

INDC(NDS)-0663 Distr. LP, NE, SK

## **INDC International Nuclear Data Committee**

## Standard Procedures for the IAEA Dust Database: Preparation, Measurements, and Upload

Prepared by

Suk-Ho Hong

National Fusion Research Institute (NFRI), Korea

January 2014

IAEA Nuclear Data Section Vienna International Centre, P.O. Box 100, 1400 Vienna, Austria

Selected INDC documents may be downloaded in electronic form from <u>http://www-nds.iaea.org/publications</u> or sent as an e-mail attachment. Requests for hardcopy or e-mail transmittal should be directed to <u>NDS.Contact-Point@iaea.org</u> or to:

Nuclear Data Section International Atomic Energy Agency Vienna International Centre PO Box 100 1400 Vienna Austria

Printed by the IAEA in Austria

INDC(NDS)-0663 Distr. LP, NE, SK

### Standard Procedures for the IAEA Dust Database: Preparation, Measurements, and Upload

Prepared by

Suk-Ho Hong

National Fusion Research Institute (NFRI), Korea

#### Abstract

An IAEA Coordinated Research Project in the years 2008-2012 was devoted to "Characterization of Size, Composition and Origins of Dust in Fusion Devices." In connection with this CRP a dust database infrastructure was created at the Max Planck Institute for Plasma Physics in Garching, Germany. Aiming at an international standardized database the present report describes standard procedures for the preparation and imaging of dust samples and for the subsequent image processing, analysis and upload into a database.

January 2014

## **TABLE OF CONTENTS**

/
8
8
8
8
9
9
0
3
3
4
5
8
9
9 0
9 0 1
9 0 1 2
9 0 1 2 3
9 0 1 3 3
9 0 1 3 3 4
9 1 2 3 4 5
9 1 2 3 4 5 6
9 1 2 3 4 5 6 7
9 0 1 2 3 4 5 6 7 8
9 0 1 2 3 4 5 6 7 8 9
9 0 1 2 3 4 5 6 7 8 9 0
9 0 1 2 3 4 5 6 7 8 9 0 1

#### **1. Introduction**

In-vessel dust particles are frequently found in present fusion devices, and they are not a great concern: they are not yet harmful for plasma operation or safety. For next step fusion devices such as International Thermonuclear Experimental Reactor (ITER), however, it is expected that the in-vessel dust will become a very important issue. Increased particle and heat flux impinging on the surface leads to much stronger plasma-surface interaction [1]. Dust production and accumulation inside the vacuum vessel may lead to serious safety and operational issues [2] by radioactive tritiated co-deposits of nano- to micrometer size, which is the main source of mobilizable dusts in tokamaks [3]. Melting and splashing of metal PFCs such as Be or W will cause somewhat different problems. Furthermore, amount of dust on hot surfaces in ITER is limited to 6 kg due to a strong chemical reactivity with air and stream (ITER accident scenario) [1]. In addition, some dusts have an impact velocity up to several km/s, which can easily damage the first wall or diagnostics.

For these reasons, it is crucial to monitor in-vessel dusts where they are created, when they are created, and how much dusts are created. The most fundamental questions for the in-vessel dust study are to find the spatial locations of the origins of dust creation and its frequency, dust velocity distribution, and quantitative measurements of amount of dusts created during a run day as well as in a campaign. In order to answer those questions and get quantitative information, dedicated diagnostics for the study have to be selected and utilized. The most common way to identify in-vessel dusts during a plasma shot is the use of CCD images. Since CCD images are obtained using wide angle optics to cover a large in-vessel area, dust creation events (DCEs) will be automatically detected and recorded. Furthermore, these DCEs are not easily detected by line-of-sight diagnostics that look at a tiny plasma volume at a fixed location such as a tomographic bolometry array or a visible spectroscopy system. In a recent publication, "a simple, fast but powerful image processing technique is developed for in-vessel dust research using existing standard visible CCD cameras already installed in fusion devices" [4]. Similar analysis software, with which the location of dust trajectories in 3D position in the vacuum vessel can be identified, is developed and the dust velocity is measured [5]. In order to estimate the amount of dust created in the vacuum vessel two different methods are available. The first is based on a microbalance modified from a capacitive diaphragm gauge [6], and the second is the electrostatic bridge using the short circuit of high voltage/current circuit caused by dust [7].

Other than these topics, another important issue to be studied and recorded is the database on the physical and chemical properties of in-vessel dusts. This type of study will give a comprehensive understanding on the reactivity of dusts. Sizes, shape and chemical composition of dusts have been investigated under the IAEA coordinated research project (CRP) on "Characterization of Size, Composition and Origins of Dust in Fusion Devices" [8]. Main diagnostics in the research are scanning electron microscopy (SEM) and energy dispersive x-ray spectroscopy (EDX). The ASDEX Upgrade (AUG) team has performed a pioneering work on the dust database using automated SEM and EDX measurement that can handle up to several thousand to ten thousand measurements per day [9].

The AUG dust database created by automated system provides better understanding on the properties, dispersion, and statistics of in-vessel dusts. However, in order to create comparable amount of dust database from other sources one has to send samples to IPP-Garching. Therefore, the creation of the database itself is limited by the operation schedule/assigned measurement time of the device at IPP-Garching. For the expansion of the creation, build up, and use of the dust database a standardized procedure for the creation of dust database has to be agreed so that each participant can create his or her own database and have it be compatible with the database created by other participants.

The aim of this document is to describe a standard procedure for the creation of a dust database and to explain the upload strategy. In section 2, the SEM measurements related to the task will be briefly described. In section 3, a standard method for the preparation of dust sample will be given. In section 4, a standard set of parameters and processing methods will be suggested. A standard processing procedure for extracting physical quantities by using ImageJ software will be described in detail in section 5. For beginners, a set of SEM images and their characteristics obtained by ImageJ analysis are given as an example in section 6. In section 7, the database upload strategy will be discussed. Finally, a summary will be given.

#### 2. A brief description on the SEM measurements parameters

Scanning electron microscopy uses a beam of highly energetic (0.1-50 keV) electrons focused on a sample surface. The impact of these electrons causes several interactions including the emission of secondary electrons, backscattered electrons, photons and X-rays, excitation of phonons, and diffraction under specific conditions. The bombardment of the electron beam is scanned in 2D, say in the X-Y plane, and an image for each process can be mapped with a suitable detector. Standard detector for SEM measurements is secondary electron detector, which records topography of the surface under observation with resolution of the order of 1-2 nm and magnification range from 10x to 500,000x. Using appropriate detectors, composition, phase, electrical, optical, thermal, and other properties can be mapped with excellent resolutions.

SEM images are provided by skilled operators. Operation of SEM including basic settings and scanning parameters depends, therefore, on the operator: they have their own preferential settings and ways of measurements. As a result, parameters such as brightness and contrast of images can be different from image to image. There is no problem of "viewing" images for the identification of surface structures, morphology, since images are considered as independent, and are not correlated with each other. However, as a source of a database, the image quality itself is a critical point: the same object measured by different settings would provide different output for the database. For instance, different brightness/contrast can result in different edge structure, which leads to a wrong measurement in length or size of the object. Therefore, standard conditions for the measurement of SEM images need to be recommended and agreed for the creation of dust database.

#### **2.1. Influence of accelerating voltage on image quality**

When electrons from the probe reach the surface of the substrate or the object under study, they start to interact with the atoms on or inside the object. The penetration depth and the area of diffusion depend strongly on the energy of the electrons and the material property of the object. Usually, a good quality image can be obtained with higher accelerating voltage and smaller electron probe. Nevertheless, the use of high accelerating voltage leads to negative effects such as smearing of surface structure, edge effects, high level of charging up, and high probability of sample damage. In addition, finer structure can be obtained by a lower accelerating voltage. The electron beam penetrates deeper and the diffusion area becomes larger with a higher accelerating voltage. These cause unwanted noise signals generated from inside of the sample. These signals cause a reduction of the image contrast and thereby smear fine structures on the surface of the object.

#### 2.2. Influence of probe current

Probe current is strongly related to the diameter of the probe: the larger the probe diameter is, the higher the probe current is. Small probe current results in high resolution and less damage to the sample. However, due to low current, contrast is somewhat poor. At high probe current one obtains better contrast, but it causes smoothing of the surface, poorer resolution and more damage on the sample.

#### 2.3. Influence of working distance and objective aperture

Working distance (WD) can be changed for various measurements. Large WD leads to the greater depth of field and low resolution while small WD results high resolution and smaller depth of field.

Smaller aperture size gives high resolution and greater depth of field. Large aperture size leads to a smaller depth of field and low resolution.

#### 2.4. Range of pixel resolution for dust database

The definition of ITER dusts is "solid particles or debris of size from 10 nm to 100  $\mu$ m". The range of the dust size is quite large, about a factor of 10<sup>4</sup>. This causes a lot of problems in measuring and quantifying in-vessel dusts. In-situ diagnostics that can cover simultaneously such a large range do not exist. Even for ex-situ measurements like SEM, it is a hard job to cover the entire range. Therefore, it is needed to determine a reasonable and good pixel resolution for the SEM measurements.

#### **3.** Preparation of dust samples

In-vessel dusts can be collected by various methods such as vacuum cleaner, sticky carbon tapes, or a silicon wafer. Usually, all of those methods are used simultaneously. For the dust database, a piece of silicon wafer would be the best collection method, and indeed silicon wafer is widely used for dust research in the fusion community as the collection substrate. It is easy to obtain and easy to handle. Due to its well-known properties it is also easy to identify the properties of dusts and to distinguish them from that of silicon wafer.

To prepare dust samples, a bare silicon wafer is cut to a smaller size using a diamond knife. By using dry nitrogen gas puff, residual silicon particles on the surface can be removed. Do not use methyl alcohol to clean the surface. Usually it leaves dot-like stripes on the surface, which are not visible by the naked eye but are clearly seen by SEM.

KSTAR utilizes two different methods to collect in-vessel dusts by using silicon wafers as shown in Fig. 1. The first one is a short term sample, which is placed inside the vacuum vessel by mid-plane manipulator in the morning, then retracted in the evening every day. The second is a long term sample, which is placed at the bottom of a vertical port during the whole campaign, then collected after the machine vent. AUG utilizes specially designed dust collection box for long term sampling of in-vessel dusts as shown in Fig. 2 [9]. A silicon wafer is attached to the bottom of the box.



Fig. 1. Short (left) and long term (right) dust sample collection in KSTAR.



Fig. 2. Long term dust sample collection in ASDEX Upgrade [9].

# 4. SEM & EDX measurements: recommended parameters and measurement procedures

Before the start of the SEM measurement, one of important parameter, pixel resolution (magnification) has to be fixed. Usually, SEM image has a pixel size of 1024×768. Depending on the magnification, corresponding size of a pixel is then determined. For instance, a 5000× magnification with 1024×768 image size is corresponding to 58.5 nm/pixel, i.e. a pixel represent a square of 60 nm × 60 nm. Therefore, this magnification can cover the full range of the ITER dust definition from 60 nm up to several hundred  $\mu$ m. The task described from here will use a magnification of 5000× as the standard magnification.

As we have mentioned before, SEM images are taken by experienced operators. Each operator has own experiences and feelings for the "measurements" of micro-meter size particles. Therefore, different results can come out by operators. In order to quantify what differences would come and what parameters should be used, a series of dust samples were measured by two different, independent operators. The purpose of the task and range of the work are explained to the operators in detail. The operators were told to get reasonable images with which one can process measured SEM images to build up the dust database, i.e. identifying shape, size, and chemical compositions. Approximately 332 SEM images were taken by both operators. The basic parameters used by two operators were summarized in table 1.

Operator	WD (mm)	EHT (kV)	Iprobe (nA)	Contrast	ontrast Brightness	
1	23-25	10	35-143	39.3-54.9	19.5-47.4	209
2	5-9	10	-	28.9-35.5	48.2-51.5	123

Table 1. The basic parameters used by two different operators



Fig. 3. Examples of SEM images from operator 1 (left) and from operator 2 (right).



Fig. 4. statistics of the parameters used by the operator 1 (Contrast and brightness)

The operator 1 has concentrated on the large difference in contrast between dusts and background to enhance the visibility of dusts, which results in good identification of the shape and size of the dusts. He has used a large WD=23-25 mm and EHT=10 kV. Contrast and brightness are in the range of 39.3-54.9 and 19.5-47.4, respectively. As a result, although the shape and the size of individual dust are clearly identified, the surface structure including its fine structure cannot be resolved. The operator 2 has concentrated more on the surface structures of dusts. He has used a WD=5-9 mm and EHT=10 kV. Contrast and brightness are in the range of 28.9-35.5 and 48.2-51.5, respectively.

Fig. 4 shows the statistics of the parameters used by the operator 1. The level of contrast shows a well-defined peak around 45. Cumulative counts indicates that more than 80 % of the measurements have been performed with a contrast under 45. The level of brightness is somewhat broad which has two peaks at 25 and at 35. Images with a brightness 35 show the background slightly, but not that different from that with the brightness 25, since contrast was adjusted as well.

Fig. 5 shows the statistics of the parameters used by the operator 2. Both contrast and brightness are in narrow zones compared with that of the operator 1. The level of contrast is concentrated around 29-33, while the level of brightness shows a sharp peak at around 50. Furthermore, dusts in the SEM images are much clear than that from the operator 1.



Fig. 5. statistics of the parameters used by the operator 2 (Contrast and brightness)

Magnification	WD (mm)	EHT (kV)	Contrast	Brightness
5000	5-9	10	~32±4	50±2

Table 2. Suggested standard set of parameter for dust database measurements

Summarizing the findings from two operators, a standard set of SEM measurement can be determined and suggested as in table 2. In any case, if the settings in table 2 are not suitable for the dust measurements, some variation should be made (operator's own decision).

After the SEM image measurement, chemical composition of individual dust can be measured by EDX. In the case of AUG, this measurement was automated by a software developed at AUG. Since other institutes have no such software, EDX measurements are performed separately by operators. Therefore, there is another task to be done for the dust database: dusts measured by EDX have to be enumerated. Fig. 6 shows an example of this procedure. Each dust measured by EDX are counted and enumerated on a copy of the SEM image to match both measurements for the dust database. Corresponding results from EDX measurement are listed in table 3.



Fig. 6. Original SEM image (left), indices of EDX measurement (right)

	1. <i>C-K</i>	2. <b><i>O-K</i></b>	3. <i>Si-K</i>	4. Fe-L
5. Point 1	6. 5.45	7. 3.43	8. 87.65	9. 3.47
10. <i>Point 2</i>	11. 4.95	12. 4.53	13. 88.00	14. 2.52
15. Point 3	16. 5.98	17. 4.01	18. 85.36	19. 4.65
20. Point 4	21. 8.10	22. 7.50	23. 81.53	24. 2.87

Table 3. Weight % of chemical components found in dusts in Fig. 6.

#### 5. Processing SEM images with ImageJ

ImageJ is a software for the processing of various images captured by microscopy. Basic features of ImageJ are 1) identifying objects, 2) measuring characteristics of objects, and finding statistics on objects: ImageJ provide information on calculated area and pixel value statistics, measure distances and angles. It can create also density histograms and line profile plots. Just like other image processing tools, ImageJ supports standard image processing functions including contrast manipulation, sharpening, smoothing, edge detection and median filtering. For more information can be found at developer's web page [10].

#### 5.1. General remarks on image processing

In general, image processing is characterized by the word "pattern recognition". The "pattern" in an image is recognized by identifying the target object called "foreground" that show differences among "static objects" in sequential frames called "background". In many cases, an image processing procedure starts with the transformation/treatment of pixel information contained in the image by logical filters. This is a mathematical transformation of numbers in an array of, e.g.,  $1024 \times 768$ . From a technical point of view, images are multi-dimensional matrices (or arrays) containing numbers. Displays and monitors display this numbers at their pixel location to reconstruct an image on the screen. Therefore, total number of pixels indicates the size of the array (e.g.  $1024 \times 768$ ) and one set of matrix for each color. Logical filters are, thus mathematical functions for certain mathematical

transformation of given array: Numbers can be added, subtracted, multiplied or divided. Depending on the type of the filters (types of transformation), certain information in the image can be enhanced, or removed. Note that, such transformation can affect negatively, so that unwanted information can be created or certain information can be destroyed.

#### 5.2. Useful Macro: SetScaleFromTiffTag

SEM images are usually saved as TIFF format. This format contains additional information tag on the scale of the image which can be used to measure the size and length of dusts in nm unit. Original ImageJ distribution (version 1.47) doesn't contain such macro. One can download the macro "SetScaleFromTiffTag" from the ImageJ website under the link <u>http://rsbweb.nih.gov/ij/macros/</u>. Installation of the macro is simple: just download the macro into the macro directory under the "macros" directory of ImageJ. To install the macro in the ImageJ, click pull-down menu **Plugins**  $\rightarrow$  **Macros**  $\rightarrow$  **Install**, then select the macro file from the directory. After the installation, a menu "SetScaleFromTiffTag" appears in the Macros menu (see Fig. 7).



Fig. 7. Plugins pull-down menu before (left) and after (right) the installation of the macro "SetScaleFromTiffTag"

To use the macro, open a TIFF image, then select Plugins  $\Box$  Macros  $\Box$  SetScaleFromTiffTag. If the macro runs correctly, it will read the tag and converts the unit from pixel to nm. Fig. 8 shows the image information before and after the use of the macro. The information before the use of the macro, all units are based on pixel numbers, whereas they are converted in nm length scale unit by applying the macro. Note that, the effect of the macro will be lost once user has closed the TIFF image: After opening of a new image, user should rerun the macro to convert the units.



Fig. 8. Image information before (left) and after (right) the use of the macro "SetScaleFromTiffTag"

#### 5.3. Quick start: Working procedure with ImageJ

To process an image, target file has to be opened. Select **File**  $\rightarrow$  **Open**, then select an TIFF image. Once the file is opened, move the mouse point into the image. By pressing and holding left mouse button and move around, a **yellow square** will be created. This square is the working area. Fig. 9 shows an example of the selection of a working area. Note that, not entire dusts in the image are selected in this example.



Fig. 9. An example of the selection of a working area.

After the selection of the working area, open the Threshold menu by selecting **Image**  $\rightarrow$  **Adjust**  $\rightarrow$  **Threshold**. The threshold pop up window is opened and the image will be changed into **red** color with white dusts as shown in Fig. 10. Select **Dark background** in the Threshold pop up window. The image will be then inverted as shown in Fig. 11.



Fig. 10. Opening the threshold pop up window. **Red** background with white dusts.



Fig. 11. Inverted Threshold by selecting "Dark background".

2011040,000 F	🛓 Threshold 📃 🗖 💌
•	
	<ul> <li>↓ 116</li> <li>↓ 255</li> </ul>
	Default
	🔽 Dark background
Norman State Stat	Auto Apply Reset Set

Fig. 12. Adjusting lower and upper level of threshold.

Now, adjust lower and upper level of threshold to fill areas of dusts with red dots as shown in Fig. 12. By changing lower level of the threshold, the areas inside dusts are filled by red dots. As the lower level decreases, a lot of additional red dots will suddenly appear at the background. Find an optimum value of lower level at which the areas inside dusts are filled with minimum "noisy red dots" at background. In order to process, select **Analyze**  $\rightarrow$  **Analyze particles**. A pop up window "Analyze Particles" appears as in Fig. 13. Select **pixel units**, enter size parameter from **5-infinity**, show "**Outlines**". Then Click **OK**.

🛓 Analyze Particles								
Size (nm^2):	5-Infinity							
Pixel	units							
Circularity:	0,00-1,00							
Show:	Outlines 🗸							
🔽 Display rest	ults 🔲 Exclude on edges							
Clear result	s 🔽 Include holes							
🗆 🗆 Summarize	Record starts							
🗆 Add to Mana	ager 🔲 In situ Show							
	OK Cancel Help							

Fig. 13. Pop up window "Analyze Particles".

The size parameter **5** is determined as follows: In order to avoid "noise count" (noise pixel), which might come from contrast or other reasons, 4-neighbor criteria has to be applied which defines the border (edge) of a dust particle: If a pixel is surrounded by 4-neighbors, then it is considered as a part of one particle (pixel connectivity). With this arguments, 4 pixels are chosen to be minimum size (actually 4-neighbor mean 5 pixels in total), and 4 pixels with a resolution of  $1024 \times 768$  are corresponding to a dust size of about ~120 nm.

Now, two different windows will appear (Fig. 14). The first one is the "Outline" figure and the second is the results of the measurement: The outline figure is the figure representing outlines of dusts found inside the working area with particle identity number. In this example, 5 dusts were found. Properties of particles in result pop up window are the summary of parameters selected in **Analyze**  $\rightarrow$  **Set Measurements.** The result can be exported as ASCII text file format by selecting File  $\rightarrow$  **Save as.** Using other software tools such as Origin, or Sigma plot, more detailed statistics can be obtained.



⊈ R	🛓 Results										
File	File Edit Font										
	Label	Area	Mean	StdDev	Mode	Min	Max	X	Y	XM	YM 🔶
1	20110620_02.tif	1727871.446	177.459	38.227	161	107	254	37117.200	8574.829	37164.906	8527
2	20110620_02.tif	41221.585	135.083	13.675	116	116	165	42277.347	7409.281	42278.365	7409
3	20110620_02.tif	3438567.232	203.873	32.509	215	116	254	22059.814	18859.685	22064.616	1885 ≘
4	20110620_02.tif	5887816.419	203.854	29.906	179	116	254	38639.651	24224.258	38605.325	2424
5	20110620_02.tif	4019104.557	192.698	28.447	170	116	253	18283.665	26213.999	18299.167	2623
											+ +

Fig. 14. "Outline" figure, its zoomed in image (upper), and the results of the "Analyze Particles" (lower).

#### 5.4. Matching indices between ImageJ result and EDX

First of all, the file name for the EDX measurement has to be given. As the file name of the SEM images, the file name should be given as the rule: [DEVICE][YEAR][SAMPLE LOCATION][SAMPLE ID][IMAGE ID][EDX].dat. The contents will be index of the particle (see Fig. 6), weight % of the EDX measurements.

index	1. C - K	2. 0 - K	3. Si - K	4. S - K	5. F e- L	6. C u- L	7. B a- L
8. 1	9. 4 7. 3 0	10. 0. 3 7	11. 5 2. 3 3				
12. 2	13. 3. 6 4	14. 2 2. 8 4	15. 6. 4 3	16. 0. 8 6	17. 5 7. 6 4	18. 6. 0 3	19. 2. 5 6

Table 4. EDX file contents for the dust database.

#### 6. An example of use of database: an exercise

Sample SEM images are provided with this document for an exercise. Sample dusts collected from KSTAR are evaluated by using the standard procedure suggested in this report as an example: Short term dust samples were collected by midplane manipulator as shown in Fig. 1 (one silicon wafer per day). 20 images were taken and analyzed. Each image was separately treated by hand using ImageJ. Note that, the file name has been created by a certain rule as follows: [DEVICE][YEAR][SAMPLE LOCATION][SAMPLE ID][IMAGE ID]. In this example, [DEVICE]=KSTAR, [YEAR]=2011, [SAMPLE LOCATION]=D-port, [SAMPLE ID]=1 or 2, [IMAGE ID (Areal ID)]=1 to 10.

Open each image in ImageJ, and perform the analysis. After the analysis, more than 400 dusts will be found in total. Save each result of the measurements from individual SEM image as ASCII files and read them in Origin or Sigma plot to extract meaningful statistics. In the example, 467 dusts were found. The results were processed further by Origin.



Fig. 15. Statistics of occupied area in  $\mu m^2$  unit.



Fig. 16. Statistics of circularity of dusts.



Fig. 17. Statistics of roundness of dusts.



Fig. 18. Statistics of elongation of dusts.

Fig. 15 shows the count of occupied area in  $\mu m^2$  unit. This parameter shows effective size of dust particles collected. Not that, we cannot calculate the volume of a dust using this effective size, since we have no information on height of the dust. About half of the dusts (231 dusts) found in the exercise were smaller than 0.05  $\mu m^2$  which is equivalent to ~ 250 nm in diameter. There were some larger size dusts, more than 10  $\mu m^2$ , but the numbers are very small.

To describe shape of the particles, three shape descriptors can be examined: circularity, roundness, and elongation. Circularity is defined as  $4\pi^*$ area/perimeter<sup>2</sup>, where perimeter is the length of the outside boundary of the dust. Circularity has a value between 0 and 1. A value of 1.0 represents a perfect circle while it indicates elongated shape as the value approaches to 0.0. Not that the validity of the circularity breaks down with very tiny dust particles: As the size (occupied area) of the dust becomes smaller, the shape of the dust cannot be described correctly. This lead to an artificial effect on the interpretation of the circularity. Roundness is defined as  $4^*$ area/( $\pi^*$ major axis<sup>2</sup>), or the inverse of the aspect ratio (major axis/minor axis). This shape descriptor tell us how round is the edge of the dust. Elongation is the ratio between longest to shortest distance vectors from the object's centroid to its boundaries.

Fig. 16, 17, and 18 show the statistics on shape descriptors. From the circularity, a large portion of dusts have high circularity of around 0.8. Roundness of 0.5, and elongation of 0.7.

#### 7. Upload strategy

Creating dust database by using the procedure prepared by AUG dust database starts from the uploading individual SEM images to the server. Each SEM image is then analyzed by ImageJ in the remote server, and saved there. It is somewhat time consuming task since the analysis is strongly

dependent on the speed of network connection. Once the session is lost during the analysis, the entire analysis should be repeated. In order to overcome such difficulty, SEM images should be analyzed locally by each end user and then files containing analyzed information should be uploaded.

Several files have to be uploaded: 1) Original SEM image, 2) Outlined image, 3) EDX indexed image, 4) ImageJ output file, 5) EDX file, 6) Other diagnostic files. Therefore, it is important to name the files in a clever way to identify each file and its contents. As we have used previously, files should be named by following rules.

[DEVICE][YEAR][SAMPLE LOCATION][SAMPLE ID][IMAGE ID][DIAGNOSTIC ID]

- 20. [DEVICE] indicates, from which machine the sample is originally come. For instance, [DEVICE]=KSTAR
- 21. [YEAR] indicates the year when the dust sample is collected. Note that, there could be more than one campaign in a year. However, there is one campaign per year in most of cases, year indicates also the campaign.
- 22. [SAMPLE LOCATION] is the geometric place in the vacuum vessel, where the silicon wafers were placed. For instance, [SAMPLE LOCATION]=D-port.
- 23. [SAMPLE ID] indicates the identification of the specific sample at the [SAMPLE LOCATION], since there would be more than one silicon wafers for the sampling.
- 24. [IMAGE ID (Areal ID)] reveals the areal point where the SEM measurement is made. In a sample, user can divide the sample area to identify where the SEM measurements are made.
- 25. [DIAGNOSTIC ID] is needed for the identification of the diagnostic data file.

#### 8. Summary and conclusion

In this document, we have introduced a standard procedure for the building up IAEA dust database from non-automated SEM and EDX measurements. SEM operators are recommended to use the suggested standard magnification, WD, EHT, contrast, and brightness. By using ImageJ and given procedure, the SEM images can be analyzed. Uploading analyzed database files created at local site to the remote server at IAEA, other users can use the dust database. Note that, upload function is not ready for the use, yet. This will be finished soon.

#### References

[1] S. Rosanvallon, F. Onofri, P. Delaporte, CFP/NTT-2007.030, "Assessment of in-vessel dust measurement and removal techniques for ITER", TW6-TSS-SEA5.1 Delivrable 5 - Final Report

[2] "Generic Site Safety Report – Volume III – Radiological and Energy source terms – July 2004", G 84 RI 3 R0.2.

[3] "Generic Site Safety Report – Volume VIII – Ultimate safety margins – July 2004", G 84 RI 7 R0.2.

- [4] S.-H. Hong, C. Grisolia and P. M. Garbet, Plasma Phys. Control. Fusion 51 (2009) 075013
- [5] Y.U.Nam, Review of scientific instrument 81 (2009) 093505
- [6] G. Counsell and C. Wu, Carbon Workshop (Hohenkammer Castle, Germany, Sept, 2000).
- [7] C. Skinner et al., Rev Sci Instrum. 81 (2010) 10E102.
- [8] <u>http://www-amdis.iaea.org/CRP/Dust/</u>
- [9] <u>http://www-amdis.iaea.org/CRP/Dust/Presentations3/Dust3-Rohde-IPP-2011-11-30.pdf</u>
- [10] http://rsbweb.nih.gov/ij/

## **Appendix: Examples**

## Example #1



Image information

Image Name: Base(2) Accelerating Voltage: 10.0 kV Magnification: 5000

#### **EDX** measurement



Weight %

	С-К	O-K	Si-K	S-K	Fe-L	Cu-L	Ba-L
Base(2)_pt1	47.30	0.37	52.33				
Base(2)_pt2	3.64	22.84	6.43	0.86	57.64	6.03	2.56

	С-К	<i>O-K</i>	Si-K	S-K	Fe-L	Cu-L	Ba-L
Base(2)_pt1	67.62	0.40	31.99				
Base(2)_pt2	9.67	45.58	7.31	0.86	32.96	3.03	0.59



Image information
-------------------

Image Name: Base(4)

Accelerating Voltage: 10.0 kV

Magnification: 5000





Weight %

	C-K	<i>0-K</i>	Si-K	Fe-L		C-K	<i>0-K</i>	Si-K	Fe-L
Base(4)_pt1	5.45	3.43	87.65	3.47	Base(4)_pt1	11.78	5.57	81.03	1.62
Base(4)_pt2	4.95	4.53	88.00	2.52	Base(4)_pt2	10.63	7.32	80.89	1.17
Base(4)_pt3	5.98	4.01	85.36	4.65	Base(4)_pt3	12.86	6.47	78.51	2.15
Base(4)_pt4	8.10	7.50	81.53	2.87	Base(4)_pt4	16.46	11.45	70.84	1.25



Image Information Image Name: Base(7) Accelerating Voltage: 10.0 kV Magnification: 5000

**EDX measurement** 



Weight %

	C-K	<i>0-K</i>	Si-K	Cr-K	Fe-L
Base(7)_pt1	8.01	4.78	83.69		3.52
Base(7)_pt2	18.33	8.97	61.64	2.02	9.05

	C-K	<i>0-K</i>	Si-K	Cr-K	Fe-L
Base(7)_pt1	16.64	7.45	74.33		1.57
Base(7)_pt2	34.05	12.50	48.97	0.87	3.62







Weight %

	С-К	<i>0-K</i>	Si-K	Fe-L
Base(8)_pt1	4.59	4.36	86.71	4.35
Base(8)_pt2	5.22	4.33	86.12	4.33
Base(8)_pt3	5.64	4.85	81.75	7.76

	С-К	0-K	Si-K	Fe-L
Base(8)_pt1	10.00	7.13	80.83	2.04
Base(8)_pt2	11.30	7.03	79.66	2.01
Base(8)_pt3	12.28	7.93	76.15	3.64







Weight %

	С-К	0-K	Si-K	Fe-L
Base(9)_pt1	8.22	5.11	82.74	3.93
Base(9)_pt2	6.73	6.55	81.28	5.44

	C-K	<i>0-K</i>	Si-K	Fe-L
Base(9)_pt1	17.02	7.94	73.29	1.75
Base(9)_pt2	14.14	10.34	73.06	2.46







Weight %

	С-К	<i>0-K</i>	Si-K	Fe-L
Base(16)_pt1	6.71	8.20	78.25	6.84
Base(16)_pt2	3.04	4.58	88.76	3.62
Base(16)_pt3	8.66	7.85	76.60	6.89

	С-К	<i>0-K</i>	Si-K	Fe-L
Base(16)_pt1	14.04	12.88	70.00	3.08
Base(16)_pt2	6.71	7.60	83.96	1.72
Base(16)_pt3	17.75	12.08	67.14	3.04



Image in	nformation
----------	------------

Image Name: Base(18)

Accelerating Voltage: 10.0 kV

Magnification: 5000





Weight %

	C-K	<i>O-K</i>	Na-K	Si-K	Fe-L
Base(18)_pt1	59.09	13.99	0.89	13.57	12.47
Base(18)_pt2	4.81	2.75		88.16	4.28
Base(18)_pt3	6.82	4.19		84.51	4.48

	C-K	<i>0-K</i>	Na-K	Si-K	Fe-L
Base(18)_pt1	75.23	13.37	0.59	7.39	3.41
Base(18)_pt2	10.58	4.53		82.86	2.02
Base(18)_pt3	14.49	6.68		76.78	2.05







Weight %

	С-К	<i>0-K</i>	Si-K	Fe-L
Base(23)_pt1	33.87	4.26	59.76	2.10
Base(23)_pt2	26.75	3.40	67.01	2.85

	C-K	<i>0-K</i>	Si-K	Fe-L
Base(23)_pt1	53.69	5.08	40.52	0.72
Base(23)_pt2	45.67	4.36	48.93	1.05







Weight %

	С-К	<i>N-K</i>	0-K	Si-K	Fe-L
Base(26)_pt1	48.29		2.02	48.43	1.27
Base(26)_pt2	16.83	2.29	3.65	75.30	1.94

	C-K	<i>N-K</i>	0-K	Si-K	Fe-L
Base(26)_pt1	68.22		2.14	29.26	0.39
Base(26)_pt2	31.08	3.62	5.06	59.47	0.77



Image Name: Base(29)

Accelerating Voltage: 10.0 kV

Magnification: 5000





Weight %

	C-K	<i>0-K</i>	Si-K	Fe-L
Base(29)_pt1	29.74	2.38	66.13	1.75
Base(29)_pt2	39.80	6.07	51.48	2.64
Base(29)_pt3	4.66		95.34	

	C-K	0-K	Si-K	Fe-L
Base(29)_pt1	49.42	2.97	46.99	0.62
Base(29)_pt2	59.45	6.81	32.89	0.85
Base(29)_pt3	10.26		89.74	

Nuclear Data Section International Atomic Energy Agency Vienna International Centre, P.O. box 100 A-1400 Vienna, Austria E-mail: nds.contact-point@iaea.org Fax: (43-1) 26007 Telephone: (43-1) 2600 21725 Web: http://www-nds.iaea.org