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粉體의 付着性 測定에 있어서의 最近 進步

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Recent Advances in Measurement of the Adhesive Force of Powdered Materials

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I. Recent Advances in Measurement of the Adhesive Force of Powdered Materials

Since the late 1950's many studies have been made on the adhesive and cohesive properties of particulate solids. However, most of these studies were performed separately and no attempt has been made to compare the results of these measurements. In May 1977, it was decided that the Working Party on the Powder Mechanical Properties of the Society of Powder Technology, Japan, should engage in a joint study to measure the adhesive force of several kinds of standard powders. Since then, measurements using different methods have been carried out in various laboratories and the results have been compared. In this paper, the work of our Working Party during the past four years is described.

Materials—Five kinds of powders of commercial origin were chosen as the samples. Fig. 1 shows the particle size distributions of those powders measured by the centrifugal sedimentation method.

Measurement Method

Tensile Strength Measurement—1) Vertical Tensile Method: A representative of this method was described by Shinohara and Tanaka [1] in 1975, in which a cell was split horizontally by apply ing a vertical tensile load.

Fig. 2 shows the equipment developed by Yano, Shirahase, Hayashida and Arakawa[2], in which a digital balance is used for measuring the tensile load. Gas paths are provided in the base of the cell holder and the moisture content of the testing powder can be changed by regulating the humidity of the flowing gas.

Suzuki, et al.[3] devised equipment for measuring both the tensile strength and shear strength using ring cells of same dimensiones. The parts for tensile test are shown in Fig. 3.

韓·日 제제 기술 워크샵(1984. 10. 29~30 코리아나 호텔, 약학회 주최)에서 발표되었음.

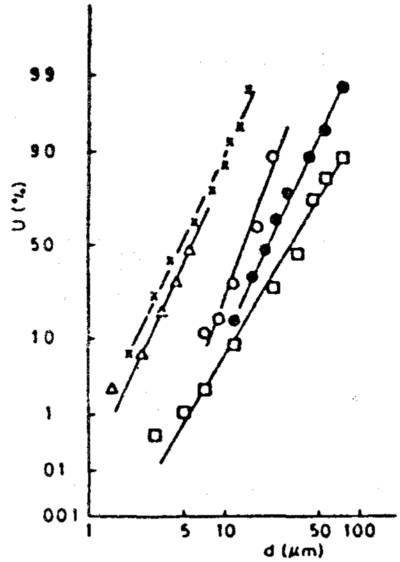

Fig. 1. Particle size distributions for the samples used.
Calcium carbonate, Heavy P-30 : ×, P-70 : ○, BF-300 : ●. α-Alumina WA-2500 : △ Lactose : □

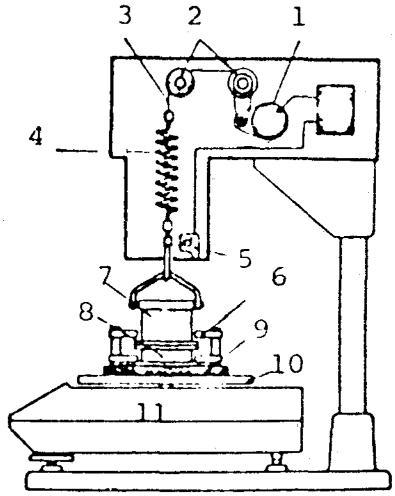

Fig. 2. Tensile strength measuring equipment.
1. Motor, 2. Pulley, 3. Wire, 4. Spring, 5. Optical switch, 6. Cell stopper, 7. Movable upper cell, 8. Fixed lower cell, 9. Cell holder, 10. Pan, 11. Electronic reading balance.

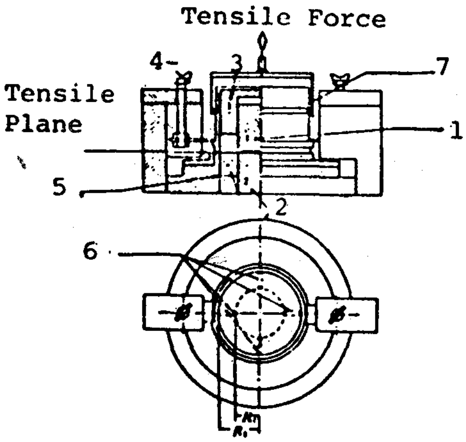

Fig. 3. Tensile pest ring cell.
1. Movable ring cell, 2. Fixed ring cell, 3. Piston, 4. Fixed screw, 5. Filter paper 6. Shear blade.

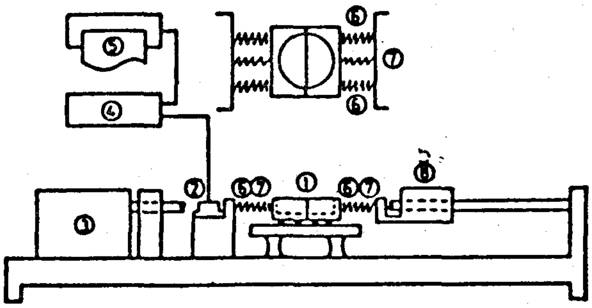

Fig. 4. Modified bearing-type apparatus.
1. Split cell, 2. Strain meter, 3. Motor and transmission, 4. Amplifier, 5. Recorder, 6. Pusher, 7. Spring for pulling, 8. Moving stage

2) Horizontal Tensile Method; The equipment introduced by Ashton et al.is widely noted. Jimbo and Yamazaki[4] proposed a new idea to reduce bearing friction and improve the coupling mechanism of a split cell. The modified bearing-type apparatus is shown in Fig. 4, where both sides of the cell are on bearings and coupled with pushers.

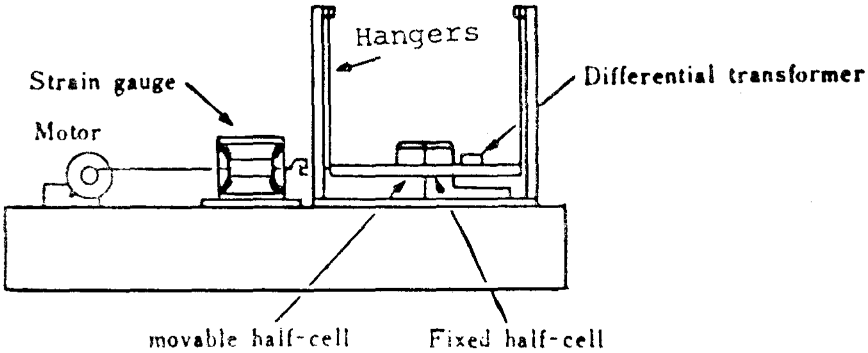

Fig. 5. Tensile strength tester equipped with hanging strips.

The equipment developed by Fujii, Kaya, Urayama, and Yokoyama[5] is shown in Fig. 5. This is designed to make the blank value as small as possible by suspending the movable side of the cell with hangers. The displacement of the cell in the horizontal direction is measured by a differential transformer.

3) Diametral Compression Test; Takahashi et al. determined the tensile strengths of briquetted materials (tablets) by means of the diametral compression test. This test consists of com pressing tablets diametrically between two flat platens of the material testing instrument. If the tablet fails solely due to tension, the tensile strength σ is given by:

$$\sigma=2P/\pi Dt$$

where P is the applied load, D is the tablet diameter, and t is the tablet thickness.

Shear Strength Measurement— 1) Constant Load Method: The procedure proposed by Jenike is a representative of this method. A number of shear tests have been performed using the Jenike cell or ones similar in principle. Nevertheleess,there are some problems even in this method. It is well known that a powder bed expands or contracts when it is sheared, which depends on the material being tested and the initial state of packing. It is therefore desirable to measure the vertical displacement and shear stress together with the horizontal strain of the testing powder. A direct shear tester with which these amounts can be measured simultaneously was developed by Terashita, Miyanami and others[7] (Fig. 6). They also measured the normal stress near the shear plane directly using pressure transducers.

2) Constant Volume Method; This method was established by Aoki and Tsunakawa.[8] When the volume of a powder bed is kept constant during the shearing procedure, cha-

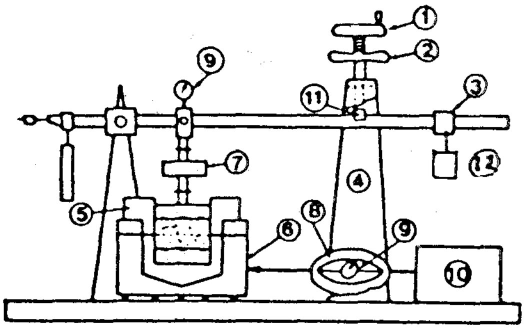

Fig. 6. Direct shear tester.
1. Handle, 2. Stopper, 3. Beam, 4. Supporter, 5. Fixed shear cell, 6. Movable shear cell, 7. Load transducer 8. Proving ring 9. Dial gauge, 10. Motor 11. Clamp 12. Weight

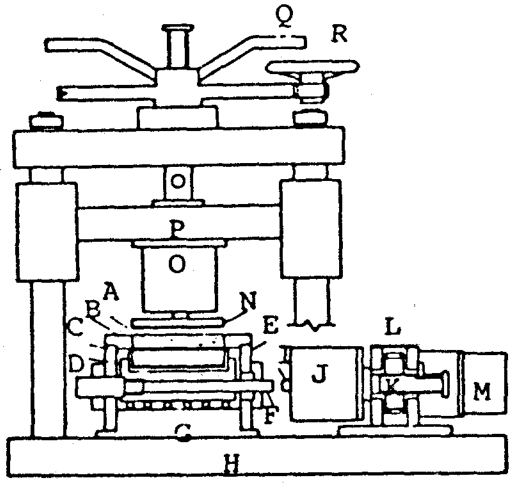

Fig. 7. Direct shear tester equipped with a press loading system.
A. Specimen, B. Metal ring (Fixed cell), C. Metal mold (Movable cell), D. Base mold, E. Guide ring, F. Shaft, G. Plate, H. Press base, I. Loading pin, J. Load transducer, K. Screw shaft, L. Reduction gear, M. Syncronus motor, N. Lid, O. Load transducer, P. Arm, QR Handle

nges in both the shear and normal load with horizontal strain are observed. Therefore, when non-cohesive materials are tightly packed, a yield locus (YL) can be obtained with only one shear test by recording both loads on an X-Y recorder.

Tsunakawa and Saegusa[9] proposed a procedure of obtaining YL of a cohesive powder in one test. The apparatus used is illustrated in Fig. 7. As described previously, Suzuki et al. used ring cells to measure both the shear and tensile strengths of cohesive powders.

3) Other Methods: The tilting cone method proposed by Lowes and Perry and the tilting box method by Hayashi and Minami were reexamined by Otsuka et al. in order to obtain the shear strength for loosely packed powders.

Single Particle Separation Method—The centrifugal method has been widely used for determining the adhesive forces of particles to substrates. Recently, Jimbo et al.[10] have developed the vibration and impact method, in which a vibrator and a tapping device are used respectively. Otsuka et al.[11] have extended the range of the separation force in the impact method using a shock-testing machine.

Results and Discussion

Tensile Strength and Shear Strength—The results of the measurements of tensile and shear strengths with different equipments in various laboratories are summarized in Fig. 8 (a) ~(e). As can be seen in these figures, even though the measured values were obtained by different methods and workers, they almost coincide with each other.

Adhesive Force of a Single Particle—Matsuda et al. measured the adhesive forces between particles and the surface of the tablet made from the same material by the centrifugal method. The values obtained are approximately 30~100 times less than those of the adhesive force to a flat glass surface.

Conclusion

It is considered that the methods for determining the adhesive forces of powder particles have approached the stage of practical use. Some of the equipment described in this review are now

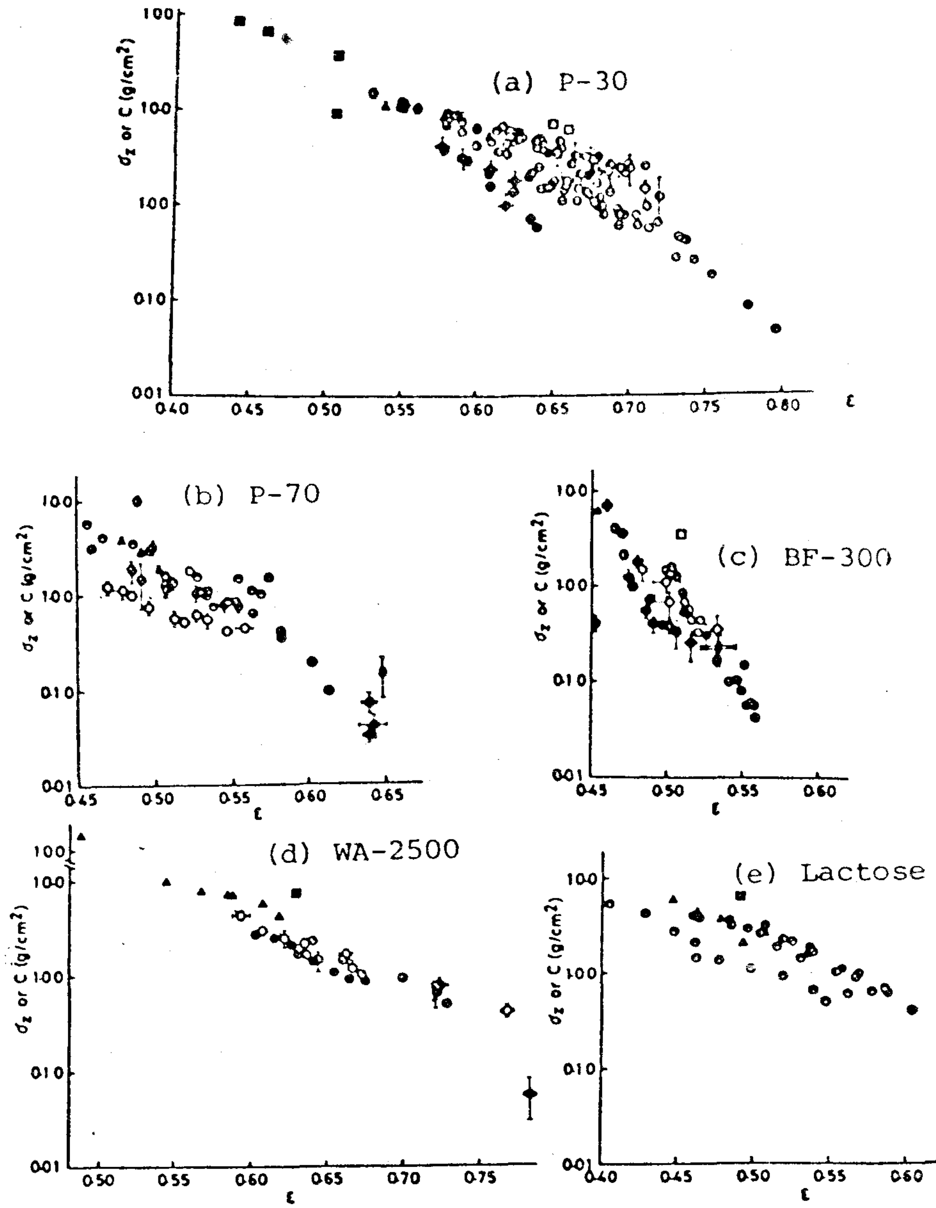

Fig. 8. Variation of tensile strength σz or shear strength C with porosity ε.

commercially available. However, there still remain some problems in the standardization of the measuring procedure by which reproducible results can be obtained. I wish to thank all the members of the Working party for providing the data and stimulating discussions on this review.

II. Effect of Temperature on the Adhesive Force of Organic Powders

II-1. The Effect of Temperature on Tensile Strength and Surface Area of Some Organic Powders

Using ethyl p-hydroxybenzoate (Et-POB) and methyl p-hydroxybenzoate (Me-POB), the effect of temperature on tensile strength as the surface area was examined. It was found that the tensile strength (σ) increased and the surface area (S_w) decreased with an increase in heating time. These results can be explained in terms of the sintering mechanism.

The tensile strength for both powders showed maxima between a homologous temperature of 0.9 and 0.95. This observation suggests that the packing structure of a powder bed alters at temperatures closer to their melting point; that is, agglomerates may be formed by a marked increase in adhesive force between particles.

There is an approximately linear relationship between the reduction in surface area(ΔS_w) and tensile strength. This may be attributed to the fact that the increase in tensile strength is due to the increase in contact area between particles which is proportional to ΔS.

II-2. Measurements of the Adhesive Force between Particles of Powdered Organic Substances and a Glass Substrate by Means of the Impact Separation Method. I. Effect of Temper-ature

The effect of temperature on the adhesive force between particles of powdered organic substances (butyl p-hydroxybenzoate and sulfadimethoxine) and a glass substrate was investigated by means of the impact separtion method using a pendulum-type shock testing machine.

At elevated temperature, a remarkable increase in adhesive force with heating time was observed. The adhesive force at a given heating time increased with rising temperature and a linear relationship existed between the logarithm of adhesive force and the reciprocal of temperature (°K) for each sample. The results were interpreted in terms of the growth of a solid neck between a particle and the substrate.

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