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Innovation and Development of Study Field Nanomaterials at the Technical University of Liberec

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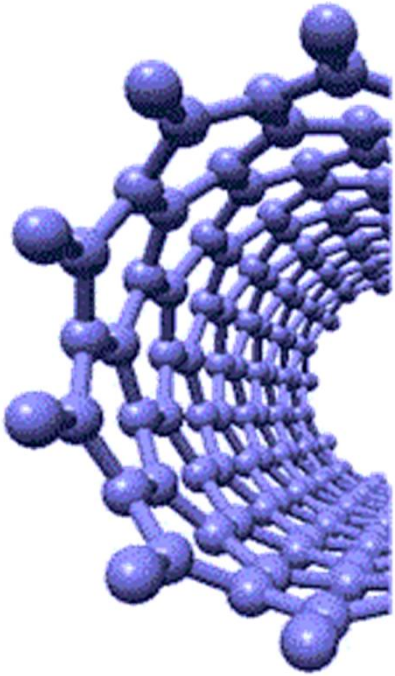

These materials have been developed within the ESF project: Innovation and development of study field Nanomaterials at the Technical University of Liberec

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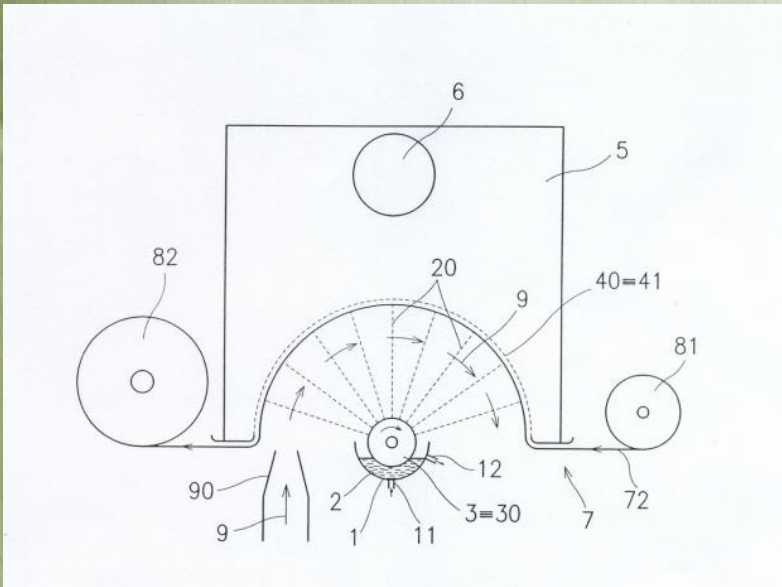
Testing of filtration properties of polyimide nanofibre layers

NANOSPIDER™ technology



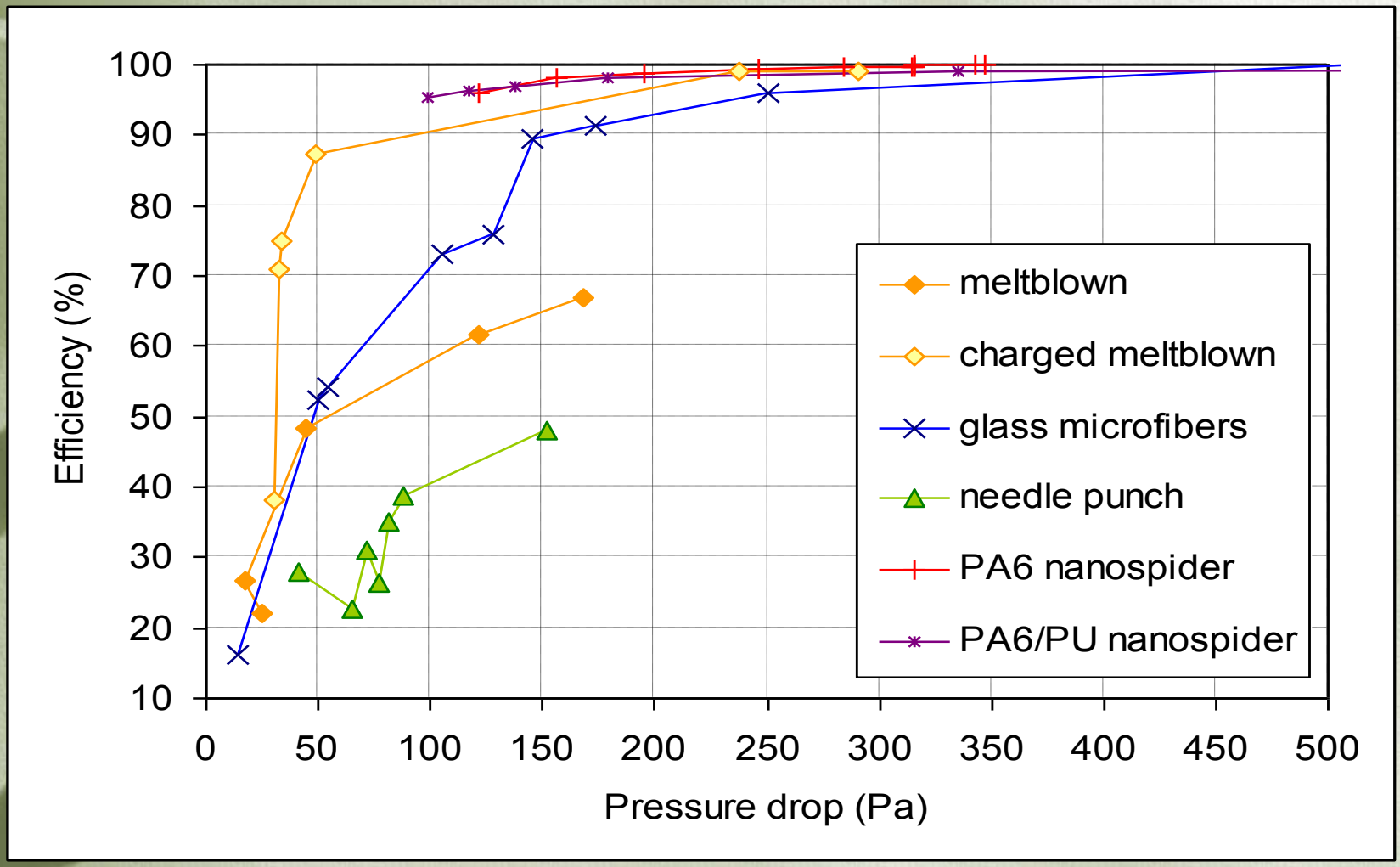
- Electrospinning from the free liquid surface
- Multiple jet production

Protection of know-how

- September 9, 2003
  Patent PS3386CZ
- September 8, 2004
  PCT/CZ2004/000056
- March 17, 2005
  WO2005/024101

Nanofiber applications

Air filtration



Electrospinning of PI nanofibres

Polyimides

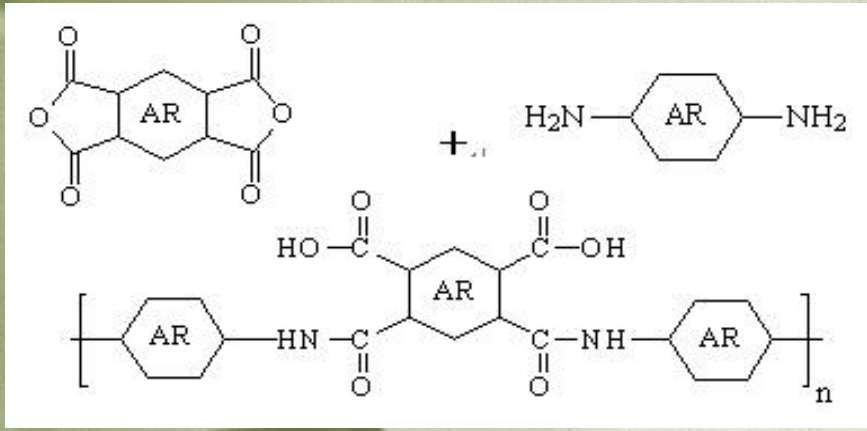


Fig.: Synthesis of the polyamic acid (PAA)

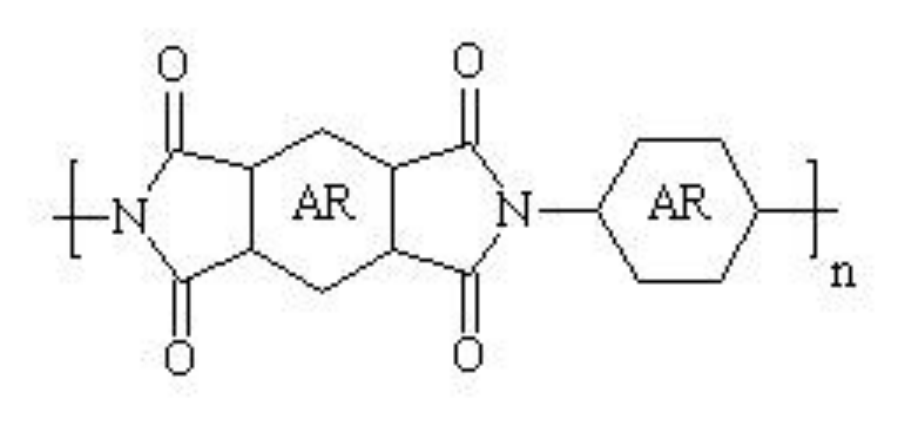


Fig.: Structural unit of aromatic polyimide

Properties of PI

- Thermoset polymers
- Good thermal stability and chemical resistance
- Flame retardant
- High tensile modulus

Electrospinning of PI nanofibres

Preparation procedure

- Preparation of polyamic acid ((PAA) precursor) solution
- Electrospinning of precursor solution
- Bonding of the nanofiber sheet with carrier layer
- Imidization

Testing of filtration material

- SEM control (fibre diameters, defects)
- Thermal stability tests (short and long time stability)
- Filtration efficiency and pressure drop

Electrospinning of PI nanofibres

Dissolving of PAA

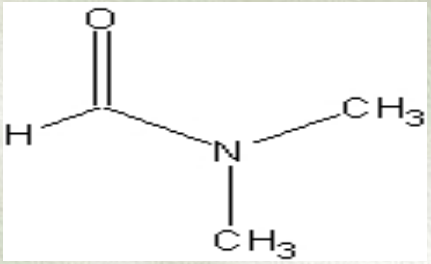


Fig.: N,N-dimethyl formamide

Solvent: N,N-dimethyl formamide

- Testing of various solution concentration (optimization of electrospinning process)

Approximate concentration range: 20-23 % w/w (solution viscosity affects morphology of produced nanofibers)

Imidization

- Sequenced heat treatment
  - 60° C - 12 hours
  - 100° C - 1 hour
  - 150° C - 1 hour
  - 200° C - 1 hour
  - 250° C - 1 hour

Preparation of composite nanofiber filters

Bonding of nanofiber layer

Fig.: Tests of bonding of PI nanofiber layer on the glass fabric substrate

- Poor mechanical properties of nanofiber membrane
  (small fibre diameters, low area weight)
- Composite structure with glass woven fabric provides better mechanical performance

- Self-adhesion (pressure activated)
  - for single use applications, insufficient for long period filtration
- Cerambind adhesive
  - ceramic adhesive for high temperatures
  - chemically affects un-imidized nanofiber layers (technology complication)

Testing of composite nanofiber filtration material

Thermal stability

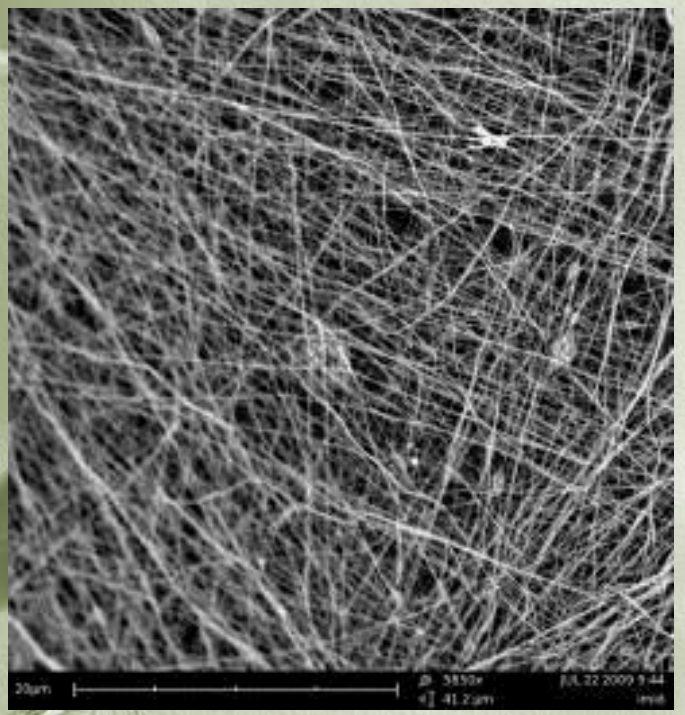


Fig.: PI Nanofibre layer after 4h at 265° C

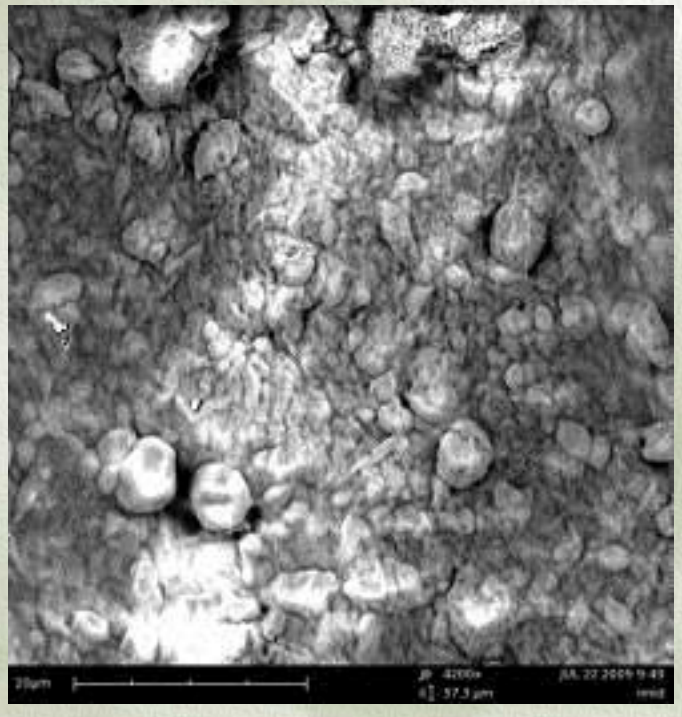


Fig.: PI Nanofibre layer after 4h at 300° C

- Long term test confirmed stability of the PI nanofiber layer for 16h at 265°C (SEM evaluation)

Testing of composite nanofiber filtration material

Filtration properties

Tested properties:

- **Filtration efficiency**:

$$E = m_1/m_2 \,.\, 100$$

(1), where m_1 is the amount of captured particles and m_2 is the amount of all particles upstream the filter.

- **Pressure drop:**

$$p = p_1 - p_2$$

(2), where p_1 is pressure upstream the filter and p_2 pressure downstream the filter.

Testing of composite nanofiber filtration material

Filtration properties

- Test method specified for the high efficiency filters
  - NaCl aerosol particles with the mean size 0,6. 10^{-6} m
  - All tests made according to the British Standard BS 4400

Samples code	PI solvent concentration	Imidization	Pressure bonding activation (better fixation on the textile substrate)
21,5NN	21,5	no	no
21,5YN	21,5	yes	no
21,5YY	21,5	yes	yes
22NN	22	no	no
22YN	22	yes	no
22YY	22	yes	yes
22,5NN	22,5	no	no
22,5YN	22,5	yes	no
22,5YY	22,5	yes	yes

***Table 1**: Description of tested samples.*

Corresponding standards	BS 4400 (Method for sodium chloride particulate test for respirator filters)
	EN 143 (Respiratory protective devices – Particle filters – Requirements – testing – marking)
Filtration test area	100 cm^2
Filtration face velocity	5 m/min
Filtration flow	50 l/min
Particle concentration	up to 13 mg / m^3
Size of particles	up to 2 μm, mean value 0,65 μm

***Table 2:** Parameters of filtration test*

Testing of composite nanofiber filtration material

Filtration properties

Sample code	Filter efficiency (%)	Filter pressure drop (Pa)	Assumption of nanofiber layer pressure drop (Pa)*
21,5NN	91,65	493	132
21,5YN	88,2	677	316
21,5N	**90,54**	**639**	**278**
22NN	95,9	531	170
22SN	96,02	705	344
22N	**97,18**	**635**	**274**
22,5NN	84,32	444	88,6
22,5SN	92,06	740	379
22,5N	**95,36**	**628**	**267**
Glass woven substrate only	13,5	361	

***Table 3**: Filtration properties of tested samples*

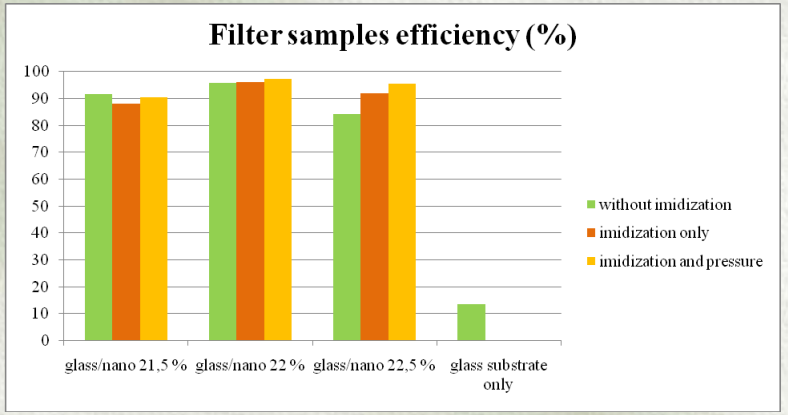


Fig.: Filter efficiency of tested samples

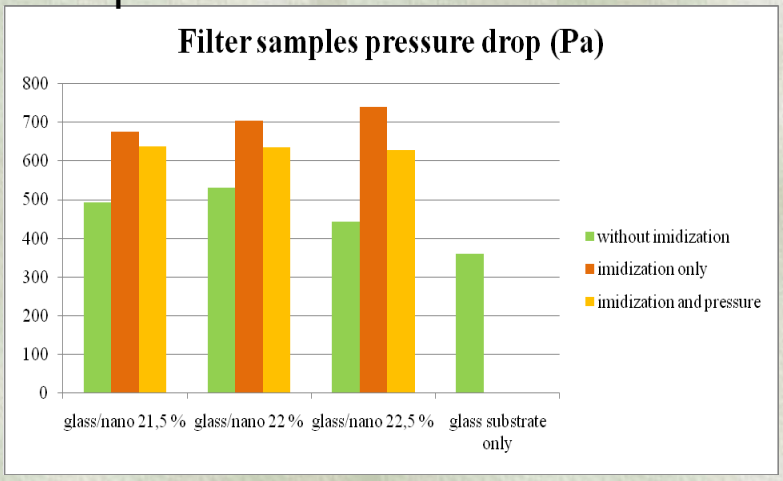


Fig. 2: Filter pressure drop of tested samples

Conclusions

- PI nanofibres are applicable for high temperature, high efficiency filtration

- Long-term thermal stability of PI nanofiber layer is confined by 265° C
  - due to small dimensions of nanofibres
- The best filtration properties reached by electrospinning of the 22 % w/w concentration of PAA solution
  - the filter efficiency is the highest and pressure drop is reasonably low.
- After imidization process
  - the filter efficiency is changing slightly
  - the pressure drop rapidly increases.
  - nevertheless the imidization process is essential for the stability of the PI nanofiber layer