

**Bureau of Mines
Report of Investigations 5203**



**LABORATORY RESULTS ON TESTING
MINERAL-WOOL RAW MATERIALS**

BY H. KENWORTHY AND M. L. MORELAND

United States Department of the Interior — March 1956

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UNITED STATES DEPARTMENT OF THE INTERIOR
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SUMMARY

Laboratory experiments were conducted on several materials to determine their suitability for the production of mineral wool. By blending raw charges in the proper proportions and in some instances by adding lime, acceptable wools were made from samples originating in Alaska, Florida, Texas, and Virginia. It was shown that satisfactory wools can be produced from many kinds of raw materials if the ratios of acidic and basic constituents are properly maintained.

INTRODUCTION

The Federal Bureau of Mines has facilities at the Mississippi Valley Experiment Station, Rolla, Mo., for examining and testing mineral-wool raw materials. Samples are evaluated through small-scale wool-blowing experiments, aided by petrographic, chemical, and physical analyses of the raw materials and finished wools. When enough experimental data have been accumulated they are made public through Government reports and periodicals. This report of investigations follows that published in 1951^{3/} and presents test data on samples received and examined since 1951.

The utilization of large tonnages of insulating materials is one of many conservation practices advocated and encouraged by the Bureau of Mines. Mineral wools have many applications in connection with conservation of heat and power. They have other excellent qualities, such as fire and corrosion resistance, sound deadening, and complete resistance to molds or decay, and they form structural barriers against insects and vermin.

Because of its low bulk density and the fact that it is deformed by crushing mineral wool is in a high transportation-tariff class. For this reason, mineral-wool plants are found near the markets of population centers, subject to the limitations dictated by climate, fuel, raw materials, and industrial activities. Since raw materials suitable for producing mineral wool are widespread, interest in evaluation of such materials is evinced on a national basis.

LABORATORY INVESTIGATIONS

Analyses

Petrographic analyses were conducted on most of the samples submitted to obtain physical information and to identify the chief minerals present.

Chemical analyses generally were made on raw materials to determine the relative quantities of acidic and basic compounds present.

^{3/} Gorski, C. H., White, O. D., and Moreland, M. L., Raw Materials for the Mineral-Wool Industry: Bureau of Mines Rept. of Investigations 4821, 1951, 8 pp.

These preliminary analyses were the bases for formulating charges. They indicate the type and amount of material required as an additive to balance and flux the experimental charges.

Sample Preparation

Sands or finely divided raw materials were used in the as-received condition. Coarse raw materials were crushed through 10-mesh; and, if blending was necessary, the desired portions were thoroughly mixed before they were charged into the heated melting crucible. The crucible was made from crucible-grade solid graphite rod stock. Burnt lime made by heating a high-grade limestone was used in certain experiments.

Converting Raw Materials to Mineral Wool

Major variables to be considered when converting raw materials to mineral wool are: Charge composition, pouring temperature, molten stream temperature, pouring rate, type of nozzle, blowing medium, and blowing pressure.

Charge compositions that fall within certain wide ranges analytically will result in satisfactory wools. Minimum and maximum limits of composition have been calculated by Thoenen,^{4/} Logan,^{5/} and Goudge.^{6/} Similar figures have been published by Lamar, Willman, Fryling, and Voskuil.^{7/} The latter base their calculations on four component diagrams, backed by experiments with synthetic mixtures of nearly pure compounds. Their results are repeated here as ranges of composition over which mineral wool may be blown. These ranges are: SiO₂ 35-65, R₂O₃ 0-33, CaO 5-50 and MgO 0-32. The R₂O₃ group is definitely amphoteric in character; and, although usually calculated as an acid in acid-to-base determinations, the group will exhibit basic qualities in the presence of strongly acidic materials.

Acid-to-base ratios are a factor in controlling the viscosity of a melt and hence greatly influence the average fiber diameter of the blown wool. As viscosity of the molten material increases, fiber diameter of the finished wool increases. Size of shot becomes larger with an increment in viscosity. Viscous melts are usually associated with acidic conditions. As basic constituents of a melt are increased the viscosity decreases, the molten material pours in a broken stream, fiber diameter of the wool decreases, fibers are shorter, and more flakes and dust appear in the blown wool.

Another consideration of prime importance is temperature. Within limits, the viscosity of a molten charge may be regulated by varying the temperature. An increase in temperature increases the fluidity or decreases the viscosity of a melt. As previously stated, average fiber diameter is determined primarily by viscosity. More shot is formed when the wool is blown at low temperatures.

^{4/} Thoenen, J. R., Mineral Wool: Bureau of Mines Inf. Circ. 6142, 1929, 13 pp.

^{5/} Logan, W. N., The Mineral-Wool Industry in Indiana: Preprint AIME, 1932.

^{6/} Goudge, M. F., Raw Materials for the Manufacture of Rock Wool in the Niagara Peninsula of Ontario: Canadian Bureau Mem. Ser. 50, 1931, 18 pp.

^{7/} Lamar, J. E., Willman, H. B., Fryling, C. F., and Voskuil, W. H., Rock Wool From Illinois Mineral Resources: Illinois Geol. Survey Bull. 61, 1934, 262 pp.

Molten stream temperature at the instant before impact with the blowing jet depends on the charge temperature, the height of fall from the crucible lip to the blowing jet, and the size of the molten stream. The longer the free-fall measurement and the thinner the poured stream, the more the stream will be cooled before it contacts the blowing jet. Viscosity increases with cooling, hence fiber diameter is affected by the height of stream fall.

Rate of pour relates to the amount of material that may be passed through a furnace and may be given in pounds per minute, tons per hour, seconds per unit of weight, etc. Under conditions prevalent in the laboratory, rate of pour affected fiber diameter but very little; however, as the rate of pour was increased, shot formation increased. Shot formation rapidly increases as the amount of molten material delivered to the blowing jet exceeds the potential energy of the steam to convert the fused material to fiber.

Types of blowing nozzles are numerous, and no universal opinion exists on the most meritorious design. Flat nozzles, Vee nozzles, and ring nozzles with modifications through use of slits or holes have been advocated for blowing. Some economy in the use of blowing medium can be expected with nozzles designed with this characteristic in mind. Lower pressure drop across the nozzle will occur with smaller openings. The common blowing mediums are air and dry steam. On a volume basis air is less expensive to produce than steam; however, steam provides a longer viscous range while the molten material is being fiberized. Thus when steam is used as a blowing medium more time is provided on fiberizing the melt as it is poured. The interval when molten droplets are in a position to be fiberized is extremely short, so any increment provides a higher conversion from melt to wool.

Blowing pressure at the nozzle is a factor that influences the average fiber diameter and shot content when mineral wools are manufactured. As nozzle pressure is decreased the fiberized material becomes larger in diameter, and shot content increases. An example illustrates this. During blowing experiments on a copper slag an average fiber diameter of 2-1/2 microns was obtained at a nozzle pressure of 45 p.s.i. The shot content was 50 percent. Lowering the nozzle pressure to 30 p.s.i. and the pouring temperature by only 35° produced a wool having an average fiber diameter of 4 microns and an attached shot content of 70 percent. Free shot increased in the same order.

Charges of about 300 grams of raw materials were placed in the graphite crucible, which had previously been heated to the desired melting temperature, and rapidly melted at a predetermined temperature by a small, high-frequency induction furnace. Because black body conditions were not present within the crucible and a thin white fume was generally emitted from the charge and, in addition, readings were taken on the charge surfaces, the optical temperature readings were considered as relative and are shown as "apparent temperatures." Temperatures were held at the predetermined level for a few minutes before pouring to assure thorough liquation, uniform temperature, and homogeneity of the melt. During pouring optical temperature readings were taken of the stream of molten material before it came into contact with the blowing jet. The time required to pour the charge was measured with a stopwatch. The size of the molten stream that may be satisfactorily fiberized determines the rate of pouring a melt. Furnace capacity is, of course, limited by the melt-down rate and optimum pouring rate. The laboratory wool-blowing apparatus is shown in operation in figure 1.

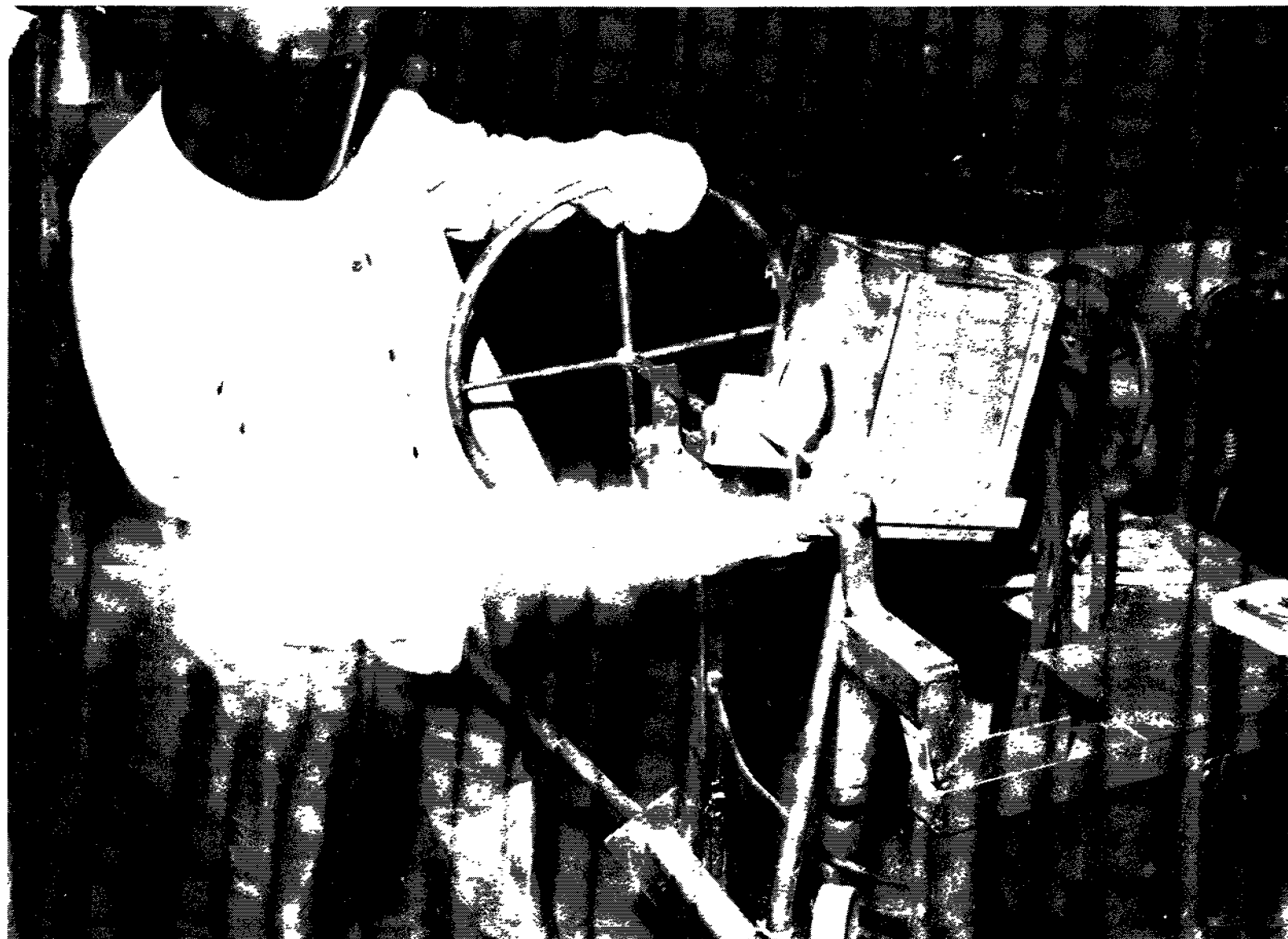


Figure 1. - Pouring and blowing mineral wool with experimental apparatus.

The molten charge was blown as it fell in a thin uniform stream just in front of a Vee nozzle from which live steam issued at 30 to 50 pounds per square inch. The impact of the steam on the liquid caused fiber and shot to be formed. This was wafted by the water vapor into a large, sheet-iron horn from which it was later collected for examination and evaluation. A highly skilled operator poured the liquid and manipulated the steam jet manually. No oils were sprayed onto the wool.

Evaluation of Finished Wool

The matted wool fibers and the loose shot were removed from the horn separately and carefully weighed. The percentage of "free" or "loose" shot was determined on the basis of the total weight of the two products. The blown wool also contained a substantial quantity of included shot, which is subsequently designated as "attached shot." Attached shot occurred largely as spherical, ellipsoidal, or pear-shaped droplets, which were usually attached to a fiber by a short tapered section of wool. The percentage of the wool that was true fiber was determined by hydraulically crushing a weighed portion of the wool under 8,000 p.s.i. to break the shot free from the fiber, then hydraulically removing the broken fiber from the shot. A sample was crushed by placing it in a machined-steel cylinder and inserting a close-fitting plunger to which pressure was applied with a Carver press.

Separations were made by using 500-cc. burettes as hydraulic classifiers. Uniform flow control of the incoming classifier water was maintained by using calibrated siphons consisting of lengths of glass tubing bent into a J shape and inverted. Siphon discharge rates were made variable by cutting the shorter leg of the siphon to different lengths. The short leg was immersed in the burette washer column by hanging it over the lip of the burette. Siphons were used in multiple, generally 2 or 3, thus providing flexibility to the rate of flow, which was adjusted to the average fiber diameter.

Microscopic examination of the separated shot and fiber shows that this method gave a superior, though not perfect, differentiation between the two physical phases. This method of true fiber determination usually showed several percent more shot than determinations by other accepted methods. Often these methods did not remove the extremely fine shot from the fiber; hence, some shot of little insulating value has been reported in technical reports as fiber. Microphotographs of mineral wool, separated shot, and fibers are shown in figures 2, 3, and 4. Subject matter is from Alaskan test 51. Magnifications are 200 diameters.

Average diameter of the wool fiber is probably the primary criterion in evaluating mineral wools. This physical determination was made by measuring the diameter of a number of fibers and calculating an arithmetical mean. Measurements were made with a microscope fitted with a filar micrometer eyepiece and a rotating mechanical stage.

Among other factors affecting the quality of mineral wool are: Shot content, color, odor, stability of the chemical composition, bulk density, and fiber brittleness. Shot content has little effect on insulating qualities but does increase bulk density and is extremely important to the manufacturer, as it is a direct measurement of the conversion efficiency of his equipment. For general use, colors, may vary from white to black. The most common obnoxious odor will be caused by quantities of sulfur in the wool, which, under certain conditions, may gradually convert to hydrogen sulfide. Use of ingredients with a low sulfur content will usually obviate this formation. Glassy wools sometimes contain enough alkalis to cause chemical instability. This may be evinced by crazing and cracking of the individual

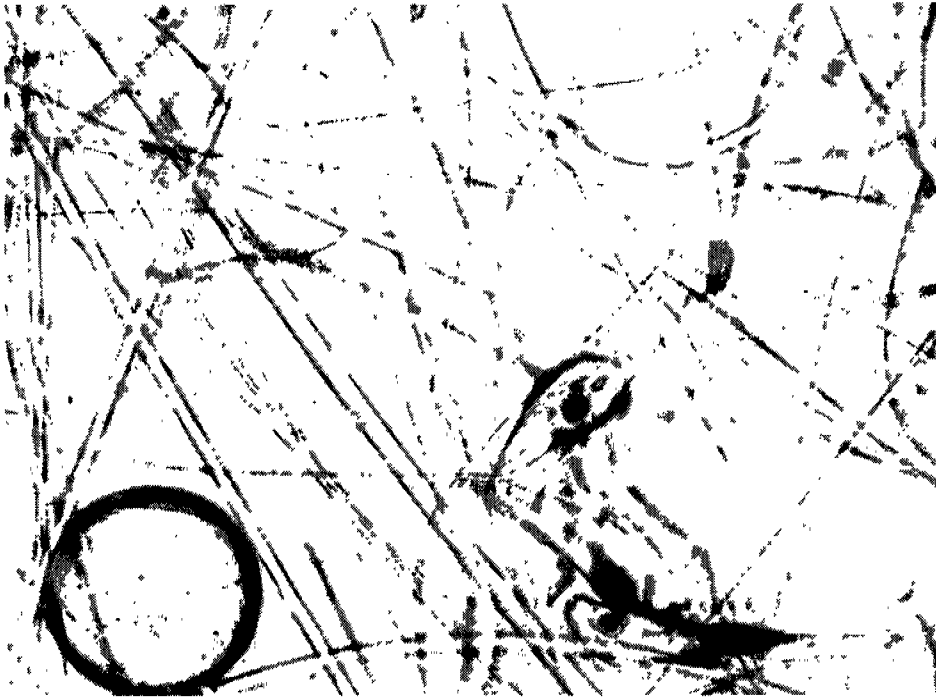


Figure 2. - Raw wool consisting of fiber and attached shot.

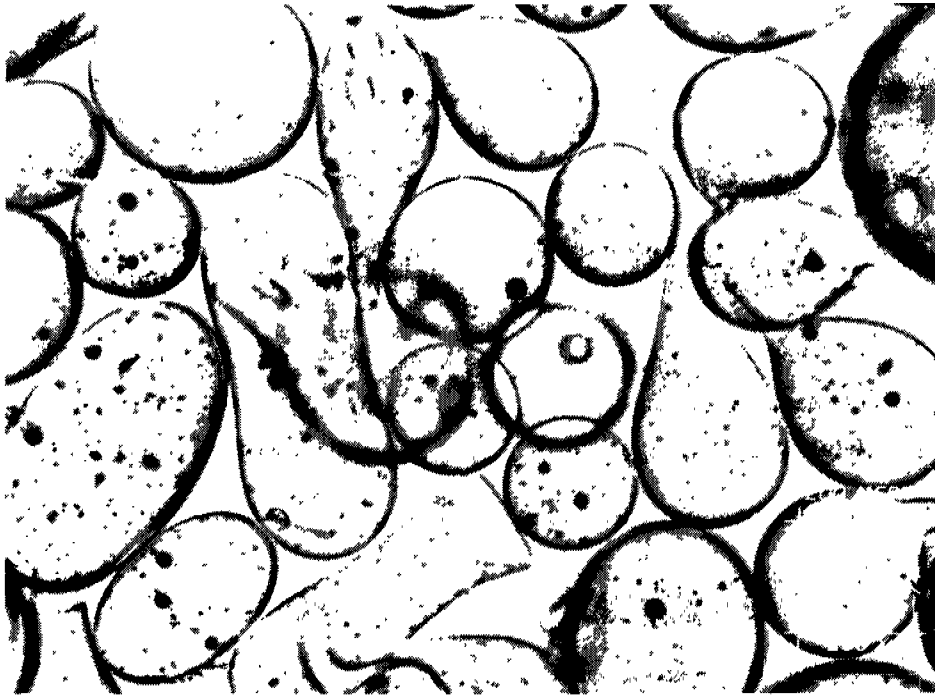


Figure 3. - Shot after elutriation separation.

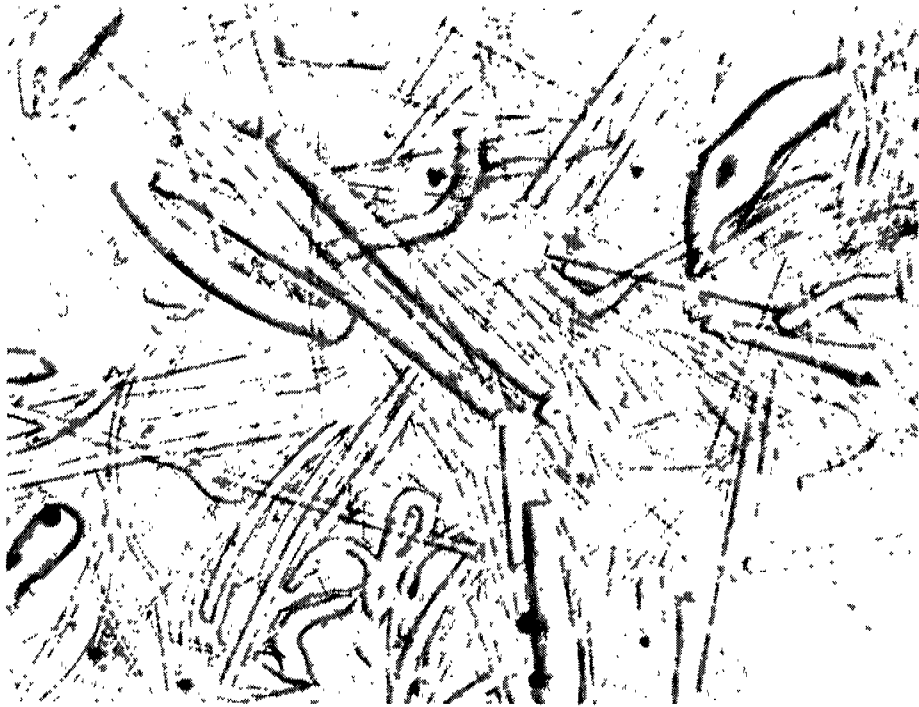


Figure 4. - Fiber after elutriation separation.

fibers or in some instances by efflorescence. An appreciable period usually elapses before symptoms of chemical instability are noticeable. Bulk density depends to a great extent on the handling of wool after it is blown; thus, from the same wool, different manufacturers would have finished products somewhat variable in bulk density. For this reason, no bulk density figures are reported in this paper. Fiber brittleness affects the handling qualities and not the insulating qualities of a mineral wool. Addition of a small quantity of atomized oil lubricates the fibers and increases the flexibility and resiliency of the mass.

Shot-content and fiber-diameter characteristics of laboratory-produced mineral fibers given on the following pages may be compared with the following data on a few commercially produced mineral wools. A sample of eastern-manufacture, formed, block-wool insulation, which had been fiberized by spinning, contained only 4.5 percent of shot. The fiber diameter averaged 6.5 microns, and the fibers were bound together by 12.5 percent of binder. Mineral wool of a southern manufacture was formed by blowing and contained 66 percent of shot with fibers averaging 7.9 microns in diameter. A wool produced by a well-known eastern company contained 66.5 percent of shot and averaged 7.8 microns in fiber size. The Midwest was the source of a mineral wool that averaged 9.5 microns in fiber diameter and contained 66.0 percent of shot. A sample of dark mineral wool was obtained from a mail-order house. This wool averaged 4.8 microns in fiber diameter and contained 66.7 percent of shot. Pelletized mineral wool produced in the Midwest contained 47.3 percent of shot, and the average fiber measured 6.0 microns in diameter. The uniformity of the shot content of the blown wools is noteworthy. Likely the ultimate efficiency in fiberization by blowing is being approached with commercial-sized production units.

Alaska

A shipment consisting of limestone and shale from Alaskan deposits, adjacent to and readily accessible by the Alaskan railroad, was received for evaluation. Chemical analyses of the materials, as furnished by the Bureau of Mines Juneau laboratory, are presented in table 1.

TABLE 1. - Analyses of Alaskan samples, percent

	Insol.	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Ign. loss	P ₂ O ₅	SO ₃	CO ₂	Na ₂ O	K ₂ O
Limestone, raw	2.8	0.8	0.7	1.2	51.0	3.4	41.0	0.002	0.18	39.4	0.4	0.7
Limestone, calcined (calculated).	4.7	1.3	1.2	2.0	85.0	5.7	-	-	-	-	-	-
Shale	75.5	60.0	15.1	9.0	.4	.07	4.3	.32	.21	-	-	-

Interest in mineral wool made from Alaskan resources stems from the facts that no wool is produced in Alaska, this type of insulation can be well utilized during severe Alaskan winters, and transportation costs are quite high on finished insulation imported from the States. Disadvantages of an Alaskan mineral-wool industry include the lack of volume demand owing to low population, undeveloped distribution facilities, and an adverse fuel situation for melting cupola-furnace charges.

Mixtures containing 35 to 80 percent of shale and the balance limestone were melted and blown to mineral wool. Optimum mix was found to be near 50-50. Data pertinent to experiments that produced acceptable wool are shown in the first portion of table 2.

TABLE 2. - Mineral wool from Alaskan shale and limestone

Test No.	Weight, percent		App. temp., °F.	Steam pressure, p.s.i.	Pouring time, seconds	Shot, percent		Fiber diam., microns	Remarks
	Shale	Lime-stone				Free	Attached		
33	50	50	2,810	40	34	13	42	10.5	Good-appearing white wools
41	50	50	2,900	40	22	15	50	9	
50	45	55	2,800	40	20	17	46	9	
51	45	55	2,850	32	27	13	42	8	
47	40	60	2,690	40	29	20	47	8	
30	80	20	2,760	-	-	-	-	-	Too viscous to pour.
37	70	30	2,950	42	45	11	-	-	Very coarse fiber.
38	70	30	3,000	48	60	11	-	16	Coarse and brittle.
34	60	40	2,850	42	36	13	-	-	Charge too viscous.
35	60	40	2,870	45	50	10	32	13	Coarse fiber.
36	60	40	2,900	44	46	15	-	-	Do.
31	50	50	2,700	44	34	9	36	11	Slightly coarse.
32	50	50	2,760	48	36	9	36	14	Coarse fiber.
42	50	50	2,670	42	30	16	41	13	Do.
49	45	55	2,760	42	30	14	56	9	High shot.
40	40	60	2,600	45	27	16	45	13	Coarse fiber.
48	40	60	2,650	44	21	24	60	-	Pour too fast.
39	40	60	2,700	45	33	15	52	11	Slightly coarse.
46	40	60	2,760	44	22	22	60	5	High shot.
44	35	65	2,700	42	25	-	-	-	Charge too basic. Poured in drops and lumpy.
43	35	65	2,750	42	24	-	-	-	Do.
45	35	65	2,830	42	-	-	-	-	Do.

Using over 50 percent shale in the charges produced an acidic condition that resulted in viscous molten charges that poured poorly and blew into coarse fibers. Incorporating less than 40 percent of shale in the charge resulted in a blend that was too basic. Molten charges in this condition will not pour in a continuous stream. The molten material discharged in lumps and drops, which upon blowing produced either excessive quantities of shot or coarse, short, brittle fiber.

Florida

Samples of three raw materials were submitted in a group. These were: (1) A phosphatic smelter slag from electric furnace production of metallic phosphorus, (2) a staurolite concentrate from mineral-dressing operations, and (3) a siliceous kaolin clay from an actively mined deposit.

Petrographic examinations showed: (1) The slag to consist chiefly of psuedo-wollastonite interspersed with a few high-phosphorus metallic inclusions; (2) the concentrate was largely staurolite, kyanite, sillimanite, and tourmaline, with small amounts of other heavy minerals; (3) the light clay material contained considerable free quartz mixed through the kaolin matrix. Chemical analyses of the samples are shown in table 3.

TABLE 3. - Analyses of Florida samples, percent - Rand

	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	P ₂ O ₅	F	TiO ₂
Slag	42.1	1.28	0.16	51.6	0.7	2.3	2.5	4.2
Staurolite ...	27.5	48.6	14.7	.25	2.1	-	-	-
Clay	78.8	15.0	.7	.31	.35	-	-	-

Utilizing the chemical analyses to compare possible blends of 2 or 3 of these materials with the ternary diagram for Al₂O₃-SiO₂-CaO shows that it should be possible to blow mineral wool from a number of blend combinations. In charge-balance calculations the iron oxide in the staurolite was considered equivalent to lime.

A variety of blends of the phosphate slag, staurolite, and clay was found to produce acceptable wool. The testing had two chief objectives: One was to attempt to produce superduty wool from the staurolite concentrate alone, and the other was to use the phosphate slag as a primary constituent to the fullest extent.

Direct melting and blowing of the staurolite concentrate were unsuccessful up to 3,600° F. This material began to fuse about 3,200° F., with all minerals apparently molten at 3,400° F.; however, an increase of 200° to 3,600° did not lower the viscosity appreciably. When the crucible was tilted, the charge flowed like a plastic rather than a liquid. Alumina and magnesia crucibles were fluxed by the charge. Silicon carbide and graphite crucibles reacted rather violently with the iron oxide phase of the charge. Large quantities of gas were given off, often blowing much of the charge from the crucible, especially at temperatures above 3,000°. Some metallic iron was formed. By blending in small percentages of fluxes, as shown in table 4, blowable melts were obtained. Finished wools were slowly heated until softening of the fibers was noted. The data in the column "First fiber deformation" shows that these wools, although not of Superduty grade with respect to heat, are far superior to white wools and better than most dark wools in heat-resisting qualities. The first portion of the table consists of the satisfactory experiments.

Results of experiments in which phosphatic slag was successfully blown to mineral wool after blending with either staurolite concentrate or clay are shown in table 5. The table is divided into two parts. The first part presents data for experiments that gave the most satisfactory wools. The second part deals with tests that gave marginal and undesirable results. The wool produced in these experiments was white and slightly fleecy; that is, it contained a few particles of flats or flakes.

A suite of two samples, consisting of an impure limestone and a "river sand," was submitted for experimental evaluation.

The materials were examined petrographically. The limestone was found to consist chiefly of clay, quartz, feldspar, and dolomite, whereas the sand was relatively clean, fine-grained material containing only silica plus scattered grains of iron oxides and iron-stained quartz. The fine grain size of the sand would limit the amount that could be satisfactorily added to a cupola furnace without some type of agglomeration treatment.

Partial chemical analyses, shown in table 6, were made on the two materials.

TABLE 4. - Mineral wool from Florida staurolite concentrate

Test No.	Weight, percent				App. pour temp., °F.	Steam pressure, p.s.i.	Wool properties			
							Temp., °F.		Attached shot, percent	Fiber diameter, microns
	Staurolite	SiO ₂	CaO	CaF ₂			First fiber deformation	Became molten		
12	68	16	16	-	3,100	52	1,795	2,770	59	5.4
21	70	17	5	8	3,230	50	1,850	2,930	51	8.5
22	76	15	3	6	3,540	50	1,900	2,950	58	7.3
13	80	8	12	-	3,450	52	1,760	2,950	46	9.1
14	82	6	12	-	3,315	50	1,830	3,020	51	6.3
15	84	3	13	-	3,365	50	1,905	2,965	45	7.4
16	86	-	14	-	3,360	44	1,875	2,910	48	6.1
17	88	-	12	-	3,405	50	1,875	2,830	55	7.0
9	70	15	15	-	3,180	40	1,840	2,770	50	8.4

Test No.	Weight, percent				App. pour temp., °F.	Steam p.s.i.	Pour time, seconds	Shot, percent		Remarks
	Staurolite	SiO ₂	CaO	Coke				Free	Attached	
1	100	-	-	-	3,100	-	-	-	-	Charge not molten.
-	100	-	-	-	3,600	-	-	-	-	Too viscous to pour.
2	90	10	-	-	3,430	-	-	-	-	Do.
3	80	20	-	-	3,330	-	-	-	-	Gas forced charge from crucible.
4	80	20	-	-	2,800	Magnesia crucible		-	-	Charge eroded through crucible.
5	80	20	-	-	3,420	Alumina crucible		-	-	Do.
318	78	17	-	5	3,450	-	-	-	-	Viscous after iron reduction.
318A	59	27	10	4	3,530	-	-	-	-	Do.

Test No.	Weight, percent				App. pour temp., °F.	Steam p.s.i.	Pour time, seconds	Shot, percent		Remarks
	Staurolite	SiO ₂	CaO	Coke				Free	Attached	
6	60	26	10	4	2,950	42	15	43	53	Poured quickly upon fusion. Fiber, 9.3 microns.
7	68	22	10	-	3,150	40	24	-	-	Very coarse fiber.
8	<u>1/70</u>	15	10	3	3,150	42	28	-	56	Fiber, 8.7 microns.
9	70	15	15	-	3,180	40	26	-	50	Fiber, 8.4 microns.
10	70	15	15	-	3,270	49	16	23	61	Fiber, 6.2 microns.
11	72	12	16	-	3,277	38	21	31	62	Fiber, 6.1 microns.
18	91	-	9	-	-	-	-	-	-	Gas forced charge from crucible.
19	78	10	5	7	3,500	-	-	-	-	Too viscous to pour.
20	74	10	8	8	3,370	-	-	-	-	Do.
21	70	17	5	<u>2/8</u>	3,230	50	18	48	51	Fiber, 8.5 microns.

1/ Plus 2 percent Na₂CO₃.2/ Fluorspar.

TABLE 5. - Mineral wool from Florida slag, clay, and staurolite

Test No.	Weight, percent			App. pour temp., °F.	Steam pressure, p.s.i.	Pour time, seconds	Shot, percent		Fiber diam., microns	Remarks
	Slag	Clay	Staurolite				Free	Attached		
3	90	-	10	2,755	42	23	5	44	5.7	
6	80	-	20	2,595	49	25	13	43	7.1	
10	70	-	30	2,610	48	29	14	43	7.6	
17	90	10	-	2,550	42	23	18	50	9.2	
21	80	20	-	2,600	40	20	16	46	10.7	
30	100	-	-	2,800	44	25	-	-	-	Charge too hot.
31	100	-	-	2,750	45	29	36	58	3.5	Excessive shot.
32	100	-	-	2,550	-	-	-	-	-	Fine fibered.
18	90	10	-	2,575	35	21	32	44	9.1	About half molten.
19	90	10	-	2,530	40	15	36	34	6.7	Excessive shot.
20	80	20	-	2,550	42	18	22	44	9.3	Do.
22	80	20	-	2,760	32	18	22	36	13.0	Fair results.
23	70	30	-	2,750	32	21	18	44	15.0	Coarse wool.
24	70	30	-	2,700	35	27	17	40	11.5	Do.
25	70	30	-	2,800	35	27	11	48	11.2	Fair results.
26	60	40	-	2,750	38	30	13	-	23.9	Do.
27	60	40	-	2,800	38	28	12	-	20.2	Coarse and shotty.
28	60	40	-	2,850	46	42	10	-	-	Do.
29	50	50	-	-	-	-	-	-	-	Very coarse.
4	90	-	10	2,725	43	25	24	45	7.2	Poor melt.
5	90	-	10	2,610	50	24	25	51	11.3	High free shot.
7	80	-	20	2,500	49	27	16	40	10.4	Coarse and shotty.
8	80	-	20	2,450	42	22	22	52	10.8	Fair results.
9	70	-	30	2,550	48	35	13	41	10.2	Excessive shot.
11	70	-	30	2,670	40	31	15	-	-	Slow pour.
12	60	-	40	2,660	42	31	12	-	-	Incomplete melt.
13	60	-	40	2,710	42	25	12	-	-	Do.
14	60	-	40	2,790	44	23	16	-	12.1	Do.
15	50	-	50	2,750	42	23	15	-	26.1	Wool full of flakes.
16	50	-	50	2,790	44	27	13	-	-	Coarse and flaky.
34	60	20	20	2,700	49	23	13	38	13.9	Do.
33	60	20	20	2,750	48	29	15	37	12.6	Coarse.
35	60	20	20	2,800	47	25	11	41	14.0	Rather coarse fibers.
2	34	33	33	2,750	48	29	15	37	12.6	Coarse.
										Rather coarse, poor pour.

TABLE 6. - Analyses of Florida samples, percent - Bailey

	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO
Limestone	34.7	33.4	-	13.9	1.0
River sand	98.6	-	1.56	-	-

A study of these analytical results indicates it is improbable that satisfactory mineral wool can be produced from any possible blends of these raw materials. There is an imbalance of basic and acidic constituents consisting of a shortage of dolomite or the basic phase and an excess of alumina.

Preliminary experiments, in which the total charge consisted of impure limestone, produced very coarse wool. Although amphoteric in nature, the refractoriness of the alumina content caused the viscosity of the molten material to be such that the blown fibers were very coarse and glassy. The high alumina content of this material made it necessary to add both silica and lime to make a blend of oxides that would lie within the mineral-wool producing area of the Al_2O_3 -CaO-SiO₂ ternary diagram. Results from blowing blended charges of limestone, river sand, and burned lime are shown in table 7.

The first part of the table presents the more satisfactory experiments, whereas the latter portion shows the marginal and unsatisfactory tests.

TABLE 7. - Mineral wool from Florida limestone and silica sand

Test No.	Weight, percent			App. temp., °F.	Steam pressure, p.s.i.	Pour time, seconds	Shot, percent		Fiber diam., microns	Remarks
	Lime-stone	Sand	CaO				Free	Attached		
2	65	15	20	2,910	48	13	32	46	6.9	High free shot.
4	50	26	24	2,642	48	17	23	36	7.3	Good wool.
5	45	40	15	3,047	50	19	23	41	6.9	High temperature.
1	100	-	-	3,090	50	16	-	-	-	Too viscous to blow.
3	58	20	22	2,760	48	18	13	49	11.6	Slightly coarse.
6	39	45	16	3,050	50	22	15	32	11.0	Fair wool.
7	51	46	3	3,450	50	19	-	-	-	Too viscous to blow.

Missouri

Two rock samples were received with a request to determine if either were a natural wool rock.

Petrographic examination proved both rocks to be dense, argillaceous dolomite containing a few calcite stringers and finely divided siliceous material, probably from the disintegration of chert. A composite of the rocks was analyzed chemically, with the results shown in table 8.

TABLE 8. - Analysis of Missouri rock composite, percent

SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Ign. loss	TiO ₂
11.1	1.6	0.9	26.8	18.5	40.1	0.14

The analytical determinations, verified by the petrographic studies, indicated that these were not naturally occurring wool rocks. The ratio of acidic to basic constituents is not suitable for making mineral wool. By adding silica to these rocks a blend could undoubtedly be obtained that would form wool upon blowing.

No blowing experiments were conducted on this material, since analytical examinations showed it was not a natural wool rock, and the submittor did not

request determination of additive constituents necessary to produce a blowable mineral-wool mixture.

Tennessee

Four rock samples were submitted from commercial quarries. The samples were designated as gray, pink, cedar marble, and "shale."

Petrographic examination of the materials indicated all were mineralogically similar, consisting chiefly of calcium carbonate with some clayey material and a small amount of iron oxide.

To show more precise compositions, a portion of the cedar marble and a portion of the shaly textured rock were analyzed chemically with the results given in table 9.

TABLE 9. - Analyses of Tennessee samples, percent

	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Ign. loss
Cedar marble	5.86	2.02	1.42	47.9	0.01	39.7
"Shale"	5.82	1.98	1.42	47.8	.01	39.4

The analyses show that it would be impossible to produce mineral wool with these materials alone, since the "shale" is not a shale and siliceous or acidic constituents are very deficient.

Blowing experiments were not made on these materials.

Texas

During experimental extraction of residual tin, tungsten, and iron from a tin slag it was noted that the extracted residues or slags had desirable characteristics for wool blowing.

Petrographic examination of the slag residues showed them to consist of glassy solid solutions with no distinguishable crystalline compounds.

Analysis of a typical slag after treatment in the molten state with chlorine and sulfur to volatilize tin and iron, but before blowing to mineral wool, showed the following percentages.

TABLE 10. - Analysis of Texas tin slag, percent

Sn	WO ₃	SiO ₂	Al ₂ O ₃	FeO	CaO	MgO	TiO ₂
0.04	0.17	50.6	11.8	2.0	27.2	0.7	2.4

Blowing tests were made on 100 percent slag charges and slag with the addition of 10 percent lime. All charges made a rather fine, soft wool. Increasing the fiber diameter by lowering pour temperature or by increasing the rate of pour tended to produce more shot. The slag-lime blend permitted a faster pour rate and a lower pour temperature with only a slight increase in shot content.

Results of typical tests are shown in table 11.

TABLE 11. - Mineral wool from Texas tin slag

Test No.	Weight, percent		App. pour temp., °F.	Steam pressure, p.s.i.	Pour time, seconds	Shot, percent		Fiber diam., microns	Remarks
	Slag	CaO				Free	Attached		
1	100	-	2,870	42	43	16	47	4.4	Rather fine fiber.
2	100	-	3,035	40	37	18	57	5.7	High shot.
3	100	-	3,080	44	42	17	45	4.7	Good wool.
4	90	10	2,870	42	26	21	53	5.0	Do.

Virginia

Three samples of quarrying waste slates were received for experimental testing. They were marked No. 1 Green, No. 2 Purple, and No. 3 Hard.

Mineralogically, the samples consisted essentially of chlorites and contained small amounts of iron oxides and quartz. Chemical analyses of the materials are shown in table 12.

TABLE 12. - Analyses of Virginia samples, percent

	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Ign. loss
No. 1 Green ...	51.7	28.7	9.9	0.27	1.83	4.8
No. 2 Purple...	54.5	29.1	9.6	.40	1.82	4.4
No. 3 Hard	55.2	25.1	9.7	.43	1.82	3.9

These analyses indicate that it would be necessary to blend basic materials with the slates to produce a mixture that could be blown into mineral wool.

The addition of proper quantities of lime to any of the three slates or mixtures of slates produced blends that were readily blowable to acceptable mineral wool as evaluated in the laboratory. Results of representative tests on each slate are presented in table 13.

TABLE 13. - Mineral wool from Virginia slates

Test No.	Type slate	Weight, percent			App. temp., °F.	Steam pressure, p.s.i.	Pour time, seconds	Shot, percent		Ave. fiber diameter, microns	Remarks
		Slate	CaO	SiO ₂				Free	Attached		
26	Green	61	39	-	2,820	49	35	10	34	8.0	
25	Purple	58	42	-	2,810	49	35	16	48	10.5	
22	Hard	61	39	-	2,865	50	30	17	33	9.7	
18	Green	32	-	-	-	-	-	-	-	-	
	Purple	32	36	-	2,780	49	27	17	48	10.0	
1	Hard	78	22	-	2,800	42	25	61	-	-	Very poor pour.
2	Do.	74	26	-	2,800	43	37	23	-	-	Very coarse fiber.
3	Do.	67	33	-	2,810	49	33	25	-	-	Lumpy pour.
4	Do.	87	-	13	2,800	-	-	-	-	-	Would not pour.
5	Do.	61	26	13	2,800	-	-	-	-	-	Incomplete melt.
6	Purple	78	22	-	2,765	42	30	12	-	-	Coarse, brittle fiber.
7	Do.	74	26	-	2,790	49	32	13	-	-	Do.

TABLE 13. - Mineral wool from Virginia slates (Con.)

Test No.	Type slate	Weight, percent			App. temp., °F.	Steam pressure, p.s.i.	Pour time, seconds	Shot, percent		Ave. fiber diameter, microns	Remarks
		Slate	CaO	SiO ₂				Free	At-tached		
8	Purple	70	30	-	2,860	49	35	8	21	21.7	Low shot, coarse.
9	Do.	67	33	-	2,840	50	33	15	33	16.5	Coarse fiber.
10	Do.	67	19	14	2,840	-	-	-	-	-	Would not pour.
11	Green	78	22	-	2,835	-	-	-	-	-	Too viscous to pour.
12	Do.	74	26	-	2,900	50	19	19	25	14.5	Fiber too coarse.
13	Do.	70	30	-	2,860	41	27	18	38	11.3	Fair wool.
14	Do.	67	33	-	2,830	42	32	16	40	11.6	Do.
15	Do.	64	36	-	2,840	50	32	14	38	10.7	Medium wool, low shot.
16	Purple	32	-	-	-	-	-	-	-	-	-
	Green	32	36	-	2,900	40	32	20	34	11.5	Fair wool.
17	Purple	43	-	-	-	-	-	-	-	-	-
	Green	24	33	-	2,800	41	22	22	38	10.7	Good wool.
19	Do.	64	36	-	2,785	50	20	22	38	10.2	Do.
20	Do.	48	-	-	-	-	-	-	-	-	-
	Purple	19	33	-	2,860	42	40	17	34	12.5	Slightly coarse.
21	Hard	38	-	-	-	-	-	-	-	-	-
	Green	29	33	-	2,850	49	23	18	34	10.0	Good wool.
23	Hard	58	42	-	2,815	50	36	18	49	10.8	Fair wool.
24	Purple	61	39	-	2,820	50	35	17	46	11.7	-
27	Green	58	42	-	2,810	49	36	19	53	8.5	-

Discussion

A common method of forming mineral-wool fibers consists of blowing molten streams of fluxed material with steam or air. During the conversion of the liquid charge to wool fibers many misshapen solids are formed that are termed "shot." As shown by the tables of laboratory tests and tests of commercial wools, shot often amounts to over 50 percent of the weight. Development of a method of fiber formation that would convert this shot to useful wool during the blowing or other fiber formation operation, at little or no additional cost, would contribute markedly to the industry through the more efficient utilization of raw materials and the conservation of fuels, manpower, and shipping expense.

APPENDIX

Information concerning the source of the samples herein reported follows in table 14.

TABLE 14. - Source data, raw samples

State	Locale	Submittor	City	Samples
Alaska	-	Federal Bureau of Mines	Juneau	Limestone. Shale.
Florida	Nichols Starke Edgar	Lenox H. Rand	Orlando	Slag. Staurolite. Clay.
Florida	Chattahoochee	Frank D. Bailey	Chattahoochee	Limestone. Sand.
Missouri	Jefferson City	L. D. Thompson	Jefferson City	Dolomites.
Tennessee	Knoxville	Carl V. Stafford	Knoxville	Marbles. Shale.
Texas	Texas City	Federal Bureau of Mines	Rolla	Slag.
Virginia	Arvonnia	Owen R. Jeffrey	Arvonnia	Slates.