CLOSED FIELD MAGNETRON SPUTTER DEPOSITION OF CARBIDE AND NITRIDES FOR OPTICAL APPLICATIONS

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ABSTRACT

Magnetron Sputtering has many advantages over conventional evaporation processes for the deposition of carbides and nitrides for optical applications. The sputtering process is “cold”, making it suitable for use on the widest range of substrates including temperature sensitive substrates and polymers. Moreover, the drum format provide more efficient loading for high throughput production.

In contrast to previous reactive dc sputtering strategies the Closed Field process does not require a separate ion or plasma source for activation. Neither does it require the vacuum chamber to be separated by vacuum pumps or baffles into deposition and reaction zones. Reaction occurs on the target and all the way round the substrate carrier resulting in low absorption.

The use of the Closed Field and unbalanced magnetrons creates a magnetic confinement that extends the electron mean free path leading to high ion current densities. The combination of high current densities with ion energies in the range ~30eV creates optimum thin film growth conditions. As a result the films are dense, spectrally stable, supersmooth and low stress.

This paper presents optical, durability and stress data for CFM sputtered carbides and nitrides.

INTRODUCTION

Magnetron sputtering has developed rapidly over the last decade to the point where it has become established as the process of choice for the deposition of a wide range of applications. Closed field magnetron sputtering (CFM)\(^1,2\) is an exceptionally versatile technique, suitable for the deposition of high quality, well adhered films of a wide range of materials at commercially useful deposition rates.

It has been reported previously\(^3,4\) that “closed field” reactive magnetron sputtering produces dense, spectrally stable metal-oxide optical coating material with refractive indices typically close to that of the bulk material.

Sputter deposition of non-oxide materials is becoming important for applications such as displays, photovoltaics and durable infra-red anti-reflection coatings. Specific non-oxide film requirements for specific applications are listed as follows.

**Photovoltaics**\(^5\)
- Contacts: NiV, Mo, SbTe
- Absorbers: CdTe, CIGS, a-Si: H
- Passivation: SiN\(_x\)
- Anti-Reflection: Si\(_3\)N\(_4\)

**Durable Infra-Red Anti-Reflection Coatings**\(^6\)
- Phosphides, diamond-like-carbon, germanium carbide, and nitrides

**Displays**\(^7\)
- Passivation: SiN\(_x\)
- Anti-Reflection: Si\(_3\)N\(_4\)
In this paper we report new results that show CFM sputtering of non-oxide materials based on nitride and carbides materials.

**REACTIVE CLOSED FIELD MAGNETRON (CFM) SPUTTERING**

The reactive closed field magnetron sputtering process was developed in 1991 by Teer and has been used successfully for some time in the field of tribological coatings. Recently, the process control has been developed to enable sub-nanometre thickness precision for application to multilayer optical coatings.

![Diagram](image)

**Figure 1** The “closed field” magnetron sputtering process does not need a separate ion source. Adjacent magnetrons are made opposite polarity to trap the plasma all the way round the drum. This ensures that all the metal deposited by the magnetrons is converted into metal-oxide.

The closed field process is illustrated schematically in Figure 1. The closed field configuration was developed to increase the ion current density in the plasma by arranging that neighbouring magnetrons are of opposite magnetic polarity. Using this arrangement, the deposition volume in which the substrates are located is surrounded by linking magnetic field lines. This traps the plasma region, prevents losses of ionising electrons, and results in significant plasma enhancement. The closed field system produces enhanced reactivity due to the high ion current density (>1mA.cm\(^{-2}\)). The ion energy is determined by the induced Voltage on the substrate carrier which is typically in the range (20-30)\(\text{eV}\). This combination of high ion current density and low ion energy produce ideal conditions for growth of optical quality thin films.

During reactive oxidation in the closed field process, the oxygen is admitted into the unbalanced magnetron plasma. Target oxidation occurs but is controlled using target Voltage control with feedback to the mass flow of oxygen. Surface charging is overcome by using pulsed dc power.

The key advantage of the process is that no separate ion source, plasma source or microwave ion source is required. Nor is it necessary to partition the working vacuum chamber into deposition and reaction zones. This simplifies the system, reduces cost, and improves reliability. It also makes it economical to scale the technology to virtually any batch size.

**THE CLOSED FIELD MAGNETRON (CFM) SPUTTERING SYSTEM FORMAT**

The thin film materials presented in this paper were deposited using a CFM450 system from Applied Multilayers Ltd. The system shown in Figure 2a. It uses a 250mm diameter vertical drum substrate carrier and up to four 405mm linear magnetrons. The drum is divided into a number of segments on which the substrates are mounted. Access is made through a large hinged external door.

The distance between the magnetron and the segmented drum surface is 100mm. Curved substrates are loaded behind apertures in the segment to ensure a reasonably constant average target to substrate distance.

The system used in these trials was pumped using a 2000 litres/sec BOC Edwards Diffstak.

A Meissner trap is used for rapid removal of water vapour and reduced pumpdown times.
The system is also fitted with a rotating shutter to expose the substrate to the appropriate target. The shutter is also used to enable targets to be conditioned without exposure to the substrates. A schematic of the drum/shutter configuration is shown in Figure 2b.

**Figure 2a** CFM450 AR coating system from Applied Multilayers Ltd.

**Figure 2b** Drum/shutter configuration

Deposition work below is based on use of a 10% tin (by weight) indium tin sputter target.

**OPTICAL**

**Silicon Nitride**

The transmission of a closed field magnetron sputtered silicon nitride (sputtered silicon in nitrogen reactive gas) thin film is shown in Figure 3.

**Figure 3** The spectral transmittance of a silicon nitride thin film

Figure 4 shows refractive index and absorption coefficient dispersive data for silicon nitride.

**Figure 4** Refractive index and absorption coefficient dispersive characteristic for silicon nitride
Figure 5 shows refractive index data for silicon oxynitride as a function of oxygen/nitrogen ratio region.

**Germanium Carbide**

Germanium carbide is formed by sputtering of germanium in a hydrocarbon reactive gas.

Figure 6 shows the germanium carbide refractive index (@ 10μm) as a function of hydrocarbon to argon gas ratio.

**Diamond Like Carbon**

Optical constants for diamond like carbon (DLC) are shown in Figure 7.

DLC is deposited by CFM sputtering graphite in a hydrocarbon reactive plasma.

**STRESS**

Film stress was evaluated from measured substrate curvature before and after film deposition. Stress was calculated from the Stoney formula. Curvature was measured along two perpendicular axes across the wafer. Typical values are indicated in Table 1.

<table>
<thead>
<tr>
<th>Method</th>
<th>CFM Sputtered</th>
<th>RF Sputtering</th>
<th>PECVD</th>
</tr>
</thead>
<tbody>
<tr>
<td>GexC (1-x)</td>
<td>-150MPa</td>
<td>-800MPa</td>
<td>-0.5 to -1GPa</td>
</tr>
<tr>
<td>DLC</td>
<td>-120MPa</td>
<td>N/A</td>
<td>-0.7 to -1.1GPa</td>
</tr>
<tr>
<td>Si3N4</td>
<td>-100MPa</td>
<td>-300MPa</td>
<td>-300 to -500MPa</td>
</tr>
</tbody>
</table>

Table 1: Measured stress for a range of oxide films (negative value indicates compressive stress)

Method – Deflection of thin coated substrate. Error ±5%

**ENVIRONMENTAL & MECHANICAL PERFORMANCE**

Environmental performance for GeC and DLC on germanium substrates are indicated in Table 2.
Test | Method | Specification
--- | --- | ---
Adhesion | Scotch Tape Test | Mil-C-48497A para 4.5.3.1
Humidity | 24hrs, 50°C, 95% RH | Mil-C-48497A para 4.5.3.2
Severe Abrasion | 50 strokes | Mil-C-48497A para 4.5.35.1
Salt Spray | 24hrs | Mil-C-675C para 4.5.9

Table 2 Environmental performance of GeC coating on germanium substrate

Hardness levels have been assessed between 12 to 20GPa, for film thicknesses ranging from 1 to 10um.

DISCUSSION

Closed field reactive magnetron (CFM) sputtering has been used for many years to produce highest quality tribological coatings. The same basic process produces transparent nitride and carbide coatings with outstanding optical, durability and environmental properties. Also the process is capable of producing low stress, dense, super-smooth coatings with low optical scatter. These properties are all derived from the fundamental advantage of the closed field strategy inherent in the combination of high ion current density combined with low ion energy.

The CFM process can be exploited in batch format or in-line format. The process does not require an auxiliary ion or plasma source and without this overhead, the batch systems are scaleable to meet the demands for a small development system through to high throughput production systems. The in-line format is particularly appropriate to the high volume production demands for displays and photovoltaics.

REFERENCES

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