

Investigation on the Method of Ag Addition into TiO₂ coating Deposited on Unglazed Ceramic Tile

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ABSTRACT– Addition of Ag into TiO₂ coating was carried out by using two different methods. The first method involved Ag addition by dipping the TiO₂ coating into Ag nanoparticles solution at the last stage of the AgTiO₂ coating preparation, while the second method utilized AgNO₃ precursor that was added during the TiO₂ sol preparation. The produced AgTiO₂ coating by both methods was then characterized by X-ray diffraction method (XRD), scanning electron microscope (SEM) and energy dispersive spectroscopy (EDS). It was found that utilization of the second method (by adding AgNO₃ precursor into the TiO₂ sol) had produced TiO₂ coating with uniform distribution of Ag throughout the coating. Presence of Ag is observed at the surface of the coating as well as in the coating layer itself. While, AgTiO₂ coating produced via the first method (by dipping the TiO₂ coating into Ag nanoparticles) exhibits presence of Ag only in the coating layer.

1. INTRODUCTION

Titanium (IV) oxide, TiO₂ has been known for its photocatalyst and can serve as an antibacterial treatment, deodorization and water purification. However, it cannot be denied that the limitation of TiO₂ that is only reactive under UV irradiation and the fast recombination of hole–electron pairs which taking place within nanoseconds. Hence, to overcome the limitation, metal/metal oxide such as Fe, Pt, CuO, and Ag were introduced into TiO₂ particles [1]. Among all those metal, Ag was frequently chosen metal ions studied because it is the most promising metal ions collaborated with TiO₂. Various researches in developing a coating technique with proper surface structure and morphology to improve environmental decontamination has been carried out in adding metal oxide into TiO₂ coating. For example, M/TiO₂ (M = Ag, Au, Pt) nanocomposite thin films were deposited by co-sputtering method on quartz glass, or indium tin oxide (ITO) glass substrates by Yoon J.W. et al. [2]. Modification of TiO₂ nanotubes of Ag nanoparticles by polyol method at low temperature is said to be a convenient, versatile and low-cost method for the synthesis of metal nanostructures on a large scale [3]. Also, deposition of TiO₂ thin films on silicon wafers with different silver content via sol gel by spin-coating technique by Yu B. et al. [4] had found to promote uniform distribution and strong attached of silver

nanoparticles to the mesoporous TiO₂ matrix. The sol-gel synthesis of TiO₂ and Ag-TiO₂ coatings had also provide corrosion resistance and reduce bacterial adhesion when deposited on titanium substrate [5]. Besides, Ag/TiO₂ coating layer on a stainless steel substrate had been studied by Wafa et al. [6] using spray coating technique. Despite all the approaches, the addition of Ag into the TiO₂ coating itself is not yet comprehensively studied in terms of its method and amount of Ag needed for optimized the antimicrobial and photocatalytic performance. Specifically, there is no study reported of AgTiO₂ coating deposited on ceramic tile substrate. Therefore, in this paper, the investigation on the method of Ag addition in TiO₂ coating was conducted specifically to study on its effect towards its crystallinity and microstructure.

2. METHODOLOGY

Material and method. Generally, for the preparation of TiO₂ sol, two types of solutions (Sol A and Sol B) were prepared as previous method [7]. In order to introduce Ag silver ions, two different methods were utilized. The first method is by 10 times dipped TiO₂ coating into 1000 ppm Ag nanoparticles solution. Another method involved the addition of 10 mol % AgNO₃ precursor into the TiO₂ sol preparation followed by 5 times dipped of the prepared AgTiO₂ sol during the dip-coating procedure. Coating were deposited on unglazed ceramics tile (20 mm x 10 mm x 5 mm) with surface roughness of 5.5 ± 0.2 µm measured using portable profilometer (Mitutoyo SJ-301). The coating procedure was carried out at 30 mm/min dipping speed and 5s dwelling time. The AgTiO₂ coating dried for 24 hours followed by oven dry for 30 minutes at 110°C. The coated tiles then heat treated at 500°C, rate 2°C/min for 1 hour. The crystallinity of the coated tiles measured by GAXRD at 2θ range of 10° – 80° with glazing angle of 4° using the PANalytical X'PERT PRO MPD Model PW 3060/60 with Cu Kα of 1.54060Å°. Morphology and element analysis were examined by Scanning Electron Microscope (SEM) coupled with an Energy-Dispersive X-ray Spectrometer (EDX) (JEOL model JSM-6010PLUS/LV).

3. RESULTS AND DISCUSSION

Crystallinity and microstructure analysis. AgTiO₂ coating produced via dipping the TiO₂ coating into Ag nanoparticles solution method exhibited anatase phase with dominant crystalline peaks at angle 2θ of 25.4°, 48° and 53.9° (Fig. 3.1(a)). Meanwhile, by using AgNO₃ precursor, the presence of Ag peaks are overlapping with the anatase peak, which support the existence of Ag⁰ at 44.2° and 64.3° and Ag₂O at 32.3° (Fig 3.1 (b)). The existence of Ag oxide in the coating results from thermal decomposition of AgNO₃ during the calcination process [8].

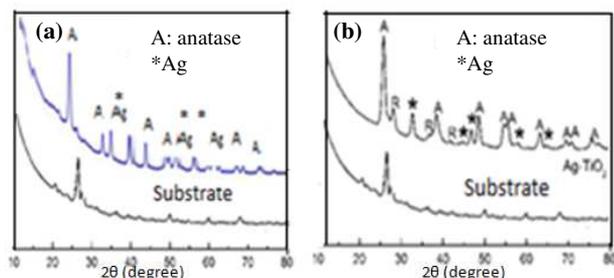


Fig 3.1: XRD pattern of AgTiO₂ coating (a) Dipping into Ag nanoparticles solution and (b) Using AgNO₃ precursor

The surface morphology of AgTiO₂ coating via dipping the TiO₂ coating into the Ag nanoparticles solution in Fig 3.2(a) exhibited more dense structure of a homogenous coating while coating produced using the AgNO₃ precursor in Fig 3.2 (b) appeared as a layer with pores, few agglomerates are distributed randomly and exhibit cracks.

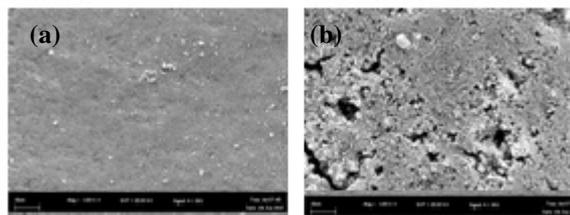


Fig 3.2: Surface morphology of AgTiO₂ coating on ceramic tile (a) Dipping into Ag nanoparticles and (b) Using AgNO₃ precursor

The SEM cross section analyses, shows that AgTiO₂ coating produced by dipping the TiO₂ coating into Ag nanoparticles, a lower percentage of Ag presence on the surface (0.15 wt %) and within the film (0.12 wt %) (Fig 3.3 (a)) compared to the TiO₂ coating produces via the addition of AgNO₃ precursor which shows 4.98 wt % Ag on the surface and 2.39 wt % within the film (Fig 3.3 (b)). Thus, it can be deduced that the method of adding AgNO₃ precursor in the sol promoted higher percent of Ag in the produced AgTiO₂ coating compared to the method of dipping the TiO₂ coating into Ag nanoparticles solution. The presence of silver deposited on the surface was due to the released of Ag⁺ ions from silver nanoparticles in dark and UV irradiation [9]. Here, the TiO₂ thin film also acted as a barrier layer preventing free ions release thus implies that the silver ions diffuse into the lattice of TiO₂.

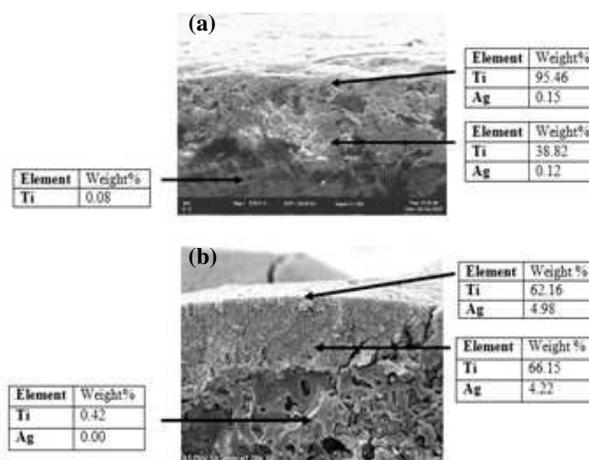


Fig 3.3: Cross sectional morphology and EDX spectrum of ceramic tile (a) Dipping into Ag nanoparticles and (b) Using AgNO₃ precursor

4. CONCLUSIONS

In summary, the addition method of Ag using AgNO₃ precursors into the TiO₂ sol had produce AgTiO₂ coating layer with the existence of Ag particles throughout the coating. While, the dipping method of TiO₂ coating into Ag nanoparticles solution shows less presence of Ag both on the surface and in the coating. Hence, further work will utilize the method of Ag addition via the AgNO₃ precursor method to proceed study on the AgTiO₂ coating deposited on unglazed ceramic tiles.

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