Enhanced Corrosion Behavior of Reinforcing Steel in Concrete Using Titanium Nano-composite Thin Films

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ABSTRACT

In this study, a thermionic vacuum arc (TVA) system is performed in order to apply nano-coatings on the structural rebar using titanium (Ti), titanium dioxide (TiO_2) and titanium diboride (TiB_2). The corrosion reactions occur on the reinforcement surface and in this process, the coatings acting as barriers on the reinforcement play an important role in blocking these reactions. The thin film coatings are nano-structured, compact and homogeneous. The deposition time of thin film in this technique is very short. After the implementation of these coatings on reinforced concrete samples, accelerated corrosion tests were done so that the effectiveness of coatings against corrosion can be examined. These experiments showed that nano-coatings increased the corrosion resistance of the structural steels used in reinforced concrete structures. During the harmful effects that threaten the reinforced sample, the impact of coatings has emerged and reduced the corrosion of steel reinforcements. The best results were obtained with TiB_2-coated sample with 48% lower corrosion current and this coating also expanded the damage occurrence time by 119% compared to uncoated rebar.

KEYWORDS: Concrete, Nano-coatings, Corrosion, Reinforcement, Titanium diboride.

INTRODUCTION

In line with the expansion in the construction sector, concrete technology also developed (Topçu and Ateşin, 2016). The long-lasting concrete condition is required in addition to the production of high-strength concrete. One of the most important points needing to be solved is that the rebar in the reinforced concrete structure is corroded for various reasons (Ababneh et al., 2009).

In recent years, the world frequently experienced horrific and destructive effects of earthquakes and such natural disasters cost many lives and properties. Although the cause of the great destruction caused by earthquakes in the society is not well known, the most important reason is corrosion; that is, the loss of bearing capacity by rusting and rotting of the steel rebar in the carrier systems of reinforced concrete buildings. These earthquakes once again revealed that they are not earthquakes that kill people, but they decay buildings. Research shows that most of the injuries and deaths in earthquakes are caused by people being trapped under collapsed buildings.

The effect of corrosion starts with micro-cracks in the concrete and slowly, the bearing capacity is reduced (Yan et al., 2019). Due to corrosion, the bond strength (adherence) between steel and concrete is decreased, as well as ductility. Corrosion affects the durability of rebar; therefore, usage of this kind of corroded structural element would not be reliable and if this situation persists, the element is not able to complete its lifetime predicted in the design stage. As a result of corrosion, the structure becomes vulnerable to earthquakes (Bicer et al., 2018).

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In recent years, many methods have been tried to protect rebar from corrosion (Koch, 2017). While some of these methods provide temporary protection, some of them significantly increase cost (Hou et al., 2017). The method of coating the reinforcement has been tested by many researchers before with many different materials (Cheng et al., 2021; Tobbala et al., 2020). Corrosion-resistant parts can be produced especially by applying a suitable coating method and heat treatments to materials with good durability. Coating with high density and good adherence can supremely increase the corrosion resistance of the substrate (Hayatdavoudi and Rahsepar, 2017; Jinwei et al., 2020). Coatings are mainly made on iron and steel-based metal in order to obtain a protective layer against corrosion. Metals are coated with different methods, such as hot dipping, spraying, ion implantation or plasma.

Many researchers have reported that decrease of bond strength is the main problem on epoxy-coated reinforcing bars according to the laboratory tests and also epoxy-coated rebars are expensive in comparison with normal rebars (Chang et al., 2002). Rebars coated with epoxy should be placed directly in the mold without being damaged. However, some defects in the coatings cannot be prevented (Maya-Visuet, 2015). If these defects are not repaired, they cause pitting corrosion in the reinforcement. Also, in damaged places on the coating, in case of corrosion of steel, it can remove the entire coating layer for both paint and plastic coatings. Application of such coatings is risky and unsafe. Over and above, these coating techniques are not long-lasting and require constant maintenance over time.

Afshar et al. (2020) investigated the corrosion resistance of rebars by using different mineral admixtures and different types of steel. They also applied various types of coating on rebars, such as hot-dip galvanization, polyamide epoxy and alkyd-based primer in order to see their effects against corrosion. They experimented on samples with electrochemical methods and compressive strength tests. They observed that the best result for corrosion resistance is given by rebars coated with zinc-rich epoxy primer included with silica fume and fly ash. They also found that samples with polyurethane gave the highest strength and the lowest corrosion rate. According to their study, the alkyd-based primer was the most economical coating system; however, it was the weakest coating type for anti-corrosion.

Bernard et al. (1993) studied the corrosion resistance of zinc-coated steels using Raman spectroscopy. Test specimens are divided into two groups; those painted with epoxy resin and unpainted ones. After 7 days of immersion in NaCI solution, they observed a large diversity of corrosion products on uncoated steels. These products were zinc oxide and zinc hydroxide. In terms of painted samples, they identified zinc hydroxy chloride and zinc oxide under the paint blisters. This means that there is a different type of corrosion reactions happening under the epoxy film. It is seen that the properties of paint are able to change the mechanism and thus the nature of the corrosion products. Nevertheless, they saw that the corrosion process for both coated and uncoated specimens depends on the chloride concentration.

According to Zhou et al. (2014) SiO_2 nanoparticles increase the corrosion resistance and provide a barrier property with aspect of low corrosion rates and high impedances. Corrosion studies were performed in 3.5 wt% NaCl solution with mild steel. Arukalam et al. (2018) used the nano ZnO polysiloxane in the marine environment. In this study, the coating shows super-hydrophobic properties with low surface energy. Nanoparticles also show great corrosion resistance performance with impedance modulus higher than 10^9 Ω cm^2. Di et al. (2016) have performed corrosion studies in 3.5 wt% NaCl solution for 1 hour by using graphene oxide/zirconia dioxide as corrosion-resistant hybrid coatings. They found that the coating has a superior barrier effect, a high specific surface and an excellent corrosion resistance on steels.

Cao et al. (2020) fabricated a coating on steel consisting of bio-based polybenzoxazine which has corrosion resistance. They saw that polybenzoxazine coating provides good durability properties against adverse environment. The coating was immersed in a 3.5% NaCl aqueous solution and showed a significant corrosion resistance. As a result, nanoparticles of coating developed a passive layer on the reinforcement and this is a promising investigation for corrosion protection.

Al-Zahrani et al. (2002) investigated steel reinforcement corrosion by using polymer-modified and waterproofing coating in order to evaluate the mechanical properties of concrete specimens. They used the accelerated corrosion test on coated and uncoated concrete specimens. Also, the physical properties of concrete specimens were evaluated for five months by heating-cooling and wetting-drying cycles. They determined the physical properties of concrete specimens with the help of chloride permeability, water absorption and adhesion tests. The results of the accelerated corrosion test obviously showed that polyurethane elastomer-based waterproofing coating provides better corrosion resistance than in specimens coated with other waterproofing materials.

In this study, we used the thermionic vacuum arc (TVA) system in order to apply nano-coatings on the structural rebar using Ti, TiO_2 and TiB_2 materials. The corrosion reaction occurs on the reinforcement surface. In this process, the coatings acting as barriers on the reinforcement play an important role in blocking these surface reactions. The coatings are nano-structured, compact and homogeneous. The deposition time of thin film in this technique was very short. Accelerated corrosion tests were performed in order to determine the durability properties of reinforced concrete samples.

Experimental Study

Materials and Properties

In the experimental study, CEM I 42.5 R Portland cement was used which was produced by Eskişehir Cement Factory and satisfied the ASTM C150/C150M-21 standard (ASTM, 2021). The test results of the sieve analysis of aggregates are given in Figure 1. Limestone-based crushed sand (0-4 mm), crushed stone I (4-11.2 mm) and crushed stone II (11.2-22.4 mm) aggregates obtained from local quarries were used. The specific weights of crushed sand, crushed stone I and crushed stone II aggregates were found to be 2.67, 2.70 and 2.70, respectively. The sieve analysis of aggregates is shown in Figure 1. In this study, aggregates were used at the percentages of 50% sand, 15% NO I, 35% NO II.

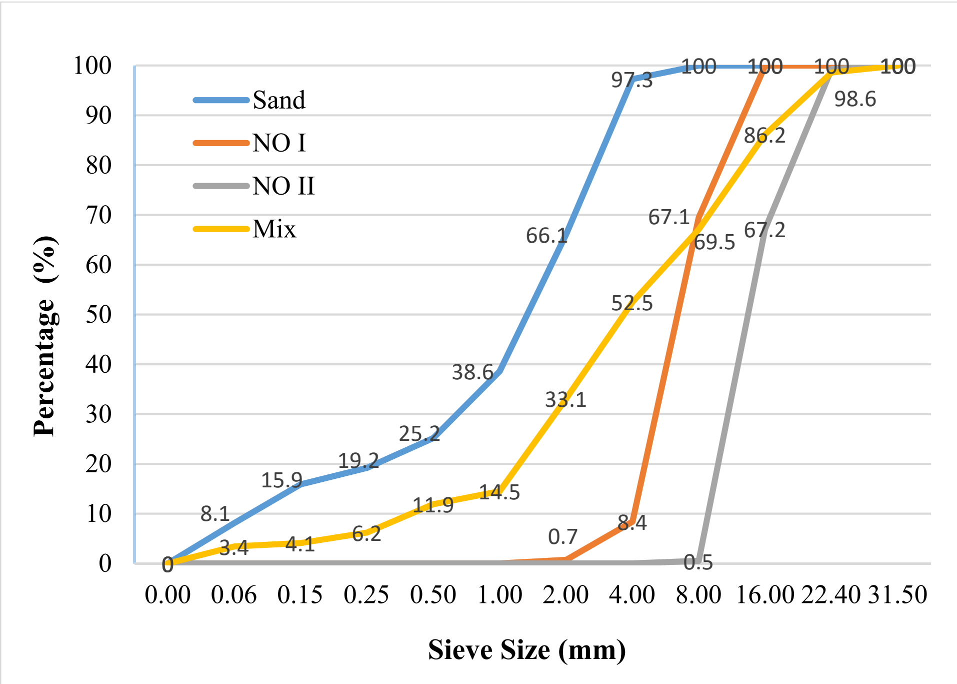

Figure (1): Sieve analysis of aggregates

In the production of reinforced concrete samples, 16-mm-diameter deformed B420C reinforced concrete steel was used (ASTM, 2020). Length of the reinforcements was 30 cm for all the specimens. Industrial sodium chloride salt was used in the accelerated corrosion tests conducted to determine the corrosion performance of reinforced concrete samples.

Curing Conditions, Mix Design and Specimen Preparation

In order to determine the durability properties of concrete, 100x200 mm cylinder concrete specimens were produced. The concrete casting process and the concrete mix properties were convenient to standards. Cylindrical samples were produced separately with reinforcement for each coating series. 30 cm long rebars were placed at the center of the cylinders. The rebar was 15 cm left, on top of the concrete for each specimen. The produced concrete samples were kept in molds in the laboratory environment for 24 hours. Standard curing was applied to the concrete samples until the 28th day in lime-saturated water pools at 20 ± 2 ºC. Concrete mixing proportions are given in Table 1.

Table 1. Concrete mixing proportions (kg/m^3)

Cement (kg)	Water (kg)	Sand (kg)	Limestone No. I (kg)	Limestone No. II (kg)	Unit Weight
400	200	843.50	284.51	621.35	2349.36

Experimental Procedure

Producing Thin Films with Thermionic Vacuum Arc System

The thermionic vacuum arc (TVA) system is a physical vapor deposition (PVD) system. PVD coating technique can be expressed as the process of bonding the solid raw material to the material to be coated in a controlled manner by turning it into high energy and plasma. Although the TVA system contains a vacuum arc, the cathodic vacuum arc has a very different operating principle than other coating systems in the literature. The thin film production phase in the TVA system begins with the production of thermal electrons, then these electrons produce plasma and a plenty of high-energy ions appear. This is an ionic medium and the procedure happens under high vacuum. Plasma formation corresponds to the arc plasma region. That is why the system is called TVA. The TVA system is also used for electron beam evaporation (Pet et al., 2017). Table 2 shows the parameters of the TVA system used in this study during the application of the coatings.

Table 2. TVA parameters for the Ti, TiO_2 and TiB_2 coatings

Parameter	Unit	Ti	TiO_2	TiB_2
Filament Current	Ampere	20.7	21.5	19
Discharge Current	Milliampere	50	200	50
Ignition Voltage	Volt	1000	1300	900
Pressure	torr	$8x10^{-5}$	$8x10^{-5}$	$8x10^{-5}$
Time	Min	10	0.5	14

Recently, the quality of covering the surface using this technique with different materials has considerably improved (Pat et al., 2018). The advantages of the TVA technique are high quality, high purity, low roughness and high adhesion compared to other plasma-assisted techniques. In addition, coatings with a thickness of 2-100 nm do not change the thickness of the component to which they are applied (Pat et al., 2017).

TVA is an anodic plasma generation technique that grows thin film for many applications (Şilik et al., 2017; Cetin et al., 2013; Korkmaz et al., 2012). In this manuscript, we used the non-reactive TVA technique, in order to produce thin films on reinforced concrete rebars. Figure 2-a and Figure 2-b show the closed and open state of the system before the experiment. After the reinforced concrete samples are coated with nanostructures, the durability of these coatings on the rebar surface against corrosion was ready to experiment. The method and coating material that we use represent a unique system. The effectiveness of the coatings has been investigated by experimental methods mentioned in the following sections. In this study, rebar samples were coated with 3 different materials. Each coating showed an increased corrosion resistivity and reduced the corrosion rate differently.

Figure (2): (a) Outside view of thermionic vacuum arc system and (b) Opening view of the system

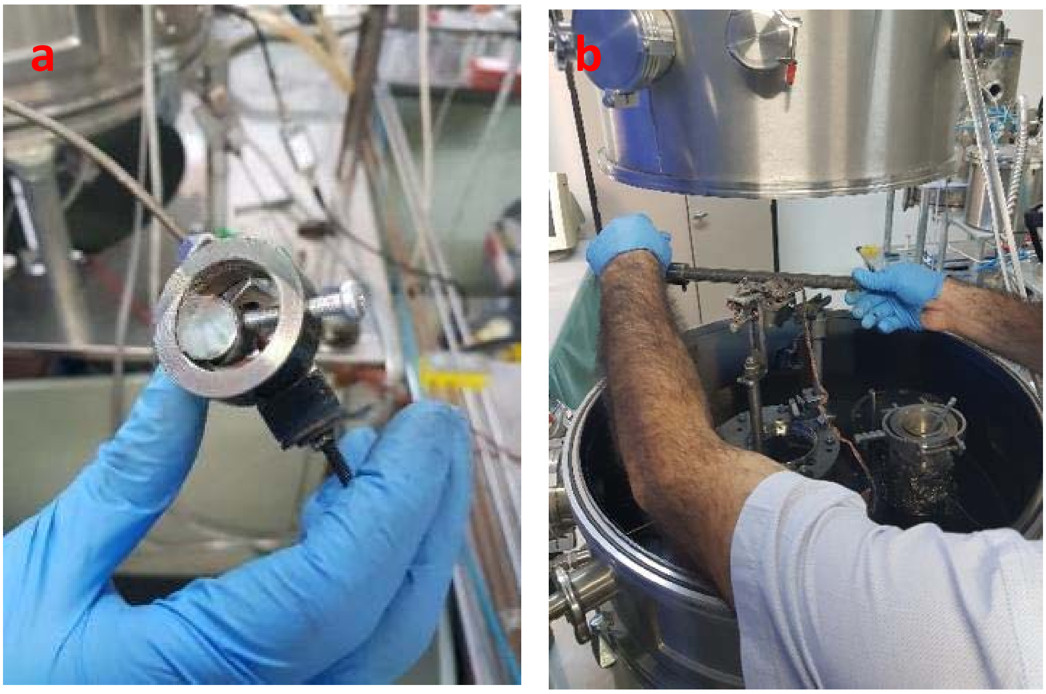

Figure (3): Coating process of the reinforcing steels in TVA system; (a) Coating the specimens for surface analysis and (b) Rebar coating for accelerated corrosion test

In Figure 3, the coating process of reinforcing steels in the TVA system was shown. Figure 3-a shows the 1-cm thick rebar samples that were coated for SEM analysis. After the rebar samples were carefully placed in the system as shown in Figure 3-b, the system was closed and the valves around it were tightened. Those 30-cm long samples were coated for the accelerated corrosion test. The coating processes were carried out neatly, so that no empty space was left on the reinforcement. Subsequent to these processes, the samples were embedded in the concrete without damaging the coating.

Accelerated Corrosion Tests

The working principle of the accelerated corrosion device is based on creating an electrochemical circuit between the anode and cathode by giving an external current to the reinforcement (Topcu and Uzunömeroğlu, 2020; Bazán et al., 2018; Topcu and Uzunömeroğlu, 2021; Chen et al., 2020; Hay and Ostertag, 2020). The accelerated corrosion test started by applying a voltage of 20 V. Increasing the voltage accelerates the corrosion test and shortens the time of the corrosion process. The setup of the devices and containers used in the accelerated corrosion test is shown in Figure 4.

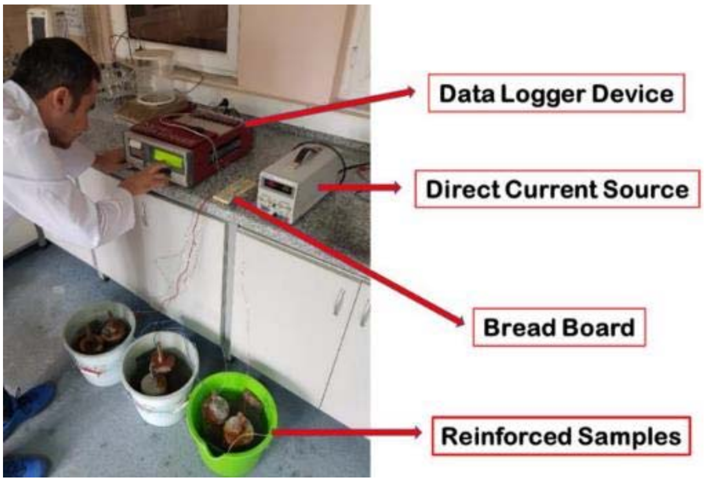

Figure (4): Applying the accelerated corrosion test on reinforced concrete samples

Implementation of this test is started with immersing the reinforced specimen into 4% sodium chloride solution which is reaching a half of the concrete sample. Specimens were monitored during the experiment till the first corrosion crack appeared on the surface. Meanwhile, current variation was recorded by the data logger device. When the specimens crack over time, the current increases suddenly indicating the occurrence of cracking. Cracks occurring by time on reinforced concrete and variation of applied current were recorded every 2 minutes for all concrete cylinders. Three specimens were made for each coated rebar and tested at the age of 28 days.

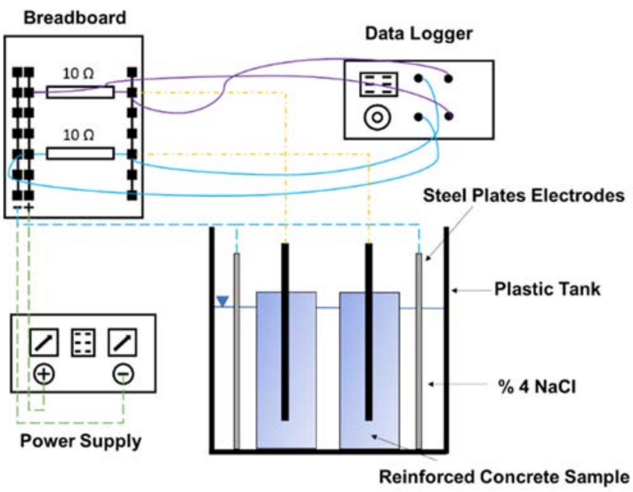

Figure (5): Schematic representation of accelerated corrosion test

The accelerated corrosion test is shown schematically in Figure 5. The experiment was set up so that two samples could be tested in each tank. The system is protected against power cuts so that the data logger device can record the data much properly. Interruptions in the current may cause the time-dependent corrosion current graph to be incorrectly obtained.

EXPERIMENTAL RESULTS AND DISCUSSION

Analysis of Thin Films

In order to make the surface analysis, specific rebar samples were produced and the surface of these samples was polished before the experiment. The thickness of the rebars was 2cm (Fig. 6).

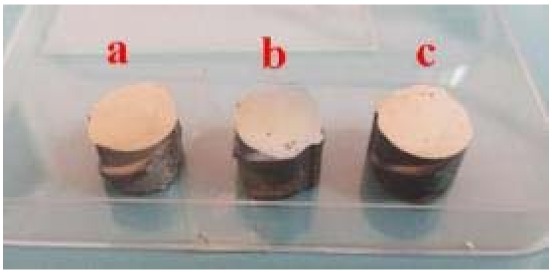

Figure (6): (a) Ti, (b) TiO_2 and (c) TiB_2 nano-coated rebars for surface analysis

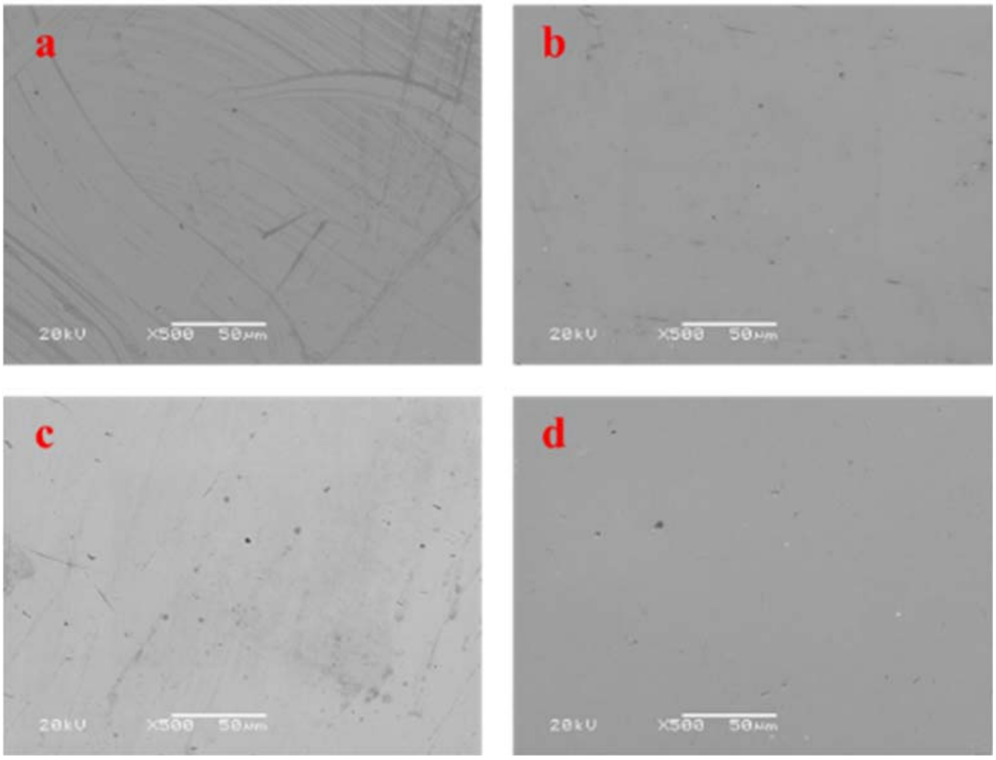

Figure (7): SEM analysis for (a) un-coated, (b) Ti, (c) TiO_2 and (d) TiB_2 nano-coated samples

Figure 7 shows the results of the SEM images. When Figure 7-a is examined, the lines of the polishing device on the control sample can be seen. In Figure 7-b, the titanium component coated on the same control sample is seen. Compounds containing titanium and titanium oxide coated the rebar surface as a thin film and covered all traces in the control sample.

Titanium diboride can fill even nano-sized spaces thanks to the boron element in the compound. When Figure 7-d is observed, it can be seen that the smallest voids on the base metal are filled with nano-coating plasma. All parts of Figure 7 are given at the same magnification for easy comparison.

Table 3 shows the proportions of elements in the surface of rebars. As can be seen, Ti spectrum was detected on the reinforcement surface. As a result of the analysis in Figure 8, 0.372 % Ti compound was found on the surface. This means that the thin-film coating is formed properly on the reinforcement surface. These coatings protected the reinforced concrete reinforcement from harmful effects, such as chloride.

Table 3. EDX analysis of the un-coated and coated rebars

Element	Control	Ti	TiO_2	TiB_2
Fe	100	99.820	91.070	99.867
Ti	-	0.180	0.372	0.133
B	-	-	-	0.000
O	-	-	8.558	-

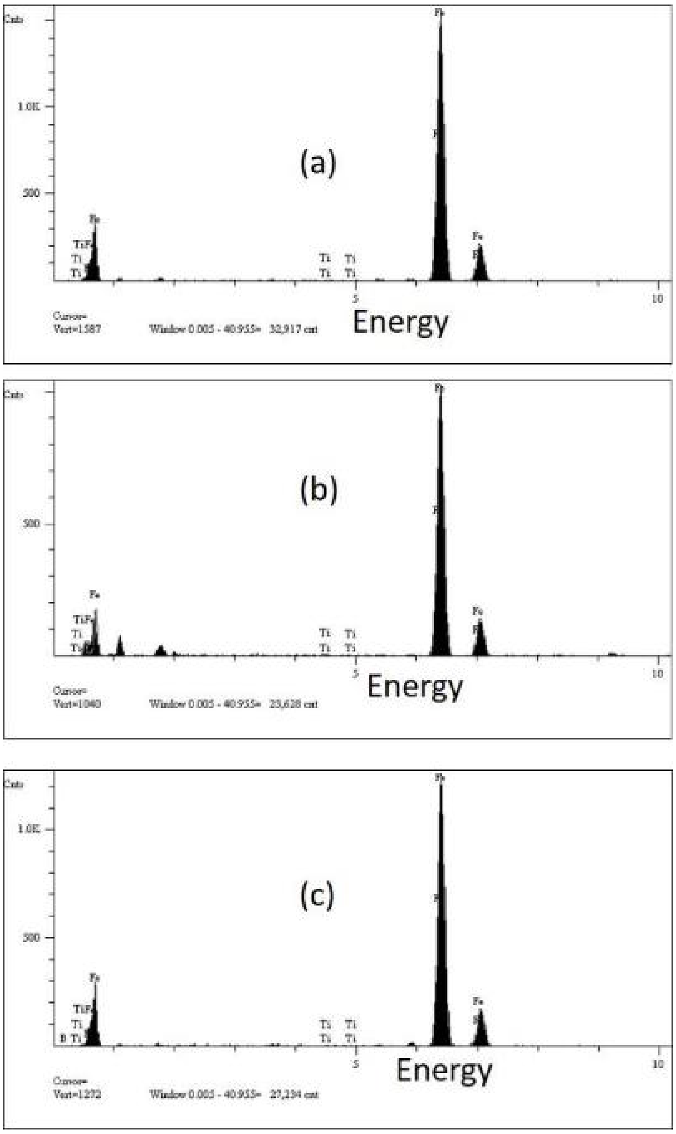

Figure (8): EDX analysis spectrum of rebar samples coated with (a) Ti, (b) TiO_2 and (c) TiB_2

As a result of EDX analysis in Figure 8, a thin film with a nano-crystalline structure formed on the surface. This crystalline structure is the coating itself and all reinforcements are covered in this way. The elements coated on the reinforcement completely envelop the surface and protect the reinforcement from corrosion.

Accelerated Corrosion Test Results

As a result of the accelerated corrosion test used within the scope of this study, the damages on the reinforced concrete samples and the time-dependent corrosion current graph are given below. The volume of corrosion products which formed as a result of corrosion reactions is greater than the volume of the steel before it undergoes corrosion. As a result, the volume expansion in the steel damaged the reinforced concrete samples as in Figure 9.

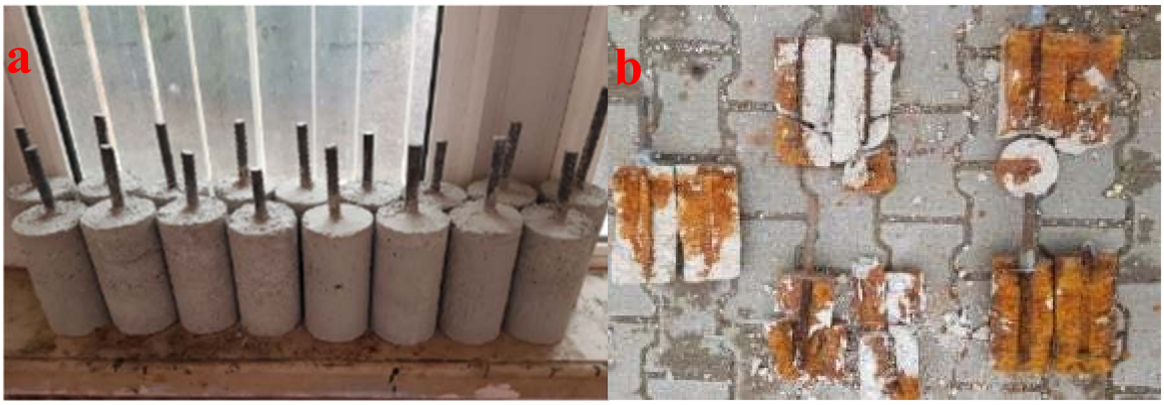

Figure (9): Damaged concrete samples (a) before and (b) after the accelerated corrosion tests

The images of the coated steel samples after cleaning with HCl acid are shown in Figure 9. As can be seen, mass losses have occurred in the reinforcements due to corrosion. According to the accelerated corrosion test results, all of the coated reinforcements gave better results compared to the uncoated control reinforcements.

When Figure 10 is examined, it is seen that the highest corrosion damage occurred in uncoated reinforcements, while the lowest corrosion damage occurred in titanium diboride-coated reinforcements. The coating with the lowest corrosion resistance is titanium. With this study, it can be said that as the quality of thin-film coating increases, the corrosion resistance improves.

According to the initial corrosion current values obtained from accelerated corrosion tests, it was seen that coated reinforcements gain significant corrosion resistance. In the uncoated reinforcements, there was a sudden increase in current on the first days and it was observed that the corrosion current values decreased on the following days. On the first days of the experiment, the corrosion current values continued steadily for coated reinforcements. However, on the following days, internal cracks occurred in the samples and the current reached high peaks and continued at high levels (Figure 11).

In Figure 10, when the first crack occurred in the concrete sample (according to the data logger), the current line in the graph increased sharply. In this study, the concrete quality of all the samples was the same. So, formation of the cracks just depended on the type of coating. According to Figure 11, it is seen that the most corrosion-resistant coating was TiB_2. It was also observed that the uncoated reinforcement was corroded for a very short time, like five days.

Initial corrosion currents and damage formation times (depending on these currents) are shown in Figure 12. As seen in the figure, the sample with the highest initial corrosion current absorbed by the system is the uncoated sample. Since the current applied by the accelerated corrosion test system is directly proportional to the corrosion potential, this means that the samples that absorb less current will be less corroded. Damage occurrence times are also seen on the same graph. Damage occurrence times increased according to the coating type, while damage occurred earlier in samples with uncoated rebar. In the control and Ti-coated samples, crack formation started and damage occurred earlier. However, TiO_2-coated and TiB_2-coated samples remained uncracked for 8 and 11 days, respectively. Since the concrete quality is the same for all the samples, the effectiveness of the coating is clearly visible in the samples that crack more lately. Within the scope of this study, it was observed that the most resistant coating against corrosion was TiB_2.

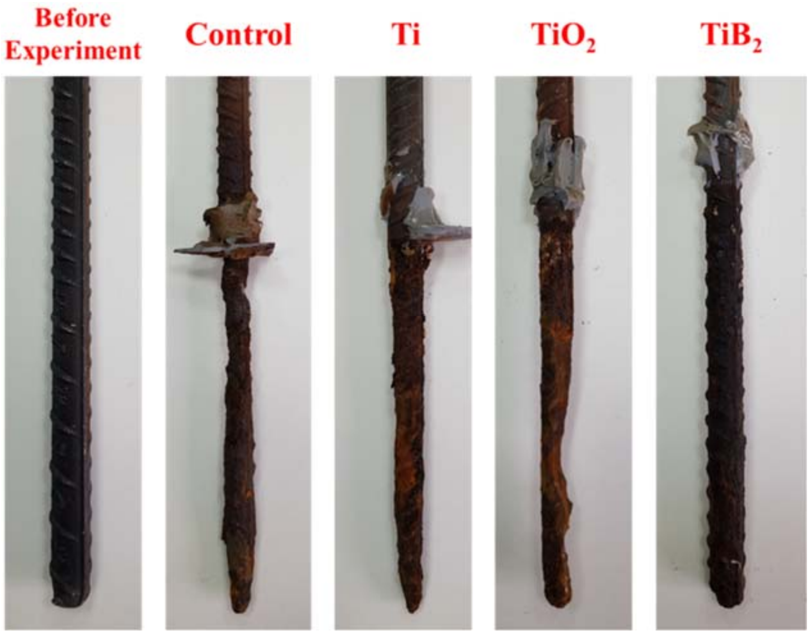

Figure (10): Coated reinforcements after the cleaning with HCI

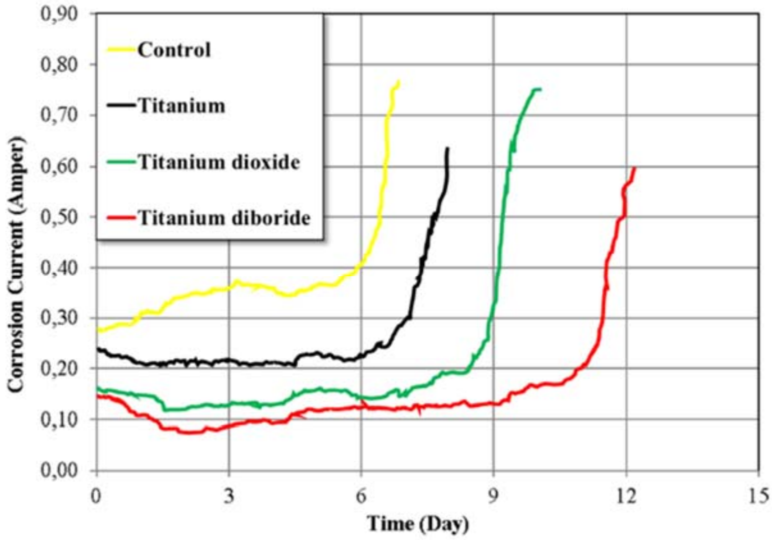

Figure (11): Variation of time-dependent corrosion current according to coating

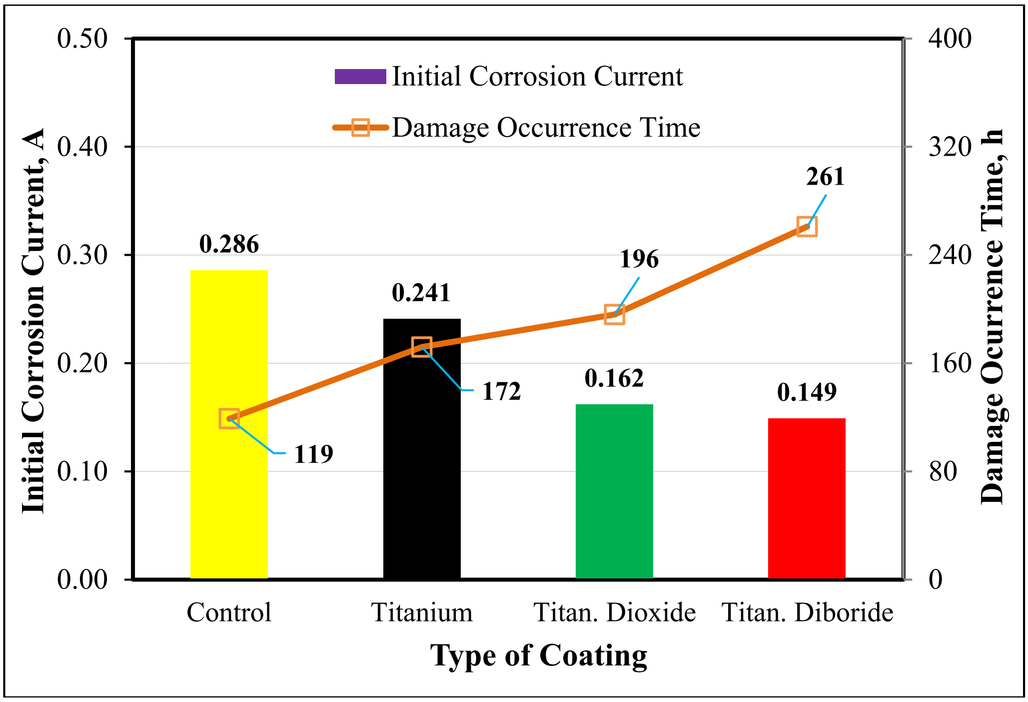

Figure (12): Initial corrosion currents and damage occurrence times of samples

CONCLUSION

TVA is an innovative system that is used for producing thin films. With this technique, we tried to protect reinforced concrete that can be damaged due to the loss of alkaline passivation and corrosion. Reinforcement corrosion, which progresses rapidly with the effect of chlorine, especially in the regions near the sea, may be stopped with these coatings. Consequently, the purpose of this research is to prevent the loss of lives caused by the collapse of buildings (especially due to earthquakes). In this experimental study, the importance of rebar coatings against the damage caused by corrosion to concrete has been revealed. Methods developed for protection against corrosion are either ineffective or require high costs in terms of implementation. Some methods are difficult to implement in terms of integration into the construction field. The point that distinguishes this study from other methods is that the TVA method, which is used for different purposes, is used for the first time in the literature to cover the rebars used in reinforced concrete structures.

In this manuscript, the TVA technique was utilized for creating nano-sized thin films on rebar surfaces. Experiments showed that against the threat of an aggressive environment, titanium-based nano-coatings are increasing the corrosion resistance of structural steels. Thin films that formed on rebar surfaces have protected the reinforcements, suspending the occurrence of corrosion reactions. As a result of this study, the best result was observed for TiB_2-coated sample with lower corrosion current and longer damage occurrence time.

The most effective way to avoid the destructive effects of corrosion is to avoid the occurrence of corrosion reactions from the very beginning. The cost of preventing corrosion at the beginning of the construction process is much less compared to the costs after corrosion occurs. In addition, the loss of lives and property that may be caused by corrosion should be kept in mind. By using this system, reinforcements can be produced as resistant to corrosion and there is no need for additional methods and costs to protect the structure from the harmful effects of corrosion. It is thought that this study will shed light on future applications.

Declaration of Competing Interest

On behalf of all authors, the corresponding author states that there is no conflict of interest regarding this research.

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