Microwave Plasma Assisted Magnetron Sputtering of Infrared Multilayer Thin Film Bandpass Filters for application in non-dispersive infrared gas sensors

Jonathan Pomfret[1, 2,*], **Des Gibson**[1,3], **Lewis Fleming**[1,3], **Shigeng Song**[1,3] ,
Greg McGann[1,3], **Sam Ahmadzadeh**[1], **Peter Mackay**[2]
[1]*Institute of Thin Films, Sensors and Imaging, Scottish Universities Physics Alliance*
School of Computing, Engineering and Physical Science. University of the West of Scotland, Paisley PA1 2BE Scotland, UK
[2]*G&H PLC, Dowlish Ford, Ilminster, TA19 OPF, UK*
[3]*Albasense Ltd, Paisley, PA1 2BE, Scotland,, UK*
**Corresponding author Jonathan Pomfrets: B00408581@studentmail.uws.ac.uk*

Abstract: Infrared bandpass filters deposited using microwave plasma assisted pulsed DC magnetron sputtering are described. Optical, thermo-optical properties and spectral response across a range of environmental conditions suitable for use in NDIR gas sensors are demonstrated.

1. Summary

Non-dispersive infrared (NDIR) sensors are based on mid-infrared absorption spectroscopy [1], providing benefits such as cost, sensitivity and integration as a miniaturised gas sensor configuration. Realisation of an NDIR sensor based on spectral differentiation requires a modulated mid-infrared source and detector optopair, covering the wavelength range of interest [2]. Band pass filters (BPF) discriminating specific wavelengths from the incident radiation are a key NDIR sensor component, shaping the spectral characteristics of the source/ detector optopair for both sensing and reference channels, enhancing NDIR gas sensor performance and stability. As such BPF`s for NDIR gas sensor application must be durable and spectrally drift- free over a wide temperature range (typically -40 to +50°C) and environmental conditions.

BPF`s deposited using traditional high temperature electron beam deposition methods lack the required durability and spectrally drift-free performance required by NDIR gas sensors. Refractive index stability of the layers is critical in minimizing the thermal drift of filter performance [3] with layer density and porosity key factors in determining refractive index thermal shift [4]. This work describes high throughput BPF deposition using a novel near room temperature drum based Microwave Plasma-Assisted pulsed DC Sputter (MPAS) deposition process [5-7]. This process has previously been demonstrated to produce highly dense, void free films [8]. BPFs comprise germanium (Ge) and silica (SiO_2) alternating high and low refractive index layers.

Thin film multilayer optical coatings were prepared using a novel microwave plasma-assisted pulsed reactive DC magnetron technique. The apparatus was equipped with a complete microwave generator system comprising a 6 kW microwave magnetron head, tuner, and microwave delivery tubes. In this work, thin films of germanium (Ge) and silicon dioxide (SiO2) were obtained by sputtering from Ge and silicon (Si) bulk sputtering targets, each with 99.999% (5 N) purity.

The deposition rate and thickness of the film were monitored in real-time by quartz crystal thin film thickness monitors (Inficon IC5, Inficon Inc., Bad Ragaz, Switzerland). Argon is used as a sputter process gas, and oxygen and hydrogen as reactive gases. The flow rate of gas was accurately controlled by an MKS mass flow controllers (MKS Instruments Inc.,Andover, MA, USA). The chamber body was heated by hot water to liberate any residual H2O molecules trapped on the chamber walls, thereby increasing their probability of being trapped by Meissner cooling coils (Telemark polycold model 3600, Telemark Inc, Battleground, WA, USA).

Table 1. The deposition parameters used to grow thin films of Ge and SiO2. Both materials were run in power control

Material	Ar Flow ($sccm^{-1}$)	O_2 Flow ($sccm^{-1)}$	H_2 Flow ($sccm^{-1}$)	Power (kW)	Current (A)	Voltage (V)	Microwave Power (kW)	Pulsed DC Frequency (kHz)	Deposition rate (nms-1)
SiO2	190	35	0	3.5	7.5	475	2.75	46	0.1
Ge	190	0	10	2.5	6.1	410	0	46	0.06

Optical transmittance measurements at normal incidence were carried out using a Perkin Elmer 983G dual beam dispersive infrared spectrophotometer. Variable temperature measurements were obtained on the same instrument using a variable temperature transmission cell from Specac equipped with ZnSe windows (part no. GS21525-Z). 983G was computer controlled using filmstar MEASURE from ftg software[9].Thin film filters were designed using filmstar DESIGN thin film design software[9].

2. Results

Environmental testing was carried out according to MIL-C-48497a [10]. Filter transmittance was measured before and after environmental testing. Adhesion and abrasion tests were carried out using Lens coating hardness kit from Summers Optical. Humidity and temperature tests carried out in a WKL temperature and climatic test system.

Table 2. Environmental test parameters

Test	MIL-C-48497a paragraph	Conditions	Result
Adhesion	4.5.3.1	Tape	PASS
Humidity	4.5.3.2	49C 95% RH	PASS
Temperature	4.5.4.1	-62C to 71C 2 hours each	PASS
Moderate Abrasion	4.5.3.3	50 strokes cheesecloth	PASS
Severe Abrasion	4.5.5.1	20 strokes eraser	PASS
High Temperature bake		350°C 2 hours	PASS

4 filters were deposited for use at 3300nm for the detection of methane. Longwave pass filter (LWPF), Wideband pass (WBPF), notch filter (NF) and narrow bandpass filter (NBPF). LWPF & WBPF were deposited on 0.5mm thick 3” Silicon wafer, NF & NBPF on 0.25mm thick 3” fused silica wafer. Spectral response of filters are shown in figure 1 and their optical performance summarized in table 3.

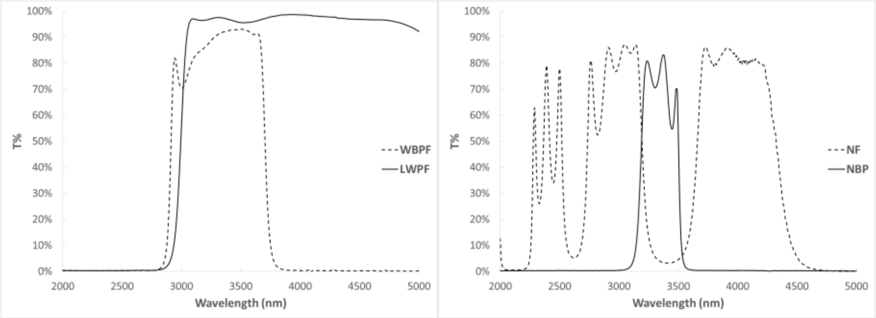

Fig. 1. Measured spectral characteristics of filter set.

Table 3. Optical performance of filter set.

Narrow bandpass	Wide bandpass	Longwave pass	Notch filter
Centre wavelength: 3336nm	Half power point: 2910nm	Half power point: 2995nm	Centre wavelength: 3374nm
Half bandwidth: 321nm	Half power point: 3705nm	Max Transmission: 98.7%	Half bandwidth: 478nm
Max Transmission: 83.1%	Max Transmission: 92.9%		Max Reflection: 94.3%
3” wafer filter thickness uniformity: ±0.25%			

NBPF has been measured in the temperature range -60C to 100C. Results are presented in figure 2. Centre wavelength shift of filter with temperature was fitted using linear regression model in minitab 21. This gave a temperature dependence of CWL of 0.139 nm/°C with an R^2 value of 98%.

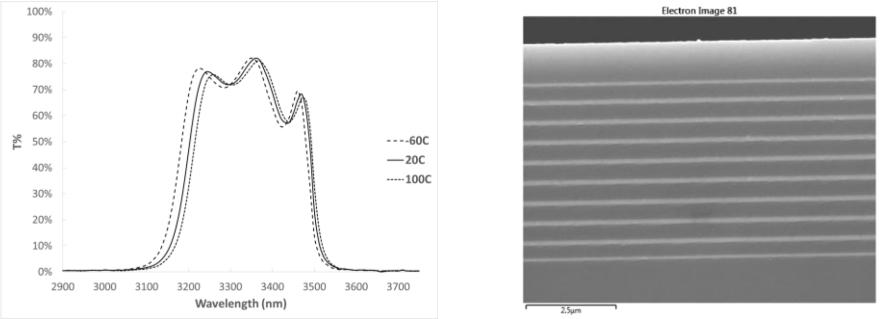

Fig. 2a. Measured spectral characteristics of NBPF with variable temperature, 2b. SEM image of SWP coating

SEM image shows the layers have a dense amorphous near-bulk structure. Layer at bottom of SEM image is a combination of Fused Silica substrate and initial SiO2 layer. These cannot be distinguished, further highlighting the near-bulk properties of the film. Single Layers of SiO_2 and Ge were also measured in the temperature range -60C to 100C and modelled by applying a thermo-optical coefficient $\frac{dn}{dt}$. SiO2 showed no discernable variation with temperature, similar to results previously reported [4], which implies figure is close to bulk value (8.9E-6 @ 3.2μm and 300K) [11]. Ge index variation was fitted giving $\frac{dn}{dt} = 4.91E^{-4}$, close to reported bulk values [12]. Applying this values to model of filter gave temperature dependence of CWL of 0.244 nm/°C, exceeding measured value of 0.139 nm/°C, implying that the embedded $\frac{dn}{dt}$ is even lower than single layer result[13].

3. Conclusion

In this work set of infrared optical filters for use in NDIR gas detection have been designed and fabricated using a microwave plasma-assisted pulsed reactive DC magnetron (MPAS) process. Minimised thermal spectral shift of filter is due to the thermo-optical coefficients of layers approaching bulk values.

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