Jan. 6, 1953

L. PARKER ET AL

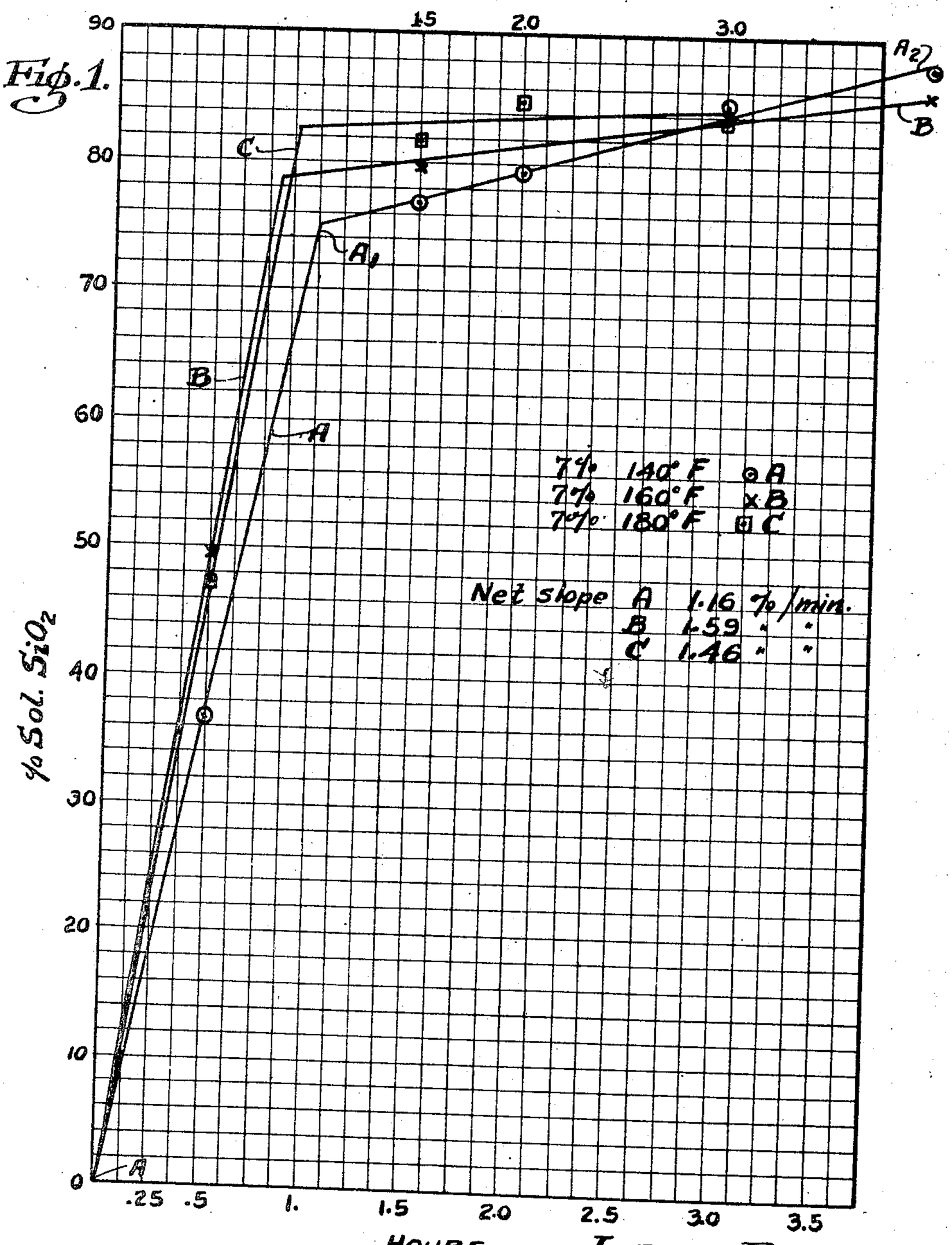
2,624,658

METHOD FOR FORMING SILICA FIBERS

Filed Aug. 8, 1949

8 Sheets-Sheet 1

Fig. 1.



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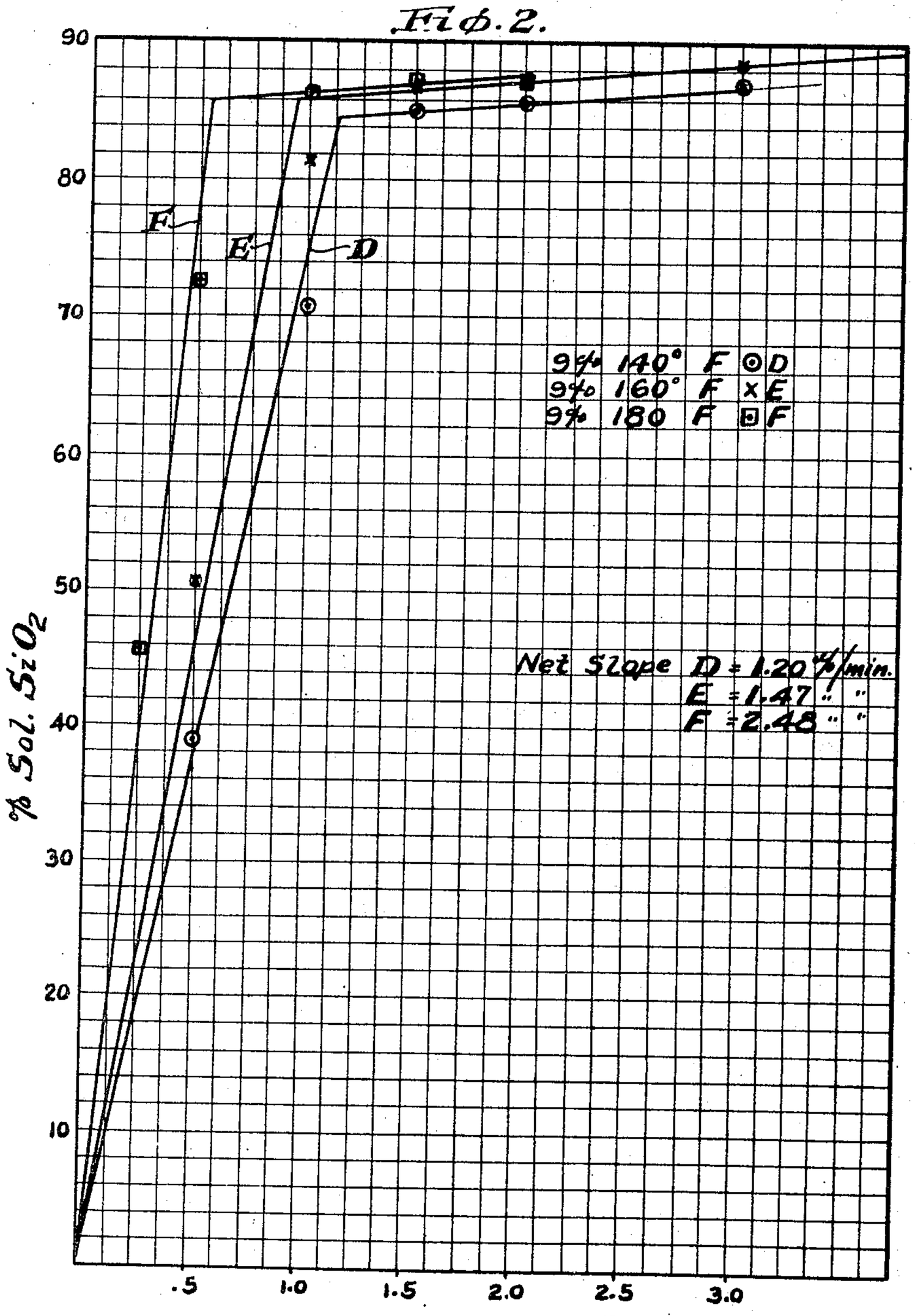
2,624,658

METHOD FOR FORMING SILICA FIBERS

Filed Aug. 8, 1949

8 Sheets-Sheet 2

Fig. 2.



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METHOD FOR FORMING SILICA FIBERS

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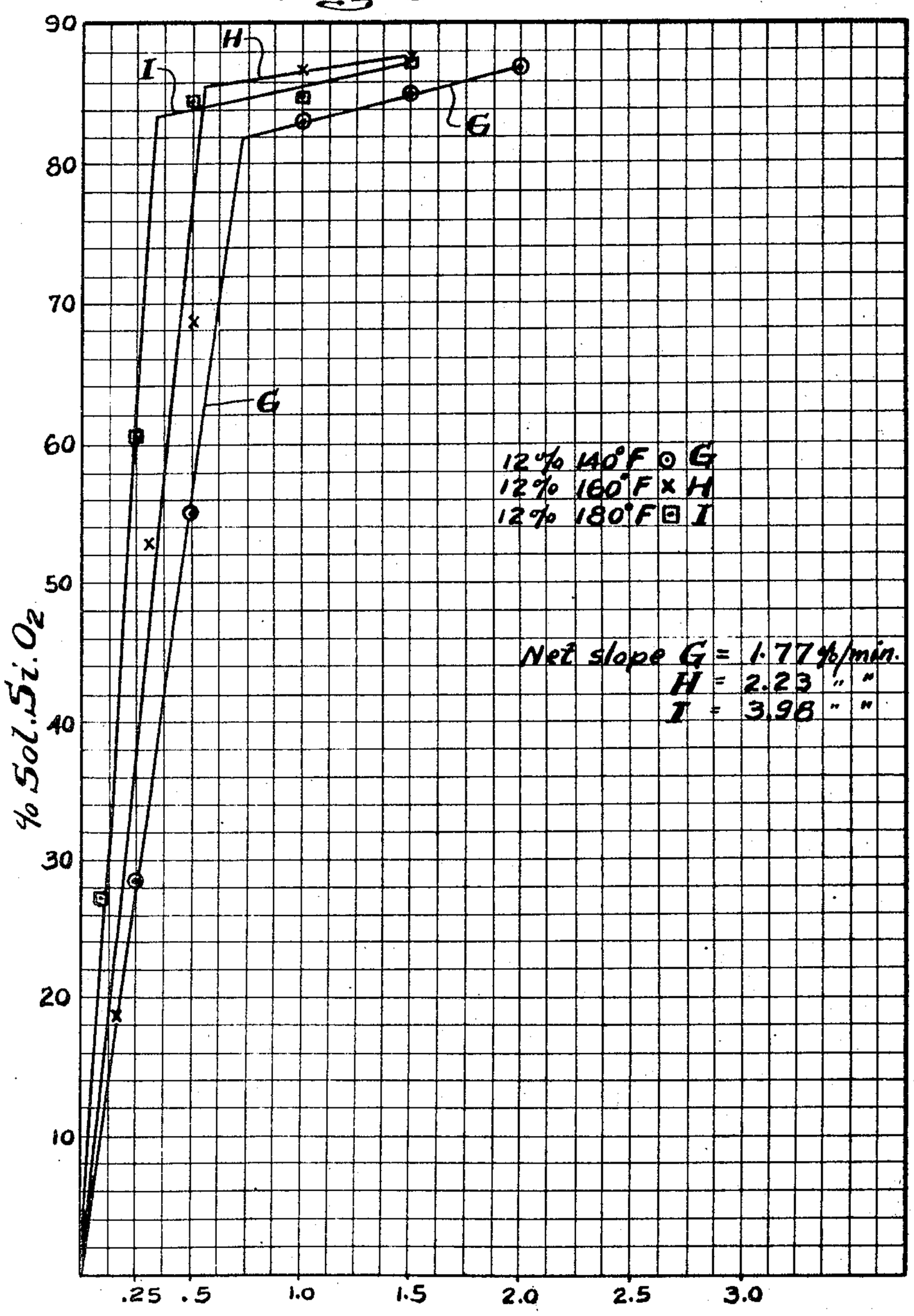
Fig. 3.



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METHOD FOR FORMING SILICA FIBERS

Filed Aug. 8, 1949

8 Sheets-Sheet 4

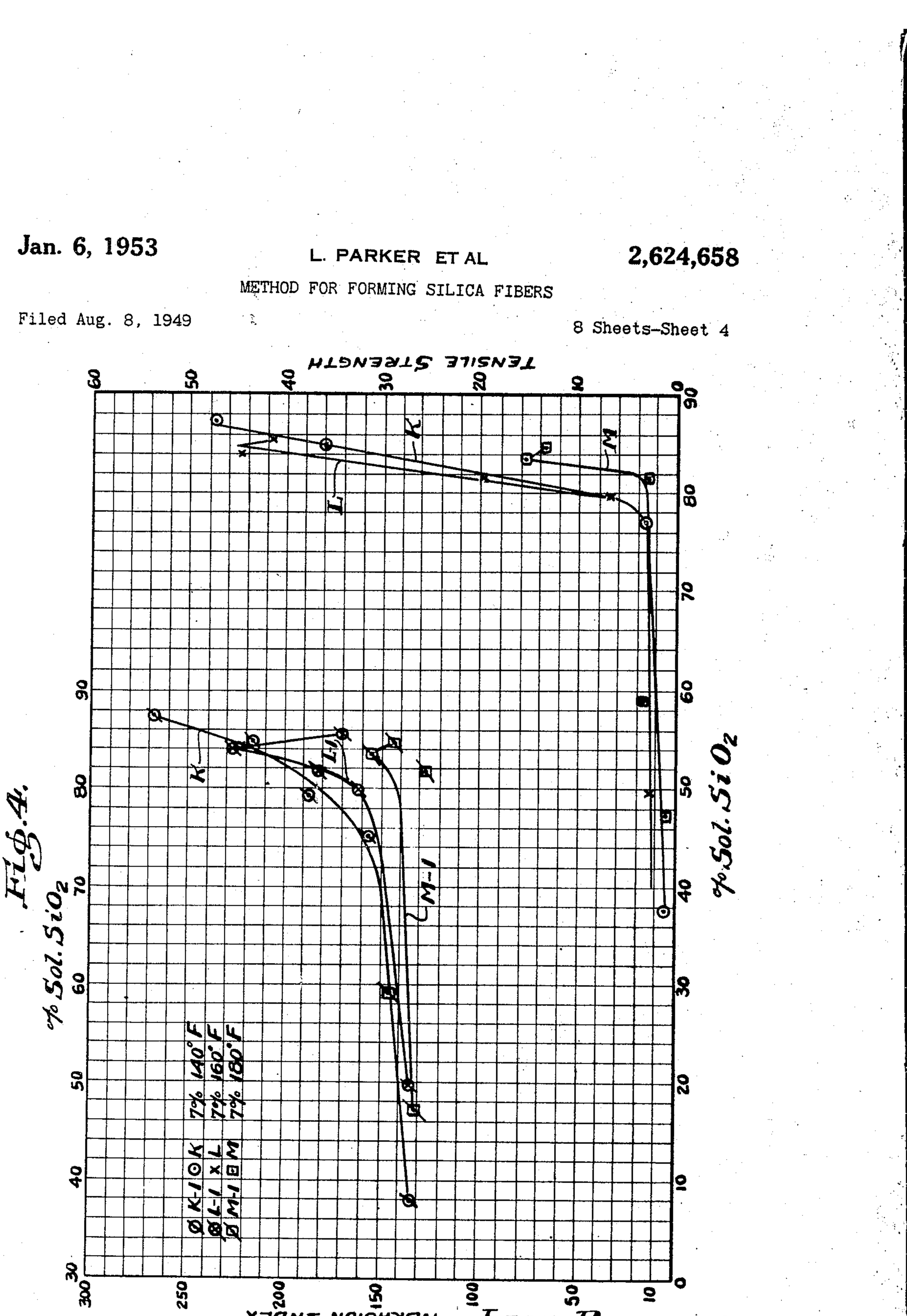

Fig. 4.

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METHOD FOR FORMING SILICA FIBERS

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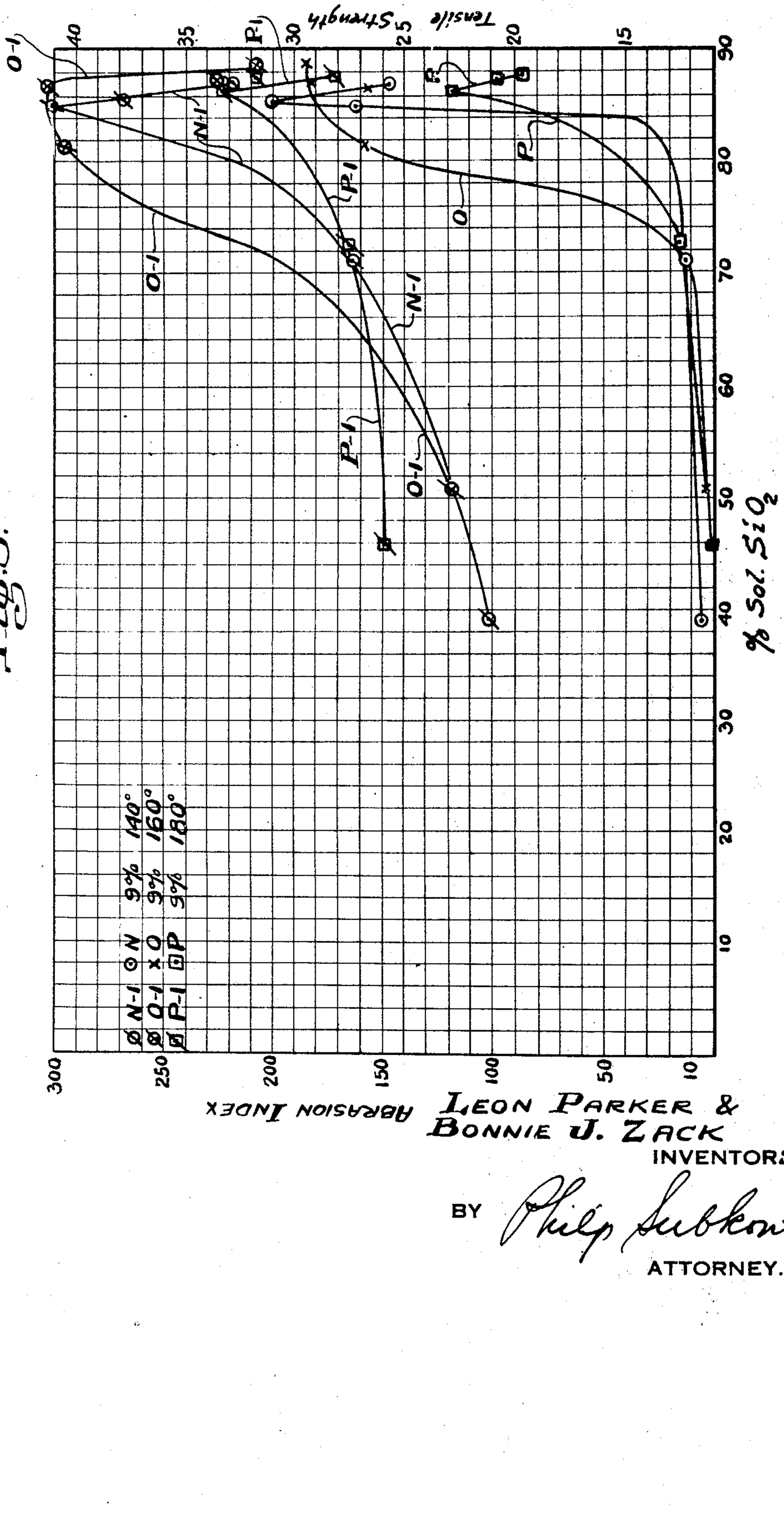

Fig. 5.

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METHOD FOR FORMING SILICA FIBERS

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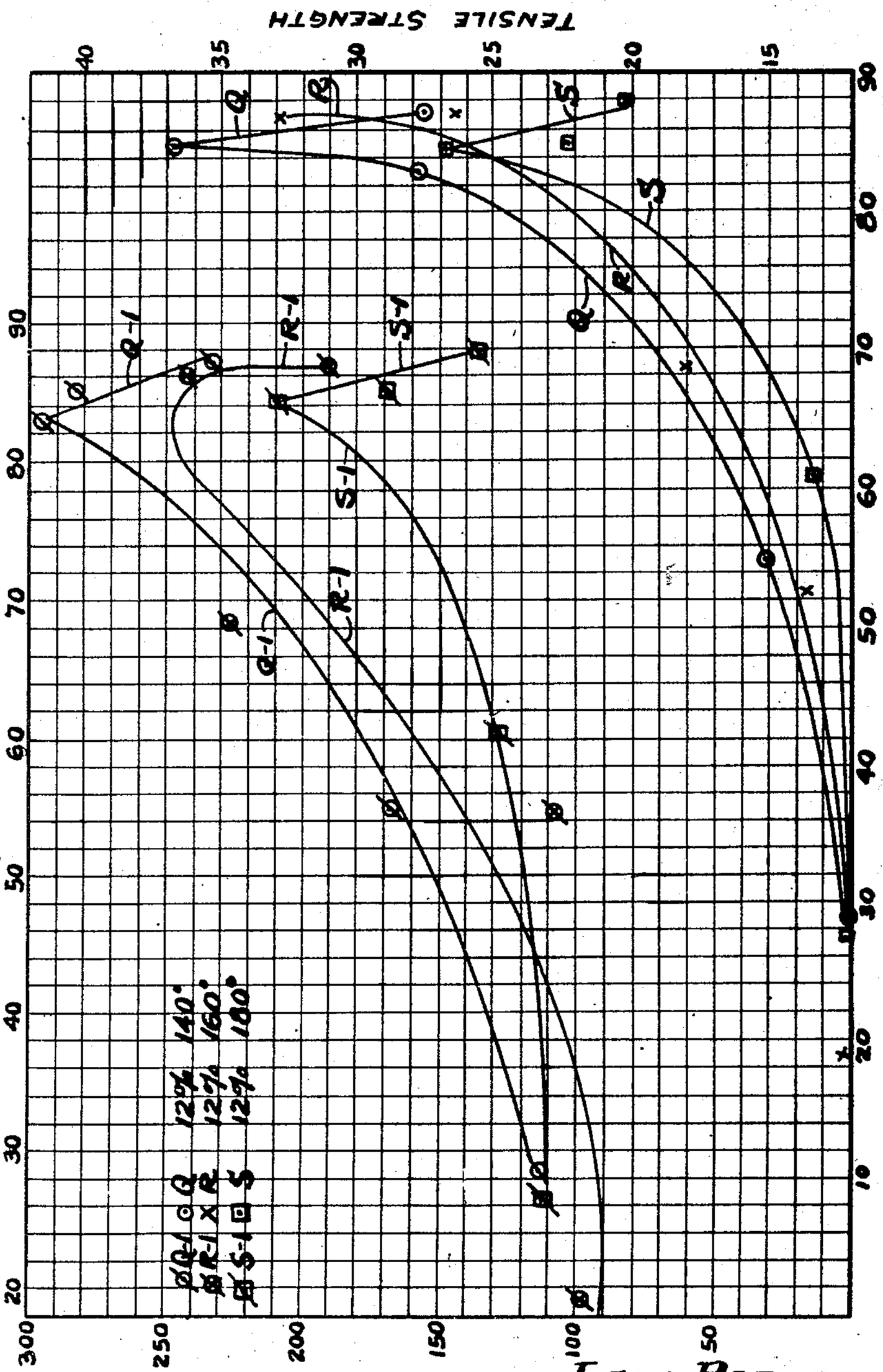

FIG. 6.

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METHOD FOR FORMING SILICA FIBERS

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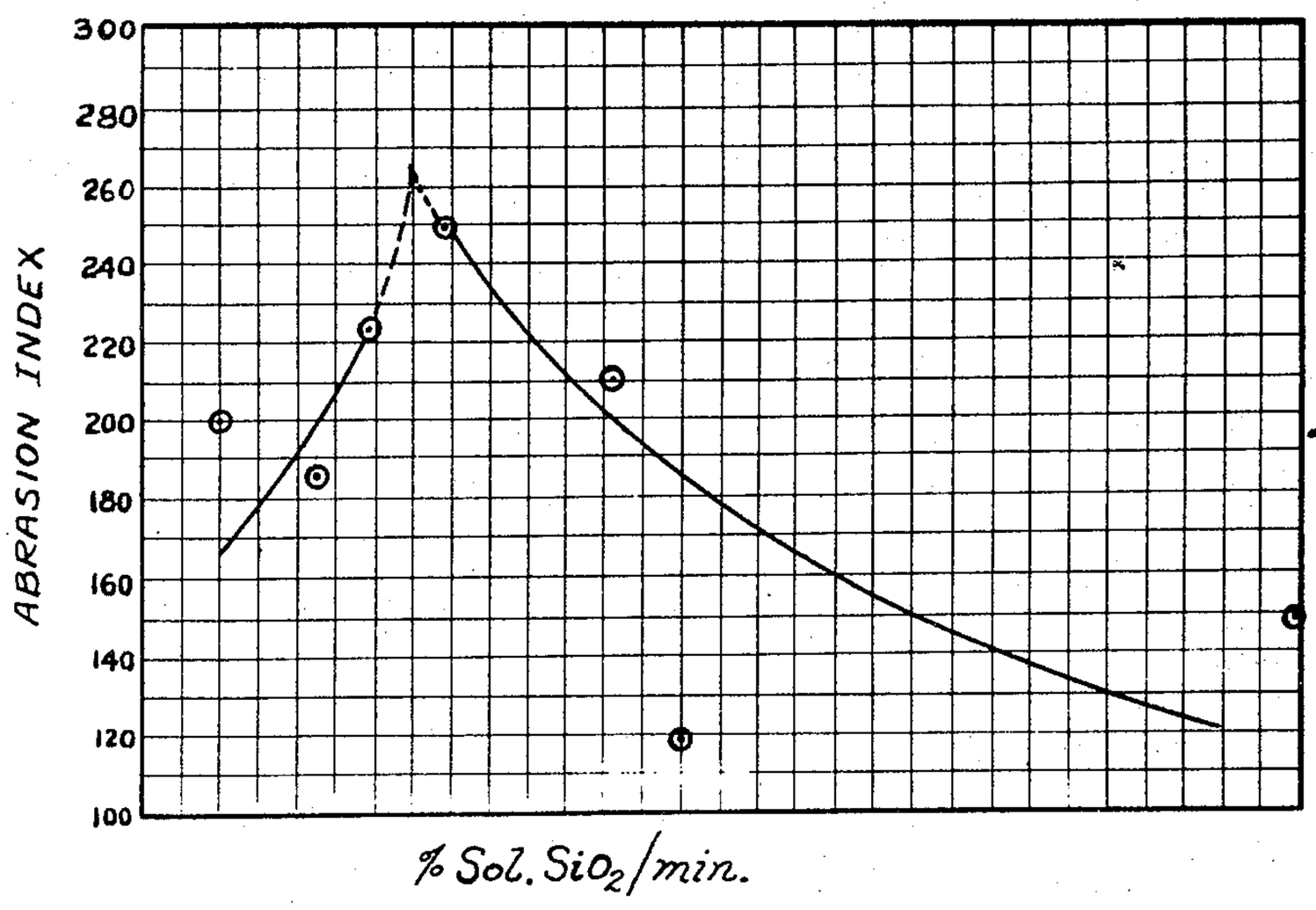

Fig. 8.

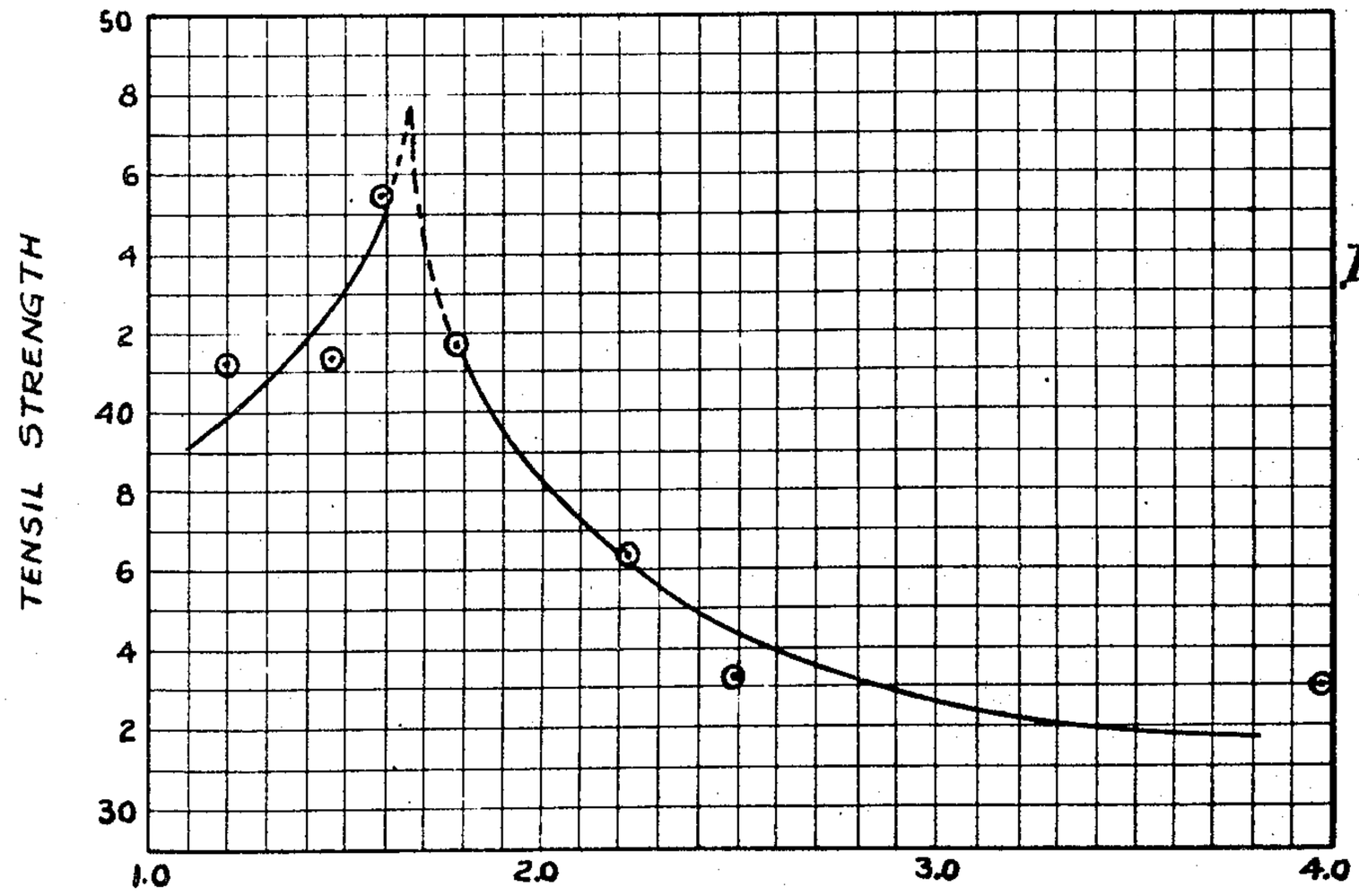

Fig. 7.

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METHOD FOR FORMING SILICA FIBERS

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Fig. 9.

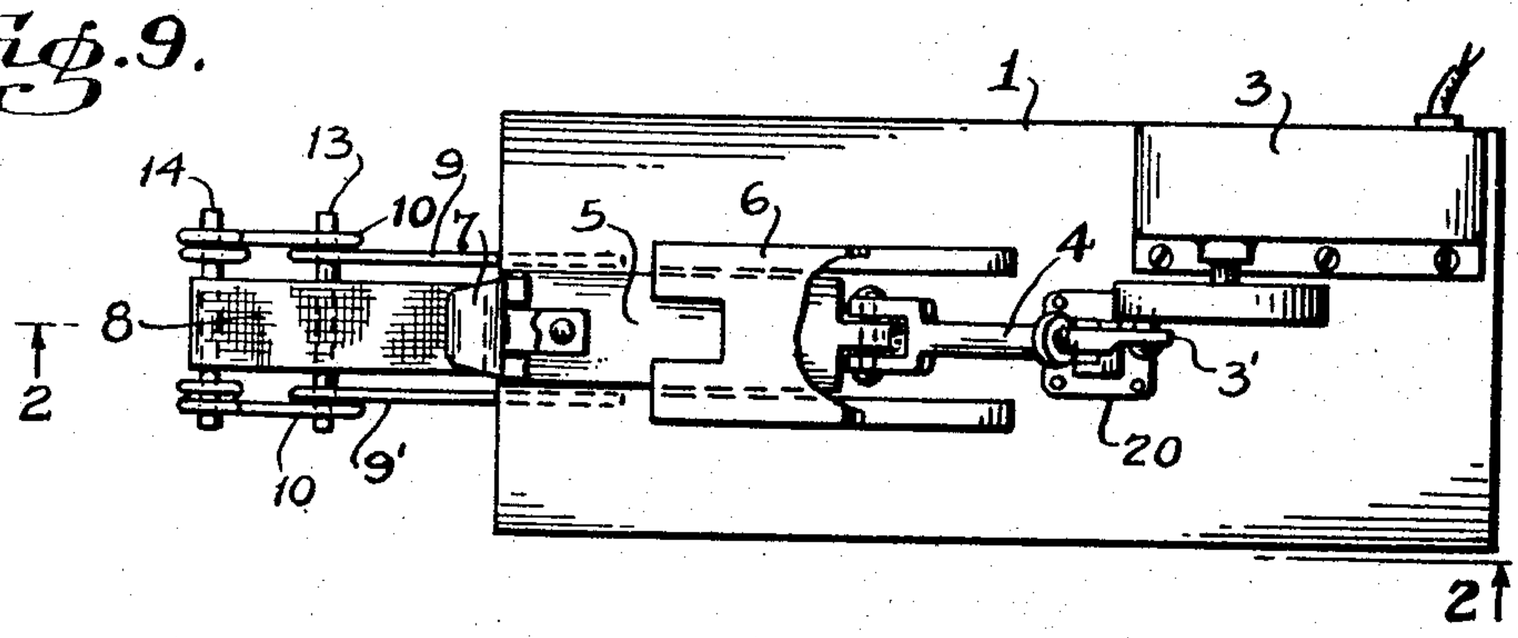


Fig. 10.

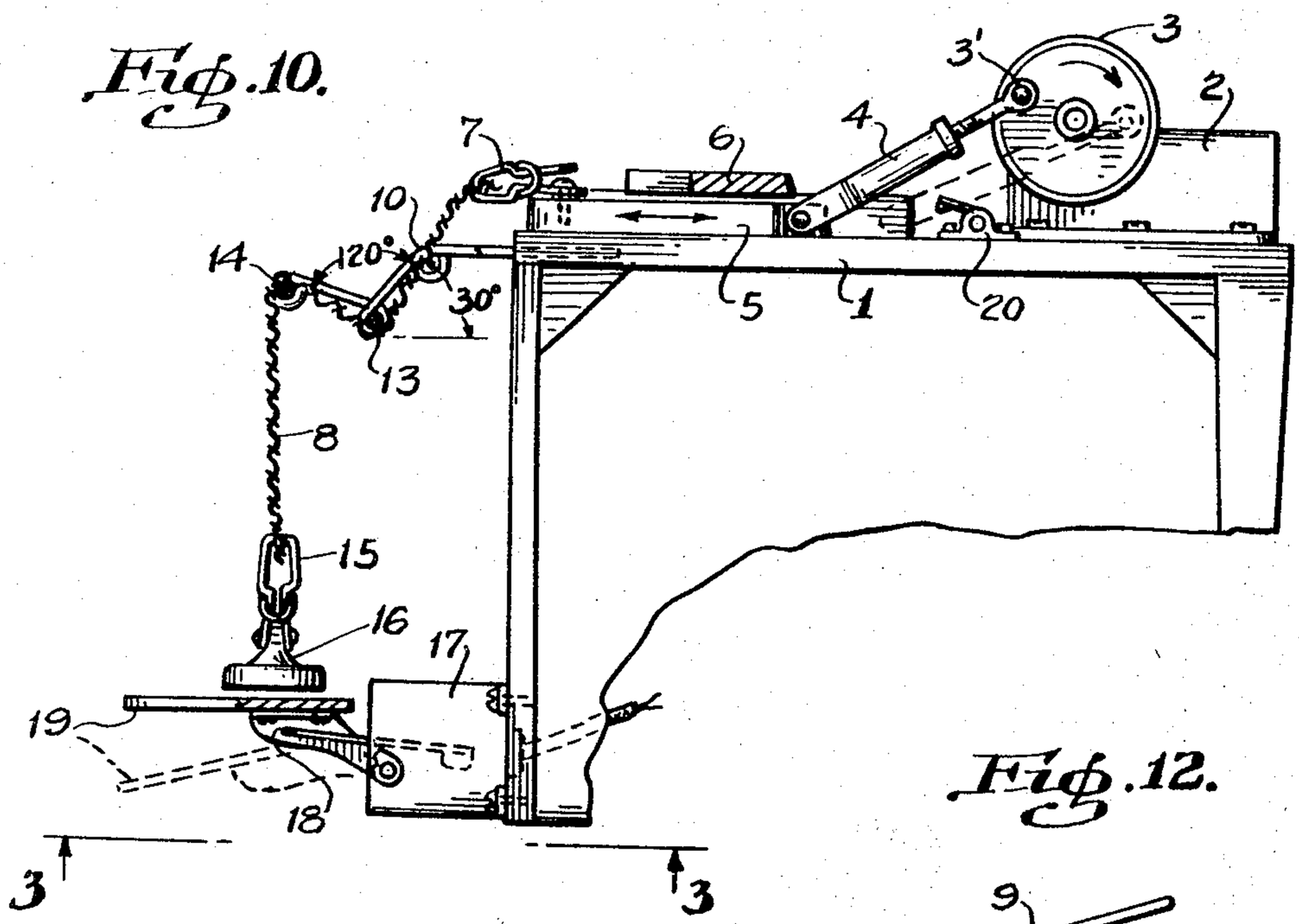


Fig. 12.

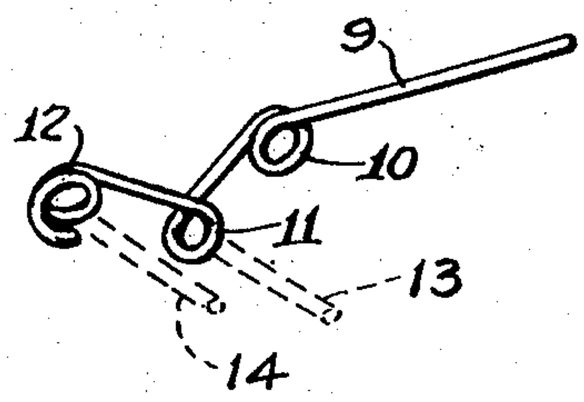
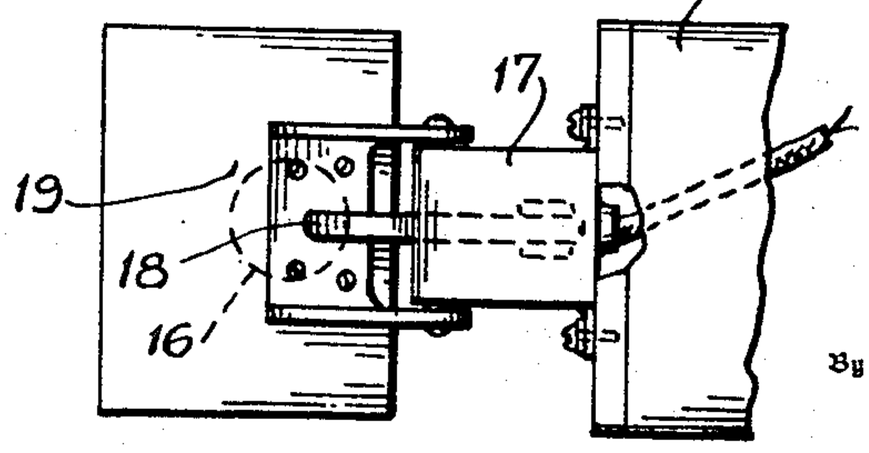


Fig. 11.



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Patented Jan. 6, 1953

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UNITED STATES PATENT OFFICE

2,624,658

METHOD FOR FORMING SILICA FIBERS

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Application August 8, 1949, Serial No. 109,206

15 Claims. (Cl. 41—42)

1

This application is a continuation-in-part of application Serial No. 74,935, filed February 7, 1949, now abandoned.

It has been shown that glass fibers may be leached to remove the non-siliceous oxides and thus produce fibers of high silica content, the silica content depending upon the degree of extraction of the fibers. Depending upon the composition of the fibers, these non-siliceous oxides may be extracted either by plain water or with acids. While glass fibers of various compositions may be so extracted by both neutral water and acid waters, the boro-silicate glasses which are usually employed in forming glass filaments for weaving into textile materials which have less than 70% silica content may be extracted without previous preheating of the glass structure. Such filaments may be leached without such heat treatment and since they are usually of less than .001'' in diameter, they may be leached to remove the acid soluble oxides other than silica without destruction of the fiber. The glasses which lend themselves best to drawing into fiber contain about 56% or less of silica, about 22% or less of alumina, about 5% or more of boron oxide and about 22% or less of second group metal oxides, especially lime and magnesia. Such fibers, without preheating, may be readily leached with acid to remove the metal oxides other than silica.

The resultant product contains some water of hydration in the neighborhood of about 8% to 11% and may be dehydrated by heating to temperatures of about 1000° F., preferably in the region of about 1400° to 1600° F. By proper control of the acid extraction process the resultant fibers after firing contain high silica content and may be as high as 90% and even substantially 99.9% of silica as determined by the hydrofluoric acid method.

The above process and the effect of the glass composition and degrees of treatment in producing such extracted glass fibers are described in the Parker and Cole application, Serial No. 669,098, filed May 11, 1946, now Patent No. 2,491,761, and United States Patent No. 2,461,841 issued to Nordberg, to which reference may be had for a more full discussion of this process.

2

In our previous application Serial No. 74,935, filed February 7, 1949, we have shown that the process of extraction of the boro silicate glasses containing alumina and second group metal oxides, as previously described, results in a transformation of the silica of the glass from a form which is substantially insoluble in sodium carbonate solution into a form which is soluble in sodium carbonate solution. We have also shown that the percentage of the silica which is present in such soluble form depends upon the degree of extraction to which the glass has been subjected. The glass structure, that is the fiber, maintains its form during the extraction and during the firing, but there is a large impairment in the cohesive force of the fiber structure. Thus, the tensile strength and the resistance to abrasion of the extracted glass fibers of yarn, rope or cloth is materially reduced, in fact, to but a small fraction of the original tensile strength or abrasive resistance.

The degree of extraction of the non-siliceous oxides of the glass depends upon the time, temperature and concentrations employed, and by controlling these factors the degree of extraction may be controlled.

In said application Serial No. 74,935, it was disclosed that there is an unexpected reversal in the depreciation of tensile strength as the leaching progresses and as more non-siliceous oxides are extracted; that is, as the concentration of the sodium carbonate soluble silica in the glass increases. Thus, as the leaching progresses and there is a conversion of a portion of the vitreous silica (which is insoluble in sodium carbonate solution) into the sodium carbonate soluble form, the tensile strength of the fiber structure is rapidly decreased, but as the leaching continues and the concentration of carbonate soluble silica in the leached glass increases there is a reversal in the trend and the tensile strength appreciates as the content of sodium carbonate soluble silica increases, but if this leaching continues with a further increase in the content of the sodium carbonate soluble silica there is a further reversal in the trend and the tensile strength again depreciates.

It was found as the result of an extensive experimentation in leaching of such boro silicate alumina glasses containing second group oxides that while the tensile strength of the leached fabric is less than that of the original glass fabric as the concentration of the sodium carbonate soluble silica in the leached glass increases beyond about 30% (on a volatile free basis), the tensile strength increases, reaching a maximum in the region of about 70% to 86% (on a volatile free basis) sodium carbonate soluble silica in the leached glass at which a maximum tensile strength is obtained, and that as the concentration of the sodium carbonate soluble silica increases beyond about 86% the tensile strength diminishes rapidly, falling to about as much as 50% of the maximum tensile strength in going from 86% to 89% (on a volatile free basis) soluble silica in the leached glass.

Summarizing the discovery of our present application we have found by further study of this phenomenon that the value of the maximum of tensile strength obtained by carrying the extraction of the glass to develop a soluble silica content within the range of 70% to 87%, depends upon the rate of attack of the glass structure by the acid. We have found that the maximum tensile strength is obtained if the development of sodium carbonate soluble silica in the leached glass is at a rate within the range of about 1 to 2.5% (on a volatile free basis) per minute. If the rate is controlled within these limits, then we obtain the maximum tensile strength. We have thus found that at any given rate of attack a maximum tensile strength is developed when the leaching is controlled to give a leached glass containing soluble silica in the range of 70% to 86% (on a volatile free basis), but the magnitude of the maximum is greatest if the rate of attack is within the limits given. We have also found that the development of abrasion resistance parallels that of tensile strength. Thus by controlling the rate of attack to within the said limits of 1% to 2.5% of sodium carbonate soluble silica per minute, we obtain the maximum abrasion resistance if we control the leaching to produce a leached glass having soluble silica in the range of 70% to 87% (on a volatile free basis).

It was thus found that in order to develop a leached glass structure of maximum tensile strength and maximum abrasion resistance, it is desirable to control the conditions of treatment to obtain a rate of attack to develop sodium carbonate soluble silica in the leached glass structure at the rate of about 1% to 2.5% per minute and to continue the attack to develop a content of sodium carbonate soluble silica in the glass structure in the range of about 70% to 87% of the leached glass.

Why these phenomena should occur we cannot explain. We merely record the discovery of their presence and because of these unexpected discoveries we were able to control the leaching of ahe glass fibers to obtain the optimum strength and optimum abrasion resistance.

The control of the rate of attack may be readily obtained by controlling either acid concentration, where acid is used, or temperature or both, as will be further described below. These discoveries will be further elucidated with the following description of our experiments when taken together with the charts, in which

Fig. 1 is a plot of the data of our experiments showing the below defined development and the rate of development of the soluble SiO_2, as hereinafter defined, with time when the fabric is treated with 7% acid at temperatures of 140°, 160°, and 180° F., respectively;

Fig. 2 is a chart similar to Fig. 1 for the treatment with 9% acid at tempreatures of 140°, 160°, and 180° F.;

Fig. 3 is a chart similar to Figs. 1 and 2 for the treatment with 12% acid at temperatures of 140°, 160°, and 180° F., respectively;

Fig. 4 is a plot of the effect of the conrentration of soluble silica upon the tensile strength and the abrasion index obtained in the treatment of fabric with 7% acid at 140°, 160°, and 180° F.;

Fig. 5 is a plot similar to Fig. 4 for the experiment with 9% acid at 140°, 160°, and 180° F.;

Fig. 6 is a plot similar to Figs. 4 and 5 for the effect of the treatment with 12% at 140°, 160°, and 180° F.;

Fig. 7 is a plot of the rate of development of soluble silica against the maximum tensile strength obtainable at such reaction rates;

Fig. 8 is a similar plot showing the effect of the rate of development of soluble silica with the maximum abrasion index obtainable at such various reaction rates;

Fig. 9 is a plan view of the machine for determining the abrasion index;

Fig. 10 is a section, partly in elevation, on line 2—2 of Fig. 1;

Fig. 11 is a detail of a bottom elevation of line 3—3 of Fig. 2; and

Fig. 12 is a detail perspective view.

In order to illustrate our discovery the following experimentations which underly our discovery may be taken as illustrative of our invention and not as a limitation thereof.

Samples of glass fiber cloth showed the following characteristics: The thread had an average diameter ranging from 0.038 to 0.040, and the number of threads per inch warp 20, fill 18. The fibers were made up of a multiple of fine filaments, each of the filaments having a dimension ranging from 195×10^{-6} inches to 260×10^{-6} inches.

A number of samples of the cloth, cut contiguously from one piece of cloth, was subjected to a series of treatments. A group of such samples was exposed to treatment with 7% acid at 140° F.; another group was subjected to treatment with 7% acid at 160° F.; and a third group was subjected to treatment with 7% acid at 180° F. This was also repeated with different groups of samples of the same cloth, cut from the same contiguous portions of a bolt, with concentrations of 9% acid at 140° F., at 160° F. and at 180° F. Another group was treated with 12% acid at 140° F., another group with 12% acid at 160° F., and another group with 12% acid at 180° F. The temperature was controlled to be constant during the treatment and such a large excess of acid was employed that the change in concentration of the acid during treatment was insignificant.

Similar conditions of treatment were used for each group, except for the change in temperature and concentration. The batches were immersed in a large container containing the acid and allowed to remain quiescent during treatment. Samples were removed at different times from the treating solution for analysis in each experiment. Each sample when removed was washed with distilled water until it was chloride free, then dried at 100° F. One-half of each of the samples were retained for analysis and test and the other portion was fired at 1600° F. for one-half hour and subjected to test and analysis.

The fired sample was subjected to analysis and test as indicated below.

The original glass cloth had the following composition:

The glass fiber had 2.09% organic matter, being the lubricant employed in the weaving. The material was burned off and the loss in weight (2.09%) is here reported as lubricant. The residual 97.91% had the following composition on a lubricant free basis:

	Percent
SiO_2	53.95
Al_2O_3	16.14
Fe_2O_3	.56
CaO	15.96
MgO	4.10
B_2O_3	8.20
Na_2O	.88
	99.79

The samples were leached as above for various times, temperature and acid concentrations, using a bath volume sufficiently large that no appreciable change in acid concentration occurred during leaching. The samples were all washed free of chlorides with distilled water.

The unfired samples were analyzed as follows:

The samples were air dried and then dried for one hour at 95° C., weighed in a closed dish, and subjected to the following analysis:

(1) *Per cent loss on ignition.*—A sample of leached cloth was accurately weighed in a platinum crucible and placed in a muffle at 1600° F. for one-half hour. Loss in weight was due to water of hydration plus lubricant on the fibers.

(2) *Per cent lubricant.*—An accurately weighed sample of glass cloth was put in the muffle at 900° F. until all of the lubricant was burned off. Loss in weight was lubricant. The per cent lubricant was found for one sample and assumed to be the same for all other samples. Subtracting this per cent from the per cent loss on ignition gives the per cent water of hydration.

(3) *Per cent soluble material (hydrated silica plus lubricant).*—A sample of leached cloth accurately weighed was dissolved in 100 ml. of hot 4 N Na_2CO_3. After solution was complete the residue was filtered off, washed to neutrality with distilled water, ignited, and weighed. The weight of residue subtracted from the weight of the initial sample gave the weight of soluble material, and the per cent was calculated on the basis of the weight of initial sample.

(4) A portion of the sample, fired for determination of loss on ignition, was weighed, and total silica in this weighed sample as determined as follows:

To the weighed fired sample approximately 2 to 3 mls. of dilute H_2SO_4 plus 20 mls. of HF were added and evaporated to dryness. This evaporation was repeated until the residue reached constant weight. The loss divided by the weight of the fired sample multiplied by 100 is reported as per cent silica.

(5) The tensile strength was determined as follows:

A Scott Tester, Model DH (motor driven, pulling rate 10''/min.), having a capacity of 0–400 pounds and using a 1'' front jaw and 3'' back jaw was employed. Ten separate strips of the cloth sample, each 3'' x 6'', with the long dimension along the warp, were run in each determination

and the average of the ten samples reported as the tensile strength of the sample. The ends of the strips were, prior to pulling, sealed with an adhesive to prevent fraying of the ends.

The abrasion index was determined as follows on the fired samples. The abrasion machine is illustrated in Fig. 9.

Mounted on base 1 is a motor 2 operating a crank wheel 3 carrying an eccentric crank pin 3' and a crank arm 4 connected to a slide 5 slidably positioned in the guide 6. The slide carries a clamp 7 to which the fabric to be tested is clamped. A flexible bracket 9 and 9' formed of wire is mounted on the edge of the base, and each wire is looped into loops 10, 11, and 12. Polished rods are loosely mounted in the loops 11 and 12 and the fabric 8 to be tested is threaded underneath 13 and over 14 and descends, held by a weight 16 connected to the end of the fabric. A platform in the form of a plate is mounted underneath the weight 16. The platform is hinged on an arm 18 which is connected to a switch in the motor circuit. A revolution counter is connected to the crank wheel 3.

The arm moves back and forth in a 2¼'' stroke in a horizontal plane at the rate of nine complete strokes per minute, i. e., the crank wheel rotates at 9 R. P. M. The angles of contact with the rods are indicated on the drawing. The rods are placed approximately 1¾'' on center with the result that each side of the cloth is abraded by one rod except in the middle for a space of about ½'' where the abrasion is against both sides. A 100 gram weight is employed. The reciprocation of the slide draws the fabric over the polished rods, which rotate as the cloth is drawn over them under the tension of the weight. The weight reciprocates above the plate without touching the plate 19. When the cloth is abraded to reduce the tensile strength sufficiently so that the cloth breaks, the weight falls and this opens the switch and stops the motor.

All the samples used in the abrasion test were cut ten threads wide and ten inches long along the fill of the cloth. The abrasion index is reported as the number of complete cycles, i. e., revolutions of the crank at which the weight falls. Duplicate samples were run for check.

Table I gives the following data:

Column 1, sub-column A gives the per cent of acid employed in the treatment. Sub-column B gives the temperature at which the treatment was carried on. Sub-column C gives the duration of the treatment.

Column 2 gives the per cent loss of material on ignition and includes the lubricant present in the original fabric and the water hydration, and column 3 is the difference between the per cent of the material soluble in sodium carbonate and this volatile fraction, and therefore is the per cent present as soluble silica calculated to a volatile free basis. This is the quantity referred to throughout this specification and in the figures as per cent soluble silica.

Column 4 gives the per cent of the silica present in the fired sample, that is, the sample fired at 1600° F. in one-half hour, as determined by the above test procedure. This test procedure does not differentiate between boron and silica and this silica may include a small fraction of boron.

Column 5 is the abrasion index as described above.

Column 6 gives the tensile strength as determined by the above identified test procedure.

Table I

	1 Leaching conditions			2 Percent volatile portion	3 Percent soluble silica	4 Percent total silica (fired)	5 Abrasion (fired only)	6 Tensile strength (lbs. fired)
	a	*b*	*c*					
	Pct.	*Deg.*						
(A)	7	140	½ hr.	5.53	37.80	62.46	5	26.7
			1½ hr.	9.12	76.99	92.75	16	31.6
			2 hr.	9.91	79.32	94.64	35	37.7
			3 hr.	9.57	84.69	98.45	180	43.3
			4 hr.	8.63	87.39	98.84	238	53.8
(B)	7	160	½ hr.	6.43	49.59	71.95	12	27.1
			1½ hr.	9.96	79.88	97.23	35	32.3
			2 hr.	10.31	81.75	97.71	99	36.4
			3 hr.	10.83	84.01	98.72	223	45.4
			4 hr.	9.70	85.58	99.01	208	34.1
(C)	7	180	½ hr.	6.74	47.14	71.89	6	26.3
			1 hr.	7.87	59.19	76.58	18	29.1
			1½ hr.	8.97	81.90	95.22	13	25.3
			2 hr.	9.12	84.76	97.59	69	28.5
			3 hr.	9.53	83.41	98.49	79	31.3
(D)	9	140	½ hr.	6.12	38.98	71.03	5	21.1
			1 hr.	9.80	70.79	88.23	12	27.4
			1½ hr.	9.99	85.16	98.81	162	41.2
			2 hr.	10.19	85.46	98.90	200	37.9
			3 hr.	9.11	86.94	99.08	138	32.9
(E)	9	160	½ hr.	7.97	50.41	72.52	3	22.9
			1 hr.	11.11	81.67	95.87	159	40.7
			1½ hr.	9.55	86.77	98.93	157	41.3
			2 hr.	9.75	87.08	99.05	181	33.7
			3 hr.	8.78	88.53	99.13	185	31.8
(F)	9	180	15 min.	6.14	45.86	70.32	1	25.9
			½ hr.	8.88	72.57	91.52	16	27.5
			1 hr.	9.59	86.52	98.24	119	33.2
			1½ hr.	9.92	87.39	99.12	98	31.8
			2 hr.	9.71	87.55	99.12	86	28.1
(G)	12	140	15 min.	4.13	28.44	52.97	2	23.3
			½ hr.	7.41	54.94	79.29	31	28.8
			1 hr.	9.77	82.96	97.27	159	41.6
			1½ hr.	10.06	85.11	98.88	249	40.2
			2 hr.	8.74	87.08	98.93	156	35.2
(H)	12	160	10 min.	3.80	18.65	50.57	3	21.8
			20 min.	7.16	52.30	75.16	16	22.8
			½ hr.	8.79	68.42	87.87	60	34.7
			1 hr.	8.66	86.86	98.84	210	36.3
			1½ hr.	9.91	86.73	99.12	143	31.1
(I)	12	180	5 min.	4.33	27.39	56.74	1	23.1
			15 min.	9.23	60.74	82.48	14	24.8
			½ hr.	9.01	84.38	98.24	149	33.0
			1 hr.	11.31	84.77	99.01	102	28.9
			1½ hr.	8.44	87.90	99.11	83	25.7

Referring to Figs. 1, 2, and 3, broken lines A to I, inclusive, (charts) the values of column 3 against 1–C (Table I) for the experiments using 7% acid at 140° F., 160° F., and 180° F., respectively (Fig. 1), 9% at 140° F., 160° F., and 180° F. (Fig. 2); 12% at 140° F., 160° F., and 180° F. (Fig. 3), it will be observed that the original samples had 1% or less of glass soluble in sodium carbonate, calculated upon a volatile free basis. The only other material which apparently is soluble is the lubricant portion to the extent of 2.09%. It will also be observed that the soluble silica is developed according to two processes. There are apparently two reactions occurring at different rates.

The major process for the development of soluble silica is at a very rapid rate and it is accompanied by a slow process which develops soluble silica at a much lower rate. This is illustrated by the broken lines formed by two intersecting lines, one of large slope and one of small slope. Thus, the slope of lines A–A1 is very steep while the slope of the lines A–1—A–2 is less steep. The major portion of the soluble silica is developed along the line A–A1 during the first hour. The process proceeds in the next three hours developing only a small amount of soluble silica along line A–1—A–2. This phenomenon is identical in the lines B to I, inclusive. Following the usual chemical kinetic criteria, the net slope of the process occurring during the first hour, i. e. along A–A1 is obtained by subtracting the slope of the line A1—A2 from the slope of the line A–A1. Similarly, the net slope is obtained on each of the lines B to I, inclusive. This net slope is the reaction rate of the major or fast reaction of production of soluble silica expressed as per cent silica per minute.

On Figs. 4 to 6, inclusive, and curves J to S, inclusive, is plotted the values of the abrasion index against the values of the per cent soluble silica of the samples tested. In other words, these are plots of values of column 5 against column 3 of Table I for each of the tests reported. In curves J′ to S′ is plotted the value of the tensile strength against the values of the per cent soluble silica in the samples tested, i. e., value in column 3 against the values in column 6.

It will be observed that in each of the cases, curves J to S and J′ to S′, the original cloth had a tensile strength and abrasion index in excess of any of that of the extracted cloths. The tensile strength and the abrasion index of the cloth are largely depreciated by leaching and firing. Further, it will be observed as the leaching progressed and as the soluble silica developed to above about 30%, the tensile strength and abrasion index appreciated, coming to a maximum when the soluble silica was in the range of about 70 to 87%.

Depreciation again sets in and is sharper when the per cent of soluble silica is 2 to 4% greater than it is at the value of the soluble silica at the maximum percentage as the soluble silica falls to almost 50% of the maximum.

Reference to Table II will indicate that the maximum for the abrasion index occurs at substantially the same percentage as the maximum for the tensile strength, confirming the phenomena with regard to tensile strengths.

Table II

Leaching conditions		Reaction rate percent sol. SiO_2/min.	Tensile strength		Abrasion index	
(*a*) Conc., percent acid	(*b*) Temp., ° F.		(*a*) Max. att.	(*b*) percent sol. SiO_2 max. att.	(*a*) Max. att.	(*b*) percent sol. SiO_2 max. att.
7	140	1.16	48+	87.39	238+	87.39
7	160	1.59	45.4	84.01	223	84.01
7	180	1.46	31.5+	83.41	79+	83.41
9	140	1.20	41.2	85.16	200	85.46
9	160	1.47	41.3	86.77	181	87.08
9	180	2.48	33.2	86.52	119	86.52
12	140	1.77	41.6	82.96	249	85.11
12	160	2.23	36.3	86.86	210	86.86
12	180	3.98	33.0	84.38	149	84.38

Table II gives the value of the maximum tensile strength and the maximum abrasion index obtained at the various rates of attack in the experiments. In columns 1*a* and 1*b* is given the conditions of the experiment in acid concentration and temperature. Column 2 gives the rates of reaction (net slope) in per cent of soluble silica (volatile free basis) per minute. Column 3*a* gives the maximum recorded tensile strength and 3*b* the per cent soluble silica of the sample (on a volatile free basis) showing the maximum tensile strength; column 4*a* gives the maximum recorded abrasion index, and column 4*b* the per cent soluble silica (on a volatile free basis) of such sample.

It will be observed that the per cent soluble silica at the maximum is in the range of 82.96% to 87.08%. The values for the 7%-140° F. experiment and for the 7%-180° F. were not carried far enough to show the depreciation, i. e., in these

tests the per cent soluble silica at the turning point is in excess of those indicated.

In Fig. 8 is plotted the values of column 4 as against 4*b*, and in Fig. 9 is plotted the values of column 3*a* against 3*b*. The dotted portions are extrapolated. It is apparent that in both of these figures the highest value of the maximum tensile strength and abrasion index is in the region between 1.6% per minute and 1.8% per minute soluble silica and that there is a large appreciation in the value as the rate rises above about 1% per minute to reach a maximum in the region of 1.6% to 1.8% per minute and again falls off as the value of about 2.5% is reached, and in this range of about 1 to 2.5% per minute will be obtained the highest values of the maximum.

It appears that there is thus another criteria for the development of maximum tensile strength and abrasion index in addition to the limitation of the development of the soluble silica within the range of 70% to 87%, to wit, that not only should the per cent soluble silica be controlled to be within the range of 70% to 87% and preferably in the range of about 78 to 87%, but there is a rapid increase in the value of the maximum tensile strength and abrasion index available as the rate of reaction increases from about 1 to 2.5% of soluble silica per minute.

It therefore appears that it would be desirable in extracting fabric made of glass fibers that the reaction rate be controlled in the region of about 1 to 2.5% of soluble silica per minute and preferably that it be held at the region of 1.6 to 1.8% of soluble silica per minute, and in this manner the highest tensile strengths will be developed and an extracted fabric of the greatest abrasion resistance also developed.

While the exact values of the optimum range of per cent soluble silica and reaction range may be somewhat different for various samples of cloth, braid, tape, or rope, woven, braided or knitted in different manners and for leachable glass of different compositions and for different rates of attack, the discoveries herein described are believed applicable thereto and the methods herein disclosed will permit those skilled in the art to repeat these tests on the fabric under consideration to select the best rates of attack and to select the per cent soluble silica at which the attack is to be stopped to obtain material of the highest tensile strength and abrasion resistance desired for the use of the material.

Reference has been had to the use of HCl acid as an example of the leaching solutions. As reference to the above prior art will indicate, the glass may also be leached, depending on its composition, with water and, if acid leachable, with other acids whose salts of the glass-forming oxides, other than SiO_2 present, are soluble or dispersable and therefore removable. Such other acids as heretofore referred to include HNO_3, acetic acid, trichloracetic acid, all of which will be comprehended within the term "acid." However, because of its utility and ease of handling and the solubility of its salts, the acid employed in the above experiments, i. e., hydrochloric acid, is the preferred acid to use.

While we have described a particular embodiment of our invention for the purpose of illustration, it should be understood that various modifications and adaptations thereof may be made within the spirit of the invention as set forth in the appended claims.

We claim:

1. A process for leaching glass fibers, which comprises leaching glass fibers with an acid solution at a leaching temperature to remove the non-siliceous metal oxides from the glass fibers and to convert vitreous silica into sodium carbonate soluble silica, continuing the leaching until the content of sodium carbonate soluble silica in the leached fibers, when calculated on a volatile free basis, is in excess of 30% and less than 89%, interrupting the leaching of said fibers, and washing said leached fibers.

2. A process for leaching glass fibers, which comprises leaching glass fibers with an acid solution at a leaching temperature to remove the non-siliceous metal oxides from the glass fibers, and to convert vitreous silica into sodium carbonate soluble silica, continuing the leaching until the content of sodium carbonate soluble silica in the leached fibers, when calculated on a volatile free basis, is in the range between 70% and 88%, interrupting said leaching of said fibers, and washing said leached fibers.

3. A process for leaching glass fibers, which comprises leaching glass fibers with an acid solution at a leaching temperature to remove the non-siliceous metal oxides from the glass fibers and to convert vitreous silica into sodium carbonate soluble silica, continuing the leaching until the content of sodium carbonate soluble silica in the leached fibers, when calculated on a volatile free basis, is in excess of 82% and less than 88%, interrupting said leaching of said fibers, and washing said leached fibers.

4. A process for leaching glass fibers, which comprises leaching glass fibers with an acid solution at a leaching temperature to remove the non-siliceous metal oxides from the glass fibers and to convert vitreous silica into sodium carbonate soluble silica, continuing the leaching until the content of sodium carbonate soluble silica in the leached fibers, when calculated on a volatile free basis, is in excess of 30% and less than 89%, interrupting said leaching, washing said leached fibers to remove the acid, and firing said fibers to dehydrate said fibers.

5. A process for leaching glass fibers, which comprises leaching glass fibers with an acid solution at a leaching temperature to remove the non-siliceous metal oxides from the glass fibers and to convert vitreous silica into sodium carbonate soluble silica, continuing the leaching until the content of sodium carbonate soluble silica in the leached fibers, when calculated on a volatile free basis, is in excess of 70% and less than 88%, interrupting said leaching, washing said fibers to remove the acid, and firing said fibers to dehydrate said fibers.

6. A process for leaching glass fibers, which comprises leaching glass fibers with an acid solution at a leaching temperature to remove the non-siliceous metal oxides from the glass fibers and to convert vitreous silica into sodium carbonate soluble silica, continuing the leaching until the content of sodium carbonate soluble silica in the leached fibers, when calculated on a volatile free basis, is in excess of 82% and less than 88%, interrupting said leaching, washing said fibers to remove the acid, and firing said fibers to dehydrate said fibers.

7. A process for leaching glass fibers, which comprises leaching glass fibers with an acid solution to remove the non-siliceous metal oxides by said leaching from said glass fibers and to convert vitreous silica into sodium carbonate soluble silica at a rate of from about 1 to 2.5% per minute, calculated on a volatile free basis, continu-

ing the leaching until the content of the soluble silica, on a volatile free basis, is in excess of 30% and less than 89%, interrupting said leaching of said fibers, and washing said leached fibers.

8. A process for leaching glass fibers, which comprises leaching glass fibers with an acid solution to move the non-siliceous metal oxides by said leaching from said glass fibers and to convert vitreous silica into sodium carbonate soluble silica at a rate of from 1 to 2.5% per minute, calculated on a volatile free basis, continuing the leaching until the content of the soluble silica on a volatile free basis is in excess of 70% and less than 88%, interrupting said leaching, and washing said leached fibers.

9. A process for leaching glass fibers, which comprises leaching glass fibers with an acid solution to remove the non-siliceous metal oxides by said leaching from said glass fibers and to convert vitreous silica into sodium carbonate soluble silica at a rate of from 1 to 2.5% per minute on a volatile free basis, continuing the leaching until the content of the soluble silica on a volatile free basis is in excess of 82% and less than 88%, interrupting said leaching, and washing said leached fibers.

10. A process for leaching glass fibers, which comprises leaching glass fibers with an acid solution to remove the non-siliceous metal oxides by said leaching from said glass fibers and to convert vitreous silica into sodium carbonate soluble silica at a rate of from about 1 to 2.5% per minute, on a volatile free basis, continuing the leaching until the content of the soluble silica on a volatile free basis is in excess of 30% and less than 89%, interrupting said leaching, washing said leached fibers, and firing said fibers to dehydrate said fibers.

11. A process for leaching glass fibers, which comprises leaching glass fibers with an acid solution to remove the non-siliceous metal oxides by said leaching from said glass fibers and to convert vitreous silica into sodium carbonate soluble silica at a rate of from about 1 to 2.5% per minute, on a volatile free basis, continuing the leaching until the content of the soluble silica on a volatile free basis is in excess of 70% and less than 88%, interrupting said leaching, washing said leached fibers, and firing said fibers to dehydrate said fibers.

12. A process for leaching glass fibers, which comprises leaching glass fibers with an acid solution to remove the non-siliceous metal oxides by said leaching from said glass fibers and to convert vitreous silica into sodium carbonate soluble silica at a rate of from 1 to 2.5% per minute, on a volatile free basis, continuing the leaching until the content of the soluble silica on a volatile free basis is in excess of 82% and less than 88%, interrupting said leaching, washing said leached fibers, and firing said fibers to dehydrate said fibers.

13. A process for leaching glass fibers, which comprises leaching glass fibers with an acid solution to remove the non-siliceous metal oxides by said leaching from said glass fibers and to convert vitreous silica into sodium carbonate soluble silica at a rate of 1.6 to 1.8% per minute, on a volatile free basis, continuing the leaching until the content of the soluble silica on a volatile free basis is in excess of 30% and less than 89%, interrupting said leaching, washing said leached fibers and firing said fibers to dehydrate said fibers.

14. A process for leaching glass fibers, which comprises leaching glass fibers with an acid solution to remove the non-siliceous metal oxides by said leaching from said glass fibers and to convert vitreous silica into sodium carbonate soluble silica at a rate of from 1.6 to 1.8% per minute, on a volatile free basis, continuing the leaching until the content of the soluble silica on a volatile free basis is in excess of 70% and less than 88%, interrupting said leaching, washing said leached fibers, and firing said fibers to dehydrate said fibers.

15. A process for leaching glass fibers, which comprises leaching glass fibers with an acid solution to remove the non-siliceous metal oxides by said leaching from said glass fibers and to convert vitreous silica into sodium carbonate soluble silica at a rate of from 1.6 to 1.8% per minute, on a volatile free basis, continuing the leaching until the content of the soluble silica on a volatile free basis is in excess of 82% and less than 88%, interrupting said leaching, washing said leached fibers, and firing said fibers to dehydrate said fibers.

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