ELECTROSPINNING OF NANOFIBER MATS

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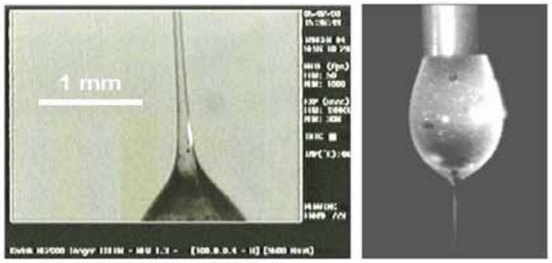
BACKGROUND

Electrospinning is a process where continuous polymer fibers with diameters in the sub-micron range are produced through the action of an applied electric field imposed on a polymer solution. Electrospun fibers have a greatly reduced fiber diameter and a high surface area to volume ratio compared to current non-woven techniques.



High Speed images of e-spinning

APPLICATIONS

The low fiber diameter (50-500 nm)* makes them excellent candidates for use as transparent tougheners in optical resins.
Other possible uses include scaffolding for tissue engineering, and nanoelectronics, due to their high (10x-100x) surface area to volume ratio

Material	Diameter (nm)
Typical Non-wovens	$1000 - 5\times10^{-5}$
Electrospun Fibers	50 – 500*
Visible Light	400 – 700

* Deitzel, J. M., et al., *Polymer* 42 (1): 261 (Jan 2001)

OBJECTIVES

1. Formulation of consistent process control parameters of electrospinning for future use in large scale production
2. Microscopic analysis of the fiber mat to determine the effect of solution composition on fiber morphology.
3. Determination of the optical properties of resin infused fiber mats.

APPARATUS

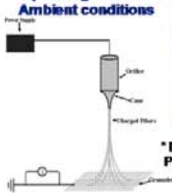
Electrospinning apparatus

MATERIALS USED

Solution composition	• 85/15 and 75/25 of THF/DMF • 5% w/w Urethane	•Pure formic acid •5% w/w Nylon 6,6
Reason for solvent	• THF has high volatility • DMF has low volatility	•Formic acid will not damage the fiber
Reason for fiber	• Urethane is an elastomer. • It is frequently used as a tougheuer.	•Nylon has a highly ordered crystalline structure. •Limited data exist for transparent nylon.

SPINNING PARAMETERS

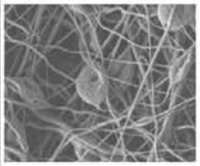
Parameters Studied
Tip to target distance
Ambient conditions



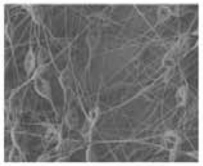
* Deitzel, JM and et al : POLYMER 42: (1) 261 JAN 2001

Average Temperature	Average Humidity	Needle Distance	Current
71 °F ± 2.1	66 % ± 4.2	7 cm	7-8 KV

NANOFIBER MORPHOLOGY



3000x SEM picture of 85/15 spun Urethane
Fiber diameters are not uniform.
Beads of varying size and shape are present in this sample.



3000x SEM picture of 75/25 spun Urethane
Fiber diameters appear smaller and more uniform that the 85/15 solution.
Beads are smaller, yet more are present and are generally of uniform shape.

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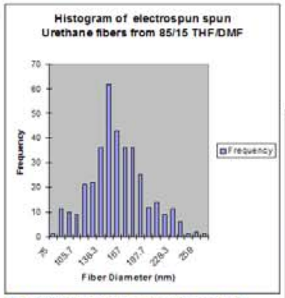
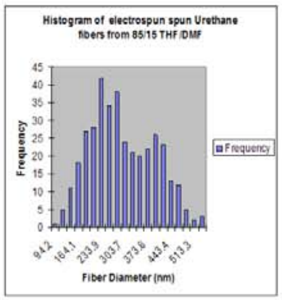
NANOFIBER MORPHOLOGY



The 75/25 solution is evenly distributed



A possible bi-modal distribution exists here

OBSERVATIONS FROM ELECTROSPINNING PROCESS

All prepared samples were examined using Scanning Electron Microscopy.
Measurements of fiber diameters were recorded to help determine the morphology of fibers based on solution composition.
Bead morphology occurred in the Urethane fibers, but not in the Nylon fibers.

Sample	Diameter (nm)	95% C.I. (nm)	Skew	Bead Area (μm^2)	Bead Propensity (Bead/324.5cm^2) *
85/15	285 ± 96.2	192	0.306	44.2 ± 17.7	8 ± 2
75/25	153 ± 35.7	71.4	0.345	16.5 ± 2.85	40 ± 6
Nylon	174 ± 32.5	65.0	0.513	na	0

* 324.5 cm^2 is the area of the SEM picture at 3000x

OBSERVATIONS FROM ELECTROSPINNING PROCESS

Dust in the fume hood can be caught within the fiber mat, which can cause contamination.
The density of the fiber mat is greatest perpendicular to the point below the needle, but decreases away from that point.
The bead morphology structure is prevalent throughout the urethane fiber mats, and is influenced by solution composition.

RESIN INFUSION

In order for the composite to be transparent, the fiber mat must be completely wetted.
One-square-inch samples of fiber were coated in resin and placed between two Frekote-coated glass slides.
Three methods were used during the curing period

Method Used	Vacuum Applied	Pressure used	Post Cure
First method	Yes	No	Yes (130°C for 2h)
Second method	Yes	Yes (5 lbs)	Yes (130°C for 1h)
Third method	No	Yes (30 lbs)	No

VINYL ESTER RESULTS

First Method
- Post curing caused a yellowing of the resin
- A large number of voids are present in the system

Second Method
- The fiber mat did not wet completely, causing small dry spots.
- Placing the sample in a vacuum did not remove all of the voids.

Third Method
- Small dry spots are in the fiber matrix
- Using pressure seems to force out most of the voids.
- Dust is present in the resin

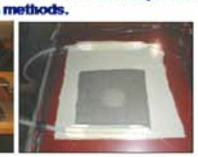
CONCLUSIONS

- Fiber diameter and bead morphology can be related to the composition of the solvent used in the electrospinning process.
- All three methods provide some degree of transparency.
- Voids are present in the composite matrix, which may be caused by air trapped in the fiber mat itself.
- The gel time of Vinyl Ester is too short to allow the samples to remain in vacuum for extended periods.
- The varying thickness of the fiber mats do not allow the resin to infuse completely into the fiber mat.
- Extreme post curing of the samples can cause discoloration of the composite.

FUTURE WORK

- Designing a clean environment in which to spin
- Control of the thickness and quality of the fiber mats.
- Elimination of voids in the composite.
- Mechanical and transmission tests of the composites
- Scaling up of production methods.

Drum method



Micro-VARTM

ACKNOWLEDGEMENTS

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