**UNIVERSIDADE DE SÃO PAULO
ESCOLA DE ENGENHARIA DE SÃO CARLOS**

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**Imageamento de banda estreita (NBI) para cápsulas
endoscópicas: filtros ópticos na fonte de luz ou no sensor
de imagem?**

São Carlos

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de imagem?**

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“Life is like riding a bicycle. To keep your balance, you must keep moving.”
Albert Einstein

ABSTRACT

GRANADO, T.C. **Narrow-Band Imaging (NBI) for endoscopic capsules: optical filters on light source or on imager?**. 2017. 104p. Dissertation (Master) - Escola de Engenharia de São Carlos, Universidade de São Paulo, São Carlos, 2017.

The Narrow-Band Imaging (NBI) technique allows the detection of lesions not identified with white light. Medical studies revealed that the wavelengths of 415 nm and 540 nm of the electromagnetic spectrum correspond to the peaks of hemoglobin absorption's band. The wavelength of 415 nm is located in the blue region of the electromagnetic spectrum, allowing the visualization enhancement of superficial veins. Moreover, the wavelength of 540 nm is located in the green region of the spectrum, and it is related to the visualization of sub-epithelial vessels. Initially, this work aims the design, simulation, fabrication (as well as establish all the ideal settings for the depositions) and characterization of optical filters for the wavelength around 415 nm and 540 nm, through the application of the Fabry-Perot principle. Additionally, this work aims the investigation of two hypotheses: the first one is test the optical filters on the image sensor, and the second one is to test them on the light sources, in order to determined which adaptation technique is better for improving the image contrast of the blood vessels.

Keywords: Narrow-band Imaging. Endoscopic Capsules. Optical filters.

RESUMO

GRANADO, T.C. **Imageamento de banda estreita (*Narrow-Band Imaging - NBI*) para cápsulas endoscópicas: filtros ópticos na fonte de luz ou no sensor de imagem?**. 2017. 104p. Dissertation (Master) - Escola de Engenharia de São Carlos, Universidade de São Paulo, São Carlos, 2017.

A técnica de *Narrow-Band Imaging (NBI)*, ou imageamento por banda estreita, permite a detecção de lesões que não podem ser identificadas com a luz branca. Estudos clínicos revelaram que os comprimentos de onda de 415 nm e 540 nm do espectro eletromagnético correspondem aos picos de banda de absorção da hemoglobina. O comprimento de onda de 415 nm localiza-se na parte azul do espectro eletromagnético, o que permite uma melhor visualização de vasos superficiais. Além disso, o comprimento de onda 540 nm localiza-se na faixa verde do espectro e está relacionado com a visualização de vasos sub-epiteliais. O intuito deste trabalho é o projeto, simulação, fabricação (assim como estabelecer os parâmetros ideais para as deposições) e caracterização de filtros ópticos para os comprimentos de onda próximos de 415 nm e 540 nm, com aplicação do princípio físico de *Fabry-Perot*. Posteriormente, pretende-se investigar duas hipóteses: a primeira é a de testar os filtros ópticos no sensor de imagem, e o segundo é a de testá-los nas fontes de luz, para verificar qual técnica de adaptação seria melhor para aumentar o contraste entre os vasos sanguíneos.

Palavras-chave: Imageamento de banda estreita. Cápsulas Endoscópicas. Filtros ópticos.

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LIST OF ABBREVIATIONS AND ACRONYMS

AFI	Autofluorescence Imaging
CCD	Charge-Coupled Device
CCS	Center of Semiconductor Components
CE	Capsule Endoscopy
CMOS	Complementary Metal-Oxide-Semiconductor
CT	Computadorized Tomography
DI	Deionized
EC	Endoscopic Capsule
FWHM	Full Width at Half Maximum
GI	Gastrointestinal
H_2O	Water
HCl	Hydrochloric Acid
HF	Hydrofluoric Acid
H_2O_2	Hydrogen Peroxide
H_2SO_4	Sulfuric Acid
INCA	National Cancer Institute
InGaN	Indium Gallium Nitride
IQ	Chemistry Institute of Araraquara
IQSC	Chemistry Institute of Sao Carlos
LED	Light Emitting Diode
LSI	Laboratory of Integrable Systems
MRI	Magnet Resonance Imaging
MS	Health Ministry
NBI	Narrow-Band Imaging

NH_4OH	Ammonium Hydroxide
P	Phosphorus
PDT	Photo-dynamic Therapy
RC	Resonance Cavity
RGB	Red-Green-Blue
SEM	Scanning Electron Microscope
SIM	System of Informations of Mortality
UICC	International Unity Against Cancer
USB	Universal Serial Bus
USP	University of Sao Paulo
WLB	White Light Videobronchoscopy
WHO	World Health Organisation

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1 INTRODUCTION

This chapter introduces the endoscopic techniques, followed by the evolution of the conventional endoscopy and finishing with the capsule endoscopy. The chapter focuses on the optical technology applied to the endoscopic techniques, such as NBI (Narrow-Band Imaging), and how this technology can be incorporated to the capsule endoscopy.

1.1 Endoscopic Techniques

Endoscopic techniques are methods of diagnosis and treatment of diseases used by health professionals. It is important to remember that the endoscopic techniques comprise several diagnostic areas, namely the gastrointestinal tract, respiratory tract, urinary tract, female reproductive system, body cavities, among others. The endoscopic techniques have become essential in the diagnoses (SHAHIDI et al., 2002), and also in minimally invasive surgeries, such as laparoscopy (LEE et al., 2013), which offers low associated risk for the patient. The endoscopy was shown to be superior in the sense of offering low risk to the patient, when compared to other imaging techniques, namely Computed Tomography (CT) requiring X-ray emission, or Magnetic Resonance Imaging (MRI) (NAGARAJAN et al., 2013), requiring the generation of magnetic fields of high intensity (SAVAGE, 2013).

The gastrointestinal (GI) tract endoscopy technique is divided into upper endoscopy and lower endoscopy, allowing access and visualization of some parts of the GI tract, such as the esophagus, stomach and some limited parts of the intestine. In this context, Figures 1.1 (a) and (b) illustrate two types of endoscopic procedures, i.e. upper endoscopy (CHIU, 2013) and lower endoscopy, known as colonoscopy (KAHI et al., 2014). It can be noted in the Figures 1.1(a) and (b) that there is a large region inaccessible to the endoscopic procedure. This restriction limits the possibility of detecting common anomalies such as bleeding, ulcers and tumors, while they are in the early stages. In this way, the opportunity to control or even cure diseases without complex treatments is lost, in many clinical cases (PAN; WANG, 2011).

The lack of accurate diagnosis often results in the degradation of symptoms and the rapid evolution of the disease (CAFFREY et al., 2008). To address this problem in 2000, the concept of endoscopic capsule (EC) was developed. This concept revolutionized gastroenterology and simultaneously promoted a new diagnostic perspective for some parts of the GI tract that previously could only be achieved through surgery (as it is the case of certain parts of the small intestine) (IDDAN et al., 2000). Figure 1.1 (c), shows the EC range in the small intestine, where most of the time it is not possible to be reached with the conventional endoscopes.

Figure 1.1 – Diagnostic coverage areas with conventional endoscopy and endoscopic capsule; (a) Upper endoscopy; (b) Lower endoscopy; (c) Reach of the endoscopic capsule in areas where the conventional endoscopic is not possible.

Source: Own authorship.

The EC is a biomedical device with reduced dimensions and tablet-like ([QURESHI, 2004](#)). Figure 1.2 illustrates the components that constitute a EC. It incorporates a camera, a lighting system and a wireless transmission, thus allowing for minimally invasive diagnoses compared to traditional upper or lower endoscopy examinations (which require patient preparation). As an example, the small bowel diagnostic test for the detection of Crohn's disease (associated with inflammatory bowel disease) is of high quality when performed with the EC ([ROSA et al., 2010](#); [SCHULMANN; HOLLERBACH; SCHMIEGEL, 2003](#); [VODERHOLZER et al., 2005](#)), when compared to the conventional endoscopic. The EC can reach intern parts of the small bowel that the conventional endoscope cannot, leading to a better diagnostic.

Approaches to the control of locomotion have been published, among them the work presented by Carpi F. *et al* ([CARPI; GALBIATI; CARPI, 2007](#)), which proposes a practical solution with the use of a complete EC locomotion platform using magnetic field. This solution does not increase the size of the EC and features an outer magnet supported by an articulated arm controlled by the physician. In this way, it is possible to obtain a higher density of EC images in pathological sites of the GI tract.

Moreover, the locomotion control of the EC opens new perspectives for functionalities such as Narrow-Band Imaging (NBI) ([RASTOGI et al., 2008](#)), Photo-Dynamic Therapy (PDT) ([DOLMANS; FUKUMURA; JAIN, 2003](#)), and Confocal Endomicroscopy (CE) ([KIESSLICH et al., 2006](#)), because the EC can be stopped at the exactly site of the lesion and capture better images than it would only following the peristaltic movements. Therefore, the diagnostic ability present in conventional endoscopy can be improved by the

Figure 1.2 – Components of an endoscopic capsule: (a) optical dome; (b) lens holder; (c) lens; (d) white LEDs; (e) a CMOS chip camera; (f) batteries; (g) a UHF band radio transmitter; (h) antenna.

Source:([QURESHI, 2004](#)).

innovation of optical techniques to aid in diagnosis as NBI and subsequently coupling in endoscopic capsules, which consists of the less invasive way of endoscopy of the GI tract. The commercial available ECs use white Light Emitting Diodes (LEDs) to illuminate the GI tract mucosa ([IDDAN et al., 2000](#)). However, some mucosal lesions are not visible when white light is used to make the diagnosis. The EC locomotion is passive, that is, it occurs according to the natural mobility of the food bolus conditioned by the peristaltic movements of the digestive system ([QURESHI, 2004](#)).

1.2 Narrow Band Imaging

The Narrow-Band Imaging, or NBI, is a digital optical technique for contrast enhancement of endoscopic images. The idea of NBI was conceived in May 1999 by the National Central Hospital of Japan and by *Olympus Medical Systems* (Tokyo, Japan) ([GONO, 2008](#); [GONO et al., 2004](#)). A study using a multi-spectral camera with a high power light source was carried out to confirm this idea. In this study, Kazuhiro Gono volunteered himself to do the first tests. The study revealed then that the use of the narrow band of 415 nm could increase the contrast of blood capillary images ([GONO, 2008](#)).

In 2001, Sano *et al.*, ([MACHIDA et al., 2004](#)) described the first clinical utility of NBI for GI endoscopy. The NBI technology emerged from the need of detecting lesions which were

not able to be observed in the white light. Therefore, the technique is used to increase the endoscopic image contrast by capturing real-time images and using a system composed of cameras and optical filters.

The first release of the technique was in 2005, with a system developed by Olympus Medical Systems ([GONO, 2007](#); [MUTO et al., 2009](#); [KUZNETSOV](#); [LAMBERT](#); [REY, 2006](#)). Since then, most countries that perform GI endoscopies have begun to use the combined NBI to the usual endoscopic techniques in clinical trials. There are currently two different types of NBI techniques in operation: one with a sequential Red-Green-Blue (RGB) light system with three broadband optical filters that thus cover all the wavelengths of the visible spectrum, while the other uses a Charge-Coupled Device (CDD) camera with small color filters for each pixel. Both models use a xenon-lamp ([GONO, 2007](#); [MUTO et al., 2009](#)).

1.2.1 Technique

The NBI is a real-time image capture technique developed over the last few decades. In this technique, the optical filters are positioned on top of the endoscopic light to create narrow-bands of wavelengths of the light spectrum in the blue color (in the range of 400-430 nm centered at 415 nm) and in the green color (in the range of 530-550 nm centered at 540nm) ([BROEK](#); [FOCKENS](#); [DEKKER, 2007](#)).

Figure 1.3 presents the configuration of a conventional system with white light compared to a NBI system. The system is composed of a light source, usually xenon, a rotative RGB filter, and a NBI filter. In the upper configuration, white light passes directly through the RGB rotating filter, thus generating a broadband light spectrum in the range of blue, green and red. In the lower configuration, the white light first passes through the NBI filter and subsequently the RGB rotating filter, thus selecting two narrow-bands of the light spectrum: blue and green ([KAZUHIRO, 2015](#); [GONO, 2008](#)).

The different wavelengths have different behaviours in the biological tissues. When light penetrates a biological tissue, part of it is reflected off the surface while the other part is diffused. When it comes to particles such as cell nuclei and organelles, multiple light scattering occurs. As a result, light propagates diffusely in the tissue. This propagation is determined by the length of the incident wave.

As the red light has broad wavelength, its diffusion is also wide. The blue light with shorter wavelength has a smaller diffusion range (Figure 1.4). Some of the scattered light can be absorbed by the blood. More specifically, the hemoglobin protein present in the blood is a type of chromophore capable of absorbing light with wavelengths in the blue and green range. This is because the hemoglobin absorption peaks are located at the wavelengths of 415 nm and 540 nm. For this reason, the NBI system uses wavelengths centered at 415 nm and 540 nm to coincide with the two absorption peaks of hemoglobin ([KAZUHIRO, 2015](#); [GONO, 2008](#)).

Figure 1.3 – Scheme of the NBI system compared to the white light system.

Source: Adapted from (KAZUHIRO, 2015).

Structures such as blood vessels have high hemoglobin content absorb light at wavelengths of 415 nm and 540 nm. In this way, they appear darker, creating greater contrast between the surrounding mucosa, which appears brighter when it reflects light. The blue light, with a wavelength of 415 nm from the electromagnetic spectrum, allows obtaining a superficial image of the mucosa, in which the network of superficial capillaries can be observed.

The green light, wavelength at 540 nm, allows a deep penetration into the mucosa, thus allowing observation of the subepithelial vessels (GONO, 2007; KUZNETSOV; LAMBERT; REY, 2006; SONG et al., 2008). Figure 1.5 illustrates the concept of the NBI technology with the green light (540 nm) and the blue light (415 nm) in contact with the tissue. It can be observed that the blue light has much shallower penetration than the green light.

The endoscopic investigation using NBI can be limited in cases of stagnant saliva, sticky mucus or a very thick layer of hyperkeratosis (excessive protein production, such as keratin, causing the mucosa to harden). In such situations, an accurate diagnosis of the mucosal surface may be impossible to observe, and in this way the advantages of the NBI technique may be lost (LUKES et al., 2013).

1.2.2 Applications

The application of the NBI technique is present in different areas of medicine. Although most of the applications are geared towards the detection of cancer in the GI tract (KARA;

Figure 1.4 – Wavelengths related to each color. A long wavelength is related to the reddish appearance, while a short wavelength is related to the bluish appearance.

Source: Adapted from (GONO, 2008).

BERGMAN, 2006; TANAKA; SANO, 2013; MACHIDA et al., 2004; CAPELLE et al., 2010; EAST et al., 2008; AL., 2005; MUTO et al., 2010; RUBIN; HEINRICH; CORLESS, 2007; STAMATAKOS et al., 2009), because they were the first attempts to apply the NBI technique, there are also promising studies in the diagnosis in other areas, such as bronchoscopy (HERTH et al., 2009) and also in gynecology (KISU et al., 2012).

Subsequently, the areas in which NBI tests have shown promising results in the improvement of diagnosis are indicated. However, it is important to emphasize that the focus of this dissertation is the application of the NBI technique in the detection of GI tract anomalies, due to the intention of integrating the NBI technique into endoscopic capsules.

1.2.2.1 Gynecology

The NBI technique showed great utility in the diagnosis of endometrial lesions and endometriosis, during experimental results in the area of gynecology. In gynecological endoscopy, the technique allows the contrast enhancement of the microstructures and capillaries of the uterine mucosa, in order to maximize the visual identification of lesions (KISU et al., 2012). Up to 2012, NBI technique was usually combined with hysteroscopy (uterine cavity examination) to identify endometrial lesions, and laparoscopy (minimally invasive surgery to operate structures and organs of the abdominal cavity) to study

Figure 1.5 – Illustration of the concept of the NBI technique in interaction with human tissue, by varying the wavelength of the incident light.

Source: Own authorship.

peritoneal lesions.

Figure 1.6 shows the difference between two images of endometrial cancer. In the Figure 1.6 (b) is possible to identify the capillaries vessels on the endometrial surface (darker color), which do not appear in the white light examination (Figure 1.6 (a)). Thus, the NBI technique proved to be superior to the conventional endoscopy because it allows the visualization of microvascular structures and atypical vessels in malignant lesions.

Although the studies about NBI with application in this area are advancing and have positive results, as shown in (ERCAN et al., 2011; FANFANI et al., 2010; FANFANI et al., 2011; FARRUGIA; NAIR; KOTRONIS, 2007; FUJII et al., 2010; KURODA et al., 2010; SURICO et al., 2010), large-scale studies are still required to confirm the validity in gynecological applications. The excellent detection of endometrial lesions further suggests that this method has great potential for a new approach in optical biopsy.

1.2.2.2 Bronchoscopy

In the case of lung cancer, it is believed that there is a possibility of cure of the disease, when the detection is done in cells of significant size in the intra-epithelial stage. Herth et al. (HERTH et al., 2009) compared three different technologies in bronchoscopy: NBI, Autofluorescence Imaging (AFI) and White Light Video-bronchoscopy (WLB), in order to analyze the effect of each technique in the diagnosis of pulmonary neoplasia in the

Figure 1.6 – Comparison of the visualization of endometrial cancer: (a) image obtained by hysteroscopy with white light; (b) image obtained by hysteroscopy combined with NBI. With NBI technique, salient papillary lesions and flexed atypical vessels can be observed (green coloration).

Source: (KISU et al., 2012).

pre-invasive stage. Figure 1.7 shows the comparison of the results with (a) white light investigation and (b) NBI investigation. The image from the AFI technique is not showed.

Figure 1.7 – Comparison of anormal finding: (a) image obtained in white light mode; (b) image obtained in NBI mode shows abrupt termination of the veins. The result of the biopsy of the same areas resulted in carcinoma.

Source: (HERTH et al., 2009)

The study showed that the detection of squamous angiogenic dysplasia by NBI, using rigorous criteria of bronchoscopy, may be more advantageous than the AFI and WLB in the early detection of lung cancer. The NBI technique seems to increase the specificity of the diagnosis without compromising sensitivity. This may perhaps be explained by the fact that angiogenesis is more specific than changes in extracellular matrix proteins and

that thickening of the superficial mucosa in the identification of intra-epithelial neoplasia (HERTH et al., 2009).

1.2.2.3 Diagnosis of the gastrointestinal tract

It is necessary to understand the parts of the GI system and the organs that are part of it, before trying to understand the GI cancer. The cancer can occur anywhere in the GI system, from the esophagus to the anus. The cancer developed in parts coated with glandular cells of the GI tract is called adenocarcinomas (SOCIETY, 2014). Most cancers of the GI tract, such as esophagus, stomach, colon and rectum are of this type. Other parts of the GI tract, such as the upper esophagus and the final part of the anus, are lined with squamous cells. Cancers that begin in this type of cell are called squamous cell carcinoma (SOCIETY, 2014).

It is necessary to analyze the estimated incidence of GI cancers to show the importance of the innovation of more accurate diagnostic techniques in the early stages of the disease. The Ministry of Health (*Ministério da Saúde - MS*), together with the Secretary of Health Surveillance, annually carries out the process of collecting data on deaths in Brazil with support from the Mortality Information System (*Sistema de Informações de Mortalidade - SIM*). Table 1.1 is based on data from 2013 of SIM and estimates from the National Cancer Institute (*Instituto Nacional do Câncer - INCA*) from 2014. It shows the number of deaths from the various types of GI cancer of the year 2013, as well as the estimates of new cases for 2015.

Table 1.1 – Numbers of deaths by type of cancer in the GI tract in Brazil in 2013 and estimates of new cases for 2015.

Cancer Site	Number of deaths in 2013(SIM)		Estimates for 2015 (INCA)		Estimates for 2016/17 (INCA)	
	Men	Women	Men	Women	Men	Women
Stomach	9142	5040	12870	7520	12920	7600
Esophagus	6203	1727	8010	2770	7950	2860
Colorectal	7387	8024	15070	17530	16660	17620
Anus	106	242	*	*	*	*

Each type of GI cancer and the global and national incidence rates are detailed as following, as well as the relationship between the incidence of a particular cancer and the risk factors and the main diagnostic techniques used in the detection of tumours.

Gastric Cancer: the latest worldwide estimate of stomach cancer has resulted in approximately one million new cancers in 2012, making it the fourth most frequent type in men (631 thousand new cases) and the fifth most frequent type in women, totaling 320

thousand new cases. The risk of stomach cancer is directly associated with dietary habits such as food additives and high sodium intake. In addition, it was also associated with prolonged infection by the bacterium *H. pylori*, in which the estimate showed that in 82.5% of the patients with stomach cancer there was the presence of this bacterium. This type of cancer is usually detected by conventional endoscopy (PARKIN; BRAY; DEVESEA, 2001; NOGUEIRA et al., 1993).

Esophageal cancer: esophageal cancer affects more than 450 thousand people in the world each year (FACINA, 2014). Studies have shown that alcohol and tobacco use are risk factors for esophageal cancer in both sexes (CASTELLSAGUE et al., 1999; SCHLECHT et al., 2001; GUERRA et al., 2005). It is usually detected by conventional upper endoscopy.

Colo-rectal cancer: these tumors usually start from polyps and benign lesions that grow on the inner wall of the large intestine. In Brazil, INCA's 2014/2015 estimate was 15070 new cases of colon and rectum cancer in men and 17530 in women. Usually this cancer is detected by colonoscopy (FACINA, 2014).

Although there are already techniques for the detection of tumors in the GI tract (esophagogastroduodenoscopy, colonoscopy, enteroscopy), many of them are not very efficient in the quality of contrast of the image. The NBI technique is a type of image enhancement that is usually used to examine the GI tract. The studies of (KARA; BERGMAN, 2006; FERREIRA, 2012; KUZNETSOV; LAMBERT; REY, 2006; CAPELLE et al., 2010) show, for example, that after detection of an endoscopic lesion, the NBI contrast enhancement technique is then used. The NBI technique has been shown to be effective primarily in the detection of metaplasia and dysplasia of Barrett's esophagus disease. Figure 1.8 shows that although the image acquired by the conventional endoscopy technique is brighter, the contrast offered using the NBI technique is better.

The NBI technique was also effective in the diagnosis of colo-rectal cancer, especially in the identification of colo-rectal polyp grooves (MACHIDA et al., 2004; EAST et al., 2008). As the peaks at 415 nm and 540 nm correspond to the bands of hemoglobin absorption frequencies, it is possible to highlight the presence of vascular patterns, and even detect valleys as structures (TANAKA; SANO, 2013). This process of pattern recognition was identified by a Japanese consensus. Because the NBI technique uses high contrast imaging to detect tissue vascularization, it was possible to observe the difference between normal and injured tissue (TANAKA; SANO, 2013).

In particular, squamous cell carcinomas that appear on the epithelial surface of the esophagus are usually difficult to identify. Therefore, the study from (MUTO et al., 2010) revealed that the index of successful diagnoses increased with the use of the NBI technique. The estimate of this study showed a sensitivity of 100% of cancer detection for the regions at the neck and head level, and 97.2% for the esophagus. The accuracy of the NBI technique for the diagnosis of superficial cancer was 86.7% for the areas at the neck and head level,

Figure 1.8 – Intestinal metaplasia in a short segment of the Barrett’s esophagus: (a) image obtained with the broadband filter and enlargement presents poor contrast in the epithelial ridges (arrow); (b) image obtained with NBI and magnification in the same area (arrow). Intestinal metaplasia is characterized by long sinuous epithelial ridges, separated by grooves.

Source: INCA ([KARA](#); [BERGMAN](#), 2006).

and 88.9% for the esophagus.

1.3 Motivation

It can be clearly observed from the study of extensive literature, that the NBI technology increases the diagnostic capacity of several pathologies, such as: real-time characterization of polyp histology ([HIRATA et al., 2007](#); [RASTOGI et al., 2009](#)), classification of specific patterns of Barrett’s esophagus ([GONO et al., 2004](#)), assessment of ulcerative colitis ([MATSUMOTO et al., 2007](#)), early detection of lung cancer ([HERTH et al., 2009](#)) and also detection of endometrial lesions ([ERCAN et al., 2011](#); [FANFANI et al., 2010](#); [FANFANI et al., 2011](#); [FARRUGIA; NAIR; KOTRONIS, 2007](#); [FUJII et al., 2010](#); [KURODA et al., 2010](#); [SURICO et al., 2010](#)).

With the advent of the wireless endoscopy capsule, the gastroenterology was revolutionized and new perspectives of diagnosis into the GI tract have raised. Thus, when it comes to the application of the NBI technique in the diagnosis of diseases of the GI tract, its inclusion in the EC combines the inherent advantages of endoscopic capsule diagnosis with improved visualization of the vascular patterns of the NBI technique.

In the context of NBI technology, there are two options for its implementation in the capsule endoscopy: it can be used commercial LEDs and then adapt them with optical filters, or the LEDs can be fabricated with the transmittance peaks at the wanted wavelength. The fabrication of optical filters offers a much cheaper alternative to the idea of fabricating LEDs with exactly transmittance peaks. Considering the scenario described before, it was decided to go for the option of fabricating the optical filters for adaptation into

Figure 1.9 – Comparison of a colo-rectal adenomatous polyp (with a high risk of developing into cancer) showing the vascular patterns: (a) image obtained by conventional colonoscopy; (b) image obtained by chromoendoscopy (application of dye by aerosolization); (c) image obtained by NBI. In Figure (c) it is possible to observe the vascular patterns and the epithelial layer that covers them.

Source: (TANAKA; SANO, 2013).

the commercial endoscopic capsules. For that matter, different methods and processes have been tried until the final functional version of the filter was reached. It is intended to fabricate a technological solution based on photonics for the integration of NBI technology in endoscopic capsules already existing in the market, without modifying them constructively in terms of design.

It is hoped that this solution will allow more accurate diagnosis of several pathologies, as well as the therapeutic intervention in less accessible places of the GI tract, as is the case of the small intestine. Figures 1.10 and 1.11 show the schematic design for the integration of the optical filters as a photonic module that can be adapted in the endoscopic capsules. The solution consists into two optical filters of dielectric materials TiO_2 (titanium dioxide) and SiO_2 (silicon dioxide) fabricated on top of glass substrate, which combined with light sources, result into two light beams centered at 415 nm and at 540 nm.

The idea of fabricating optical filters instead of using LEDs whose peak transmittance are at the wavelengths of 415 nm and 540 nm is due to two main reasons: the manufacture of LEDs with peak transmittance at specific wavelengths is much more expensive than using

Figure 1.10 – Scheme of how the NBI filters can be integrated in the capsule's photonic module.

Source: Own authorship.

Figure 1.11 – Side view of the NBI photonic module schematic and how the fabricated optical filters can be adapted to it.

Source: Own authorship.

LEDs already sold commercially and adapting them with optical filters; it was intended to alter as little as possible the constructive model of commercially available endoscopic capsules, which already contain white LEDs.

Besides the clinical reasons, the importance of this work also refers to the adaptation of

the commercial devices that use white light without changing its constructive form and its electronic.

1.4 Organization

This master thesis is organized into four chapters. This first chapter consisted of an introduction about imaging techniques, that can be applied to endoscopy, as well as a brief introduction about NBI technology and its major applications. Moreover, this chapter also is the dissertation summary and presents the primary motivation of this work. Chapter 2 describes the theory associated to the optical components, such as electromagnetic spectrum of light, thin-film optical filters and light sources. Chapter 3 presents the technology involved in the fabrication process of the optical filters. Chapter 4 shows the results of the characterization of the optical filters, as well as the initial tests involving the two hypotheses of this work: optical filters on the image sensor or on the light sources.

2 OPTICAL COMPONENTS

2.1 Introduction

This chapter presents the theoretical base about the light as an electromagnetic spectrum, thin-films optical filters, as well as the materials used in their fabrication. Moreover, the optical filters simulations will be also presented.

2.2 Spectral analysis of visible light

James Maxwell (1831-1879) was the responsible for making a complete mathematical description about the electromagnetic character of the light. The most acceptable theory about the light propagation is that light propagates as a wave with constant velocity $c = 3 * 10^8$ m/s in the free space. However, the propagation is different in other mediums, depending on the radiation frequency (SALEH; TEICH, 1991).

Light consists of an electromagnetic phenomenon described with the same theoretical foundation that explains all forms of electromagnetic radiation. Electromagnetic radiation behaves as electromagnetic waves and as particles, the photons. The distance between two adjacent wave peaks can be defined as wavelength (λ) of the light. The λ is given in meter(m). The frequency f of the electromagnetic wave is the number of oscillations that occur during one second. The wavelength and frequency are correlated to the Equation 2.1, where c is the light velocity in the medium given in m/s and λ_0 is the wavelength in vacuum.

$$\lambda_0 = \frac{c}{f} \quad (2.1)$$

The complete range of the electromagnetic spectrum is presented in Figure 2.1.

The range of an electromagnetic spectrum comprises the gamma rays and also the radio waves. Light occupies a space in the electromagnetic spectrum which consists of three bands: ultra-violet (from 10 to 390 nm), visible (from 390 to 760 nm) and infrared (from 760 to 1 mm) (SALEH; TEICH, 1991). Table 2.1 shows the approximate values of the wavelength bands that each color of the visible spectrum of the light comprises.

2.3 Fundamentals of the electromagnetic propagation

The basis for theoretical analysis on the electromagnetic spectrum is to study the Maxwell equations, which analyze the behavior of electromagnetic flat waves, i.e, they translate the relation existing in the electric and magnetic fields. Considering a uniform and isotropic medium (where there are uniform physical properties, and in which the refractive index

Figure 2.1 – Representation of the electromagnetic spectrum. The spectral range of light comprehends the wavelengths from 10nm (Ultraviolet rays) up to 1mm (Infrared rays).

Source: ([LABORATORIES](#),).

Table 2.1 – Approximated ranges of wavelength for different colors in vacuum.

Color	Wavelength (nm)
Violet	380 a 455
Blue	455 a 492
Green	492 a 577
Yellow	577 a 597
Orange	597 a 622
Red	622 a 780

does not vary with the orientation of the field), the Equations 2.2, 2.3, 2.4 and 2.5 are given:

$$rot(\mathbf{E}) = -\frac{\partial \mathbf{B}}{\partial t} \quad (2.2)$$

$$rot(\mathbf{H}) = \mathbf{j} + \frac{\partial \mathbf{D}}{\partial t} \quad (2.3)$$

$$div(\mathbf{D}) = \rho \quad (2.4)$$

$$div(\mathbf{B}) = 0 \quad (2.5)$$

where $\mathbf{E}$ is the electric field (or electric field intensity), $\mathbf{H}$ is the magnetic field (or magnetic field intensity), $\mathbf{D}$ is the electric displacement field and $\mathbf{B}$ is the magnetic flux density, j represents the electric current density and ρ the electric charge density. All symbols in bold represent vectors and the others are scalar. The J , $\mathbf{D}$ and $\mathbf{B}$ are defined from Equations 2.6, 2.7 and 2.8:

$$\mathbf{j} = \sigma \mathbf{E} \quad (2.6)$$

$$\mathbf{D} = \varepsilon \mathbf{E} \quad (2.7)$$

$$\mathbf{B} = \mu \mathbf{H} \quad (2.8)$$

$$\varepsilon = \varepsilon_r \varepsilon_0; \varepsilon_0 = 8.854185 \times 10^{-12} \text{ Fm}^{-1} \quad (2.9)$$

$$\mu = \mu_r \mu_0; \mu_0 = 4\pi \times 10^{-7} \text{ Hm}^{-1} \quad (2.10)$$

where ε is the dielectric constant (or electric permittivity) and μ is the magnetic permittivity. In the Equations 2.9 and 2.10, ε_0 and μ_0 represent the permittivity and permeability in free space, while ε_r and μ_r are the relative permittivity and permeability of the medium, respectively. If the medium is anisotropic, the Equations 2.2 and 2.8 become more complicated, by the fact that the symbols σ , ε , and μ are tensors rather than scalars. The Maxwell equations can still be solved in order to obtain the wave equations. For this analysis, it is considered a medium free of electric and magnetic field sources, which means $j = 0$ and $\rho = 0$. Equations 2.11 and 2.12 are obtained from the solutions of the previous Equations 2.3, 2.4, 2.7 and 2.8.

The Maxwell equations for a free medium show that there are a coupling between the electric and magnetic fields, i.e., the variations in the electric field act as source of magnetic field, which consequently acts as a source of electric field and so on. In this way, the electric and magnetic fields can be analyzed with a single entity, called the electromagnetic field (FERREIRA, 2012; MACLEOD, 2010).

$$\text{lap}(\mathbf{E}) = \mu \varepsilon \frac{\partial^2 \mathbf{E}}{\partial t^2} \quad (2.11)$$

$$\text{lap}(\mathbf{B}) = \mu \varepsilon \frac{\partial^2 \mathbf{B}}{\partial t^2} \quad (2.12)$$

The light velocity v in a given medium can be related to the wave Equations 2.11 and 2.12) through the Equation 2.13.

$$v = \frac{1}{\sqrt{\mu\varepsilon}} \quad (2.13)$$

For a generic medium, the wave impedance Z is defined as the ratio between the amplitudes of electric and magnetic fields:

$$Z = \frac{E}{H} \quad (2.14)$$

In vacuum, this impedance is approximately $Z_0 = \frac{E_0}{H_0} = \sqrt{\frac{\mu_0}{\varepsilon_0}} = 376,73\Omega$ and is called the characteristic vacuum impedance. The refractive index of a dielectric medium is the ratio between its Z-wave and vacuum impedances (Equation 2.15).

$$n = \frac{Z}{Z_0} \quad (2.15)$$

The refractive index can also be expressed by

$$n = \frac{c}{v_p} = \sqrt{\frac{\mu\varepsilon}{\mu_0\varepsilon_0}} \quad (2.16)$$

where v_p is the propagation velocity of the electromagnetic wave in a different medium than the vacuum. For this reason, v_p has a lower value than the one observed in vacuum ($v_p < c$), so that the refractive index is $n > 1$. By definition, for the vacuum is $n = 1$. Generally, the magnetic properties of a medium has a negligible effect on the speed of light propagation. Thus, it can be considered $\mu = \mu_0$ (FERREIRA, 2003). Therefore, the refractive index of the medium, n , depends basically on the permittivity ε , which depends on the frequency of the incident wave. Equations 2.17 and 2.18 represent the behavior of light as wave.

$$\mathbf{E} = Re\{E_0 e^{i(\omega t - \mathbf{k} \cdot \mathbf{z} + \phi)}\} \hat{x} \quad (2.17)$$

$$\mathbf{B} = Re\{B_0 e^{i(\omega t - \mathbf{k} \cdot \mathbf{z} + \phi)}\} \hat{y} \quad (2.18)$$

where Re indicates the real part, E_0 and B_0 are the amplitudes of the electric and magnetic field oscillations, respectively. ω represents the angular frequency of the wave, ϕ is the wave phase, $\mathbf{z}$ the position vector and $\mathbf{k}$ is the wavevector, defined by the Equation 2.19:

$$|\mathbf{k}| = k = \frac{2\pi n}{\lambda_0} = k_0 n \quad (2.19)$$

where k is the wave number, k_0 is the wave number in vacuum, and λ_0 is the wavelength in vacuum. Moreover, $\mathbf{E}$ and $\mathbf{H}$ are contained in the planes xoz and $yo z$, respectively. This means that $\mathbf{E}$ and $\mathbf{H}$ are orthogonal to each other and z is the direction of the wave propagation. The frequency f , and the wavelength, λ_g , are also defined by the Equations 2.20 e 2.21:

$$f = \frac{\omega}{2\pi} \quad (2.20)$$

$$\lambda_g = \frac{2\pi}{k} = \frac{\lambda_0}{n} \quad (2.21)$$

where λ_g is the wavelength in a medium other than vacuum. It is interesting to note that an electromagnetic wave has its wavelength decreased as it propagates in a medium with growing refractive index n . In a generic medium, it can be considered an important term for thin-film optical structures: the optical admittance, which unit is siemens [S]. This concept defines how easily the light can pass through them. The optical admittance is then defined by Equation 2.22:

$$Y = \sqrt{\frac{\varepsilon}{\mu}} \quad (2.22)$$

for the specific case of vacuum, the admittance is:

$$Y_0 = \sqrt{\frac{\varepsilon_0}{\mu_0}} \quad (2.23)$$

and finally, the admittance of a generic medium can also be expressed as:

$$Y = \frac{Y_0}{n} = 2.6544 \times 10^{-3} S \quad (2.24)$$

The optical admittance of a material is numerically equal to its refractive index n . The optical admittance, or the refractive index, connects the electric and magnetic fields, through the following expression:

$$n = \frac{H}{E} \quad (2.25)$$

2.4 Thin-films interface equations

It is necessary to study each layer of thin-film, the interface created between them, and how the light propagates through them, in order to understand a multilayer thin-film structure. Generally, a thin-film is defined by a material layer with thickness up to 1 μm . The thin-film can be obtained by depositing a given material on a substrate like glass, quartz and others.

The structure of a thin-film with more than one boundary/interface, is represented by Figure 2.2, in which the main components are defined so that the structure is formed by: incident medium, thin-film structure and substrate. In order to analyze the propagation of light through the thin-film, it is convenient to establish that the symbol (+) represents the waves propagating in the same direction of the incident wave (positive sense), and the symbol (-) represents the waves propagating in opposite direction (negative sense) (FERREIRA, 2012; MACLEOD, 2010).

Figure 2.2 – Flat wavefront incident on a thin-film structure. Adapted from (MACLEOD, 2010).

There are no waves that propagate in the negative sense in the substrate considering an interface between the thin-film and substrate (Boundary b) of the Figure 2.2. Considering that, the waves on the thin-film can be summed up as the result of all waves propagating in the positive sense and the resultant of all waves propagating in the negative one. Therefore, the tangential components of electrical and magnetic field in Boundary b can be defined by the Equations 2.26 and 2.27, respectively. The index 1 refers to the medium with refractive index n_1 .

$$E_b = E_{1b}^+ + E_{1b}^- \quad (2.26)$$

$$H_b = \eta_1 E_{1b}^+ - \eta_1 E_{1b}^- \quad (2.27)$$

The components E_{1b}^+ , E_{1b}^- , H_{1b}^+ , and H_{1b}^- are defined by the Equations 2.28, 2.29, 2.30 and 2.31, respectively.

$$E_{1b}^+ = \frac{1}{2} \left(\frac{H_b}{\eta_1} + E_b \right) \quad (2.28)$$

$$E_{1b}^- = \frac{1}{2} \left(-\frac{H_b}{\eta_1} + E_b \right) \quad (2.29)$$

$$H_{1b}^+ = \eta_1 E_{1b}^+ = \frac{1}{2} (H_b + \eta_1 E_b) \quad (2.30)$$

$$H_{1b}^- = \eta_1 E_{1b}^- = \frac{1}{2} (H_b - \eta_1 E_b) \quad (2.31)$$

where η corresponds to the optical admittance. For the normal incidence, $\eta = nY_0$. For the oblique incidence it is $\eta_p = nY_0/\cos(\theta)$ for the p-polarization (the wave with electric vector is on the incidence plan); $\eta_s = nY_0\cos(\theta)$ for the s-polarization (the wave with electric vector is perpendicular to the incidence plan); θ is the incidence angle.

The electric and magnetic fields at the interface between the incident medium and the thin-film (Boundary **a**), at the same instant and at any point with identical coordinates x and y , can be determined by changing the phase factor of the wave, in order to change the displacement in the coordinate z , from 0 to $-d$.

The phase factor of the waves propagating in the forward direction (positive) will be multiplied by $e^{(i\delta)}$, whereas the phase factor of the waves propagating in the backward direction (negative) will be multiplied by $e^{(-i\delta)}$, where δ , defined by the Equation 2.32, is the phase thickness and corresponds to the phase change experienced by the wave as it crosses the distance d , that is normal to the Boundary **a**.

$$\delta = \frac{2\pi N_1 d \cos(\theta)}{\lambda} \quad (2.32)$$

The distance d is the physical thickness of the thin-film, λ is the wavelength, and N_1 is the complex refractive index, $n - ik$ (n is the refractive index, and k is the absorption coefficient).

The optical path length (OPL) is defined as the distance travelled by the light in the thin-film and is expressed by Equation 2.33.

$$OPL = N_1 d \cos(\theta) \quad (2.33)$$

The tangential components of the electric and magnetic field can be defined by Equations 2.34, 2.35, 2.36 and 2.37, respectively.

$$E_{1a}^+ = \frac{1}{2} \left(\frac{H_b}{\eta_1} + E_b \right) e^{(i\delta)} \quad (2.34)$$

$$E_{1a}^- = \frac{1}{2} \left(-\frac{H_b}{\eta_1} + E_b \right) e^{(-i\delta)} \quad (2.35)$$

$$H_{1a}^+ = \frac{1}{2} (H_b + \eta_1 E_b) e^{(i\delta)} \quad (2.36)$$

$$H_{1a}^- = \frac{1}{2} (H_b - \eta_1 E_b) e^{(-i\delta)} \quad (2.37)$$

It is possible to obtain the components E_a e H_a , by combining the Equations 2.34, 2.35, 2.36 and 2.37, which correspond to the values of electric and magnetic field at the Boundary **a**, respectively.

$$E_a = E_{1a}^+ + E_{1a}^- = E_b \left(\frac{e^{(i\delta)} + e^{(-i\delta)}}{2} \right) + H_b \left(\frac{e^{(i\delta)} + e^{(-i\delta)}}{2} \right) = E_b \cos(\delta) + H_b \frac{i \sin(\delta)}{\eta_1} \quad (2.38)$$

$$H_a = H_{1a}^+ + H_{1a}^- = E_b \eta_1 \left(\frac{e^{(i\delta)} + e^{(-i\delta)}}{2} \right) + H_b \left(\frac{e^{(i\delta)} + e^{(-i\delta)}}{2} \right) = E_b i \eta_1 \sin(\delta) + H_b \cos(\delta) \quad (2.39)$$

Equations 2.38 and 2.39 can be presented in matrix notation by Equation 2.40, also called characteristic matrix of the thin-film. This matrix relationship associates the components of electric and magnetic field incident at the Boundary **a** with the same components transmitted by the final interface, i.e., Boundary **b**.

$$\begin{bmatrix} E_a \\ H_a \end{bmatrix} = \begin{bmatrix} \cos(\delta) & i \sin(\delta)/\eta_1 \\ i \eta_1 \sin(\delta) & \cos(\delta) \end{bmatrix} \begin{bmatrix} E_b \\ H_b \end{bmatrix} \quad (2.40)$$

Thus, it can be calculated the admittance (Y) of the thin-films structure, given by Equation 2.41.

$$Y = \frac{H_a}{E_a} \quad (2.41)$$

And yet the reflectance of the boundary between the incident medium with admittance η_0 , and the admittance medium Y :

$$R = \left(\frac{\eta_0 - Y}{\eta_0 + Y} \right) \left(\frac{\eta_0 - Y}{\eta_0 + Y} \right)^* \quad (2.42)$$

When the Equation 2.40 is normalized, a new matrix is obtained, showed in Equation 2.43, where NE and NH are the electric and magnetic field at the Boundary **a**.

$$\begin{bmatrix} E_a/E_b \\ H_a/H_b \end{bmatrix} = \begin{bmatrix} B \\ C \end{bmatrix} = \begin{bmatrix} \cos(\delta) & i \sin(\delta)/\eta_1 \\ i \eta_1 \sin(\delta) & \cos(\delta) \end{bmatrix} \begin{bmatrix} 1 \\ \eta_2 \end{bmatrix} \quad (2.43)$$

In that way, with the Equations 2.42 and 2.43, is possible to write a new admittance relationship Y :

$$Y = \frac{H_a}{E_a} = \frac{C}{B} = \frac{\eta_2 \cos(\delta) + i \eta_1 \sin(\delta)}{\cos(\delta) + i (\eta_2/\eta_1) \sin(\delta)} \quad (2.44)$$

Finally, the reflectance R can be then calculated by Equations 2.44 and 2.42. Equation 2.45 is known as the characteristic matrix of the thin-film multilayer structure.

$$R = \begin{bmatrix} B \\ C \end{bmatrix} \quad (2.45)$$

All the analysis previously presented was made for a simple thin-film. However, the same methodology can be applied for the case of thin-films with multilayer structure. Figure 2.3 shows a structure with two thin-films and three boundaries. In this case, the characteristic matrix of the the thin-film N_2 will be expressed by Equation 2.46

Figure 2.3 – Structure with two thin-films and three boundaries (a, b and c).

Source: (MACLEOD, 2010).

$$\begin{bmatrix} \cos(\delta_2) & i \sin(\delta_2)/\eta_2 \\ i \eta_2 \sin(\delta_2) & \cos(\delta_2) \end{bmatrix} \quad (2.46)$$

Thus, applying Equation 2.40, it can be obtained the parameters at boundaries **b** and **a** by Equations 2.47 and 2.48, respectively.

$$\begin{bmatrix} E_b \\ H_b \end{bmatrix} = \begin{bmatrix} \cos(\delta_2) & i \sin(\delta_2)/\eta_2 \\ i \eta_2 \sin(\delta_2) & \cos(\delta_2) \end{bmatrix} \begin{bmatrix} E_c \\ H_c \end{bmatrix} \quad (2.47)$$

$$\begin{bmatrix} E_a \\ H_a \end{bmatrix} = \begin{bmatrix} \cos(\delta_1) & i \sin(\delta_1)/\eta_1 \\ i \eta_1 \sin(\delta_1) & \cos(\delta_1) \end{bmatrix} \begin{bmatrix} \cos(\delta_2) & i \sin(\delta_2)/\eta_2 \\ i \eta_2 \sin(\delta_2) & \cos(\delta_2) \end{bmatrix} \begin{bmatrix} E_c \\ H_c \end{bmatrix} \quad (2.48)$$

The characteristic matrix of the multilayer structure is showed by Equation 2.49:

$$\begin{bmatrix} B \\ C \end{bmatrix} = \begin{bmatrix} \cos(\delta_1) & i \sin(\delta_1)/\eta_1 \\ i \eta_1 \sin(\delta_1) & \cos(\delta_1) \end{bmatrix} \begin{bmatrix} \cos(\delta_2) & i \sin(\delta_2)/\eta_2 \\ i \eta_2 \sin(\delta_2) & \cos(\delta_2) \end{bmatrix} \begin{bmatrix} 1 \\ \eta_3 \end{bmatrix} \quad (2.49)$$

Finally, this result can be generalized for a multilayer structure of X layers. Then, the characteristic matrix can be the basis for multiple thin-film layers calculation, and it is define by the product of the individual matrices, as shown on Equation 2.50:

$$\begin{bmatrix} B \\ C \end{bmatrix} = \left\{ \prod_{r=1}^X \begin{bmatrix} \cos(\delta_r) & i \sin(\delta_r)/\eta_r \\ i \eta_r \sin(\delta_r) & \cos(\delta_r) \end{bmatrix} \right\} \begin{bmatrix} 1 \\ \eta_m \end{bmatrix} \quad (2.50)$$

where m denotes the substrate and δ_r is:

$$\delta_r = \frac{2\pi\eta_r d_r \cos(\theta_r)}{\lambda} \quad (2.51)$$

2.5 Structure of NBI optical filters

The detection of certain wavelengths is done through the thin-film optical filters. It is necessary to understand three statements about the performance of thin-film optical devices, in order to understand them in a qualitative way.

- 1 - The amplitude of the reflected wave at any of the boundaries between two media is given by $(1 - \rho_r)/(1 + \rho_r)$, where ρ_r is the ratio of optical admittances in the boundaries, that is, in the optical region, the ratio between the media refractive indexes (n).
- 2 - There is a phase shift of 180° when the reflection occurs in a medium with a lower refractive index than the adjacent medium. There is a phase shift of 0° when the reflection occurs in a medium with a higher refractive index than the adjacent medium.
- 3 - If the reflected light divides into two components on the bottom and upper surfaces of the thin-film, the components will recombine so that the resulting amplitude will be the difference of the amplitudes if the relative phase shift is 180° , or the sum of the amplitudes if the phase shift is 0° or multiples of 360° (MACLEOD, 2010).

The fabrication of an optical filter can be made by using a multilayer thin-films structure, in which passing and rejecting spectral bands will be produced. For that purpose, the structure from Figure 2.4 must be transformed in a structure like the one presented in Figure 2.5, which has a resonance layer between two mirrors. The called Fabry-Perot filter (Figure 2.5) consists of a structure that captures and confines light of certain wavelengths and operates as an optical transmission system incorporating feedback: light circulates or is repeatedly reflected within the mirror system without escaping.

Figure 2.4 – Illustration of a thin-films multilayer structure.

Source: (MINAS, 2004).

Figure 2.5 – Illustration of a simple Fabry-Perot resonator.

Source: Adapted from (CORREIA, 1999).

The narrow-band optical filters can be built using an assembly of thin-films of high (H_n) and low refractive (L_n) indexes, alternately deposited on a transparent substrate. This assembly produces passing and rejecting bands, and for a collimated light, the transmittance is low for all wavelengths, except for a series of narrow transmission bands (which are adjusted as desired). In this work, the optical filters have a total of 7 layers of dielectric materials. The structure of Fabry-Perot interferometers (Figure 2.6) consists of two flat and parallel mirrors, which act as reflecting surfaces and are separated by a distance d , called resonance cavity.

Figure 2.6 – Structure of a Fabry-Perot optical filter.

Source: Adapted from (MACLEOD, 2010).

When a light wave reaches the upper surface of a resonance cavity, it is divided into two waves: reflected and transmitted. This transmitted wave will then reach the bottom surface of the resonance cavity and will again be divided into two waves: one transmitted and other reflected back to the upper surface of the resonance cavity. This process will be repeated along the entire Fabry-Perot filter, and the total transmitted light will be a sum of all transmitted waves.

The sum of light transmitted in the resonance layer is the sum of all beams that have been transmitted due to constructive interference, which depends on the wavelength of the incident light and the thickness of the resonance cavity. The wavelengths that reach this resonance cavity then undergo a destructive interference and are not transmitted. The Fabry-Perot filter is considered to be ideal when it is assumed that there is no loss in both mirrors and that they are perfectly parallel to each other, and also that there is no absorption in the middle of the filter (CORREIA, 1999).

According to (MACLEOD, 2010), when the reflectance of the mirrors is high enough, the total intensity (I_t) of the light transmitted by the filter is determined by the Equation 2.52

$$I_t = \frac{I_0}{1 + F \sin^2 \delta} \quad (2.52)$$

where I_0 is the intensity of the incident beam and δ is the phase thickness. The coefficient of finesse (the Fabry-Perot function F) can be determined by Equation 2.53, where R is the reflectance. As F increases, the reflectance of the mirrors also increases, and the widths of the peaks become narrower. If the peaks are narrow, light is transmitted through the Fabry-Perot assembly only if the resonance cavity thickness (d), refractive index (n) and wavelength (λ) satisfy the Equation 2.54, where q is the peak order.

$$F = \frac{4R}{(1 - R)^2} \quad (2.53)$$

$$\delta = \frac{2\pi\eta_1 d \cos(\theta)}{\lambda} = q\pi \quad (2.54)$$

For a specific separation d between the upper and bottom mirrors, the filter will transmit (ideally) only the wavelength λ that satisfies Equation 2.54, but it is important to observe that the filter will repeat the transmission pattern when δ increases by π . In order to simplify the calculations, the peak order is considered to be 1 and the incidence normal. In that way, the thickness of the resonance cavity is calculated as:

$$d_{RC} = \frac{\lambda}{2\eta} \quad (2.55)$$

where λ is the wanted wavelength to be transmitted by the Fabry-Perot filter. The thickness of the quarter-wave layers is calculated by Equation 2.56:

$$d_{QW} = \frac{\lambda}{4\eta} \quad (2.56)$$

2.6 Thin-films optical filters materials

The first step of the fabrication of thin-films optical filters is to choose the material of the substrate that will hold the optical filters. In this work different specifications of glasses were tested as substrate. The first trial was done with microscope slides, measuring 76 x 26 x 1.1 mm. It was found that the surface roughness of this substrate was very high for the application proposed in this work, around 5000 nm rms, which is also higher than some layers of thin-film. The glass substrate B20 from Schott presented surface roughness around 2 nm rms.

The structure of thin-films was fabricated using dielectric materials: silicon dioxide (SiO_2) and titanium dioxide (TiO_2). The choice of working with these materials is due to the fact that they are materials of high adherence to the substrate, which increases the durability of the thin-film. Moreover, the SiO_2 has a low refractive index when compared to TiO_2 . This fact satisfies the necessary condition for a Fabry-Perot interferometer structure, which requires layers of materials with high and low refractive index, alternately.

Both dielectric materials offer good performance in the following characteristics: high and narrow peak of transmittance spectrum, high reflectivity and low absorption losses because of their transparency in the wavelengths of interest (415 and 540 nm). Another feature offered by the SiO_2 is that its refractive index varies very little within the visible spectrum range. In addition, the chosen materials can be easily integrated into CMOS (Complementary Metal-Oxide-Semiconductor) technology, making it compatible with microelectronic manufacturing techniques.

2.7 Optical filters simulation

The design of the optical filters considered a structure in which both mirrors (upper and bottom) were composed by dielectric materials. The multilayer structure can be represented by $HnLnHnLnLnHnLnHn$, where Hn is the high refractive index material (TiO_2), and Ln is the low refractive index material (SiO_2).

The first step on the design of the filters is to calculate the approximate thickness of each layer. Considering that the aim is to fabricate two optical filters, two different designs were performed.

For the blue filter, it was considered that the wavelength of reference was 415 nm, because that where the transmittance peak should be centralized. For the green filter the reference was considered 540 nm. The analytical calculations of the thickness of each layer were done following Equations 2.55 and 2.56.

For the central wavelength of 415 nm, $\eta_{SiO_2} = 1.45$, and $\eta_{TiO_2} = 2.65$. Therefore, the thickness of the SiO_2 layers is approximately 71 nm, the thickness of the TiO_2 layers is 39 nm, and the resonance cavity spacing is 143 nm.

For the central wavelength of 540 nm, $\eta_{SiO_2} = 1.46$, and $\eta_{TiO_2} = 2.59$. Therefore, the thickness of the SiO_2 layers is approximately 93, the thickness of the TiO_2 layers is 51 nm, and the resonance cavity spacing is 186 nm.

After obtaining these results, computational simulations were performed using the software *TFCalcTM3.5* (Software Spectra, Inc., Portland, OR). This software allows the structural optimization of the optical filters, because it includes in the simulations the material properties, and also allows to introduce the illuminant (in this case the LEDs spectral transmittance), the wavelength of reference, and the detector characteristics.

Figure 2.7 shows the environment setting of the software *TFCalcTM3.5*. For all the simulations, the following features were set:

- 1) the substrate was considered the B270, from Schott;
- 2) the incident and exit medium were considered air;
- 3) the thickness of the substrate was considered 1 mm;
- 4) the detector was considered ideal;
- 5) the incident angle (deg) was considered 0° (normal incidence).

The reference wavelength and illuminant changed according to the filter (blue or green). The achievement of good simulations depends on the insertion of the material properties, such as a table of refractive index of the material along the range of wavelengths, as well as the spectral signature of the illuminant. Both characterizations details will be shown in the Chapter 4, but there were used in the simulations, in order to have the a simulation as near as possible of the real.

After repeatedly simulations and adjusts, all the thickness were chosen in way to better fit the project requirements. It was preferred to have a blue filter simulation centralized

Figure 2.7 – Illustration of environment setting of the software *TFCalcTM* 3.5.

Source: Own authorship.

at 413 nm with high transmittance, than have it centralized at 415 nm with very poor transmittance.

Figure 2.8 presents the relative transmittance for the blue filter with the blue LED as illuminant, compared to the spectral signature of the blue LED itself. It can be observed that the transmittance peak was shifted from 410 nm to 413 nm, which was closer to the desired value 415 nm.

Figure 2.9 shows the relative transmittance of the green filter, considering the green LED as illuminant, in contrast with the green LED spectral signature. It was possible to obtain a peak centered at 540 nm, with high transmittance. The transmittance peak was shifted from 533 nm to 540 nm.

After the simulations adjustment, the final thicknesses were set and are showed on Table 2.2.

Table 2.2 – Structure of the optical filters

Layer	Material	Thickness blue filter [nm]	Thickness green filter [nm]
1	TiO_2	32	56
2	SiO_2	83	95
3	TiO_2	32	56
Resonance Cavity	SiO_2	154	176
5	TiO_2	32	56
6	SiO_2	83	95
7	TiO_2	32	56

Figure 2.8 – Relative transmittance of the blue LED along with the simulation of the blue filter.

Source: Own authorship.

Figure 2.9 – Relative transmittance of the green LED along with the simulation of the blue filter.

Source: Own authorship.

3 OPTICAL FILTERS FABRICATION

This Chapter presents the techniques used for characterization and deposition of thin-film materials and optical filters. The thin-film deposition and material characterization were performed at the Center of Semiconductor Components (CCS), University of Campinas (Unicamp), Campinas. Some of the ellipsometry characterization was performed at the Integrable Systems Laboratory (LSI), USP, Sao Paulo. The measurements of spectrophotometry were performed in the Chemistry Institute of Sao Carlos (IQSC), USP, Sao Carlos.

3.1 Cleaning Techniques

Before any thin-film deposition, there is the necessity of a cleaning step, in order to avoid that any type of dirt or particles enter the machines used for the depositions, as well as to guarantee that the sample surface is completely free of impurities, like grease and ions. Two cleaning procedures were performed: the first one was the Radio Corporation of America (RCA) cleaning, and the second one was a physical cleaning. The RCA clean consists of four steps, which are listed in the following items:

- a) Step 1: consists of removing the organic contaminants. The procedure is to soak the samples into a piranha solution/etch, i.e., H_2SO_4/H_2O_2 using a (4:1) proportion at 80°C, for 10 minutes.
- b) Step 2: Oxide strip consists of removing the silicon dioxide formed during Step 1 and a fraction of ionic contaminants. The procedure is a short immersion of the samples in a solution of HF/H_2O using a proportion of (1:10) at room temperature for about 10 seconds.
- c) Step 3: Consists of removing the remaining traces of metallic (ionic) contaminants, particles and lipids. It also leaves a thin passivity layer on the surface, which protects the sample of subsequent contaminations. The procedure is an immersion of the samples in a solution of $NH_4OH/H_2O_2/H_2O$ in a proportion of (1:1:5) at 80°C for about 10 minutes.
- d) Step 4: The final ionic clean strips alkali ions and metals $HCl/H_2O_2/H_2O$ in the proportion of (1:1:5) at 80°C for 10 minutes.

Between each step, a dionized (DI) water rinsing was performed for about 3 minutes, and the sample was dried with nitrogen. The resistivity of the DI water is 18MΩ.cm .

The organic clean consists of three steps, which are listed in the following items:

- a) Step 1: detergent rinsing consists of a liquid rinsing to remove grease. The procedure is to soak the sample in detergent for about 3 minutes.

- b) Step 2: acetone ultrasonic bath for about 10 minutes.
- c) Step 3: isopropanol ultrasonic bath for about 10 minutes.

After Step 3, a dionized (DI) water rinsing was performed for about 3 minutes, and the sample was dried with nitrogen.

The basic organic clean was experimentally found to be the better cleaning technique for glasses samples, once it does not etch the sample, leaving a flatter surface when compared to RCA clean. These results were observed under the optical microscopy, but unfortunately the pictures could not be saved. When the surface is etched, the overall surface roughness increases, which affects the parallelism between the two mirrors from the stack of layers of the optical filters.

3.2 Thin-films Characterization

3.2.1 Ellipsometry

Ellipsometry can be used to determine optical constants of a reflective surface and also the thickness of a thin-film deposited on an optical substrate. Ellipsometry is normally divided in two phases: the first one consists of determining the values of ψ (psi) and Δ (delta), and the second one consists of correlating the measured values with the optical parameters, such as refractive index (n), thin-film thickness (d) and extinction coefficient (k) (COLLETT, 1992). Figure 3.1 shows the ellipsometer Rudolph used in this work.

The ellipsometry involves the measure of $\tan(\psi)$, the change in amplitude and of Δ and the change in phase. The values of ψ and Δ are functions of the optical constants of the medium, the thin-film and the substrate, the wavelength of the light, the incidence angle, and, for the optical filters deposited into substrates, the thickness. The fundamental equation of ellipsometry is defined by the Equation 3.1:

$$\rho = \tan(\psi) \exp(i\Delta) = f(n, k, d) \quad (3.1)$$

Ideally, when ψ and Δ are measured, the variables n , k and d can be determined by calculations involving iterations. In the Equation 3.1, ρ is expressed as a general function $f(n, k, d)$. The equations to measure ψ and Δ derived from the fundamental Equation 3.1, and then the other variables in the equation can be obtained as well.

Among the advantages offered in the using of ellipsometry as a method of characterization of thin-films, it can be emphasized the ease manipulation of the ellipsometer itself, combined with the fact that it is a non-destructive experiment (it does not damage the samples), and it can be performed with small sizes of samples.

The deposition parameters can be adjust based on the results from the measurements of refractive index and thickness done by ellipsometry. It is recommend to use the ellipsometry to characterized the refractive index along the range of wavelengths, but the number

Figure 3.1 – Rudolph Ellipsometer.

Source: Own authorship.

of wavelengths that can be measured depends on the lasers sources available for each equipment.

It was possible to characterize the titanium dioxide thin-films at three different wavelengths (405 nm, 630 nm and 830 nm), because the limitations of the available equipment. These measurements were performed with the ellipsometer Rudolph, at the laboratory (LSI). Unfortunately, by the time of the characterization of the SiO_2 thin-films, the equipment at LSI was not functioning and the samples with SiO_2 were only characterized at the wavelength of 630 nm, with the ellipsometer available at the laboratory CCS (Unicamp). Facing the problem of not having the appropriate machine for doing this kind of measure, it was attempted to adjust the refractive index at the wavelength of 630 nm to a value closest to the one in literature (also measured at 630 nm), in order to have the most accurate refractive index. The refractive index for the other wavelengths were found using a interpolation function, using as reference the measured value at the wavelength of 630 nm, and other values of refractive index from literature.

Figure 3.2 shows the plot of the comparison between the refractive index of the silicon dioxide (deposited by plasma enhanced chemical vapor deposition) versus the titanium dioxide (deposited by sputtering). It is important to notice that the values of the refractive indexes for other wavelengths were required in the simulation of the optical filters using the software TFCalc, thus the need of doing the interpolation.

Figure 3.2 – Refractive indexes of silicon dioxide and titanium dioxide.

Source: Own authorship.

3.3 Deposition Techniques

Every type of deposition technique must be tested and characterized before a series of depositions. These steps are crucial to the development of a good recipe and reliability of the process. The characterization consists of knowing how to manage all the parameters required to keep the machines running. It can be a very repetitive and exhausting process, since it consists of doing various depositions and changes of settings, in order to get the best recipe possible. The SiO_2 films were deposited by ECR and PECVD processes, and the TiO_2 films were deposited by a DC sputtering. The ideal scenario would be depositing both films using the same process and the same machine, in a way that all the layers would be deposited without leaving the chambers and getting air contamination. Unfortunately, it was not possible to deposit the SiO_2 thin-films by sputtering, because the chamber reserved for this material was not functioning.

3.3.1 Electron Cyclotron Resonance (ECR)

The ECR is a microwave electron cyclotron resonance technology that offers independent control of the ion energy and ion flux. The control of the ion energy is done by a Radio Frequency (RF) or a DC biasing of the substrate holder (STEVENSON, 1995). The microwave energy is coupled to a natural resonant frequency of the electron gas in the presence of a static magnetic field. The resonant frequency is established when the electron cyclotron frequency equals the excitation frequency. This technology consists on creating a low-pressure and low-temperature discharge (in the range of <0.5 Torr), and it does not depend on having an electrode (anode and cathode).

The ECR technology can be used in etching, thin-films deposition and also broad ion beam applications. In this project, it was used for thin-films deposition. The ECR machine depends on a microwave power (frequency 2.45 GHz) to run. This power is then introduced in the plasma chamber passing through a waveguide and a window made of a fused quartz. The plasma chamber operates as microwave cavity resonator, and around its periphery are arranged the magnetic coils for ECR plasma excitation (ASMUSSEN, 1989; MATSUO; KIUCHI, 1983).

The deposition of SiO_2 thin-films was first tested using the ECR process. The depositions were performed at the Device Research Laboratory (LPD), Unicamp. The machine used in this process is shown in Figure 3.3. The deposition rate was around 5.7 nm/min, by using this technique. From six samples of SiO_2 thin-films, the refractive index varied from 1.550 to 1.563 measured at the wavelength of 630 nm. The characterization of the SiO_2 thin-films was made by ellipsometry, and the refractive index used as parameter for the iterations was 1.462. However, the real refractive index varied from the results found in literature (GAO; LEMARCHAND; LEQUIME, 2012; GAO; LEMARCHAND; LEQUIME, 2013), probably because the films were not stoichiometric (which means that there was more silicon than oxygen in the process).

The recipe followed in the deposition process was already tested multiple times by the laboratory technicians (and also provided data from previous depositions), so it was not necessary to test the reliability of the machine. The SiO_2 thin-films showed good quality, but since the ECR machine was quite old and the process was too long, it was decided to test the process with another technology, in order to check if it was possible to find a recipe that provided both the refractive index observed in the literature and a shorter process. The parameters used in these depositions are showed in Table 3.1.

3.3.2 Plasma Enhanced Chemical Vapor Deposition (PECVD)

The technique of PECVD consists of a deposition of a solid phase from a gaseous phase, forming the thin-films. After the creation of the plasma some chemical reactions occur (volatile precursor gases react or decompose on a heated substrate). The plasma can be created by Radio Frequency (RF) or by direct discharge between the two electrodes (anode

Table 3.1 – ECR parameters for the SiO_2 thin-films deposition.

Parameter	Value
Argon Flux	5 sccm
Oxygen Flux	2.5 sccm
SiH_4 Flux	125 sccm
Pressure	2 mTorr
ECR power	250 W
Deposition rate	5.7 nm/min

Figure 3.3 – ECR machine.

Source: Own authorship.

and cathode). The plasma is formed in the space between the two electrodes (a grounded electrode and a RF or DC energized electrode), which is filled with the reacting gases (PLUMMER, 2009).

The capacitive coupling between the electrodes excites the reactant gases into a plasma, which induces a chemical reaction and results in the reaction product being deposited on the substrate. The substrate is placed on the grounded electrode, which is typically heated to 250 °C or even up to 350 °C, depending on the film requirements. The basic parts of the PECVD system are shown in Figure 3.4.

The advantages of using the PECVD are low operating temperature, uniform coating of different shapes, stress reduction, deposition of hard films and is environmentally stable. The disadvantages are toxic precursors and byproducts (thus the need to check for contamination of the samples by silane SiH_4), hard to obtain stoichiometry (thus is referred as SiOx).

The PECVD process is based on silane (SiH_4). The formation of SiO_x occurs if there is an interaction of the chemical compound silane with the nitrous oxide, according to the reaction: $SiH_x + N_2O \rightarrow SiO_x (+H_2 + N_2)$. In the process, there is a formation of the hydrogen gas (H_2), and nitrogen gas (N_2), which are then evacuated at the end of the process through the gas outlet (CHAPMAN, 1980; MADOU, 2002).

Figure 3.4 – Illustration of a PECVD system.

Source: Own authorship.

The SiH_4 can be used pure or diluted in another inert gas, typically N_2 , argon (Ar) or helium (He). The pure SiH_4 was used in this work. In a PECVD process, several parameters can be controlled, such as gas flow, temperature of the sample holder. Such parameters can directly interfere in the film resulting refractive index.

The refractive index is directly related to the composition of the film, normally the correct ratio would be a Si:O ratio. If the Si content is high, the refractive index will be high. After several attempts, the deposition process of the SiO_2 thin-films was standardized according to the Table 3.2.

This technique yielded better results for the thin-films of SiO_2 , when compared to ECR. As the deposition process was still not well defined, it was necessary to test some recipes, until reaching the refractive index that was closest to the literature (GAO; LEMARCHAND; LEQUIME, 2012; GAO; LEMARCHAND; LEQUIME, 2013). The depositions of SiO_2 films were performed at the CCS. The deposition rate was around 80 nm/min (much faster than the one using ECR technique), by using this technique. The refractive index varied from 1.462 to 1.475, which was found to be very close to the values in the previous cited

literature.

Table 3.2 – PECVD parameters for the SiO_2 thin-films deposition.

Parameter	Value
N_2 Flux	1000 sccm
N_2O Flux	1000 sccm
SiH_4 Flux	5 sccm
Pressure	2 Torr
HF power	40 W
Plate temperature	400 °C
Deposition rate	80 nm/min

3.3.3 Sputtering

The sputtering system is very similar to the PECVD system (showed at Figure 3.5). In this system, an inert gas, such as Ar , is inserted into the sputtering chamber at low pressure. Afterwards, a voltage is applied between both electrodes (anode and cathode) and a plasma is created. This plasma contains neutral argon atoms and is a conducting medium. The cathode (top electrode) is the source of the material to be deposited (is this case made out of titanium), and a negative DC voltage is applied to it.

The anode is the bottom electrode and it is at the same time the sample holder. The positive ions present in the plasma are then accelerated to the negatively biased electrode, called target, and these energetic ions strike the target and sputter the target atoms. In consequence, these atoms will then travel through the plasma as vapor and be deposited onto the wafers (CHAPMAN, 1980; MADOU, 2002). The DC magnetron sputtering technique was used in this work for the deposition of TiO_2 thin-films. This configuration of sputtering allows the increase of the percentage of electrons that take part in the ionization process, leading to a increased ionization efficiency (PLUMMER, 2009).

First of all, a recognition of the machine was made to know how the process could be controlled, which means that before the final settings, repeated deposition were performed, changing one parameter at a time, leading to a more stable recipe. All the data from depositions, and how those final settings were chosen are in the Annex. The main parameters for the sputtering deposition are presented in Table 3.3, which presents the optimal settings for this kind of deposition. These settings were found to be a good recipe for matching the desired refractive indexes, which were close to the literature (SIEFKE et al., 2016).

Figure 3.5 – Sputtering system.

Source: Own authorship.

Table 3.3 – Sputtering parameters for the TiO_2 thin-films deposition.

Parameter	Value
Argon Flux	65 sccm
Oxygen Flux	40 sccm
Power	1000W
Process Pressure	20 mTorr
Magnetron	17V/1.33A
Deposition rate	4.92 nm/min

4 OPTICAL FILTERS CHARACTERIZATION: EXPERIMENTAL RESULTS AND DISCUSSION

This Chapter presents all the necessary steps done before the final characterization of the optical filters, the experimental results, as well as the techniques used.

4.1 Introduction

It is important to have an overview of the work at this point in order to understand the work-flow necessary to conclude the analysis of the optical filters. Figure 4.1 shows the sequence of work activities and indicates when each type of test was performed. The first

Figure 4.1 – Process flowchart for the design, fabrication and characterization of the optical filters.

step was to build a test Printed Circuito Board (PCB) containing all the LEDs that would be characterized. Next step was to characterize them using a spectrometer. Following the flowchart, the LEDs suitable for work were chosen. The project and simulation of the optical filters were addressed in the Chapter 2. The fabrication process and techniques were discussed in Chapter 3.

After the fabrication, a microscope analysis of the layers of the filters was performed, using a Scanning Electron Microscope (SEM). The characterization of the filters proceeded, performing the broadband test using spectrophotometry, and the narrow-band test, using spectrometry. The next step was an application test performed onto the sub-lingual mucosa.

All the work activities and techniques are explained in more details in the following sections.

4.2 Spectrometry

In this work, the Portable Spectrometer CCS200 from *Thorlabs* was used to measure the spectral signature of the light sources. The blue, green and white LEDs were fixed in a PCB, projected to have the necessary current for the light sources to work. This equipment was also used to analyze the transmittance of the filters blue and green to a narrow-band range of wavelengths (in this case, the transmittance of the filter by placing the LEDs blue and green as a light source).

The setup using for the characterization was placing the light source with a collimator right after it, in order to narrow the scattered light beams from the LED and align them with the spectrometer entrance. The optical fiber was not used in the setup, because its aperture was very narrow. Thus, the collimator directed the light directly into the spectrometer aperture, providing easier alignment.

The basic function of a spectrometer (Figure 4.2) is to capture light, divide it into spectral components, digitize the acquired signal in function of its wavelength, and transfer the signal to display it in the computer. The first step of this process is to direct the light to the spectrometer through the aperture of the optical fiber.

This aperture causes the occurrence of the effect of the captured light. This effect results in some areas being darkened. In many spectrometers, the divergent light is collimated by a concave mirror and then directed to a diffraction grating. This diffraction grating disperses the wavelengths of light at slightly different angles, and is then focused by a second concave mirror and then projected onto the detector.

Figure 4.2 – Thorlabs portable spectrometer CCS200 coupled with the optical fiber. Range of wavelength detection from 200 nm to 1000 nm.

Source: ([THORLABS](https://www.thorlabs.com),).

Once the image is projected onto the detector, the photons are converted into electrons, which are then digitized and read by a PC through the Universal Serial Bus (USB) port

or by the serial port. The software then makes an interpolation based on the number of photons present in the detector and on the linear dispersion of the diffraction grating, in a way of creating a calibration to allow the data to be plotted. Usually the data is plotted in function of the wavelength under a determined spectral range.

The spectrometer CCS200 has a spectral range of wavelength detection from 200 nm to 1000 nm, a bandwidth spectral accuracy of < 2 nm at 633 nm, sensitivity of the CCD of 160 V/(lx.s), and integration time of 10 μ s - 60 s. For more technical information, may the manual of the equipment be consulted ([THORLABS](#),).

4.3 Spectrophotometry

The characterization of the optical filters was also done using the spectrophotometer CRAIC (Figure 4.3), from the Department of Chemistry of the University of Sao Paulo (IQSC). The spectrophotometry follows the same functioning of the spectrometer, but it is a more robust equipment, as it has three kinds of lamps and a microscope coupled to the equipment. This equipment was used to analyze the transmittance of the filter itself to a broad band range of wavelengths (in this case, a xenon lamp). The results are shown in the following sections.

Figure 4.3 – Spectrophotometer Craic. On the left side it is shown the detector, the light source and the microscope attached to the equipment. On the right side it is shown the lamps and the frontal image of the microscope showing the sample holder.

Source: Own authorship.

4.4 Light Sources Characterization

The characterization of the light sources, i.e., illuminants, is called spectral signature and it can be done by using a spectrometer. This step consists of analyze the electromagnetic

spectrum of each LED, in order to know the range of wavelength that each light source can operate. The result of a spectral signature is a graph that shows the emitted light intensity by wavelength.

The light sources used in this work were chosen according to the final objective of the project, which was fabricated optical filters for the endoscopic capsules. In this context, the light sources should be as small as possible to be integrated into the small capsules. For this work, the illuminants are high-efficiency LEDs. These LEDs are build using indium gallium nitride (InGaN), phosphorus (P), with dimensions of 1.6 mm x 0.8 mm x 0.55 mm (length x width x height) from *Mouser Electronics* ([MOUSER...](#)) and from *Dualcom*. A total of eight LEDs (including green, blue, and white ones) were characterized. After performing the characterizations, the green LED (peak centered at 533 nm) from Dualcom and the blue LED (peak centered at 410 nm) from Mouser were chosen, because their transmittance peak matched the desired wavelengths and bandwidth proposed in this work. The results are shown in the following sections.

Figure 4.4 presents the spectral signature of the green LED characterized using the spectrometer. The green LED has its peak centralized at 533 nm. This LED was chosen because it comprehends the wavelength of interest (540 nm) with high relative transmittance, around 88%. Figure 4.5 shows the spectral signature of the blue LED. The blue LED has

Figure 4.4 – Spectral signature of the green LED light source.

Source: Own authorship.

its peak centered at 410 nm and it comprehends the wavelength of 415 nm with relative transmittance around 65%. It is important that the spectral signature of LED comprises

the wavelength wanted for the filter to be centered, once the simulation of the optical filter will be within the LED spectral signature.

Figure 4.5 – Spectral signature of the blue LED light source.

Source: Own authorship.

4.5 SEM analysis

A high resolution SEM was used for measuring the thicknesses of the layers of the optical filters. This SEM is MEV-FEG, JEOL model 7500F, from the Laboratory of advanced microscopy (LMA), Chemistry Institute of Araraquara (IQ-UNESP).

Figures 4.6 and 4.7 present the SEM photographs showing the cross-section of the green and blue optical filters. It can be observed an acceptable deviation from the simulated thicknesses (around 2 nm). It is also demonstrated a clear separation between the layers of TiO_2 and the SiO_2 . The thin-films showed a good flatness along the cross-section. The flatness directly influences on the parallelism between the two mirrors concerning to the resonance cavity, which is a crucial characteristic for NBI filters.

4.6 Optical Filters Characterization

Figure 4.8 presents the picture of both optical filters, blue and green. The filters shows a good film quality, from the center to the borders. The presence of border-effect is common to many deposition methods, and in the Figure 4.8 the sputtering border pattern can be

Figure 4.6 – SEM photograph of the cross-section of the green filter. The thicknesses of the TiO_2 and the SiO_2 layers was set to 56 nm and to 95 nm, respectively. The resonance cavity (SiO_2) spacing was set to 176 nm. The deviation from the simulation was minimal and it can be related to the measurement error intrinsic from this technique.

Source: Own authorship.

clearly visualized. Tests of spectrophotometry were performed focusing different spots of the sample, and the best results were found in the middle of them, where the coating was more uniform.

The tests of the filters were carried out in two ways. The first way was testing the response of the filter to the light sources with specific wavelengths (415 and 540 nm). Figure 4.9 shows the setup used for the first test. The second way was testing the response of the filter to a light sources containing a wider range of wavelengths (300-800 nm). For this second option a xenon light source was employed.

The tests with the LEDs as light sources were performed using a portable spectrometer from Thorlabs, and the test with the xenon lamp were performed using a spectrophotometer from Craic. For this last test there was no setup because the machine had all the elements already in their exact places. Thus, it was just needed to place the filters on the sample holder, perform the reference measurements and the spectral transmittance of the filters is

Figure 4.7 – SEM photograph of the cross-section of the green filter. The thicknesses of the TiO_2 and the SiO_2 layers was set to 32 nm and to 83 nm, respectively. The resonance cavity (SiO_2) spacing was set to 154 nm. It can be observed that the deviation from the simulation and the fabricated filter was minimal and it can be related to the measurement error of this technique.

Source: Own authorship.

measured by the equipment.

4.6.1 Broadband characterization

4.6.1.1 Blue filter

Figure 4.10 presents the measure of transmittance for the blue filter, under a xenon light source. The measurement window was set to perform $1\mu\text{ m} \times 1\mu\text{ m}$ for each measure. This measurement showed the response of the filter for broadband light source. It can be observed that for the xenon light, the filter is centered around the 420 nm, but it still comprehends the wavelength of 415 nm with high transmittance (around 51%). The border measure showed a peak shift to 433 nm. This occurs because of the deposition pattern at the border is not so uniform as in the middle of the samples. At the border, the total thickness is thinner than desired.

Figure 4.8 – Picture of the physical optical filters; (a) On the left side the blue filter; (b) On the right side the green filter.

Source: Own authorship.

Figure 4.9 – Setup for the characterization of the optical filters with specific illuminants blue and green.

Source: Own authorship.

4.6.1.2 Green filter

Figure 4.11 shows the response of the filter to a Xenon light source. It can be observed that plots of the center measure are well centralized at 540 nm. It also shows the fringes

Figure 4.10 – Measured transmittance of the blue filter with a Xenon light as illuminant.

Source: Own authorship.

of the filter around the 400 nm. The measured done at the border of the sample is a little shifted to the right, centered at 546 nm. These measurements showed that even with the incidence of broad band Xenon light, the filter works and comprises the wanted wavelength for this project (540 nm).

4.6.2 Narrow-band characterization

The broadband measurement works as a guide for the narrow-band measurements, because it shows a wider bandwidth that the filter works. It can be observed that the broadband characterization showed the best results in the middle of the sample, which means that at these points, the transmittance spectrum comprised the wavelengths of interest (415 nm and 540 nm). Thus, the best results of the narrow-band test were at the points where the broadband comprises the wavelengths of interest.

Figure 4.11 – Measured transmittance of the green filter with a Xenon light as illuminant. It can be observed that there is even a difference between both center measures, because the measurement window is very narrow, so any slight difference or interference (like dust) can vary the result.

Source: Own authorship.

4.6.2.1 Blue filter

Figure 4.12 shows the relative transmittance of blue filter with the blue LED as illuminant. The results showed a relative transmittance of around 35% and the peak was centered at 414 nm, which is a good result comparing with the simulations. The fact that the relative transmittance was lower than the simulated was expected, because of the narrow spectral signature of the illuminant (blue LED), and also regarding the fabrication issues involved. The result showed the peak was just 1 nm deviated from the goal, that was at 415 nm. The calculated FWHM was 17 nm.

Figure 4.12 – Measured relative transmittance of the blue filter with a blue LED as illuminant.

Source: Own authorship.

4.6.2.2 Green Filter

Figure 4.13 shows the spectral relative transmittance of green filter with the green LED as illuminant. The results showed a relative transmittance of around 60%, the peak was centered at 542 nm, and the calculated FWHM was 29 nm. There was a peak deviation of 2 nm from the intended, which was a peak centered at 540 nm.

4.7 Preliminary tests on sub-lingual mucosa

The optical filters were also tested on the sub-lingual mucosa to a more realistic test. The sub-lingual mucosa was chosen because it is more accessible mucosa and it has both superficial and deep vessels (DRAKE; MITCHELL; VOGL, 2005). The tests helped to prove the initial hypothesis of this work, that there was a difference between positioning the filters on the illuminant and on the image sensor.

Figure 4.13 – Relative transmittance of the green filter with a green LED as illuminant.

Source: Own authorship.

4.7.0.1 Filter on the light source

The first test was to place the filters directly on top of the green and blue LEDs, and illuminate the sub-lingual mucosa with it. Then, a photograph was taken. Figure 4.14 shows the illustration of the setup for this test. The blue LED + blue filter was tested separated to the green LED + green filter.

Figure 4.15 shows the results of the filters on the light source. A reference picture (Figure 4.15 (a)) was taken to compare the photographs. Figure 4.15 (b) shows the photograph of the incident green light with the green filter on top, and 4.15 (c) shows the one with incident blue light with the blue filter on top. It can be observed a different vessel pattern on each picture, but the contrast between the superficial and deep vessels is not so clear.

4.7.0.2 Filters on imager

The second test was performed placing each filter at a time onto the image sensor. Figure 4.16 shows the schematic of the setup used for the test with green LED + green filter and

Figure 4.14 – Schematic of the setup for testing the filter on the light source.

Source: Own authorship.

Figure 4.15 – Tests of the optical filters onto the sub-lingual mucosa; (a) Reference picture taken under ambient light; (b) Illumination with green light and green filter on top of the LED; (c) Illumination with blue light and blue filter on top of the LED.

Source: Own authorship.

for the test with blue LED + blue filter (the emissions of light were not concomitantly). Figure 4.17 shows the results of the filters on the image sensor. Figure 4.17 (b) shows the photograph of the incident green light with the green filter on top, and 4.17 (c) shows the one with incident blue light with the blue filter on top.

It can be observed a more clear difference between the vessel pattern. On Figure 4.17 (b) the contrast between the deeper vessels is higher, and it is observed some vessels

Figure 4.16 – Schematic of the setup for testing the filter on the image sensor.

Source: Own authorship.

non-observed in the reference picture. On Figure 4.17 (c) the deep vessels cannot be observed, but there is a higher contrast between the superficial vessels.

The hypothesis of placing the filters on the imager was found to be a more efficient way of a setup for using the NBI filters. When the light beam reaches the mucosa without an intervention of a filter, the contrast is much more higher. It was found that when the filter is placed on top of the light source, it decreases the amplitude of the light beam, leading to a poor contrast between the vessels.

The tests were done in an illuminated ambient, in order to have a better focus of the camera. This preliminary test can be improved using a good quality camera set to video mode, and using a matrix of LEDs, in order to increase the area of intensity of the illumination.

4.8 Preliminary tests on ocular mucosa

After the preliminary test on sub-lingual mucosa, was realized that the ambient light (white fluorescent light) was contaminating the photographs and that the illuminating system had a poor amplitude, which could not lighten enough the surface in study. Therefore, two lighting systems were fabricated, in order to enhance the illuminated area, and also to enhance the quality of the images. With this two new systems, it was chosen to make the test on the ocular mucosa.

Figure 4.17 – Tests of the optical filter onto the sub-lingual mucosa; (a) Reference picture taken under ambient light; (b) Illumination with green light and green filter on top of the image sensor; (c) Illumination with blue light and blue filter on top of the image sensor.

Source: Own authorship.

4.8.0.1 Filter on the light source

Figure 4.18 (b1) and (b2) show the tests with blue and green illumination, respectively, onto the sub-lingual mucosa, with the filters positioned on the light sources. For this system, the positioning of the optical filters on top of the LEDs showed great contrast enhancement and focus of the superficial vessels with the blue illumination.

4.8.0.2 Filters on imager

Figure 4.18 (c1) and (c2) show the tests with blue and green illumination, respectively, onto the sub-lingual mucosa, with the filters positioned on the image sensor. The positioning of the optical filters on top of the image sensor showed a less focused image, but still with contrast enhancement of the superficial and deep vessels.

After changing the lightening system, it was found that when the optical filters are positioned on top of the light sources, better images (regarding contrast enhancement and focus) were obtained. It is important to observe that different results were found between the test on sub-lingual mucosa and the test on ocular mucosa. It is believed that the ambient light used in the tests on sub-lingual mucosa was contaminating the images, and thus leading to non-realistic results. After the improvement made in the lighting system, it was possible to observe that the results differed from the previous test and were much better.

Figure 4.18 – Tests of the optical filter onto the sub-lingual mucosa; (a) Reference picture taken under ambient light; (b1) Illumination with blue light and blue filter on top of the light source; (b2) Illumination with green light and green filter on top of the image sensor; (c1) Illumination with blue light and blue filter on top of the image sensor; (c2) Illumination with green light and green filter on top of the image sensor.

Source: Own authorship.

5 CONCLUSIONS

Previous researches with NBI technology have shown great advance when coupled with conventional endoscopy, for the detections of lesions in the gastrointestinal tract, and also in the diagnosis of endometrial and lung lesions. It is important to notice that sometimes, the conventional endoscopy for the GI tract cannot reach some portions of the small intestine. The concept of capsule endoscopy has revolutionized gastroenterology and simultaneously promoted a new diagnostic perspective into the GI tract.

It is believed that the incorporation of the NBI technology into the capsules will be a great upgrade in the diagnosis, in the sense of detecting lesions non-visible with the white light that most endoscopes have, in areas that the endoscopes cannot reach.

This master thesis presented the complete design, fabrication and characterization of two optical filters using the NBI technology. This work also aimed the study of two hypotheses regarding the optical filters, involving the difference in the adaptation of the optical filters: 1) Adapting the filters on the imager sensor or 2) on the light sources. The preliminary tests showed that the better setup was placing the filter on top of the image sensor.

The main objective of this work was fabricating two optical filters using the NBI technology, with specific illuminants and bandwidth centered at the wavelengths of 415 nm and 540 nm, the absorption peak of the hemoglobin. Thus, with these two filters would be possible to observe two different vessel patterns, the superficial and the deep.

The blue filter was centered at 414 nm, really near to the simulations (415 nm) with relative transmittance of 33%, with the blue LED as illuminant, and a FWHM of 17 nm. The green filter was centered at 542 nm (also near to the simulations at 540 nm) with a relative transmittance of 62% with the green LED as illuminant, and a FWHM of 29 nm. Both filters showed good results regard the transmittance peak and the FWHM.

The NBI filters fabrication presented in this work is a part of a bigger project, which is the incorporation of this technology into the endoscope capsules. The whole project steps is presented in Figure 5.1 and as following the complexity of the tasks involved are discussed.

The fabrication and characterization of optical filters requires a very methodological work, because it involves multiple tasks, and some of them are even dependent on each other. The work-flow must be very clear and organized.

The development of this project has highlighted the complexity and challenges associated with the fabrication of optical filters. This project required a cooperation of four different laboratories (CCS, LSI, IQ and IQSC), located in three different cities. The majority of the resources were located at the clean room of the CCS (Campinas), and most of the process required a technician assistance (cleaning of substrate, machine operation), which means the access to the processes and the time to repeat them was very limited and dependent

Figure 5.1 – Block diagram of the complete incorporation of the NBI technology into the endoscopic capsules.

Source: Own authorship.

on third-party personnel.

Another difficult encountered during this work was the low machines availability for depositions and for characterizations. Most of the time they were broken, or repairing, which made all the processes slower than necessary, and sometimes this fact lead to not finish the measurements.

This work showed the completion of most tasks, which are pointed out with a green check mark in the flowchart (Figure 5.1). The four last tasks can be accomplished in future works. As the example in the flowchart shows, the optical filters can be adapted into a photonic module, which contains the light sources, the optical filters, and an image sensor. Moreover, the photonic module can be validated, and then adapted into endoscopic capsules. This step requires a fine adaptation, taking into account that the ECs can be commercial

capsules, or this step could also require the construction of the capsule (leading to a long process). Finally the ECs could be validated and be ready for use in humans.

Another idea of future work is to integrate the NBI optical filters for the analysis of other vessels patterns, like the sub-lingual mucosa or eyes.

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Annex

ANNEX A – DATA FROM SPUTTERING DEPOSITION OF TiO_2 THIN-FILMS

After obtaining the data from the characterization of the thin-films by ellipsometry, it was calculated the absolute, relative and percentage errors of each sample, i.e., how much the measured value distanced itself from the ideal value that was intended in the deposition. From the data collected at each deposition, it was possible to measure the deposition rate and thus adjust it to a determined value, which would provide a smaller error between the measured and deposited thin-film thickness.

The data were organized into table to facilitate the analysis and evolution of the characterization process. The *Sample* column refers to which was the sample (1, 2, 3 or 4). The *Sample measurement location* refers to measurement points by ellipsometry, i.e., if the measurement was performed in the center or on both borders of the samples. The *Wanted d* column refers to the ideal thickness of the thin-film that was expected to be deposited. The *Measured d* column refers to the thickness of the thin-film measured by ellipsometry after each deposition. The *Refractive Index* column refers to the value n of the thin-film measured by ellipsometry. Finally, the $|Ea|$, Er and $E\%$ columns refer to the absolute, relative, and percent error calculations, respectively.

The first deposition is always a test of how the system works, and how the parameters are related. Thus, there is no need of doing a fine adjustment at this point. It was tested two samples of each thickness, and as a start, there were chosen the thicknesses of 100, 80, 60 and 40 nm. It must be pointed out that the sputtering machine did not have the crystal quartz in order to measure the deposition thickness, neither any control of the thickness. In that way, there was a need to adjust all the parameters and also the deposition rate.

Table A.1 presents the data from the first deposition of TiO_2 thin-films. It can be inferred from Table A.1 that the percentage error was really high, and the refractive index varied from 2.43 to 2.49. The average thicknesses measured by ellipsometry were 83.95, 66.70, 51.77, and 35.70 nm, in contrast with the wanted ones 100, 80, 60 and 40 nm, respectively.

Tables A.2 and A.3 present the data from the second deposition of TiO_2 samples with 100, 80, 60, and 40 nm. This step consisted in a more fine adjustment, and with this purpose, more samples were tested.

The average thicknesses measured by ellipsometry were 84.06, 67.98, 51.82, and 35.30 nm, in contrast with the ideal ones 100, 80, 60 and 40 nm, respectively. For the 100 nm samples, the refractive index varied from 2.44 to 2.49, depending where the measurement was done (at the center or the border). It was expected to have different thickness and refractive index from the center to the border of the sample, because the sputtering machine induces a circular pattern on the samples. The percentage error was still a little high for the

Table A.1 – Sample data from the first deposition for the thicknesses of 100, 80, 60 and 40 nm. Sample of TiO_2 .

Sample	Sample measurement location	Wanted d (nm)	Measured d (nm)	Refractive index	$ Ea $ (nm)	Er	$E\%$
1	center	100	84.8	2.48	15.2	0.15	15.20
1	center	100	83.1	2.49	16.9	0.17	16.63
2	center	80	66.7	2.44	13.3	0.14	13.83
2	center	80	67.5	2.43	12.5	0.16	15.63
3	center	60	51.8	2.44	8.2	0.14	13.67
3	center	60	51.8	2.44	8.2	0.14	13.67
4	center	40	36.1	2.43	3.9	0.10	9.75
4	center	40	35.5	2.43	4.5	0.11	11.25
Average measured thickness of the 100 nm samples: 83.95 nm							
Average measured thickness of the 80 nm samples: 66.70 nm							
Average measured thickness of the 60 nm samples: 51.77 nm							
Average measured thickness of the 40 nm samples: 35.70 nm							

application of optical filters.

Tables A.4 presents the data from the third deposition of TiO_2 samples with 100 and 80 nm. For the 100 nm samples, the refractive index varied from 2.51 to 2.55. The maximum divergence between the measured thickness and the ideal was 9.2 nm, and the average measured thickness was 94.88 nm. For the 80 nm, the refractive index varied from 2.47 to 2.50. The maximum divergence from the wanted thickness was 4.2 nm, and the average thickness was 78.97 nm.

Table A.5 presents the data from the third deposition of TiO_2 samples with 60 and 40 nm. For the 80 nm samples, the refractive index varied from 2.44 to 2.46. The maximum divergence between the measured thickness and the ideal was 3.3 nm, and the average measured thickness was 57 nm. For the 40 nm, the refractive index varied from 2.43 to 2.44. The maximum divergence from the wanted thickness was 4.4 nm, and the average thickness was 37.45 nm.

Table A.6 presents the data from the third deposition of TiO_2 samples with 100 and 80 nm. For the 100 nm samples, the refractive index varied from 2.53 to 2.58. The maximum divergence between the measured thickness and the ideal was 6.1 nm, and the average measured thickness was 97.34 nm. For the 80 nm, the refractive index varied from 2.47 to 2.50. The maximum divergence from the wanted thickness was 5.2 nm, and the average thickness was 80.68 nm.

Table A.7 presents the data from the third deposition of TiO_2 samples with 60 and 40 nm. For the 80 nm samples, the refractive index varied from 2.44 to 2.47. The maximum divergence between the measured thickness and the ideal was 3.3 nm, and the average

Table A.2 – Sample data from the second deposition for the thicknesses of 100 and 80 nm. Samples of TiO_2 .

Sample	Sample measurement location	Wanted d (nm)	Measured d (nm)	Refractive index	$ Ea $ (nm)	Er	$E\%$
1	center	100	84.7	2.47	15.3	0.15	15.30
1	border1	100	86.1	2.48	13.9	0.14	13.90
1	border 2	100	81.8	2.44	18.2	0.18	18.20
2	center	100	83.9	2.47	16.1	0.16	16.10
2	border1	100	86.7	2.48	13.3	0.13	13.30
2	border 2	100	81.6	2.44	18.4	0.18	18.40
3	center	100	83.9	2.46	16.1	0.16	16.10
3	border1	100	86.2	2.49	13.8	0.14	13.80
3	border 2	100	81.6	2.44	18.4	0.18	18.40
4	center	100	83.7	2.46	16.3	0.16	16.30
4	border1	100	86.1	2.48	13.9	0.14	13.90
4	border 2	100	82.2	2.45	17.8	0.18	17.80
Average measured thickness of the 100 nm samples: 84.06 nm.							
1	center	80	68.1	2.45	11.9	0.15	14.88
1	border1	80	70.4	2.46	9.6	0.12	12
1	border 2	80	66.2	2.43	13.8	0.17	17.25
2	center	80	68.1	2.45	11.9	0.15	14.88
2	border1	80	70	2.46	10	0.13	12.50
2	border 2	80	65.6	2.43	14.4	0.18	18
3	center	80	68	2.45	12	0.15	15
3	border1	80	70.2	2.46	9.8	0.12	12.25
3	border 2	80	65.2	2.43	14.8	0.19	18.50
4	center	80	68.3	2.45	11.7	0.15	14.63
4	border1	80	70.6	2.46	9.4	0.12	11.75
4	border 2	80	65.1	2.42	14.9	0.19	18.63
Average measured thickness of the 80 nm samples: 67.98 nm.							

measured thickness was 59.39 nm. For the 40 nm, the refractive index varied from 2.43 to 2.44. The maximum divergence from the wanted thickness was 2.9 nm, and the average thickness was 40.94 nm.

After adjusting the deposition rate for the thickness of 40, 60, 80 and 100 nm, samples with thicknesses of 30, 50, 70 and 90 nm were characterized. The Table A.8 shows the measured and calculated values for the 90 and 70 nm. For the samples of 90 nm, the variation of the refractive index was from 2.48 to 2.58. The maximum divergence from the ideal value was 5.5 nm. The average thickness was 90.74 nm. The samples of 70 nm had average measured thickness of 74.72 nm, the refractive index ranged from 2.43 to 2.48, and the highest divergence from the ideal was 14.2 nm.

Table A.9 presents the measurements of the fourth deposition for the thickness of 50 and

Table A.3 – Sample data from the second deposition for the thicknesses of 60 and 40 nm. Samples of TiO_2 .

Sample	Sample measurement location	Wanted d (nm)	Measured d (nm)	Refractive index	$ Ea $ (nm)	Er	$E\%$
1	center	60	52	2.43	8	0.13	13.33
1	border1	60	53.4	2.44	6.6	0.11	11
1	border 2	60	50.9	2.42	9.1	0.15	15.17
2	center	60	51.9	2.43	8.1	0.13	13.33
2	border1	60	53.2	2.44	6.8	0.11	11.33
2	border 2	60	50.3	2.42	9.7	0.15	15.17
3	center	60	51.5	2.43	8.5	0.14	14.17
3	border1	60	53.5	2.44	6.5	0.11	10.83
3	border 2	60	50.1	2.42	9.9	0.17	16.50
4	center	60	52	2.44	8	0.13	13.33
4	border1	60	53.2	2.44	6.8	0.11	11.33
4	border 2	60	49.8	2.42	10.2	0.17	17
Average measured thickness of the 60 nm samples: 51.82 nm.							
1	center	40	35.2	2.42	4.8	0.12	12
1	border1	40	36.5	2.43	3.5	0.09	8.75
1	border 2	40	34.5	2.42	5.5	0.14	13.75
2	center	40	35.3	2.42	4.7	0.12	11.75
2	border1	40	36.2	2.43	3.8	0.09	9.50
2	border 2	40	34.5	2.42	5.5	0.14	13.75
3	center	40	35.4	2.42	4.6	0.12	11.50
3	border1	40	36.1	2.42	3.9	0.10	9.75
3	border 2	40	34.2	2.41	5.8	0.15	14.50
4	center	40	35.4	2.42	4.6	0.12	11.50
4	border1	40	36.2	2.43	3.8	0.09	9.50
4	border 2	40	34.1	2.41	5.9	0.15	14.75
Average measured thickness of the 40 nm samples: 35.30 nm.							

30 nm. For the samples of 50 nm, the variation of the refractive index was from 2.44 to 2.48. The maximum divergence from the ideal value was 6.4 nm. The average thickness was 53.69 nm. The samples of 30 nm had average measured thickness of 33.07 nm, the refractive index ranged from 2.43 to 2.44, and the highest divergence from the ideal was 4.9 nm.

Although the results from the samples of 90 and 70 nm presented good results and a minor percentage error, the samples with 50 and 30 nm were still presenting a percentage error up to 14%. Thus, another batch of depositions were made adjusting again the parameters. Table A.10 presents the results for the thicknesses of 90 and 70 nm. The average thicknesses were 91.01 nm, and 73.55 nm, respectively. The refractive index range from 2.48 to 2.52 for the 90 nm samples, and from 2.45 to 2.48 for the 70 nm samples. The percentage error

Table A.4 – Sample data from the third deposition for the thicknesses of 100 and 80 nm. Samples of TiO_2 .

Sample	Sample measurement location	Wanted d (nm)	Measured d (nm)	Refractive index	$ Ea $ (nm)	Er	$E\%$
1	center	100	94.3	2.53	5.7	0.06	5.70
1	border 1	100	97.2	2.54	2.8	0.03	2.80
1	border 2	100	91.9	2.51	8.1	0.08	8.10
2	center	100	94.4	2.53	5.6	0.06	5.60
2	border 1	100	97.3	2.55	2.7	0.03	2.70
2	border 2	100	92.6	2.52	7.4	0.07	7.40
3	center	100	93.6	2.54	6.1	0.06	6.10
3	border 1	100	97.9	2.54	2.1	0.02	2.10
3	border 2	100	90.8	2.53	9.2	0.09	9.20
4	center	100	96.1	2.53	3.9	0.04	3.90
4	border 1	100	100.1	2.53	0.1	0.00	0.10
4	border 2	100	92.1	2.52	7.9	0.08	7.90
Average measured thickness of the 100 nm samples: 94.88 nm							
1	center	80	78.4	2.48	1.6	0.02	2.00
1	border 1	80	80.8	2.48	0.8	0.01	1.00
1	border 2	80	75.8	2.47	4.2	0.05	5.25
2	center	80	78.9	2.49	1.1	0.01	1.37
2	border 1	80	81.7	2.49	1.7	0.02	2.13
2	border 2	80	76.2	2.48	3.8	0.05	4.75
3	center	80	79.8	2.49	0.2	0.00	0.25
3	border 1	80	82.0	2.50	2.0	0.03	2.50
3	border 2	80	77.6	2.49	2.4	0.03	3.00
4	center	80	78.8	2.49	1.2	0.02	1.50
4	border 1	80	80.7	2.50	0.7	0.01	0.88
4	border 2	80	77.0	2.48	3.0	0.04	3.75
Average measured thickness of the 80 nm samples: 78.97 nm							

decreased for both kind of samples.

Table A.11 shows the results for the thicknesses of 50 and 30 nm. The average thicknesses were 53.67 nm, and 32.83 nm, respectively. The refractive index range from 2.44 to 2.45 for the 50 nm samples, and from 2.43 to 2.44 for the 70 nm samples. The percentage error decreased for both kind of samples, considering the center measurements. It can also be noticed that the refractive index did not have a wide range of variation as in the previous samples.

After all the deposition changes and settings, the results turned out satisfactory for starting to deposit TiO_2 thin-films. It can be seen that the percentage error did not completely zeroed, but it is acceptable for small scale fabrications.

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