Wet-formed nonwoven webs from high-performance fibers

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While wet forming is a technology ideally suited to producing isotropic or directional nonwoven webs from both organic and inorganic fibers, its success depends on a correctly sized system with appropriate capabilities.

The origin of wet-forming nonwoven webs is firmly rooted in paper technology. Much early work in this area was conducted on converted paper machines, many of which were totally unsuited to the process for which they are now being used.

Fortunately for the wet-forming sector, innovative machinery builders were able to devise systems which enabled man-made fibers to be dispersed in water, reduced to a very low dilution to ensure good fiber distribution, and then to form these very dilute suspensions of fibers into coherent, often self-bonding webs.

Machines such as the inclined wire, developed from the fourdrinier paper machine, allowed the large volumes of water required for low forming consistencies to be handled easily (**Figs. 1 and 2**). An alternate development, whose origin belonged more to the conventional cylinder mold machine, was a roll former (**Figs. 3 and 4**).

The relationship between these various process configurations was examined by Magill (*1*), and **Fig. 5** is taken from this study. This plot of product and process types shows that roll formers are remarkably versatile; our own work has shown that the range covered with inorganic fibers can equal the performance of the inclined-wire machine, except with longer fibers (above 15 mm), which are seldom required in the type of products studied.

Our studies, therefore, concentrated on a basic roll former configuration shown in **Fig. 6**, which was successfully employed in the production of webs from 15 g/m^2 to 1000 g/m^2. This basic former could be extended by means of a wire section to provide the facility for multilayer operation.

Fibers

"High performance" can be applied to a wide variety of fibrous materials now available because there is no generally accepted definition. The generalization can be applied to fibers with high modulus, to fibers with high temperature stability, or to others that have high tensile strength.

For the purpose of our studies we have included the following fiber types:

- Glass microfibers
- Silica fibers
- Ceramic fibers
- Polyimides
- Carbon.

Glass microfibers

Glass microfibers are produced from boro-silicate-type glass consisting of sand, borax, feldspar, soda ash, fluorspar, barium carbonate, zinc oxide, and cullet.

These materials are fed into a natural gas-fired furnace at a certain ratio which produces a uniform molten flow of glass. The molten glass is formed into 16-mm-diameter marbles which are then formed into fibers. The fibers are created using the flame attenuation method; the marbles are remelted in a pot and drawn through a bushing. The formed fibers are then exposed to a high temperature, high velocity gas flame. This resoftens and attenuates the fiber to a fine diameter. The fibers are collected in a chamber and removed by a metal-link screen.

By varying the components, different types of glass may be produced. Typical chemical analyses of three different glass types are shown in **Table I**.

An important characteristic to be considered in the selection of glass microfibers for wet-formed nonwovens is the weight loss from the fibers when exposed to acid, alkali, or prolonged exposure to water at elevated temperatures. Results from laboratory tests on different types of microfibers are shown in **Table II** (*2*).

Probably the most important physical characteristic of glass microfibers of concern to the web producer is the fiber diameter. Manufacturers normally produce microfibers with grade designations. The data from one major producer are shown in **Table III**.

The fiber diameter distribution samples of microfiber can be evaluated for fiber diameter using the scanning

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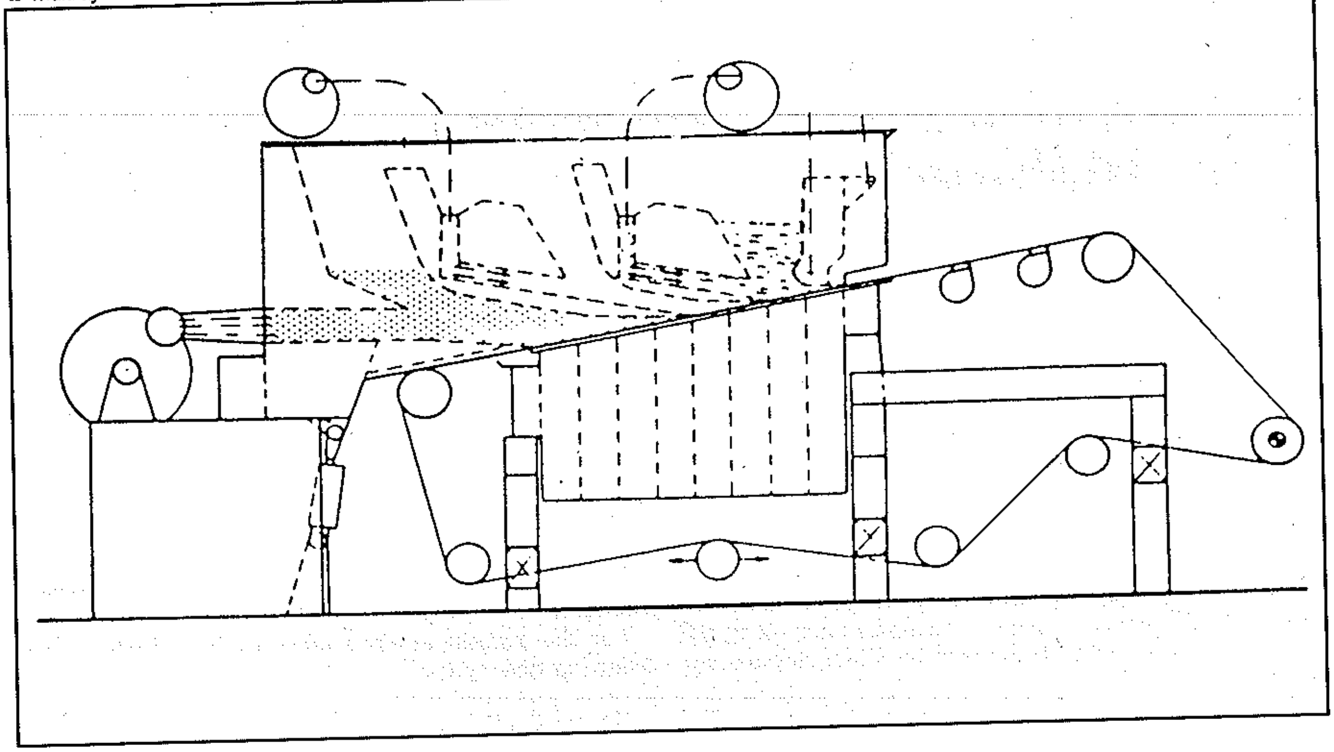
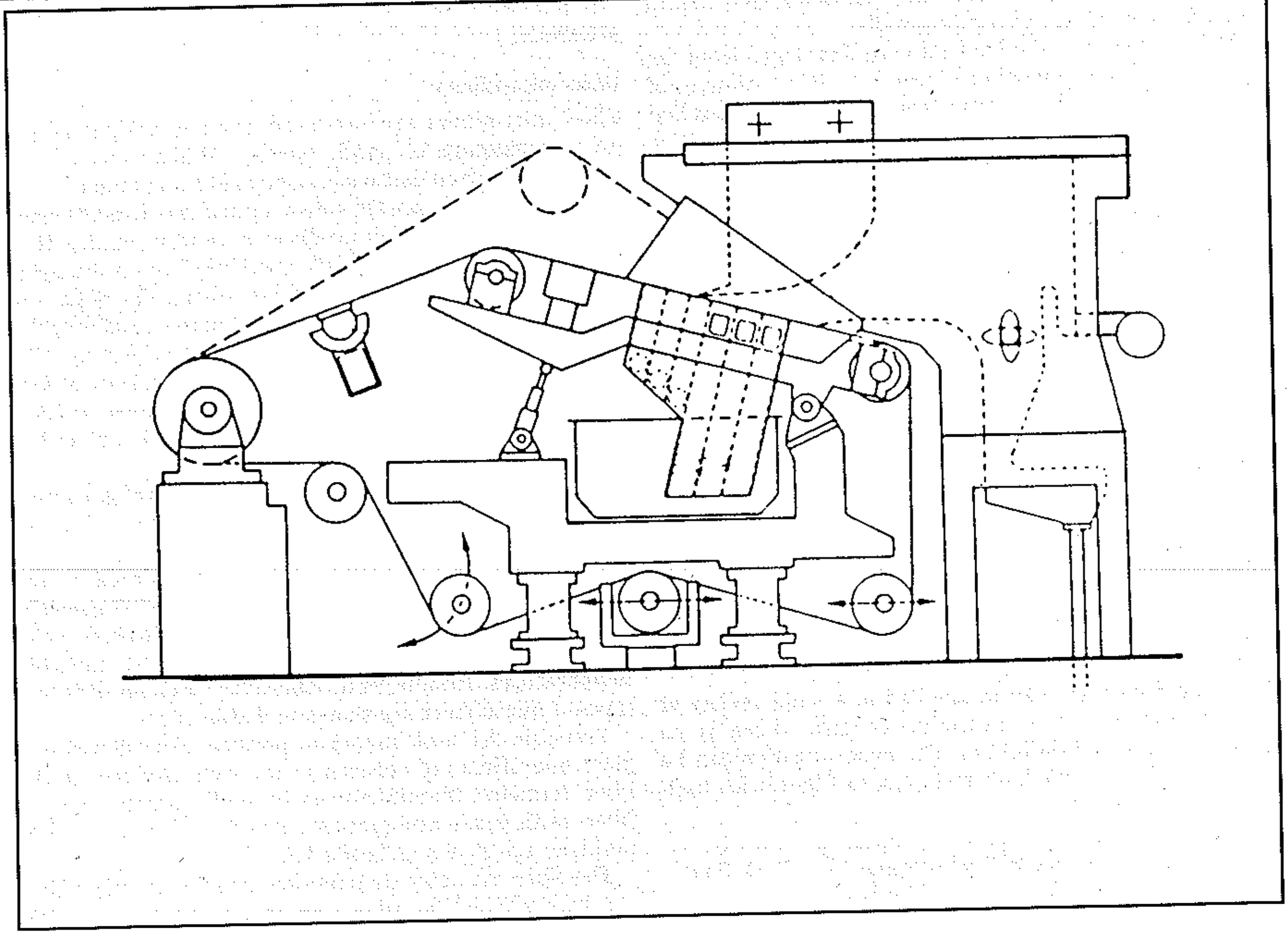
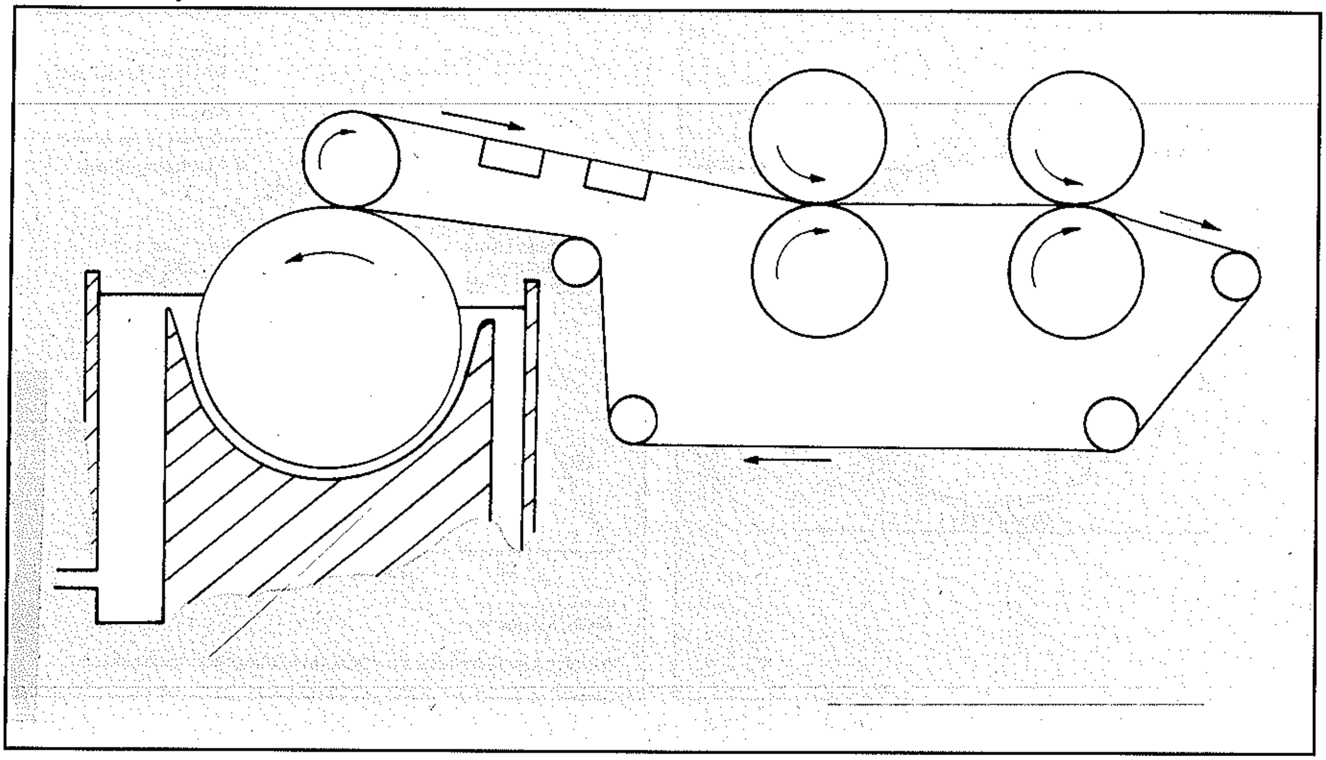
1. Multilayer inclined-wire machine



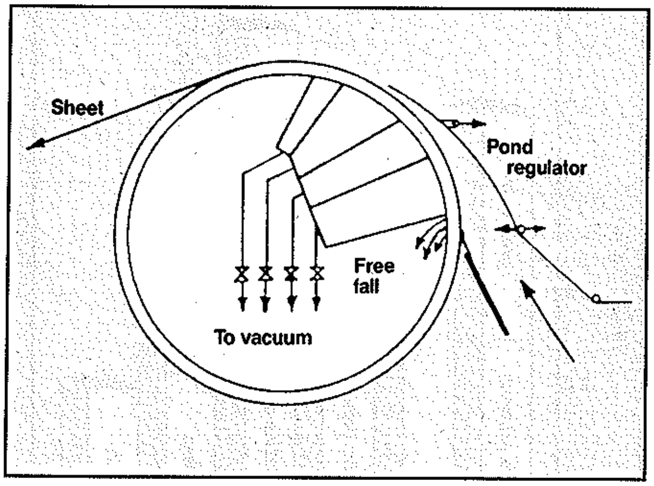
2. Inclined-wire machine



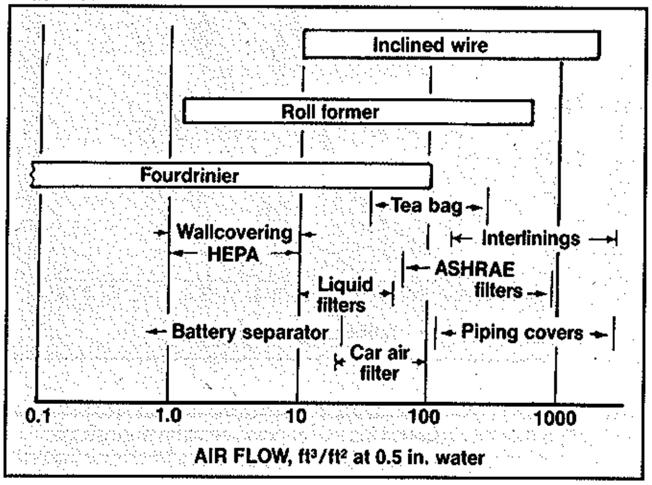
4. Conventional cylinder mold machine



3. Roll former



5. Comparison of flat-wire fourdrinier, roll former, and inclined-wire machines



electron microscope (SEM). Results of such an evaluation on the same manufacturer's fibers (shown in Table III) are given in **Figs. 7 and 8**.

These results are prone to some error in the top end of the distribution since the higher-diameter fibers are not "seen" by the SEM on a representative basis. The curves shown, however, give a reasonably good idea of the relative diameter distributions of these products.

The curves shown in Fig. 7 give the diameter distribution on a weight basis. For air filtration applications, the mean surface diameter is more meaningful. Figure 8 shows the surface diameter distributions of the various grades taken from the SEM data of Fig. 7.

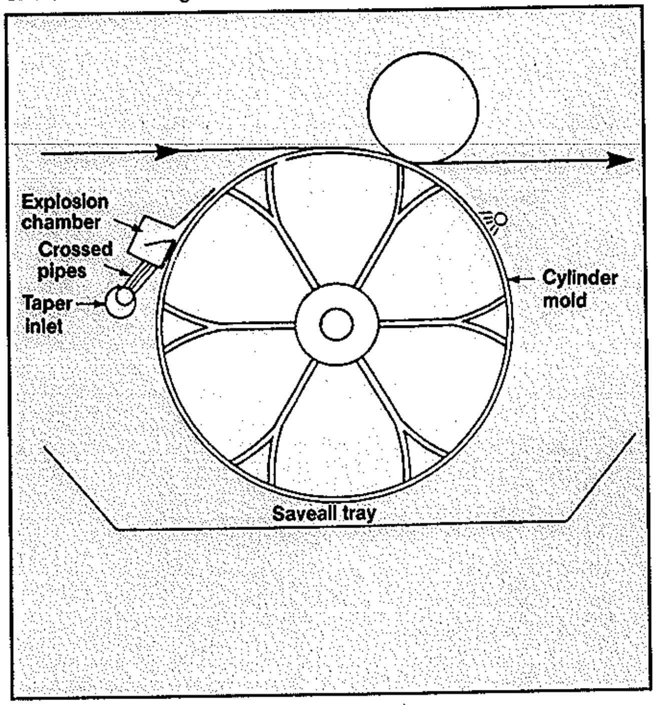
Alumina fibers

A number of developments have taken place recently that have resulted in several lower-cost alumina fibers being available. Fine-diameter (3 μm) alumina fibers possess high strength and modulus. The fibers can be formed into a web which can then be used in metal-matrix composites.

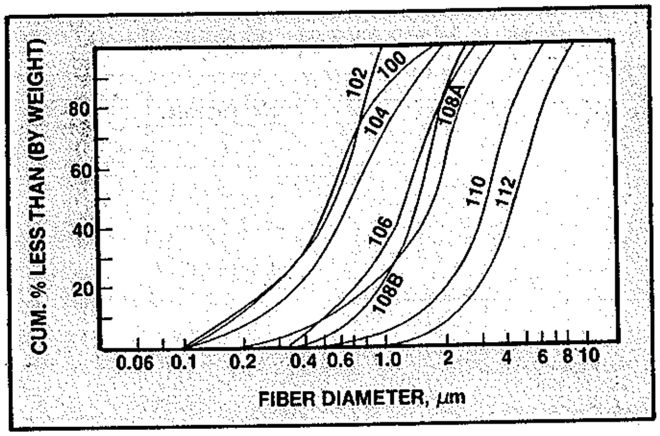
The polycrystalline fibers comprise 95% alumina and 5% silica and are available in two grades: low density and standard density. The important properties of these two fibers are shown in **Table IV**.

Costs will be in the order of $110/kg, compared with current Japanese fibers at $500/kg. This lower cost, combined with their excellent resistance to molten light alloys, should make these fibers of great potential use (*3*).

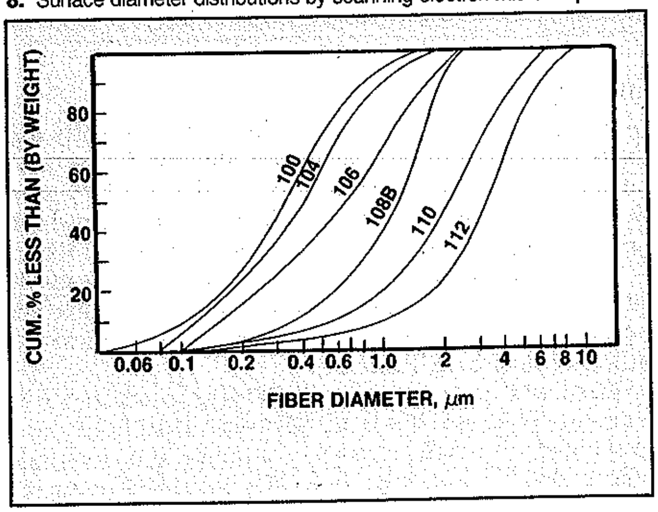
6. Roll former configuration



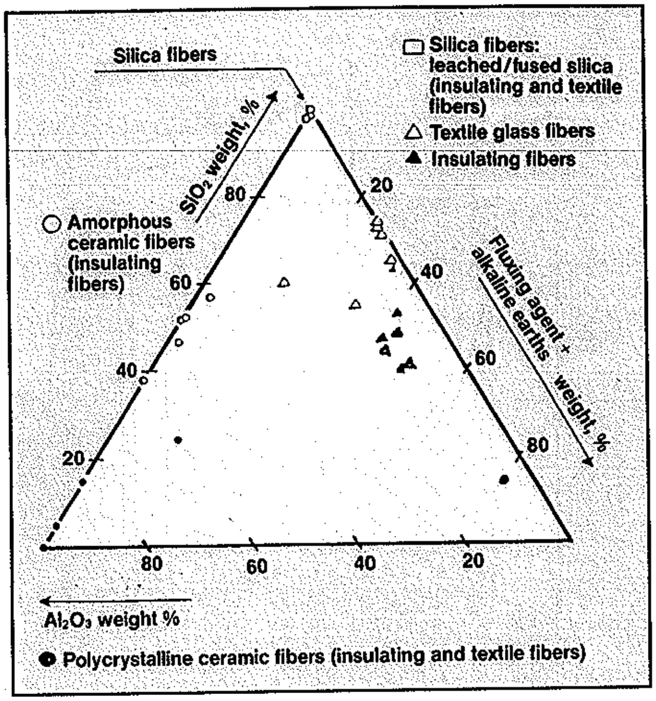
7. Fiber diameter distribution by scanning electron microscope



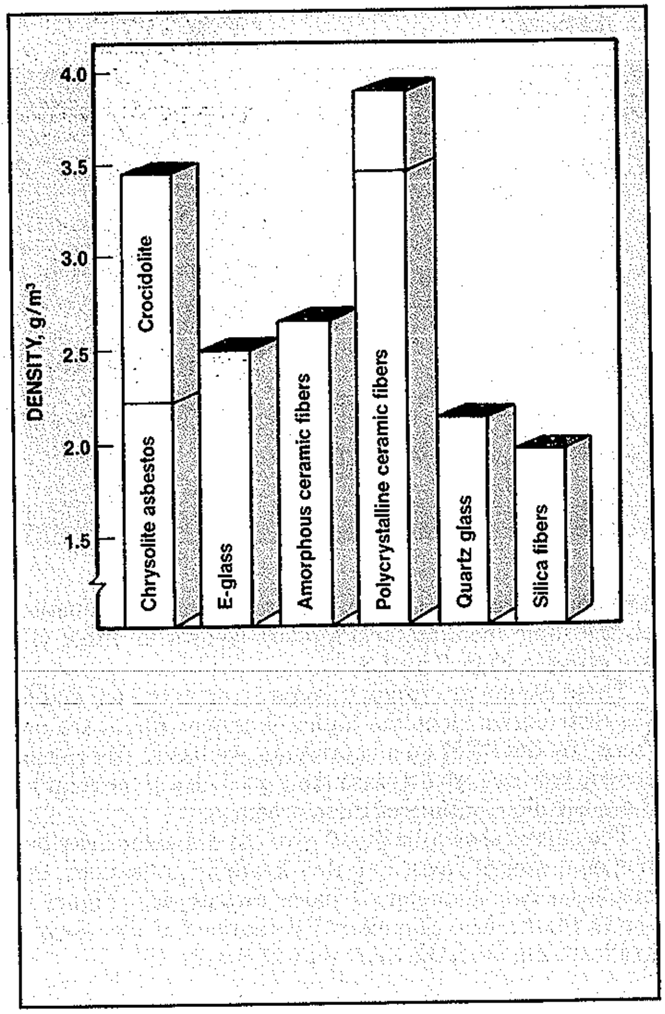
8. Surface diameter distributions by scanning electron microscope



9. Three-phase diagram of silica fibers



10. Comparison of the density of inorganic fibers



I. Typical analyses of different types of glass

Method of analysis	475 Borosilicate glass	753 Borosilicate glass	E glass (electrical, alkali-free)
SiO_2	57.9	63.4	54.4
B_2O_3	10.7	5.6	8.0
Na_2O	10.1	14.6	0.4
Al_2O_3	5.8	5.3	14.1
Fe_2O_3	.1	.2	0.3
BaO	5.0	...	...
ZnO	3.9	...	...
K_2O	2.9	1.1	...
CaO	2.6	6.1	17.4
MgO	0.4	3.0	4.7
F_2	0.6	0.7	0.2
TiO_2	...	...	0.5

II. Laboratory test results on weight loss of different fiber types

		475	753
Percent weight loss in 1.0 *N* H_2SO_4 for 1 h at room temperature	Code 106	3.59	4.10
	Code 112	0.83	1.01
		475	753
Percent weight loss in 1.0 *N* H_2SO_4 for 1 h at 100°C	Code 106	6.17	5.89
	Code 112	0.90	0.81
		475	E glass
Percent weight loss for 1 μm fiber, 1 h boil, 0.1. N NaOH		8.14	9.7
		475	E glass
Percent weight loss for 5μm fiber, 1 h at 100°C H_2O (tap water)		1.2	1.3

III. Microfiber grade designations

Code 100	0.29–0.32 μm[a]
Code 102	0.33–0.38 μm
Code 104	0.39–0.53 μm
Code 106	0.54–0.68 μm
Code 108B	0.69–1.1 μm
Code 108B	1.2–2.4 μm
Code 110	2.5–4.0 μm
Code 112	2.6–3.8 μm[b]

[a]Based on typical values obtained using Williams Freeness Test (FG - 436-209): calibrated by B.E.T. surface area, Krypton absorption.

[b]Determined by Micronaire J-M Test No. 436-202.

IV. Properties of alumina fibers

	Low density	Standard density
Density, g/cm³	2.0	3.3
Elastic modulus, GPa	200	300
Tensile strength, GPa	2.0	2.0
Maximum service temperature, °C	900	1600

Silica fibers

Synthetic inorganic fibers may be divided into three categories:

- Amorphous fibers
- Polycrystalline fibers
- Monocrystalline fibers (normally called whiskers).

This type of classification shows the wide ranging composition of inorganic fibers running from carbon fibers to tungsten fibers, with an important group of amorphous textile fibers made from silica as part of the system.

Figure 9 compares the composition of glass and ceramic fibers with amorphous silica in a three-phase diagram of alumina, silica, and fluxing agents such as alkaline earths or boron oxide (*4*).

Table V lists some important properties of recently available amorphous silica fibers.

The density of the silica filaments and fibers is within a range of 1.8 g/cm³ to 2.0 g/cm³, a property which naturally counts in those fields of application where saving in weight is of importance. They are the lightest inorganic fibers or filament yarns known (**Fig. 10**). The following advantageous properties are now available in amorphous silica fibers because of the high chemical purity of 99.6% SiO_2 that can be achieved:

- Good thermal resistance (up to 1100°C)
- Low specific weight (2.0 g/cm³) compared with other inorganic fibers
- Good thermal and electrical insulation properties
- Very low thermal expansion coefficient achievable.

Silica fibers find applications in composite materials as illustrated in **Table VI**. Web forms of silica fibers (wet formed nonwovens) are an appropriate way of handling these fibers.

Ceramic fibers

Ceramic fibers are available in many forms. The type used in these studies was a washed fiber, which removes much of the nonfibrous material (known as shot) that is present. Prior to use in these trials, it was washed further using

V. Properties of silica fibers

Type of silica fiber	*Low temp.*	*High temp.*
Density, g/cm^3	1.8	2.0
Spec. pore volume, mL/g	0.2	...
Average pore diameter, nm	2	...
Tensile modulus (E), GPa	13	56
Tensile strength (R_H), MPa	250	800
Elongation at break (E_H)	2.0	1.4
Reciprocal loop ratio (*d*), %	6.5	2.6
Shrinkage		
to 600°C, %	6.6	0.1
to 1000°C, %	9.6	1.2

VI. Silica fiber applications in composite materials

Composite materials (organic matrix)

Thermal and electrical composites
- Aviation, astronautics
- Automotive industry
- Electronic industry

Composite materials (inorganic matrix)

Thermal and electrical ceramics
- Electrical industry
- Aviation, astronautics
- Furnace building

VII. Properties of ceramic fibers

Typical physical properties	
Color	White
Continuous use limit	1260°C (2300°F)
Melting point	1790°C (3260°F)
Normal packing density	96–1922 kg/m^3 (6–12 lb/ft^3)
Approximate shipping density	64–96 kg/m^3 (4–6 lb/ft^3)
Fiber diameter	2–3 μm (mean)
Specific gravity	2.73 g/cm^3
Specific heat at 1093°C (2000°F)	1130 J/kg°C (0.27 Btu/lb °F)
Fiber surface area	0.5 m^2/g

VIII. Typical chemical analysis of ceramic fibers

Typical chemical analysis	
Al_2O_2	49.2%
SiO_2	50.5%
Na_2O	0.2%
Fe_2O_3	0.06%
K_2O	0.03%
TiO_2	0.02%
Leachable chlorides	<10 ppm

a waterfall method to remove the last traces of shot. This method was moderately successful. Even though we were willing to accept a relatively high fiber loss, we could not entirely eliminate the last small portion of shot material, which eventually made its way into the sheet.

In some applications this can be a problem, but the webs produced during the program were, compared to commercially available samples, essentially shot-free.

Ceramic fibers provide:

- High temperature stability
- Low thermal conductivity
- Low heat storage
- Excellent thermal shock resistance
- Light weight
- Superior corrosion resistance.

Table VII shows typical physical properties for washed ceramic fiber, and **Table VIII** gives typical chemical analysis figures.

Figure 11 illustrates the thermal conductivity of a 96-kg/m^3 structure of ceramic fibers. Nonwoven structures of 1 mm and 2 mm most often are used to provide effective, lightweight, low density, thermal barriers.

High-performance organics

A wide range of organic fibers are now available, at least in experimental quantities, to be investigated.

These range from the meta-aramids such as DuPont's Kevlar® fiber, the para-aramid (Technora®) polyimides, polyethermides, polyoxamides, and polyether ether ketones.

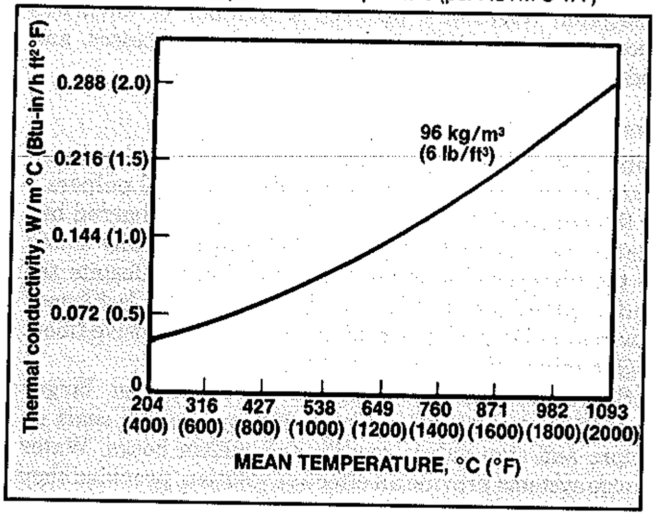
Others, such as novoloid fibers (cross-linked phenol-aldehyde fiber) have been known for some time and still can be used effectively as reinforcements for resin-based lining systems. These fibers are unlike most organics because they have an amorphous three-dimensional network polymer structure.

Using any of these fibers in wet-formed nonwovens requires that the polymer be available as a precisely-cut staple fiber, at an appropriate decitex (weight in grams of 10,000 m of single filament of the fiber; normally not greater than 3.3 dtex), and that it can be water dispersed.

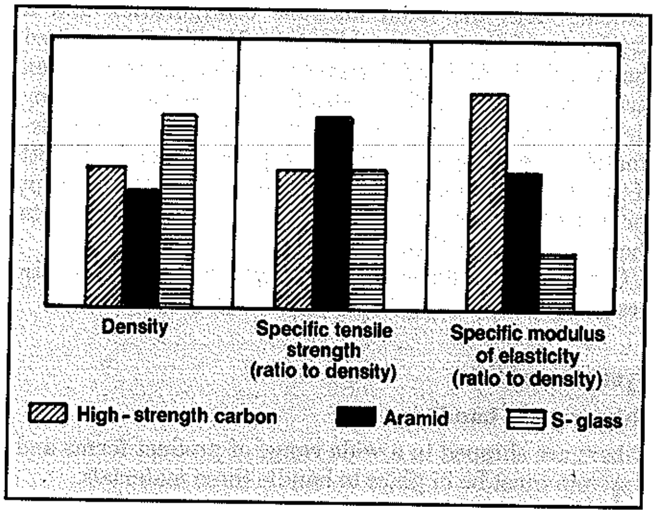
Each fiber presents a different set of requirements for web dispersion and some (Kevlar, for example) can be mechanically treated in a refiner system to produce a highly fibrillated fiber structure. This is a particularly useful aspect of these fibers derived from the structure of the fibers themselves.

It is, therefore, possible to produce webs of the Kevlar fiber that are sufficiently entangled to require very little

11. Thermal conductivity vs. mean temperature (per ASTM C-177)



12. Comparison of structural fibers



additional binder for some lightweight applications. An illustration of how the aramid fibers relate to carbon and glass is shown in **Fig. 12** (*5*).

Bonding

High-performance fibers do not exhibit an inherent ability to bond. With the lower-micron fiber diameters in glass there is often sufficient entanglement of these relatively long (in relation to their diameter) fibers to provide enough wet web strength for the web to form. However, unless some other form of bonding is created within the structure, it normally has insufficient strength to be of much practical use.

In the case of glass microfibers, the use of acid dispersion conditions promotes sufficient bonding between the fibers to produce adequate web strength without the need for additional binder systems. Stassen (*6*) provides some basic information on the mechanisms in this type of bonding, and White and Moore (*7*) showed how the dispersant effect of low-pH systems could be used to advantage in the development of ceramic and mineral-wool fiber webs.

In the case of other inorganic fibers, there is often a need to provide two levels of bonding.

Green strength binders are required to allow handling and to give flexibility to the fiber web for initial processing. Often these binders are subsequently removed by firing the web above the decomposition temperature of the binder system used. Work on the use of synpulps in this type of application with inorganic fibers has recently been reported (*8*), and these, together with low-melting-point polyesters PVA and bicomponent fibers, are the synthetic binders of choice for these nonwoven webs.

It is feasible to use a whole variety of other binders, but in many instances processing difficulties outweigh lower binder costs.

For permanent "in use" inorganic binders, the precipitation *in situ* of appropriate inorganic materials such as silica acid or aluminum hydroxide, can provide a totally inorganic system capable of a high degree of control.

Addition of inorganic binders by means of spraying the wet web has been successfully applied, but operational problems such as the spraying of sodium silicate solutions have prevented wider use.

For high-performance materials such as ceramic fiber, it is possible to use a lower-melting-point glass fiber system as the binder fiber. This can be effective in producing a well bonded, totally inorganic web.

High-performance organics pose different bonding problems, which are largely determined by the end use to which the web is to be put. Most of the organic binders used with inorganic fibers that will work with the organics and special-purpose binder systems are beginning to be developed (*9*).

Web forming

In theory any one of a number of processes currently used to form nonwoven webs could be used to handle high-performance fibers.

In practice, wet forming has proved to be a particularly suitable route for forming these fibers into a full range of fiber webs in terms of weight and in terms of directionality. It is quite feasible to produce a lightweight 10-g/m² web that is 100% isotropic or to produce other webs with a machine direction-cross direction ratio of 10:1.

However, the major problem to be addressed is not one of process technology per se, but one of appropriateness of scale. With fibers often costing $500/kg and more, there is a need to take advantage of the inherent characteristics of wet forming to produce extremely uniform webs, but to be able to do this on a scale appropriate to the costs of the fibers and binders involved and the requirements of the marketplace.

The concept of a small, highly flexible and readily changeable unit that can accommodate frequent cleanups and grade changes, pH ranges from 2 to 10, and operate with fibers from submicron diameter to 10 μm or more at fiber lengths from below 1 mm to 12–15 mm was developed.

Process technology

The process technology used in this work was based on the cylinder mold and developments from it, notably a roll former. Initial developments were concentrated on glass microfiber structure and nonwovens based on rayons (*10*) utilizing both the mold machine configuration and a Rotiformer. In a recent publication, Magill (*1*) examined the capabilities of three systems for wet-forming nonwoven structures. Figure 5 illustrates the ranges, as defined by air flow measurements on the web, that can be approximately covered by each system. A presentation by Elliott of the Herty Foundation (*11*) discusses a Rotonier concept for wet-formed nonwovens which is a similar concept to those developed in this program.

Considerable development work was involved in designing a small, flexible wet-forming system.

System outline

Shipping and handling

Fibers are shipped in a wide range of product forms and a system must be in place to handle these materials.

Good housekeeping, always an important consideration, is doubly important with fibers costing $500/kg and more.

Correct storage and stock control is also essential, as is accountability for fiber use and possible system loss.

Quality control

Quality control of all input materials is mandatory. Much improvement has been made in the cutting, packaging, and shipment of fibers, but problems can and do occur. Inorganic fibers can produce a number of surprises because of their difficult manufacturing processes, and these substandard materials must be removed.

Stock preparation

Fiber dispersion systems used have included rubber lined, stainless steel roll bars and small stainless steel pulpers.

Rubber lined, stainless steel roll bars, wide barred bed plates, and Hollander beaters have proved ideally suited to the dispersion of small batches of fibers. Despite the small quantities handled (5 kg), these "old fashioned" devices are ideal when working with very expensive fibers and demineralized water systems.

Small stainless-steel pulpers work well for larger quantities, but abrasive fibers destroy flexible seals on rotors and stop valves readily and within hours. Water purge systems provide an effective solution, provided they are kept under control.

A variable-speed drive is particularly useful in such a system.

Stock flow and storage

Piping systems were adopted that are fabricated in stainless steel with flangeless joints. This is achieved by having one section of pipe machined to fit into the second

Appendix: web characteristics

The web characteristics of sheets produced on this system are revealed in Tables IX–XIII.

IX. Manufacturing conditions of glass microfibers

Conditions			
Glass	100% Manville Code 106		
Water	Demineralized		
pH	3.4 with hydrochloric acid		
Dispersion	1.98% Consistency		
Storage	1.04% Consistency		
Mixing	0.06% Consistency		
Forming	0.03% Consistency		
Forming speed, m/min		50	72
Web moisture at pickup, %		88	90
Web moisture at press, %		82	84
Web moisture at dryers, I		58	65
Web moisture at dryers, S		33	58
Web moisture at reel, %		1.2	10.5
Basis weight, g/m²		52	52

X. Glass microfiber webs

Ref.	*F*	*A*	*A1*	*A2*
Manville 106 microfiber	100%	100%	100%	100%
Basis weight, g/m²	17	52	75	75
Thickness, μm	0.035	0.25	0.40	0.40
Density, g/cm³	0.50	0.48	0.52	0.52
Porosity, s/300 mL	3	12	14	13
Air flow, cm/s	50	50	50	50
Pressure drop, mm/Hg	2	14	10	12
Wet burst, g/cm²	N/A	140	210	210
Tensile, g/15 μm	120	540	680	600

section, producing a near-perfect internal seal. All other systems tried have resulted in one problem or another, and the high cost of the stainless fittings is more than justified.

Where required, good agitation is necessary, and care must be exercised with storage tanks so fibers do not break down with time.

Accurate consistency control by a microprocessor system is essential. Valves are always a problem in a wet-formed nonwoven system; avoid all unnecessary valves.

Linking accurate fiber weight control and accurate volume control of all water addition points with a microprocessor, it is possible to control the stock feed pump flow to deposit exactly the required amount of fiber to the flow box.

With consistency controlled throughout the system, the right web weight is automatically achieved without large recirculation systems, control valves, and dead spots in the system.

Accurate and known stock flow conditions make it easy to adjust pH and add chemicals and binders by proportional flow. If the stock flow stops or is stopped for any reason, the additive lines are automatically shut down and prevent system contamination. Control of the water volume and chemistry is critical (*6*).

With a system requiring frequent grade changes, it is also easy to program the system to automatically wash through, preventing deposit buildup within the system or the possibility of excessive corrosion.

Sheet forming

To achieve a wide range of web weights, including multilayer webs, a number of primary forming devices were constructed. These were based on:

- A contraflow cylinder mold
- A restricted-flow version of a cylinder mold
- A roll former that could be run as a cylinder or extended by the addition of a secondary forming fabric to accommodate multilayer secondary flow boxes.

The system was 0.5 m wide and could produce continuous web from 10 kg of fiber.

Web weights between 10 g/m² and 1000 g/m² were achievable, and multi-ply products up to three layers thick could be produced.

Speed control and sectional draw control were accurately monitored and data from all subsystems, such as vacuum, continuously monitored and controlled.

Webs were often "consolidated" by using wet presses fully felted with batt-on-base felts and very sensitive pneumatic controllers to facilitate almost zero roll-load effect at the nip.

XI. Commercially available glass microfiber filtration media

Grade	Basis weight, g/m²	Thickness, μm at 53 kPa	Porosity, Gurley s/100 mL	Pore size, μm Max.	Min.	Mean	Dry tensile, g/1.5 cm	Wet tensile, g/1.5 cm	Klemm test, S*
GF/A	53	260	4	4	1.5	1.9	240	110	2:15
GF/B	143	675	12	4	1.0	1.3	1050	715	2:33
GF/C	53	260	7	4	1.3	1.7	270	177	2:40
GF/D	120	675	3	12	4	6	390	290	1:0
GF/F	75	420	19	2.5	0.6	0.8	690	268	2:17
GF/AVA	48	303	3	9	2	3	640	324	2:23

*Time to rise 7.5 cm.

XII. Ceramic fiber web properties

Fiber	100% ceramic fiber water cleaned to remove most "shot." Average shot content less than 5%.			
Binder	Aluminum sulfate/ammonium hydroxide precipitation to produce *in situ* Aluminum hydroxide			
Fiber dispersion, pH	4.3			
Binder, % as aluminum sulfate	20			
Forming, pH	8.8	8.5	8.8	9.0
Basis weight, g/m²	106	120	150	182
Density, g/cm³	0.30	0.30	0.35	0.35
Permeability factor, m³/m²/s	400	234	210	200
Tensile, g/15 mm	95	108	215	234

XIII. Aramid fiber web properties

Fiber	Kevlar type 29—fibrillated pulp
Basis weight, g/m²	16.0
Thickness, mm	0.137
Density, g/cm³	0.11
Permeability factor, m³/m²/s	475
Tensile, g/15 mm	200

Drying was either over poly tetra fluoro ethylene-covered (*PTFE*) drying cans or with a through-dryer fitted with a carrier-wire system.

Reeling was performed with a separate drive-tension-controlled center wind.

Conclusions

We have examined a wide range of organic and inorganic high-performance fibers.

We have examined the requirements of these products in the marketplace and concluded that while wet forming is a nonwovens technology ideally suited to handle these fibers and to produce the range of web weights required in both mono- and multilayer structures, systems of appropriate size and capabilities are essential if product quality and process economic conditions are to be met.□

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