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Synthesis and Microstructure $CaTiO_3$ coating by Sol-Gel Spin-Coating Process

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ABSTRACT

Recently, Calcium Titanate has been introduced as a bioactive bioceramic with acceptable mechanical and better biological properties compared to hydroxyapatite for orthopaedic implant applications. In this study, $CaTiO_3$nano-structure coating was produced by sol-gel spin-coating route for biomedical applications. Calcium oxide and titanium isopropoxide were used as a precursor for the sol-gel spin-coating. After coating process, the specimen was subjected to heating in oven at 100°C for 24 hours and the sample was heated at 800°C for 2 hours. The phase structure and surface morphology of coating were investigated by X-ray diffraction (XRD) and scanning electron microscopy (SEM). Finally, it concluded that the uniform crack-free nano-structured $CaTiO_3$ coatings could be used for the biomedical application.

Keywords— Sol-Gel Spin Coating, $CaTiO_3$, Microstructure, Bioactive coating

I. INTRODUCTION

The main purpose of surface modifications for biomaterials is to improve tissue responses in a living body because tissue biomaterial reactions are interfacial phenomena which are governed by surface properties of the biomaterial. Ceramic coatings are often applied to facilitate osteogenesis on metallic biomaterials. Among ceramics, hydroxyapatite (HA) is the most popular coating material [1–5]. Many researchers have demonstrated good osteogenesis on HA-coated metals [6–9], and HA-coated titanium prepared with a plasma-spraying process has been used clinically [10–12]. However, fractures at the HA titanium interface and in the HA layer itself are often degraded after long-term use in the human body [13]. Accidents caused by these fractures result in a loss of the biomaterial-bone fixation. Consequently, clinical use of the HA-coated titanium has decreased in recent year.

Recently, some of the present authors succeeded in developing a bioactive calcium titanate (CaTiO3) coating which can activate osteogenesis on titanium [14-17]. The bioactive $CaTiO_3$ film was prepared by radiofrequency (RF) magnetron sputtering with a CaTiO3 target in an argon atmosphere and post-annealing at 873 K in air [16,17]. The prepared film was crystallized into perovskite-type CaTiO3, and the chemical composition of the film was almost in accordance with that of stoichiometric CaTiO3. A remarkable feature of the bioactive CaTiO3 film was that the thickness was about 50 nm [17]. The thickness was 1/1000 that of plasma-sprayed HA coating. This thickness made it possible to improve the mechanical strength of the film itself. However, the post-annealing in air yielded not only crystallization of the CaTiO3 film but also the formation of a titanium-oxide layer in the interface between the film and the titanium substrate because of the oxidation of titanium, resulting in a change in the interface properties.

It was showed that the adhesion strength of the CaTiO3 film increases with a decrease in the thickness of the interfacial TiO2 layer [15]. By thinning the TiO2 layer up to halfits thickness, the adhesion strength estimated by the tensile test increased by approximately 40%. Likewise, Kobayashi et al. reported that in the case of sodium titanate film, the formation of an interfacial TiO2 layer formed by heating weakened the adhesion strength [18]. Consequently, in order to obtain a nondestructive bioactive CaTiO3 layer, development of new coating process without formation of the thick oxide layer is required.

The objective of the present work is to synthesis of CaTiO3 thin film using sol-gel process in which calcium oxide (CaO) and Titanium isopropoxide (C12H2804Ti)as starting materials, ethanol as the dispersed medium, ethylene-diamine-tetra-acetic acid (EDTA) as chilling agent for the reaction. X-ray diffraction (XRD) and Scanning Electron Microscopy (SEM) analysis were carried out to study the microstructural and morphological behavior of CaTiO3 thin films.

II. EXPERIMENTAL PROCEDURES

2.1 Preparation of $CaTiO_3$ Solution

The precursor materials used for the synthesis of $CaTiO_3$ were Calcium oxide (CaO) and Titanium (IV) isopropoxide ($Ti(OC_3H_7)_4$), EDTA, Ethanol and Acetic acid. Initially, CaO shall disperse in ethanol. The dispersed medium should be kept on a hot plate by maintaining a temperature at 90^{o}Cand the suspension should be stirred by an electromagnetic stirrer. Following this, an equivalent amount (keeping the Ca/Ti ratio same as $CaTiO_3$) of Titanium Isopropoxide solution should be added drop wise in the dispersed CaO medium. Then a catalyst, namely concentrated acetic acid shall be added toit. The stiring should continue for an hour. A few drops of a chelating agent (EDTA) should be added to the solution .This mixture should be allowed to stir for about 2 hours. The resulting solution is the $CaTiO_3$solution as depicted in figure 1.

Figure 1- CaTiO3 sol produced by sol gel Process.

2.2. Preparation of Thin Films

The $CaTiO_3$ thin films were prepared on titanium substrates, given in figure 2(a-b). The solution should be dropped on the well cleaned Titanium substrates and the substrates should be allowed to rotate at 3000 rpm for 3 minutes. After each coating CaTiO3 films should be dried at 100oC for 24 hours then heated in furnace at the temperature 900oC for 1 hour and left for the furnace cooling for 24 hour.The spin-coating and drying process should be repeated for three times.

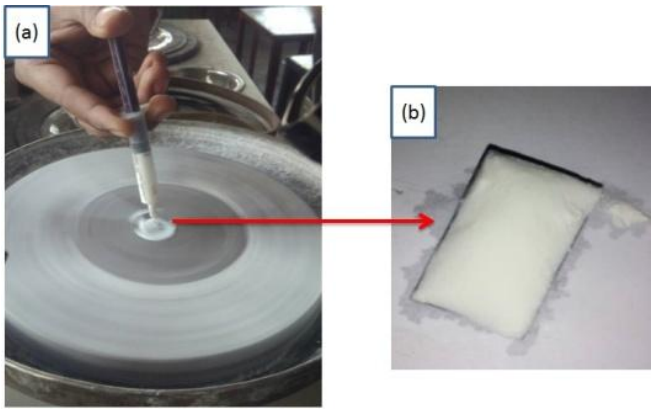

Figure 2- Top view of (a) coating during processing, (b) coated $CaTiO_3$ on Ti substrate.

2.3 Characterization of Deposited Thin Films

The $CaTiO_3$ thin film was analysised through the different characterization technique to study the composition, microstructures and morphology. X-ray diffraction (XRD) analysis was performed on a diffractometer (SHIMADZU, XRD700) using CuKα radiation for all analyses at 40 kV and 30mA in order to identify the phases of the films. The XRD patterns were recorded in the 2Θ range= 20°-120° using a step size of 0.02° and a counting time of 5s per step. The microstructural and compositional characteristics of powder particles were investigated by SEM (Scanning ElectronMicroscopy) attached with energy dispersive X-ray spectroscopy (EDS, OXFORD).

III. RESULTS AND DISCUSSION

3.1. Phase Analysis

Figure 3depicted XRD peaks of the crystalline $CaTiO_3$ films after drying at 100^{o}C for24 hours, which indicates the characteristic peaks corresponding to crystalline $CaTiO_3$is accurately matched to the peaks from JCPDS card number 03-0805. There were some traces of TiO_2in the filmpresent at (224) and (216) plane whereas $CaTiO_3$ present at (110), (220), (310) and (330) plane.

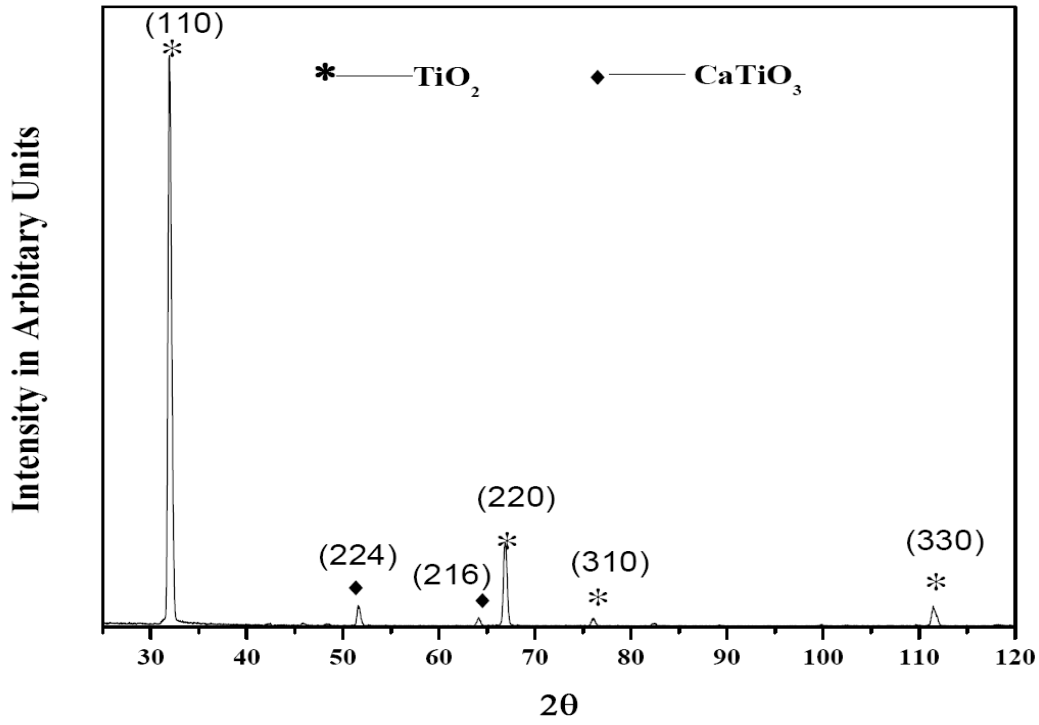

Figure 3- XRD analysis for $CaTiO_3$ thin film prepared by sol gel processing.

3.2. Morphological Analysis

Scanning electron microscope (SEM) is a promising technique for the topography study of thin film samples, as it provides valuable information regarding the size and shape of the particles or grains and also gives the information about the growth mechanism. The SEM image of $CaTiO_3$ thin film deposited on Titanium substrate is shown in Figure 4. This was indicated the uniform epitaxial growth of $CaTiO_3$ grains having size in the range of 10 µm in length and 5µm in width closely packed clusters covered on the substrate surface.

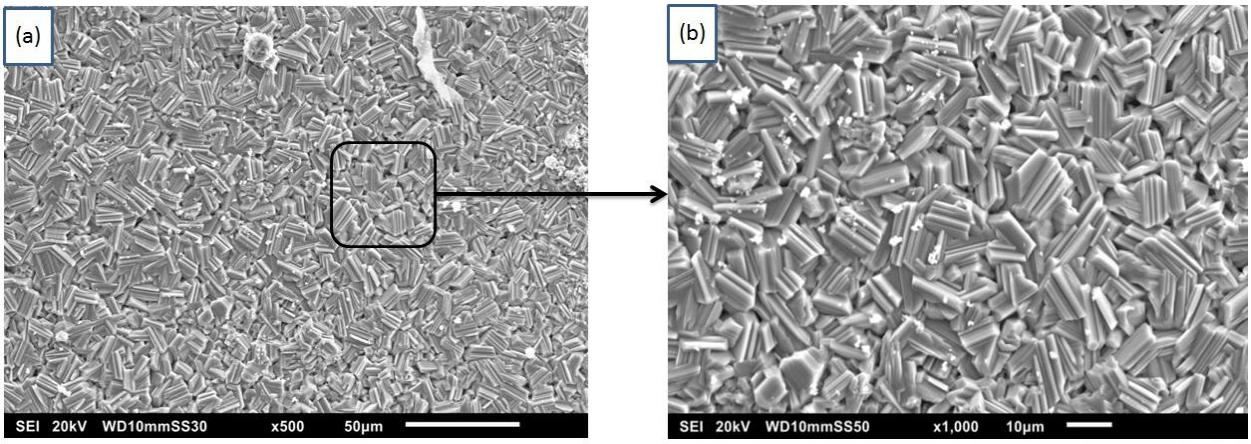

Figure 4- Microstructure and surfacemorphology of $CaTiO_3$ thin film at magnification (a) 500X and 1000X.

In the figure 5, the energy dispersive analysis of X-rays (EDAX) patterns of above SEM image of $CaTiO_3$ thin film conforms the calcium, titanium and oxygen elements present in the coated substrates.

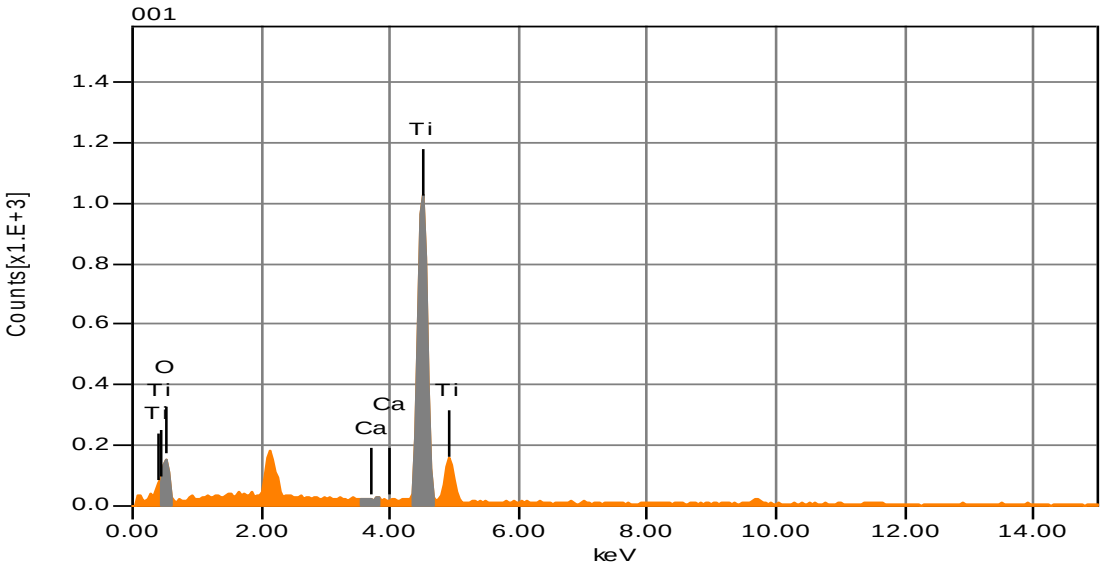

Figure 5- EDS pattern of $CaTiO_3$ thin film

IV. CONCLUSION

Based on the experimental results and analysis, the following conclusions have been presented.$CaTiO_3$ thin film is successfully prepared by sol gel spin coating process.

1. From XRD analysis confirms presence of crystalline $CaTiO_3$ phases after heat treatment at 900^oC for 1 hour. It also shows presence of some traces of TiO_2 phases.
2. The surface morphology obtained from SEM micrograph showed that the epitaxial growth grains with the uniform shape & size of 10µm in length and 5µm in width.
3. This indicates that the $CaTiO_3$ thin film can be used for the better biocompatibility and osteoconductivity of titanium alloy for the biomedical application (Dental and Hip Implant).

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