

# Institute of Photonics and Electronics v.v.i. (www.ufe.cz) Technology of Optical Fibers FILANO team

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# Ústav fotoniky a elektroniky AV ČR, v.v.i.







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#### ZÁKLADNÍ VÝZKUM:

fotonika

- vláknové lasery a zesilovače, optická vlákna
- optické biosenzory
- státní etalon času, detekce pole živých buněk

100 FTE



# Outline

Intro

Optical fibers

**Technologies**Preform preparation – MCVD & others silica properties – MCVD vers. conventional fiber drawing

ApplicationTelecommunications, fiber lasers,<br/>amplifiers, sensors

Summary LABO

MCVD, fiber drawing, sol-gel, magnetron sputtering



# **Optical fiber**

Optical fiber : dielectric structure, L<< r, n<sub>core</sub> > n<sub>clad</sub> [W. Snell +1626, J. Tyndall +1893] attenuation, dispersion

**Optical losses in optical fibers** 

- trasparency of 3 mm of window-glass  $\approx$  2 km of optical fiber



## **Purity of material**



- 1. Per Analysis PA (99 99,5 %)
- 2. Semiconductor PP (99,9995 %)
- **3.** Ultra-pure FO Optipur / for trace analysis [ppb]

 $\% - 10^{-2}$ 

ppm – 10<sup>-6</sup> (parts per million)

ppb – 10<sup>-9</sup> (parts per billion) : content of impurities acceptable in FO Optipur materials Ultra-pure technologies - CVD !



# **Optical fiber preparation**







# Ultra-pure technologies CVD - Chemical Vapor Deposition

#### A(g) + B(g) = AB(s)



Goal : starting materials (g) or (l) can be purified (E.g. distilled)



## **Preform preparation**

#### **MCVD – (Modified) Chemical Vapor Deposition**



- Sequential sintering of thin glassy layers (of thickness 1-20 µm) onto inner wall of silica substrate resulting in bulk material – preform [S. R. Nagel, 1982]
- high purity (~ 10<sup>1</sup> ppb) high preciseness (better than 1 %)



# **MCVD process model**

#### **1. Vaporization of starting materials**

• 
$$V_{XCI4} = V_{Ox} * P_{XCI4}^{o} / (P - P_{XCI4}^{o}) \dots$$
 boiling point SiCl<sub>4</sub>=56°C

#### 2. Oxidation

- 1<sup>st</sup> -order kinetics, t = 0.02 s
- Chemical equilibrium :
- SiCl<sub>4</sub> (g) + O<sub>2</sub>  $\rightarrow$  SiO<sub>2</sub> (s) + 2Cl<sub>2</sub>

conversion ~ 0.95 – 0.99 (1500 °C)

• GeCl<sub>4</sub> (g) + O<sub>2</sub>  $\rightarrow$  GeO<sub>2</sub> (s) + 2Cl<sub>2</sub>

conversion ~0.5 – 0.6 (1600 °C), f(t,  $x_{SiCl4}/x_{GeCl4}$ )

#### 3. Deposition



#### **MCVD process model**



Temperature field during deposition





# **MCVD process model**

#### **Process parameters :**

#### Variable :

- flow rates (Si, Ge, P, B, F, Ox ...)
- deposition temperature

#### Adjustable :

- temperature of starting materials (liquids)
- burner speed
- pressure
- rotation speed of the substrate tube
- substrate tube dimensions

[McChesney and Nagel, 1982, Wood, 1987, Kirchhof, 1986]



#### **MCVD output parameters**



# Tomography of the refractive-index profile of preform

- High purity material due to FO-Optipur purity starting materials.
- High quenching rate ranging from 10<sup>2</sup> to 10<sup>3</sup> °C/s !



# **MCVD - doping of silica**



[A.B. Chynoweth, 1979, M. Shimizu, 1986, Y. Ohmori, 1983, S. H. Wemple, 1973, H. Wehr 1986, I. Kasik, 2005, K. Sanada, 1980, M. M. Karim 1994]



#### **Other CVD technologies (ultra-pure)**





#### **Other technologies : sol-gel**



#### No melting, disorder imprinted.

[J. McKenzie (US), J. B. McChesney, 1997, A. Pope (US), 1993, M. Guglielmi (It), J. Livage (F), R. Almeida (P), S. Ribeiro (Br), B. McCraith (Ir), J. Brinker (US), S. Sakka (J), V. Matejec & J.Mrazek (CZ)]



# Comparison

#### **CVD** (Chemical)

# **x PVD** (Physical)

MCVD OVD etc. DC magnetron sputtering vacuum evaporation etc.

#### Layer thickness

1 – 10<sup>1</sup> µm

1 - 10<sup>1</sup> **nm** 

(however, both are reported as "thin layers")

# **Deposition rate**

HIGH

LOW

#### Products



Layers only



#### Comparison x co

## conventional

#### **Starting materials**

gaseous (g) or liquid (l)

(M)CVD

melting point of oxides different

(s) solid state melting point comparable

#### **Purification methods**

distillation

recrystallisation, remelting









**Annealing ~ 1120-1180** °**C** [www.Heraeus, M. B. Volf, 1987, A. B. Chynoweth, 1979, M. Ohashi, 1992, O. V. Mazurin, 1980, K. Shiraki, 1993]





[A. B. Chynoweth, 1979, O. V. Mazurin, 1980, S. H. Wemple, 1973]



# Properties(M)CVDxconventional

#### **Optical properties - transparency**



Dependence of absorption at UV on technology of silica production [Safibra, 2010 & M. B. Volf, 1987]



# Properties(M)CVDxconventional

#### **Optical properties – refractive index**



 $n_{633 \text{ nm}, 20 ^{\circ}\text{C}} = 1.457$  $n_{10.6 \,\mu\text{m}, 20 ^{\circ}\text{C}} < 1$  [www.heraeus.de]

1.48 < n < 1.95 [www.schott.com]



# Comparison : GLASS (M)CVD x conventional

**GLASS** : solid state material, amorphous, usually produced by quenching of melt, in glassy state (stable below  $T_q$ ) [Hlaváč, 1981]

#### **Structure**

amorphous

amorphous

short-distance order (< 1 nm) longer-distance disorder (>1 nm) no X-ray crystallographic signal nano-structure imprint feasible (glass/glassceramics)

#### **Production - kinetics**

sintering + melting & quenching ~10<sup>2</sup> -10<sup>3</sup> °C/s melting + quenching ~10°C/min

**VŠCHT – ÚSK, 2013** 

[G. Tammann, 1933]





- higher porosity •
- lower density •

Temperature **VŠCHT – ÚSK, 2013** 

Transformation

area

**Crystal** 



# **Drawing of optical fibers**



# **Comparison of fibers**

### optical

#### X

# soft-glass

**SILICA** (doped with  $GeO_2$ ,  $P_2O_5$ ,  $B_2O_3$  ...) High productivity (relative)

- Purity FO Optipur grade
- Chemical durability
- Precise geometry (<0.5 μm)</li>
- Low optical loss ~ 0.2 dB/km
- Strength ~ 5 GPa due to coating
- Use in **OPTICS**

• (CaO, MgO)-Al<sub>2</sub>O<sub>3</sub>-B<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub>

- $Na_2O CaO (Al_2O_3) SiO_2$
- High productivity low processing temperature, cheap starting materials
- Thermo-insulating properties
- Chemical durability
- A geometry allowing weaving
- Strength ~ 2.5 GPa (without coating, temporary)
- Use as insulators and textiles



#### **Application**





### **Telecommunications**



Testing of 200 km telecom line





#### SM 1300, 1550 nm

GI - technology transfer VÚSU Teplice, Hesfibel TR

1981 – 1<sup>st</sup> demonstration of CZ optical fiber – UFE/URE/JLS



# Telecommunications : fiber lasers and amplifiers





#### **High power fiber lasers**



Intensity of light Sun 63 MW/m2 1W-fiber laser 12.7 GW/m2



Welding, cutting < 2kW savings, fast process

Er- fiber laser, pulsed 197 fs, 5m resonator Liekki





### **Fiber-optic sensors**

Small devices capable of continuous and reversible monitoring of (bio)chemical species and their concentration



Principle : change of properties of the light due to chemical (physical) changes of medium.



#### **SUMMARY**

#### MCVD

#### Suitable for the preparation of :

- silica-based materials, doped (up to 50 mol%)
- **few-component** (up to ~ 6 components) materials
- materials for photonics, optics, optoelectronics
- materials of high-level purity (~ 10<sup>1</sup> ppb)
- products requiring high preciseness of geometry (better 1 %)



# **SUMMARY**

- 1. OF technology : preparation of structures of high preciseness from materials of ultra-high purity (impurities in ppbs only).
- 2. OF preparation in two steps : preform preparation and fiber drawing.

(M)CVD technique (preform) makes possible to prepare multilayered tailored structures of suitable level of purity.

- 3. Fibers conventional (passive) and specialty (active).
- 4. Research of optical fibers (CR) :





## References

- **J. M. Senior** : Optical fiber communications Principle and practise, Pearson Education Limited, Harlow, England, 2009.
- A. Mendez, F.T. Morse : Specialty optical fibers handbook,

Elsevier Science & Technol, USA, 2006.

- J. Schrofel, K. Novotný : Optické vlnovody, SNTL, 1986
- **Saaleh,** Fotonika (1 4), Matfyzpres
- S. R. Nagel, J. B. McChesney, K. L. Walker : An overview of the MCVD process and performance, IEEE J. Quantum Electron. QE-18 (1982) 459-477
- Československý časopis pro fyziku 1/2010, 4-5/2010, 1/2011
- Jemná mechanika a optika 55 (2010)
- Sdělovací technika 3/2011



# Uplatnění v oboru



UCHP - Oddělení aerosolových a laserových studií : Laserová ablační příprava nanostrukturovaných prášků, RNDr. Vladislav Dřínek, CSc., PROJEKT MAGISTERSKÉHO/BAKALÁŘKÉHO STUDIA

UCHP - Oddělení aerosolových a laserových studií : Laserová ablační příprava nanostrukturovaných prášků v kryogenních atmosférách RNDr. Vladislav Dřínek, CSc., PROJEKT DOKTORSKÉHO STUDIA

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