PARTICLE SIZE DISTRIBUTION OF DUST COLLECTED FROM ALCATOR C-MOD

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ABSTRACT

There are important safety issues associated with tokamak dust, accumulated primarily from sputtering and disruptions. The dust may contain tritium, it may be activated, chemically toxic, and chemically reactive. The purpose of this paper is to present results from analyses of particulate collected from the Alcator C-MOD tokamak located at Massachusetts Institute of Technology (MIT) in Cambridge, Massachusetts. The sample obtained from C-MOD was not originally intended for examination outside of MIT. The sample was collected with the intent of performing only a composition analysis. However, MIT provided the INEEL with this sample for particle analysis. The sample was collected by vacuuming a section of the machine (covering approximately 1/3 of the machine surface) with a coarse fiber filter as the collection surface. The sample was then analyzed using an optical microscope, SEM microscope, Microtrac FRA particle size analyzer. The data fit a log-normal distribution. The count median diameter (CMD) of the samples ranged from 0.3 μm to 1.1 μm with geometric standard deviations (GSD) ranging from 2.8 to 5.2 and a mass median diameter (MMD) ranging from 7.22 to 176 μm .

I. INTRODUCTION

This study provides information on the dust size distribution and composition produced during operation of the fusion tokamak reactor Alcator C-MOD. The Fusion Safety Program is primarily interested in dust products in tokamaks because of the potential for redistribution of the dust during both maintenance operations and during postulated accidents and the potential for chemical reactions in oxidizing

environments during accident scenarios.¹ C-MOD dust is relatively unique because C-MOD has a molybdenum first wall. This is in contrast to previous studies of dust from experimental machines with graphite first walls, i.e. General Atomic's DIII-D² and Princeton Plasma Physics Laboratory's TFTR.³

Although the C-MOD sample was not collected for the purpose of particle size distribution analysis, it still provides valuable preliminary data and influenced further sampling and analysis procedures used at C-MOD and other tokamaks. Section II, Sample Preparation, provides a description of the processing steps performed on the dust sample prior to analysis. Section III, Optical Analysis, describes the C-MOD analysis methodology. The results of analysis completed using the Scanning Electron Microscope (SEM) are presented in Section IV, SEM Photographs and Analysis. The results of analysis completed using the Microtrac FRA volumetric particle size analysis instrument are presented in Section V, Microtrac Analysis. Section VI contains the conclusions.

II. SAMPLE PREPARATION

C-MOD personnel used a commercial vacuum cleaner fitted with a coarse fiber 2 μm filter to collect the tokamak dust. The entire surface of the plasma facing wall was vacuumed with this apparatus. Very small particulate on the surfaces would have passed through the filter and avoided collection, or become embedded in the fibers and were not available for analysis.

The sample received from C-MOD personnel for examination contained a variety of materials, including hair, paper, and other foreign materials. This dust was sieved with a 1.680 mm sieve and a 250 μm sieve to

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remove unwanted materials, i.e., the hair, paper, and other foreign materials. The sieved dust was divided into three samples, labeled C-MOD 1-3, 1-4, and 1-5, then small amounts from each were vacuumed onto 0.02 μm filters for optical analysis. The remaining samples were placed in vials for surface area analysis using BET (a gas adsorption method).^{4,5} Following BET analysis, dust on the sides and bottom of the sample vials was removed and placed on SEM mounting stubs for further analysis. The bag that the sample was placed in for shipping to the INEEL was washed with deionized water and the mixture saved for Microtrac FRA analysis. This liquid sample was then filtered through a 0.02 μm filter for optical analysis. Sample descriptions are provided in Table 1. The optical analysis completed on samples 1-3, 1-4, and 1-5 represents the best estimate of the C-MOD particle size distribution from the sample collected. This is because it required the least amount of processing prior to analysis and contains the most particulate.

Table 1. Description of Sample Preparation.

Sample	Description
C-MOD 1-3	These samples came from the 0.02 μm filters used during vacuuming after the sample was sieved.
C-MOD 1-4	
C-MOD 1-5	
C-MOD SEM Bottom Smpl 1-12 Side Smpl 1-13	The dust that passed through the sieves was placed in a tube or vial for analysis by BET. After the BET analysis was performed, samples from the side and bottom of the BET tube were taken and examined using SEM.
C-MOD 1-10 a & b	Following washing of the shipping bag, the captured liquid was analyzed in the Microtrac FRA instrument (sample 1-10a). The liquid was then filtered through a 0.02 μm filter for optical analysis (samples 1-10b).
C-MOD 1-11 a & b	These samples were washed off the BET tube.

III. OPTICAL ANALYSIS

The optical microscope analysis samples were analyzed according to the methods outlined in Carmack et al. (1997)² and Carmack et al. (1998).³ Photographs were taken at 50x, 100x, 200x, 500x and 1000x in four

different areas on each sample filter. Then each image was analyzed using NIH-Image, an image analysis program written by the National Institute of Health.⁶ Examples of the photographs used in the NIH-Image analysis are shown in Figures 1 and 2. The images shown in Figures 1 and 2 are taken from analysis of sample 1-4. The photographs from optical analysis of samples 1-3, 1-5, 1-10b, and 1-11b are visually similar to Figure 1.

200x

Figure 1. Photograph of C-MOD dust sample 1-4 at 200 times magnification.

1000x

Figure 2. Photograph of C-MOD dust sample 1-4 at 1000 times magnification.

These images undergo analysis using NIH-Image so individual cumulative distributions can be constructed. Before creation of these distributions, a Kruskal-Wallis statistical test is performed to ensure that the data from different photographs represents the same underlying distribution. If data from one or more photographs does not pass the test with the other sets, the data are not used in the analysis. An example distribution created from a

passing data set is shown in Figure 3. The table in Figure 3 shows the magnification at which the photographs were taken. The $D_{15\%}$ represents the diameter at which 15 percent of the particles (by count) are smaller and 80 percent are larger. The $D_{84\%}$ represents a value at which 84 percent of the particles are smaller than, the $D_{50\%}$ is the count median diameter value, and the R^2 is the least squares fit correlation coefficient for a straight line.

Figure 3. Individual magnification cumulative distributions for C-MOD 1-10b.

As described in Carmack et al. (1997),² the data from each magnification are combined (using a scaling factor based on the magnification at which the data were acquired) to build an overall distribution for each sample. Seventy-two total photographs were used to create the cumulative distributions for the five samples. Figures 4 and 5 show the final distributions constructed for samples 1-5 and 1-11b from the data obtained in this analysis. A log normal distribution is commonly used in particle size analysis because most industrial aerosols tend to be lognormal in distribution. When the results are plotted on a semi-log scale the resulting curve represents a straight line.³ Each of the log plots in this report use a straight line to represent the fit to the particle size distribution. Ninety five percent confidence intervals were calculated and are shown in Figures 4 and 5 as dashed lines. The upper confidence interval is labeled +95% Confidence, the lower confidence interval is labeled -95% Confidence, and the data is labeled Data in each of the straight line fit graphs. Figures 4 and 5 show somewhat larger confidence bands at smaller particle sizes. This is

due to the loss of small particles during the collection procedure. Table 2 shows the results of all of the samples analyzed. The CMD, GSD, and MMD are all shown in Table 2. The MMD is the mid-point of mass for the range of particles in that sample.

Figure 4. Cumulative log probability distribution generated with 95% confidence intervals for C-MOD 1-5.

Figure 5. Cumulative log probability distribution generated with 95% confidence intervals for C-MOD 1-11b.

Table 2. Final results of the analysis.

Sample Number	CMD (μm)	GSD	Calculated MMD (microns)
C-MOD 1-3	0.7	3.5	87.0
C-MOD 1-4	0.6	3.5	67.7
C-MOD 1-5	1.1	3.6	176
C-MOD 1-10b	0.3	2.9	7.22
C-MOD 1-11b	0.5	2.8	15.0

It is important to note that samples 1-10b and 1-11b may not be entirely representative of C-MOD dust because of the manner in which each was processed for analysis. Both of the samples were taken from the sides of their containers after the sample dust had been removed: either the bag used for shipping for sample 1-10b or from the BET tube analysis sample 1-11b. This particulate was more consistent with a residue of particulate left on these surfaces rather than the particulate removed from the original sample. Samples 1-10b and 1-11b represent a smaller diameter subset of the particulate represented in Samples 1-3, 1-4, and 1-5.

IV. SEM PHOTOGRAPHS AND COMPOSITION ANALYSIS

Energy dispersion spectroscopy (EDS) and Induction Couple Plasma Mass Spectroscopy (ICP-MS) were used for composition analysis of the C-MOD dust samples used in BET analysis. The SEM analyses presented in this report should not be used for detailed particle size results because they do not represent the entire distribution. Figures 6 and 7 show examples of two of the SEM photographs. ICP-MS indicated that 80 percent of the mass contained in the sample was boron. The samples analyzed contained different elements including iron, chromium, silicon oxide, oxide, calcium, and magnesium. The particle labeled Mo in figure 6 is molybdenum.

V. MICROTRAC ANALYSIS

Four samples of the C-MOD dust were analyzed for particle volume in the Microtrac FRA particle size analyzer. The Microtrac machine uses a laser to record the volume of the particles while they are suspended in a water solution. The resulting cumulative distribution for sample 1-11a is shown in Figure 8. The y-axis on this figure used the $\log D_v$ instead of the $\log D_p$. The Microtrac analysis uses a volume based diameter, D_v , while the other method uses a count based diameter, D_p . In the figure the number of runs performed for that sample is indicated by lettering them in order. Multiple runs (typically 2 to 4) were performed to ensure consistent results and ensure that the sample was not skewed by bubbles floating in the analysis volume. Table 3 shows the CMD, GSD, and MMD for both sample 1-10a and 1-11a.

Figure 6. SEM C-MOD photo of particles from sample 1-13 at 3,000x magnification.

Figure 7. SEM C-MOD photo of particles from sample 1-12 at 5,000x magnification.

Figure 8. Cumulative log probability distribution generated for the C-MOD sample of particles washed off the bag surface.

Table 3. Final results of the Microtrac analysis.

Sample Number	GSD	MMD (microns)
C-MOD 1-10a	3.07 - 3.47	8.88 - 9.94
C-MOD 1-11a	2.90 - 2.99	22.5 - 24.7

VI. CONCLUSIONS AND FUTURE WORK

The results most representative of C-MOD dust obtained during this analysis effort were obtained from the optical analysis of samples 1-3, 1-4, and 1-5. These samples contained the largest amount of particulate removed from the sample bag. All of the other samples represent a portion of the total particulate available for analysis. The SEM information indicates that small particles are present in C-MOD. Also, the SEM analysis implies potential particle formation by condensation in C-MOD indicated by the spherical shapes observed.

Additional dust samples were recently collected from the Alcator C-MOD tokamak. The samples are currently undergoing evaluation and the results will be available in the summer of 1998.

VII. ACKNOWLEDGMENTS

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