New Method for Evaluation of Addition to Both the Surface and the Core of Toner Particles -Toner Analysis Using a Particle Analyzer-

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Abstract

We have developed a particle analyzer system, the model PT1000, which analyzes particulates using helium microwave-induced plasma (He-MIP). The PT1000 can measure data for components, composition and size of individual particles for several thousand particles at a time. We applied this system to toner powder analysis and successfully analyzed the coated additive concentration and the degree of dispersion of coated and embedded additives. We detected differences in samples that could not be identified with other analytical techniques.

1. Introduction

In electrophotography, toner plays a key role and interacts with all subsystems. Improving the performance and stability of toner greatly improves the performance and quality of printing. However, the only techniques available for analyzing toner were surface analysis using an SEM, particle size measurement, or electric charge 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.

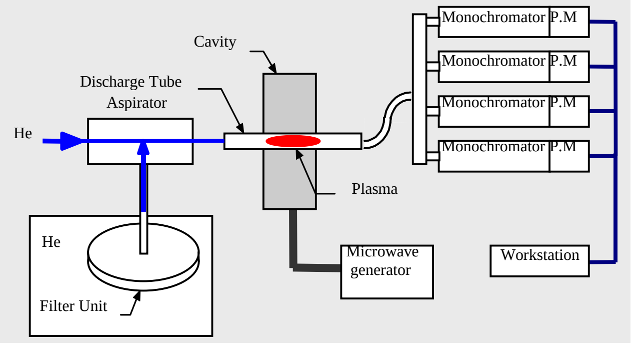

Figure 1. Configuration of Particle Analyzer PT1000

Recently, we have developed the PT1000 Particle Analyzer System. The PT1000 is a particulate analyzing system using helium microwave-induced plasma and is used mainly for analysis of particles in clean rooms(1).

This time, we have successfully analyzed toner from an entirely new aspect by using the PT1000 for analysis of toner particles. This system 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. The use of the system enables changes in toner properties, that could not be identified with any other techniques, to be measured with high sensitivity.

2. Experimental

2.1 System Configuration

Figure 1 outlines the measuring system used. A filter that has collected particulates is placed in a helium environment, and a nozzle above the filter scans the filter in detail. At the scan width of 16 mm, a maximum of 15 scans can be performed. The filter is 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 emission cavity one by one. The plasma cavity is a Beenakker type electromagnetic resonant cavity(2) in the TM_{010} resonance mode and produces a helium plasma at atmospheric pressure. The input is 150 W and the frequency is 2.45 GHz. It is reported that, under these conditions, the electron density is 5.8×10^{14} /cm^3 and the excitation temperature is 3600 K.

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

The significant features of this system are direct introduction of particles into plasma and the use of atmospheric-pressure helium plasma. Direct introduction of particles into plasma not only simplifies sample preparation but also enables the property of each individual particle to be evaluated. In addition, atmospheric-pressure helium plasma has a high excitation energy as shown in Table 1 and, theoretically, excitation of all the elements is possible. As a result, halogen and light elements that cannot be analyzed with sufficient sensitivity using the X-ray fluorescence or ICP emission technique can be analyzed with high sensitivity[3].

Table 1. Excitation and ionization energies of atoms (eV)

Energy	He	Ar
E_1	21.9	11.6
E_{1M}	19.8	11.5
E_i	24.5	15.8

E_1:	The lowest excitation energy level. Transition from the ground state to this level is permitted.
E_{1M}:	The lowest metastable excitation energy level.
E_i:	Ionization energy from the ground state.

2.2 Equivalent Particle Size

Figure 2 shows the general idea of an equivalent particle size. Existing particulates vary in shape and are not usually composed of a single component. This system measures the intensity of emission of each element and computes the diameter of a true sphere that emits the same intensity of light. This computed diameter is then displayed as the particle size. Namely, for coated and embedded additives, the diameters of true spheres equivalent to the total amount of the coated additive and the total amount of the embedded additive, are obtained as the sizes.

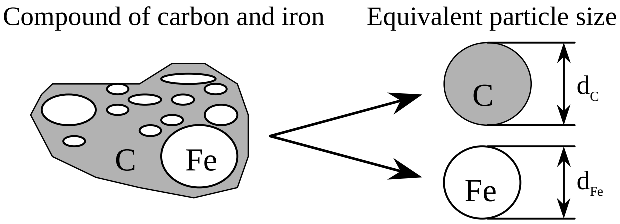

Figure 2. Equivalent Particle Size

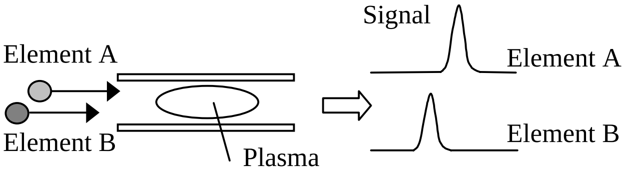

Figure 3a. Emissions of Single-element Components

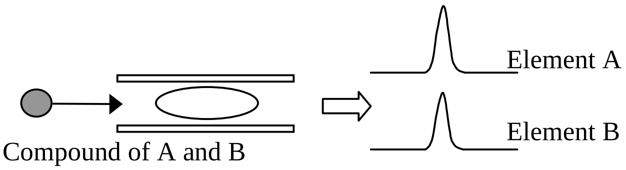

Figure 3b. Emissions of Compound

2.3 Synchronization

Figures 3a and 3b show light emission from particulates suctioned into the nozzle. When single-element components, element A and element B in Figure 3a, emit light, there is no correlation in time. In contrast, when a compound of elements A and B emits light, each element of the compound emits light at the same time as shown in Figure 3b. This means that by observing the simultaneity in emitted lights, compounds and single-element components can be distinguished. By measuring toner particulates, embedded additives and coated additives adsorbed into the base material can be distinguished from free materials.

2.4 Correlation Between Toner Properties and Data from PT1000

Let us assume there is an ideal toner particulate whose surface is coated by silica of an identical thickness, regardless of the size. The relationship between the base material, carbon, and the coated additive, silicon, fits the curve $y = x^{2/3}$ as shown in Figure 4a. If the additive silica is embedded inside the base material and the ratio of the concentration of silicon to the base material is constant, then the ratio of the emission intensity of silicon to that of the base material is also constant. Hence, the distribution fits a straight line as shown in Figure 4b. On the contrary, when silica is free, silica is not present in the base material, so the resin and silicon independently emit light and there is no simultaneity in emission time. As a result, silicon is detected on the Y axis only, as shown in Figure 4c.

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

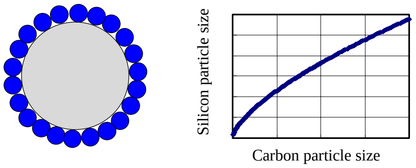

Figure 4a. Ideally Coated Model

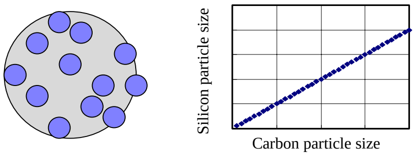

Figure 4b. Embedded Additive

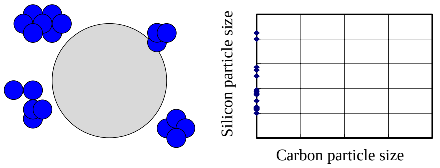

Figure 4c. Free Additive

3. Results and Discussion

3.1 Analysis of Coated Substances

Figure 5 shows an example of measuring toner particles of resin coated with silica. The figure indicates the distribution of silicon 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 silicon.

If this dispersion width is broad, it signifies that the coated thickness of the coating substance silica is not uniform and the coating varies widely from thin to thick. If the dispersion width is narrow, it signifies that the coated thickness is uniform.

In addition, counts on an axis indicate a substance that did not emit light simultaneously. For example, by examining the data on the Y-axis, we can obtain information on free silica that is not attached to the resin.

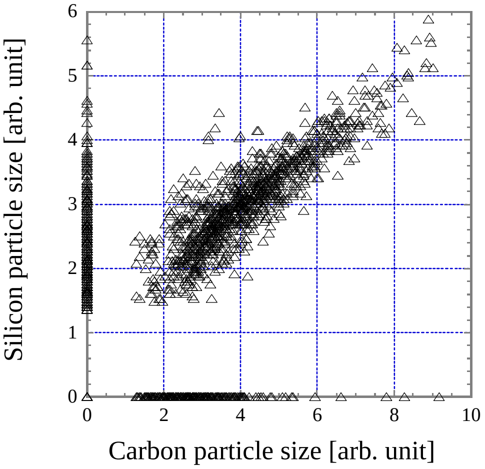

Figure 5. Dispersion of Toner Particles with Coated Additive Silica

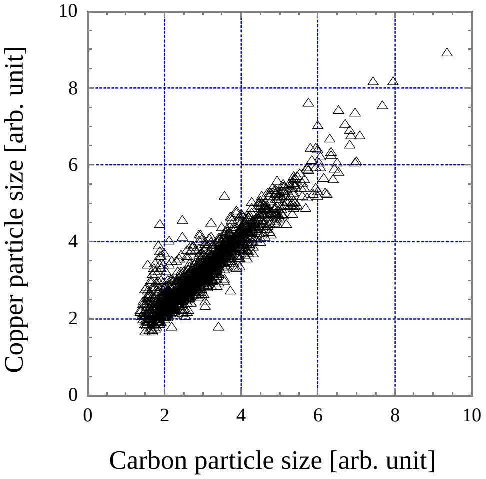

Figure 6. Dispersion of Toner Particles with Embedded Additive Copper

3.2 Analysis of Embedded Substances

Figures 6 and 7 show examples of measuring toner particles of resins with embedded copper and iron respectively.

Copper is found in organic compounds and thus homogeneity of resin and copper is better. In contrast, iron particle size is large and homogeneity of resin and iron is not so good.

Measurements with the PT1000 show that the dispersion width for copper is narrow as shown in Figure 6 indicating that copper is uniformly distributed in the resin. In contrast, the dispersion width for iron is very broad as shown in Figure 7 indicating that iron is scattered but localized in some positions. That is, these figures accurately represent the states of embedded copper and iron.

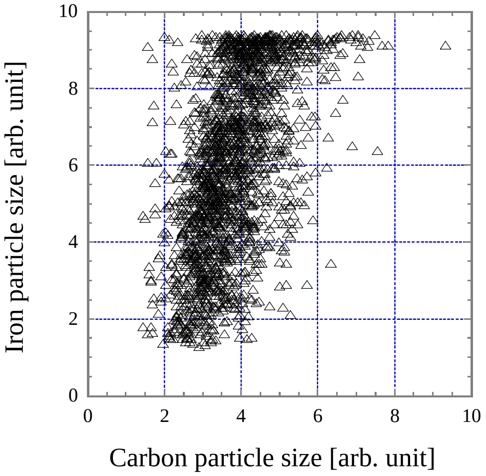

Figure 7. Dispersion of Toner Particles with Embedded Additive Iron

3.3 Changes in State through Acceleration Test

Figures 8 to 11 show the results of analyzing virgin toner with coated silicon additive and the same toner after an acceleration test.

3.3.1 Coated Additive

The distribution of the coated additive silicon of the virgin toner clearly fits the relation $y = x^{2/3}$ which indicates a coated additive (Figure 8). After the acceleration test, however, the distribution has changed to a straight line, which indicates an embedded additive (Figure 9).

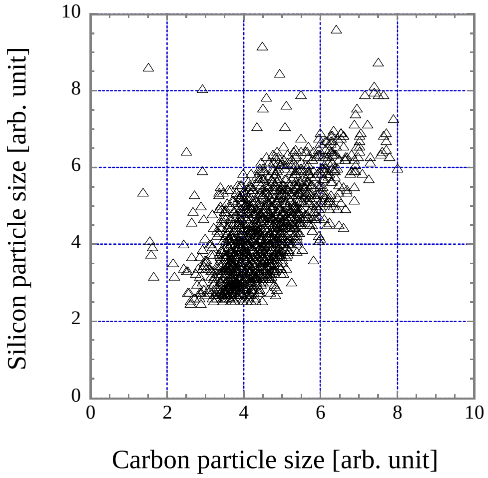

Figure 8. Dispersion of Coated Additive Silica for Virgin Toner

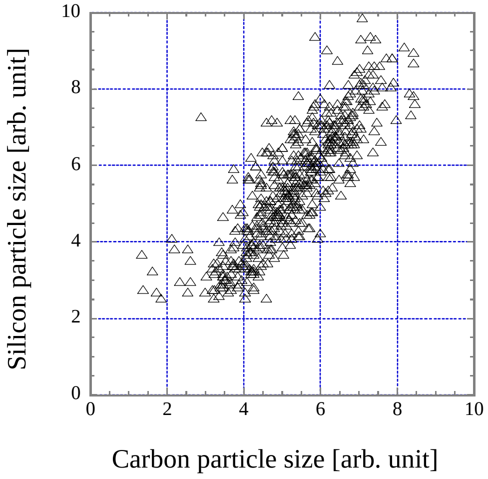

Figure 9. Dispersion of Coated Additive Silica after Acceleration Test

Namely, the acceleration test caused a change in the state of the silica; the silica coated on the toner surface became embedded within the base resin.

3.3.2 Distribution of Particle Size of Base Resin

Figures 10 and 11 show the distribution of particle size of the resin before and after the acceleration test. On the X axis is the particle size of the resin and on the Y axis the counts. In comparison to the virgin toner (Figure 10), the particle size of the largest counts becomes larger after the acceleration test (Figure 11). From this, we know that the number of smaller toner particles decreased during the acceleration test and larger toner particles became the majority.

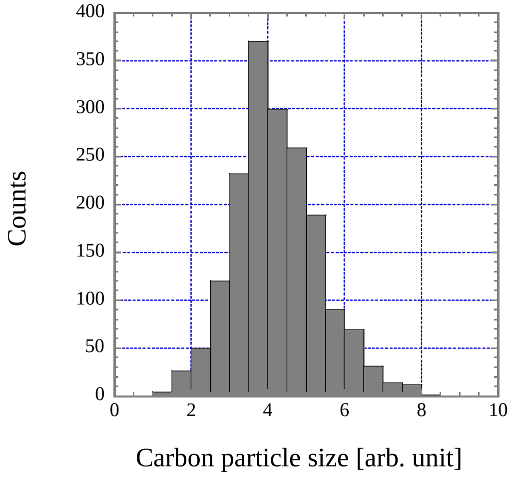

Figure 10. Distribution of Base Resin for Virgin Toner

The cause of this change in the particle size distribution is assumed to be either a decrease in the number of small toner particles due to particle size selectivity, or generation of large toner particles due to cohesion of resin particles. Further time-series analyses may clarify the cause of toner deterioration.

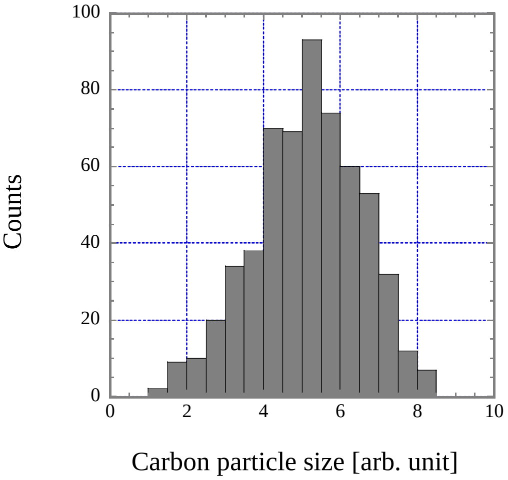

Figure 11. Distribution of Base Resin after Acceleration Test

4. Conclusion

This system enables the degree of dispersion of coated and embedded additives, which has been very difficult to measure, to be analyzed and to acquire information on free substances. Toner degradation and the charging mechanism may be clarified by evaluating toner properties using an entirely different approach from existing analysis techniques.

References

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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 New Business Operations Center of Yokogawa Electric Corporation.