

Characterization of Microstructures Found on the Surface of and Within Toner Particles -Particle Analyzer Dispersion Evaluation-

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Abstract

We have developed a particle analyzer system, the model PT1000, which analyzes particulates using helium microwave-induced plasma[1-2]. The PT1000 can simultaneously measure the three-dimensional components, composition and size of individual particles without requiring any preparatory treatment. Applied to the evaluation of toner particles, the PT1000 enables changes in toner properties, that could not be identified with any other techniques, to be measured with high sensitivity. This paper discusses the correlation between variations in toner particle properties and analysis results using the PT1000, and presents the results of example measurements using new methods of analyzing property changes during the photo-developing process and so on.

1. Introduction

As printers and printing methods move toward color and digital printing, the performance of individual toner particles has become increasingly important in order to maintain printing quality. Consequently, the size, components and composition of individual particulates need to be analyzed. However, the only existing techniques available for analyzing toner were TEM analysis using toner-embedded resin, electric charge measurement, or dielectric constant measurement, and it was very difficult to directly analyze the amount of coated additives and to measure the degree of dispersion of coated and embedded additives.

The PT1000 introduces toner particles into the plasma one by one, and can analyze the components, composition and size of individual particles for several thousand particles at a time. As a result, the PT1000 can measure the concentration of additives coated on toner particles and the degree of dispersion of both coated and embedded additives as well as analyze free substances that had been very difficult to achieve[3-4].

The significant features of the PT1000 include:

Direct introduction of particles into plasma
Analysis requiring less than one microgram of sample
Use of atmospheric-pressure helium plasma

Direct introduction of particles into the plasma not only simplifies sample preparation but also enables the properties of each individual particle to be evaluated. Since less than one microgram of sample are required, the process inside the printer can be analyzed. In addition, because atmospheric-pressure helium plasma has high excitation energy, not only metallic elements but also nonmetallic elements such as carbon and silicon can be measured with high sensitivity. Theoretically, all elements including halogen and light elements can be measured.

2. Principle

2.1 System Configuration

Figure 1 outlines the system. A filter that has collected particulates is placed in a helium environment, and a nozzle above the filter scans the filter in detail. At a scan width of 16 mm, a maximum of 15 scans can be performed with the filter spun slightly after each scan. Particulates suctioned into the aspirator separate into their cohesion substances at the sonic velocity region of the aspirator, and are then introduced into the plasma one by one. The plasma cavity is a Beenakker type electromagnetic resonant cavity in the

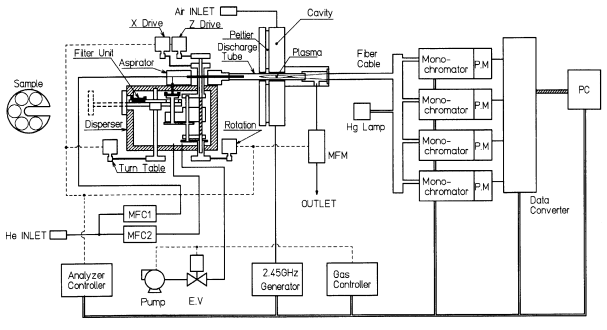

Figure 1. System Configuration

TM010 resonance mode and produces a helium plasma at atmospheric pressure[5]. The input is 150 W and the frequency is 2.45 GHz. It is reported that, under these conditions, the electron density is 5.8 x 10^{14} cm^{-3} and the excitation temperature is 3600 K.

The particulates rushing into the plasma are decomposed to atoms and excited, and therefore emit light. The emission spectra are fed into four spectroscopes through optical fibers. The elements are determined from the wavelengths of emission, the number of particulates from the number of occurrences of emission peaks, and the particle sizes from the peak height. These measured data are transmitted to a workstation. Each component of the system is controlled by the workstation so that the measurement is performed completely automatically.

2.2 Correlation Between Toner Properties and Data from PT1000

Let us assume there is an ideal toner particulate whose surface is coated by silica of identical thickness, regardless of the size. The relationship between the base material, carbon, and the coated additive, silica, fits the curve $y = x^{2/3}$ as shown in Figure 2a. If the additive silica is embedded within the base material and the ratio of the concentration of silica to the base material is constant, then the ratio of the emission intensity of silica to that of the base material is also constant. Hence, the distribution fits a straight line as shown in Figure 2b. On the contrary, when silica is free, silica is not present in the base material, so the base material and silica independently emit light and there is no simultaneity in emission time. As a result, silica is detected on the Y axis only, as shown in Figure 2c.

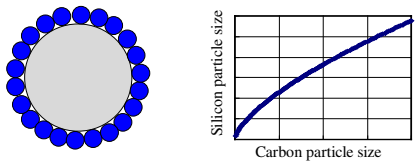

Figure 2a. Ideally Coated Model

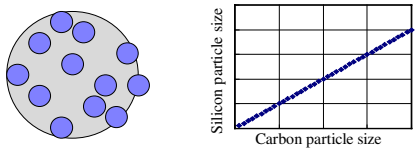

Figure 2b. Embedded Additive

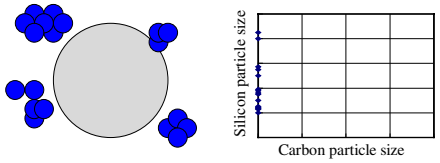

Figure 2c. Free Additive

As discussed above, observation of the distribution of synchronized emissions enables the properties of the embedded and coated additives to be measured.

3. Results and Discussion

3.1 Analysis of Coated Substances

Figure 3 shows an example of measuring toner particles of carbon (the base material) coated with silica. The figure indicates the distribution of silica that simultaneously emitted light with carbon. Values on the X-axis are proportional to the equivalent particle size of carbon and values on the Y-axis are proportional to the equivalent particle size of silica.

The width of this dispersion appears wider when silica is coated unevenly, hence the uniformity of the coating thickness of the coated material can be determined by analyzing this dispersion width.

Counts on the Y-axis indicate silica particulates that did not emit light simultaneously, namely, free silica that is not attached to the carbon.

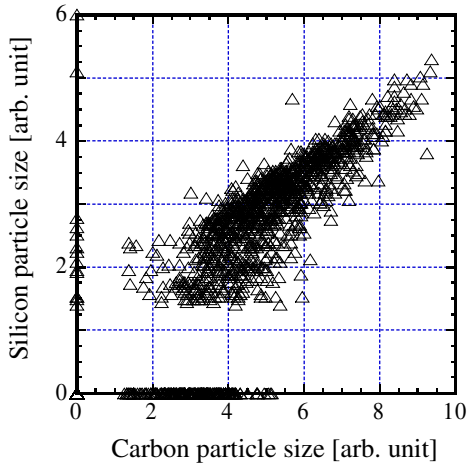

Figure 3. Results of Analysis Using the PT1000

3.2 Analysis of Embedded Substances

Figures 4 and 5 show examples of measuring toner particles in which CCA was embedded by different methods. For the CCA used here, part of the contained organic compounds was substituted by heavy metal so that the observed dispersions of carbons and substitute heavy metal could be used to evaluate the dispersion of CCA.

From the measured results, the dispersion in Figure 4 has a narrower width than in Figure 5, indicating that CCA is embedded more evenly for the samples of Figure 4.

The absolute deviations are quantified values of deviations from the perfectly uniform dispersion state. In this manner, the dispersion of an embedded substance, which has been difficult to analyze, can be evaluated

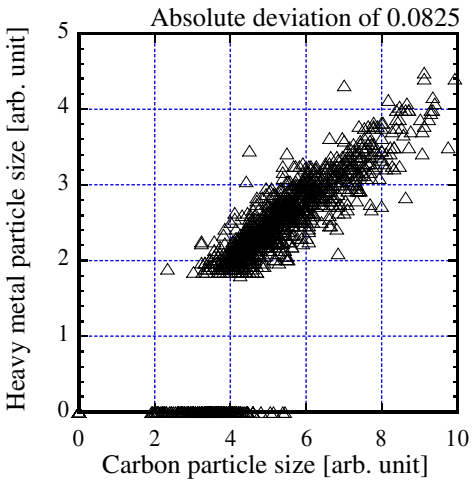

Figure 4. Dispersion of CCA(Recipe A)

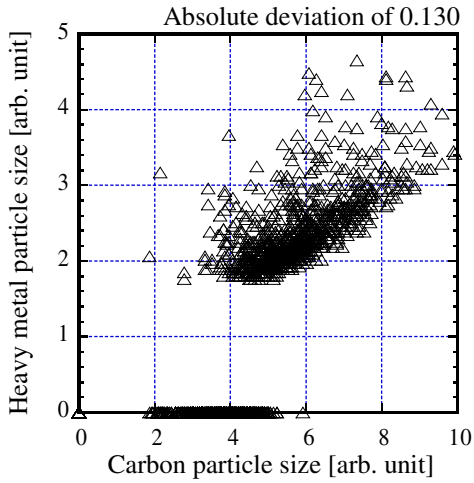

Figure 5. Dispersion of CCA(Recipe B)

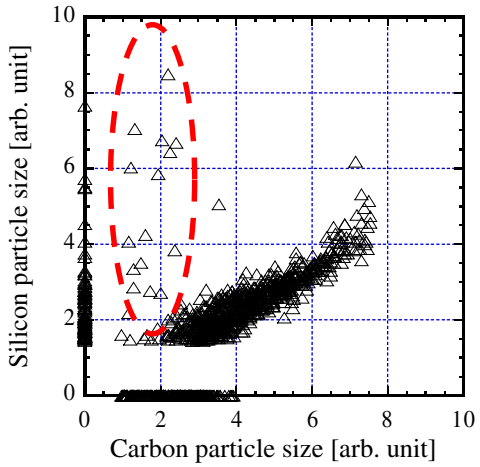

Figure 6. Dispersion of Coated Additive Silica(NG lot)

qualitatively by using the PT1000.

3.3 Analysis of NG Lot

Figure 6 shows the result of analyzing toner of an NG (no good) lot. No significant difference can be seen in the concentration of coated silica and the degree of dispersion near the middle. Nevertheless, the NG lot represents the presence of a large amount of silica-rich particulates. It can be assumed that, because of the large difference in ability to carry electrical charge, the silica-rich particulates greatly affect the printing performance.

After the manufacturing conditions had been modified based on these results, the printing performance was improved.

3.4 Process Analysis

3.4.1 Virgin Toner and Toner on Drum

Figures 7 to 8 show the results of analyzing virgin toner and toner on a printer drum. The former toner was directly sampled from unused toner, and the latter from toner adhered onto a printer drum. The X-axis shows the particle sizes of toner, and the Y-axis the counts. In comparison to the virgin toner, the toner on a drum shows higher peaks of particle size. This means that, in the process by which toner particles adhere to the drum, large particles are selectively adhered.

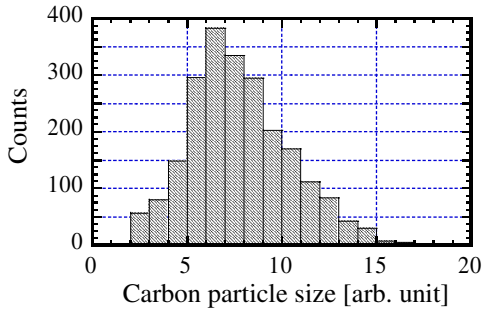

Figure 7. Particle Size of Virgin Toner

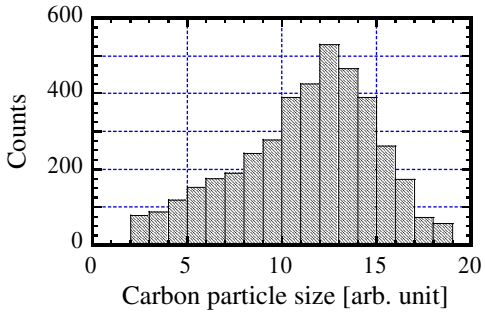

Figure 8. Particle Size of Toner on Drum

3.4.2 Relationship between Number of Pages Printed and Properties of Toner on Drum

Figure 9 shows the relationship between the number of pages printed and the ratio of free particles in the coated substance, and Figure 10 shows the relationship between the number of pages printed, concentration of silica, and absolute deviation. The printing performance was poor when the 2000th page was printed; however, the print conditions were changed after this, and the performance was improved and had recovered when the 4000th page was printed. The ratio of free particles in the coated substance is obtained by dividing the count of free coated substance by the count of total coated substance.

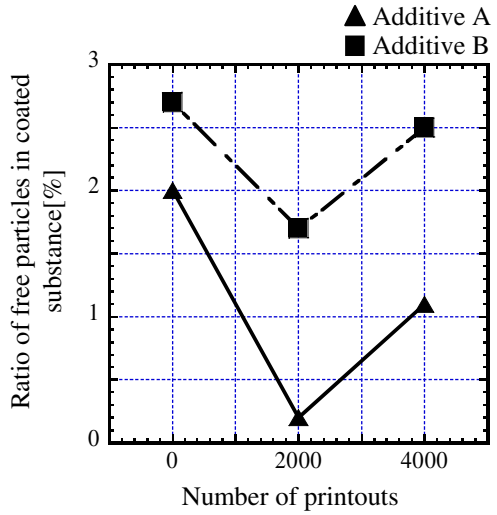

Figure 9. Relationship between Number of Printouts and Ratio of Free Particles in Coated Substance

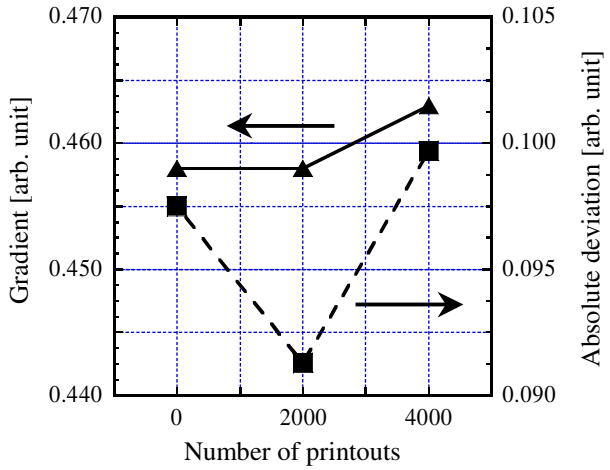

Figure 10. Relationship among Number of Printouts, Concentration of Silica, and Absolute Deviation

As shown in Figure 9, the ratio of free particles decreased towards printing of the 2000th page where the printing performance was poor, and then recovered toward printing the 4000th page to almost the same level as when no page had been printed. The low absolute deviation at the point of printing the 2000th page shown in Figure 10 means a narrow dispersion width of silica, which indicates that the dispersion of coated substance for the same size of toner particles became less. From this, we can consider that the printing performance of toner is affected by the ratio of free coated substance and the degree of dispersion of coated substance.

4. Conclusion

By analyzing the degree of dispersion of coated and embedded additives as well as analyzing process changes inside the printer, this system can clarify the changes in toner particles up to the output and the deterioration process. We intend to improve the analysis method, and thus raise the quality of digital printing and boost the speed of development.

References

1. H. Takahara, M. Iwasaki and Y. Tanibata, *IEEE IM*, vol. **44**, no. 3, June, p. 819, 1995.
2. Eileen M. Skelly Frame, Y. Takamatsu and T. Suzuki., *Spectroscopy*, (1), 17-22 (1996)
3. T. Suzuki, Y. Ujigawa and H. Takahara, *IS&T-s NIP 14: Intl. Conf. on Digital Printing Technol.*, Oct 18-23, 1998, p. 635
4. T. Suzuki, Y. Ujigawa and H. Takahara, *Proceedings of Pan-Pacific Imaging Conference/Japan Hardcopy ë98*, 1998, p. 285
5. Beenakker, C. I. M., *Spectrochim. Acta*, **31B**, 483(1976)

Biography

In 1989, the author acquired a degree in physics from Gakushuin University. In 1991, he acquired a Master of Science in post-graduate studies in physics from the Natural Science Research Laboratory, Gakushuin University. He joined Yokogawa Electric Corporation in 1991, and works as a software engineer. Presently he is developing particle analyzers in the Particle Analyzer Business Center of Yokogawa Electric Corporation.