ELSEVIER

Journal of Chromatography A, 716 (1995) 135–139

JOURNAL OF
CHROMATOGRAPHY A

# Integrated optical detection cell based on Bragg reflecting waveguides

Thomas Delonge, Henning Fouckhardt*

*Technische Universität Braunschweig, Institut für Hochfrequenztechnik, Abt. Optoelektronik, Postfach 3323, D-38023 Braunschweig, Germany*

**Abstract**

In modern capillary electrophoresis systems with optical detection, the light beam is usually transmitted through the capillary crosswise. The disadvantages of this system are the small absorbent path length and the large amount of stray light. A new concept for a planar integrated optical detection cell with light guided in the liquid a defined length between 1 mm and 1 cm along the capillary is proposed. Using ARROWs (anti-resonant reflecting optical waveguides) or high-reflecting $TiO_2$–$SiO_2$ layers (Bragg layers), low-loss waveguiding can be established with confinement of the wave energy within the liquid exceeding 95%. Advantages of this system are an increased path length and a high signal-to-noise ratio while maintaining small inner dimensions below 20 $\mu$m × 20 $\mu$m. The stray light may be easily suppressed by masks on either side of the light path, which can be used as input slits for a spectrometer.

## 1. Introduction

Modern analytical systems use capillaries with an inner diameter of about 50–100 $\mu$m. Thinner capillaries promise an increased detection speed, better selectivity and smaller amounts of both solution and solvent. In most available capillary systems, the light is transmitted through the capillary crosswise. The light is either monochromatic for single-wavelength detection or emitted from a broadband source for spectral detection with a typical wavelength range from 190 to 600 nm. In order to have sufficient transparency for short UV wavelengths, the capillary material is usually fused silica ($SiO_2$).

The major disadvantages of this set-up are the short path length which is given by the inner diameter of the capillary and the large amount of "false" or "stray" light (Fig. 1), i.e. light going through the capillary wall instead of the fluid and entering the detecting element. This light decreases the signal-to-noise ratio and the dynamic range of the detector.

The attributes of a new sensor should be an increased path length and an effective reduction

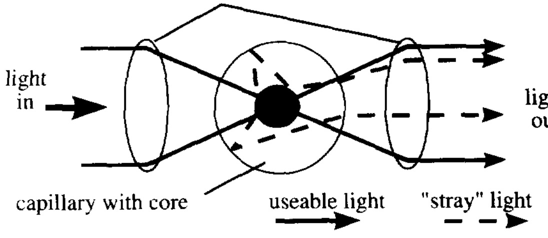

Fig. 1. Standard cross-capillary detection: disadvantages are a short path length and a large amount of stray light.

---

* Corresponding author.

*SSDI* 0021-9673(95)00611-7

of false light while maintaining a small inner diameter.

## 2. Standard approaches

One commercially used approach to increasing the path length is to widen the capillary locally to a bubble (Hewlett-Packard). This widening is limited to three to five times the inner diameter of the capillary and the problem of the false light remains.

Another solution is to guide the light within the solvent in the direction of the column by normal Fresnel reflections [1,2]. However, this leads to significant losses. A further approach might be to guide the light in the capillary wall by total internal reflection at the wall/analyte interface. The interaction between the light and the analyte takes place only by the small evanescent fields extending into the liquid. This requires long interaction lengths. Yet another solution would be to guide the light within the liquid along the capillary, but with a low-loss waveguiding mechanism. For chromatographic detection the analyt is usually dissolved in water; this aqueous medium has a lower refractive index than fused silica. To achieve total reflection, either a solvent with a higher refractive index than the material of the capillary (e.g., salt solutions with Teflon PFA or PFE) or coating of the inner wall of the capillary with a low-index material can be chosen.

In the first case, there are two disadvantages: the aqueous salt solution is undesirable in many analytical measurements and the Teflon materials mentioned above are not sufficiently transparent at shorter UV wavelengths. In the second case, the coating material used is an amorphous fluoropolymer, Teflon AF, which has a lower refractive index than the aqueous solution. The disadvantage of this system is a high loss at lower wavelengths between 190 and 230 nm.

In the following, we present a waveguiding concept for analytical applications which ensures low-loss waveguiding, but allows the application of capillaries with walls having a higher refractive index than the liquid.

## 3. ARROW–Bragg waveguiding

The principle of the Bragg waveguides [3,4] is a reflection at the interfaces of layers with alternating higher and lower refractive indices, which bound the actual core of the waveguide. The refractive indices of the layers may be lower than, equal to or even higher than the refractive index of the core.

The design of the thicknesses of the layers can be carried out with standard methods such as the transfer-matrix method [5] combined with optimization algorithms. Using the transfer-matrix method, the complex propagation constant of the eigenmodes of a planar system with arbitrarily stacked layers can be calculated. In first order, the real part of the propagation constant determines the field and intensity profile and the imaginary part of the propagation constant determines the attenuation related to the propagation length. With this information, the transmission of the examined structure can be calculated.

A special class of Bragg waveguides are the anti-resonant reflecting optical waveguides (ARROWs) [6,7]. The principle of the ARROW is shown in Fig. 2. In the case of the Bragg reflecting waveguide, the core with the lowest refractive index of the structure is surrounded by

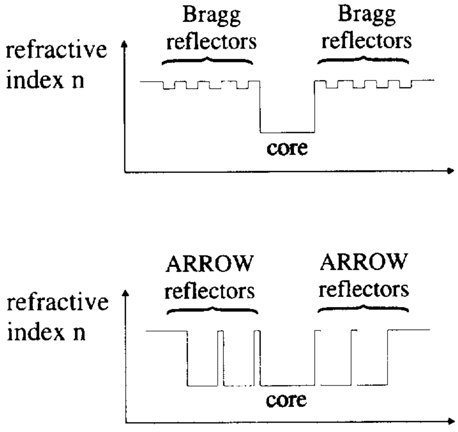

Fig. 2. Principles of Bragg and ARROW waveguides. Top: Bragg waveguides on both sides of the core made of layers with alternating higher and lower refractive indices. Bottom: ARROW waveguide with even-numbered reflectors on both sides of the core having a refractive index comparable to the refractive index of the core.

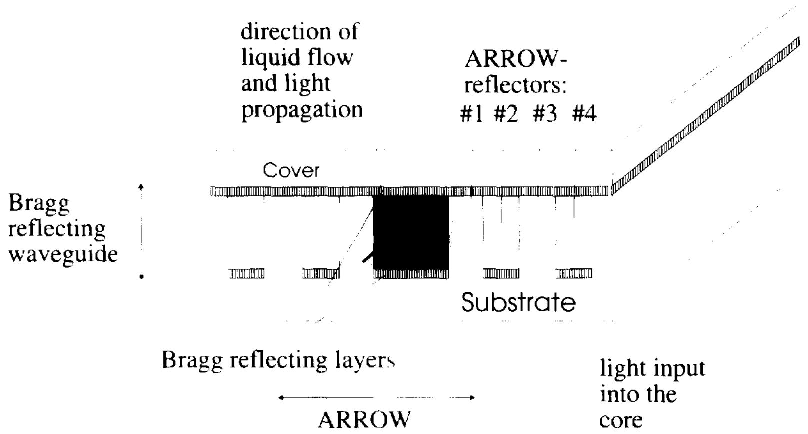

Fig. 3. Integrated optical detection cell as a combined Bragg–ARROW waveguide. In the horizontal direction the waveguiding is established by an ARROW structure; in the vertical direction Bragg layers are used.

thin layers of media with alternating higher and lower refractive indices. All reflection layers have a higher refractive index than the core. Here we use pairs of layers made of $TiO_2$ and $SiO_2$. The refractive indices of the materials are wavelength dependent. For the considered wavelength region between 180 and 600 nm, the refractive indices lie in the ranges $n = 1.7–4.3$ ($TiO_2$) and $n = 1.45–1.58$ ($SiO_2$). We stress, however, that any material which can be deposited on the substrate can be used. The substrate itself should be transparent for high coupling of the light into the core.

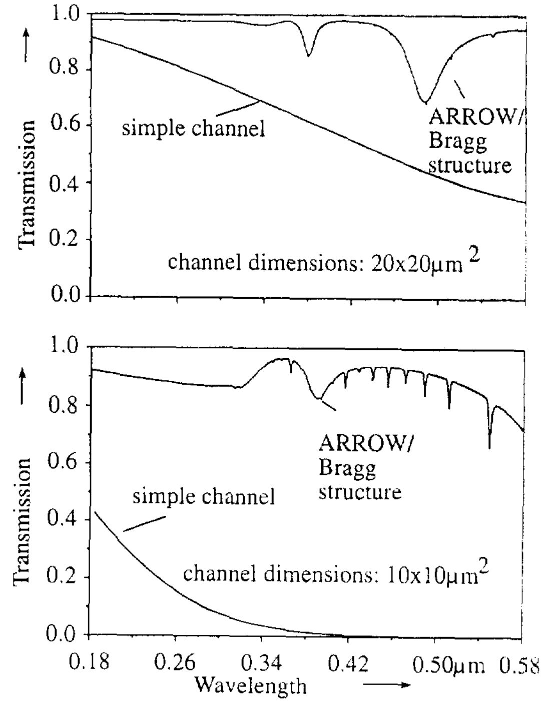

Fig. 4. Transmission of the combined ARROW–Bragg structure compared with a simple channel with cross-sectional dimensions of 20 μm × 20 μm and 10 μm × 10 μm. The absorbent path length is 1 cm.

The thicknesses of the Bragg reflecting layers depend on the core width, the refractive indices and the wavelength range. The principle of the ARROW is based on reflecting the light back into the core by specially designed pairs of coupled Fabry–Perot reflectors. The first reflector, turned to the core, consists of an arbitrary material with a higher refractive index than that of the core. The even-numbered reflectors on both sides of the core have a refractive index comparable to that of the core. For the highest reflection (anti-resonance), the thickness of these reflectors should be an odd multiple of half the thickness of the core. Fig. 3 depicts a cross-sectional view of an integrated absorption sensor with a combined ARROW–Bragg reflecting waveguide. In the horizontal direction waveguiding is achieved by an ARROW structure and in the vertical direction by Bragg reflecting layers. The core is filled with the fluid to be analysed; the even-numbered ARROW reflectors at both sides of the core are filled with a fluid with a refractive index comparable to that of the core.

Fig. 4 gives the calculated transmission of the ARROW–Bragg structure and a simple channel (waveguiding by Fresnel reflections according to Manz et al. [1] and Verpoorte et al. [2]). The path length is 1 cm and the cross-sectional dimensions of the structure are 10 μm × 10 μm and 20 μm × 20 μm, respectively. The combined ARROW–Bragg structure offers a significantly higher transmission than the simple channel, especially at higher wavelengths and smaller dimensions. For cross-sectional dimensions of 20 μm × 20 μm the gain in transmission is up to a factor of 2. The difference between the transmission of the two concepts increases drastically when the dimensions are reduced further. The second part of Fig. 4 shows the calculated transmission for cross-sectional dimensions of 10 μm × 10 μm. The transmission of the simple channel is lower than 10% for wavelengths over 300 nm while the transmission of the combined ARROW–Bragg waveguide is higher than 70% over the whole wavelength region between 180 and 580 nm. At 350 nm the transmission is enhanced by a factor of 15, which leads to a significantly higher signal-to-noise ratio (by a factor of ca. 4 for this wavelength). The sharp dips in the transmission characteristic are related to the resonant states of the horizontal ARROW structure and can be suppressed by an appropriate calibration. Moreover, the stray light can be easily suppressed by masks on one or both sides of the end facets of the detection cell, which increases the signal-to-noise ratio and the dynamic range even further.

## 4. Fabrication

The described structure is well suited for planar integration on different kinds of substrates. The technology will be the same as used for microelectronics, micromechanics or integrated optics. Combined with hybrid integration

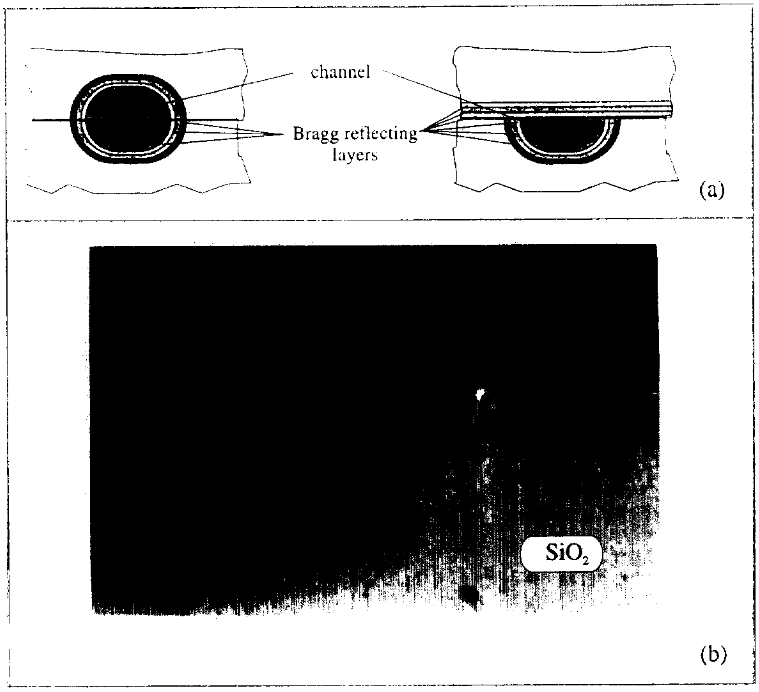

Fig. 5. (a) Alternative structures with waveguiding by Bragg reflecting layers in all dimensions and (b) photograph of a wet-chemically etched semi-cylindrical channel with a depth of 10 μm and a width of 30 μm.

of photodetectors, micromechanical pumps and valves, this structure opens the door to an analysis system on a single chip.

The channels in the substrate and cover are formed by standard processes, e.g., photolithographic structuring of resists spun on to the surface of the substrate with a subsequent wet, plasma or ion etching. The Bragg reflecting layers can be deposited by evaporation or, in the case of crystalline substrates, by epitaxial growth. The substrate and cover are fixed together by glueing or anodic bonding.

The horizontal ARROW structure requires a sophisticated structuring based on ion-etching techniques. A simpler method is wet chemical etching. Owing to the isotropic character of the fused silica, only (nearly) cylindrical-shaped channels are possible. In this case, the waveguiding has to be established by Bragg reflectors only. A typical structure is shown in Fig. 5 (in the photograph without Bragg layers). The etched substrate has a width of 30 $\mu$m and a depth of 10 $\mu$m. A channel can be formed either by using a symmetrical structure as superstrate or by using a simple plate as cover. The Bragg reflecting layers may be deposited by thermal evaporation of solids or chemical vapour deposition.

## 5. Conclusion

We have presented a new concept for an integrated-optical detection cell for capillary electrophoresis and liquid chromatography. The detection cell offers low-loss waveguiding within the analyte especially when small cross-sectional dimensions of the channel are desired. The concept is based on ARROW and/or Bragg reflecting structures, which are well suited for planar integration of a CE system "on a chip". Although the concept presented requires greater technological effort than standard cross-column detection, it promises a substantially higher sensitivity owing to the increased path length and the high transmission combined with reduced stray light.

## References

[1] A. Manz, D.J. Harrison, E. Verpoorte and H.M. Widmer, Adv. Chromatogr., 33 (1993) 1–65.

[2] E. Verpoorte, A. Manz, H. Lüdi, A.E. Bruno, F. Maystre, B. Kratinger, H.M. Widmer, B.H. van der Schoot and N.F. de Rooij, Sensors Actuators B, 6 (1992) 66–70.

[3] P. Yeh, A. Yariv and C.-S. Hong, J. Opt. Soc. Am. A, 67 (1977) 423–438.

[4] P. Yeh, A. Yariv and C.-S. Hong, J. Opt. Soc. Am. A, 67 (1977) 438–448.

[5] J. Chilwell and I. Hodgkinson, J. Opt. Soc. Am. A, 1 (1984) 742–753.

[6] M.A. Duguay, Y. Kokobun, T.L. Koch and L. Pfeiffer, Appl. Phys. Lett., 49 (1986) 13–15.

[7] Y. Kokobun, T. Baba and T. Sakaki, Electron. Lett., 22 (1986) 892–983.