

3.4 μm “Flip-chip” LEDs for Fiber Optic Liquid Sensing

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Abstract

We have characterized 3.4 μm wavelength InAsSbP/InAs light emitting diodes grown as double heterostructures by liquid phase epitaxy with a 300 μm wide aperture coupled with As_2S_3 naked fibre. The output power, far field pattern and spectral characteristics depend on chemical composition of liquid being in contact with fibre making feasible evanescent wave analysis of liquids contenting water and/or hydrocarbons, e.g. alcohol.

Optical transmission measurements are widely used for analysis of gases since absorption features are «fingerprint» of most substances. These measurements are effective for open path trace analysis in the mid-IR spectral region (3-5 μm) where atmosphere is transparent and most gases have fundamental absorption bands. The above methods are useful also for analysis of liquids, however transparency of the latter is much smaller than that for the gases. This limits the path to several hundreds of micrometers [¹] and obviously is an obstacle for performing trace measurements.

On the other hand the light can be localized in a very thin channel due to internal reflections in a fibre and allow decreasing the interaction with the undesired substances and objects like dust particles distributed in air or floating in a liquid. Such materials as sapphire, silver halides [²] and many others can be used for the fiber.

The interaction with the substance under investigation is realized through absorption of the evanescent field, which exponentially decreases in the vicinity of a fibre surface in contact with a mixture. Being an integral part of radiation traveling along the fibre axis this evanescent part is sensitive to the presence of an absorber at the fibre surface. The number of internal reflections is being sufficient the output from the fibre end depends on absorber concentration and fibre length.

Fiberoptic Evanescent Wave spectroscopy (FEWS) has numerous applications including detection of flammable gases [³] and analysis of hydrocarbons in water [⁴, ⁵] however only few instruments were reported to employ LED source operating in the mid-IR region. This is partly explained by low coupling efficiency due to nonoptimized constructions used so far. McCabe [³] reported the use of InGaAs LED with top contact emitting at 3.3 μm . Direct viewing of the actived LED by fibre coupled with InSb detector enabled the authors of [³] to detect propane of 1% v/v at a fibre length of 23 cm. The above coupling of LED and fibre is characterized by low coupling efficiency and that's why the output power from a 1 m long fibre was in the order of 1 μW only.

Direct “gluing” of the fibre end to an LED surface by polymer compounds is often used for devices operating in the near IR region [⁶]. However, there is very limited information on the mid-IR LEDs with construction that is appropriate for the above coupling. Recently we have developed several “flip-chip” LED constructions [⁷, ⁸] that are convenient for coupling with additional optical elements including fibre. However, to the best of our knowledge there were no reports on direct coupling of a mid-IR LED with a fibre.

Here for the first time we report on efficient coupling of mid-IR LED and fibre and first experiments on liquid sensing using mid-IR A^3B^5 LEDs.

Fig.1 presents scheme of “flip-chip” LED whose surface layer is composed from transparent substrate or “window”. Transparent glue with high refractive index ($n=2.6$) is sandwiched between LED and 500- μm thick As_2S_3 fibre without protection layers as received

from Institute of Chemistry of Pure Materials RAS (N.Novgorod, Russia). The important feature of the LEDs is that the substrate is transparent for the light making possible close contact of the p-n junction and a heatsink with high heat dissipation efficiency. Such

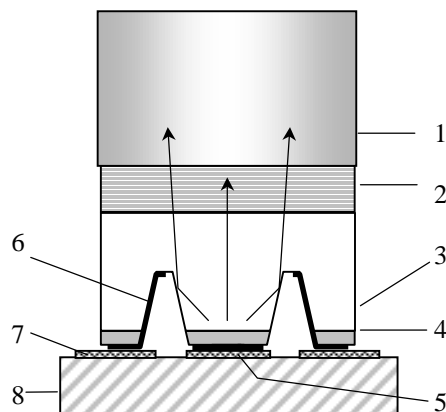


Fig.1 - Scheme of a "fibre" LED, 1- optical fibre, 2- optical "glue", 3- transparent n-type substrate or "broad band window", 4- active zone of the device (p-n junction) 5- anode connected with bonding area, 6-gold contacts (cathodes), 7- Pb+Sn – bonding areas, 8- semiinsulating silicon submount (header)

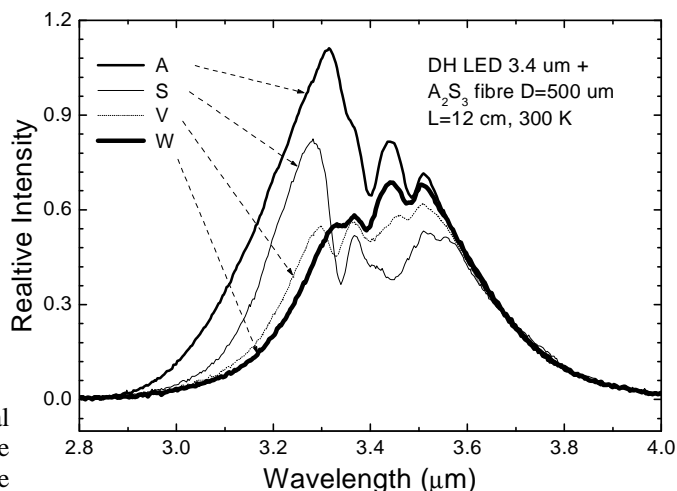


Fig.2 - LED emission spectra recorded as coming from 15-cm long As_2S_3 fiber merged into air (A), alcohol (S), vodka (V) and water (W) at room temperature

constructions usually contain heavily doped n^+ -InAs substrate with Moss-Burstein shift of the absorption edge [8,9].

The output of the assembly shown in Fig.1 at $I = 500$ mA amounts at the fiber end to $100 \mu\text{W}$ and is already two decades higher than previously reported value [3] showing evidently the advantage of the proposed fibre LED construction.

The assembly with 15 cm long As_2S_3 fiber was installed in a cell having the possibility to be filled with liquid and to merge 12 cm of fibre length into a liquid. Inasmuch as the fibre surface was "naked" the output radiation was sensitive to the liquid presence as shown in Fig.2 where LED emission spectra are presented for unfilled cell ("A") and three liquids contacting fibre: water ("W"), alcohol ("S") and a 40% mixture of the latter two (that is, vodka) ("V"). For the sake of convenience we normalized spectra by amplitude in the vicinity of $\lambda = 3.6\text{--}3.9 \mu\text{m}$ where we didn't expect absorption by the liquids used. However, as seen from Fig.2 we were facing some absorption at $3.4 \mu\text{m}$ even when fibre surface was free from liquid. This absorption feature is the intrinsic property of a fibre as confirmed by the vendor. This, however, was not an obstacle to observe spectrum distortions associated with hydrocarbon vibration – rotational absorption at liquid presence. As seen from Fig.2 relative fibre transmission at $3.4 \mu\text{m}$ is increasing in the row: alcohol, vodka, water clearly indicating a possibility of quantitative analysis of the mixture by LED emission spectrum recording or registering several signal corresponding to different wavelengths. In the latter case there is always a choice for the band to use since as seen from Fig.2 the "shortwave" shoulder of the LED emission contains information on water absorption ($\sim 3 \mu\text{m}$) as well: the transmission at $3\text{--}3.3 \mu\text{m}$ evidently indicates the water presence since it decreases on alcohol concentration decrease/water concentration increase.

It is well known that the sensitivity of the FEWS strongly depends on geometrical parameters of the fibre in particular on its diameter: the smaller is the diameter the bigger is the evanescent part of the emission and higher is the number of reflections at the fibre/analyte heterojunction. The latter can be illustrated by the dependence of far field pattern of the light leaving fibre end on a liquid presence shown in Fig.3. Shown in Fig.3 are far-field patterns of

uncoated LED ($\lambda \sim 3.4 \mu\text{m}$) (1), LED coupled with fiber and merged into the air (2) and alcohol (3) correspondingly. Initially broad diagram is narrowing by the fibre presence

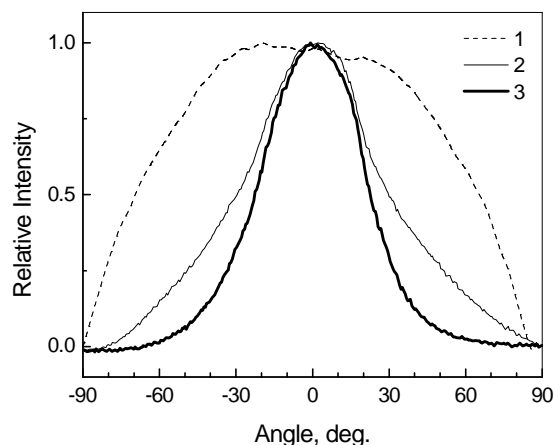


Fig.3 - Far filed pattern of “naked” (1) and coupled with 15-cm long fibre $3.4 \mu\text{m}$ LED exhibited to air (2) and alcohol (3)

since only totally reflected modes are coming out of the fibre. The presence of an absorbing liquid has two effects: a) the decrease of refractive index contrast (Δn) at the fibre surface with the corresponding decrease of the cone of possible transmitting rays b) the increase of absorption of the rays travelling with high number of internal reflections, that is, for the rays with high angles from the fibre axis. Both effects result in the narrowing of the far field pattern for the fibre merged into liquid as clearly seen from Fig.3.

It is worth to mention that the above measurements have shown no hysteresis of the liquid influence on spectra indicating

their potential for quantitative liquid analysis of oil and water mixtures by using e.g. sapphire fibres with high stability with respect to chemicals and high temperature conditions.

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