

# Spring 2015 Glass Processing Course

## *Lecture 14. Fiberglass Processing*



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Lectures available at:  
**[www.lehigh.edu/imi](http://www.lehigh.edu/imi)**

Sponsored by US National Science Foundation (DMR-0844014)

# Outline



- Brief background
- Quick note on viscosity and liquidus temperature
- Continuous filament fiber
  - Process overview
  - Bushings design and operation
  - Importance of sizing
- Wool fiber (discontinuous)
  - Rotary process (internal centrifuge)
  - Cascade process (external centrifuge)
  - Flame attenuation process (pot & marble)
- More about viscosity and liquidus temperature
- A few other process variations



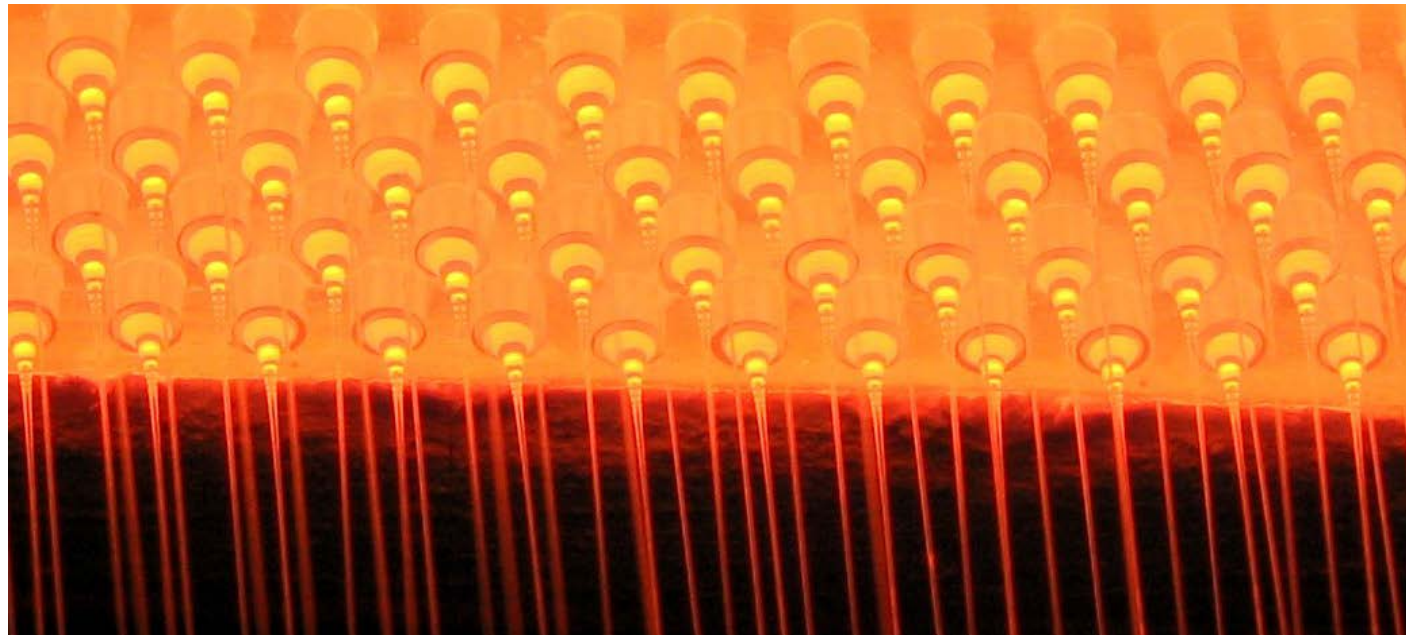
# Stretching Glass is Fun



- The first fibers might have been made by accident
- Glass fibers have been made for many centuries, originally for art and decoration



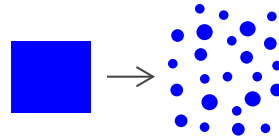
- Fiber started to be manufactured for practical uses in the late 1800's
- The process was improved and scaled up over decades
- Manufacturing improvements continue to help expand the global usage of glass fiber



# Fiber Forming

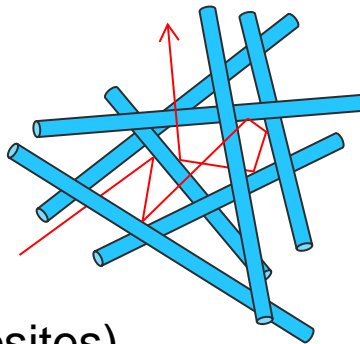
What is the fundamental purpose?

## Making surface area

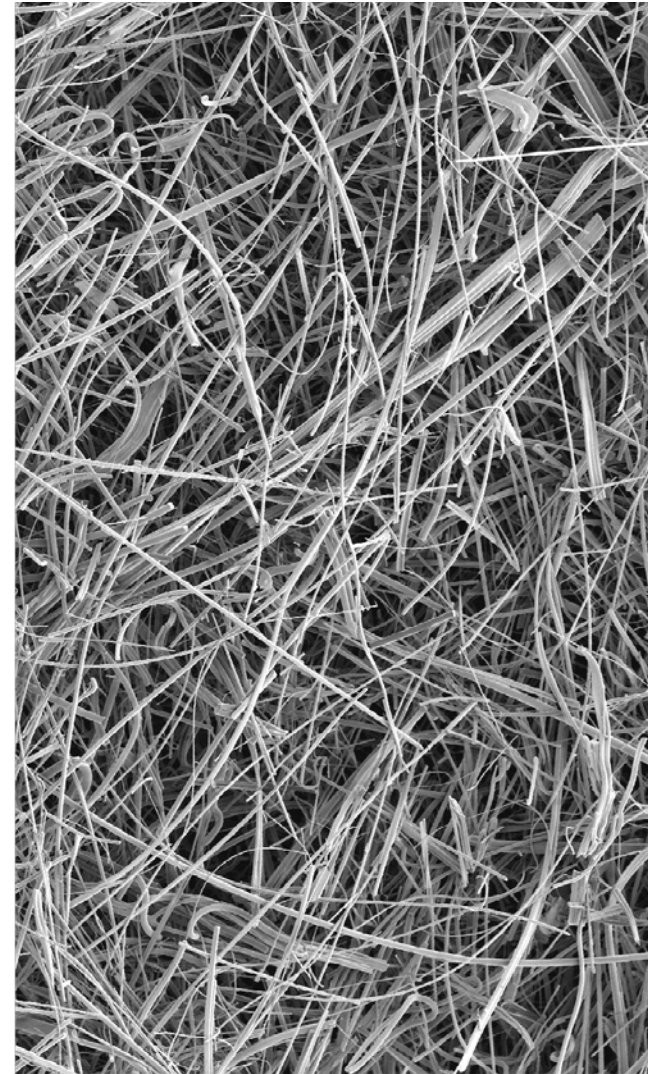


- 0.05 to 15 square meters per gram
- A large wool manufacturing line can produce 2000m<sup>2</sup>/s or 170km<sup>2</sup> per day
- Interface, coupling, filtration

## Making tortured paths



- Acoustic paths
- Filtration paths (air, liquid)
- Fracture propagation paths (composites)
- Thermal paths (conduction, convection, radiation)



# Continuous Filament vs. Wool



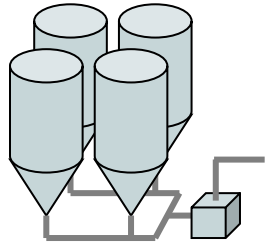
	<b>Continuous Filament</b>	<b>Wool (Discontinuous)</b>
<b>Processing</b>	Pulled in a continuous filament parallel with other strands (often chopped downstream)	Process inherently creates discontinuous sections of fiber that have somewhat random orientation
<b>Typical Fiber Diameters</b>	4-30 micron	0.2 to 10 micron
<b>Glass Types</b>	<u>E-glass</u> , C-glass, R-glass, S-glass, AR-glass, A-glass, D-glass, basalt, others	Soft alkali borosilicates, mineral wool, modified slag & basalt, RCF, others
<b>Uses</b>	Reinforcement (chopped fiber, rovings, wovens, non-wovens) filtration, separation, facers, thermal insulation, fireblocking	Thermal insulation (blanket, board, pipe, paper), acoustic insulation, filtration, separation



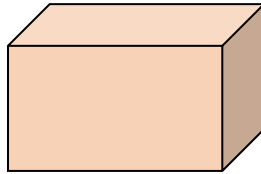
# Generalized Process Flow



Batching

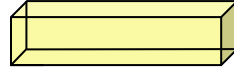


Melting



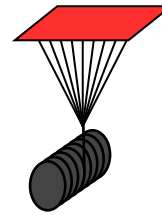
1 to 300  
tons/day, gas  
and/or electric

Conditioning



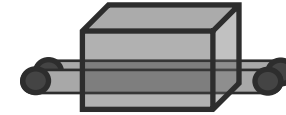
Forehearth, dwell  
tank, or just melt  
stream

Fiberization



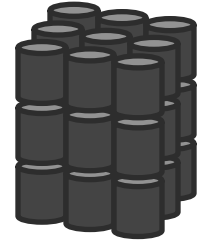
Bushing, rotary,  
cascade, flame  
attenuated

Cold (Warm)  
Processing

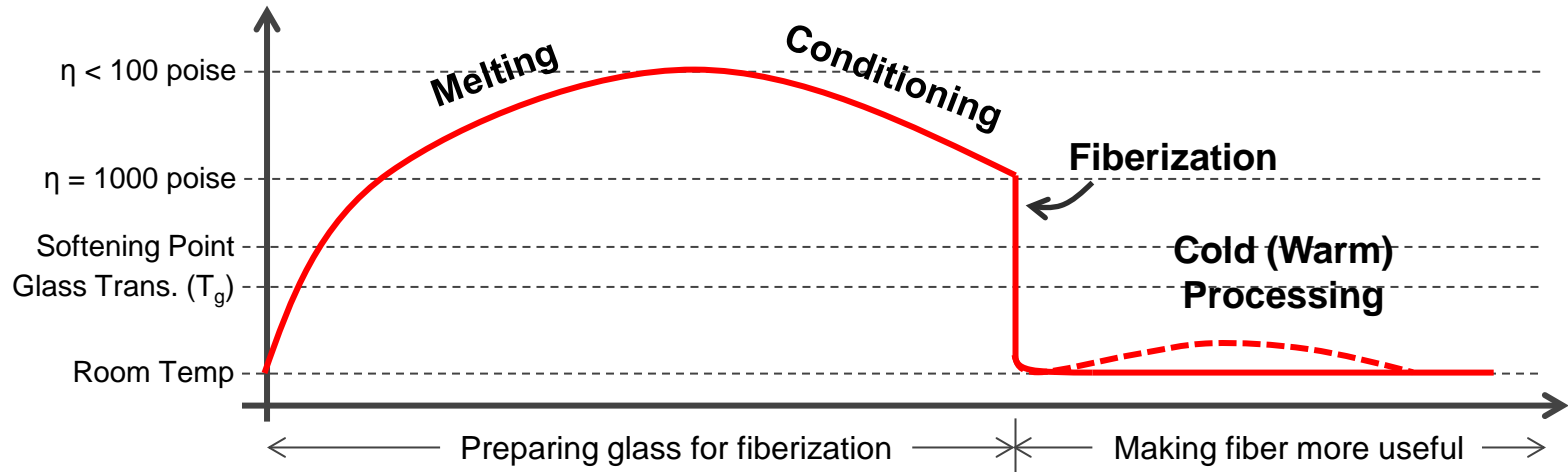


Winding, chopping,  
sizing, binding,  
twisting, assembling,  
pressing, screening,  
drying, curing

Packaging

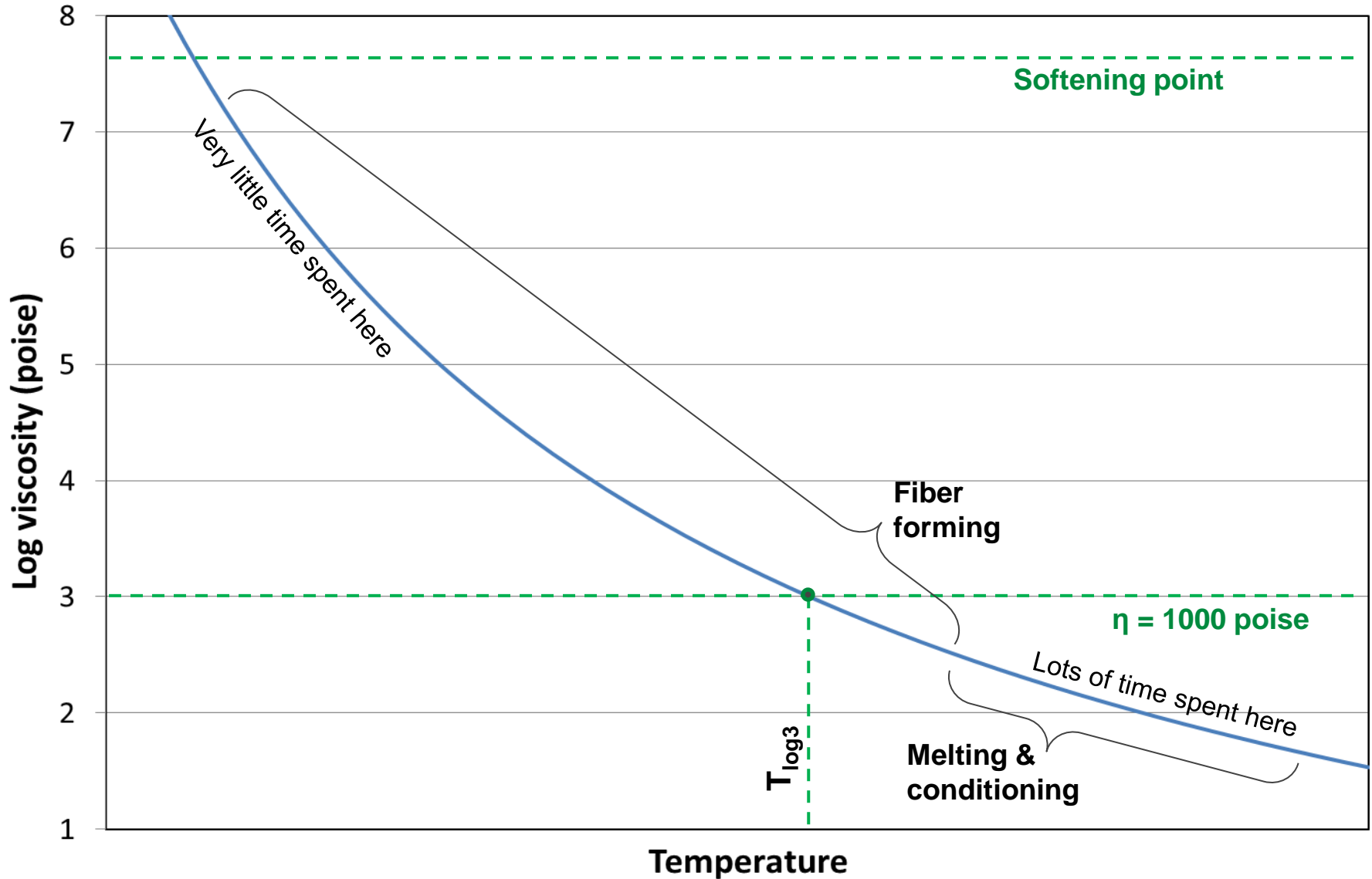


Temperature  
(Viscosity)



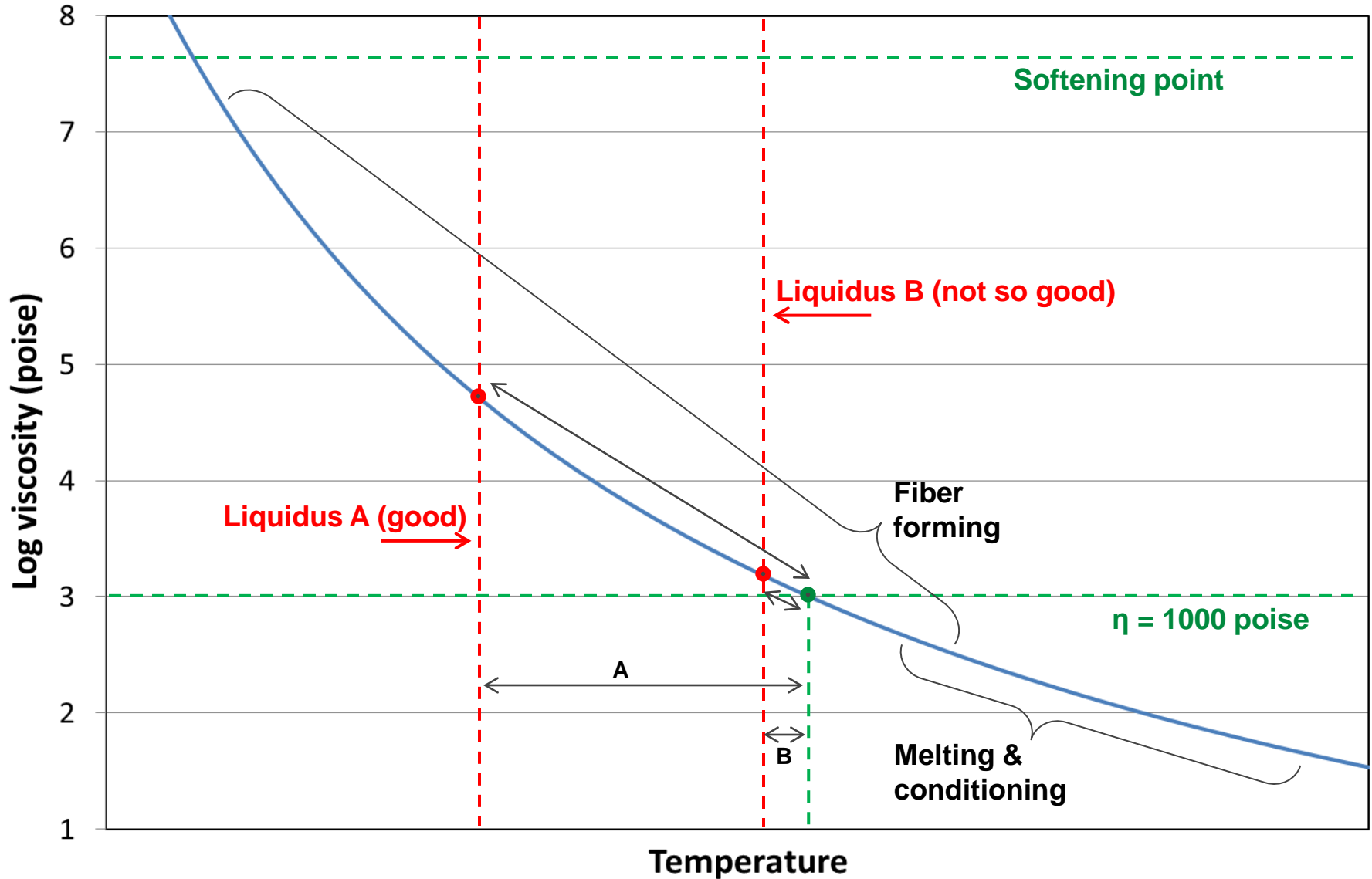
Time

# Viscosity



- Glass passes through the fiber forming viscosity range very fast, so in many cases the slope of the viscosity curve is not a key glass design parameter
- Liquidus temperature relative to viscosity can be a much more critical parameter.....

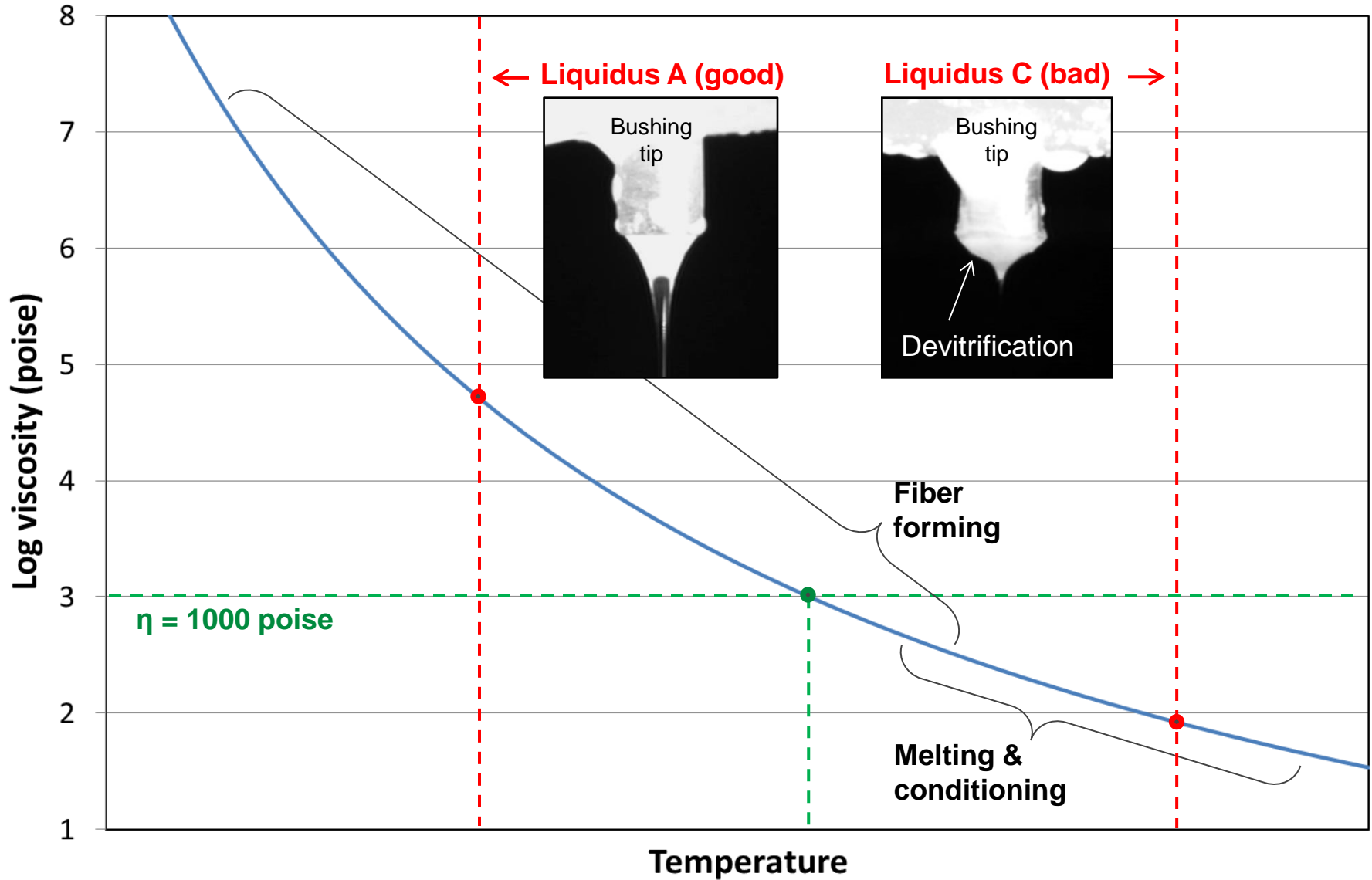
# Viscosity & Liquidus Temperature



- The liquidus temperature relative to a viscosity reference point such as  $T_{\log 3}$  is important for glass chemistry design
- As liquidus temperature increases, the risk of devitrification increases



# Viscosity & Liquidus Temperature



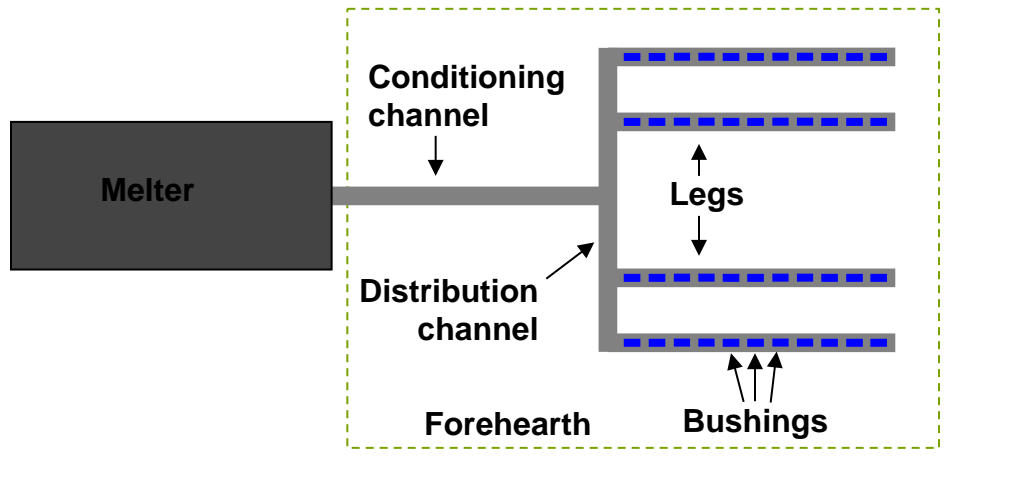
- Liquidus temperature can vary widely depending on composition
- Some processes are more tolerant of high liquidus temperature glass

# Continuous Filament Process

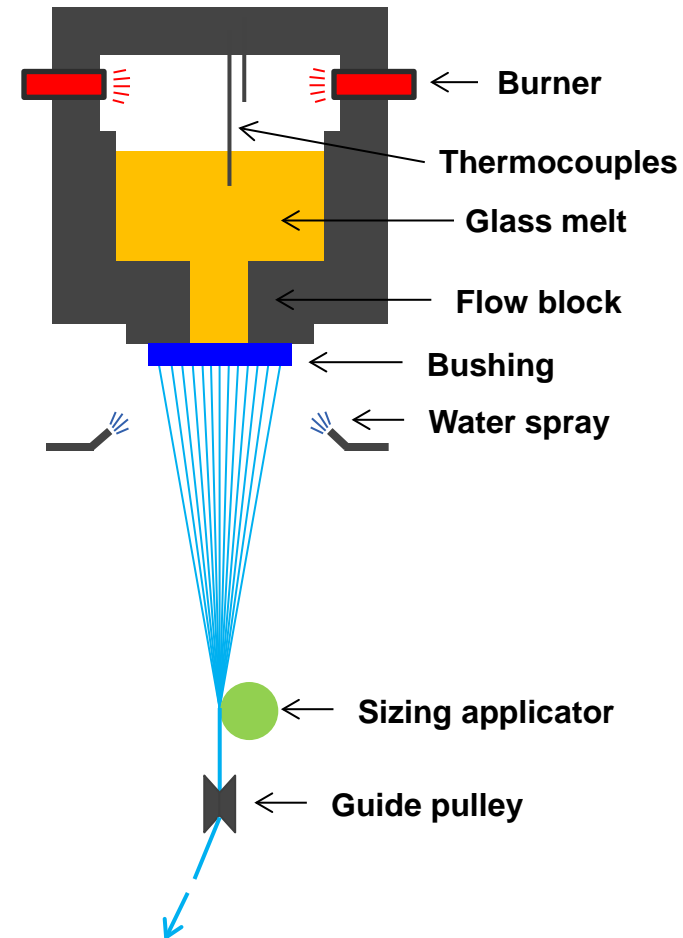


## Typical E-glass Process

Melter & Forehearth Layout (top view)



Leg/Bushing Cross Section (end view)

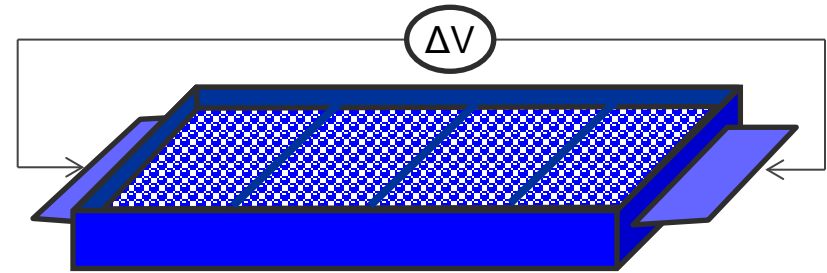


- Originally marble re-melt, now almost all direct melt
- Many different forehearth layouts
- Forehearth designed to deliver target glass temperature and head pressure above each bushing
- Enclosed and conditioned forming room(s) underneath legs
- Electrically heated precious metal bushings

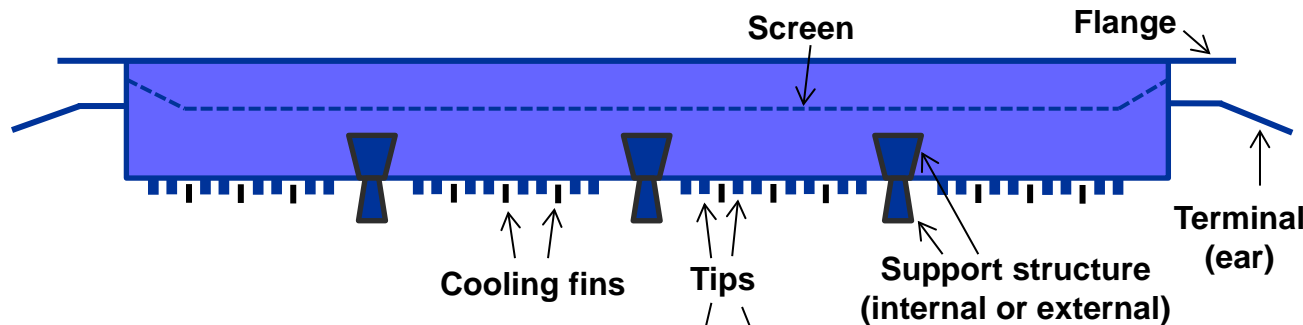
# Continuous Filament Bushings



- Platinum alloyed with 5-25% rhodium for high temperature strength
- Low voltage applied across bushing for resistive heating (high current)
- Usually mounted into a frame and surrounded on sides with refractory for insulation and support
- Screen improves temperature distribution and reduces effective head pressure

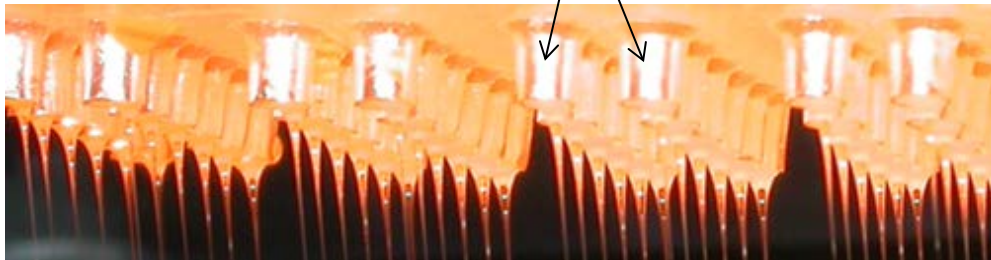


- 500 to 7000 tips per bushing
- Uniform temperature across the tip plate is critical for uniform flow per tip



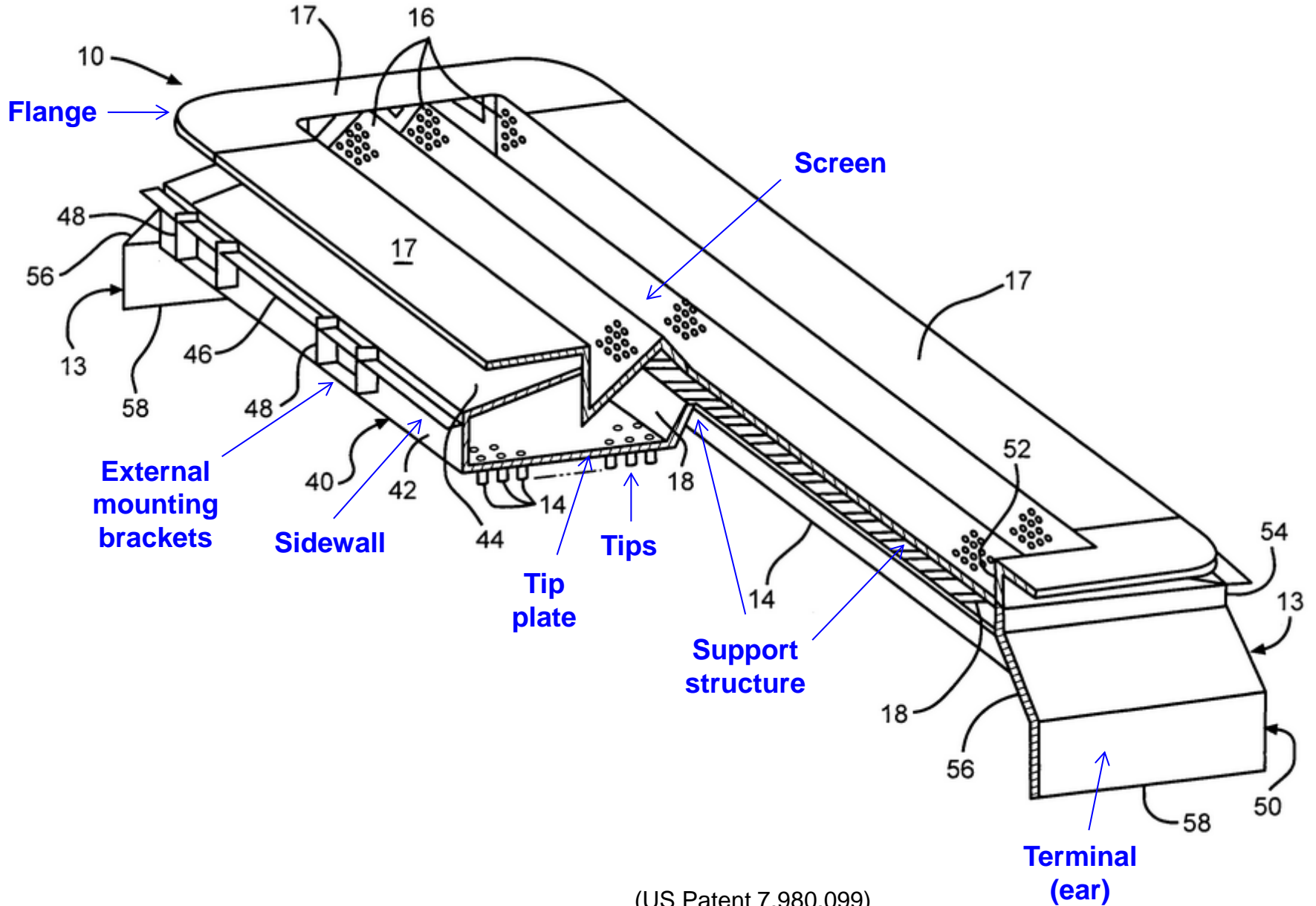
## Design Considerations

- Tip dia. and length
- Tip number and spacing
- Screen thickness and flow resistance
- Support structure
- Cooling fins
- Part thicknesses



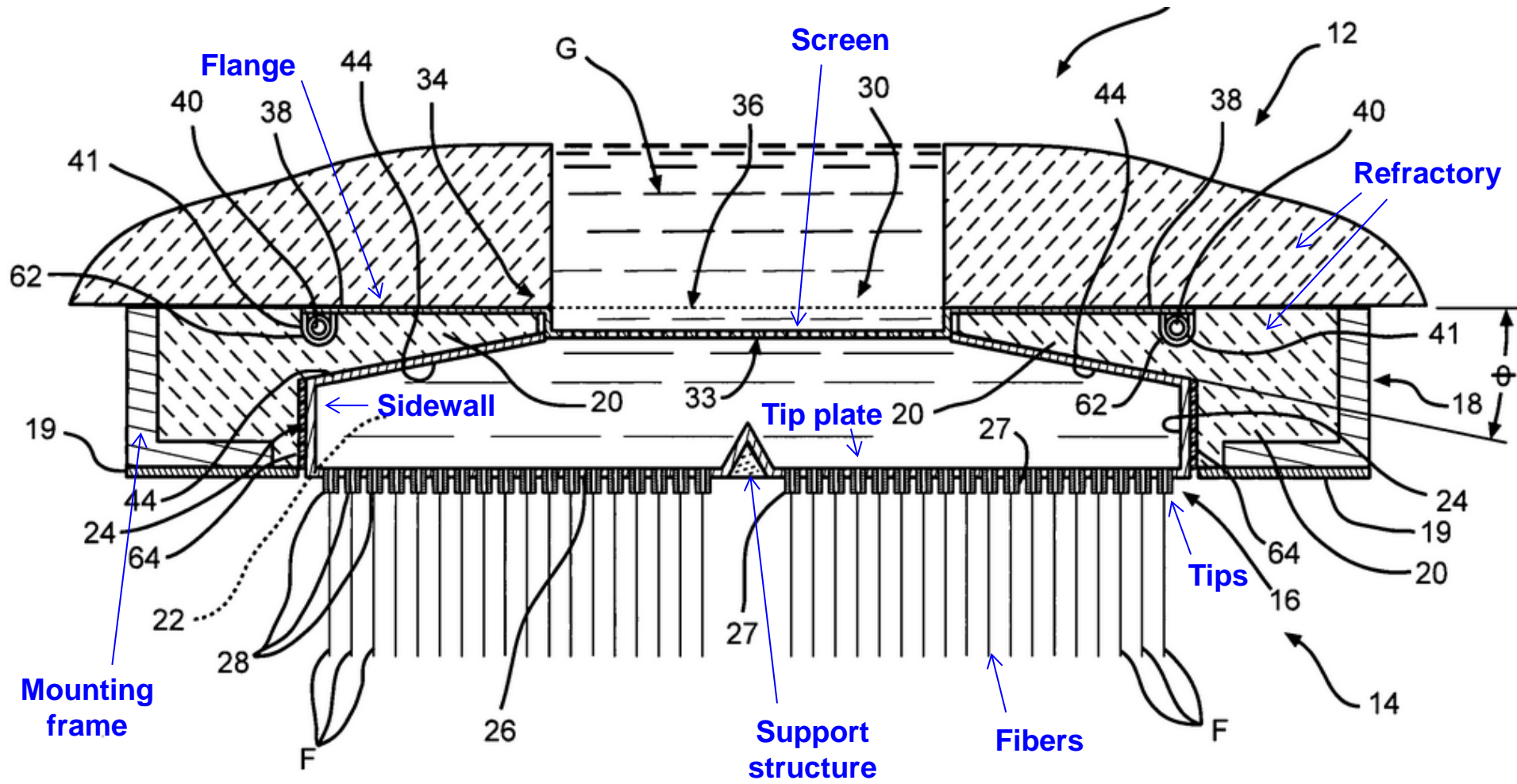
(cooling fins not shown for clarity)

# Bushing Design Examples

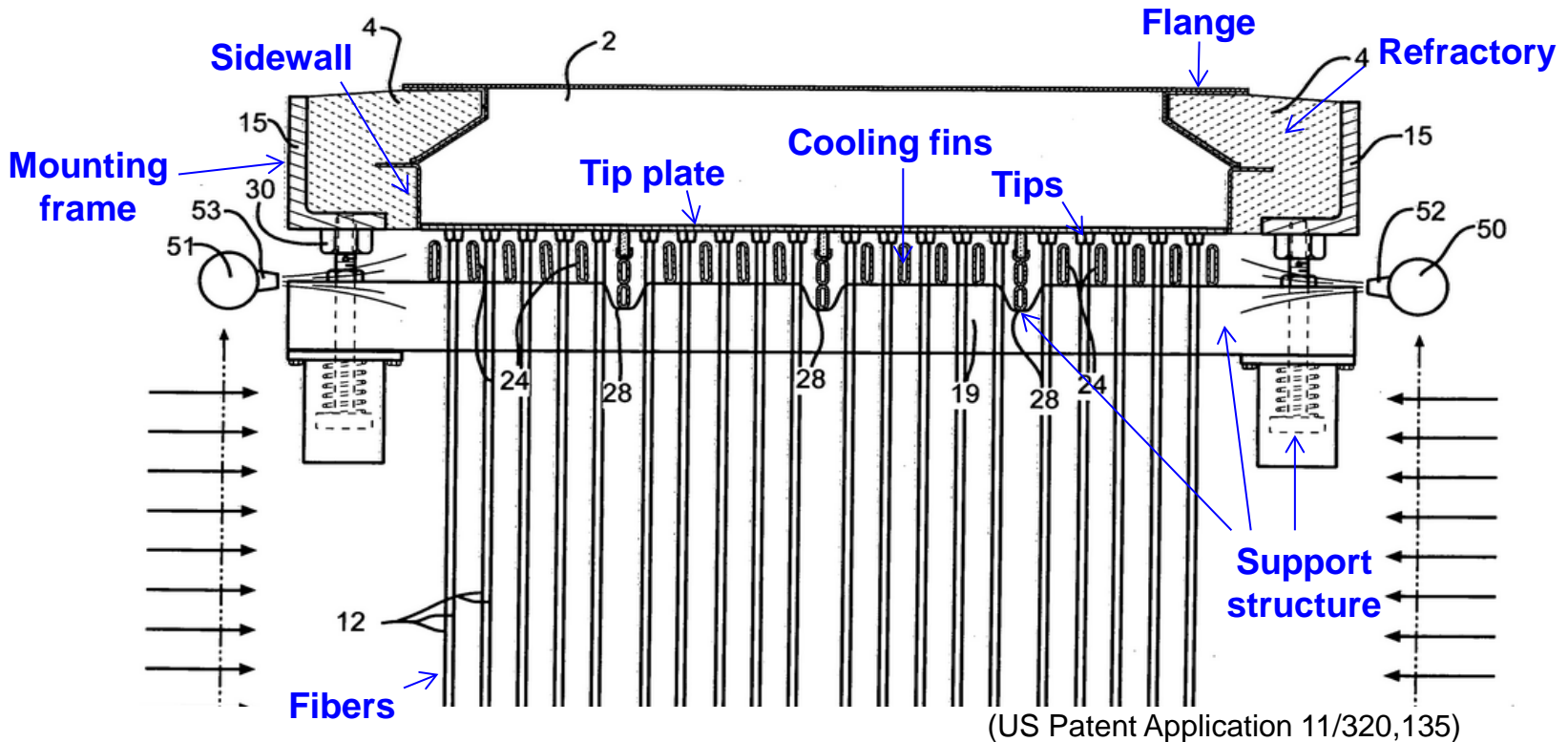
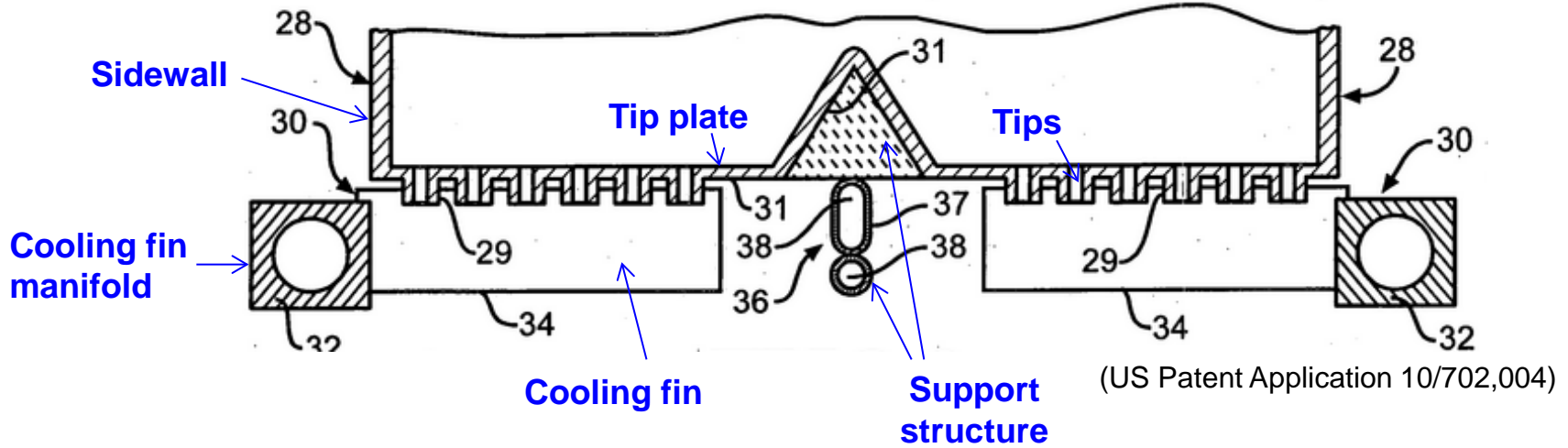


(US Patent 7,980,099)

# Bushing Design Examples



# Bushing Design Examples



# Bushing Operation



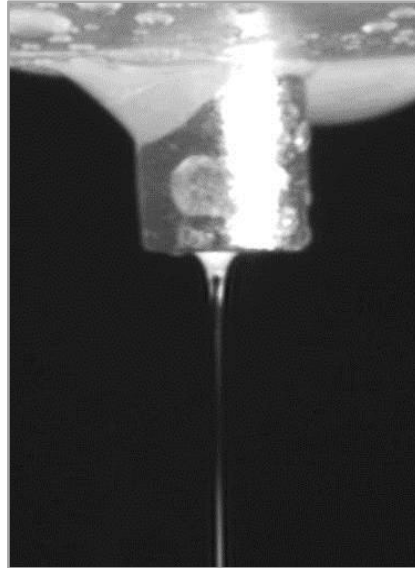
## Fiber Diameter

### Flow

- Effective head pressure
- Tip geometry (dia., length)
- Glass temperature (visc.)

### Pulling Speed

- Winder or chopper



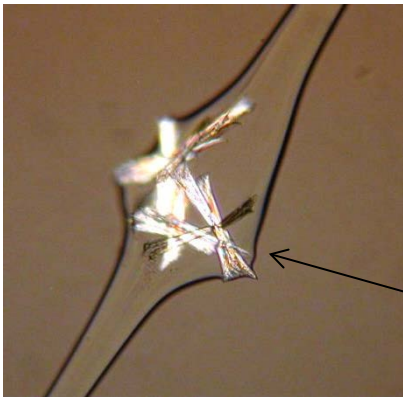
(estimated tip exit viscosity)  $\rightarrow \eta = 10,000$  poise

High viscosity results in high fiber forming stress

$\eta = 1,000$  poise

$\eta = 200$  poise

Low viscosity can lead to cone instability



Operation can be very sensitive to defects in the glass

### • **Stones**

- Refractory
- Batch/batch reactants
- Devitrification
- Contamination

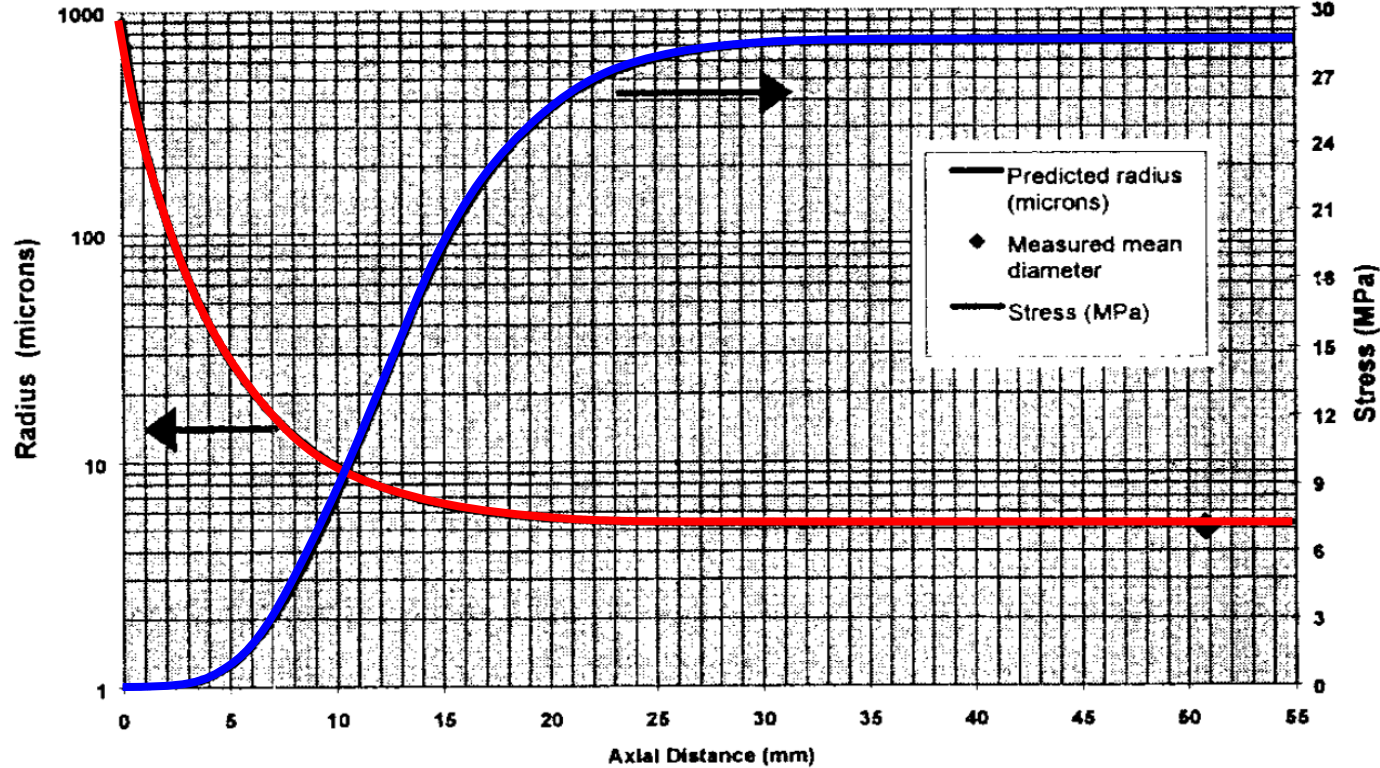
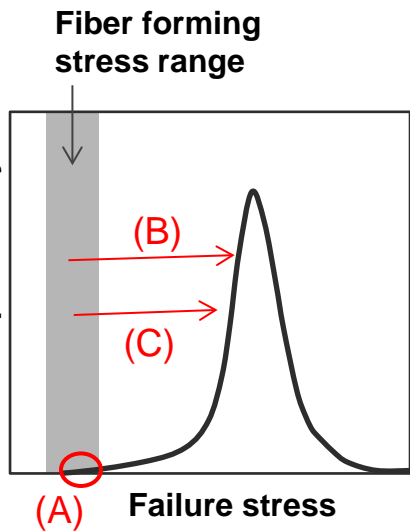
### • **Seeds**

- **Inhomogeneity (cord)**

# Fiber Forming Stress and Breaks



Modeling is used to predict stress profiles with varying conditions and designs



- A) Sample size (length of fiber) is so large that a weak spot is eventually found
- B) Defects cause an increase in fiber forming stress to the point of break
- And/or
- C) High stress from a cold tip

- A bushing is like an in-line high speed tensile strength test
- 100,000 to 500,000km of fiber per hour per bushing is tested
- Stress level from 10-200MPa at temperatures from RT to  $>T_g$ .

S. Rekhson et al, "Attenuation and Breakage in the Continuous Glass Fiber Drawing Process", *Ceramic Engineering & Science Proceedings*, vol. 25, No. 1, page 179.



# Tips and Contact Angle



- Tips are typically 1.0 to 2.5mm inside diameter
- Tips size needs to match process requirements
- Tip size strongly influences flow rate and fiber forming stress
- Some tips have restricted outlets to reduce stress but still have sufficiently high flow rate
- The wetting (contact angle) of the molten glass on the alloy can influence forming cone shape
- Wetting can also influence bead formation when a fiber breaks
- It is desirable to have the glass quickly form a bead that drops down to allow for bushing restart

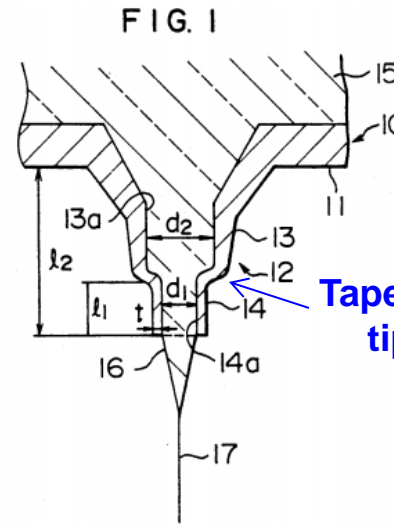
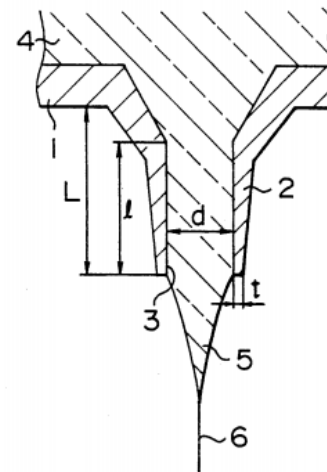


FIG. 3  
PRIOR ART



Non-wetting layer  
(Pt-Au alloy)

FIG. 2

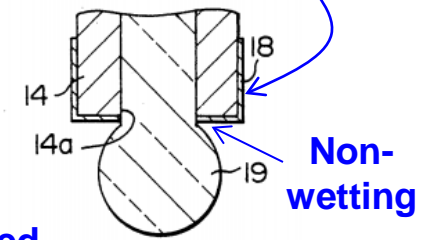


FIG. 4  
PRIOR ART  
Wetting  
("flooding")

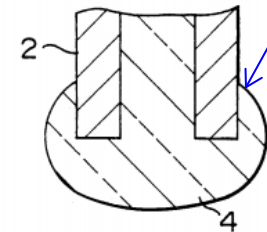
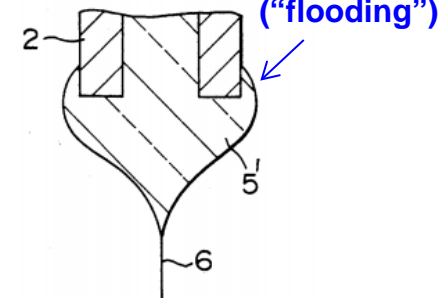


FIG. 5  
PRIOR ART  
Wetting  
("flooding")

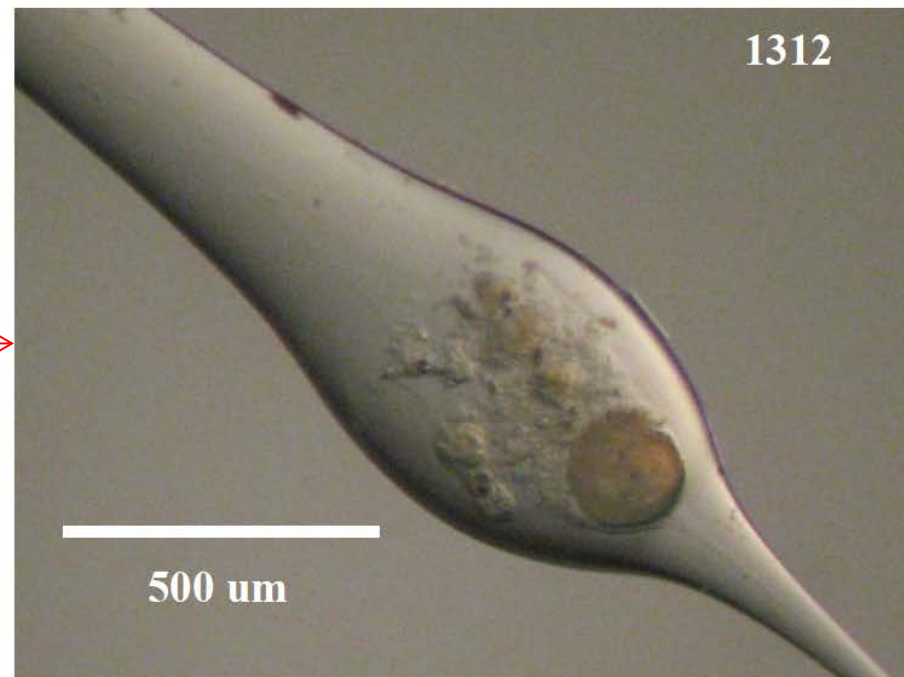
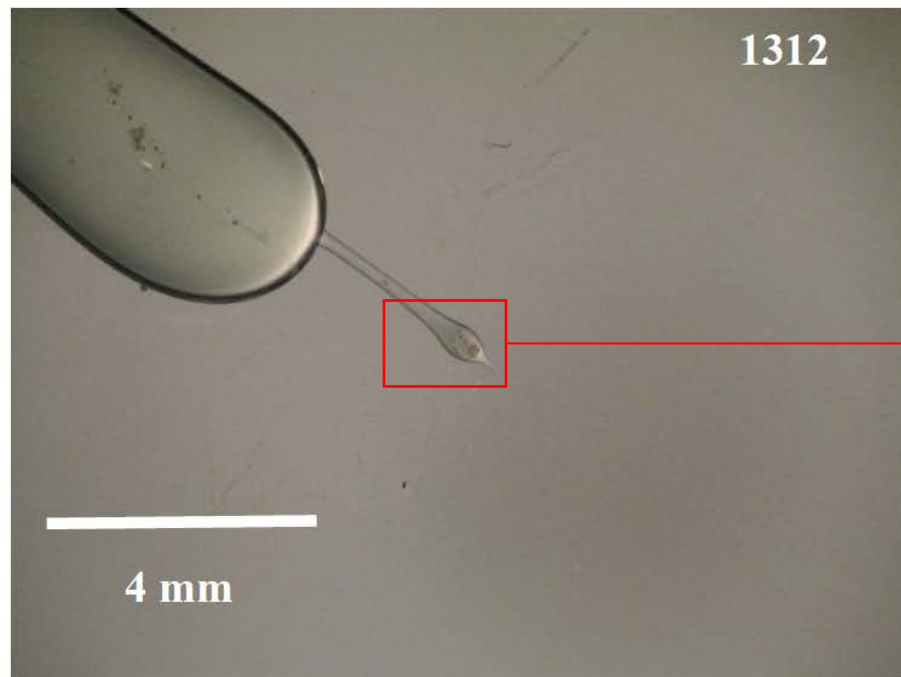


# Collecting Breakout Defects



**Collecting and identifying the cause of a break can be very difficult**

- In some cases the tip that broke first can be identified and the bead collected
- Optical microscopy and SEM/EDS can help with identification

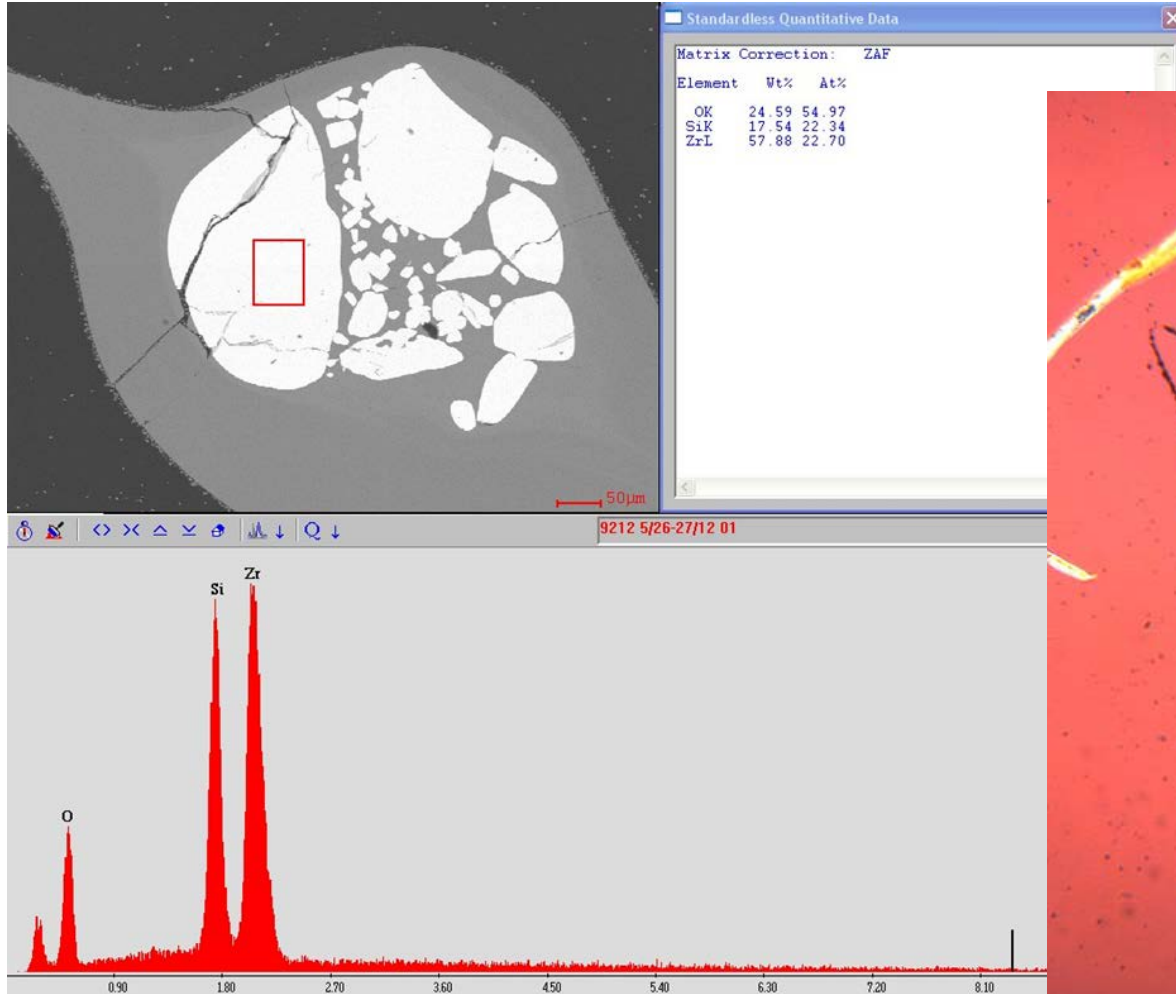


Al-Si defect from contaminant

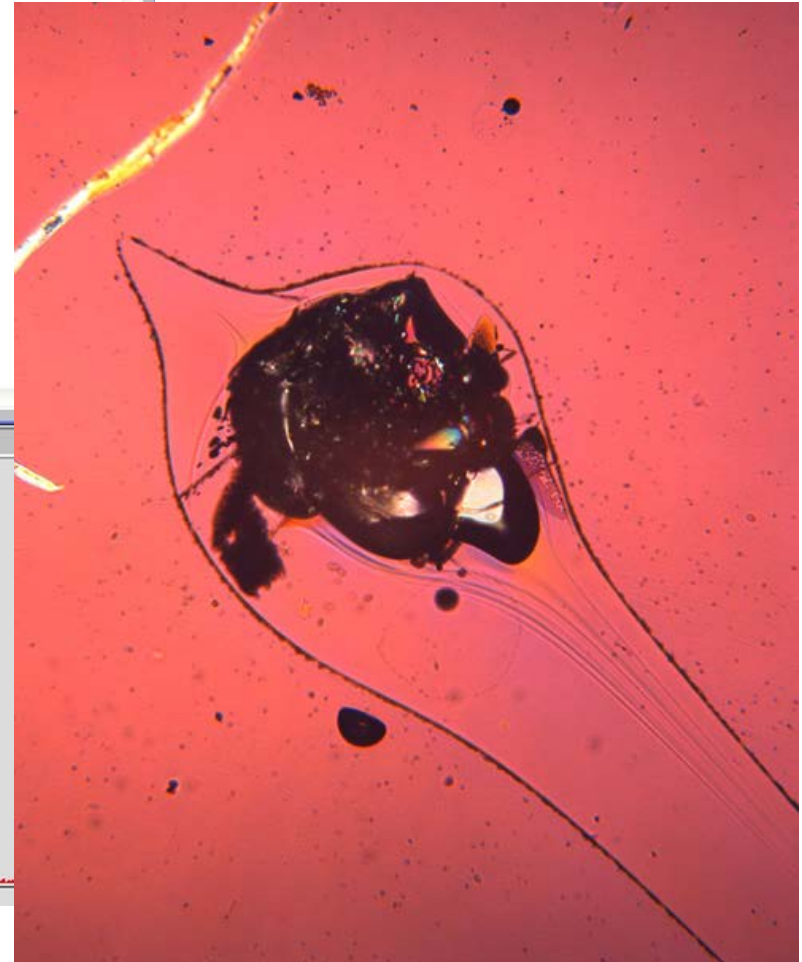
# Identifying Defects



SEM/EDS

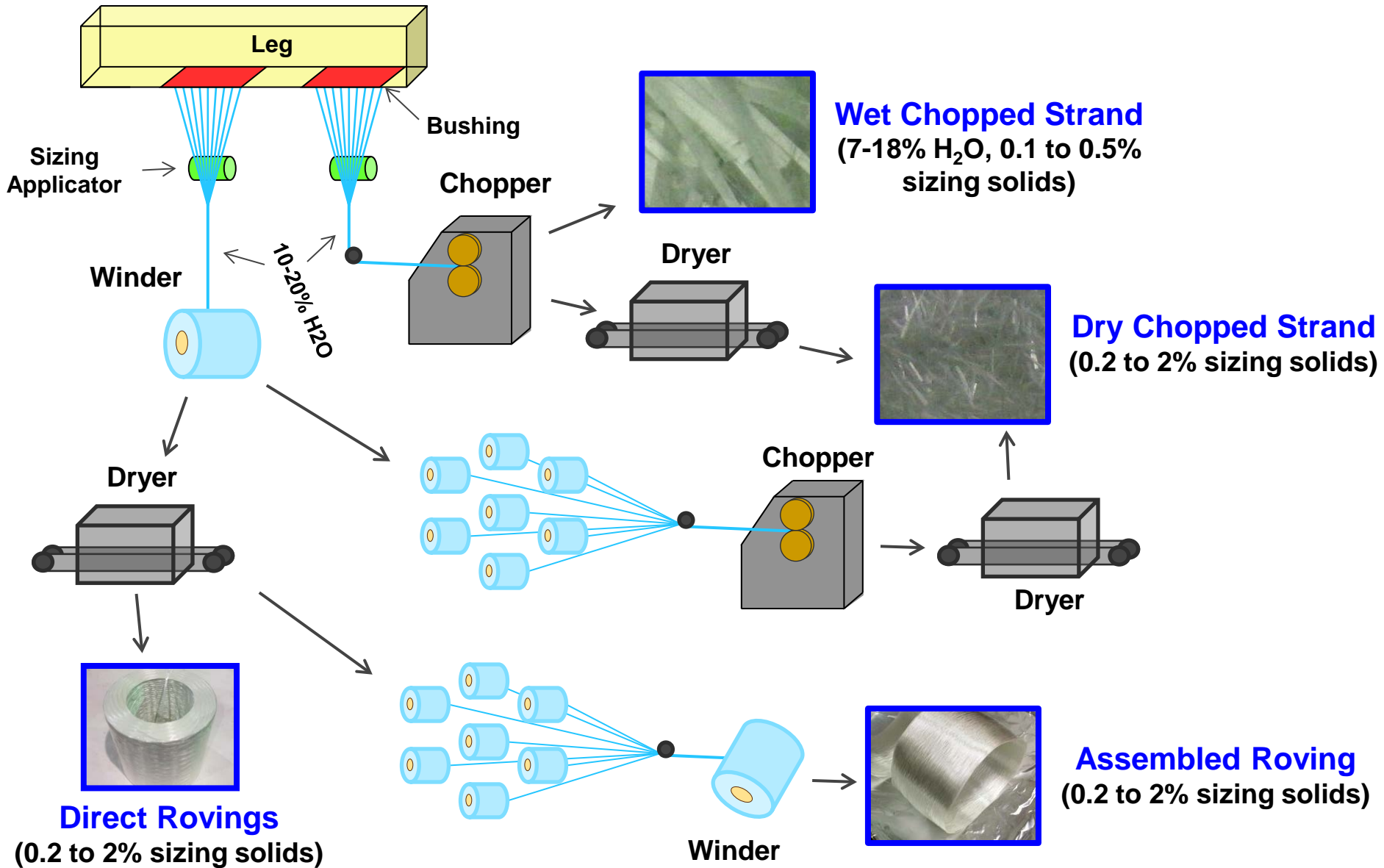


Optical Microscopy



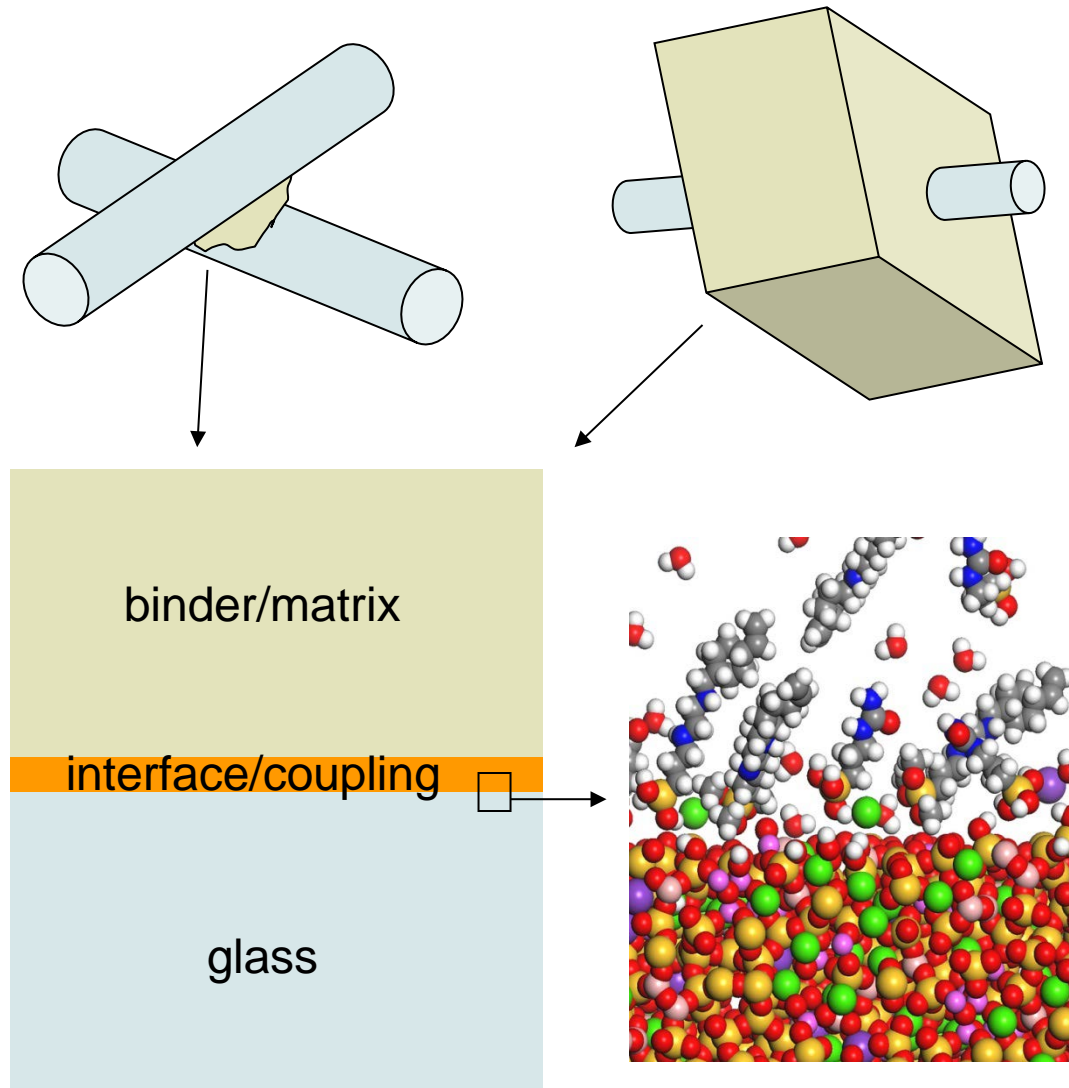
Defect source: zircon refractory

# Continuous Filament Process



# Continuous Filament Sizing

- Tailored to specific application
- Critical for downstream processing, handling, and physical performance
- Key roles
  - Protection from damage (physical & chemical)
  - Lubrication
  - Bundle/strand integrity
  - Dispersion
  - Coupling
- 1 to 10 components
  - Silane (e.g. amino-propyl silane)
  - Film-formers
  - Lubricants
  - Enhancers
  - “Magic ingredients”
  - Other

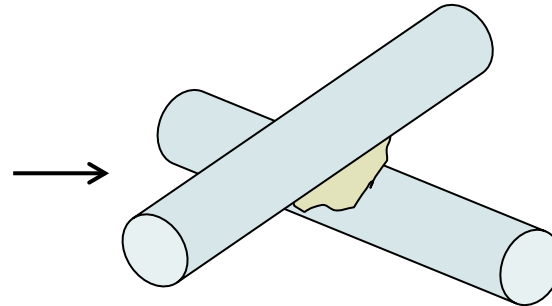


# Sizing Performance: Coupling



Role of silane in the moisture resistance of a fiber/resin bond

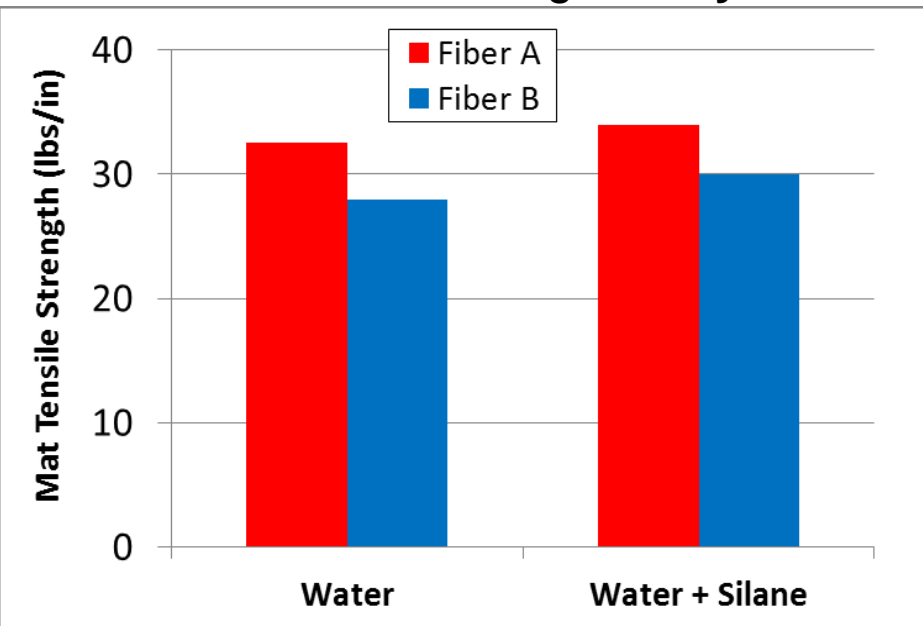
Wet chop fiber



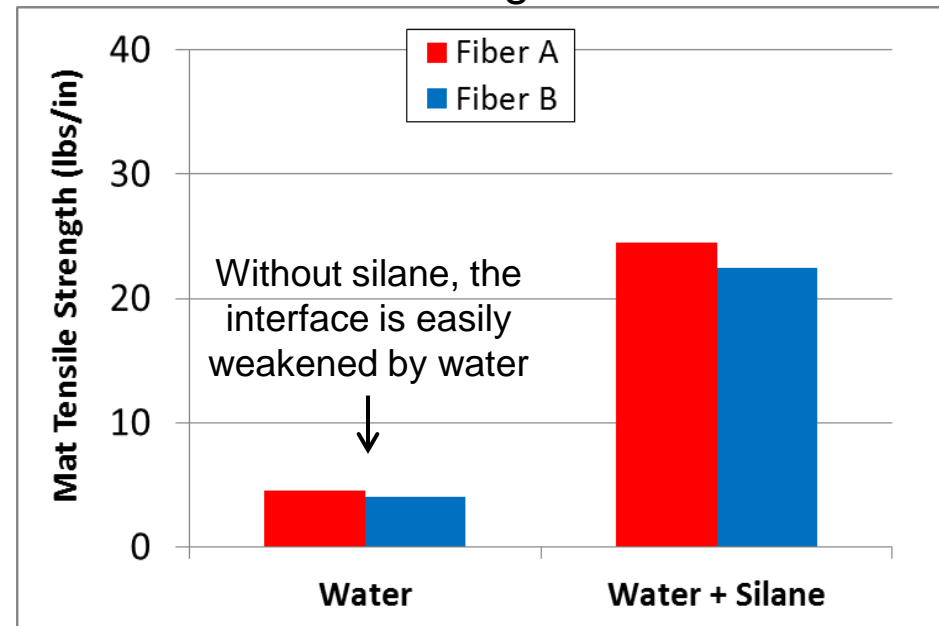
Non-woven mat



Mat tensile strength – dry



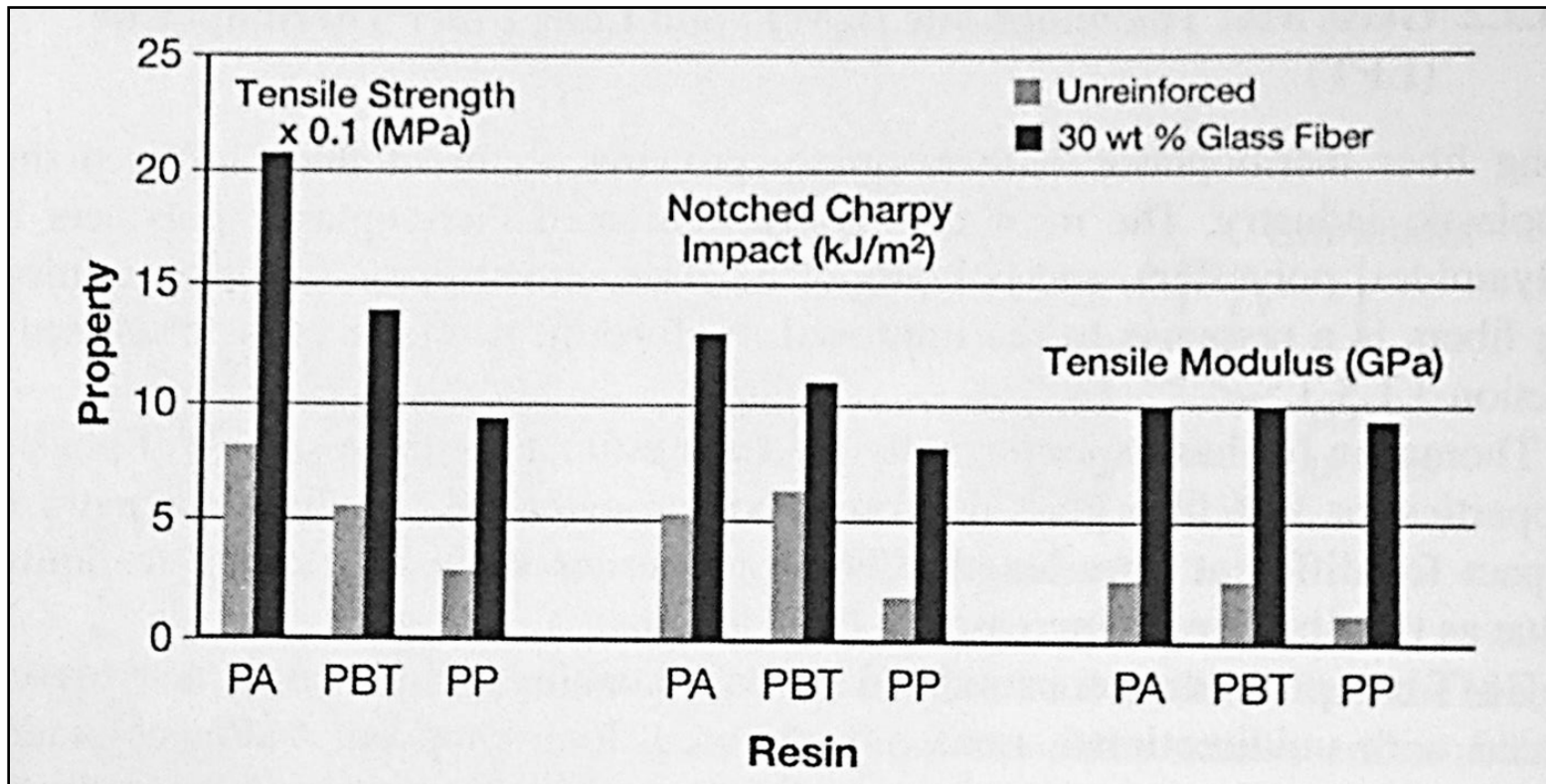
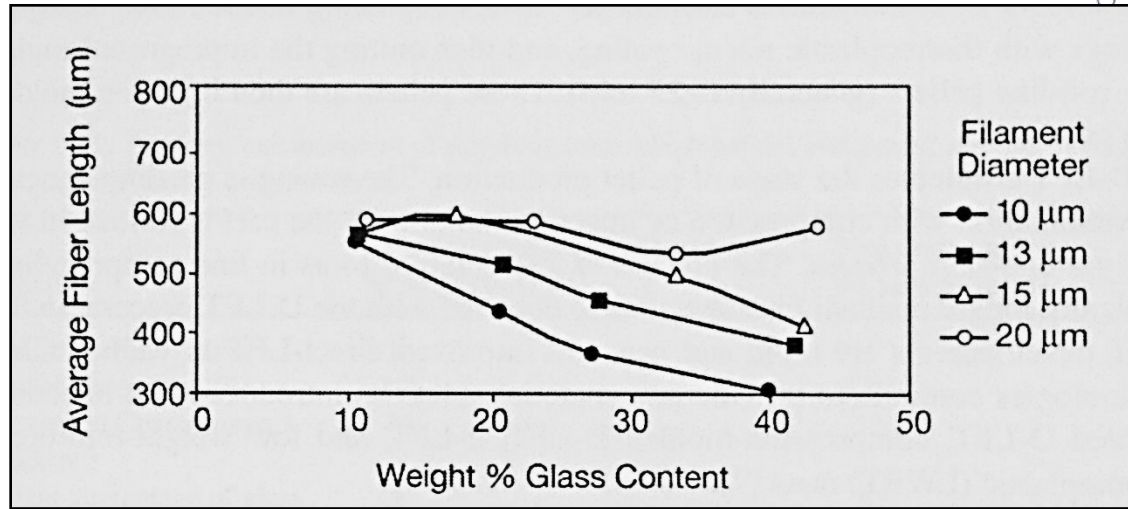
Mat tensile strength – hot/wet



# Application Example: Reinforced Thermoplastic



- Proper sizing chemistry and processing is very important for composites
- Chopped fiber is further broken down in compounding but still delivers a significant increase in strength



J. H. A. van der Woude and E. L. Lawton, 'Composite Design and Engineering' in *Fiberglass and Glass Technology*, F. T. Wallenberger and P. A. Bingham (Editors), Springer, New York, pp. 125-173, 2010

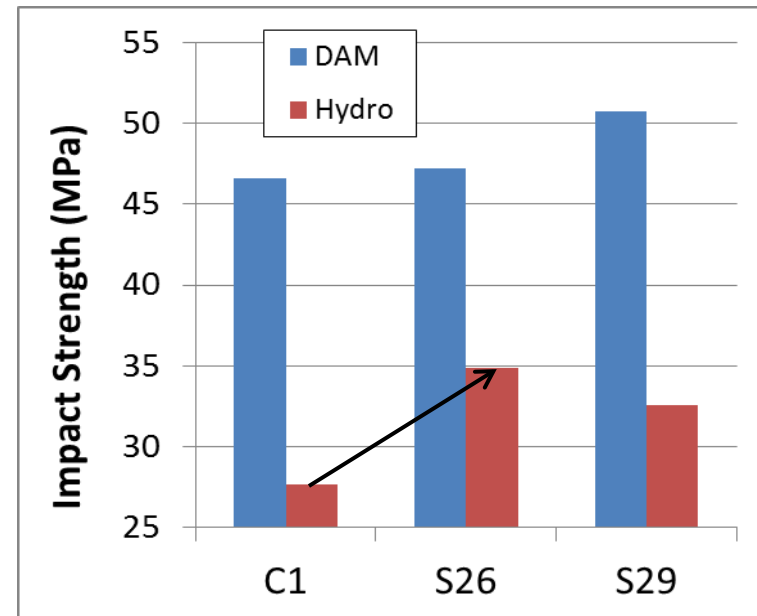
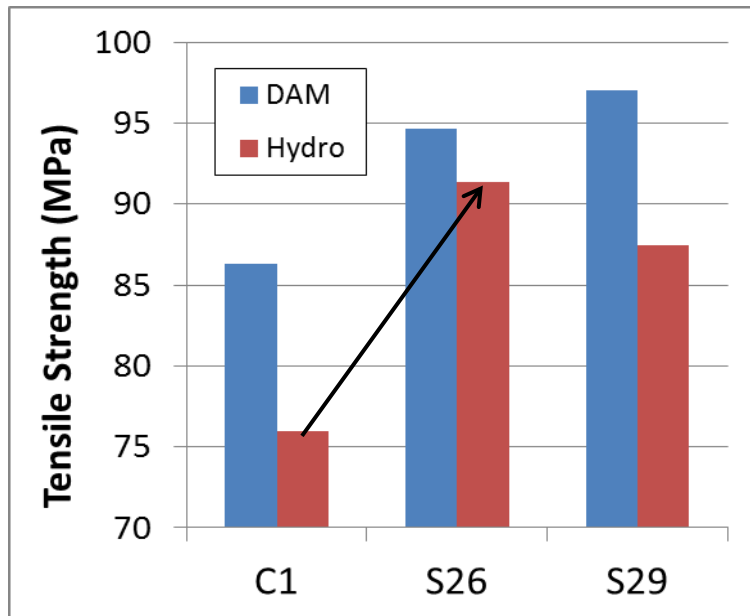
# Sizing Chemistry Example



## Sizing for chopped strand polypropylene reinforcement fiber

		C1	S26	S29
Film-former →	<b>Modified Polypropylene Emulsion</b>	40.71	43.00	43.00
Coupling agent →	<b>Aminopropyltriethoxysilane</b>	5.70	5.69	5.69
“Enhancers” (lubrication, wetting, hydrophobic interphase promotion, etc)	<b>Saturated Fatty Acid</b>	15.20	15.15	15.15
	<b>NH<sub>4</sub>BF<sub>4</sub></b>		1.54	0.68
	<b>Na<sub>2</sub>HPO<sub>4</sub></b>			1.45
	<b>Water</b>	306.70	337.81	347.76

30% chopped fiber, 67.8% polypropylene resin, 2.2% polypropylene additive



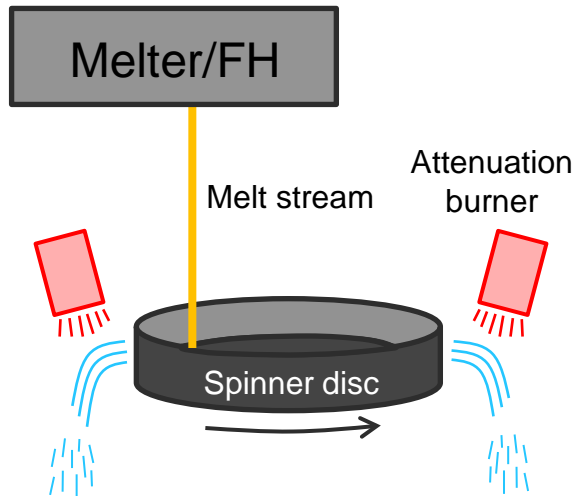
(Data from US Patent 7,732,047)



# Wool Processes

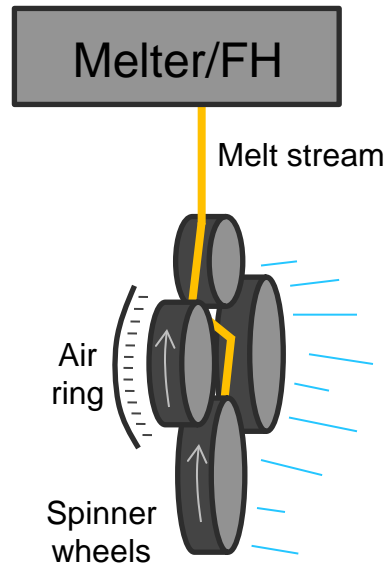


## Rotary (Internal Centrifuge)



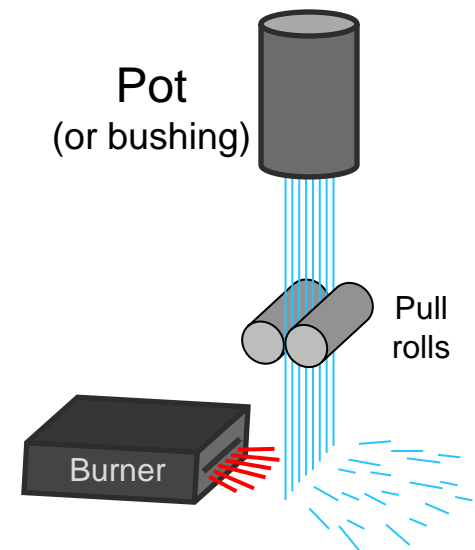
- High throughput
- 1.5 to 8 $\mu$ m average diameter
- Good fiber quality
- Low temperature (soft) glasses
- Sensitive to liquidus
- High energy

## Cascade (External Centrifuge)



- Very high throughput
- 3 to 10 $\mu$ m average diameter
- Poor fiber quality (shot)
- Mineral wool and high temp. (refractory) fiber
- Tolerates very high liquidus
- Low energy

## Flame Attenuation (Pot & Marble)



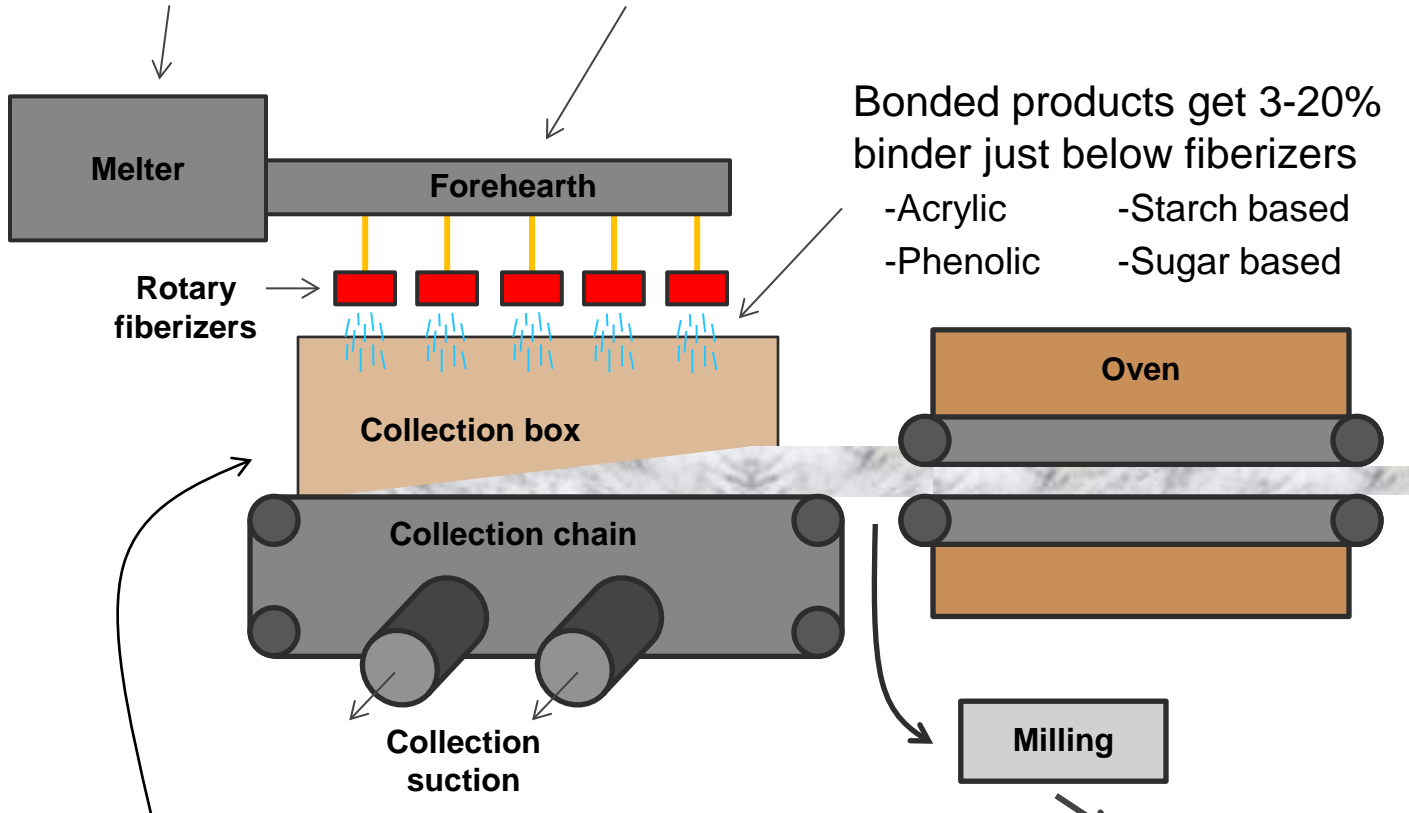
- Low throughput
- 0.1 to 6 $\mu$ m average diameter
- Very good fiber quality
- Capable of relatively wide viscosity range
- Very sensitive to liquidus
- Very high energy

# Rotary Wool Process



Typically direct melt with gas or electric melters.

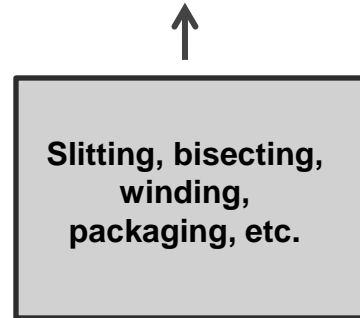
Melt conditioning can range from full forehearth to simple diverter troughs and small holding vessels



Bonded products get 3-20% binder just below fiberizers

- Acrylic
- Starch based
- Phenolic
- Sugar based

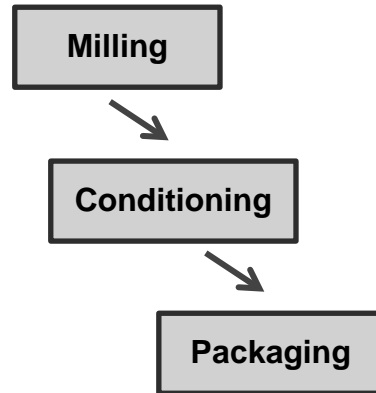
## Boards & Blankets



Many collection box designs

- 1-10 fiberizers per box
- Collection by chain or drum
- 1-3 collection boxes per line
- Air lappers to distribute fiber

## Loose-Fill

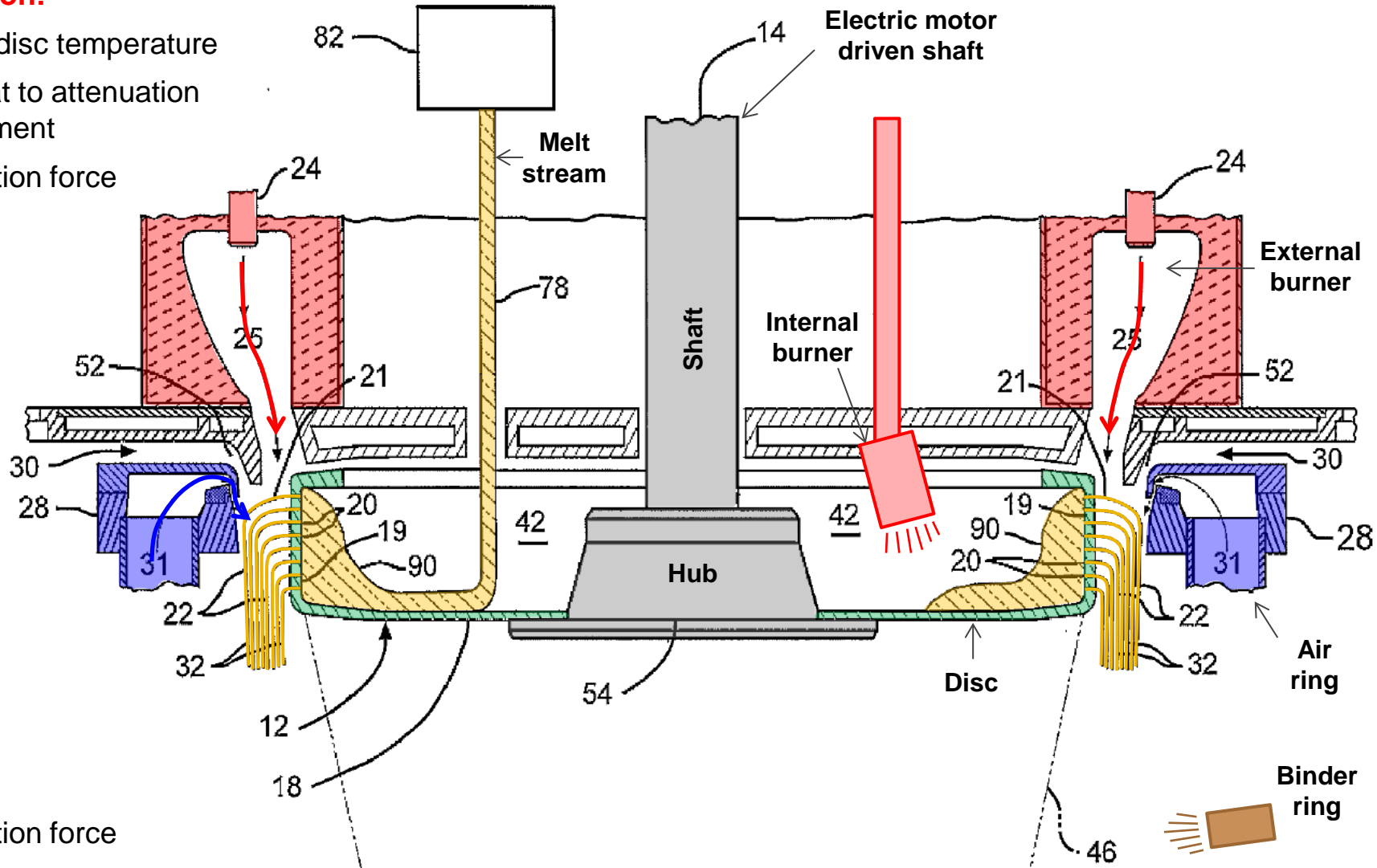


# Rotary Fiberizer Details



## Combustion:

- Control disc temperature
- Add heat to attenuation environment
- Attenuation force



(US Patent 8,250,884)

## Air Ring:

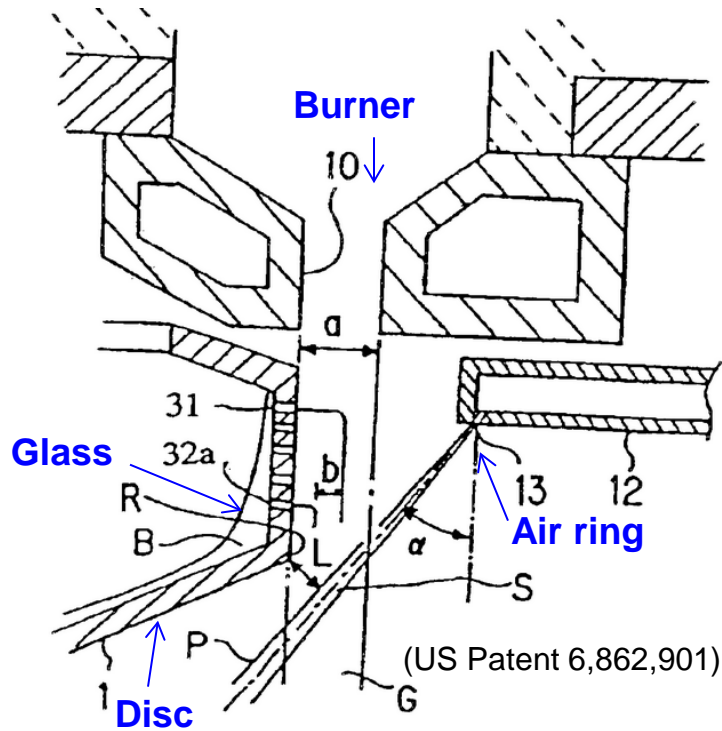
- Attenuation force
- Containment of fiberization
- Containment of column



# Complex Fiberization Environment



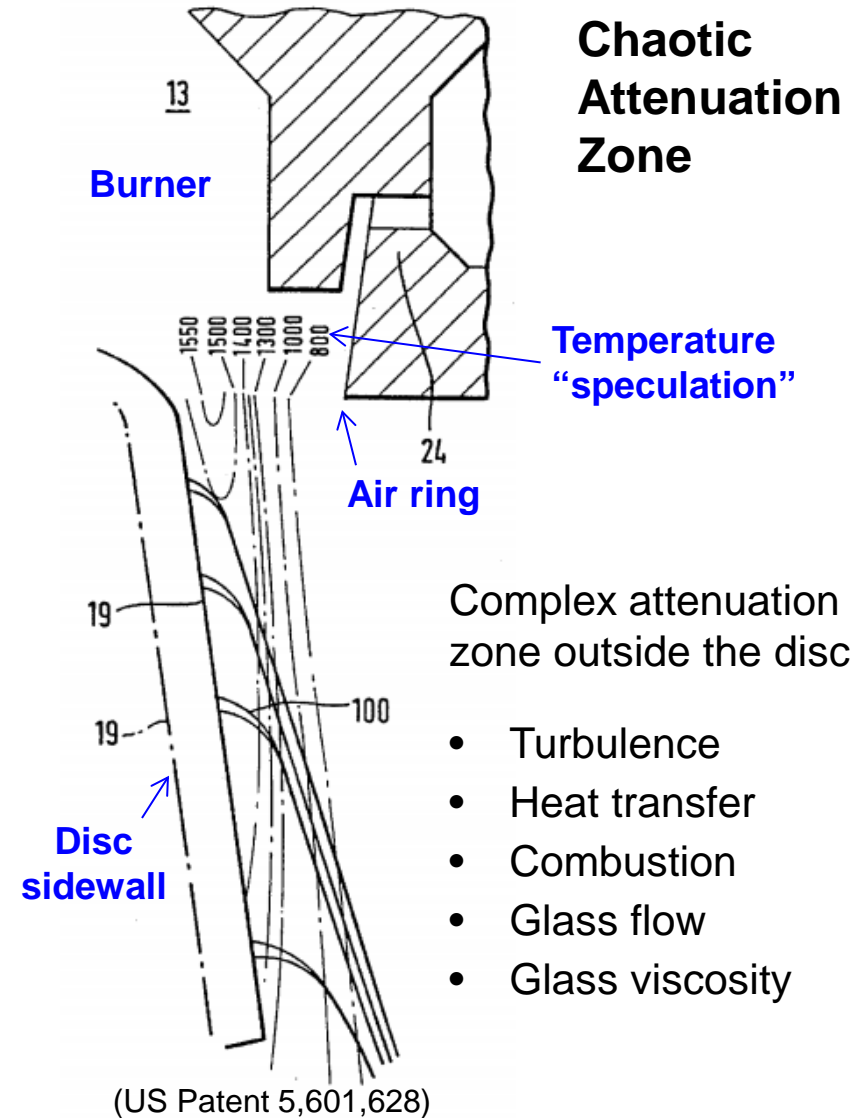
The life of a disc is harsh and short



- High temperatures
- High stress from rotation (500-1000g's)
- Molten glass corrosion
- Combustion environment

A more challenging strength, ductility, and corrosion environment than jet turbine blades

Good materials science challenge



Complex attenuation zone outside the disc

- Turbulence
- Heat transfer
- Combustion
- Glass flow
- Glass viscosity

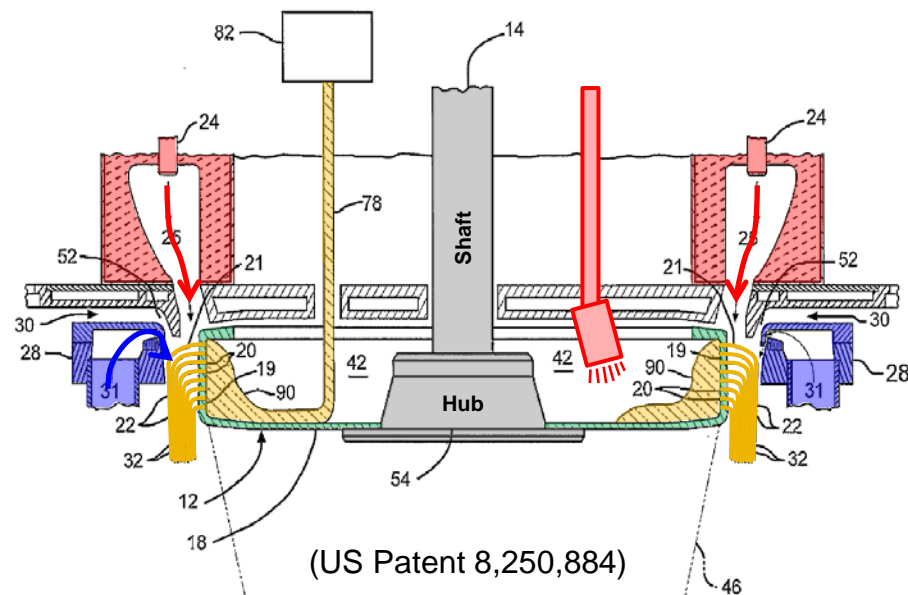
Good modeling challenge

# Rotary Wool Design

*It is challenging to develop an optimized design because rotary fiberization is a very complex system of interactions and competing parameters*

## Some Considerations

- Glass melt properties (viscosity, liquidus, heat transfer)
- Product requirements (thermal, mechanical)
- Disc alloy (composition, melting and forming process)
- Disc dimensions (diameter, thickness, wall height, flange, etc.)
- Hole pattern (diameter, count, profiling, banding, etc)
- Internal & external combustion energy and placement
- Air ring placement and pressure
- Conditions for disc removal (disc life)
- Glass stream temperature
- Disc throughput (loading)
- Disc rotational speed
- Number of fiberizers in collection box
- Number of collection boxes per line
- Fiber laydown pattern (column formation)



# Rotary Wool Parameters



## Some Typical Ranges

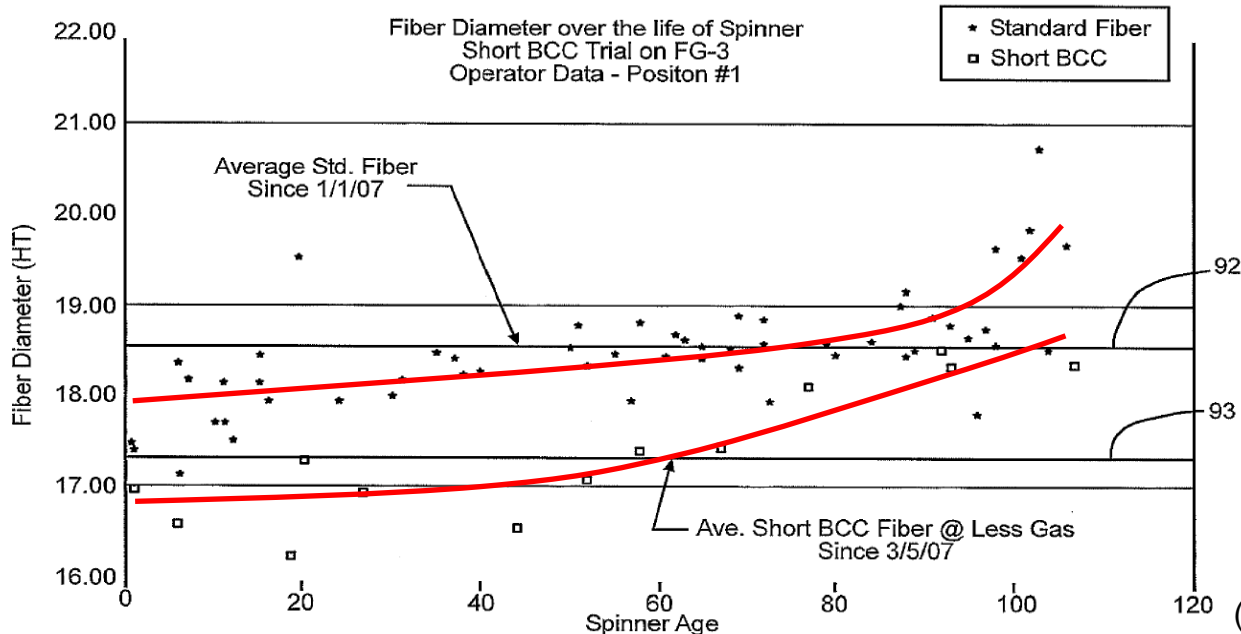
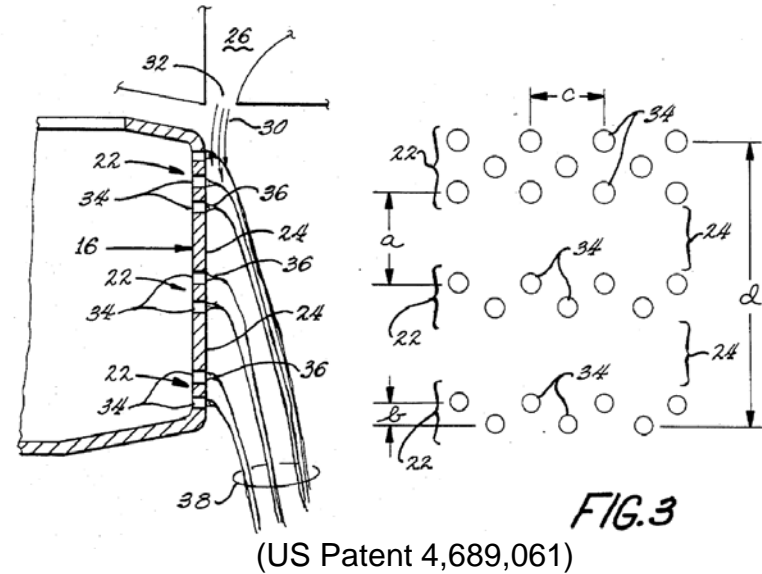
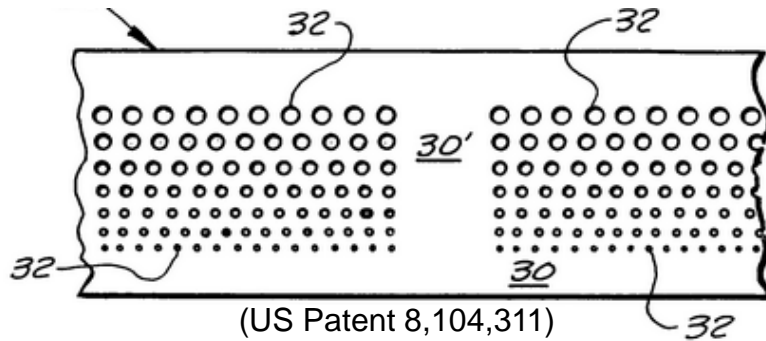
- Glass melt viscosity (temp. at 1000poise) 900 to 1100°C
- Glass melt liquidus temperature 700 to 1100°C
- Average fiber diameter 1.5 to 8  $\mu\text{m}$
- Average fiber length difficult to characterize
- Disc diameter 25 to 100 cm
- Disc alloy Co-Ni-Cr superalloy
- Number of holes thousands
- Hole diameter 0.4 to 1.0 mm
- Disc rotational speed 1500 to 2500 rpm
- Disc throughput 100 to 1100 kg/hr
- Disc life 20 to 400 hours
- Number of fiberizers in collection box 1 to 10
- Number of collection boxes per line 1 to 3

# Rotary Fiberization



## Hole Size Profiling

Top holes are typically larger because the glass coming out of the top holes will experience more attenuation than the bottom holes



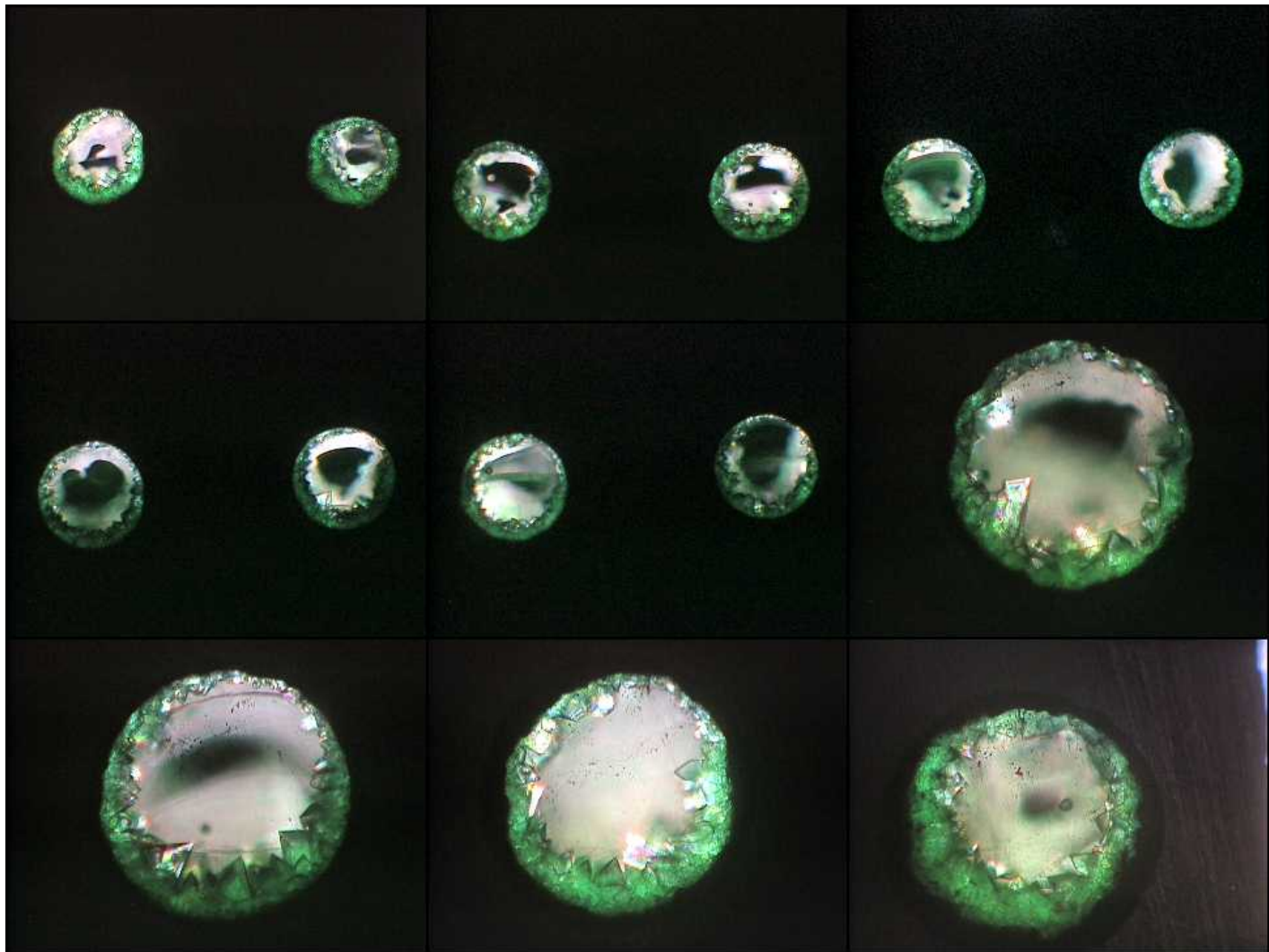
## Hole Wear

Hole size increases over the life of the disc, causing a fiber diameter increase if no process adjustments are made

# Devitrification in Discs

**Glass chemistry or process upsets can cause devitrification in discs**

Can occur in cold corners of disc or in holes as shown below





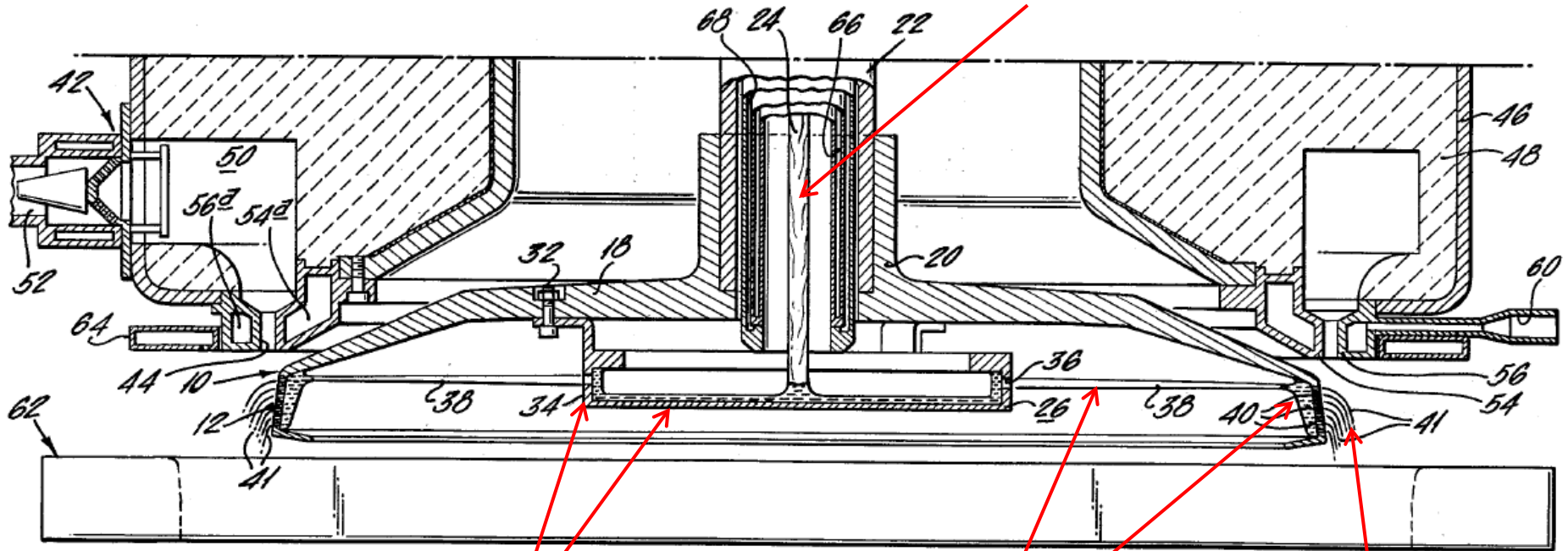
# Rotary Design Variations



There are many variations, including this “upside-down” disc design

*Fig. 1.*

A) Glass melt stream enters through center of hollow disc shaft



B) Melt stream collects in small interior slinger disc with large holes around the sidewall

C) Glass from slinger disc flies outward to collect in large “upside-down” disc

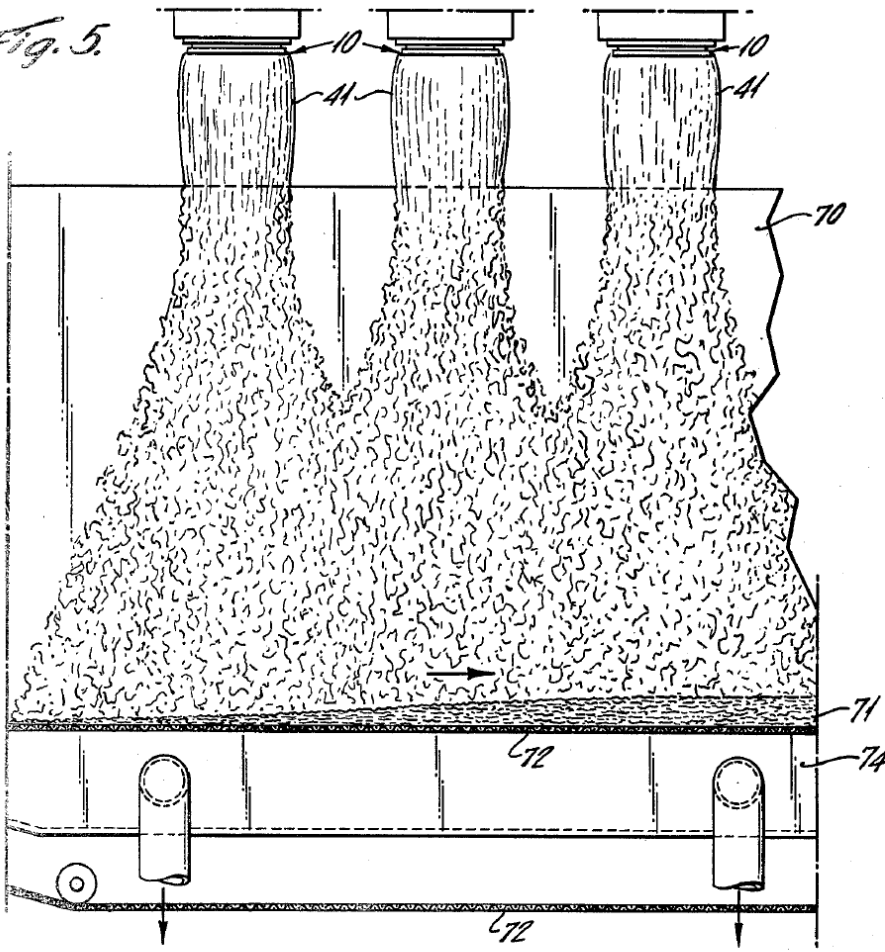
D) Fiberization occurs in a similar fashion to other discs

# Collection Designs



Typical

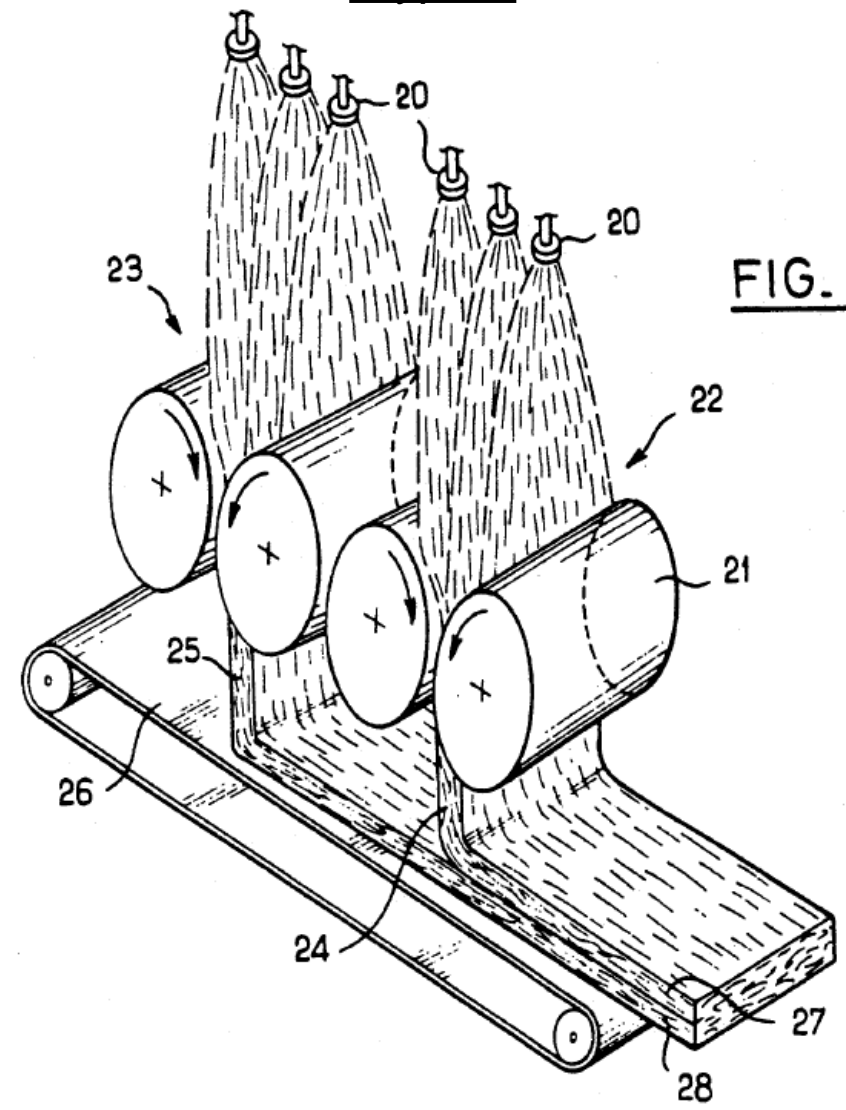
*Fig. 5.*



(US Patent 4,451,276)

Atypical

FIG. 3

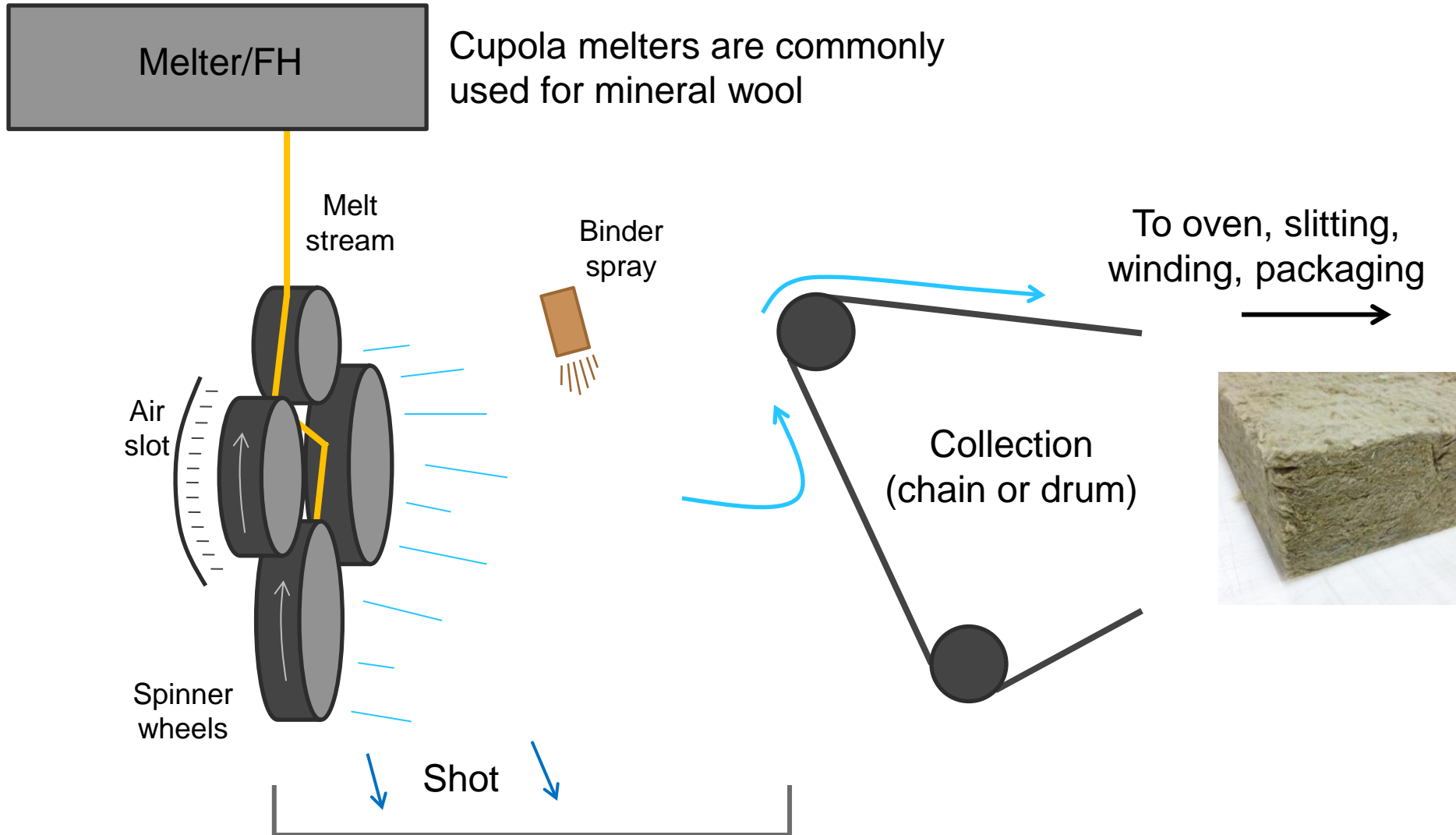


(US Patent 5,268,015)

# Cascade (External Centrifuge) Process



The cascade process is commonly used for mineral wool and other high liquidus temperature glasses



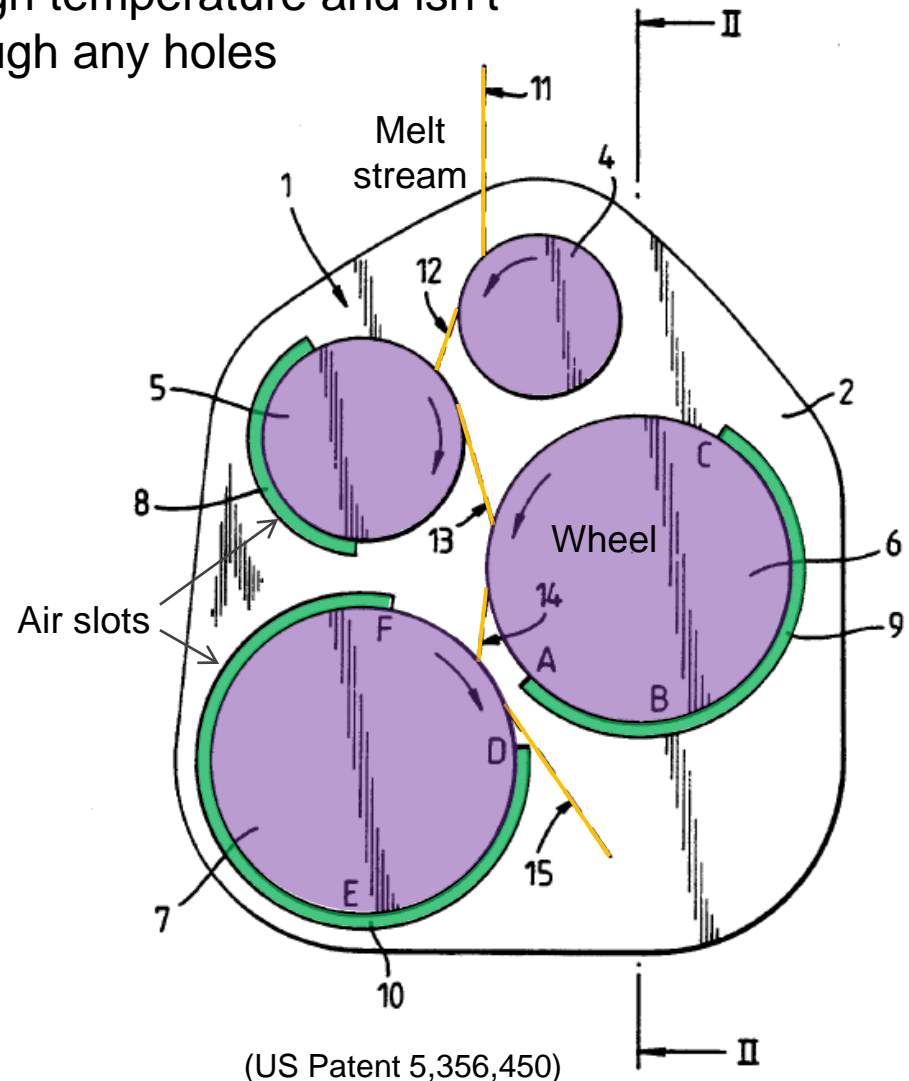
# Cascade Process Detail



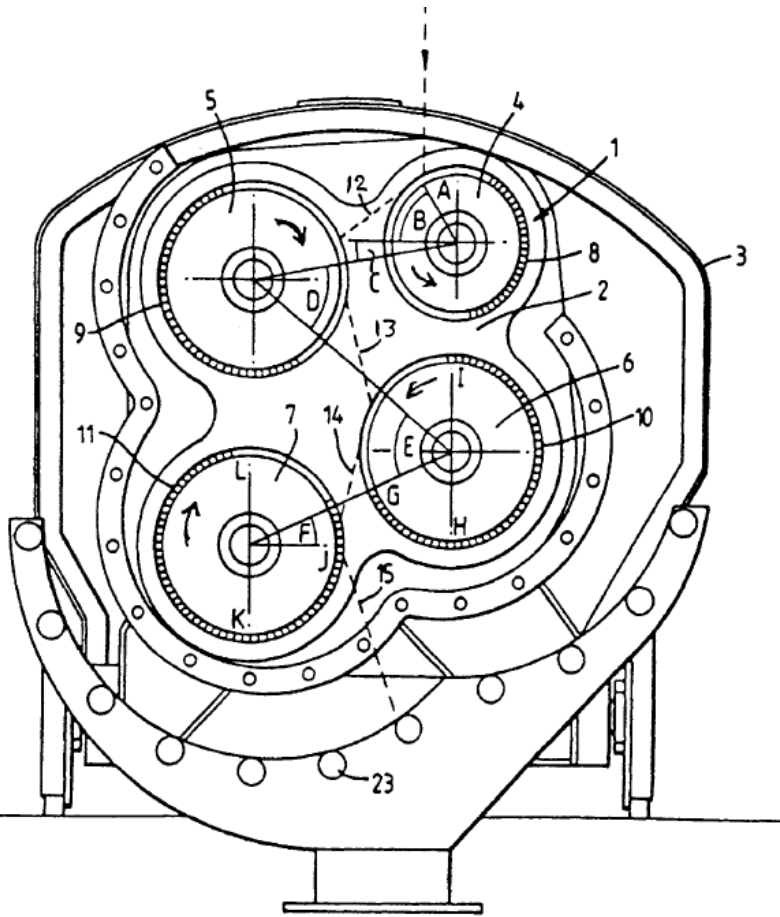
Used for glasses with high liquidus temperature because the melt doesn't have an opportunity to devitrify

The melt stream drops onto the wheels at high temperature and isn't held in a containment device or pushed through any holes

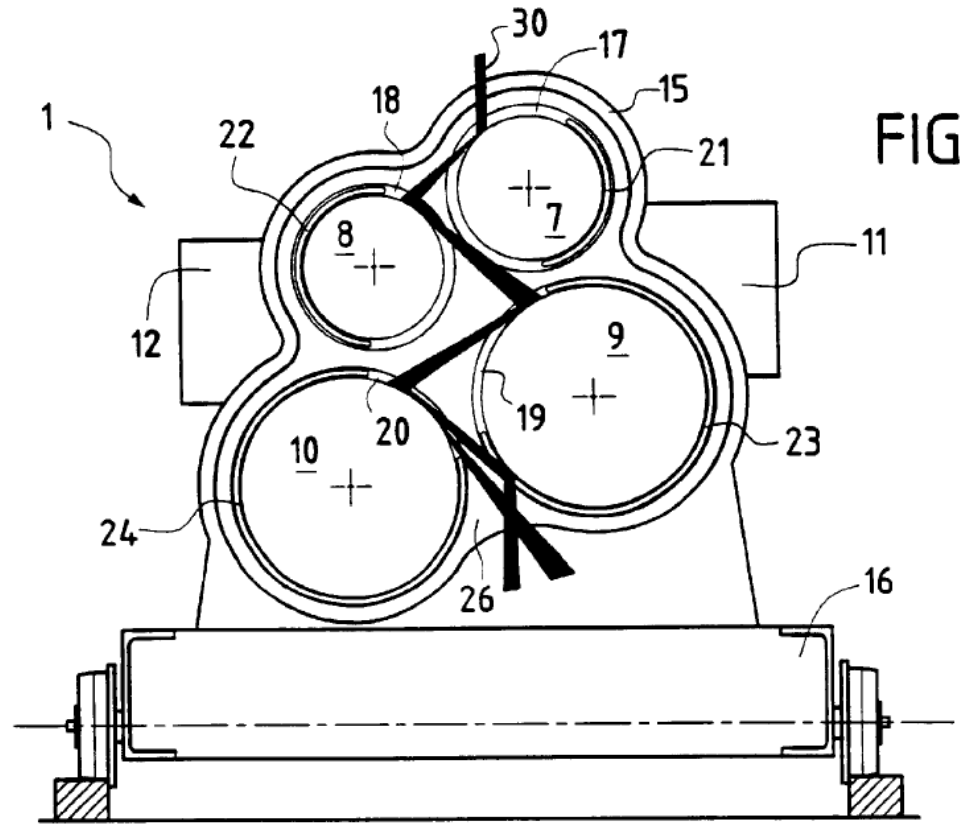
- Melt stream temperature is  $>1400^{\circ}\text{C}$
- Viscosity is 15-40poise at the first wheel
- 15-50cm diameter wheels
- 3000-9000 RPM wheel speed
- Air slot around 30-80% of wheel
- Throughput of 2000-7000kg/hr



# Cascade Design Examples



(US Patent 5,954,852)



(US Patent 6,536,241)

# Shot and Fiber Quality



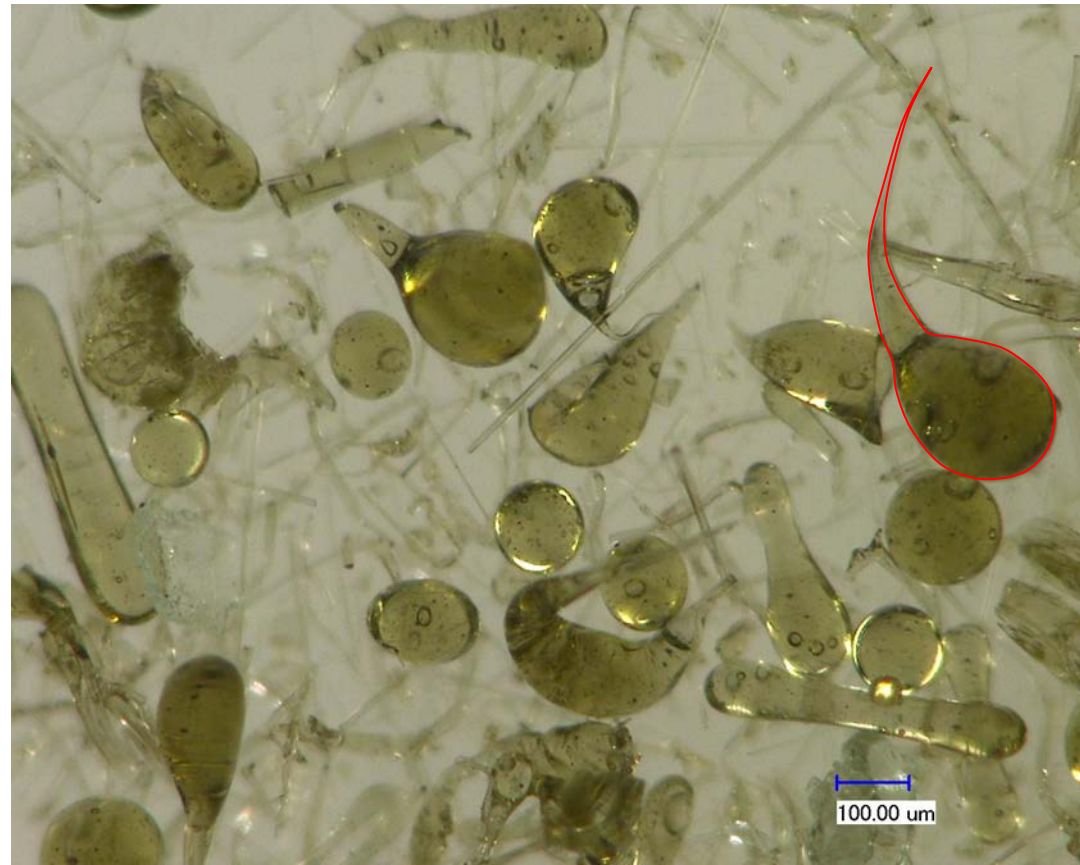
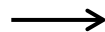
## Cascade Advantages: High liquidus temperature glasses and high iron glasses

- Fiber has better high temperature and fire resistance performance
- Lower cost glass raw materials

## Cascade Disadvantage: Fiber quality generally not as good as rotary

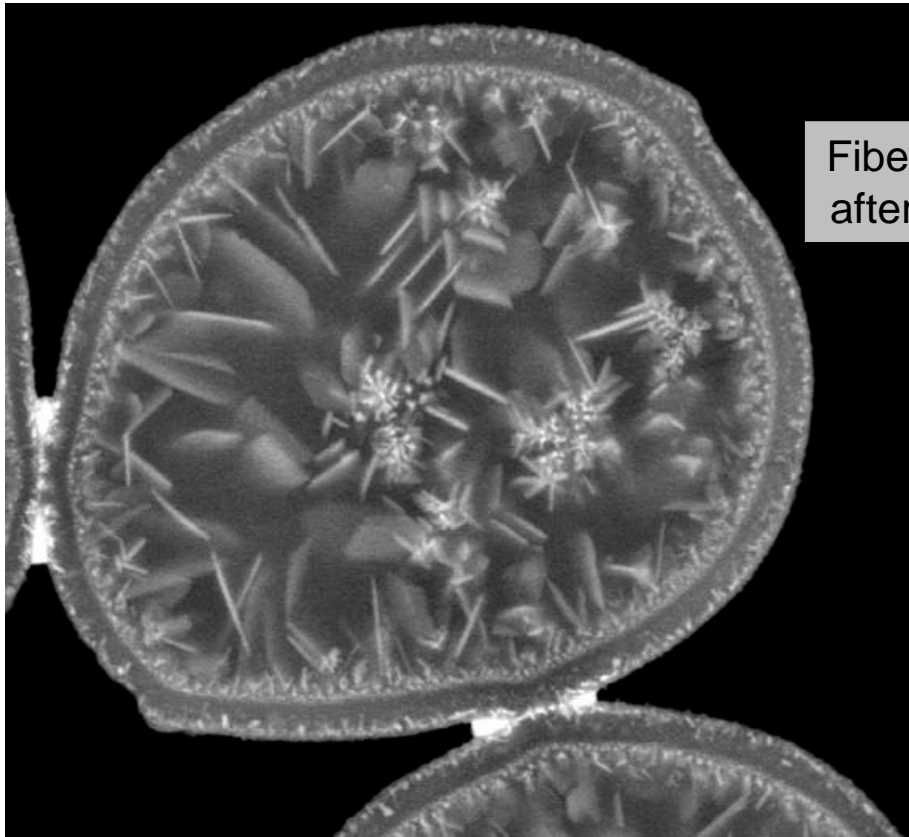
- Higher shot content
- Lower aspect ratio, shorter fiber
- Higher thermal conductivity for the same density (due in part to shot)

**Shot** – Non-fiberized pieces of glass, often with attenuation “tails”

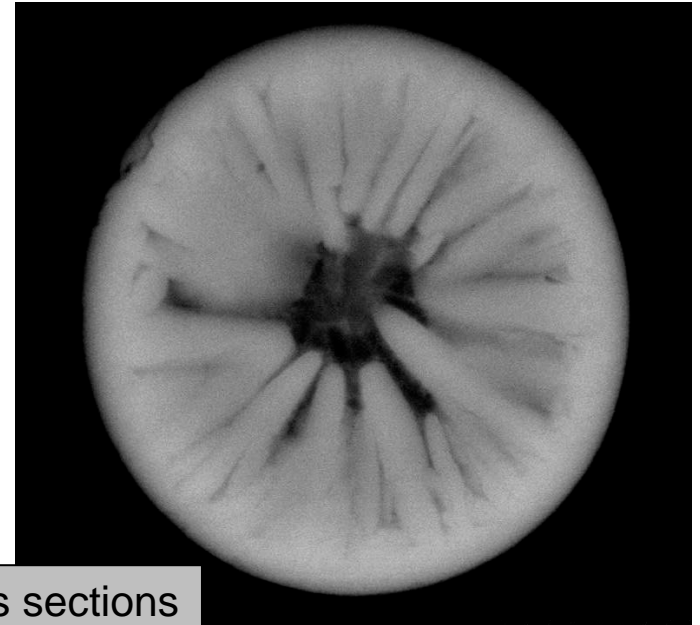


# Mineral Wool Crystallization

- High liquidus temperature glass fibers can crystallize upon reheating
- Can lead to desirable high temperature & fire resistance properties
- Illustrates how challenging these glasses can be for fiberization processes



Fiber cross sections  
after heat treatment

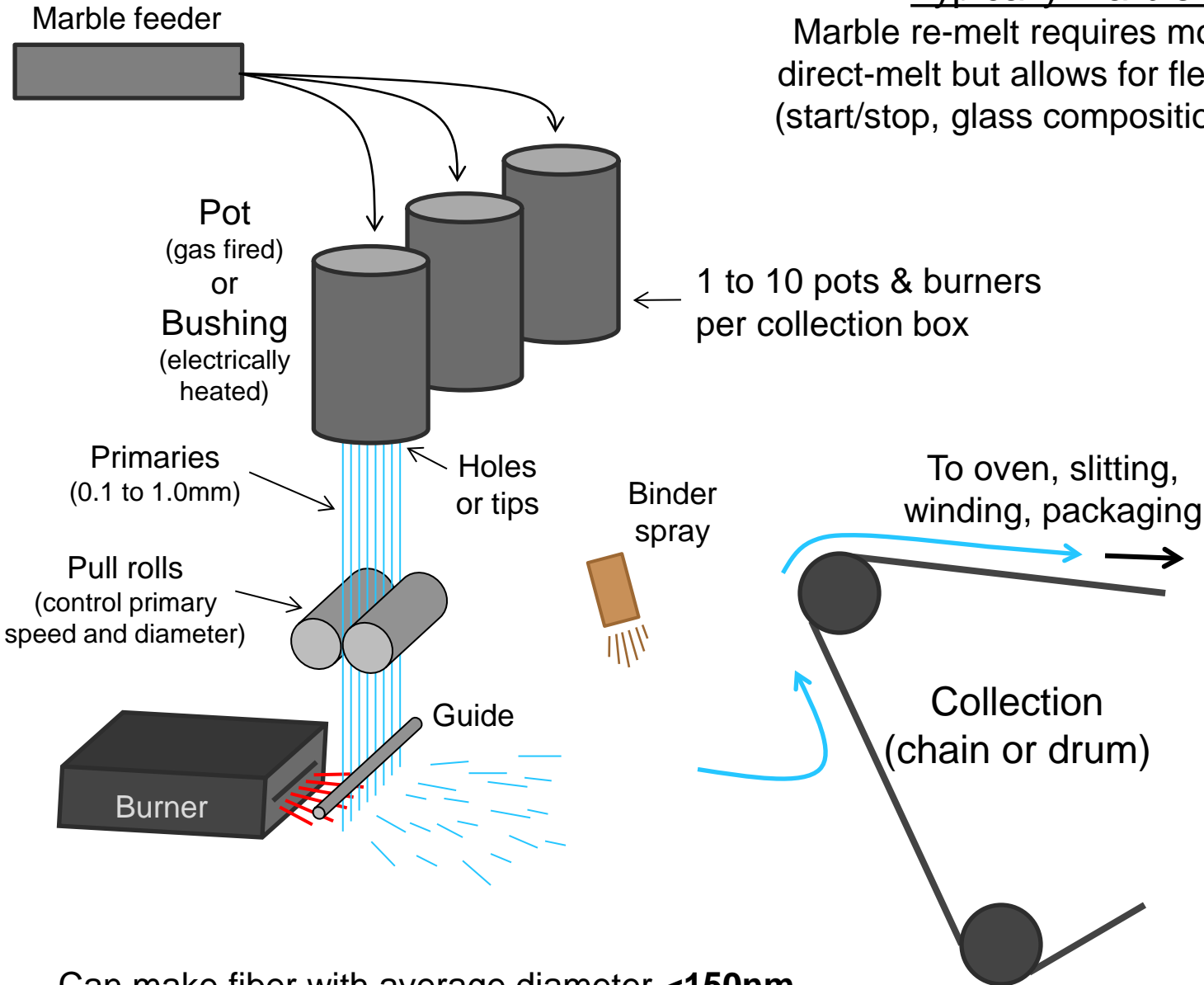


# Flame Attenuation (Pot & Marble)



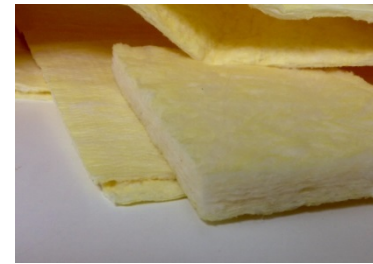
## Typically marble re-melt

Marble re-melt requires more energy than direct-melt but allows for flexible fiberization (start/stop, glass composition changes, etc.)



1 to 10 pots & burners per collection box

Bonded Microfiber



Non-bonded Microfiber



Can make fiber with average diameter <150nm

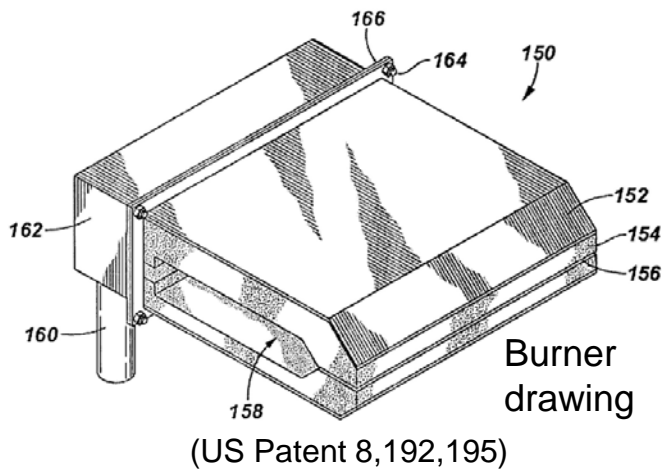
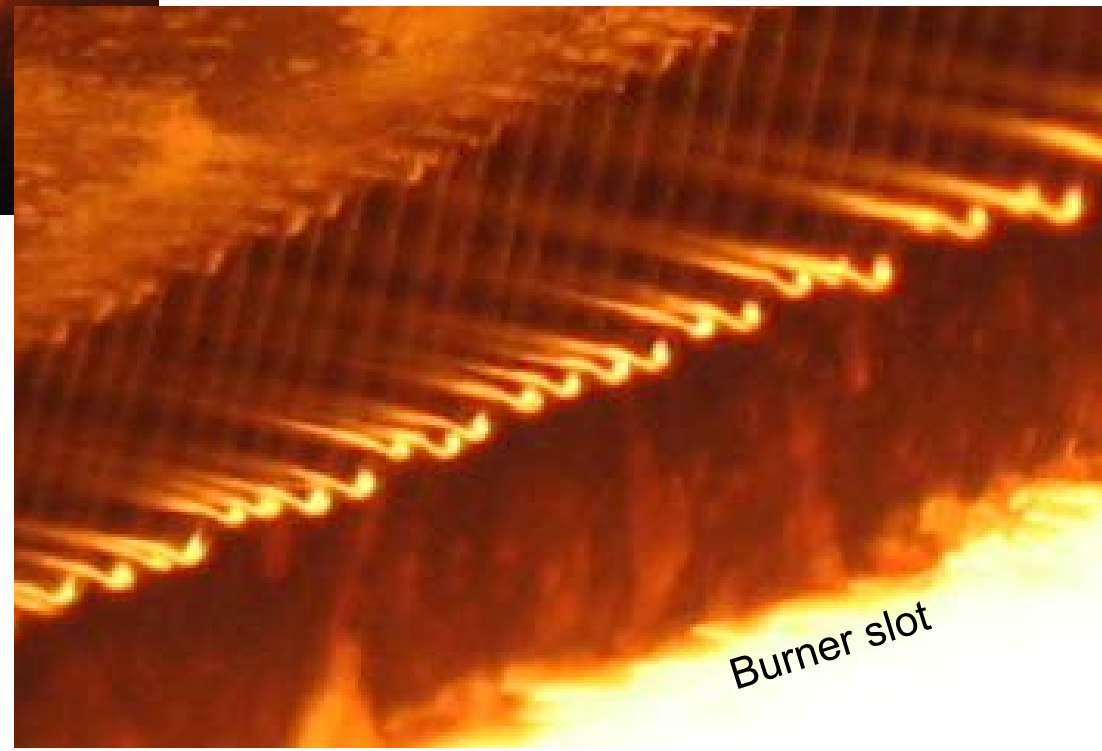
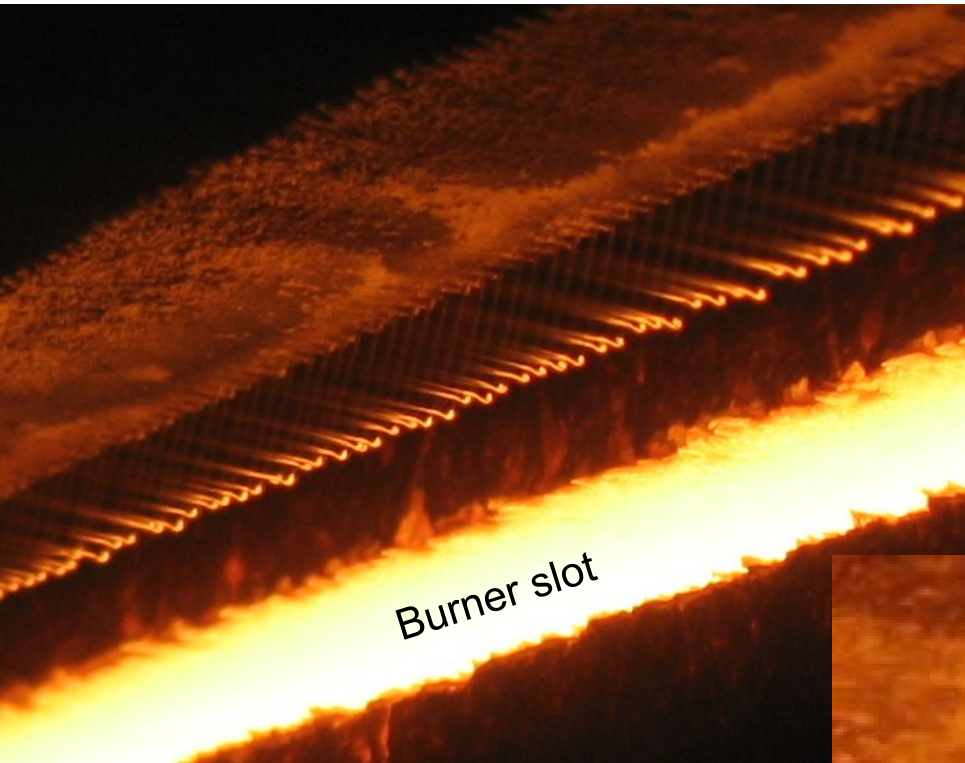


# Flame Attenuation Close-Up

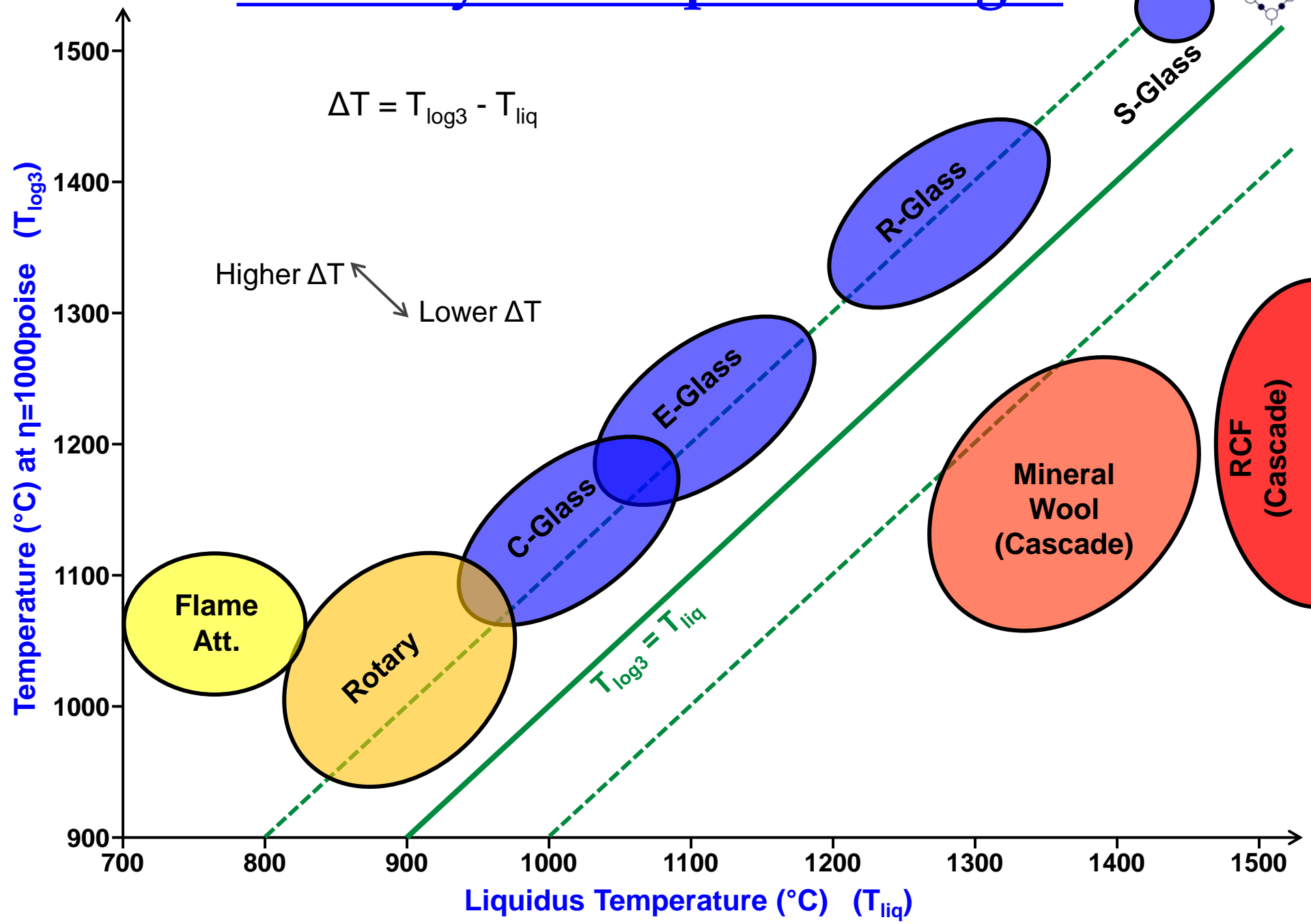


Attenuation influenced by:

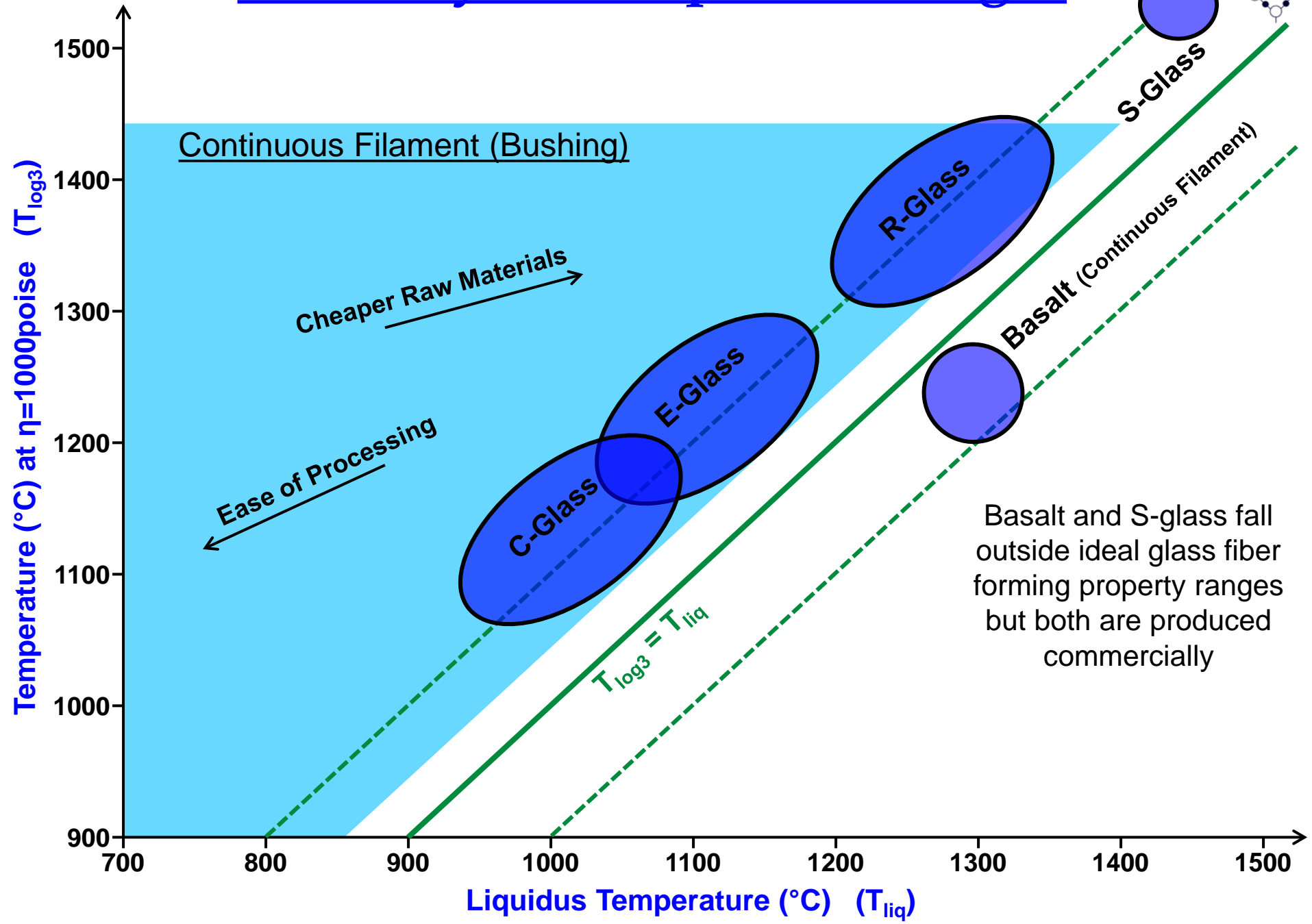
- Primary diameter
- Primary speed
- Primary spacing
- Burner temperature
- Burner pressure
- Burner distance
- Burner slot size



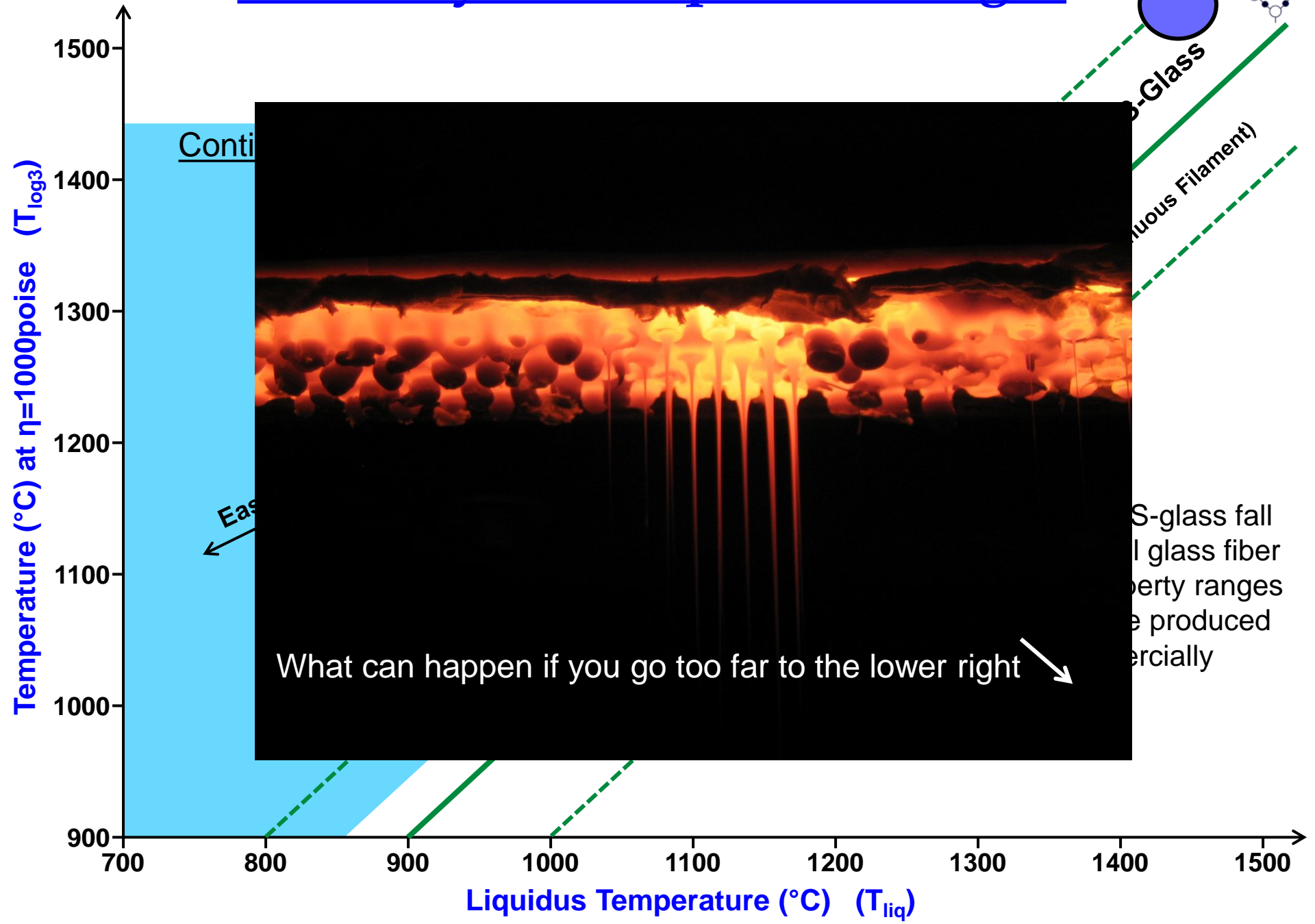
# Viscosity and Liquidus Ranges



# Viscosity and Liquidus Ranges



# Viscosity and Liquidus Ranges



Continuity

Temperature ( $^{\circ}\text{C}$ ) at  $\eta=1000\text{poise}$  ( $T_{\log 3}$ )

E-glass

S-Glass

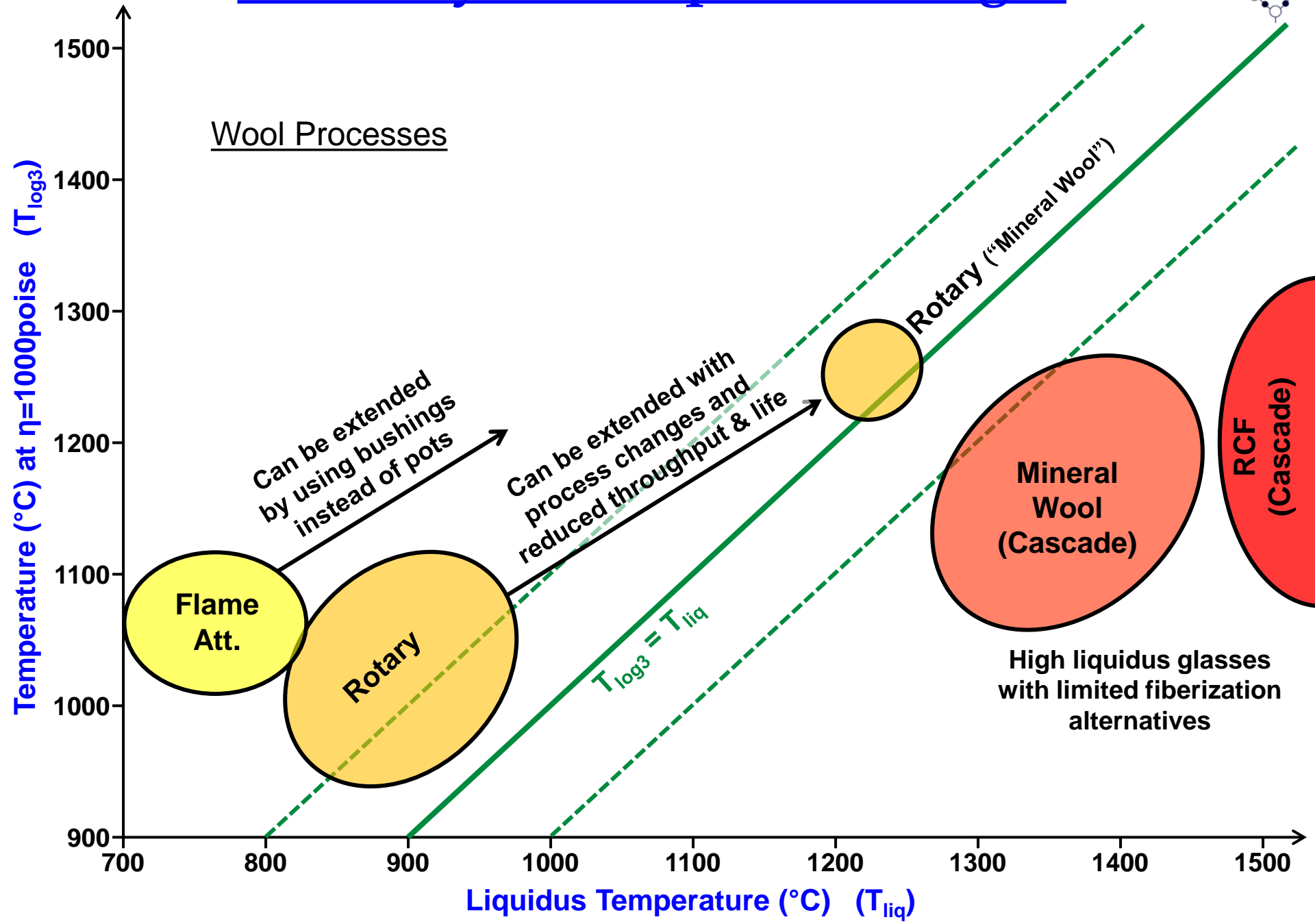
Continuous Filament

S-glass fall  
 l glass fiber  
 property ranges  
 e produced  
 ercially

What can happen if you go too far to the lower right

Liquidus Temperature ( $^{\circ}\text{C}$ ) ( $T_{\text{liq}}$ )

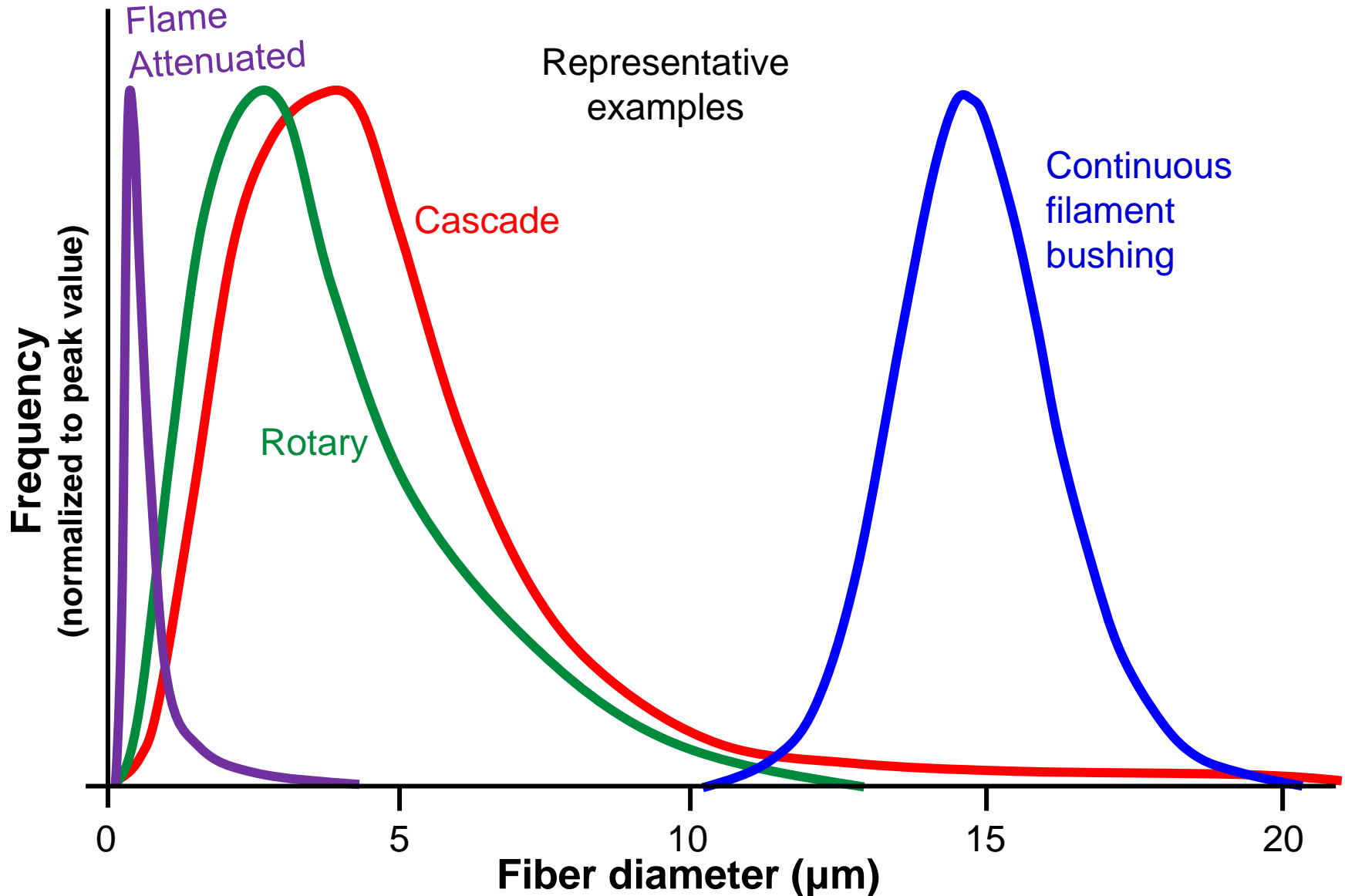
# Viscosity and Liquidus Ranges



# Fiber Diameter Distributions



Diameter distribution size and shape can vary widely depending on the process and how it is operated



# Glass Composition Considerations



**Glass compositions are designed to achieve viable balance of properties**

- Raw material costs
- Viscosity
- Liquidus temperature
- Melting enthalpy
- Electrical conductivity (melt)
- Surface tension (melt)
- Fiber tensile strength (and retention)
- Elastic modulus
- Chemical durability
  - Acidic
  - Neutral
  - Basic
  - Unique environments
- Radiative heat transfer (melt & fiber)
- Density
- Refractive index
- Surface reactivity (sizing, binder, resin compatibility)

Items on this partial list will vary in importance depending on process and application



## Future Development

- Compositions and processes are continually being modified and improved
- Improved product properties
- Energy reduction
- Efficiency improvement
- New markets/applications

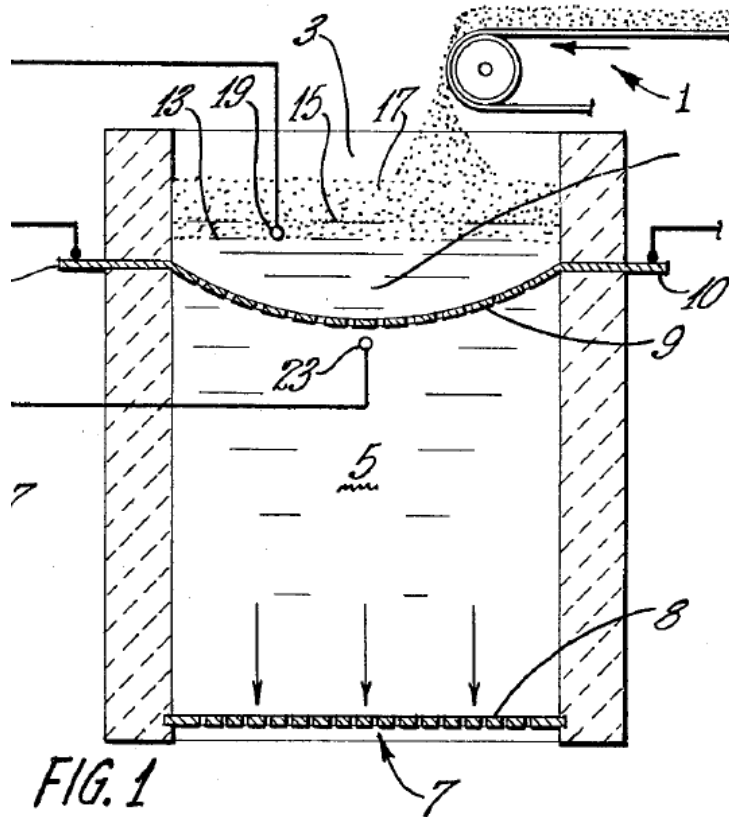
# Other Methods & Variations



(with varying degrees of practicality)

## Paramelter

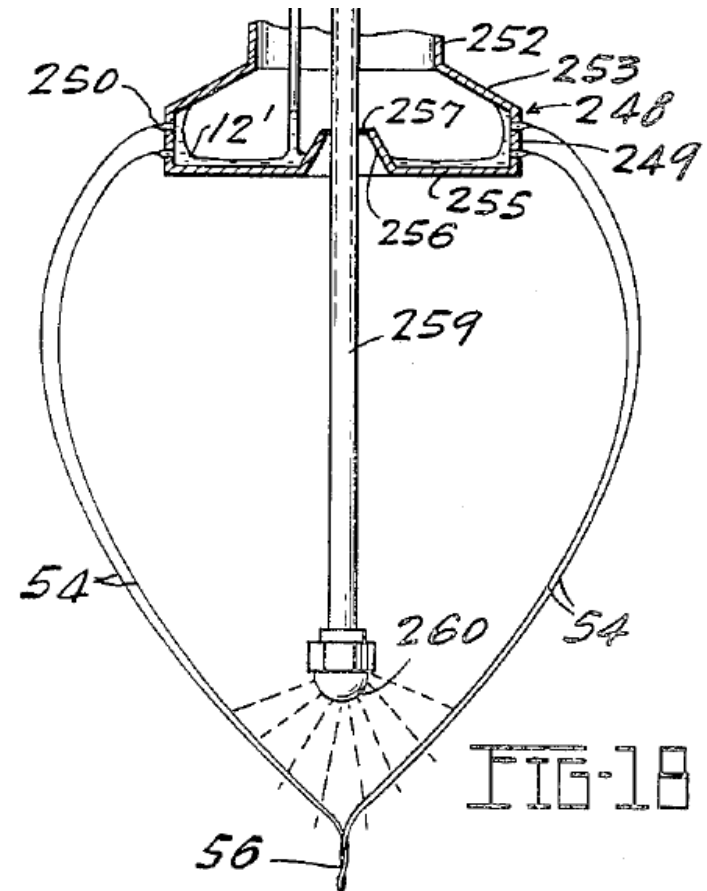
Small self-contained unit with batch feed on top, electrically heated, and a small CF bushing on the bottom



(US Patent 4,262,158)

## Rotary Continuous Filament

Using a rotary disc as a rotating bushing to collect twisted continuous filament strands



(US Patent 3,250,602)



# Other Methods & Variations

(with varying degrees of practicality)



## Hollow Fibers

### Continuous filament

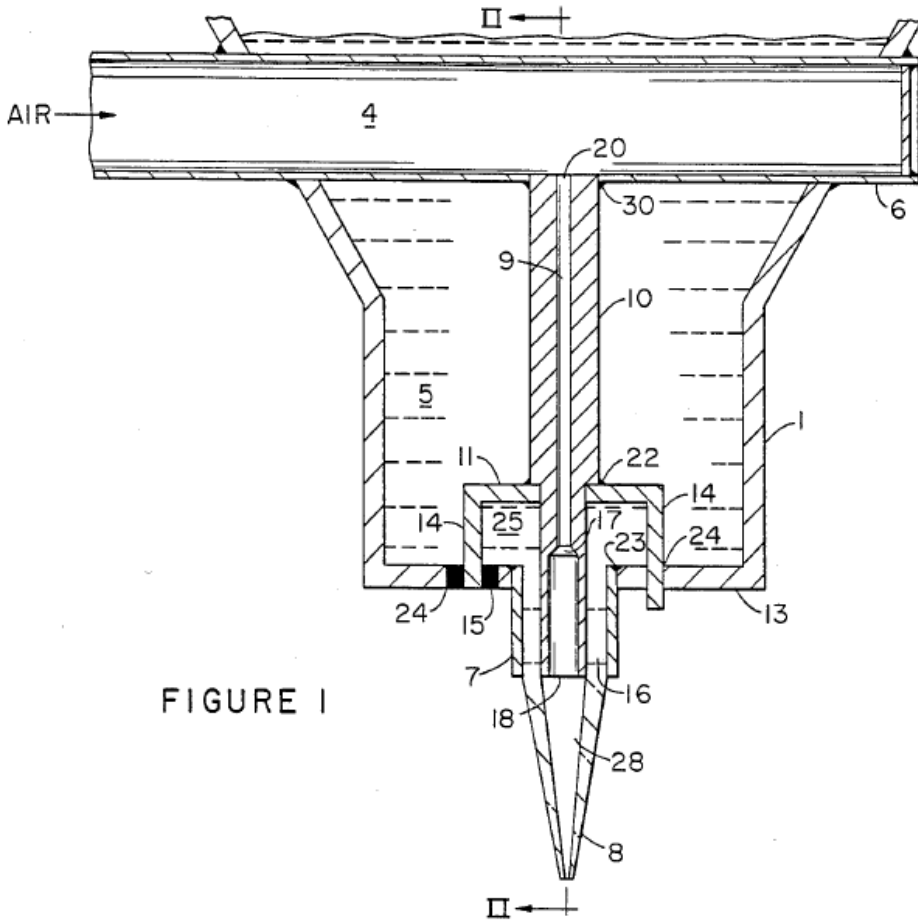
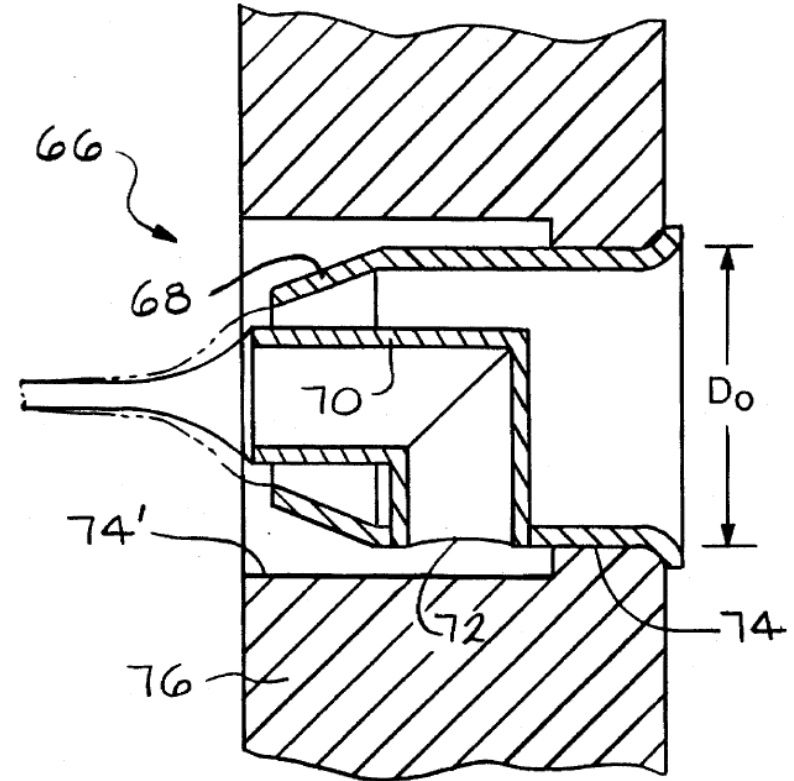


FIGURE I

(US Patent 4,758,259)

### Rotary



(US Patent 5,674,307)

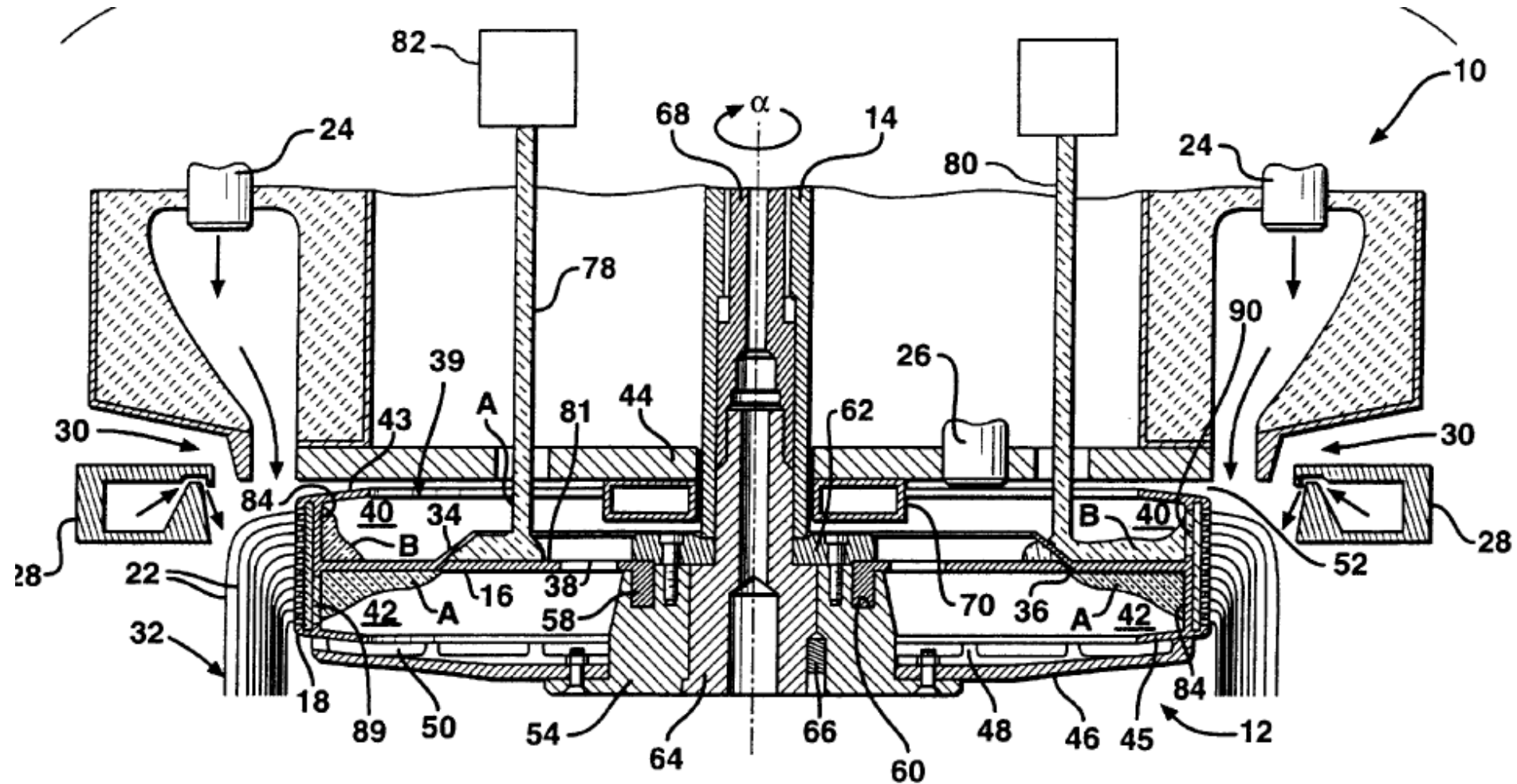
# Other Methods & Variations



(with varying degrees of practicality)

## Bi-Component Fibers

Rotary process

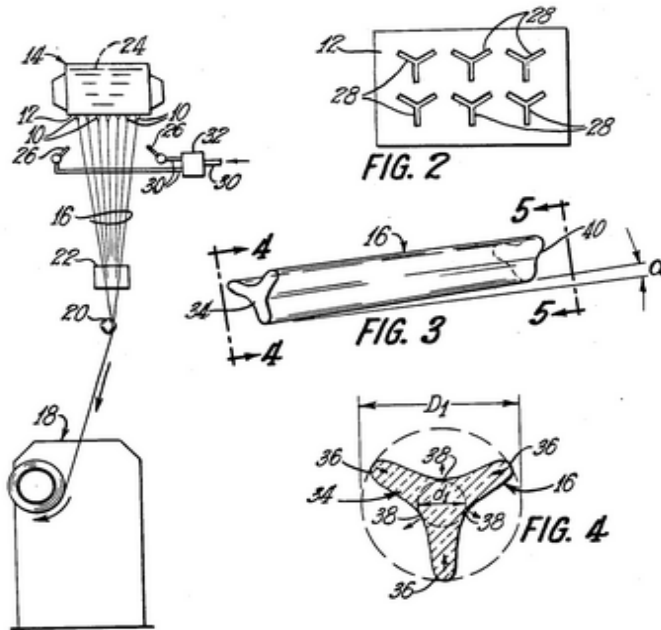


(US Patent 6,167,729)

# Other Methods & Variations



- Basalt continuous filament (low throughput, small bushings)
- Rotary “Mineral Wool” (low throughput, high energy, extremes for disc metallurgy)
- Sliver process (large drum, collection blade)
- Silica glass fiber (acid leach, draw from pre-form, sol-gel spun)
- Steam blown mineral wool (predecessor to cascade process)
- Non-round fiber cross section (oval or tri-lobed)



(US Patent 4,666,485)

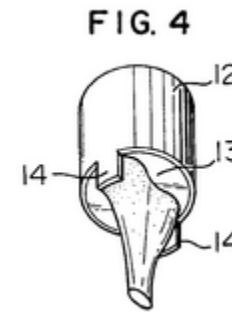


FIG. 4

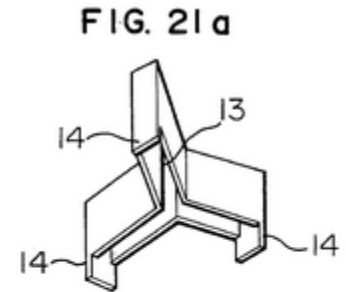


FIG. 21a

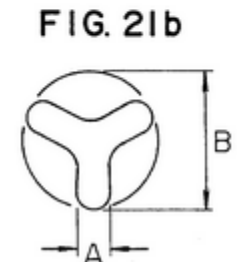


FIG. 21b

(US Patent 5,462,571)

There are many variations on these methods (lots of strange and interesting patents)

# References and Suggested Reading



## **Books**

J. G. Mohr and W. P. Rowe, *Fiber Glass*, Van Nostrand Reinhold Company, New York, 1978

F. T. Wallenberger and P. A. Bingham (Editors), *Fiberglass and Glass Technology*, Springer, New York, 2010

K. L. Loewenstein, *The Manufacturing Technology of Continuous Glass Fibres*, Elsevier, 1993

## **Articles**

F. T. Wallenberger, J. C. Watson, and H. Li, "Fiber Glass", ASM Handbook, Vol. 21: Composites (#06781G), 2001

S. Rekhson et al, "Attenuation and Breakage in the Continuous Glass Fiber Drawing Process", *Ceramic Engineering & Science Proceedings*, vol. 25, No. 1, page 179

S. Krishnan and L. R. Glicksman, "A Two-Dimensional Analysis of a Heated Jet at Low Reynolds Numbers", J. Am. Soc. Mech. Engineers, vol. 35D, page 355, 1971

## **US Patents**

Continuous Filament

7,980,099

5,017,205

7,732,047

Wool (Discontinuous)

8,250,884

4,451,276

5,356,450

**Thanks for your attention!**



**Questions?**

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Lectures available at:  
**[www.lehigh.edu/imi](http://www.lehigh.edu/imi)**