


EFFECT OF TiO₂ LAYER THICKNESS ON ELECTRODE DEGRADATION IN REDOX FLOW BATTERIES

 Shukhrat Ch. Iskandarov*,  Ilyos Kh. Khudaykulov,  Temur K. Turdaliev,

 Usmonjon F. Berdiyev,  Sardor A. Tulaganov, Boburjon R. Kakhramonov

Institute of Ion-Plasma and Laser Technologies named after U.A. Arifov, Academy of Sciences of the Republic of Uzbekistan, 100125, Durmon yuli st. 33, Tashkent, Uzbekistan

**Corresponding Author e-mail: iskandarov@iplt.uz*

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In this work, TiO₂ coatings of varying thicknesses were deposited on carbon felt fibers via ALD (Atomic Layer Deposition), and the mechanical and electrochemical properties of the electrodes were studied. The experimental results show that the TiO₂ coating effectively protects and enhances the stability of carbon wet electrodes. While a 4% mass loss was observed on the untreated cathode electrode, this decreased to 1.6-1.7% with a TiO₂ coating. When the coating thickness was increased from 50 nm to 300 nm, no significant change in mass loss was observed, which indicates that even thin coatings provide effective protection. The mass loss in the anode electrode was relatively small, ranging from 2% in the untreated state and 0.5-1.1% in the coated states. This is explained by the lower anode potential than the cathode and the low sensitivity of the V²⁺/V³⁺ redox reaction at the anode. It was found that the titanium dioxide coating plays an important role in increasing the electrodes' electrochemical degradation and corrosion resistance, as well as extending battery life. It was also shown that a 50 nm thick TiO₂ coating can provide effective protection, whereas very thick coatings can limit electron mobility. These results confirm that TiO₂ coatings are one of the promising solutions for protecting electrode materials in vanadium flow batteries.

Keywords: *Electrodes; Electrocatalysts; Redox flow batteries; Energy storage systems; Metals; Metal oxides*

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INTRODUCTION

Currently, the main advantage of RFB technology is that it allows the use of various redox couples (for example, vanadium, iron-chromium or zinc bromide ions). The most widely studied and effectively used in practice is the fully vanadium redox battery (VRFB). This technology allows for efficient energy storage by using vanadium ions from a homogeneous element as an electrolyte [1]. The main components required for the operation of RFB include electrolytes [2], electrodes [3] and membranes [4].

Numerous scientific studies by scientists around the world have identified carbon-based materials that not only exhibit superior electrical conductivity for vanadium redox battery electrolytes but are also the most cost-effective materials for use in both the negative and positive half-cells of vanadium redox batteries. Commonly used carbon-based materials for RFBs include graphite felt (GF), carbon paper (CP), carbon nanotubes (CNTs), graphene oxide (GO), graphite, and graphene.

Graphite felt (GF) has a three-dimensional (3D) porous structure with high specific surface area [5] and many outstanding properties, such as excellent electrical conductivity, flexibility, corrosion resistance, and electrochemical stability [6,7]. Therefore, it has been widely studied as an electrode for energy and environmental protection, including vanadium-reduction-flow batteries (VRFBs) [8,9].

To improve the electrochemical activity of GF, the hydrophobic surface of the original GF should be changed to a hydrophilic surface, and in addition, the GF should maintain its natural good electrical conductivity at the same time. Several methods have been successfully used to modify the surface of GF, including plasma treatment [10], chemical cleaning [11], thermal treatment [12,13], nitrogenization treatment [14], carbon nanomaterial-based modification [15, 16], nanostructured metals [17], and metal oxides [18], among others.

In addition, the modified GF should exhibit long-term hydrophilicity stability and excellent chemical stability to protect electrochemical devices such as vanadium redox flow batteries (VRFBs), aluminum-ion batteries [19], etc., from corrosive electrolytes.

TiO₂ is an n-type semiconductor with natural hydrophilicity and excellent chemical stability in acidic media, and is used in various applications such as solar cells, photocatalysts, and sensors [20]. In addition, TiO₂ has been reported to enhance the wettability of the catalyst layer and is also chemically stable [21].

The hydrophobic surface of GF can be changed to hydrophilic by coating with TiO₂ films. When TiO₂-modified GF is applied to electrochemical devices, the TiO₂ surface coating can withstand the corrosion of a strong acidic electrolyte.

ALD is an advanced technique for growing highly conformal thin films on the surfaces of three-dimensional complex structures and nanostructures with atomic-level thickness control and excellent coating uniformity. Therefore,

TiO₂ films of different thicknesses can be uniformly coated on the surface of GF by ALD technology with precise control of the film thickness at the nanoscale.

In this work, we have grown a nano-coated TiO₂ film (with different thicknesses) on the surface of GF, an ultra-thin TiO₂ film with nanocrystalline structure through atomic layer deposition (ALD) to modify the surface of GF, which not only imparts hydrophilicity to GF, but also ensures sufficient conductivity of ALD-TiO₂ modified GF (ALD-TiO₂/GF)[22]. With its good electrochemical properties, the ALD-TiO₂/GF electrode is expected to have potential applications in vanadium redox flow batteries (VRFB), aluminum-ion batteries, and other electrochemical energy storage devices.

METHODS AND EQUIPMENT

Electrode modification: The sample was coated with titanium dioxide using thermal atomic layer deposition (ALD). The process was performed using a precursor pair of titanium tetraisopropoxide (TTIP) and deionized water. The deposition parameters were as follows: nitrogen flow rate 120 cm³/min, reactor pressure approximately 60 Pa, substrate temperature approximately 250 °C, and cycle duration 8 s. Each ALD cycle consisted of the following five steps: initial purge; 0.5 s TTIP pulse; 3 s nitrogen purge; 1.5 s H₂O pulse; and a final 3 s nitrogen purge. Under these conditions, the growth per cycle (GPC) was determined to be 0.2 nm. A detailed description of the deposition process can be found in the literature below [23].

Electrolyte preparation: We used recycled V₂O₅ powder, sulfuric acid, and deionized water to prepare a 1.5 mol/l vanadium electrolyte. For this purpose, we ground the recycled V₂O₅ powder in a special mill and passed it through a 400 μm sieve to separate the V₂O₅ oxide powder of the same size for melting. In addition, a single-cell membrane electronic block (MEB) was assembled to test the electrode system.

Laboratory model of the membrane electronic block (MEB): We used a Nafion-117 membrane as a separator between the prepared graphite bipolar plate to assemble the MEB. We used a 2 mm thick silicone rubber gasket as the compression layer for the graphite bipolar plate, with a working area of 42 mm × 40 mm. Instead, two identical electrodes of the same dimensions and 3 mm thickness, made of carbon felt (Zibo Ouzheng Carbon Co., Ltd., China), were employed to assemble a single-cell MEB. To prevent the graphite bipolar plate from breaking and to compress it with uniform force, an external compression mold was made using HP panels. This HP panel not only protects the graphite plate from cracking but also uses a 0.5 mm thick copper sheet as a collector between the graphite plate and the HP panel to remove electrons generated by the oxidation-reduction reaction at the electrodes [24].

RESULTS AND DISCUSSIONS

Carbon felt was used as the electrode. Figure 1 below shows an image of carbon felt coated with titanium oxide using the ALD method. Figure 1 (left) shows a general image of carbon felt coated with titanium dioxide (TiO₂) using the ALD (Atomic Layer Deposition) method (200 μm). Figure 1 (right) shows an image of carbon fiber coated with titanium dioxide (TiO₂) using the ALD (Atomic Layer Deposition) method (10 μm). Here we can observe a change in the color of the fibers and an increase in their brightness. This optical effect is observed due to a change in the light scattering properties of the ray's incident on the surface of titanium dioxide.

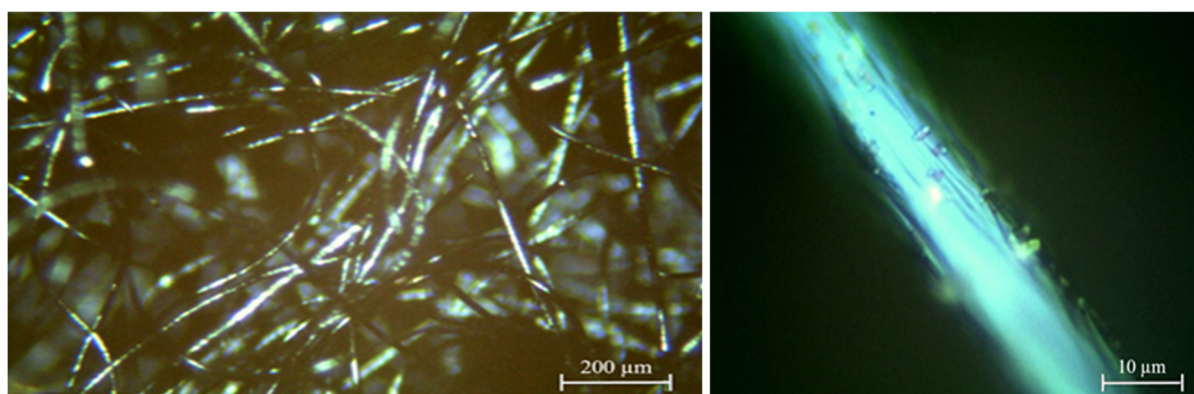


Figure 1. Optical microscope image of carbon felt. General view of the TiO₂ coated sample; (left), image of carbon fiber after TiO₂ coating; (right)

The images show that the fibers change color after being coated with titanium dioxide using ALD. This phenomenon is related to the interaction of light with the surface, the refractive index of titanium dioxide, and optical dispersion.

Since titanium dioxide has a high refractive index, the ability of the fibers to scatter light increases, making them appear brighter. As shown in the image above, the difference between the carbon felt coated with titanium oxide by ALD is visually apparent.

However, we conducted another method, Raman spectroscopy, to verify that the surface of the carbon felt was indeed coated with TiO₂ by ALD. The results of our analysis are presented in Figure 2. The red dashed line below shows

the Raman spectrum of the carbon felt without any treatment. The blue line shows the Raman spectrum of the carbon felt with a thin layer of TiO_2 , that is, modified.

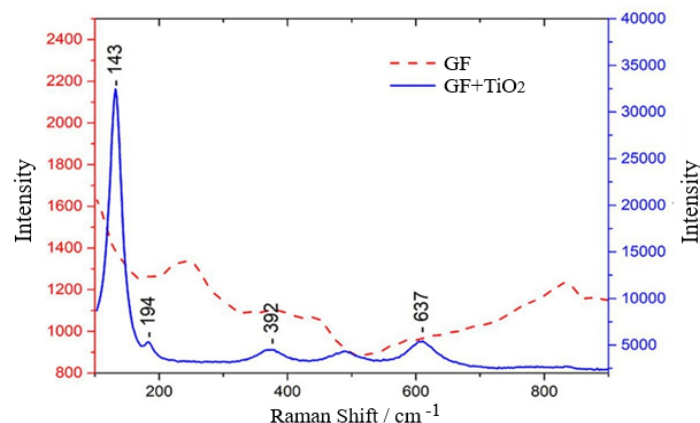


Figure 2. Raman spectra of carbon felt with and without TiO_2 nanocoating

In this graph, the peaks in the red dashed lines that characterize the sample without carbon felt treatment are not clearly distinguished by their intensity. However, we can observe characteristic Raman peaks in the blue line on the TiO_2 -coated, i.e. modified electrode. Here, 143 cm^{-1} (main peak) is characteristic of the anatase phase of TiO_2 , indicating the presence of nanostructured TiO_2 on the carbon felt surface [25]. The remaining peaks at 194 cm^{-1} , 392 cm^{-1} , 637 cm^{-1} are also associated with nanoscale structures of TiO_2 , reflecting their phonon vibrational properties. From the above optical microscope image and Raman spectrum results, it is clear that a TiO_2 anatase phase coating was formed on the surface of the carbon felt. Thus, we assembled a laboratory model of a single-cell vanadium flow battery to test our modified electrode by coating the TiO_2 coating on the surface of the carbon felt. We conducted a series of test experiments to test the resistance of the TiO_2 coated electrode to the charging and discharging processes.

We conducted our experiments by testing modified electrode materials with different thicknesses. The mechanism of the change in the open-circuit voltage curve over time during the charging process of our modified electrode with different thicknesses is shown in Figure 3 below.

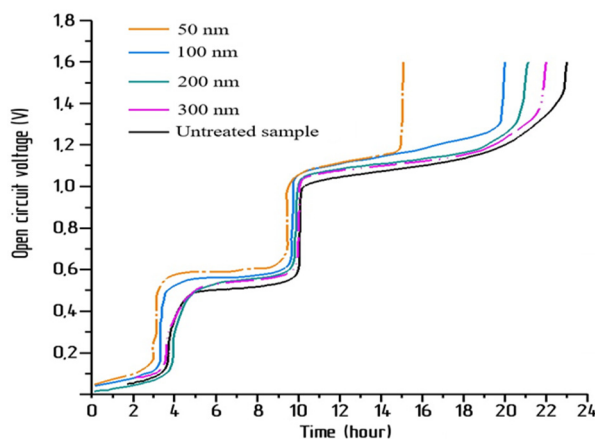


Figure 3. Graph of the open circuit voltage versus charging time of electrodes with TiO_2 coatings of different thicknesses during the first charge

Several studies have been conducted to prevent electrolysis corrosion of the carbon felt surface during the operation of a vanadium flow battery. Here, since the environment is acidic, a certain amount of corrosion is observed on the surface of the electrodes. For this reason, a material with good electrical conductivity and resistance to acidic environments is required as an electrode material. During charging and discharging (direct chemical oxidation), electrolysis can further accelerate the corrosion of the electrode surface. To avoid this problem, researchers are trying to modify the electrode surface with various metal oxides.

As mentioned above, a 50-300 nm thick TiO_2 coating was applied to the carbon felt surface using the ALD method, compared to an untreated sample. Figure 3 shows that the charging time for the untreated sample is 23 hours. For the modified electrode with a thickness of 50 nm, the charging time was reduced to 15.2 hours (1.5 times). At 100, 200 and 300 nm, the charging time is almost approaching the charging time of the untreated sample. This is because in the modified electrode with a thickness of 50 nm, TiO_2 fully demonstrates its catalytic and hydrophilic properties. At 100, 200 and 300 nm, the movement of electrons is hindered. This, in turn, causes the charging time to increase.

In addition, the studies were carried out several times, and after each study, the mass of the electrodes was measured. Initially, samples coated with TiO₂ coatings of 50 nm, 100 nm, 200 nm, and 300 nm thickness were selected and the mass loss (degradation) of each sample at the anode and cathode was measured over 300 cycles. Figure 4 presents the mass loss of the cathode and anode electrodes after modification with different thicknesses, compared to the initial sample.

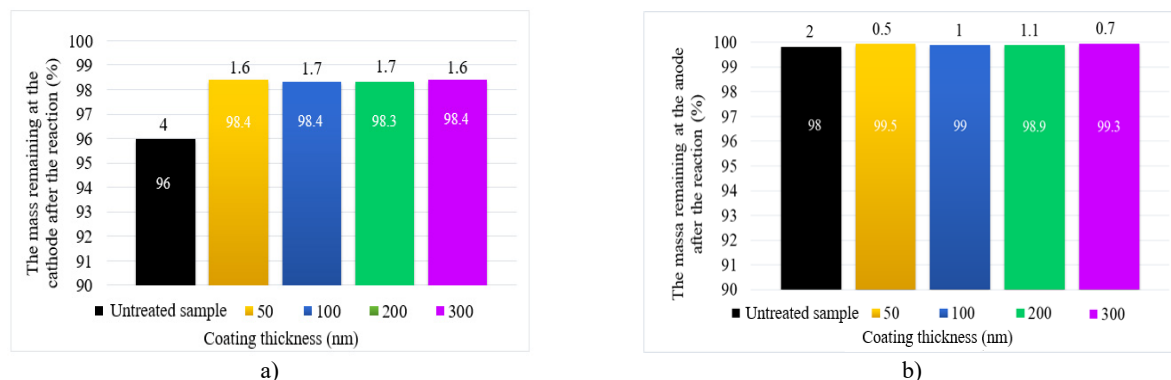


Figure 4 Histogram of electrode degradation (mass loss) during the first charging process. (a) Mass loss in untreated and modified cathodes; (b) Mass loss in untreated and modified anodes

In Figures 4a and 4b, the black columns represent the initial sample (without titanium oxide coating), where electrode degradation reaches up to 4%. In the samples with a TiO₂ coating, the mass loss decreased sharply, falling within the range of approximately 1.6–1.7%. Due to the chemical stability of the TiO₂ coating, it protects the electrode surface from corrosion. In samples with a coating, passivation is formed, which limits the reaction of soluble material with the electrolyte. There was no significant difference between the coating thicknesses of 50 nm and 300 nm, which indicates that the carbon felt was effectively protected with a thin layer of TiO₂.

Thus, cathode electrodes coated with titanium dioxide allowed to reduce mass loss by up to 2.3 times. Increasing the thickness can prevent the release of electrons from the electrode surface. This confirms the effectiveness of the 50 nm TiO₂ coating in protecting the electrode materials.

As can be seen from Figure 4-b), the mass loss in the anode system of vanadium flow batteries is very low - 2% for the untreated sample, and in the case of TiO₂ coating, we can observe a mass loss in the range of 0.5–1.1%. The anode process in vanadium flow batteries occurs through the V²⁺ - V³⁺ reaction. This process usually does not cause significant material loss, since the electron exchange does not significantly change the structure of the anode electrode.

CONCLUSIONS

The study's results confirmed that titanium dioxide (TiO₂) coatings effectively enhance the mechanical and electrochemical stability of carbon felt electrodes. In the initial case, a 4% mass loss was observed in the cathode, but this decreased to 1.6–1.7% when the TiO₂ coating was applied. The fact that increasing the coating thickness from 50 nm to 300 nm did not result in a significant difference in mass loss indicates that even thin coatings can provide effective protection. The mass loss in the anode electrode was relatively small, initially at 2%, and in the range of 0.5–1.1% when the TiO₂ coating was applied. This is explained by the low sensitivity of the V²⁺/V³⁺ reaction at the anode in vanadium flow batteries, and by the lower anode potential relative to the cathode. Titanium dioxide coating increases the resistance of electrodes to corrosion and electrochemical corrosion, extending their service life. In coated samples, mass loss in cathode electrodes was reduced by a factor of 2.3. Thin coatings (50 nm) can also provide effective protection, but very thick coatings are likely to restrict electron mobility.

TiO₂ coating plays an important role in protecting electrode materials in vanadium flow batteries. Coatings protect the anode and cathode electrodes from mechanical and electrochemical corrosion, ensuring long-term stability of batteries. Therefore, TiO₂ coating is a promising method for improving the efficiency of energy storage systems in the future.

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ORCID

Shukhrat Ch. Iskandarov, <https://orcid.org/0000-0002-3002-9141>; Ilyos Kh. Khudaykulov, <https://orcid.org/0000-0002-2335-4456>
Temur K. Turdaliev, <https://orcid.org/0000-0002-0732-9357>; Usmonjon F. Berdiyev, <https://orcid.org/0000-0003-2808-0105>
Sardor A. Tulaganov, <https://orcid.org/0000-0003-1881-2165>;

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ВПЛИВ ТОВЩИНИ ШАРУ TiO₂ НА ДЕГРАДАЦІЮ ЕЛЕКТРОДІВ В ОКИСНОВІДНОВНИХ ПРОТОЧНИХ АКУМУЛЯТОРАХ

Шухрат Ч. Искандаров, Ильос Х. Худайкулов, Темура К. Турдалиев, Усмонджон Ф. Бердиев,
Сардор А. Тулаганов, Бобурджон Р. Кахрамов

Институт ионно-плазмовых та лазерных технологий имени У.А. Арифова, Академия наук Республики Узбекистан,
100125, вул. Дурмон йули, 33, Ташкент, Узбекистан

У цій роботі покриття TiO_2 різної товщини були нанесені на вуглецеві волокна фетру методом ALD (атомно-шарове осадження), а також досліджені механічні та електрохімічні властивості електродів. Експериментальні результати показують, що покриття TiO_2 ефективно захищає та підвищує стабільність вуглецевих мокрих електродів. Хоча втрата маси необробленого катодного електрода спостерігалася на рівні 4%, цей показник зменшився до 1,6-1,7% після нанесення покриття TiO_2 . Коли товщина покриття збільшилася з 50 нм до 300 нм, суттєвої зміни втрати маси не спостерігалось, що свідчить про те, що навіть тонкі покриття забезпечують ефективний захист. Втрата маси анодного електрода була відносно невеликою, коливаючись від 2% у необробленому стані до 0,5-1,1% у покритому стані. Це пояснюється нижчим анодним потенціалом, ніж у катода, та низькою чутливістю окисно-відновної реакції $\text{V}^{2+}/\text{V}^{3+}$ на аноді. Було виявлено, що покриття з діоксиду титану відіграє важливу роль у підвищенні електрохімічної деградації та корозійної стійкості електродів, а також у подовженні терміну служби батареї. Також було показано, що покриття TiO_2 товщиною 50 нм може забезпечити ефективний захист, тоді як дуже товсті покриття можуть обмежувати рухливість електронів. Ці результати підтверджують, що покриття TiO_2 є одним із перспективних рішень для захисту електродних матеріалів у ванадієвих проточних батареях.

Ключові слова: *електроди; електрокаталізатори; окисно-відновні проточні батареї; системи накопичення енергії; метали; оксиди металів*