel ≅ 88.47 µm



$[Sc]_{3\text{-}4}$ = 68.11 pixel ≅ 86.88 µm

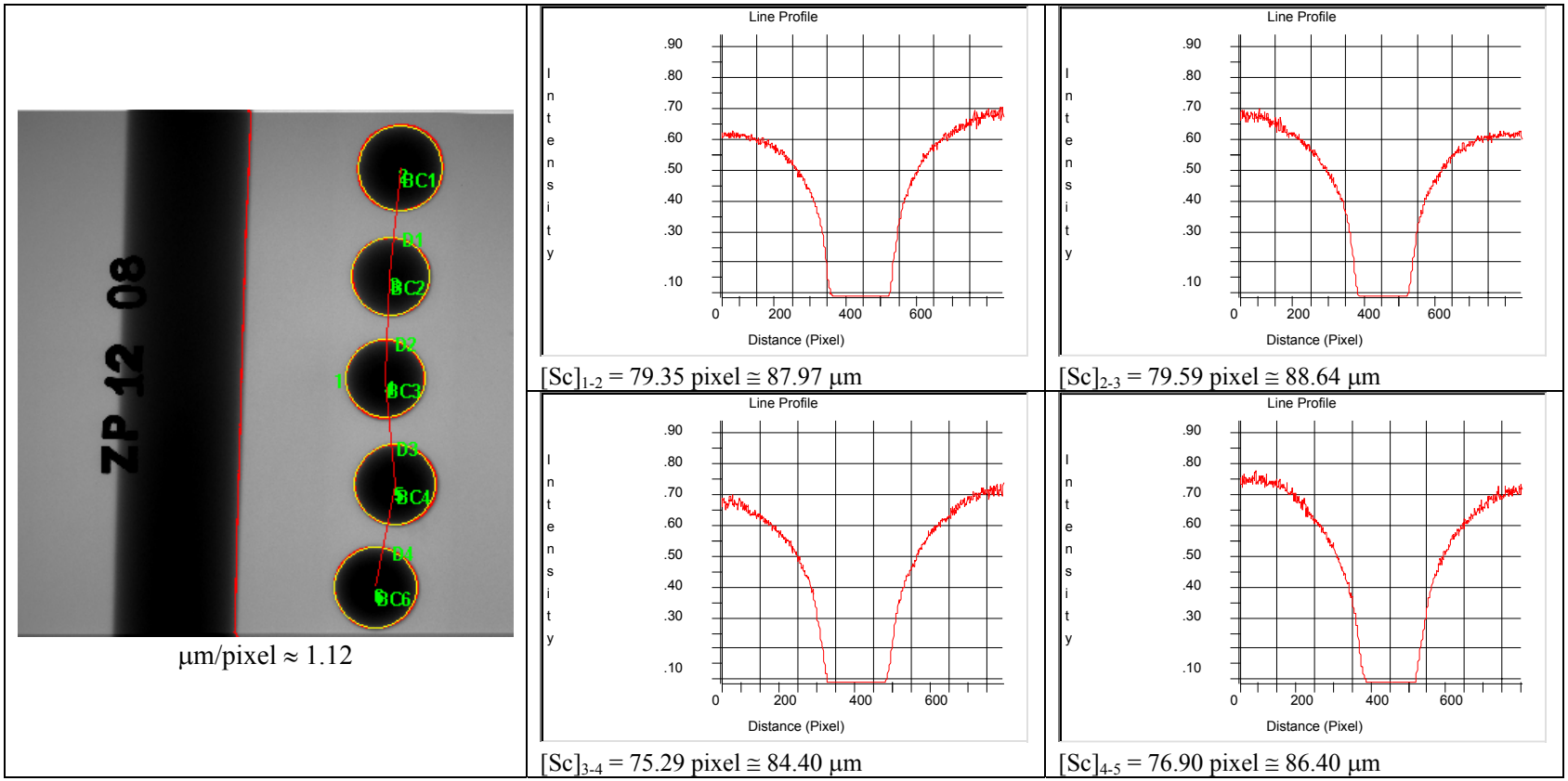


$[Sc]_{4\text{-}5}$ = 70.01 pixel ≅ 89.07 µm



$[Sc]_{5\text{-}6}$ = 68.03 pixel ≅ 86.55 µm

Figure 11. Density line profiles for the set ZP 12-07



µm/pixel ≈ 1.12

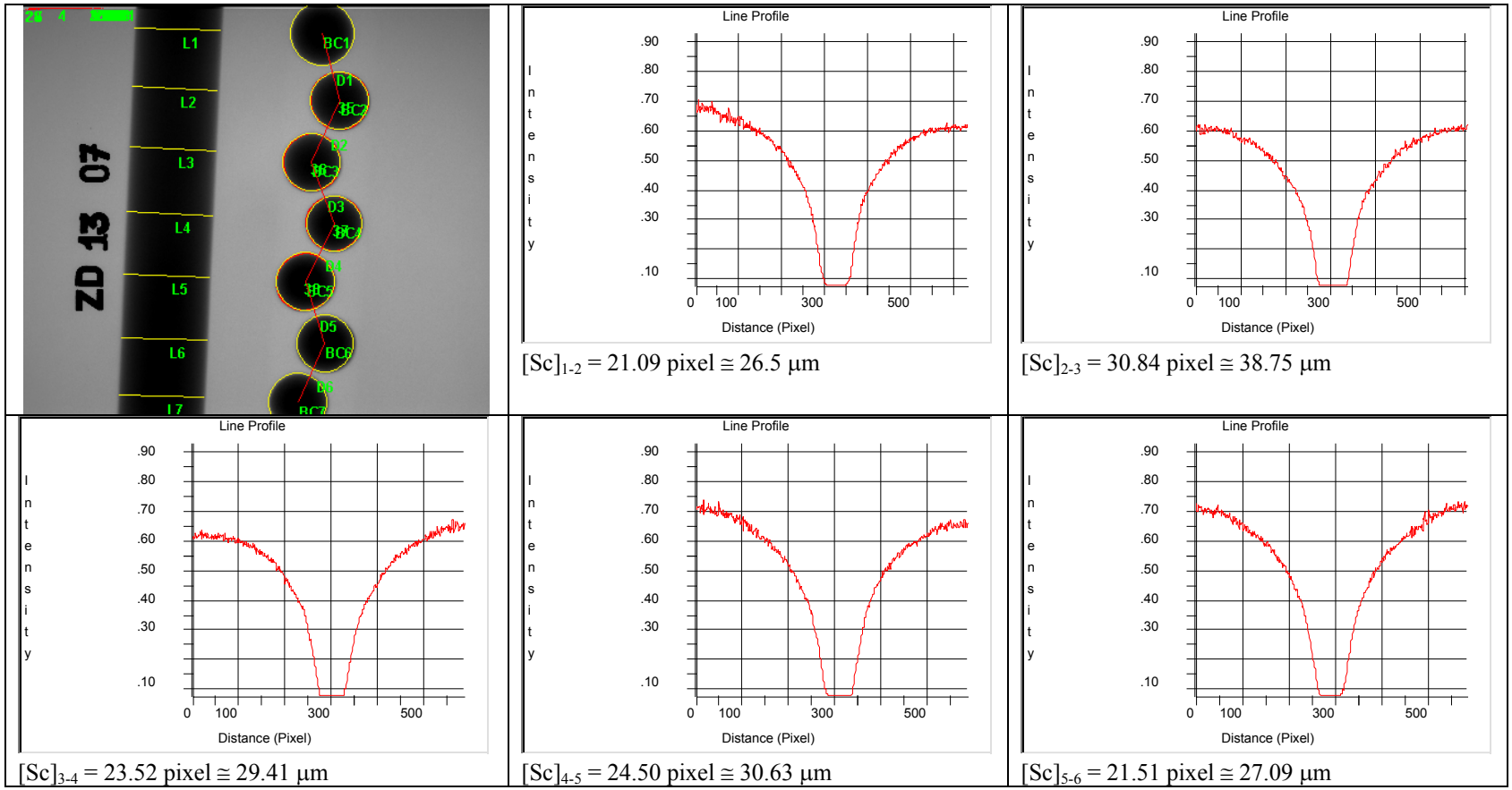
$[Sc]_{1\text{-}2}$ = 79.35 pixel ≅ 87.97 µm

$[Sc]_{2\text{-}3}$ = 79.59 pixel ≅ 88.64 µm

$[Sc]_{3\text{-}4}$ = 75.29 pixel ≅ 84.40 µm

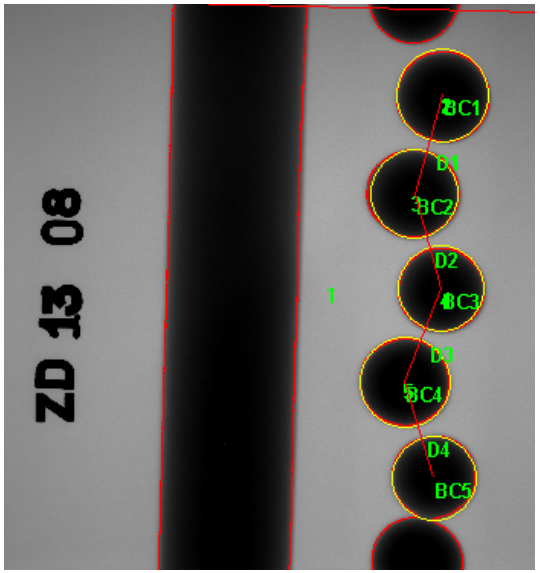
$[Sc]_{4\text{-}5}$ = 76.90 pixel ≅ 86.40 µm

Figure 12. Density line profiles for the set ZP 12-08

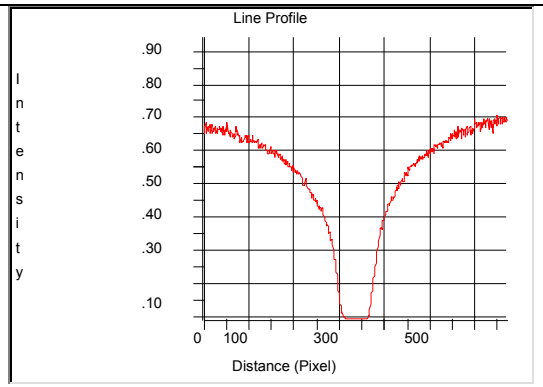


µm/pixel ≈ 1.25

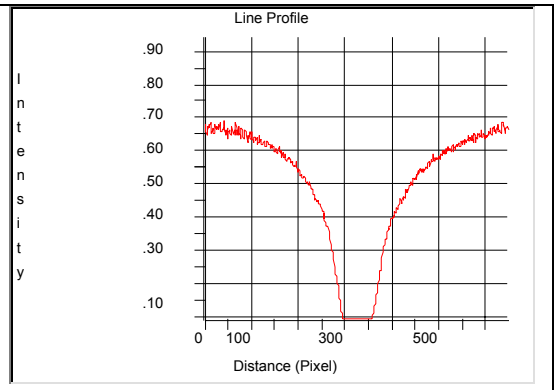
Figure 13. Density line profiles for the set ZD 13-07



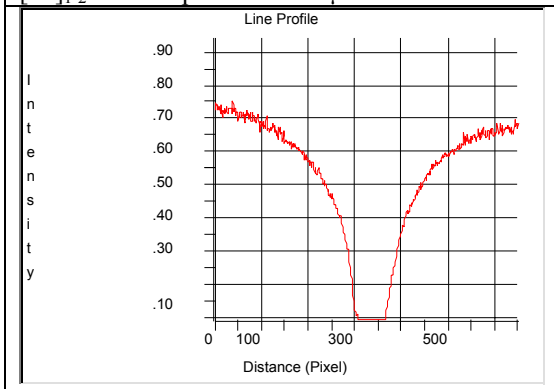
μm/pixel ≈ 1.12



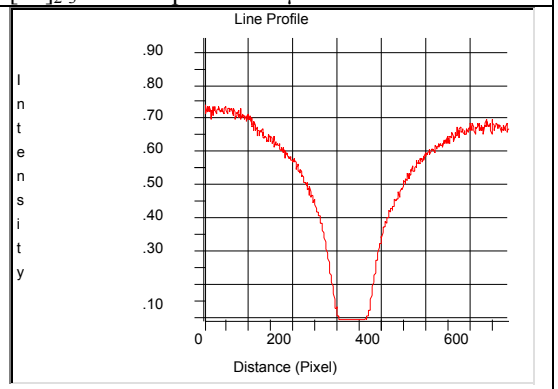
$[Sc]_{1\text{-}2}$ = 24.51 pixel ≅ 27.48 μm



$[Sc]_{2\text{-}3}$ = 28.80 pixel ≅ 32 μm

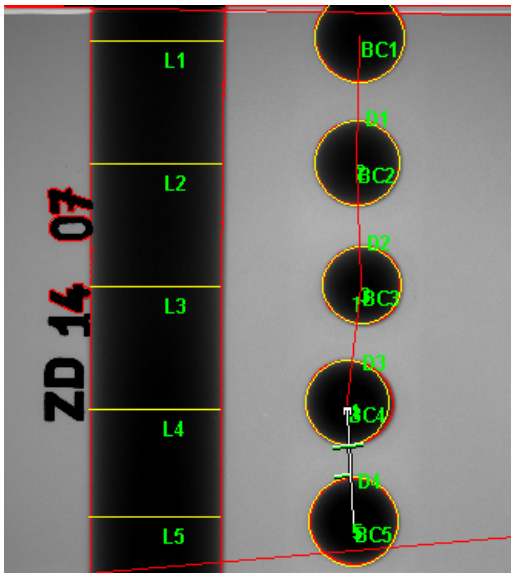


$[Sc]_{3\text{-}4}$ = 27.34 pixel ≅ 30.44 μm

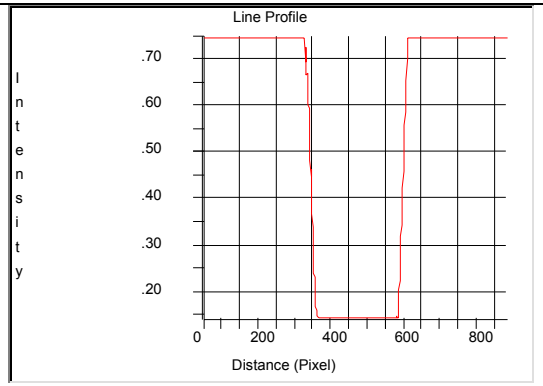


$[Sc]_{4\text{-}5}$ = 28.81 pixel ≅ 32.23 μm

Figure 14. Density line profiles for the set ZD 13-08

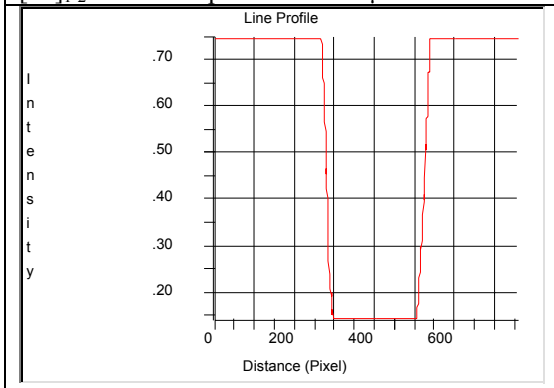


μm/pixel ≈ 1.16

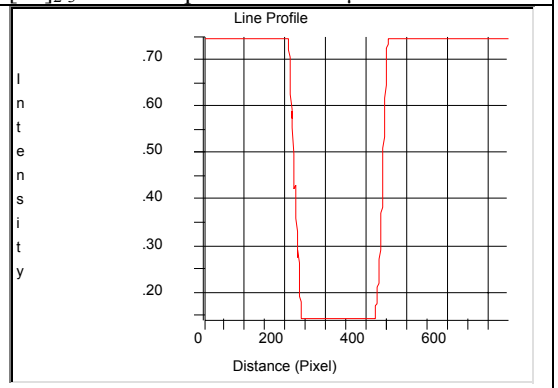


$[Sc]_{1\text{-}2}$ = 109.034 pixel ≅ 125.62 μm

$[Sc]_{2\text{-}3}$ = 119.59 pixel ≅ 140.04 μm

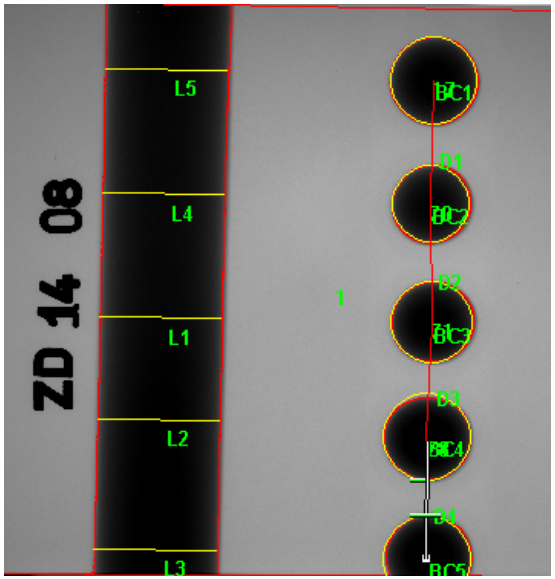


$[Sc]_{3\text{-}4}$ = 104.22 pixel ≅ 121.75 μm

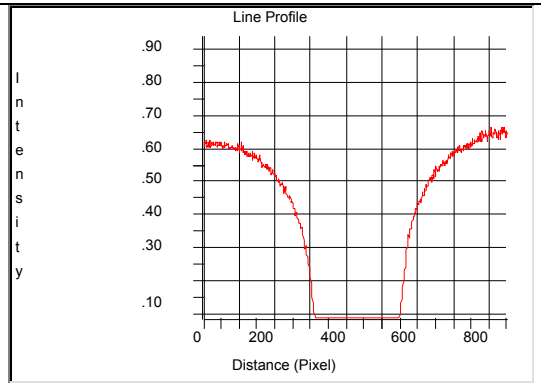


$[Sc]_{4\text{-}5}$ = 90.12 pixel ≅ 104.55 μm

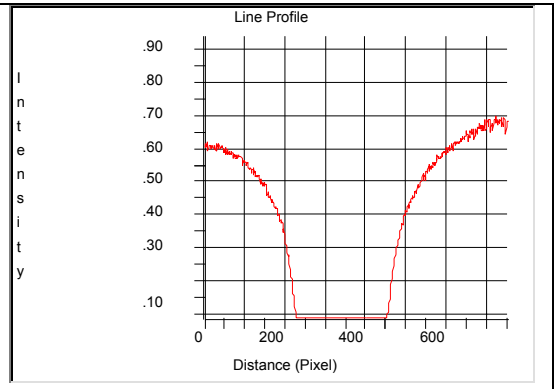
Figure 15. Density line profiles for the set ZD 14-07



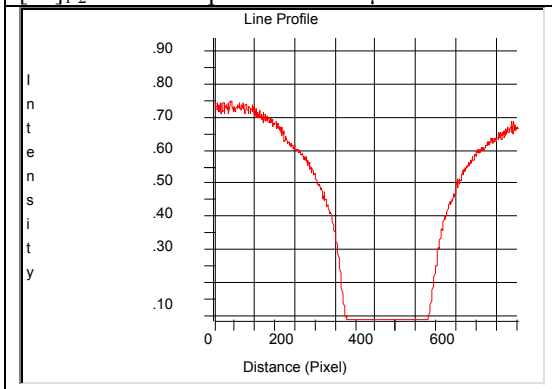
µm/pixel ≈ 1.20



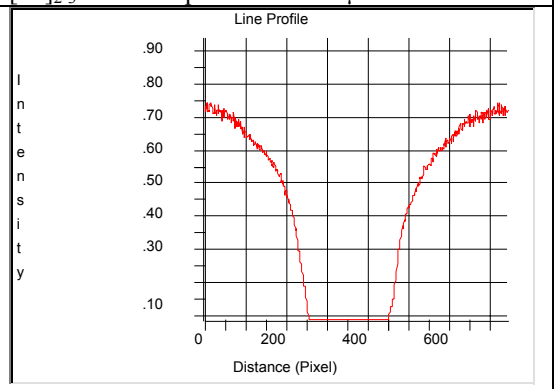
$[Sc]_{1-2}$ = 117.014 pixel ≅ 141.15 µm



$[Sc]_{2-3}$ = 112.02 pixel ≅ 135.13 µm



$[Sc]_{3-4}$ = 105.58 pixel ≅ 126.9 µm



$[Sc]_{4-5}$ = 102.02 pixel ≅ 123.51 µm

Figure 16. Density line profiles for the set ZD 14-08

Table 7. Relation coating and coating thickness from density profile analysis

	RC	D_m μm	S_c μm
ZP 12-07	0,102	859,0	87,29
ZP 12-08	0,104	878,1	91,71
ZD 13-07	0,040	764,8	30,74
ZD 13-08	0,041	751,3	30,93
ZD 14-07	0,134	957,9	128,09
ZD 14-08	0,140	961,8	135,09

As stated before the Image Pro automatically counts and measures relevant objects within the X-ray graphs. In this way kernels have been automatically determined and their radius measured. In the Table 8 the average of the "automatically measured" kernel diameters and their standard deviation can be seen. The relation between maximum and minimum radius for each particle has been also calculated, and the averaged value for each particles set is the *Radius ratio*, which represents a measure of the particles' roundness. As it can be observed, Table 8, particles are not uniformly spherical and this induces an additional error in the coating thickness computation.

Table 8. Kernel and radius ratio from automatic measurements

	d_k μm	σ	Radius ratio	σ RR
ZP 12-07	654,1	0,022	1,037	0,011
ZP 12-08	637,7	0,026	1,033	0,007
ZD 13-07	646,9	0,011	1,044	0,014
ZD 13-08	644,8	0,026	1,049	0,012
ZD 14-07	631,6	0,026	1,042	0,017
ZD 14-08	651,9	0,034	1,047	0,019

DISCUSSION

The coating thickness of the simulated particles has been determined from the image analysis of the X-ray graphs using two methods:

The first method is based on taking direct measurements on the previously calibrated image. The obtained kernel diameters range from 657 to 687 μm. The calculated external diameters are in good agreement with those measured with the projector, with differences within the tolerance obtained the measurements with the projector.

The coating thickness averaged for the three groups (ZP 12, ZD 13 and ZD 14) with different coatings are summarised in the Table 9. As it can be seen the obtained values are quite near from the values provided by the specifications.

Table 9. Direct measurement of the coating thickness

	S_c		
	specification μm	calculated μm	tolerance μm
ZP 12	95	89	3
ZD 13	40	34	2
ZD 14	135	135	5

The systematic error associated to this measurement procedure mainly comes from the error made when defining the perimeters of the different kernels in the image and the dimension of the calibration wire (see Figure 7). In particular, when selecting the contour of the kernel by a circumference the uncertainty σ_k is about the 3% and when determining the calibration wire thickness (used in the spatial calibration) a $\sigma_w \approx 2$ % is obtained. Thus the total error will be:

$$\sigma_c = \sqrt{\sigma_k^2 + \sigma_w^2} \approx 3.6\%$$

To the error associated to the measurement it has to be added the error arising from the digitisation process, which is the spatial offset in the scanning, that is estimated on approximately 1 μm.

Based on this the tolerance in micrometers for the different coating thickness is reported in the Table 9, where it can be seen that the thickness can be determined with an accuracy of 5 μm by using this procedure.

The second method makes use of some tools of the Image Pro Plus program, as the automatic measurement feature and the optical density profile along lines from particle to particle, to obtain the coating thickness. By using this method the measured kernel diameters are relatively smaller than those calculated by the first procedure, see the Table 3 and the Table 8 for comparison. The coating thickness determined by this method (Table 6 and Table 7) is also smaller than the one arising from the direct dimensional measurements.

In this case the systematic error is given by the uncertainty in the distinction made by the program itself when choosing the different grey levels that define the kernel perimeter and by the uncertainty in selecting the "kernel boundaries" in the density profile plots (from which intensity level can be considered that the kernel is finished and there is the coating). Such uncertainty depends on the particle size and roundness ranging from 5 % in the case of the group ZD 14 up to 11 % for the ZD 13 set (see Table 10).

The pixel calibration error is, as in the previous method, in the order of 2 %.

The tolerance for the coating thickness calculations using density profile analysis is stated in the Table 10.

Table 10. Coating thickness determined by density profile analysis

	specification μm	calculated μm	uncertainty %	tolerance μm
ZP 12	95	87	7	6
ZD 13	40	31	11	3
ZD 14	135	128	5	7

In addition to the systematic error there exists random errors due to:

- The fact of that the particles are not perfect spheres and coating thickness are not uniform.
- The analysed image comes from a digitisation of the X-ray film.
- The analysis are carried out inferring from images in a computer screen, this can be quite subjective depending on the visual perception of the person making the analysis, clearness of the image, etc.

CONCLUSION

In the frame of the HTR-F project the JRC/ IE have received an amount of approximately 5000 simulated particles for HTR fuel. Such particles are divided in three different groups as a function of their coating thickness. The scope of the work presented in this report was to assess the radiographic examination as a method for quality control of the coating of HTR fuel particles.

An X-ray evaluation have been performed using an "ad-hoc" particle carrier, which allows fixing the particles in contact between themselves and includes a calibration wire for later image analysis.

The coating thickness have been calculated from the analysis of the digitised radiographs by using the software ImagePro Plus to perform:

- Direct dimensional measurements
- Density profile analysis

In general, the obtained results for the coating thickness are in good agreement with the specification, showing slightly smaller values than those reported in the HTR-F Deliverable 16 “Coating of particles”.

A full analysis of the errors associated with the measurement technique has been performed. By means of dimensional measurements the thickness can be determined with an accuracy of 5 μm. Using the density line profile analysis the coating thickness is calculated with an accuracy of 7 μm.

To obtain more accurately the coating thickness possible improvements could be done within the X-ray technique. It should be desirable to perform new X-ray examination adjusting the magnification and (or) the exposure time, and compare them with these used in this report in order to find the optimal parameters to obtain X-ray graphs best suitable for the analysis with ImagePro Plus software.

Since the analysed image comes from the digitisation (scanning) of the X-ray films, connecting a digital acquisition system to the X-ray device could optimise the process, and in this way one source of random errors will be minimised.

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