

Effects of Packing Conditions on Apparent Resistivity Measurements of Polymer Powders

K. S. Choi, S. Kawai, T. Y. Kim, M. Yamaguma, T. Kodama, J.H. Joung, M. Masui and M. Takeuchi

Abstract - The apparent volume resistivity of powders measured by both the tapping and the compressing methods was compared in this study. Factors such as applied voltage, pressure, corona charging, and so on affecting the apparent volume resistivity of polymer powders were also examined experimentally. Powders of polyacrylonitrile and Nylon 11 were mainly used. The values of the apparent resistivity of polymer powders taken by the tapping method turned out to be larger than those taken by the compressing method, which indicates that the apparent volume resistivity of polymer powders depends strongly on the measurement method. The apparent resistivity of polymer powders increased with an increase in applied voltage while it decreased with an increase in pressure, tapping time, and particle size. The influence of the moisture content of powder and corona charging on the resistivity of polymer powders is also discussed.

Keywords - polymer powders, apparent volume resistivity, packing conditions

1. Introduction

The apparent volume resistivity of powders is one of the important factors which affect their charging characteristics [1]. It is necessary to estimate the resistivity of relevant powders as well as minimum ignition energy (MIE) and minimum ignition temperature (MIT) of the powders prior to handling them in order to plan protective measures against accidents due to electrostatic spark [2].

Two testing methods have long been used for measuring the apparent volume resistivity of powdered materials based on the direct current (DC) method in Japan. One method is to measure the apparent volume resistivity of a powder layer packed by tapping the specimen several times by hand (tapping method) [3]. The other is to measure the apparent volume resistivity of a powder layer compressed sufficiently between a pair of parallel electrodes (compressing method) [4]. The largest difference between the two testing methods is the configuration of powder particles between a pair of electrodes used to measure the resis-

tance. The apparent volume resistivity of powders usually depends on the packing density and the pressure applied to the powder, as well as on particle size and moisture content.

The two measurement methods of apparent volume resistivity of polymer powders were compared with each other in this study. The apparent volume resistivity of polymer powders was measured as a function of applied voltage, packing pressure, tapping time, moisture content, and so on. The influence of corona charging of the specimen on its apparent resistivity was also examined.

2. Experimental

2.1 Apparatus and method

The experimental system for the resistivity measurements consists of a resistivity test cell, an electrometer (Keithley-6512), a standard high-voltage supply (Matsusada, HSX-3R5), and a personal computer (PC) for data processing.

A schematic drawing of the test cell used for the determination of the electrical resistivity of powder is given in Fig. 1. The test cell is made up of an insulator case (polycarbonate) and three electrodes: a main electrode and a counter electrode, by which the current is measured, and a guard electrode to prevent leakage of current via the cell.

A voltage, V [V], was applied between the electrodes, and the current, I [A], was measured. The resistance, R [Ω], was given by $R=V/I$, and then the apparent volume resistivity, ρ (Ω m), was calculated using the following formula (1):

$$\rho = R \cdot S/L, \quad (1)$$

where S [m^2] is the effective sectional area of the main

We thank Dr. Teruo Suzuki and Mr. Tomofumi Mogami of Kasuga Denki, Inc., in Japan for their help with the experiments and Dr. Sang Won Choi of the Korea Occupational Safety & Health Agency for his helpful discussion. We are also grateful to Dr. Yasuyuki Tabata of the Technology Institution of Industrial Safety in Japan for continuing guidance and encouragement.

Manuscript received: June 19, 2002 accepted: Aug. 22, 2002

K. S. Choi and S. Kawai are with the Graduate School of Science and Engineering, Ibaraki University, Japan.

T. Y. Kim is with the School of Safety Science, New South Wales University, Australia.

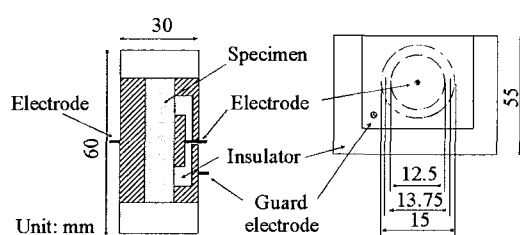
M. Yamaguma and T. Kodama are with the National Institute of Industrial Safety, Japan.

J.H. Joung is with the Department of Safety Engineering, Seoul National University of Technology, Korea.

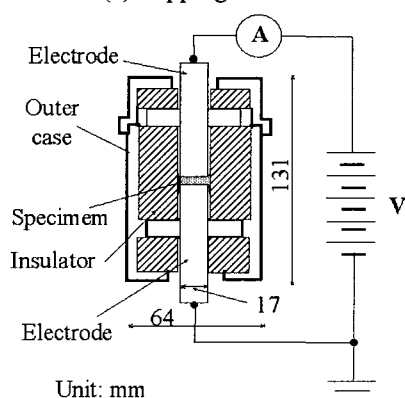
M. Masui and M. Takeuchi are with the Department of Electrical and Electronic Engineering, Ibaraki University, Japan.

electrode, L [m] is the thickness of the specimen, and R [Ω] is the resistance.

The apparent volume resistivity of a powder specimen (1 min value) was measured five times under the same conditions, and the average was calculated as the final value in this study [3]. Electric shielding was put on major parts (electrodes and electrometer) of the experimental system to enhance the accuracy of the measurements. All the measurements were carried out under the conditions of $20 \pm 3^\circ\text{C}$ and $50 \pm 5\%$ RH.



(a) Tapping method



(b) Compressing method

Fig. 1 Test cell for determination of the electrical resistivity of powders.

Table 1 List of powder specimens used in the experiments.

Specimen	Abbreviation	Shape	**M.D [μm]
Ployacrylonitrile	PAN	Round	62
Nylon 11	PA	Irregular	44
Polymethylmethacrylate	PMMA	Round	344
Polystyrene (*waste)	PS	Irregular	31
High-impact polystyrene	HIPS	Irregular	74
Polyethylene	PE	Irregular	145
High-density polyethylene (*waste)	HDPE	Irregular	85

*Waste: Specimens taken from industrial processes.

**M.D: Median diameter.

2.2 Powders

The powders used in this study were polyacrylonitrile powder, defined as electrostatic ignition experimental dust

by IEC[5]; powders of polymethylmethacrylate, polystyrene, and polyethylene; and a coating powder of Nylon 11, as listed in Table 1. Each powder is, hereinafter, referred by its abbreviation given in Table 1. The median particle size measured by the laser diffraction method (LDA Win 1.21, Wet-type) and the shape observed by SEM are also listed in Table 1.

The powders of PAN and PA were used to study the effects of several factors affecting the apparent resistivity of the powders. The other powders were used for comparing the two testing methods. All the powders were dried in a desiccator at 50°C for 24 h before the test.

2.3 Corona charging system for powders

In order to study the charge-transport behavior through the powder layer, a corona charge was put on the powder by using an electrostatic powder-coating system (Onoda, GX376)[6]. A sample powder was pneumatically conveyed to a spray gun in which a corona discharge was generated. A high voltage of -80 kV was applied to the needle-like electrode of the corona spray gun. The powder was charged when it passed the corona discharging space. The experimental results concerning the influence of corona charging are described in Section 3.6.

2.4 Surface potential decay in corona-charged powder layers

The surface potential decay in corona-charged powder layers was observed as follows: A powder specimen was packed in a grounded aluminum cup which was 50 mm in diameter and 1.5 mm in depth. The sample layer was brought under an electrode for corona discharge and was corona-charged negatively.

The corona-discharging voltage was -5 kV, and the charging time was 1 s. The surface-potential decay after the cessation of corona charging was measured with a high-level surface-potential meter (Kawaguchi Electric Works, S-2001). The measurements were performed to check the degree of charge decay rate, which corresponds to the apparent resistivity of powdered materials.

3. Results and Discussion

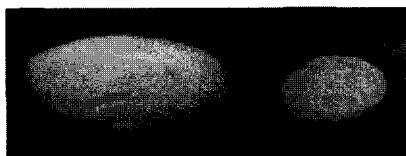
3.1 Comparison of apparent volume resistivity of powders measured by the tapping and compressing methods

The apparent volume resistivities of powder specimens measured by both the tapping and compressing methods are given in Table 2. In the case of the tapping method, the values of resistivity were obtained when the specimen of 12 g was tapped 5 times in the test cell by hand-shake.

They were obtained when the specimen was compressed at 85 MPa between a pair of electrodes in the case of the compressing method.

Table 2 Apparent volume resistivity of powders measured by the tapping and compressing methods.

Specimen	Apparent volume resistivity [$\Omega \cdot \text{m}$]	
	Tapping method	Compressing method
PAN	2.7E14	3.0E12
PA	7.8E12	4.0E11
PMMA	7.8E13	2.4E12
PS	1.7E14	1.8E13
HIPS	1.0E14	8.1E12
PE	4.0E13	2.1E11
HDPE	2.5E14	1.3E13



(a) before (b) after

Fig. 2 Comparison of outlook of PAN powder (5 g) before and after measurements (85 MPa of pressure).

The thicknesses of packed powder layers were about 10 mm and 1 mm for the tapping and compressing methods, respectively. We compared the apparent resistivities at the applied voltage of 100 V and 10 V for the tapping and compressing methods, respectively, in which the electric field applied to the specimen was nearly equal. The values of apparent resistivity for polymer powders taken by the tapping method turned out to be larger than those taken by the compressing method. That is, the value of apparent volume resistivity for powders depended strongly on the method used for measurements.

Fig. 2 shows the outlook of PAN powder before and after the measurements taken by the compressing method. It is obvious that the powder specimen was agglomerated by the pressure used in the test to form a conspicuous disk. This fact suggests that the apparent resistivity of powders depends on the contact resistance both between the electrodes and particles and among the particles.

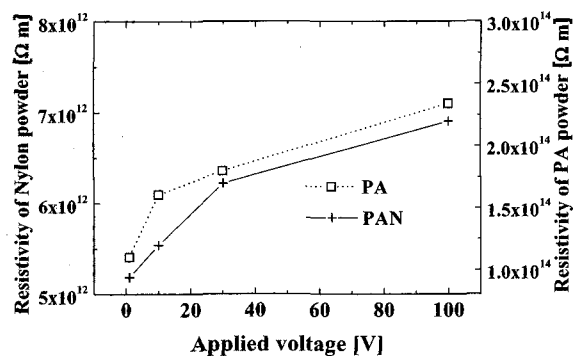
3.2 Influence of applied voltage

The dependence of the resistivity of polymer powder (PA and PAN) on applied voltage obtained by both the tapping and compressing methods is given in Fig. 3.

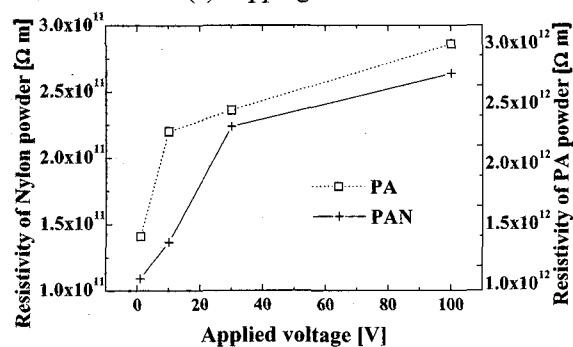
The resistivity of powder specimens was measured by applying DC voltages from 10 to 100 V for both the testing cells. The apparent resistivity of PA powder increased with an increase in applied voltage. A similar result was obtained for PAN powder.

A change in current when measuring the resistivity is

shown in Fig. 4. A high initial current flow was observed, which gradually decayed to a steady level for PA powder. The high initial current shown in the Fig. is the displacement current and represents the energy that must be absorbed to polarize the atoms of the insulator.



(a) Tapping method



(b) Compressing method

Fig. 3 Resistivity of polymer powder as a function of applied voltage across electrodes.

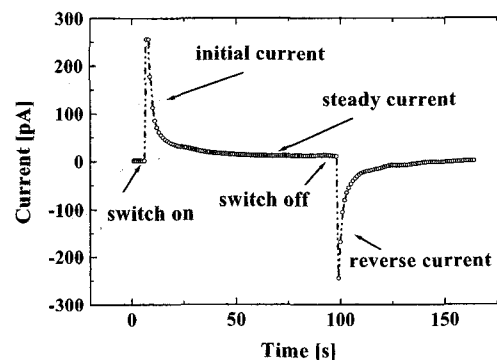


Fig. 4 The current decay in polymer powder as a function of time after applying a DC voltage in resistivity measurements (PA powder; applied voltage: 100 V)

The steady current which occurred after the first minute was equivalent to a time constant of about 100 s. When the applied voltage was turned off, a reverse current began to flow. Fig. 5 shows the traces of reverse current for various choices of applied voltage. Since the reverse current increased with an increasing applied voltage, the dependence of the resistivity of powder on the applied voltage can be

attributed to the accumulation of electrons in the powder specimens.

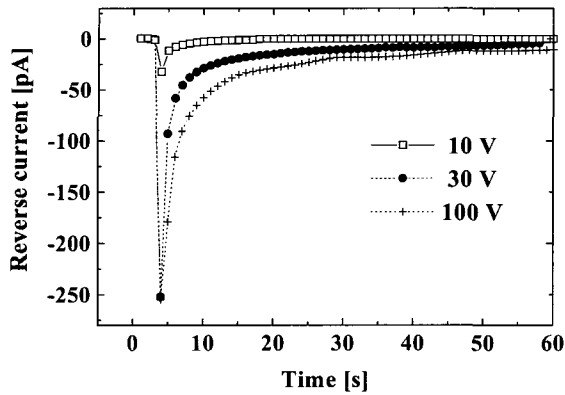


Fig. 5 Reverse current in PA powder as a function of time for various choices of applied voltage.

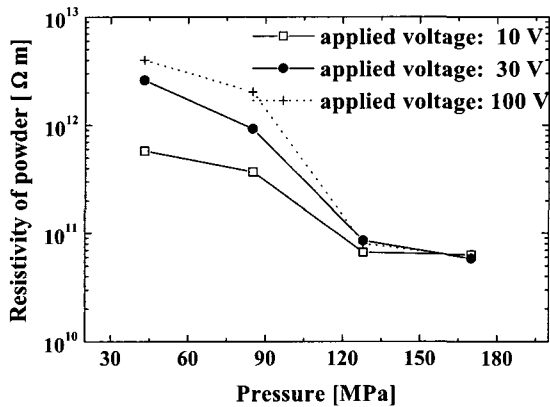


Fig. 6 Resistivity of polymer powder (PAN) as a function of pressure across electrodes.

3.3 Influence of pressure and tapping times

Fig. 6 and 7 show the effect of pressure between electrodes and tapping time, respectively, on the resistivity of the powder specimen (PAN). The resistivity of polymer powder decreased with an increase in both pressure and tapping times.

This result can be explained as follows: First, this may be attributable to an increase in contact area between the particles and electrodes, resulting in a decrease in contact resistance in the compressing method. Second, an increase in tapping time has an effect on the decrease in the porosity among particles, as shown in Fig. 8, resulting in a decrease in the apparent resistivity of polymer powders.

3.4 Influence of particle size of powder

The effects of particle size on the resistivity of polymer powder (PAN) obtained by both the compressing and tapping methods are given in Table 3. Three kinds of powders

of about 30, 50, and 70 μm in diameter were used in this study. It should be noted here that the value of the particle size was the median value.

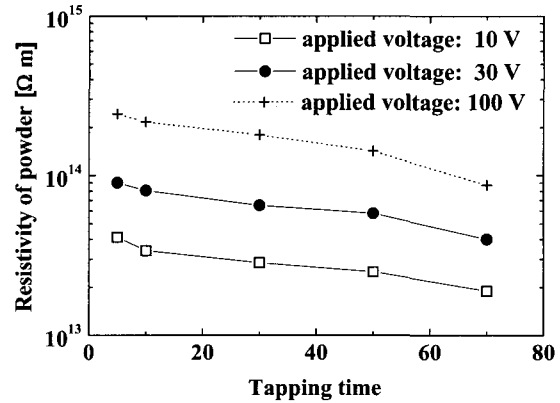


Fig. 7 Resistivity of polymer powder (PAN) as a function of tapping time.

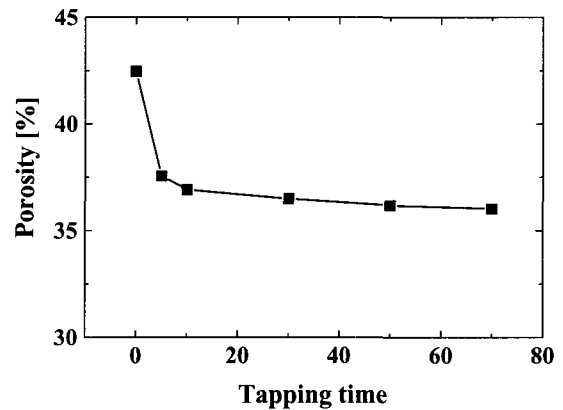


Fig. 8 Relationship between porosity of powder layer and tapping time.

Table 3 Effects of the median particle size on apparent resistivity of polymer powder (PAN) measured by the tapping and compressing methods.

Specimen size [μm]	Apparent volume resistivity [$\Omega\text{ m}$]			
	Tapping method		Compressing method	
	*10 V	100 V	10 V	100 V
30	2.4E13	3.5E14	4.9E12	9.4E12
50	1.3E13	2.1E14	3.4E12	7.2E12
70	8.3E12	1.2E14	2.6E12	5.5E12

*: Applied voltage

The apparent resistivity of sample powders taken with both the compressing and tapping methods decreased with an increase in the particle size of the samples. This result suggests that the influence of the contact resistance between particles is superior to other factors in the apparent

resistivity of powders. When the particle size is large, the contact area between particles is large, while the number of contacts between particles is small, resulting in a decrease in the resistivity of the powder.

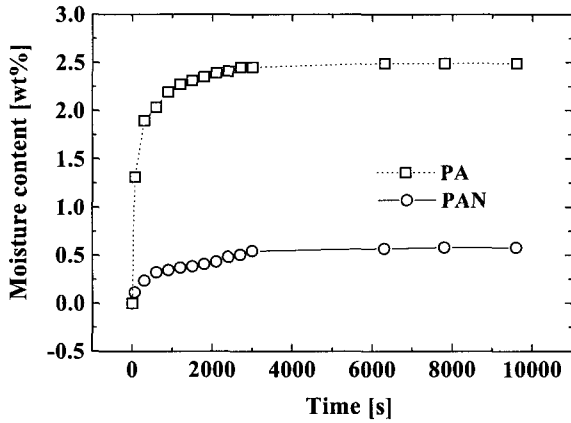


Fig. 9 Change in moisture content with time after being left exposed to ambient air.

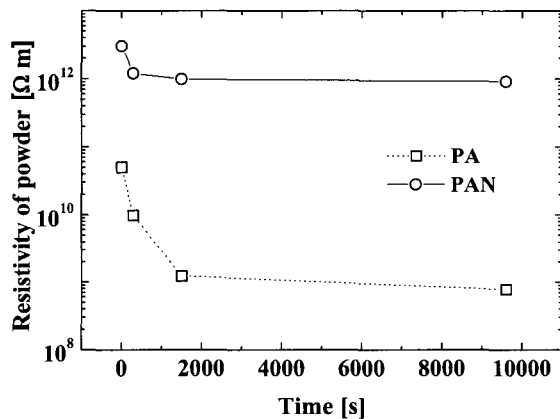


Fig. 10 Change in resistivity with elapsed time after being left exposed to ambient air for polymer powders (compressing method; applied voltage: 10 V).

3.5 Influence of moisture content of powders

After powder specimens (PA and PAN) were dried sufficiently in an evacuated oven, they were left exposed to ambient air. Fig. 9 shows the change in moisture content adsorbed on the powder specimens with time after being exposed to ambient air. The amount of moisture adsorbed on the powder specimens was weighed with a balance. Because PA powder is a hygroscopic material, a relatively large amount of moisture was adsorbed on the PA powders, while the moisture adsorption was relatively low for the PAN powders.

The resistivities of powder specimens (PA and PAN) exposed to ambient air, as a function of elapsed time, are shown in Fig. 10. The resistivities changed with the moisture content for both powders. However, the resistivity of

PA powder was more sensitive to ambient moisture than that of PAN powder. This fact suggests that electrical conduction through the moisture layer adsorbed on the surface of powder specimens decreased their apparent resistivity.

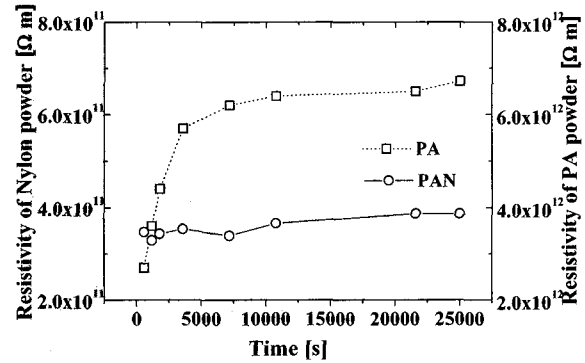


Fig. 11 Resistivity of the corona-charged polymer powder as a function of time (compressing method; applied voltage: 10 V).

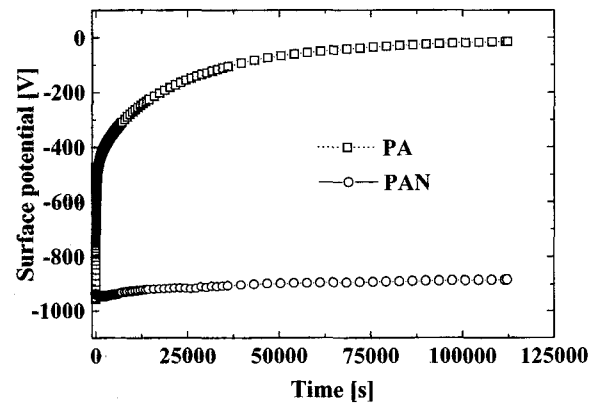


Fig. 12 Decay of the surface potential for sample powder-layers (corona discharge: -5 kV).

3.6 Influence of corona charging on the carrier transport in powder layers

Fig. 11 shows the resistivity of polymer powders (PA and PAN) which were corona charged by the electrostatic powder coating system mentioned in Section 2.3 as a function of elapsed time after corona charging. Corona charging had a large effect on the resistivity of PA powder; that is, corona charging decreased the value of the resistivity significantly, but the resistivity increased with elapsed time after corona charging. On the other hand, corona charging had little effect on the resistivity of PAN powder.

To study the corona-charging behavior of powder specimens in greater detail, the decay of the surface potential after corona charging in powder layers was observed. The results are shown in Fig. 12. The surface potential decay was relatively fast for the PA powder layer, while the decay was extremely slow for the PAN powder layer. This result suggests that charge carriers can and cannot migrate

easily in PA and PAN powders, respectively. The former contributes to charge transport in the powder layer, but the latter does not. Corona charges can be considered to work as charge carriers in the powder layer.

6. Conclusions

A comparison of the tapping and compressing methods for apparent volume resistivity measurements of polymer powders was carried out in this study. Factors such as applied voltage, moisture content, corona charging, and so on affecting apparent volume resistivity of polymer powders were also examined experimentally.

The experimental results can be summarized as follows:

- (1) The values of apparent resistivity of polymer powders taken by the tapping method turned out to be larger than those taken by the compressing method.
- (2) The apparent resistivity of polymer powders increased with an increase in applied voltage and decreased with an increase in compressing pressure between two electrodes.
- (3) An increase in tapping time decreased the porosity of powder layers, resulting in an increase in apparent resistivity.
- (4) The apparent resistivity of polymer powders decreased with an increase in particle size.
- (5) The electrical conduction through a moisture layer adsorbed on the surface of sample powders decreased their apparent resistivity.
- (6) Corona charging decreased the resistivity of the PA powder layer, in which the charge decay is relatively fast.



Kwang Seok Choi received the B.E and M.E degrees from the Seoul National University of Technology, Seoul, Korea, in 1998, 2000, respectively. And currently, studying in Ph degree at Ibaraki National University of Japan. His research interests include static electrification and dust explosions.

Tel: +81-424-91-4512, Fax: +81-424-91-7846
E-mail: choiks@anken.go.jp

Acknowledgements

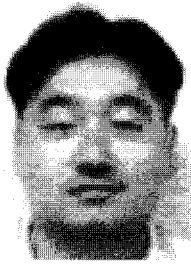
We thank Dr. Teruo Suzuki and Mr. Tomofumi Mogami of Kasuga Denki, Inc., in Japan for their help with the experiments and Dr. Sang Won Choi of the Korea Occupational Safety & Health Agency for his helpful discussion. We are also grateful to Dr. Yasuyuki Tabata of the Technology Institution of Industrial Safety in Japan for continuing guidance and encouragement.

References

- [1] K.S.Choi, M.Yamaguma, T.Kodama, J.H.Joung, D.S.Shin, N.Iwai, T.Kashiwazaki and M.Takeuchi, "*An experimental study on tribocharging of fine powders in powder coating spray gun*", KIEE International Transactions on Electro-Physical and Application, vol.11C, no.2, pp.1-5, June, 2001.
- [2] T. Hope, N. Jaeger and J. Terry, "*Safe handling of combustible powders during transportation, charging, discharging and storage*", J. Loss prev. Process Ind. vol.13, pp.253-263, 1996.
- [3] The Research Institute of Industrial Safety Japan, *Recommended practice for protection against hazards arising out of static electricity in general industries*, RIIS-TR-87-1, 1988, p.6.
- [4] Institute of Electrostatics of Japan, *Handbook of static electricity*, 1998, pp.284 and 1233.
- [5] IEC, *International standard 1241-2-3*, 1994.
- [6] K. S. Choi, M. Yamaguma, T. Kodama, J. H. Joung, J. Cross, C. S. Choi, T. Y. Kim and M. Takeuchi, "*Ignitability of powder paints in electrostatic powder coating system*", Asia Pacific Sym. on Safety Proc., vol.1, pp.297-300, November 2001.



Shintaro Kawai received the B.E and M.E degrees from the Ibaraki University, Ibaraki, Japan, in 2000, 2002, respectively. He is now working at Coper Co., Japan. His research interests include static electrification of electro photographic developers.



Tai Young KIM received B.S. degree in Safety Engineering from the Seoul National University of Technology and M.S. degree in Safety Science from the University of New South Wales in 1998 and 2002, respectively. He has been a research student at the University of New South Wales, where he has been

studying on the removal of harmful gases using plasma technology for Ph.D. degree.

Tel: +61-2-9385-5002, Fax: +61-2-9385-6190

E-mail: tykim@student.unsw.edu.au

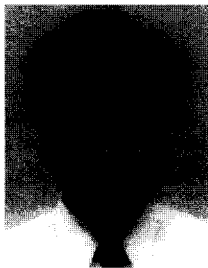


Jae Hee Joung received the B.E. M.E and D.E degrees from the Chung Ang University, Seoul, Korea, in 1979, 1981, and 1984, respectively. In 1985 he joined the Department of Safety Engineering at Seoul Nation University of Technology and professor. He has been responsible for research and development on safety management

and electrostatic safety in industrial companies.

Tel: +82-2-970-6376, Fax: +82-2-976-7479

E-mail: joung@duck.snut.ac.kr



Muzuki Yamaguma graduated from Kumamoto National College of Technology in 1979. After working for the Foreign Ministry of Japan as a communication specialist, he entered National Institute of Industrial Safety in 1989. Currently he is a senior researcher and chiefly engaged in studies on electrostatics, especially in the

field of accident prevention caused by electrostatic discharges.

Tel: +81-424-91-4512, Fax: +81-424-91-7846

E-mail: yamaguma@anken.go.jp

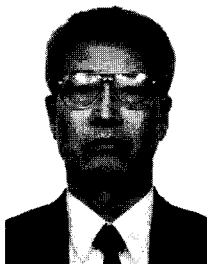


Masayoshi Masui graduated in Department of Electrical Engineering, Ibaraki University, Japan in 1962, and received the D.E degree from the Waseda University, Tokyo, Japan in 1984. He is now Professor of Department of Electrical and Electronic Engineering, Ibaraki University, Japan. His research interests include electrostatics,

electrical breakdown and electrical conduction of polymer, organic thin film gas sensor, and organic electroluminescence device.

Tel: +81-294-38-5099, Fax: +81-294-38-5275

E-mail: masui@ee.ibaraki.ac.jp

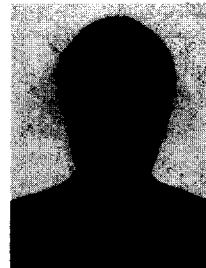


Tsutomu Kodama graduated from Electrical Engineering Department of Kyusyu Institute of Technology in 1966, entered the National Institute of Industrial Safety (NISS), Ministry of Labor and has engaged in the research work on the prevention of electrostatic hazards and nuisances for all the years. He got a position of

chief of Research Planning and Coordinating Section from 1997 to 1998, Director of Physical Engineering Safety Research Division from 1999 to 2000. He is now Director of Mechanical and System Safety Research Group in the NISS, Independent Administrative Institution.

Tel: +81-424-91-4512, Fax: +81-424-91-7846

E-mail: kodama@anken.go.jp



Manabu Takeuchi received the BSc.M.Sc and DSc. Degrees from the Tokyo Institute of Technology, Tokyo, Japan, in 1966, 1968, and 1971, respectively. In 1972, he joined the Department of Electrical Engineering at Ibaraki and Professor at the Physics Department of the University of Alverta, Canada from

1981 to 1982. His research interests include static electrification of polymer powders and photoelectric properties of semiconductor.

Tel: +81-294-38-5091, Fax: +81-294-38-5275

E-mail: takeuchi@ee.ibaraki.ac.jp