

## New microsystems-based IR gas sensing technology

Ohlckers, Per<sup>\*,\*\*</sup>, Skotheim, Terje<sup>\*</sup>, Kropachev, Aleksandr<sup>\*</sup>,  
Kunsch, Johannes<sup>\*\*\*</sup>,

<sup>\*</sup> Intex, Tucson, Arizona, <sup>\*\*</sup> Vestfold University College,  
Borre, Norway, <sup>\*\*\*</sup> Laser Components, Olching, Germany  
Per.Ohlckers@hive.no

### Abstract

*Combining Bulk Silicon Micromachining (BSM) with NanoAmorphous Carbon (NAC) thin film technology can be favourably used to make high performance MicroElectroMechanicalSystems (MEMS) devices. Intex is using this unique combination of technologies to design and manufacture a family of high performance infrared emitters with the most distinctive features being high speed with a modulation depth of more than 100 Hz, broadband IR emission from 1 to 14 micrometer, with around 10% power efficiency, and a lifetime beyond 100,000 hours. Rated input power is 950mW. This year (2011), two new versions will be launched, one with 25 % higher output IR radiation for demanding application, and a low power version with smaller chip size for low cost applications. Intex is also developing photoacoustic gas sensors. The photoacoustic principle offers high sensitivity and selectivity when used for gas sensing. Here, the design, fabrication and characterization of prototypes of micromachined silicon microphone with piezoresistive readout designed for low cost photoacoustic gas sensors are presented. The microphones have been fabricated using a foundry multiproject wafer manufacturing service. To increase sensitivity and resolution, a design based on a released membrane suspended by four beams was chosen. The microphones have been characterized for frequencies up to 1 kHz and 100 Hz, respectively. Averaged sensitivities are measured to be  $30\mu V/(V \times Pa)$  and  $400\mu V/(V \times Pa)$*

### Introduction: Thermal IR Emitters for Non-Dispersive Gas Sensors

We define here "Infrared emitters" (IR emitters) as microdevices designed and manufactured using micro- and nanotechnologies (MNT) to enable specific attractive features such as small size, high radiation efficiency, high switching speed, high reliability and low cost. IR emitters are mostly thermal broadband emitters giving heat radiation to a large degree following Planck's law as greybody emitters, where the term greybody means that the emissivity deviates from the ideal blackbody emitters exactly following Planck's law. A typical and probable the most used application for microdevice IR emitters is in Non-Dispersive InfraRed (NDIR) gas sensors [1] using the infrared gas absorption principle. Non-dispersive simply means that dispersion of different wavelengths of the radiation is not used in the sensing principle, in contrast to dispersive techniques like spectrometers.

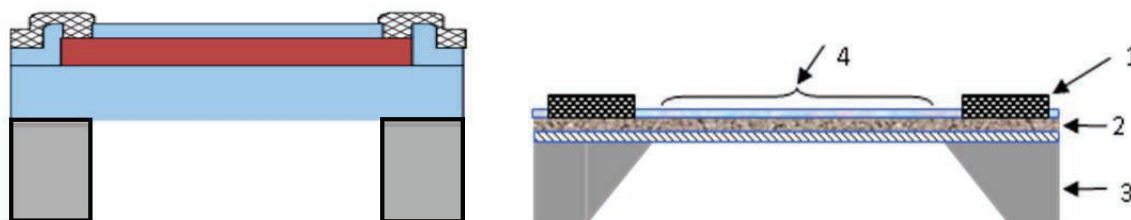


Figure 1 To the left the cross section view of the principle design of a microdevice infrared emitter with a micromachined multilayer membrane film structure. To the left a cross section view of a practical design using anisotropic silicon micromachining; the Intex IN20-1000: 1) Bonding pads; 2) NAC multilayer membrane, 3) Silicon support, 4) Active emitter area. Chip size is 3.8mmx3.8mm with chip thickness of 380 micrometer.

Most microdevice thermal IR emitters are MicroElectroMechanical System (MEMS) devices using bulk silicon micromachining (BSM) to define a thin membrane or a beam structure with low thermal mass

as a carrier structure for a resistive film that is current heated to emit greybody infrared radiation. The resistive film can be made of different materials, with polysilicon, doped nanoamorphous carbon and metal as examples of thin film materials used. A principal configuration is shown in Figure 1 where we can see a micromachined membrane with dielectric thin film layers covering both sides of the heater film structure. The dielectric films have both the function to give environmental protection and the function to set a proper stress level in the membrane or beam structure. In addition, the dielectric films may be designed to improve radiation emissivity at specific wavelengths by manipulating transmission, with constructive interference in the forward direction and destructive interference in the backward direction. In addition, emissivity can be improved by a surface film giving emissivity closer to blackbody radiation.

The carrier structure needs to have low thermal mass to achieve high switching speed and high emission efficiency, while having sufficient mechanical strength to survive the typical pulsed powering between off mode at low temperature and on mode at high temperature. Off-mode temperature would typically be 50 centigrade – at or close to the silicon frame temperature, which would be ambient temperature with the addition of a modest over temperature due to heat pick up from emitting membrane, mostly acquired by conduction and convection from the heating resistor. On-mode temperature in the membrane or beam structure will be given by the on-mode saturation temperature balance as a balance between high emission output and sufficient lifetime for the used materials. Typical in-mode saturation temperatures will be in the range from 600 to 800 centigrades.

The silicon micromachining is today mostly being done by anisotropic wet etching, giving the typical sloped cavity sidewalls at 54.7 degrees inclination, as for the Intex emitter chips shown in Figure 1. Mostly, such micromachining is done using TetraMethyl Ammonium Hydroxide and Water (TMAH) but sometimes also with potassium hydroxide (KOH) and water.

This is expected to gradually change to Deep Reactive Ion Etching (DRIE) [2], a dry etching process giving vertical sidewalls with an aspect ratio typically around 20:1, meaning a deviation from ninety degrees of less than 1 micron for each 20 micron depth, as shown in Figure 1. The main advantage will be smaller chip footprint because of the vertical etch pit sidewalls, while main disadvantage is higher processing cost, which will decrease as the DRIE technology becomes a more mature industrial process.

Also, surface micromachining could in principle be used by etching out a beam structure supported at both ends containing a thin film resistive element, e.g. polysilicon. However, no commercial emitters of such configuration are to our knowledge available – maybe because of the challenges to passivate the resistive thin film structures.

The most used NDIR technology for gas sensor is using infrared bandpass filters to select the specific absorption line for the gas to be sensed. However, the photoacoustic gas sensing principle is an NDIR technique offering higher selectivity and higher sensitivity, but they have so far been offered as large size, high cost systems based on traditional macrosystem integration [3]. By using micro- and nanotechnologies, the principle has the potential to be miniaturised and made much more cost effective. These sensors can favourably use the above described microdevice IR emitters, but need also highly sensitive and accurate micromicrophones to achieve the needed performance.

### **Intex Silicon Micromachined IR Emitters Using Nanoamorphous Carbon as Resistive Heating Element**

The IR emitter IN20-1000 from Intex is a microdevice thermal IR emitter using bulk silicon micromachining to achieve a carrier structure with low thermal mass for the resistive element generating the infrared radiation [3]. These emitters are using a proprietary nanoamorphous thin film technology to make the resistive heating element on these thermal emitters.

Nanoamorphous carbon (NAC) is a diamond-like-carbon (DLC) material that has the following major material characteristics that are important when used as resistive thin film material in IR Emitters:

- Extraordinary Yield Strength of up towards 30 times better than stainless steel and up to 5 times *better than silicon*.
- High thermal conductivity.
- Superior chemical and corrosion resistance
- Processing of DLC films compatible with most silicon processes up to 500 °C ((short pulsing to ~800°C).
- The NAC thin films can be made by different Physical Vapour Deposition and Chemical Deposition Methods, or combined methods.
- Combination of silicon MEMS with NAC thin films can be used to make devices combining the versatility of silicon processing with the unique features of NAC thin film

In addition other important features are:

- Extraordinary stiffness with Young Modulus of Elasticity of around 8 times stiffer than silicon and around 7 times stiffer than steel.
- Indentation hardness and wear resistance approaching diamond, the best among any other materials.

The emitters are packaged in TO5 metal can transistor headers as shown in Figure 2 with an open header cap. The cap can alternatively be sealed in nitrogen with an infrared filter window like calcium fluoride, sapphire, silicon etc., depending upon the spectral properties wanted for the cap window.

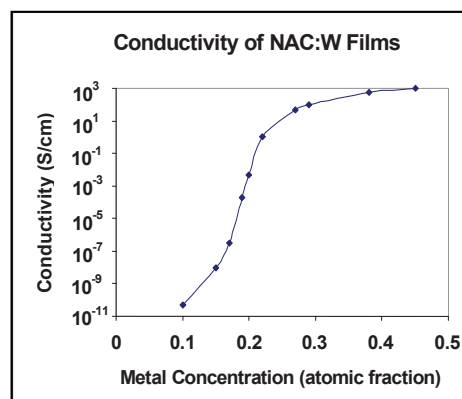
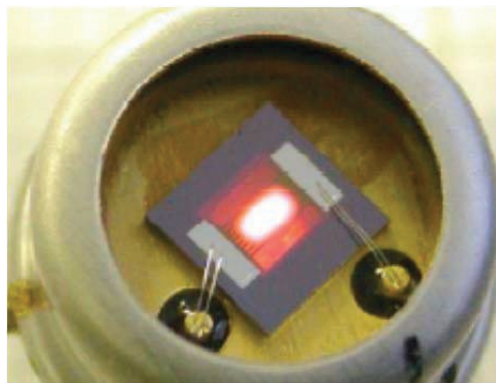


Figure 2: To the left: Picture of the Intex IN20-1000 infrared emitter. It is packaged in a metal can transistor header. The picture is taken during operation, showing the visible part of radiation from the emitting membrane. To the right: Conductivity of the NAC film as a function of the atomic fraction of tungsten as the metal additive cosputtered during the NAC deposition process.

The conductivity of the NAC film can be controlled by the atomic fraction of a metal as the resistive additive, as shown in Figure 2 with tungsten (W) as the metal additive.

The INTEX IN20-1000 emitters radiate like a greybody thermal infrared emitter, as shown in Figure 3 for different power levels from 657 to 851 mW. It can be observed that these emitters shine with high emissivity in the important wavelength band from 2 to 5 micrometer, where many gases with C-H bonds and C-O bonds have strong absorption lines making this band useful for sensing gases with such bonds, like methane and carbon dioxide. The measurements were made by Intex using FT-IR spectrometer Nicolet 6700 with Detector DTGS KBr. All these power levels are below the rated power level of 950 mW, which would give 750°C peak temperature in the centre of the membrane area. However, the emissivity at the band from 8 to 9.5 micrometer is less impressive, although still very high. To excel in that band as well, these emitters would need a high emissivity coating such as the competitor Axetris Leister is using. The source can pulse at frequencies up to 100Hz at ~50% modulation depth. High frequency pulsed sources are important for achieving good signal-to-noise

ratios (high sensitivity) in IR gas sensors. Recent specifications of the IN20-1000 (earlier MIRL17-900) are listed in Table 1. Please note that the difference between cold and hot resistance is within 5%.

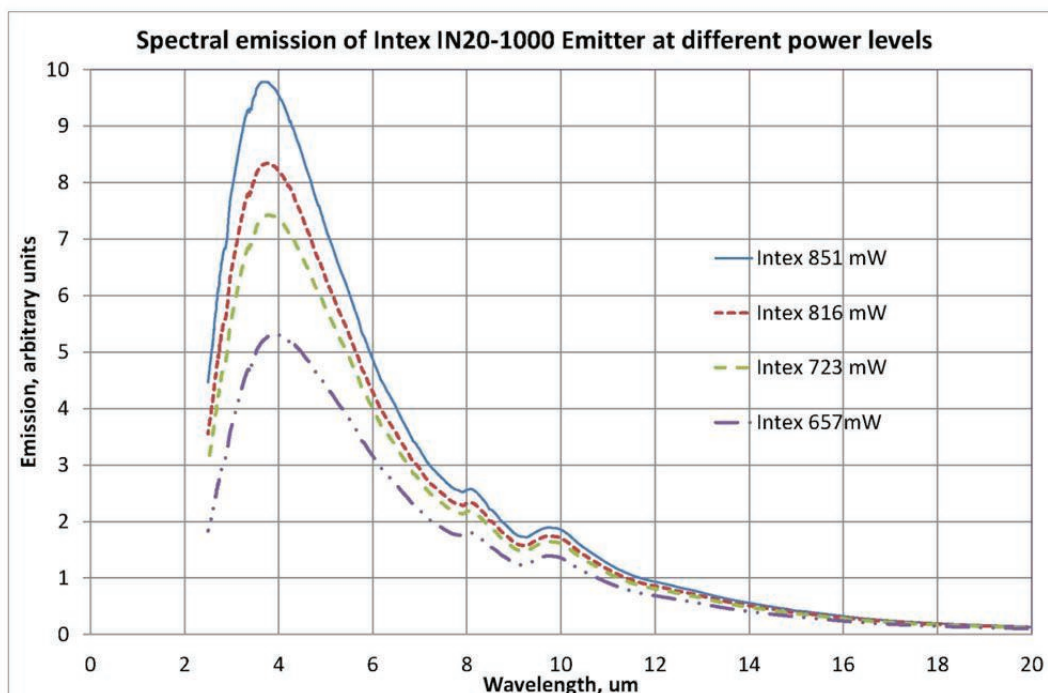


Figure 3: Spectral emission of IN 20-100 (earlier Intex MIRL17-900) at different power levels, with higher power giving higher emission and peak values at shorter wavelengths.

Parameter	Typical	Variation Limits
Spectral Output Range	1.0 - 20 $\mu\text{m}$	See graph
Emitter Surface Area	1.7x1.7 $\text{mm}^2$	
Hot Resistance	50 $\Omega$	45 - 55 $\Omega$
Drive Voltage (pulsed, bi-polar or DC)	6.9 V	6.6 - 7.2 V
Drive Current	140 mA	130 - 150 mA
Working Temperature	750 $^{\circ}\text{C}$	
Modulation Frequency	0 - 100 Hz	
Maximum Frequency at 50% Modulation	100 Hz	
Power Consumption	950 mW	930 - 1030 mW
Integrated Power Emission (Emission Efficiency)	100 mW	90 - 110 mW
Warm-up Time	<30 msec	
Decay time	<5 msec	
Lifetime	>5,000 hours at 750 $^{\circ}\text{C}$ >25,000 hours at 600 $^{\circ}\text{C}$ >100,000 hours at 500 $^{\circ}\text{C}$	Mean Time Between Failure (MTBF)

Table 1: Specifications of the Intex IN20-1000 emitter. Data apply @ 10 Hz and 50% duty cycle.

The emitter output can be focused with a parabolic reflector mounted on the header cap. In this way, improved signal-to-noise ratio can be achieved for the same power input to the emitter.

## Intex is Launching Two New IR Emitters in Year 2011

Intex will in year 2011 introduce two new emitters:

- A high power version with 1300 mW input power and 25% higher infrared radiation output than the existing Intex IN20-1000, focused towards applications needing higher IR radiation intensity.

The higher power rating is mainly achieved by increasing the IR emitting membrane from 2.0mm x 2.0mm to 2.2mm x 2.2mm. Tentative product name is IN22-1300.

- b. A low cost version with 250 mW input power for applications where low cost, small size and low power operation are most important features.

At time of writing, prototypes have successfully been made and tested. Manufacturing process qualifications and product qualifications are now being performed, and product launches are expected in Quarter 3/2011. Spectral emission tests of the new high power Intex emitter, compared with the existing IN20-1000 emitter and the Axetris Leister emitter is shown in Figure 4. The tests are done with the same test setup as described for IN20-1000 in the previous chapter.

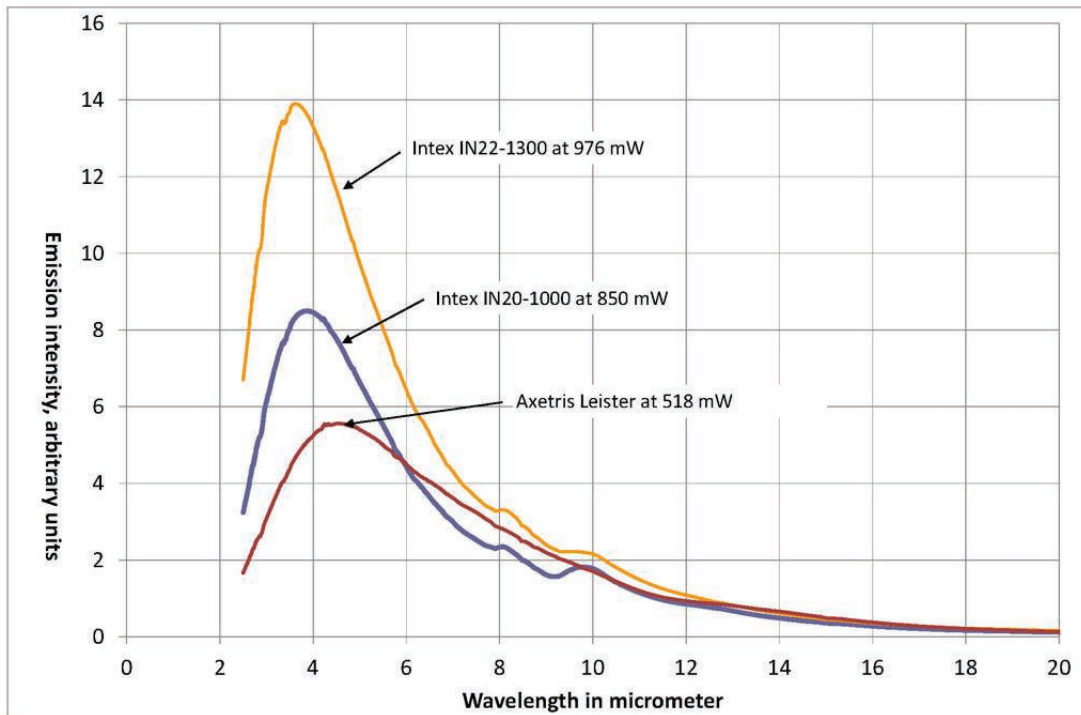


Figure 4. Spectral emissions versus wavelength of Intex IN20-1000 and the new Intex high power emitter IN-22-1300 at comparable power levels, showing that the new IN-22-1300 outshines the present emitter with about 40% higher emission.

As can be seen in Figure 4, the new Intex IN22-1300 emitter definitely outshines the other two emitters. However, it should be noted that the Axetris Leister emitter is performing excellent in the wavelength band from 8 to 9.5 micrometer, for instance favourable to be used for ethanol sensing, which has a strong absorption line at 9.49 micrometer. This can mainly be explained by the close-to-blackbody emissivity of the Axetris Leister emitters due to a proprietary high emissivity coating. On the other hand, the Intex emitters come out more favourable in the band from 2 to 5 micrometer, where for instance methane and carbon dioxide have strong absorption lines, respectively at 3.4 and 4.3 micrometers.

### Micromachined silicon microphone with piezoresistive readout designed for low cost photoacoustic gas sensors

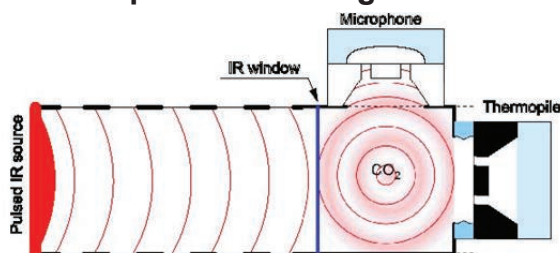


Figure 5: A miniaturized photo acoustic gas sensor system can consist of a pulsed solid state IR source, an absorption path and a reference chamber. The reference chamber is filled with the gas whose

concentration is to be measured. On top of the reference chamber, a microphone picks up changes in the acoustic pressure in the reference chamber, generated by the absorption of light in the gas. At the right end of the chamber, a micromachined thermopile is included to monitor the emitter signal over time, and hence provide long-term stability for the system.

The principal design of a photoacoustic system is shown in Figure 5. To make a miniaturised and low cost photoacoustic gas sensor, a highly sensitivity microphone with low cost is needed. Therefore, different prototypes of silicon micromachined piezoresistive microphones have been designed and fabricated by Vestfold University College in collaboration with Intex, using the MultiMEMS multiproject wafer manufacturing service from Sensoror. The principal design is shown in Figure 6.

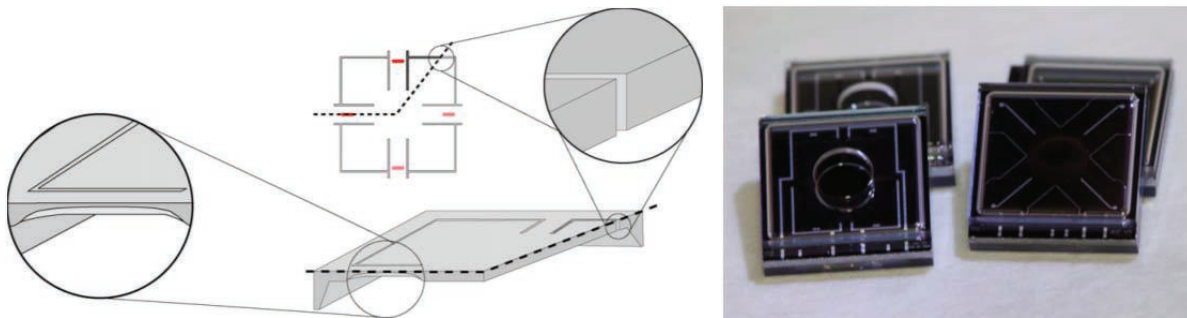


Figure 6. To the left a 3D sketch showing slightly more than one-quarter of the microphone design, the rest being identical. Surrounding the square diaphragm is a narrow slot of  $3\text{ }\mu\text{m}$  line width to make the suspension system as compliant as possible while minimizing the gas flow past the membrane to maximize sensitivity. The beams are here fabricated in thin membrane regions inside the thick membrane. To the right pictures of fabricated microphone chips.

The prototypes have been fabricated with different slot depths using Reactive Ion Etching (3 and 6 microns) and Deep Reactive Ion Etching (23 microns) and the prototypes have measured sensitivity ranging from average  $30\mu\text{V}/(\text{V} \times \text{Pa})$  to average  $400\mu\text{V}/(\text{V} \times \text{Pa})$  depending on the specific design. This means that photoacoustic gas sensors with high resolution, for instance for carbon dioxide with parts per billion resolution.

## Conclusions and further work

Intex is successfully manufacturing microdevice IR emitters for NDIR gas sensor applications with high IR intensity and high speed as highlighted features, and will in 2011 launch a new emitter IN22-1300 with 25% improved radiation intensity, and one low power version with smaller size and lower cost. Future work will focus on performance improvements and more cost effective manufacturing enabling wider market acceptance. In addition, Vestfold University College and Intex is developing high sensitive silicon microphone technology for future photoacoustic gas sensors with parts per billion resolution for gases such as carbon dioxide.

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