A Method for Determining Shot in Refractory Fibers

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Published methods for determining shot content for refractory wools and other inorganic fibers such as glass wool, slag wool, etc., were reviewed and a method of determination was devised. It is based on crushing of the wool to free the shot from the fibers and elutriating to separate the fibrous material from the shot. This procedure is suitable for use as a control test and also for evaluating wool quality. A definition of shot can be based upon a pronounced change in the slope of the elutriation curve. For all the wools tested such a change occurred, thus indicating that shot content can be defined on an empirical basis.

I. Introduction

In the production of refractory fibers, molten glassy material from a melting furnace is introduced into a blast of air or steam. Attenuation results in the production of thread-like fibers primarily, but an appreciable amount of the glassy material appears in the product in the form of globules or so-called shot. Inasmuch as this shot has no insulating value compared with the fibers, studies were made to avoid its formation by investigating various blowing techniques. In developing a process for the production of kaolin fibers, the need for a rapid and precise method for determining shot content was of great importance.

Trade specifications[1] limit by definition shot as consisting of material separated from the pulverized fibers by sieving on a 50-mesh sieve, i.e., particles that are greater than 0.3 mm. (300 μ) in diameter. However, such definition is unrealistic in not taking into account finer shot which is also objectionable. For example a commercial refractory fiber, containing only 2 to 3% shot when graded by a test using the 50-mesh sieve, actually was found to contain 30% shot when graded according to a more rigorous test method.

II. Methods for Shot Determination

Shot, although present in part as free unattached spherical particles, also may be attached to fibers and usually must be detached prior to mechanical separation from the fibers.

Shot is best freed from the wool by crushing the fibers selectively. This may be done by grinding the sample against a screen surface with a rubber stopper, as specified in a Commercial Standard test, by blowing the wool against a sieve with a blast of air, or by crushing in a special crusher like that which was used by the Bureau of Mines.[2] The last named method appears better suited for a satisfactory test procedure.

After the wool is crushed and the shot separated from fibers in the wool crushing operation, shot may be separated from the wool fragments by air or water elutriation. An air-blast method for separation was investigated.

A 20-gm. sample of wool was crushed in a special crusher, to be described later, placed between two nested 3-in., 150-mesh sieves, and the wool was blown from the shot by a high-velocity air blast directed against one of the sieve faces. This procedure, although suitable for rough checks is dusty, hard to standardize, and quite abrasive to sieve cloth.

In an original procedure for separating shot from wool by a wet method, a wool sample was disintegrated by processing it in a small paper beater. The beater blades broke up the fibers and afforded a slurry of the broken fibers and shot in water from which the wool was separated by elutriation.

At this stage in the development, a method proposed briefly by the Bureau of Mines[2] in one of its publications, was considered and further development of that test method undertaken.

III. Babcock & Wilcox Shot Tester

A 20-gm. wool sample is placed in a compression cylinder consisting of plunger and cylinder (Fig. 1). This assembly, made of hardened steel, is placed in a laboratory press and the wool is crushed by applying a load of 2000 p.s.i. on the wool. The pressure is not particularly critical and may be varied from about 1000 to 3000 p.s.i. The broken wool fragments act as a cushion to prevent shattering of the shot particles. Pressure is usually applied twice and the plunger is rotated 90° between the first and second application of the pressure.

The crushed wool sample and the contained shot is added to the water in a dispersion flask (Fig. 2); a wetting agent is added to the water when there is a water-repellent coating on the wool. This flask or dispersion chamber, with the shot trap in place and the hand used to cap the opening, is shaken until the solids are well dispersed. An elutriation column is then inserted and the water line is connected. In event that foaming is excessive, a drop of anti-foaming agent may be added to destroy the froth before connecting the column. Although a silicone anti-foam proved satisfactory, xylene was equally good.

Water pressure is applied to provide a churning action and elevate the shot and wool fragments into the column. The water flow is adjusted to the desired flow rate as the water level is brought up to the top of the column.

The water flow rate is measured either with calibrated overflow siphons or by the readings of a mercury-manometer flow meter. An overhead constant-level tank can be used to

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Presented at the Fifty-Sixth Annual Meeting, The American Ceramic Society, Chicago, Ill., April 22, 1954 (Refractories Division, No. 20).

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[1] U. S. Department of Commerce, National Bureau of Standards, Commercial Standard CS131-46 "Industrial Mineral Wool Products, All Types—Testing and Reporting," U. S Government Ptg. Office.

[2] C. H. Gorski, O. D. White, and M. C. Moreland, "Raw Materials for The Mineral Wool Industry," *U. S. Bur. Mines Rept. Invest.* No. 4821, 8 pp. (1951).

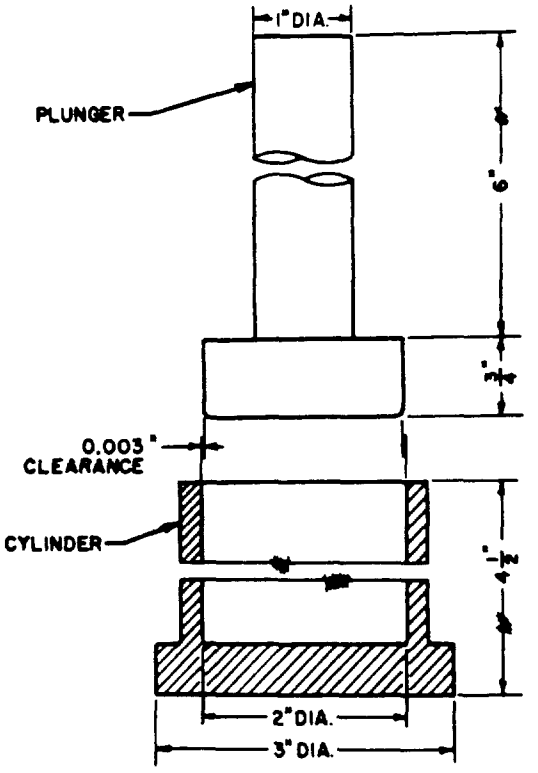

Fig. 1. Plunger-cylinder device for preparation of shot-test specimens.

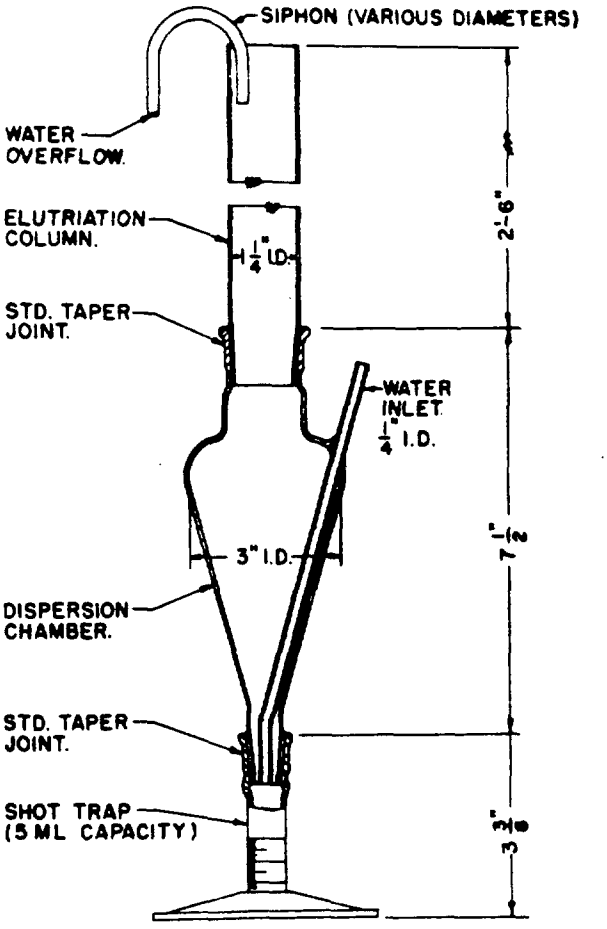

Fig. 2. Shot tester.

supply water at constant pressure to the system. In general, the water tank is not necessary if the water-line pressure is reasonably uniform.

Elutriation is continued at the fixed rate required to separate wool from shot. A water velocity of 215 cm. per minute (1700 ml. per minute from a column 1.25 inches I.D.) was found to be the most satisfactory rate to give the best separation of wool from shot, usually requiring about 5 minutes. Tests showed that there was little change in the amount of shot collected at a given flow velocity in the column after this stabilization period. This velocity of water in the dispersion chamber is sufficiently high that the fine fragments of wool are swept up into the column and out of the system.

After elutriating the wool, the water flow is stopped and the shot suspended in the water settles into the shot collector, the water is siphoned out of the system through the water-inlet opening, at a slow rate to avoid drawing out shot, and the trap is detached. The volume of shot is measured after compacting it firmly with the blunt end of a glass rod. The measured volume of shot is calibrated in terms of its weight and calculated to the shot content by weight of the sample. A calibration was made for each of various kinds of wool tested. For aluminum silicate fibers wherein the mole ratio of alumina to silica is 1:2, one cubic centimeter of wet, compacted shot weighs 1.34 gm. on a dry basis and is equivalent to 6.7% shot, based upon a 20-gm. sample of the original wool. This direct volumetric procedure eliminates the need for drying and weighing the sample to calculate the shot content.

IV. Shot Content of Fibers

Shot may range in size from several μ to $^1/_8$ inch in diameter and may include even larger glassy shards. The velocity of the ascending water column in the elutriator will determine the effective size of shot remaining in the system, which size may be calculated approximately by Stoke's Law, when the Reynolds' number for the particle is between 0.40 and 10.[3]

The calculation of shot content was made on the basis of the relationship between observed shot content and elutriation flow rate. The water velocity to give clean shot in the trap and clean wool in the overflow (Fig. 3) appeared to be 215 cm. per minute, equivalent to a shot diameter of 195 μ. For the purpose of making this photograph, both the wool and the shot were recovered.

When typical data for percentage shot separated was plotted against the flow rate of water in the column, or shot diameter as calculated from Stoke's Law, the curved lines shown in Fig. 4 were produced. These lines, although they show a difference in slope between the upper part of the curve, for wool rods, and the lower part of the curve, for shot, do not clearly show the place of change from wool to shot.

However, when similar data were plotted on log-log paper, as in Fig. 5 (each datum point is the average of several determinations), straight lines were drawn which represent a range of rods, an intermediate range, and one for shot particles. The selected flow rate of 1700 ml. per minute falls in the intermediate range. Similar plots of several refractory wools showed that this selected flow is one which may be used to compare the shot content of various wools. Some wools which may have been subjected to auxiliary cleaning will give values which indicate that some other water flow rate

[3] A. F. Taggart, Handbook of Mineral Dressing, J. Wiley & Sons, New York, 1927, p. 8-43

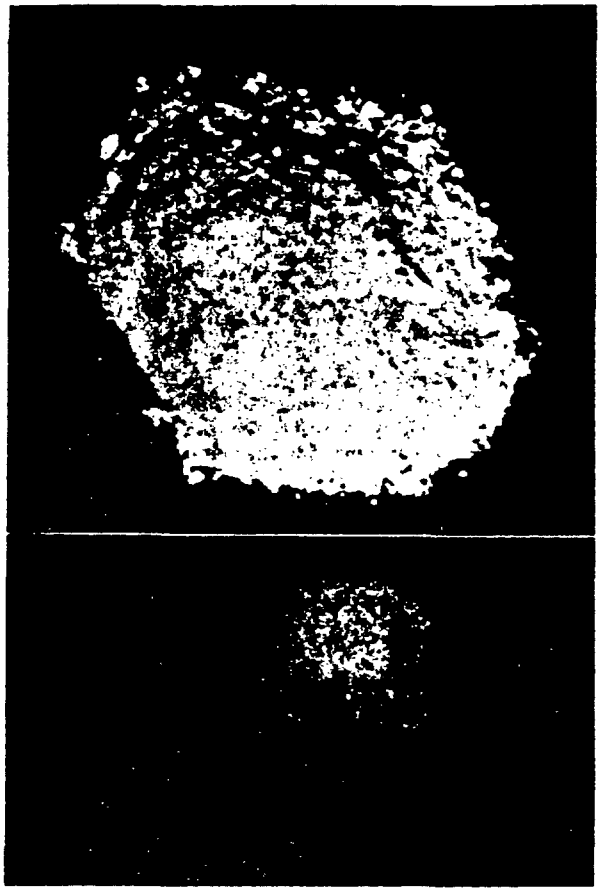

Fig. 3. Wool and shot from a separation at 1700 cc. per minute.

might be preferred to indicate the optimum shot content. However, the 1700 ml. per minute rate appeared best for comparing various wools.

In the particular column that is described, 1.25 inches in diameter, the Reynolds' number for the particle was 7.6; a condition which satisfies the requirement for applying Stoke's Law.[3] The corresponding Re value for the column was 1235, which is within the range of streamline motion.[4]

Table I summarizes the data obtained in determining the relationships between water flow in the elutriator, particle diameter calculated from Stoke's Law and Reynolds' numbers. The pipe Reynolds'[5] number defines laminar flow in the elutriator.

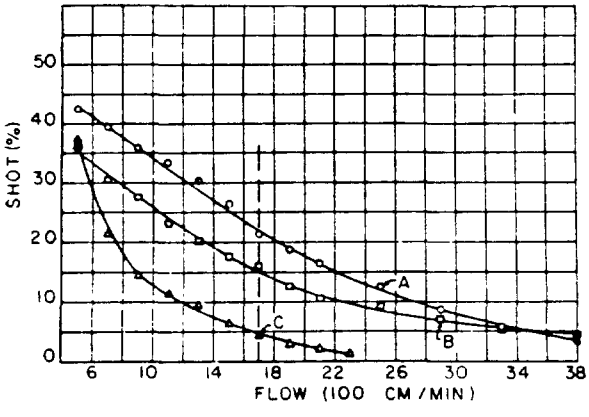

Fig. 4. Measured shot content vs. flow rate for several wools.

Table I. Elutriation Conditions Defined by Water Flow

Water flow			Particle diameter "d" Microns	Tyler Sieve No	Reynolds' No., Re	
cc./min	cm. min.	cm./sec			Particle	Pipe
500	63.1	1.052	106	140	1.21	365
700	88.4	1.473	125	120	2.00	510
900	113.6	1.894	142	102	2.92	655
1100	138.9	2.315	157	93	3.96	800
1300	164.1	2.736	170	85	5.07	945
1500	189.4	3.157	183	78	6.30	1090
1700	214.6	3.577	195	74	7.60	1235
1900	240	3.988	206	71	8.98	1380
2100	265.2	4.419	216	68	10.40	1530
2500	315.7	5.261	236	62	13.55	1820
2900	366.2	6.103	254	58	16.90	2110
3300	416.7	6.945	271	55	20.51	2400
3800	479.8	7.996	291	52	25.40	2770
4300	542.9	9.049	309	49	30.48	3130

* Underscoring represents the selected test conditions.

Stoke's formula for spherical grains is:

$$V = 2gr^2(\rho_s - \rho)/9\eta \qquad (1)$$

V = velocity of particle with respect to the liquid, cm./sec.,
g = acceleration due to gravity, 980.67 cm./sec.^{2},
ρ_s = density of particle, 2.58 g./cc.,
ρ = density of liquid, water at 24°C. = 0.997 g./cc.,
η = viscosity of liquid, water at 24°C. = 0.009142 poise (g./cm. sec.),
r = radius of particle, cm.,
d = diam. of particle, microns,
D = diam. of tube, cm.

$$d = 102.9\sqrt{V} \qquad (2)$$

$$\text{Re (particle)} = dV\rho/\eta = 0.0109\ Vd \qquad (3)$$

$$\text{Re (tube)} = DV\rho/\eta = 346\ V \qquad (4)$$

The conditions in the column are such that Stoke's Law for viscous flow applies. The particle Reynolds' number is

[3] J. M. DallaValle, Micromeritics, The Technology of Fine Particles, Pitman Publishing Corp., New York, 1943. 428 pp.; *Ceram. Abstr.*, **22** [6] 106 (1943).

[4] J. H. Perry, Chemical Engineers' Handbook. 3d edition. McGraw-Hill Book Co., Inc., New York, 1950. xv + 1942 pp.; *Ceram. Abstr.*, **1950**, July, p. 154*h*.

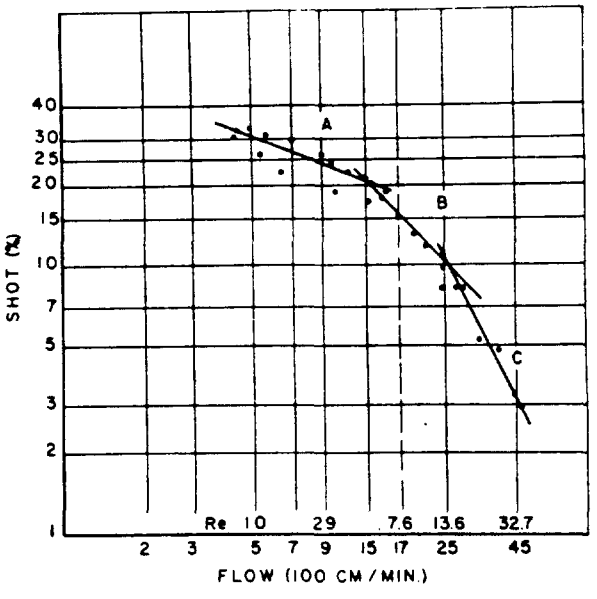

Fig. 5. Shot vs. water flow and particle Reynolds' number. (A) Range of rods; (B) intermediate range; and (C) range of spheres.

Barium Titanate and Other Ceramic Ferroelectrics: V. Theory

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Elementary theory of dielectrics is reviewed with emphasis on the atomic mechanisms involved and the concept of the internal field. The various attempts at a theoretical explanation of the behavior of barium titanate are reviewed and the present status of the theories summarized.

I. Introduction

This paper is an attempt to summarize the present state of the theoretical explanations for the ferroelectric behavior of barium titanate. This will not attempt a detailed presentation of each of the theories, but rather hope to give a general idea of the basic mechanism and formulation of each of the theories and the conclusions to which they lead. For more detailed information on the different theories, the reader is referred to the excellent critical review by Jaynes[1] or to the original papers as hereinafter cited.

II. Dielectric Theory

Before discussing the theories of barium titanate, it will be necessary to review briefly the general theory of dielectrics.

(1) Macroscopic Phenomena

If a voltage E is applied to a condenser consisting of two parallel plates with a vacuum between them, a certain amount of charge Q_f flows into the condenser (Fig. 1A). The voltage and the charge are related by the equation

$$Q_f = CE \tag{1}$$

where C is defined as the capacitance of the condenser.

If (with the same voltage applied) a dielectric material is inserted between the plates instead of the vacuum, a further amount of charge Q_b will flow into the condenser so that the total charge, Q_t, is now related to the voltage by the equation

$$Q_f + Q_b = Q_t = C'E \tag{2}$$

where C′ is the capacitance of the condenser with the dielectric between the plates. The ratio of capacitances with and without the dielectric is called the dielectric constant, K, of the material and is defined

$$K = \frac{C'}{C} \tag{3}$$

The extra flow of charge into the condenser caused by the insertion of the dielectric is due to the fact that in the dielectric there is a movement of charge—negative charges move toward the positive plate of the condenser, and positive charges move toward the negative plate. These charges neutralize the extra charge flowing into the condenser from the external circuit (Fig. 1B).

Note that the movement of the charge in the dielectric is limited. If this were not so, and the charges were free to move through the dielectric, then the material would behave as a resistance, not a capacitance.

Associated with the free charge Q_f (flowing irrespective of the dielectric in the condenser) is an electric field vector E proportional to the free charge density. Associated with the excess or bound charge Q_b (which flows because of the charge movement within the dielectric) is a polarization vector P proportional to the bound charge density. Lastly, associated with the total charge Q_t is a displacement vector D proportional to the total charge density. These vectors are related by the following equations:

[1] E. T. Jaynes, Ferroelectricity, Princeton University Press, Princeton, N. J., 1953. viii + 137 pp.; *Ceram. Abstr.*, 1954, April, p. 72d.

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(Continued from page 327)

within the limit of Re = 0.4 to 10 applicable for Stoke's Law approximations.

No doubt a separation of wool fragments from shot could be effected under condition of turbulent flow, but that application would require a less compact apparatus than has been developed here.

Table II. Sieve Analysis of Shot Separated from Kaowool

U. S. Sieve No.	Equiv. diameter of particle, microns	Shot remaining on sieve, %
20	840	3.9
30	589	14.2
40	417	33.3
50	295	63.8
70	210	84.7
100	149	95.5
140	105	98.6
170	88	99.7
200	74	99.9
−200	−74	0.1

The shot test is simple, performed rapidly, and is more rigorous in establishing limits on shot size than others that have been investigated. The limiting size of 195 μ is close to 75-mesh rather than the 50-mesh sieve size specified in the Commercial Standard test.

A sieve analysis of the shot particle size distribution is shown in Table II. Although about 15% of the shot separate is finer than indicated by the Stoke's Law calculation, nearly 100% is coarser than 105 μ. The deviation from Stoke's Law is no greater than could be expected when it is considered that the shot particles are not perfect spheres.☆

Acknowledgments

Mr. F. J. Hartwig, Babcock & Wilcox Company, contributed valuable suggestions and criticism. Mr. Allen Colley, Babcock & Wilcox Company, performed most of the test work. Mr. R. G. Knickerbocker, U. S. Department of the Interior, Bureau of Mines, furnished information about the method used in his organization for crushing wool samples.

American Ceramic Society Bulletin
Volume 34, 1955
pgs. 325-328