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High throughput low cost closed field magnetron sputter deposition of durable reflectors based on dielectric overcoated metal for application in non-dispersive infrared gas sensors

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Abstract: Optical & mechanical properties of closed-field magnetron pulsed DC sputter deposited Al, Au & Ag with Al_2O_3 protective layer are presented. Results show optical & durability performance suitable for use within NDIR optical gas sensors.

OCIS CODES: (310.1860) Deposition and fabrication; (310.3840) Materials and process characterization; (310.1860) Thin films, optical properties

1. Summary

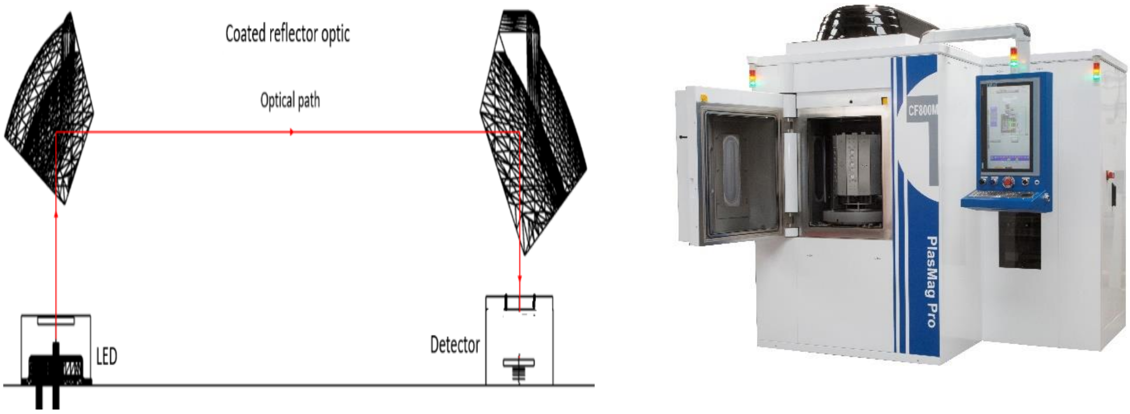
Non-dispersive Infrared (NDIR) gas sensors typically comprise an infrared light source/photodetector optopair, an optical bandpass filter to select emitted/ detected light at a wavelength tuned to the absorption of a specific gas in the optical path between source and photodetector [1]. Gas absorption wavelengths are typically in the mid-infrared (MIR) waveband (3 to 5 µm). Examples include 3.3 µm and 4.2 µm for methane (CH_4) and carbon dioxide (CO_2) respectively. For a given pathlength measured signal reduction is calibrated against gas concentration using the Beer Lambert law [2] over a range of operating temperatures.

Current NDIR gas sensors typically utilise injection moulded reflective optics [1] to guide light through the gas cell pathlength. Typically, gold thin films (thickness ≥200 nm) are used as the reflective surfaces providing a MIR high reflectance [1,3,4]. However, high price of gold material is not compatible with volume manufacture of low cost NDIR sensors. As such there is a need for alternative low-cost MIR reflective thin films compatible with deposition onto injection moulded optics and providing same or better optical (reflectance typically > 97%), adhesion, durability and environmental [5] performance compared with gold.

In this work optical thin film modelling [6] was progressed identifying both silver (Ag) and aluminium (Al) as low-cost candidate materials - providing typically a factor of 100 reduction in material cost compared with gold. Enhanced durability and environmental performance using an aluminium oxide (Al_2O_3) thin film overcoat layer was investigated and optimised. Use of drum based closed field unbalanced magnetron (CFUM) sputtering [7] provides a high throughput low temperature process - compatible with deposition onto injection moulded optics. Moreover, CFUM sputtering provides sequential deposition of the metal reflector layer and subsequent reactive sputter deposition of a thin Al_2O_3 protective layer within the same deposition cycle.

2. Modelling

Essential MacLeod optical thin film design software [6] was used to evaluate and compare various metals with gold reflector layer performance. This analysis identified aluminium & silver as low-cost candidate materials based on modelled spectral reflectance at 0° and 45° angle of incidence (AOI) - 45° AOI is relevant as NDIR gas sensor optics utilise folded optics to minimise space envelope. MIR reflectance results (mid-range MIR wavelength selected 4 µm for analysis). Modelled results indicate acceptable reflectance levels and angular performance (0 to 45°) for Au, Ag and Al - silver exhibits highest reflectance (≈99 %) and minimal change with AOI (0 to 45°) out of the three modelled metals. Influence of a thin Al_2O_3 protective overcoat layer on MIR metal layer reflectance is minimal overlayer thicknesses < 50 nm reduce reflectance by typically ≈0.1 %.



(a) **(b)**

Figure 1. **(a)** Schematic of Zemax modelled gas sensor architecture showing a single ray trace and **(b)** CFUM sputter system used to deposit protected reflector coatings onto sensor moulded optic.

An NDIR gas sensor (moulded polymer) with co-planar MIR InAsSb based light emitting diode (LED) and photodiode (PD) optopair [8] schematic is shown in figure 1a. Two parabolic reflectors operating at AOI 45° focus and direct MIR light via a 50 mm gas path. Bandpass filters (BPF) are added to select specific gas absorption spectral waveband. Zemax [9] non-sequential ray tracing modelled throughput progressed for various protected metallic reflectors. Includes influence of LED spectral output and detector spectral responsivity.

Experimental Results

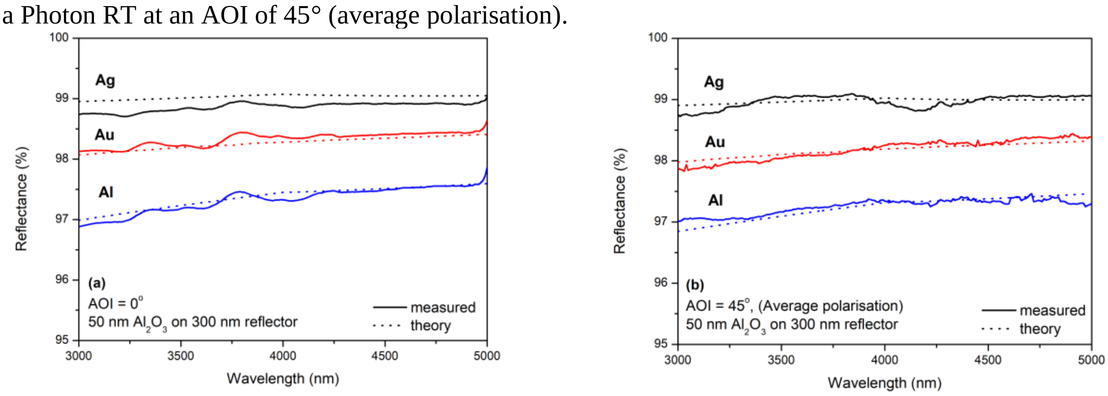
Deposition was carried out using a drum based CFUM pulsed DC reactive sputtering process with a 5-minute ion pre-clean for moulded optics. Pulsed DC sputtering is employed to suppress arcing, necessary to ensure required coating cosmetic quality is achieved. For both the Ag & Au deposited films - thin chromium bonding layer used to enhance adhesion to the injection moulded NDIR optics. Optimised deposition conditions are provided in table 1 for each metal and Al_2O_3 overcoat. Depositions carried out at near-room temperature.

Table 1. Deposition conditions for Al, Ag, Au metal layers & Al_2O_3 protective overcoat

Metal layer				Al_2O_3 layer				
Coating Material	Ar Flow (sccm^{-1})	Power (W)	Bias Voltage (V)	O_2 Flow (sccm^{-1})	Power (W)	Bias Voltage (V)	Pulsed DC Frequency (kHz)	Pulse Width (ns)
Aluminium	14	1300	30	9	1200	30	151	1536
Silver	14	500	30					
Gold	14	200	60					

Measured and Modelled MIR Reflectance

MIR reflection tests on microscope slide reference substrates and NDIR moulded sensor optics, was undertaken using a Perkin Elmer 983 infrared spectrophotometer over wavelength range of 3.0 to 5.0 µm at an AOI of 0° and a Photon RT at an AOI of 45° (average polarisation).



(a) **(b)**

Figure 2. Comparison of modelled and measured Al_2O_3 protected reflector optical reflectance at incident angles **(a)** 0° and **(b)** 45°

NDIR Sensor Test Bed (Fig 1a) Throughput Measurements

An NDIR gas sensor test bed was constructed based on Fig 1a schematic, comprising an electronic board controlling co-planar MIR LED and photodiode (PD) optopair [8]. Coated reflector polymer optic is removable, allowing test of optics with different coating designs. Measured results provided in Table 2.

Table 2. Comparison of theoretical and measured optical NDIR sensor testbed (see Fig 1a) throughput (%) - utilising III-V source and detector (wavelength 3.3um) optopair [8].

Coating design	Measured NDIR sensor optical throughput (%)	Zemax [9] modelled NDIR sensor optical throughput (%)
Al_2O_3 (50 nm)/ Ag (300 nm)	12.9	12.9
Al_2O_3 (50 nm)/ Au (300 nm)	12.7	12.8
Al_2O_3 (50 nm)/ Al (300 nm)	11.6	12.2

Adhesion, Durability and Environmental Performance

Al_2O_3 protected Ag, Au and Al reflector coatings pass the MIL-STAN environmental and durability tests listed in table 3. Tests carried out in sequential order with optical reflectance performance unchanged.

Table 3. MIL-STAN -C-48497a [5] environmental/durability test results

Specification	Description
Adhesion (tape snap test) MIL-C-48497a 3.4.1.1	No sign of coating removal when cellophane tape is pressed firmly against the coated surface and quickly removed at an angle normal to the coated surface.
Humidity (24 hr > 95% RH) MIL-C-48497a 3.4.1.2	No evidence of cracking, peeling or blistering after 24 hr exposure to 95% RH at 50ºC
Moderate abrasion (cheesecloth) MIL-C-48497a 3.4.1.3	No signs of deterioration such as streaks or scratches after 50 strokes with dry clean cheesecloth pad.

Conclusions

In this work CFUM pulsed DC reactive sputter deposited Al_2O_3 overcoated Al, Ag & Au reflectors exhibit high reflectivity (97 to 99%) in agreement with modelled reflectance. Results demonstrate Al_2O_3 protected Ag and Al reflectors can be used as low-cost replacements for Au as MIR reflector in NDIR gas sensors without compromising sensor performance.

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