

## THE EDS ANALYZE OF SiO<sub>x</sub> DEPOSITED BY PECVD

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**Abstract.** *Thin film technology and, in particular, plasma deposition for the surface modification of conventional materials is gaining increasing popularity because it allows the design of new devices with unique surface properties. The main purpose of this study is to present equipments used for SiO<sub>x</sub> layer deposition by PECVD, especially on substrates as Ti and its alloys and to analyze by EDS different plasma chemical compositions.*

Keywords: EDS, SiO<sub>x</sub>, Ti6Al4V, PECVD

### 1. INTRODUCTION

The aim of this paper is to present a review of the PECVD (Plasma Enhanced Chemical Vapor Deposition) SiO<sub>x</sub> thin films on the surface of the most used biomaterials, such as Ti and its alloys. These biomaterials offer many attractive properties, which lead to an increasing interest in using them in different areas such as medical devices and automotive parts. Regarding the medical research field, the permanent metallic prostheses or dental implants, are usually fabricated from light metal or alloys which exhibit superior mechanical properties such as tensile strength, toughness and fatigue resistance. Mainly, their excellent specific strength and outstanding corrosion resistance have made them very important [1].

PECVD is suitable for growing silicon oxide at low temperatures which is surely an advantage, as the number of applications with low temperature requirements is increasing [2].

Thin silicon oxide films are used widely in microelectronics, optoelectronics and optics for their outstanding ability to protect metals and polymers. These coatings have several advantages: they are transparent, chemically inert and have sufficient hardness. Silicon oxide is used as a protective layer in the reflectors of automobiles or mobile phones and can be used as a scratch resistant layer on transparent polymers.

Therefore, in the recent years, a special attention has been paid to organosilicons. Their compounds are non-toxic and non-explosive and hence, no special safety installation is required as in the case of silane. Tetraethoxysiloxane (TEOS) mixed with oxygen is used in PECVD of silicon oxide films for more than 30 years. TEOS-SiO<sub>2</sub> films exhibit a good step coverage even when deposited at a relatively low temperature, but it is widely known that there are several problems such as high hygroscopicity and abundance of carbon and water related impurities which affect the electrical properties of the films. TEOS-SiO<sub>2</sub> films exhibit good step

coverage even when deposited at a relatively low temperature, but it is widely known that there are several problems such as high hygroscopicity and abundance of carbon and water related impurities which affect the electrical properties of the films.

The oxidation of Si takes place in three steps: transport of the oxygen to the surface, diffusion of the oxygen through the already grown oxide and finally the reaction of the oxygen with the silicon at the interface between silicon and silicon oxide. With growing oxide thickness, the growing rate slows down because the time of the diffusion through the oxide depends on its thickness, and therefore becomes relevant for the oxidation rate. Very thin oxides can also grow at reduced pressure or in RTP systems (rapid thermal anneal). By the oxidation of the silicon, the silicon is consumed and the interface moves into the substrate [3].

Three types of PECVD systems are in use today: with a single wafer, with multiple wafers, clustered tools, and systems employing a single chamber with multiple deposition sites to achieve better film thickness uniformity. Other features being implemented include a dc or RF bias (dc: 400 kHz), in order to modify the film stress.

### 2. EXPERIMENTAL

#### 2.1 EQUIPMENT USED TO PERFORM THE PECVD PROCESS

A single chamber reactor is designed with associated plasma sealing shields. These shields avoid the deposition outside the wafer area and thus particles which may be generated within the system. RF power is applied through the electrode which injects reactants into the process chamber. A second bias, either dc or more recently, low frequency RF, can be applied to the lower electrode.

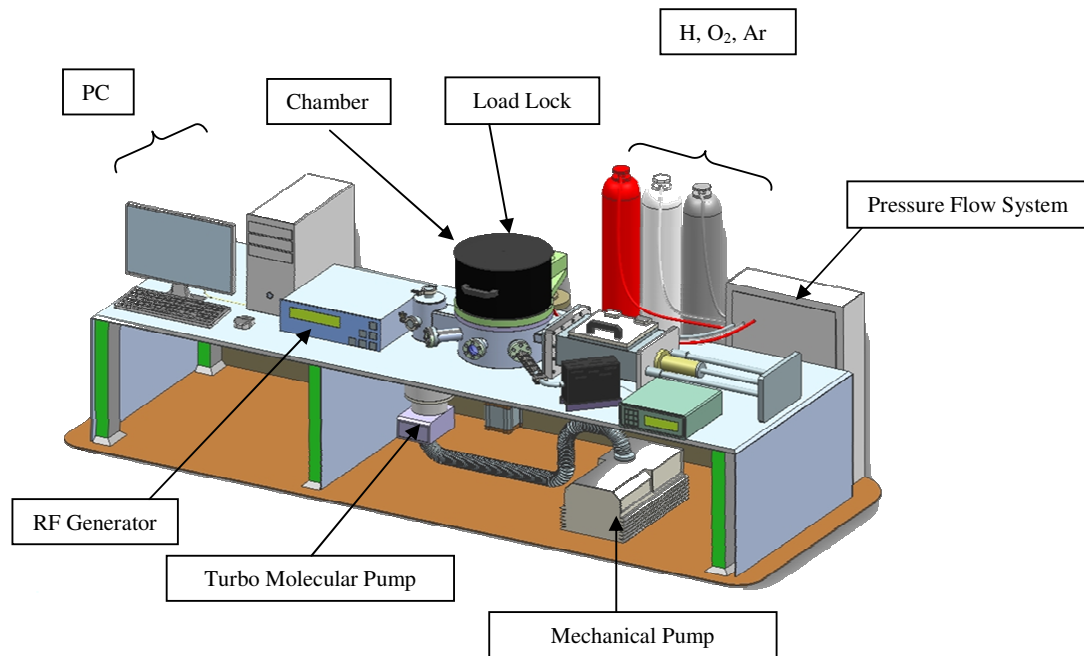


Fig. 1. PECVD silicon oxide thin film equipments.

During deposition process, some important PECVD film parameters such as: the reactant flow rates, the wafer temperature, and the RF power, are used to sustain plasma. The properties of the film can be changed also by increasing or decreasing the input power into the chamber. The bias controls the ion species bombardment of the deposited film, especially during deposition, leading to an ability to control film stress from a low compressive to a high compressive value.

In Figure 1, it is possible to observe the main components used on PECVD deposition of  $\text{SiO}_x$  thin films. The reactor presented is constructed by one cylindrical chamber with 200mm diameter and internal parallel electrodes. The above electrode is connected to a potential generator of 13,56MHz and the below one, where the samples will be placed, is connected to the ground. Using a RF generator, the plasma is formed in the reaction chamber which will contain reactive ions and radicals. The growth of the deposit starts easily because of the activation and cleaning of the surface by the intense bombarding with ions from the plasma.

Hence, the silicon oxide film adhesion and growth rate will begin when the plasma will be activated by the RF generator power. In order to obtain a high quality of the silicon oxide coating, it can be used PECVD, a versatile technique that offer the possibility to control many parameters. The most important parameters are: the adjustment of adhesion, compressive and tensile stress causing warpage, hydrogen content and density, etch ability, etch rate and selectivity in etching, step coverage as well as stoichiometry (consistence) and cleanliness of the deposited layers. The maximum thickness of the

deposit and the best uniformity of the coating, also depend on the PECVD process parameters.

A capacitive manometer is used to control the inside working pressure of the chamber of the PECVD system. The mixture of  $\text{O}_2$ , Ar, and H is introduced into the system using electronics devices such as Mass Flow Controller with a maximum flow capacitance of 200 sccm (standard cubic centimeter per minute) and for the TEOS flow will be used a Vapor Source Controller.

The vacuum from the reactor chamber is achieved by a pumping system constructed from a turbo molecular pump. The system is equipped with a controlled valve in order to ensure the possibility to change the pumping speed and thereby, the working pressure will be directly influenced. The substrate temperature is an important parameter, difficult to be controlled by PECVD. Thus, in order to manage the substrate composition by temperature, the PECVD reactor is equipped with a system of six warming lamps.

The system is also equipped with a water cooling system, which is useful in achieving an efficient sputtering process and protecting the chamber vacuum sealing.

## 2.2 PROCESS

The substrate that will be used for silicon oxide deposition is Ti6Al4V, which is one of the most commonly used titanium alloys is an alpha-beta alloy containing 6% Al and 4% V.

In order to perform the silicon oxide deposition by PECVD technology, a group of Ti6Al4V three samples

were mechanically polished on SiC paper to 2400 grit and ultrasonically cleaned in a solution containing distilled water. Silicon oxide layer is obtained after combination of: Ar + O<sub>2</sub>+ TEOS, 50W input power. There will be analyzed 3 types of coatings. Below are presented the three coated samples with SiO<sub>x</sub> by PECVD:

**Sample no. 1:**

Ar =18sccm + O<sub>2</sub>=20sccm + TEOS=1sccm (sccm-Standard Cubic Centimeters per Minute);

**Sample no. 2:**

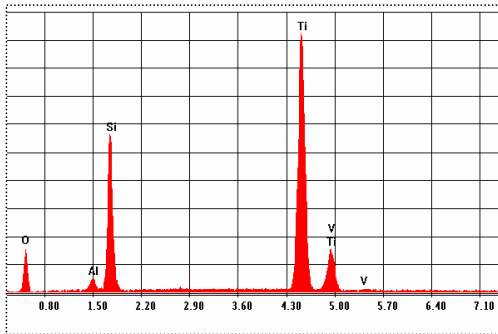
Ar= 28sccm + O<sub>2</sub>=10sccm + TEOS=1sccm;

**Sample no. 3:**

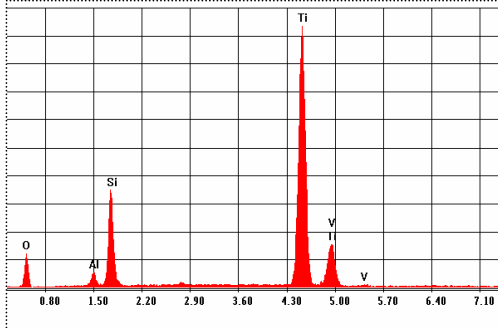
Ar =38sccm + O<sub>2</sub>=1sccm + TEOS=1sccm;

The power applied during the deposition process was 50W during 60 min. The equipment used is CVD600 PECVD – PVD (physical vapor deposition) which is running by lookout software manufactured specially in order to be controlled by an external PC.

**Sample no. 1**



**Sample no. 2**



**Sample no. 3**

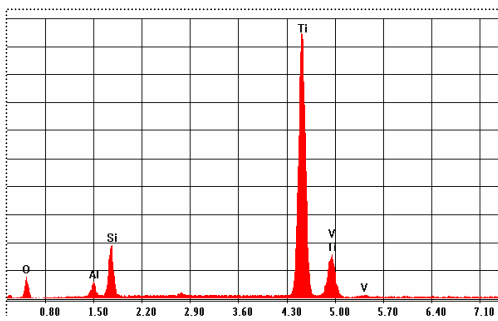


Fig. 2. EDS analysis

Table 1. Plasma composition of each sample

Element	Wt %		
	Sample no. 1	Sample no. 2	Sample no. 3
O <sub>2</sub>	42.95	42	35.01
Al	1.79	2.36	3
Si	18.6	13.53	9.51
Ti	35.74	40.87	51.04
V	0.92	1.24	1.44

In Figure 2 are presented the EDS graphics (Energy-dispersive X-ray spectroscopy) of the Ti6Al4V samples with different plasma composition.

**3. CONCLUSIONS**

PECVD is suitable for growing silicon oxide at low temperatures which is surely an advantage, as the number of applications with low temperature requirements is increasing.

In order to achieve a better understanding of the PECVD process, in the 1<sup>st</sup> part of the paper it was highlighted a theoretical study and the equipments used in this order to obtain SiO<sub>x</sub> layers. In order to highlight the equipments used in this process, were designed 3D images in Unigraphics NX5, a special soft used in industry execution design.

It was coated three samples with three different composition of plasma. The plasma composition was changed by increasing or decreasing the ration between O<sub>2</sub> and Ar. In order to evaluate the chemical composition of SiO<sub>x</sub> layer it was used EDS technique. Thereby, EDS provide us the quantity of each element of the coatings (Table 1).

Thereby, the goal of this paper is to highlight by EDS analysis that the SiO<sub>x</sub> layer chemical composition can be controlled by changing the Ar:O<sub>2</sub>:TEOS quantity components.

**4. REFERENCES**

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