Technological challenges in the development of silica-titania platform for integrated optics

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Abstract—This short report discusses the technological challenges of silica-titania (SiO_2:TiO_2) thin film deposited via a simple, cost-effective sol-gel process and dip-coating method. Sol-gel is a versatile method for producing materials by using a solution (sol) that undergoes a gelation process to form a solid network (gel). This process involves hydrolysis and condensation reactions of precursor molecules. Although the sol-gel application is simple and economical, it has some limitations. The aging of sol-gel materials causes a change in the properties and structure of a sol-gel system over time, causing a deterioration in the final properties of the waveguide thin films. Nanoimprint lithography is a cost-effective patterning technique that is only effective when fresh sol is used; otherwise, it leads to the formation of defective waveguide structures.

The sol-gel process is a well-established synthetic approach for producing metal oxide nanoparticles such as ZnO, SiO_2, TiO_2, CuO, Fe_2O_3, MnO_2, and mixed oxide composites such as SiO_2: TiO_2, ZnO: TiO_2, and SiO_2: ZnO. It was first proposed by Ebelmen, a French chemist, approximately 170 years ago [1]. Ebelmen's work is credited with establishing a sol-gel synthesis of silicon tetra isoamyl oxide from silicon tetrachloride and isoamyl alcohol. The sol-gel process provides a powderless coating of glass and ceramics [2–4]. This process can be performed by dip-coating or spin-coating methods [5–8].

The precursors used in the sol preparations are tetraethyl orthosilicate $Si(OC_2H_5)_4$ (TEOS) for Si and tetra ethoxy titanate $Ti(OC_2H_5)_4$ (TET) for Ti. The solutions are partially hydrolyzed first, then combined, and a homogenizing agent (ethyl) and catalyst (HCl) are added. The thin film is then treated further to generate waveguiding structures [9]. The sol-gel process is a wet chemical process that is combined with the dip-coating method for the deposition of SiO_2:TiO_2 high-quality thin-film, as shown in Fig. 1.

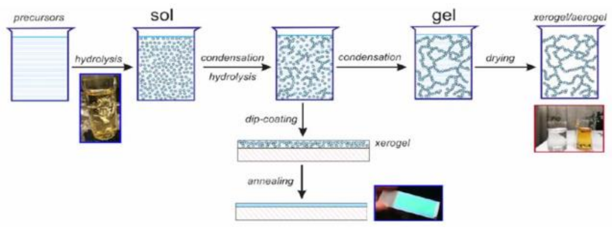

Fig. 1: Steps of sol-gel dip coating method [9].

The equipment used in thin-film deposition process is simple and straightforward forward which is why the cost of depositing high-quality thin-films on a glass substrate is significantly low as it does not require the complex devices used in chemical vapor deposition (CVD) and physical vapor deposition (PVD), which are other traditional processes. As with the other thin-film coating methods, it is critical to control the film thickness in the dip-coating method to meet the requirements and trouble-free use in integrated optics. Karasinski *et al*. determined that the thickness is controlled by substrate withdrawal speed, inclination angle, sol concentration, sol viscosity, density, and liquid-vapour surface tension are factors that affect the thickness, as shown in Fig. 2 [9].

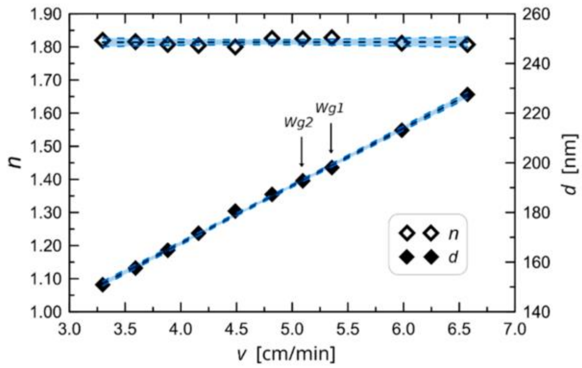

Fig. 2. The experimentally determined characteristic of waveguide film thickness (d) and refractive index (n) against change in substrate withdrawal speed (v) from the sol [9].

In addition to the simplicity and low cost of the sol-gel method, the control of the refractive index of the SiO_2:TiO_2 thin film is achieved by appropriate adjustment of the stoichiometric ratio between the leading components. Due to the high smoothness of fabricated waveguide films on a glass substrate, low diffusion loss is possible, and non-abrasive production by directly applying nano-imprint lithography (NIL) on uncured waveguide films [10–11].

The SiO_2:TiO_2 waveguide layers are deposited on a standard glass (silica) substrate for cost-cutting. The glass substrate is cleaved with a diamond cutter to obtain the end facets of the optical devices. As the glass substrate is amorphous, the end facets may not look so smooth. Therefore, after the cleaving process, the resulting surfaces may require further processing or treatment, such

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as polishing or cleaning, to ensure they meet the optical quality to reduce the insertion loss. Advanced techniques, such as dicing saws or laser cutting, are also used in the industry for more precise and automated cleaving processes. To evade such complications related to the end-facet cleaving, silica-on-silicon (SOS) substrates can also be used to deposit SiO_2:TiO_2 thin-films. The cleaving is performed manually by scribing a line and applying a force to the wafer along the scribe line, causing a controlled fracture along the crystal lattice of the silicon wafer. However, using an SOS wafer can add up to the overall cost of the photonic integrated circuit (PIC).

The sol-gel process and dip-coating method are generally suitable for producing thin films ranging from a few nanometers (~180 nm to 200 nm) in thickness. If thicker films are required, multiple coating and annealing cycles are typically needed, which can be time-consuming and may introduce defects. Scaling up the sol-gel process and dip-coating method for large-scale production can be challenging [12]. Achieving consistent film quality and uniformity across larger areas can be difficult, and the process may require substantial optimization to guarantee reproducibility [13]. The sol-gel process and dip-coating method typically involve specialized equipment and precise process control. Maintaining the required environmental specifications, such as temperature and humidity, can be demanding. The complexity of the equipment and process can increase the cost and technical expertise needed for thin-film deposition.

It is worth noting that some of these drawbacks can be addressed through process optimization, the use of additives or surfactants, and innovations in equipment and materials. However, these challenges highlight the limitations and consequences of thin-film deposition using the sol-gel process and dip-coating methods.

SiO_2:TiO_2 platform offers many advantages over other traditional technological platforms, such as Silicon-on-Insulator (SOI) and Indium phosphide (InP), as shown in Table 1 [14]. Contrary to SOI and InP, SiO_2:TiO_2 has wide transparency, making operation in the visible and near-infrared spectral range possible. Thin-film deposition in SiO_2:TiO_2 is based on a sol-gel process and dip-coating method, and this offers a very high-cost efficiency because the wafer bonding method and LP-MOCVD require expensive devices, that's why SOI and InP platforms cannot offer a high-cost efficiency. SiO_2:TiO_2 has a very high chemical resistivity, unlike SOI and InP, which are prone to oxidation. Over the past few decades, extensive research has been conducted on SOI and InP platforms, which points out that we know more about these platforms; however, SiO_2:TiO_2 is still under development and requires further research to explore its entire application range.

Table 1: Comparison SiO_2:TiO_2 platform with other platforms.

Features	**SOI**	**InP**	**SiO_2:TiO_2**
Refractive index	3.42	3.4	1.81 to 2.2
Spectral range	1.1-1.65	NIR	VIS-NIR
Technological maturity	High	High	Under development
Fabrication Method	Wafer Bonding	LP MOCVD	Sol-gel
Cost efficiency	Very high	Moderate	Very high
Refractive index tailoring	No	No	Yes (1.2-2.2)
Applications	Telecom, MEMS, Sensors	Telecom	Sensors, special applications

ICP-RIE is a selective dry etching process used in the fabrication of various semiconductor devices. Etching is employed to create non-planar microstructures such as pits, mesa formations, and sidewalls with a controlled slope [15]. The ICP-RIE approach makes use of plasma events and ion interactions with the etched material. The collision of electrically neutral atoms with electrons accelerated in the electric field produces reactive plasma because of the intense discharge in the etching gas. Chemically active radicals (uncharged atoms or molecules), ionized atoms, excited atoms, undissociated atoms (molecules), and free electrons make up the plasma. The ICP-RIE is well-developed, easy to replicate, and commercially available. However, it's extremely expensive and demands a cleanroom environment.

Wet chemical etching is the process of chemically removing a material with a liquid reactant or etchants. It may contain a chemical that dissolves the material to be scraped, or it may use a chemical mixture that first oxidizes the material and then dissolves the oxide. Materials not protected by masks are worn away by liquid chemicals. Chemical etching takes place in three basic stages. I) Diffusion of liquid etch into the structure to be removed, II) The reaction between the liquid etcher and the etched material, III) Diffusion of by-products from the reacting surface. Wet chemical etching is relatively cheap and easy to conduct. However, it's difficult to replicate due to its isotropic nature. In literature, several technological solutions, such as inductively coupled plasma-reactive ion etching (ICP-RIE) and wet chemical etching, are used to fabricate waveguide structures in SiO_2:TiO_2 platforms except for NIL. Therefore, our current focus is to promote the usage of the NIL method for the structuration of photonic devices.

Among other fabrication processes, the NIL principle is quite straightforward. A nano-scale rigid mold is pressed onto a polymeric material that is poured onto a substrate at a controlled temperature and pressure, thus creating a thickness contrast in the polymeric material. Recently, our group demonstrated the fabrication of a rib waveguide on a glass substrate via the NIL process, as shown in Fig. 3 (a–d) [16].

In comparison to conventional fabrication methods, NIL has several benefits, including high resolution, large-area patterning, affordability, and adaptability [17]. Despite its advances, NIL has certain downsides, such as the need to fabricate templates, select release agents carefully, and ensure material compatibility. The lifetime of the templates in NIL is limited due to the formation of waveguide patterns with photoresist [18]. The repeated use of the templates can lead to wear and degradation, resulting in a loss of pattern fidelity and resolution. Eventually, the templates must be replaced, adding to the overall cost of the process. This problem can be solved by using the patterns made by metals instead of photoresists. NIL is more suitable for certain materials, particularly those that can be softened or cured under pressure and temperature. This limits its compatibility with a wide range of materials. Additionally, the process may introduce stresses or damage to fragile materials, which can affect their properties or cause structural deformation.

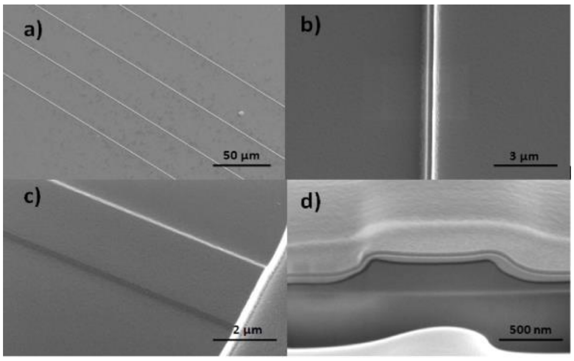

Fig. 3. SEM images of the rib waveguide fabricated via the NIL process, (a) an array of rib waveguides, (b) top-view, (c) magnified view, (d) cross-sectional view [16].

Furthermore, NIL requires a flat, smooth substrate surface for good pattern transfer. Any roughness or irregularities on the substrate can lead to imperfect imprinting or pattern distortion. This enforces stricter constraints on the substrate preparation, adding complexity and potential cost to the fabrication process [19]. However, current studies and developments in the sector are working to get beyond these restrictions and broaden the uses for this nanofabrication method. In the end, we reported a few of the most common pros and cons of the SiO_2:TiO_2 platform, as shown in Fig. 4.

Nevertheless, we believe that the SiO_2:TiO_2 platform has a huge potential, and further research is essential in the expansion of a flawless and cost-effective SiO_2:TiO_2 platform to be employed in a wide application range [20] ensuing in its commercialization soon [21].

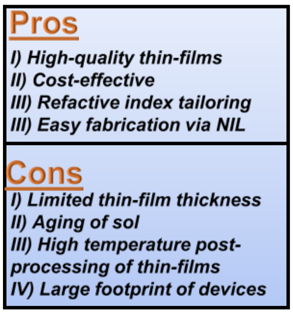

Fig. 4. Mainly known pros and cons of SiO_2: TiO_2 platform.

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