



NONWOVENS RETROSPECTIVE

The History of the Development of Melt Blowing Technology

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Abstract

Almost a half century ago development efforts were initiated by very different entities, in widely different locations, to demonstrate one step processes to convert polymer to web:

- Major fiber producers (DuPont, Freudenberg, Monsanto) began work on converting polymer (PE, PET, Nylon) into continuous "cold drawn" filaments and integrating the conversion of these filaments into a random-laid bonded nonwoven fabric.
- An oil company (Exxon), building on the earlier work (1950's) of the Naval Research Labs to produce fine fibers, began work on converting their recently commercialized PP polymer into discontinuous, or continuous, "hot drawn" filaments and integrating these filaments into a random-laid self bonded nonwoven web having average fiber sizes 2-5 microns (fine fibered webs) to 100+ fibers (coarse fibered webs).

As a result of these early development efforts, three different, but related melt spinning nonwoven processes have achieved significant commercial importance, with tremendous benefits to consumers worldwide:

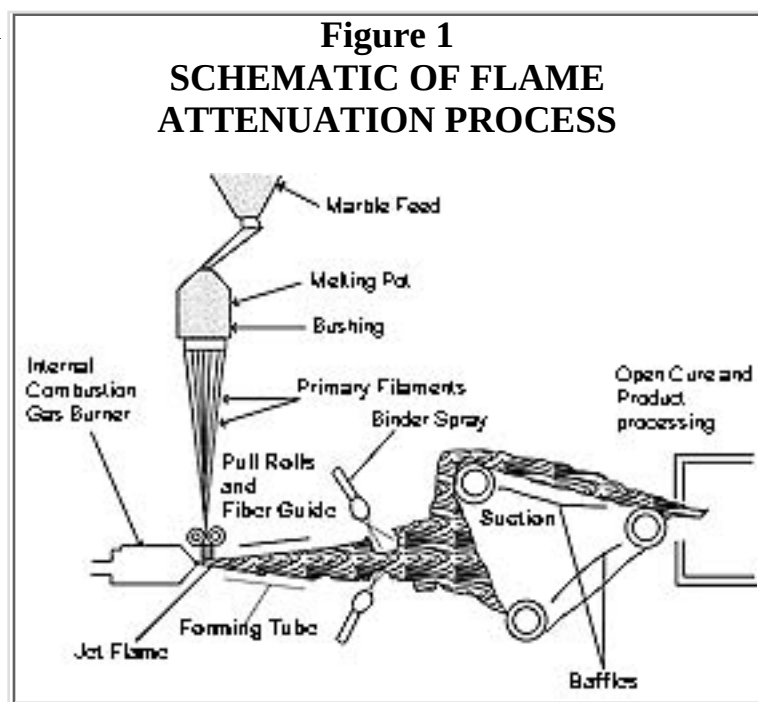
- Spunbond process
- Melt blowing process
- Flash spinning process

This presentation will summarize the development of the melt blowing process from conceptualization to current state-of-the-art. Important product, process and equipment developments are detailed in the 50 year growth of the melt blowing process from a laboratory concept to a 125 million pound a year U.S. and Canadian commercial business and substantial additional worldwide consumption. Today, spunbond and melt blown processes are used for approximately 54% of the total 18.6 million square yards U.S. nonwoven market.

Conceptualization

The concept of melt blowing of thermoplastics to form microfibers (<10 microns) was first demonstrated

back in 1954 by Van A. Went, of the Naval Research Laboratories, who were interested in developing such fibers to collect radioactive particles in the upper atmosphere to monitor worldwide testing of nuclear weapons. Went's process "using an adjustable (ram) extruder forced a molten polymer through a row of fine orifices directly into two high velocity streams of heated air and formed fibers within the gas stream when cooler, ambient air solidified with fibers" was able to make very fine fibers (0.3 micron) by operating at very high temperatures (850°F) [1, 2, 3]. Conceptually it was quite similar to the manufacture of mineral or slag wool, which was practiced as early as 1840. In the mid 1940's Owens-Corning Fiberglass developed the flame attenuation process for making microdenier glass fibers in which the drawn 1 mm diameter fibers were aligned and then attenuated by the jet flame blast from an external combustion burner (*Figure 1*). Glass fibers as fine as 0.5 micron could be made by this process [4].

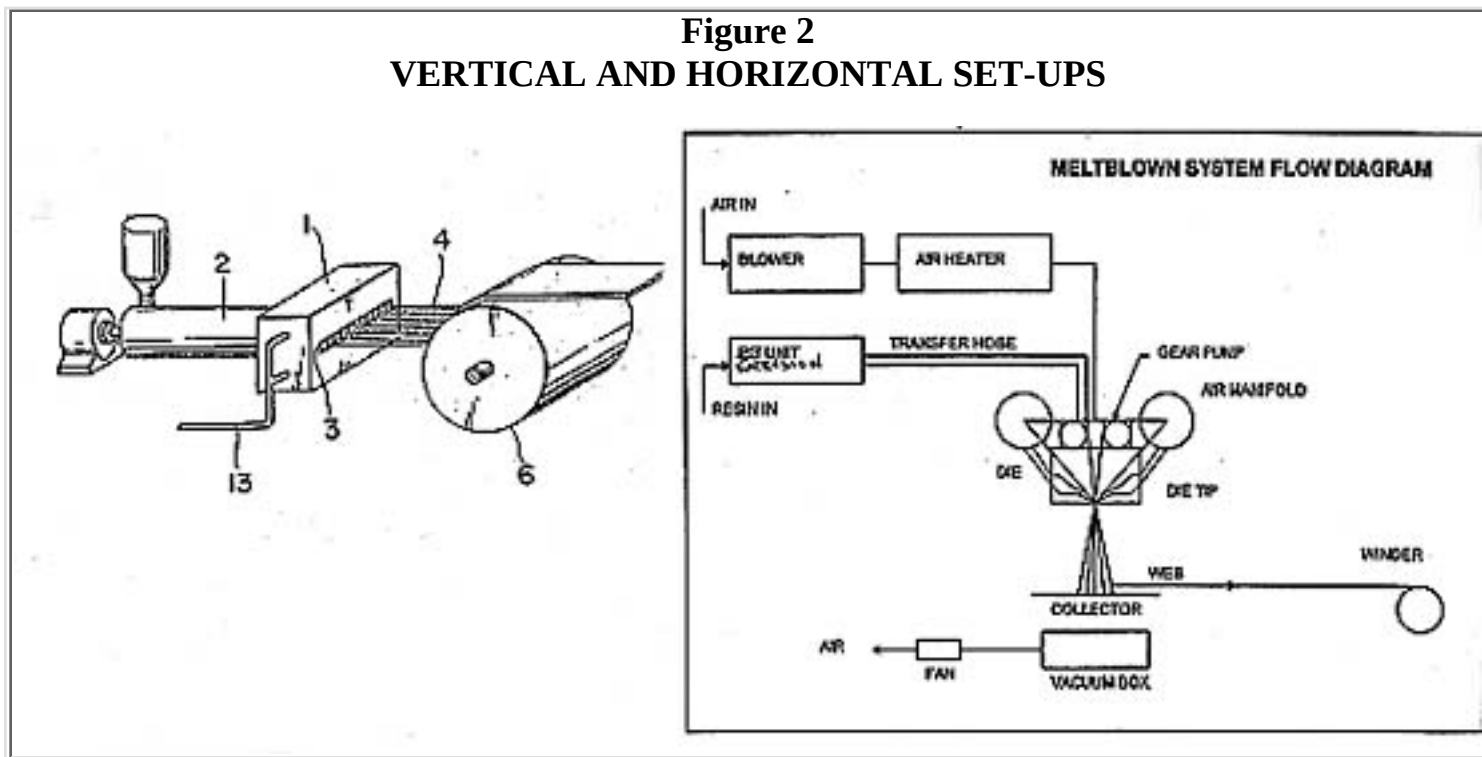


In the late 1930's and early 1940's, The American Viscose Co. investigated spray spinning of acrylonitrile and co-polymers, to make microfibers; Dow Chemical made laboratory quantities of microfiber polystyrene, while The Chemstrand Company tried spray spinning from solvent solutions of acrylonitrile polymers, but never commercialized the process [4].

Initial Commercial Demonstration

In the late 1960's and early 1970's, Exxon Research, in looking for uses for their newly commercialized polyolefin "Polypropylene" (PP) were trying to use a polypropylene reactor slurry to produce synthetic paper. Dr. Robert Buntin became aware of the Naval Research Laboratories publications and these served as a starting point for a multi year, multi million dollar project. The project culminated with a semi-works (3-40 inch dies in series) line, in several publications, with many patents and patent applications and a decision to license the technology, rather than commercialize it. In addition to providing the technology, Exxon also provided the industry a new process name, "Melt Blowing Process." Exxon's affiliate in Japan retained the right to commercialize the technology and did so quite successfully by producing and marketing Tapyrus melt blown products and became one of the earliest commercial melt blowing process practitioners. Thus, Exxon became the first to demonstrate, patent, publicize and license the use of the Went concept as a very practical one-step process to produce unique types of nonwoven webs, tow, bats, etc. [5,6,7,8]. Early successful licensees included Kimberly-Clark, Johnson & Johnson, James River, Web Dynamics and Ergon Nonwovens, followed by many other companies, including 3M.

Figure 2
VERTICAL AND HORIZONTAL SET-UPS



Although the process is conceptually quite simplistic, (Figure 2) successful production of acceptable quality webs could only be produced with precisely engineered equipment operated under a rather narrow operating window for each different product. To assist licensees, Exxon entered into agreements with two equipment manufacturers, Accurate Products and Reifenhauer, to supply equipment to licensees, after these companies demonstrated their capability to build and market such precisely engineered equipment.

So, What Is Melt Blowing?

The most commonly accepted and current definition for the melt blowing process is: "A one-step process in which high-velocity fluid, normally air blows molten thermoplastic resin from an extruder die tip onto a conveyor, or take-up screen, or substrate to form a fine fibred self-bonded web" [9]. (See Attachment A for schematic drawings) This very broad definition covers the extremely versatile melt blowing process which has the following characteristics:

- the thermoplastic resin can be any resin, including adhesives which are fibrous forming, having relatively low viscosity as it exits the die
- any blowing fluid can be used, including steam, air or other fluid
- the high-velocity air can be supersonic down to much less than sonic (subsonic)
- the fine fibers can be < than 1 micron to 100 + micron
- the die tip can be single orifice surrounded by air with multiple rows of orifices (Biax FiberFilm [10]), or single row, multi orifices with airplates on either side (Accurate, J&M) or capillaries (Lohkamp, Exxon, Mende, MPC)
- the die can be horizontal or circular (Exxon Roving Die)
- the thermoplastic resin(s) can be in solid form or in solution or slurry form
- the fibers can be continuous or discontinuous

The above definition, although quite broad, is still somewhat limited in that the melt blowing process has been used to produce precursor carbon fibers in an unbonded manner, hence the definition should include

unbonded fibers as well as self-bonded webs. Dr. Lloyd Trimble pointed out in "Melt Blown Technology Today" [11] the conventional definition of melt blowing, if expanded slightly to add the words "or another appropriate force to attenuate the filaments" the melt blowing process would include, in addition to the standard melt blowing process:

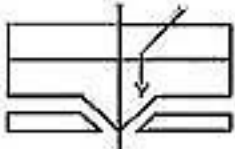
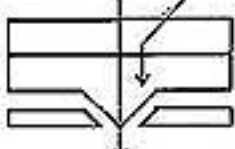
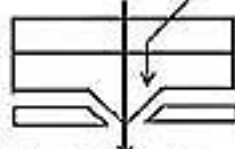
- the flash spinning process for production of Tyvek polyethylene plexifilamentary microfibrinous nonwovens practiced by DuPont [12]
- the use of electrostatic forces rather than high velocity air to attenuate the fibers as practiced by Freudenberg and by Dexter [13]
- the use of centrifugal force from a circular spinning die to form fibers similar to the process used to produce glass microfibers [4]

With this expanded definition of the melt blowing process, the potential for use of the process is enormous. There are three distinct regimes for the melt blowing process [14, 15] (Figure 3):

- very high air flow regime - most common - 2 to 5 micron average fiber size - current commercial regime
- ultra high flow regime to produce ultrafine fibers < 1 micron average fiber size - under development
- lower air flow regime to produce 1 denier + average fiber size - (adhesive range) Early Product Development Activities

The extremely fine fibers of the conventional melt blowing process result in a soft, self-bonded fabric having excellent covering power and opacity. Because of the fineness and tremendous number of fibers, melt blown webs can develop significant bonding strength through fiber entanglement. Additional fiber-to-fiber bonding can also be achieved by shortening the DCD so that the fibers are not completely solidified when they impinge the collector screen. These entangled fine fibers yield a web having a range of small pores.

Figure 3
THE THREE DIFFERENT MELT BLOWING REGIMES
(ONE METER WIDE DIES)

Regime	Fiber Size (microns)	Air Gap (mils)	Air Flow (cu m/h/m)	Throughput (g/m/h)	Possible Other Features
High Air (Conventional)  Holes: 1200 x 0.4 mm D's.	1.0-10.0	1.5	1500-1600	0.60-2.0+	Air/Water Quench
Ultra High (Development) (U.F.F.)  Holes: 1500 x 0.25 mm D's. 2000 x 0.10 mm D's.	<1.0	0-13	300 to 2000	0.01-0.10	Level unit USP 5,645,057
Lower Air (Adhesive)  Holes: 4000 x 0.50 mm D's.	10.0-200	0.05-0.15	12 to 30	0.05-10.00	Separate small dies, with internal valves

UFF=Ultra Fine Fiber

Because of these properties, one of the initial application thrusts was battery separators. Exxon developed a 3-ply calendered PP laminate (USP 4,078,124 et al.) for this end use. Roy Volkman, Riegel Products, commercialized a PP melt blown separator for lead-acid, maintenance-free batteries in the 1970's; however, the problems of re-wettability and fabric abrasion resistance slowed its acceptance. Toa Nenryo has been successful (USP 4,743,494 and 5,089,360) in overcoming some of these problems for alkaline batteries. Entek (USP 5,230,949) produced webs from highly loaded ultra-high M.W.P.E. and with subsequent extraction formed a microporous web, Asahi recently announced the use of their nylon microfiber web to try to tap the vast Ni-Cd battery market. The need for longer life batteries for electric vehicles may result in a significant market for melt blown webs as battery separators.

The many small pores and hydrophobic nature of PP webs made it a natural candidate for Oil Sorbents and Wipes. Hydrophilic additives and/or topical treatments provided hydrophilicity, which enabled companies such as Ergon Nonwovens, Sorbent Products and Kimberly-Clark to produce and market melt blown webs starting in the early 1970's. This market is continuing to grow at a 7-8% rate.

The replacement of glass fibers with melt blown fibers in facemasks and respirators was initiated by such companies as Johnson & Johnson, again in the early 1970's, and the replacement is now essentially complete. Rowland Griffin summarized some of the more important innovations at Filtration '97 [16]. Fiberweb has just been granted a patent on producing a very fine (0.8 to 1.3 micron) web for this end-use (USP 5,645,057).

One of the earliest filtration markets targeted for melt blown webs was cigarette filters. Cellulose acetate filters had been introduced in the 1950's (Kent cigarettes) and it was hoped that PP filters might supplement the cellulose acetate filters, just as OPP film had largely replaced cellophane due to its lower cost and equivalent performance. Exxon obtained U.S. Patents 3,888,610 and 3,595,245 on the direct conversion of melt blown PP into cigarette tow, utilizing a circular die; however, quality control and cost were problems. Dr. R. Berger, Filtrona, patented a sheath core, EVA/PP bi-component web for such use. (USP 5,509,430) Additional work has recently been carried out on the direct conversion of a low basis weight, >10 micron melt blown web as the feed to a conventional cigarette rod maker. Despite these early activities commercial success of melt blown webs for cigarette filters has not been achieved to date, but work is continuing [17].

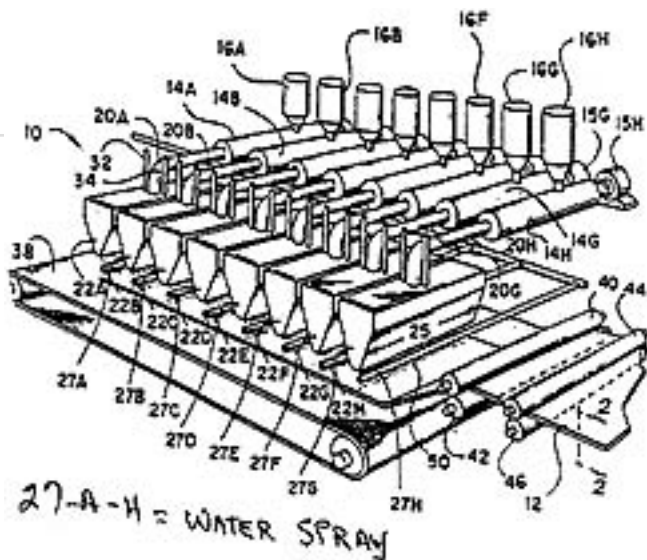


Figure 4
GRADED FILTER DIAGRAM

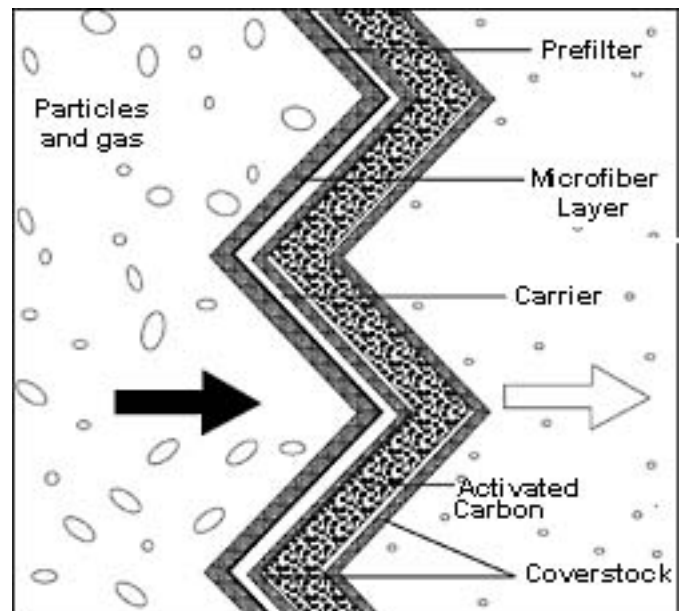


Figure 5
SCHEMATIC OF MICROFIBER WEBS FOR CABIN FILTERS

Much success has been achieved in other more conventional air and liquid filter applications of melt blown webs. Shipp et al., Kimberly-Clark obtained USP 4,714,647 on a graded filter (*Figure 4*). Pall recently obtained patent 5,582,907 on a melt blown filter, presumably for blood filtration. JML obtained USP 5,409,642 for the direct conversion of polymer into a finished tubular liquid filter. Akzona obtained two patents 4,666,763 and 4,247,498 using the Exxon melt blowing process to extrude solutions and form membranes for separation of leukocytes from blood.

Other innovative filter developments are included in the previously referenced publication of Rowland Giffin.

Other Important Product Milestones

Kimberly-Clark, the most prominent practitioner of the melt blowing process, has made many important innovations. The Brock et al. SMS patents opened up large outlets in medical fabric and surgical wrap applications. Their CoForm patented technology has been widely used in wipes. 3M, with a somewhat similar concept, commercialized Thinsulate thermal insulation, which appears to be also useful as cable insulation, as well as for outdoor sportswear and more recently for divers' underwater clothing. Biax FiberFilm uses recycled polyester bottles to melt blow building insulation. Tandec has patented an excellent electrostatic charging process (USP 5,401,446) to provide improved filtration performance at low pressure drop, while others have utilized the "triboelectric effect" [18] using two polymers of different electronegativities to achieve similar results.

Demonstration of the unique properties of melt blown E-CTFE Halar has opened up new applications in the high temperature filtration field (19). Kuss Filtration has recently introduced their Stratopore PDM fuel filters using three layers of differing fiber size, melt blown nylon 6 webs. Fiberweb, N.A. published on November 7, 1995 a statutory invention disclosure on the melt blowing of liquid crystals for use in high-temperature filtration. Freudenberg published the use of microfiber webs for cabin filters [19]. (*Figure 5*)

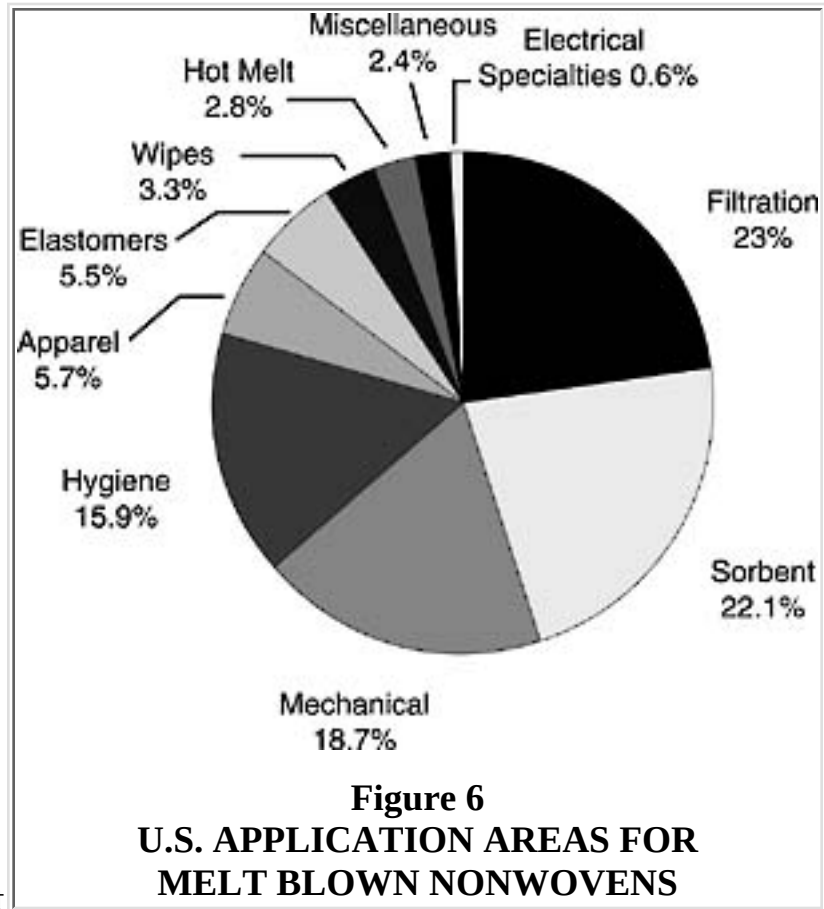
Kimberly-Clark was one of the first companies to melt blow elastomeric materials [20]. Toa Nenryo and FITESA/Exhibit 1010, Page 6 of 11

Kuraray have melt blown elastomeric webs from thermoplastic polyurethanes for such applications as hospital gloves. Pitch is now being melt blown in China to form the precursor fibers for median modulus, low cost carbon fibers. Product development work is underway in several laboratories to use melt blowing of certain polymers as the starting point for artificial leather as a follow-up to the 1980's development by Toa Nenryo.

A 1995 estimate of the U.S. application areas for Melt Blown Nonwovens is shown in *Figure 6* [21]: An increasing use is to improve the barrier and appearance by "painting" nonwoven fabric with a fine (<2.0 gsm) coating of melt blown product to substantially enhance the value of the base fabric.

Important Melt Blowing Equipment Developments

New nonwoven product developments require precisely engineered, user-friendly equipment for successful commercial production of these new products. This is particularly true in melt blowing, where the design and fabrication of the key components (die, nosepiece, air assembly, collector) are all critical. Accurate Products was the first to successfully build a 40 inch melt blowing die meeting the critical Exxon specifications (USP 4,986,743). The following summarizes other innovative equipment developments, recognizing that several major melt

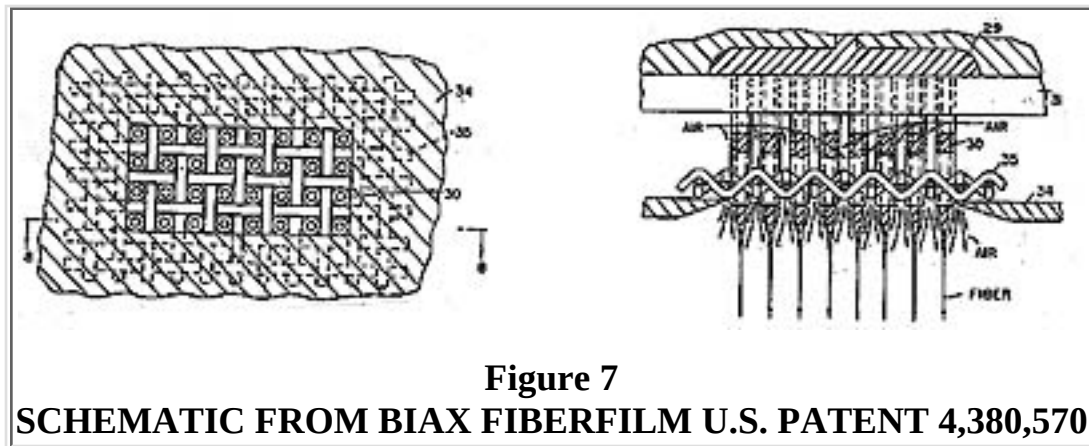


blowing producers have designed and built, or have built, their own proprietary designed equipment.

- JML, in the early 1990's, by dividing the melt blowing die into several 1 to 2 inch side-by-side units, and incorporating an internal valve in each unit along with a quick change die feature, successfully modified the conventional melt blowing die to melt blow adhesives onto substrates. This has opened up a large new outlet for "fat fibered" melt blowing fibers (USP 5,145,689; 5,692,884 and others).
- Reifenhauer has designed and built improved designs for melt blowing dies (USP 5,248,247) and for stretching and heat setting of coarse fibered melt blown webs (USP 5,609,808), which claims very high productivity. They also design and sell SMS lines.
- 3M obtained USP 5,232,770 on equipment to produce high-temperature stable nonwoven webs based on multi-layer blown microfibers. Eugene Joseph, the inventor, has also published articles on this development [22].
- Biax FiberFilm has designed and sold melt blowing equipment (*Figure 7*) that uses multiple rows of orifices to provide higher productivity (USP 5,476,616; 4,380,570) [10].
- Kimberly-Clark has patented a slot die for melt blowing (USP 4,720,252) to minimize orifice plugging.
- Chisso Corporation has patented (USP 5,290,626 and 5,511,960) for equipment to produce

conjugate melt blown webs, and for producing islands-in-the-sea type webs.

- Kimberly-Clark obtained USP 5,711,970 for an apparatus for melt blowing, spunbonding and CoForm processes that produces an in-line perturbation of the flow of the fluid (air) to produce crimped or uncrimped fibers at reduced energy costs. This appears to build on their earlier Kimberly-Clark Tribbes patent 5,695,377, which uses projections on the inside of the air plates to produce the perturbations. They state their approach is more practical than Shambaugh's patented (5,523,033) somewhat similar approach.



- Tandec has obtained patents on their in-line or off-line process to provide CD stretch to melt blown, non-elastic webs [23].
- Mende, of Mitsui Petrochemical Industries has obtained patents 5,017,112 and 5,171,512 on the use of capillary melt blowing dies.
- JML obtained USP 5,618,566 for construction of a modular melt blowing die with side-by-side interchangeable modules on a manifold so that the die length can be readily varied by adding or subtracting modules. (DuraFiber^a System [15])
- FiberWeb N.A. as patented a process and products therefrom (USP 5,783,503) to produce sub-denier and micro-denier fibers of surprising strength, barrier, and cover utilizing multi-component dies to produce continuous, easily splittable hollow fibers of low orientation (*Figure 8*). This could be an alternate process to produce microfibers.

Conclusions

Since the nearly 50 years after Wentz first produced microfibers using a small ram extruder; and since the more than 30 years after Buntin first demonstrated a microfiber nonwoven web using a small 4 inch die, the melt blowing process has become a worldwide process of choice to produce microfiber polymeric webs and "fat-fiber" adhesive webs. This global acceptance has been largely due to the innovativeness and entrepreneurial approach of individuals in both large and small companies, particularly equipment manufacturing companies. A review of the first 50 (almost) years of key events in

the melt blowing process evolution (along with spunbonding history) is shown in Figure 9. Today, the melt blowing population - including adhesives lines - is well over 300 lines, including 86 lines in China alone [26].

In summary, the melt blowing process is one of the most versatile and cost effective processes commercially available to produce microfibers, or larger when needed, and these fibers continue to be increasingly important to meet today's and tomorrow's health and environmental needs. Far from being "Beyond melt blowing" as suggested in a recent EDANA presentation [24] the melt blowing process, either alone or in combination with other processes, will continue to become even more widely used to "add value" to existing products and to enable consumers to benefit from the introduction of completely new products [25]. In 1998, consumption of monolithic and composite in the U.S. and Canada was estimated by John Starr to be 125 million pounds, about equally divided between monolithic and composite melt blown products [25].

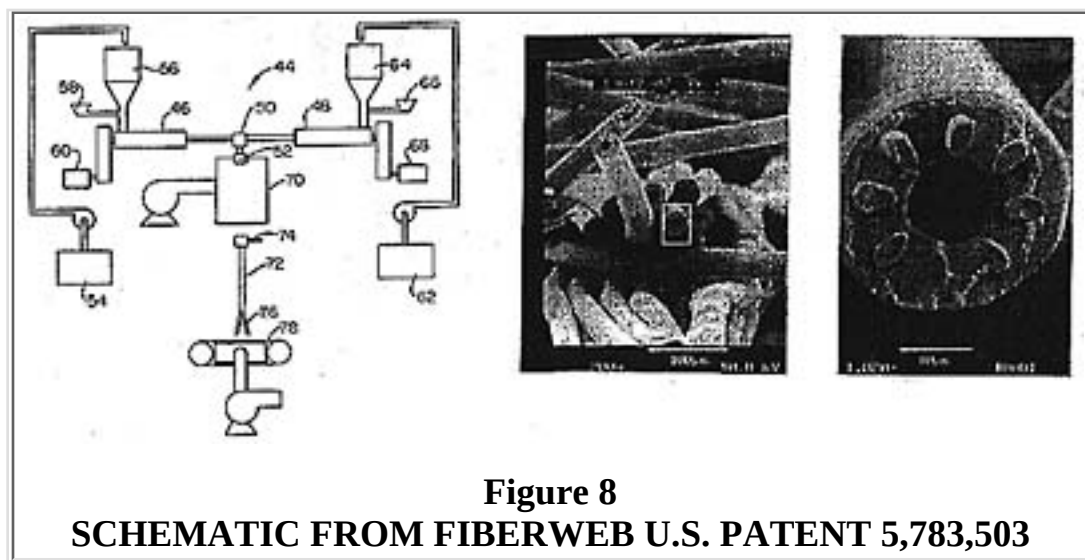


Figure 8
SCHEMATIC FROM FIBERWEB U.S. PATENT 5,783,503

Figure 9
TIME LINES FOR MELT BLOWING AND SPUNBOND DEVELOPMENT

Spunbonding - The First 50 Years (1950's-1990's)

1950's: Internal developments by major fiber producers
1967: DuPont's first SB patent - electrostatic charges to control web formation
1968: Freudenberg's first U.S. patent - (1962 filing date)
1968: Lurgi (DOCAN) process licensable (no web bonding)
1969: KC, CZ, other paper producers took out license - developed own versions
1970: Monsanto's patent on SB nylon (Cerex)
1974: Asahi obtained first US SB patent on their process
1976: KC obtained SMS Patent 4,041,203
1977: Appel & Mormon (KC) obtained Patent 4,043,739 plus

1996: Ason obtained two patents on their subdenier SB process
1997: Nippon Kodoshi, Kobelco offering SB equipment
1998: Sima, Italy offering SB equipment
1998: Hills/Ason first commercial SB lines
1998: Biax offering SB equipment
1998: Fiberweb multicomponent hollow fiber SB patent
1998: J&M combines both Melt Blowing and Spin Bonding with introduction of their MultiFil™ Composite Filament System

Melt Blowing - The First 50 Years (1951 - 1998+)

1951: Naval Research Laboratory (Concept)
1965: First 10 inch Melt Blowing line (Exxon)

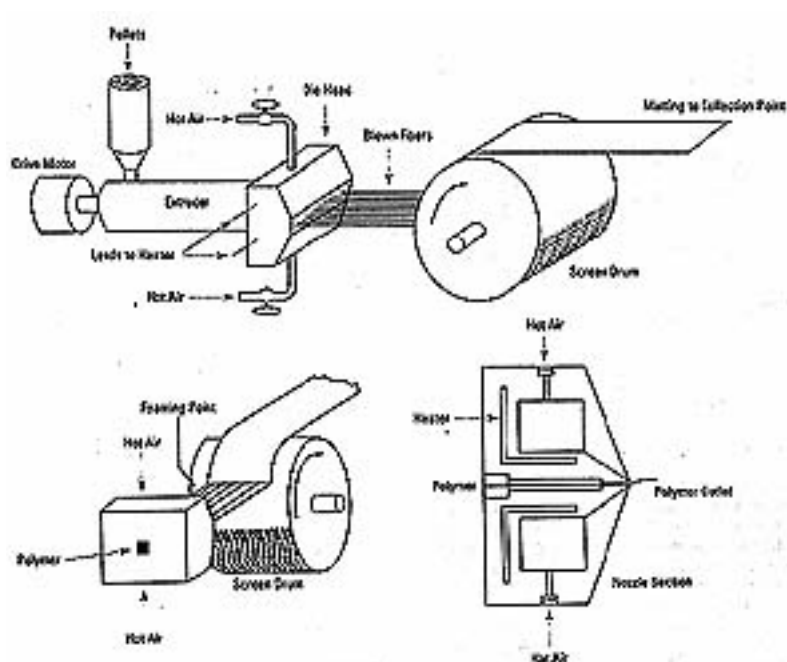
1971: First Commercial Line - U.S.
1972: First USA equipment manufacturing (Accurate Products)
1973-5: First Seven Practitioners - U.S.
1974: First Japanese Practitioner (Tonen)
1983: First Pilot Line at Tandec (University of Tennessee)
1986: First European Practitioner
1989: First Taiwanese Practitioner
1989: First adhesive melt blown line (J&M)
1989: First European equipment manufacturing (Reifenhauser)
1991: First Chinese Practitioner
1993: First Canadian Practitioner (Spill-Tech)

many others	1969: First demo on Pitch melt blown - C. Fibers	1994: First J&M polymer melt blown line
1984: Reifenhauer SB turnkey lines (REICOFIL) offered for sale	1970: First Large Scale Semi-Works Unit / 3-40 inch dies (Exxon)	1995: Tandec develops "Tantret" and "stretch" technology
1991: Amoco obtained patents on centrifugal spinning SB process		1997: Exxon transfer melt blown assets to Tandec
1991: Corovin obtained U.S. patent on their modification of SB process		1998: J&M combines both Melt Blowing and Spin Bonding with introduction of their MultiFil™ Composite Filament System

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Attachment A



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