



Y₂O₃-Doped BioGlass Coatings for Ti-6Al-4V Alloy: Insights into Degradation, Bioactivity, Ion Release, Adhesion, and Corrosion Behavior

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The Ti-6Al-4V alloy is particularly favored for orthopedic applications. However, important issues such as corrosion resistance, biocompatibility, and ion release need to be addressed. Therefore, this study aims to enhance the corrosion resistance and biocompatibility and reduce the ion release of the Ti-6Al-4V alloy by applying Y₂O₃-doped bioglass bioceramic coatings. The coatings were synthesized using the sol-gel method, including pure BioGlass and BioGlass with 1, 5, and 10 wt pct Y₂O₃. These coatings were then evaluated for their corrosion resistance, adhesion strength, degradation behavior, bioactivity, and ion release. The results showed that all coatings exhibited homogeneous, crack-free, and porous morphologies, which are favorable for osteointegration. The addition of 10 wt pct Y₂O₃ to the BioGlass coating resulted in a 52 pct increment in adhesion strength, which increased from 13.72 ± 2.80 MPa for single BioGlass coatings to 20.81 ± 2.53 MPa. All samples showed degradation values lower than 1 wt pct. Single BioGlass coatings significantly enhanced the bioactivity of Ti-6Al-4V alloy. However, the addition of Y₂O₃ negatively impacted BioGlass bioactivity, leading to a noticeable decrease as the Y₂O₃ content increased. Conversely, the presence of Y₂O₃ negatively impacted bioactivity and corrosion resistance while increasing the release of Ti ions. Specifically, the release of Ti ions increased from 79.5 ± 0.8 ppb in uncoated samples to 253.7 ± 15.3 ppb in BG-coated samples. For 1, 5, and 10 pct Y₂O₃-added samples, Ti releases were measured as 192.2 ± 33.6 ppb, 168.5 ± 62.4 ppb, and 194.3 ± 54.4 ppb, respectively. This research highlights the potential of tailored BioGlass coatings to improve the performance of Ti-6Al-4V alloys in orthopedic implant applications, particularly for enhancing bone integration and reducing implant failure.

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I. INTRODUCTION

METALLIC implants are commonly preferred in surgical operations, especially in hard tissue applications. There are well-known issues with metallic implants, such as low biocompatibility, inadequate corrosion resistance, mechanical incompatibility, high density, and the potential of ion release known for their

toxicity causing allergic tissue reactions.^[1] Titanium alloys have been shown to exhibit certain advantages over stainless steel and Co-Cr alloys, due to their low density, high strength, corrosion resistance, and biocompatibility.^[2] These alloys can be classified into three distinct categories based on their phase structure: α , $\alpha + \beta$, and β . Ti-6Al-4V, an $\alpha + \beta$ alloy, is a commonly used commercial biomaterial, but α and $\alpha + \beta$ alloys present certain disadvantages.^[3] These include a high elastic modulus compared to bone and potential toxicity, potential toxicity, tissue discoloration, inflammation, and infection depending on the alloying elements (e.g., Al and V).^[4,5] In contrast, β -type alloys are considered as alternatives for biological implants due to their non-toxic, non-allergenic properties, high strength, and bone-like elasticity.^[6–8] Despite the inherent properties of Ti alloys that render them more suitable for biomedical applications, they are bioinert materials. Therefore, various surface modifications are performed to improve bioactivity and controlled metal ion release, especially in the case of Ti-6Al-4V alloys,

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which facilitates bonding formation with the host tissue upon implantation.^[5,9] These modifications are commonly conducted using hydroxyapatite-type bioactive ceramics.^[10] The improvement of coating properties such as adhesion and corrosion resistance can be achieved by adding various elements into matrices of bioceramics.^[11] Following implantation, the mechanical properties, interaction with surrounding tissue, surface properties, and mechanical properties of the implant are all directly related.^[12] Modifying the surface of implants with bioactive ceramics such as hydroxyapatite and bioglass can provide the benefits of using high-strength alloys in implants, including improved biocompatibility and corrosion resistance.^[13,14] Consequently, the desired properties, such as adhesion, of the implant material in conjunction with the tissue with minor sacrifices or no sacrifices from corrosion resistance can be achieved.

Hydroxyapatite (HA) and bioglass (BG) are the most frequently used bioceramics to modify the implant materials' surfaces.^[15,16] HA is commonly used as a coating material due to its excellent biocompatibility and bioactivity, and its effect on bone formation is well-known.^[17–19] However, various biocompatible silica and phosphate-containing bioactive ceramics have also been developed, which can form strong bonds with surrounding tissues. The implant surface can be bonded directly to living tissues by forming a hydroxy carbonate apatite (HCA) layer with bioactive ceramics.^[20] It is well established that bioactive glasses demonstrate excellent biocompatibility and the ability to bond with bone tissue, enhancing osseointegration and expediting the healing process.^[21] Even though bioglass has potential as an alternative to hydroxyapatite, its limited adhesion strength is a significant problem that must be addressed in bioglass-based bioceramic coatings, as reported in limited studies.^[22–25] Various additives have been studied to improve the corrosion resistance, bioactivity and adhesion strength of bioceramic materials in the body and successful results have been reported.^[26–32] In these additives, yttrium and zirconium are reported as a few of the most prominent additives.^[33–35] Specifically, it has been reported that yttria (Y_2O_3) enhances the mechanical properties and corrosion resistance of bioceramic coatings while improving their biocompatibility.^[35]

The presented study aimed to improve the adhesion strength of BG-based bioceramic coatings by adding Y_2O_3 , which is not often addressed in the existing literature. While numerous studies have examined hydroxyapatite-based coatings for enhancing metallic implant performance, limited research exists on bioglass surface modifications, despite their demonstrated potential as alternatives; These limited studies suggest that bioglass coatings can improve corrosion resistance and biocompatibility, but also highlight concerns regarding inadequate adhesion strength.^[36–38] The research focused on how different amounts of Y_2O_3 added to BG-based coatings on Ti-6Al-4V substrates affected corrosion resistance, degradation, ion release, and bioactivity. Undoubtedly, the reported findings will provide valuable information for the studies focusing on developing advanced bioceramic coatings,

particularly aiming for improved performance and biointegration for long-term clinical applications.

II. EXPERIMENTAL PROCEDURE

A. Before the Coating Process

The Ti-6Al-4V alloy, composed of 90 wt pct titanium (Ti), 6 wt pct aluminum (Al), and 4 wt pct vanadium (V), in dimensions of 10×15 mm, was preferred as the substrate. Before the coating process, the metallic samples were cut to in the size of 10×15 mm and sandblasted with SiO_2 particles to increase the surface area. To remove impurities from the surface, a series of ultrasonic cleaning treatments (DW, Acetone and Ethanol) were applied, and then the samples were immersed in 25 pct HNO_3 for 1 hour.

B. Coating Process

Four different bioceramic coatings, namely Single BG, BG/ $1Y_2O_3$, BG/ $5Y_2O_3$ and BG/ $10Y_2O_3$, were coated on Ti-6Al-4V surfaces using the sol-gel process. The coating contents and sample groups are given in Table I. To prepare BG-based sols, 45S5 bioglass powders were used consisting of 45 wt pct SiO_2 , 24.5 wt pct NaO_2 , 24.5 wt pct CaO and 6 wt pct P_2O_5 , with an average particle size of $36.33 \mu m$ (as shown in Figure 1(a)). During the preparation of sols, P_2O_5 , KH_2PO_4 and $NaCO_3$ were added to enhance gelation and sintering efficiency. Concentrations of these additives were optimized through a series of preliminary tests to achieve the best performance. To prepare Y_2O_3 -doped BG coating groups, Y_2O_3 powders with an average particle size of $6.81 \mu m$ were added at three different rates (Figure 1(b)). Following the coating of Ti-6Al-4V alloys' surfaces *via* the dip-coating method, samples were allowed to dry at room temperature for 24 hours and then subjected to a pre-drying treatment at $120^\circ C$ for 180 minutes. Subsequently, all samples underwent a sintering treatment at $550^\circ C$ for 120 minutes. The sintering temperature and duration were determined through a series of optimization studies.^[38] Higher sintering temperatures (700 – $800^\circ C$) resulted in the detachment of the coating from the surface, while lower temperatures ($<550^\circ C$) led to insufficient adhesion.

C. Characterization Analysis

SEM/EDS and XRD techniques were utilized to obtain a detailed examination of the morphological features and phase compositions of the coated surfaces. Before SEM and EDS analyses, the samples were ultrasonically cleaned in distilled water for 5 minutes and then dried at $80^\circ C$ for 2 hours. To increase the surface conductivity of coated samples, they were coated with a thin layer of Au before imaging and EDS analysis. SEM analyses were conducted using a HITACHI SU3500 scanning electron microscope and EDS analyses were performed using an Oxford INCA X-ray

spectrometer at 15 kV accelerating voltage and 10 mm working distance. XRD analyses were carried out using a Rigaku Miniflex 600 X-ray diffractometer. The XRD measurements were taken in the 20-90° range with a step size of 0.02° and a scan rate of 2°·min⁻¹.

D. Degradation/Ion Release/Bioactivity Analysis

To assess the interaction of uncoated and bio-ceramic-coated samples with simulated body fluid (SBF), all samples were immersed in SBF for 4 weeks. The chemical composition of SBF is shown in Table II. The total degradation amount of each sample was determined by measuring the weight difference before and after immersion. After 4 weeks of immersion, the released Ti, V, Al, and Y ions in SBF solutions were detected using ICP-MS analysis (NexION® 2000 C PerkinElmer® Inc.). The pH value of SBF was determined to be 7.4, and the analyses were conducted at a fixed temperature of 37 °C in the ETUV. Following degradation, Ca-P nucleations were observed on the sample surfaces EDS through analysis, and the bioactivity properties of the samples were evaluated. Degradation, ion release, and bioactivity tests were performed three times, and the average values were used.

Table I. Sample Groups

Sample Code	Coating
R	uncoated
S ₁	BG single coated
S ₂	BG/1 wt pct Y ₂ O ₃ coated
S ₃	BG/5 wt pct Y ₂ O ₃ coated
S ₄	BG/10 wt pct Y ₂ O ₃ coated

E. Adhesion Analyses

Adhesion strengths of bioceramic coatings were determined by pull-out tensile test method in accordance with ISO 13779-4 standard.^[40] DP420 (3MTM Scotch-WeldTM) epoxy was used as an adhesive and 7 mm diameter Ti rods were glued to the coated surfaces with the help of the adhesive and pull-out tests were applied at 1 mm·min⁻¹ deformation rates with Shimadzu AG-X 100 kN tensile testing machine. Adhesion tests were repeated five times, then the standard deviation values were calculated, and the average values were taken into account.

F. Electrochemical Corrosion Measurements

Electrochemical corrosion measurements were conducted in 3.5 wt pct NaCl solutions using a typical three-electrode electrochemical cell. Tafel corrosion tests were carried out with Biologic SP240 potentiostat at room temperature, and coated samples served as a working electrode (WE). Ag/AgCl (3.5 M KCl) electrode was used as the reference electrode. 25 cm² ruthenium-/iridium-coated Ti was used as an inert counter electrode. Before starting each corrosion test,

Table II. The Chemical Composition of SBF Solution^[39]

Components	Amount (g L ⁻¹)
NaCl	8.035
NaHCO ₃	0.355
KCl	0.225
K ₂ HPO ₄ ·3H ₂ O	0.231
MgCl ₂ ·6H ₂ O	0.311
c(HCl) = 1 mol/l	39 ml
CaCl ₂	0.292
Na ₂ SO ₄	0.072
TRIS	6.118
c(HCl) = 1 mol/l	0–5 ml

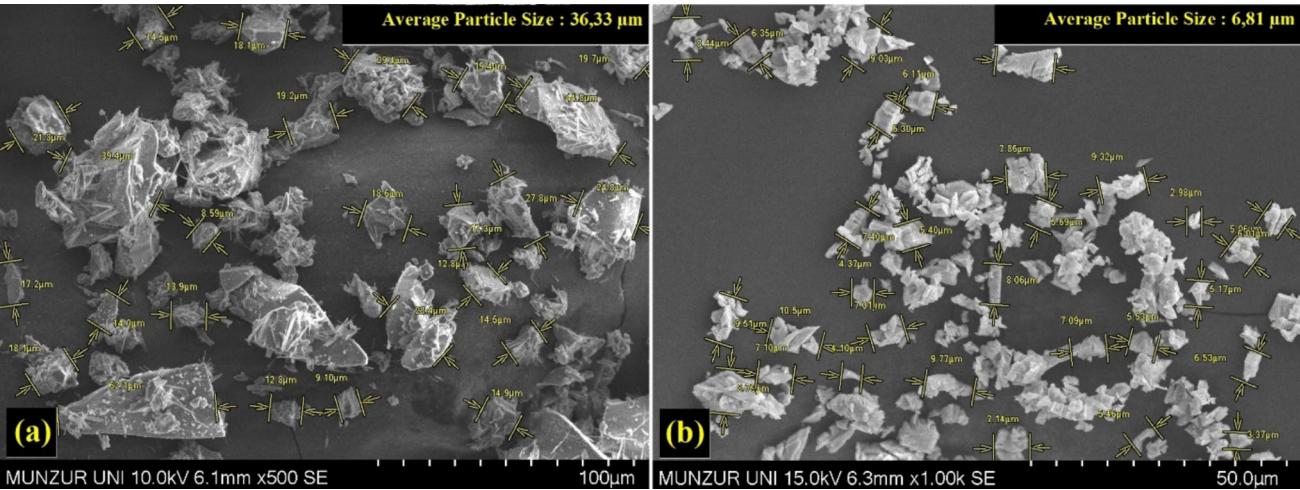


Fig. 1—SEM/EDS images of BG (a) and Y₂O₃ powders (b).

stable open-circuit potential (OCP) was measured, and then a potentiodynamic voltammetric Tafel scan at 0.166 mV s^{-1} rate was applied for $\pm 250 \text{ mV}$ of OCP. Tafel extrapolation method is applied for the calculation of Tafel elements. In short, the I_{corr} value is calculated directly from the Tafel chart. It is measured at the point where the slopes of the upper anodic curve and the lower cathodic curve intersect on the x -axis. The intersect point on the y -axis provides the E_{corr} value.

III. RESULT AND DISCUSSION

A. Microstructural and Morphological Characterization

SEM images and EDS analyses of uncoated Ti-6Al-4V, single BG and 1, 5, 10 wt pct Y_2O_3 -doped BG coatings before degradation are given in Figures 2(a)–(e), respectively. Figure 2(a) shows the aimed porosity created on uncoated Ti-6Al-4V surfaces by sandblasting with SiO_2 particles to increase the surface area and improve the coating efficiency. In the EDS analysis, the presence of Ti, V, and Al as the inherent constituents of the Ti-6Al-4V alloy along with the expected Si peak, was recorded on the surface. In addition, oxygen was observed, emerging from the expected oxide layer formed on the surface due to the effects of sandblasting and pre-surface treatments.

In the SEM-EDS analyses of the single BG coated samples, a crack-free, and porous coating was observed on the surfaces. The EDS analysis recorded the spectra of Na, Si, Ca, and O elements from the bioglass structure, as well as Ti, Al, and V spectra from the substrate material. When comparing the SEM images of 1 wt pct Y_2O_3 -doped BG to the single BG-coated sample, clear morphological changes were observed, but the yttria addition was not detected in the EDS analysis. Figure 2(d) shows the 5 wt pct Y_2O_3 -doped BG coating, where no significant morphological difference was detected, but capillary cracks were observed on the surfaces of larger grains. The EDS spectrum revealed an emergence of a Y peak with the amount of 5.41 wt pct. As for the 10 wt pct Y_2O_3 -doped BG coating, it showed a homogeneous and relatively crack-free surface morphology and an increase in the amount of Y was detected as expected. This is attributed to the improved network structure within the bioglass matrix with 10 wt pct Y_2O_3 doping, contributing to the stability of the coating and helping to distribute thermal stress and absorb energy more evenly throughout the more homogenous structure.^[41,42] The coatings retained Ti, V, and Al from the Ti-6Al-4V substrate material. In conclusion, all coatings achieved homogeneous, relatively crack-free, and porous surface morphologies, and the addition of Y_2O_3 did not cause significant morphological differences. In addition, SEM cross-sectional imaging was employed to determine the coating thickness (Figure 3). The coating thicknesses were measured to be in the range of 20–25 μm , with values of $21.4 \pm 3.2 \mu\text{m}$ and $25.4 \pm 1.7 \mu\text{m}$ for the single BG and BG/10 wt pct Y_2O_3 coatings, respectively.

B. In-Vitro Bioactivity

SEM images and EDS analyses of uncoated Ti-6Al-4V, single BG and 1, 5, 10 wt pct Y_2O_3 -doped BG coatings after biodegradation are given in Figures 4(a)–(e), respectively. When the SEM images in Figure 4(a) are examined, it is observed that there is no significant difference between the surface morphology before and after degradation. When the EDS analysis results before and after degradation states are compared, it is striking that very small amounts of Ca, P, Na and Cl are formed on the Ti-6Al-4V surface after degradation. SEM-EDS analysis results of the surface after degradation are given in Figure 4(b) for only BG coatings. When the surface morphologies after degradation are examined, approximately 0.4 μm sized spherical-shaped nanoparticles were found in the BG coatings. When the EDS analysis results were examined, it is seen that the Ca amount has increased by more than twofold after degradation. This situation can be explained by the formation of Ca-P nanostructures on the BG coating. Figures 4(c)–(e) shows the SEM and EDS analyses of the 1, 5, and 10 wt pct Y_2O_3 -doped BG coatings, respectively. All Y_2O_3 -reinforced samples exhibit similar surface morphologies to the single BG coating. Additionally, the EDS spectra of BG coatings doped with 1, 5, and 10 wt pct Y_2O_3 show that the Ca/P ratios before degradation were 0.52, 0.44, 0.54, and 0.63, and after degradation, the Ca/P ratios were calculated as 1.05, 0.93, 0.85, and 0.84, respectively. These results indicate that the apatite phase may be present in the bio-glass ceramics, and it was observed that the Ca-P formation increased slightly, especially after degradation. In all coatings, the percentages of the Ti, Al, and V elements emerging from the substrate material were found to decrease after degradation.

To further explain the bioactivity that occurs when Ca-P based apatite forms after degradation, EDS-Mapping analyses of uncoated Ti-6Al-4V, single BG, and 10 wt pct Y_2O_3 -doped BG coatings were conducted, and results are presented in Figures 5(a)–(c). On the surfaces of uncoated Ti-6Al-4V, calcium phosphate formation was detected at levels of 0.7 wt pct Ca and 0.3 wt pct P. In addition, the percentages of Ti, Al and V also showed a slight decrease after degradation. It is believed that this is raised because of low levels of bioactivity. A slight increase in the amount of O was also detected after degradation. This can be explained by the presence of the naturally formed passive TiO_2 oxide layer on the Ti-6Al-4V alloy surface, which facilitates the nucleation and deposition of Ca, P, Na, and Cl-based structures during the 28-day immersion period in SBF. Figure 5(b) shows the EDS-Mapping analysis results for the single BG coating. Compared to before degradation state, there was a more than two-fold increase in calcium (11 wt pct Ca), a significant decrease in sodium (6.4 wt pct Na), and no significant difference in oxygen. This indicates that Ca-based apatite structures, which have formed on the bioactive glass, cover the surface of the Na-based bioactive glass coating, demonstrating significant bioactivity. Similarly, there is a decrease in Ti and V values. Based on these results, it can be concluded that

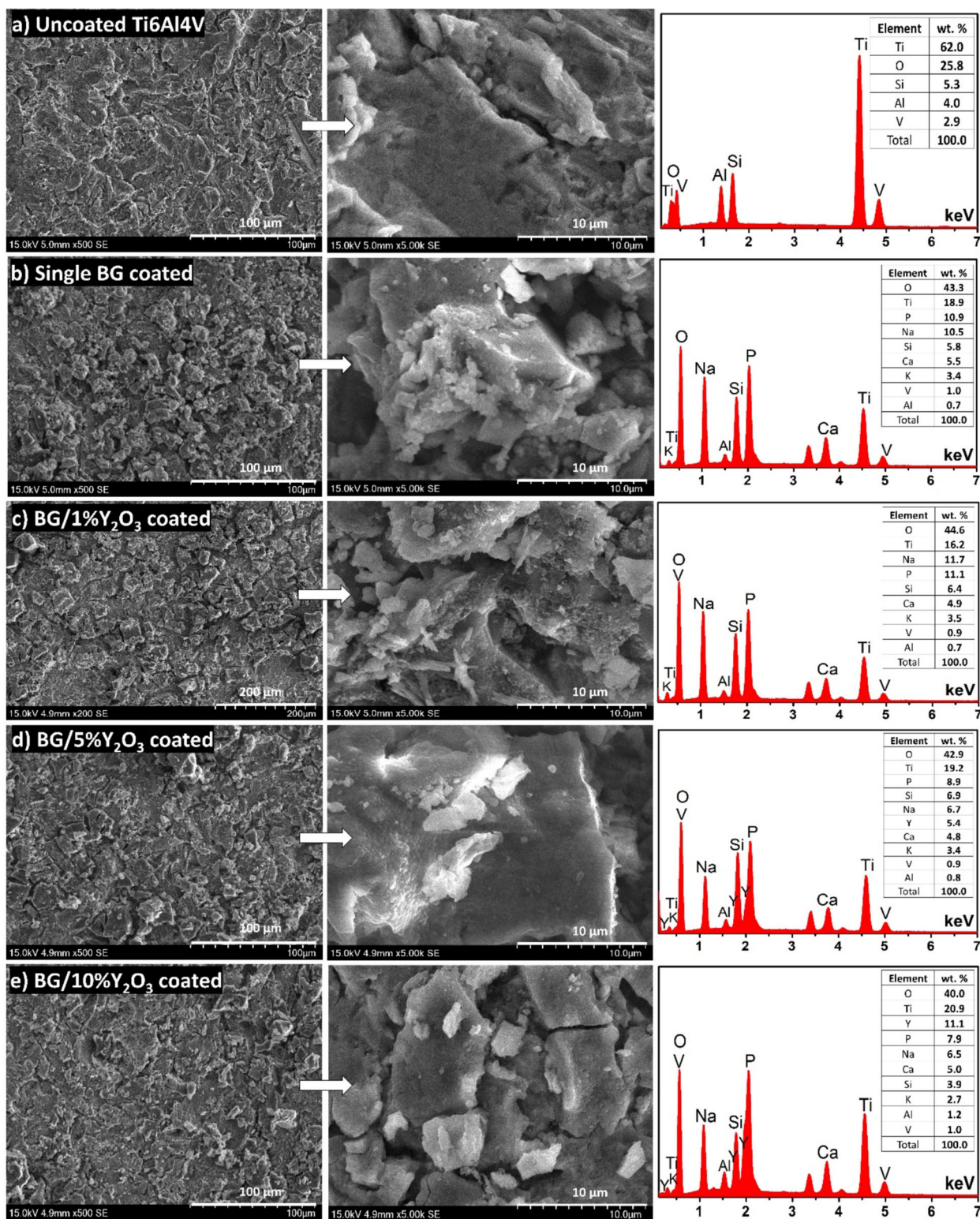


Fig. 2—SEM images and EDS spectra of surfaces before degradation.

there is a significant level of apatite bioactivity in the BG bioceramic coatings. Figure 5(c) displays the results of the EDS-Mapping analysis of the BG/10 wt pct Y₂O₃ bioceramic coatings after degradation, revealing an increase in both Na (9.5 wt pct) and Ca (8.7 wt pct).

However, upon evaluating all bioceramic coatings collectively, it was noticed that the magnitude of the increase in the Ca ratio lessened as the added Y₂O₃ ratio increased. Similarly, the decrease in the Na ratio lessened as the added Y₂O₃ ratio increased. These

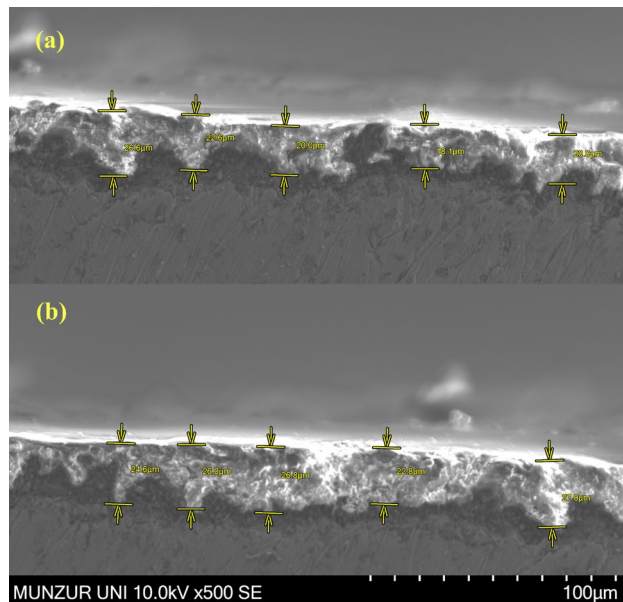


Fig. 3—Cross-sectional SEM images of (a) only BG and (b) BG/10 wt pct Y_2O_3 coatings.

results suggest that Y_2O_3 addition negatively affects the bioactivity of BG coatings causing a decrease with increasing addition ratio. The negative impact of Y_2O_3 on bioactivity may be attributed to factors related to its ionic radius and chemistry. Yttrium ions (Y^{3+}) have a larger ionic radius (212 pm) compared to other ions typically found in bioglass, such as calcium (Ca^{2+} , 99 pm) and phosphorus (P^{5+} , 98 pm). The difference in ionic radii can have a negative impact on the stability of the bioglass, potentially impeding the formation of apatite, which is a key indicator of bioactivity. Another important point is the changes in the O ratio. While a significant increase in the O ratio was observed in the uncoated sample, there was almost no change in all other bioceramic coatings. These results suggest that the amount of surface oxide on the uncoated Ti-6Al-4V surface increased during the 28-day incubation period in SBF. However, it can be concluded that the Ti-6Al-4V surface is protected by the physical barrier effect of the BG bioceramic coatings in SBF, and the O ratio did not change significantly.

The XRD patterns of the uncoated Ti-6Al-4V substrate, BG, and BG coatings doped with 1, 5, and 10 wt pct Y_2O_3 are presented in Figure 6. In the single BG coating, diffraction peaks corresponding to crystalline BG 45S5 ($\text{Na}_2\text{Ca}_2\text{Si}_3\text{O}_9$, JCPDS 22-1455), as well as characteristic α -Ti (JCPDS 44-1294) and β -Ti (JCPDS 44-1288) phases were detected. In addition to the formation of β -Ti is attributed to compositional redistribution during the heat treatment process.^[43,44] In the diffraction pattern of the BG/1 wt pct Y_2O_3 coating, Y_2O_3 is present, although not very prominently, and crystalline BG and Ti peaks are present. However, when the Y_2O_3 addition rate is increased to 5 and 10 wt pct, the presence of Y_2O_3 peaks in the coating becomes more noticeable. The intensity of XRD peaks decreased when the Y_2O_3 concentration reached 10 pct. This decrease is

attributed to the broadening or shortening of the peaks, which resulted from the higher Y doping concentration causing the coating structure to become more amorphous. The α -Ti peak is consistently observed in the XRD patterns across all coatings. This persistence is attributed to the high penetration depth of X-rays and the porous nature of the coatings, as confirmed by SEM analysis.^[45] It is important to note that the α -Ti peaks in the coated samples exhibit slight deviations in position when compared to the uncoated Ti-6Al-4V substrate. This shift may be partially explained by the crystallization behavior of the initially amorphous BG phase during sintering, which influences the diffraction signals and improves chemical bonding at the coating-substrate interface.^[38] Specifically, the peak observed at 40.48° for the uncoated Ti-6Al-4V alloy shifted to 40.34° , 40.29° , 40.35° and 40.37° for the single BG, and 1, 5, and 10 pct Y_2O_3 -reinforced BG coatings, respectively. Moreover, the full width at half maximum (FWHM) values for these peaks were recorded as 0.20, 0.32, 0.29, 0.30, and 0.32, respectively. The increase in FWHM values relative to the uncoated Ti-6Al-4V substrate suggests a possible cause for the observed peak shift, which is likely related to the changes in crystallinity. The diffraction patterns obtained from the uncoated Ti-6Al-4V alloy, BG, and Y_2O_3 /BG coatings are consistent with those reported in the literature.^[21,37,46,47]

The information in Table III shows the extent of degradation in samples after immersing them in SBF for four weeks. The uncoated sample did not experience any weight loss. However, when 1 wt pct Y_2O_3 was added, the degradation increased to 0.66 wt pct compared to the 0.34 wt pct weight loss in single BG coatings. Interestingly, adding 5 wt pct Y_2O_3 resulted in nearly the same degradation as single-BG coatings. These findings indicate that there is no clear relationship between the change in Y_2O_3 content and degradation. The larger ionic radius of Y is expected to negatively impact the stability of the bioglass, leading to more degradation with a higher concentration of Y_2O_3 . However, the results show that there is no definite correlation between the amount of Y_2O_3 and the severity of degradation. The low degradation amounts and high standard deviation values also suggest that a four-week immersion period is not sufficient to fully assess the impact of Y_2O_3 . Longer-term SBF tests are needed to gain a complete understanding of how Y_2O_3 affects the degradation mechanisms of bioglass coatings.

In metallic implants, corrosion can lead to prosthetic loosening and the release of metal ions, which poses health risks to humans. In the case of Ti-6Al-4V alloys, the release of Ti, Al, and V ions was observed in Hank's solution.^[48] After 28 days of immersion in SBF, the results of Al, Ti, V, and Y ion release for different sample groups are shown in Figure 7. Uncoated samples showed a small amount of Ti ion release at the ppb level, while bioceramic coatings significantly increased the Ti ion release. Interestingly, the addition of the Y slightly reduced the Ti release, but overall, BG-based bioceramic coatings increased the Ti ion release in Ti-6Al-4V alloy. The effect of Y_2O_3 addition on Y ion release was not linear. For example, 1 wt pct Y_2O_3 addition resulted in

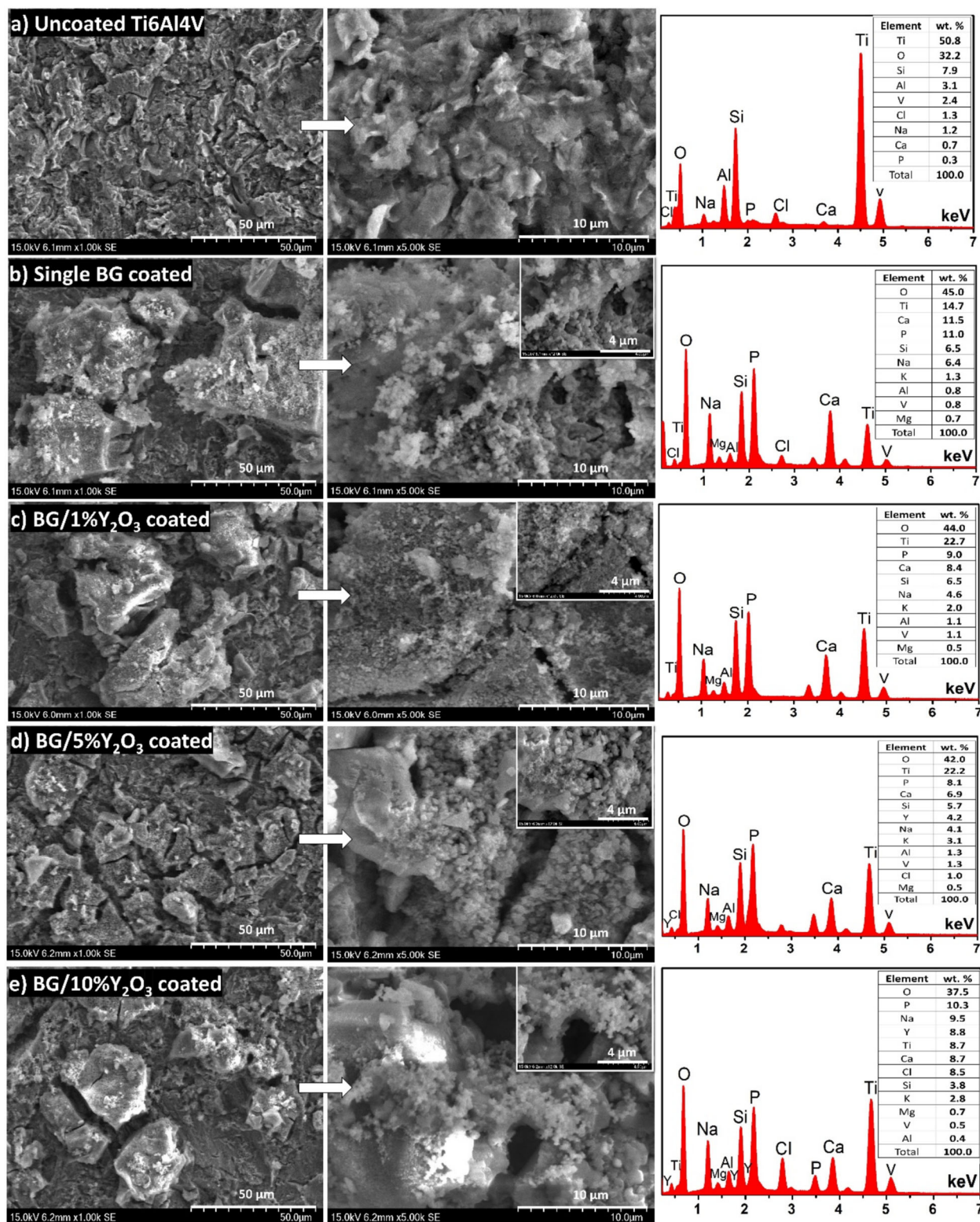


Fig. 4—SEM images and EDS spectra of surfaces after degradation.

305.9 ppb of Y ion release, but increasing it to 5 wt pct raised the release amount to 1870.3 ppb. However, 10 wt pct Y₂O₃ addition resulted in lower Y ion release compared to the 5 wt pct addition. Other studies have found that bioceramic coatings prevent ion release by

acting as a physical barrier.^[49–51] However, this study showed the opposite. The stagnant regions of SBF due to the porous structure of the bioceramic coatings caused a slight increase in ion release by increasing the corrosive effect.

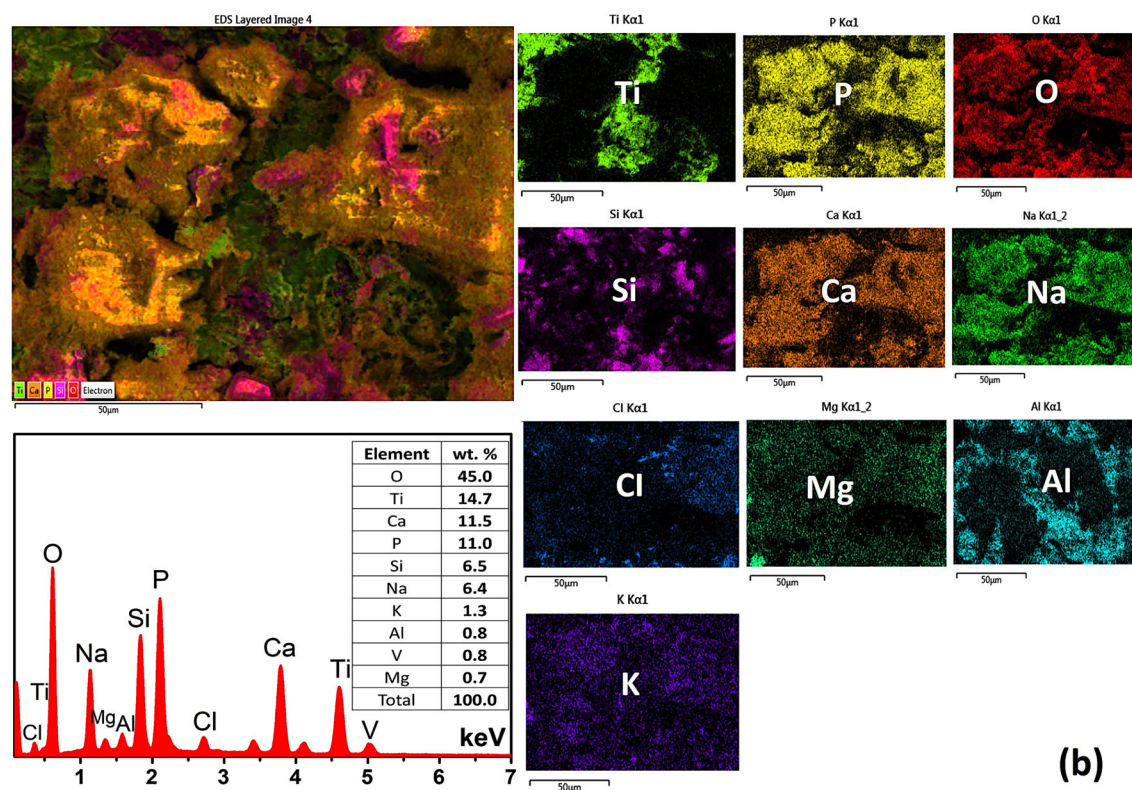
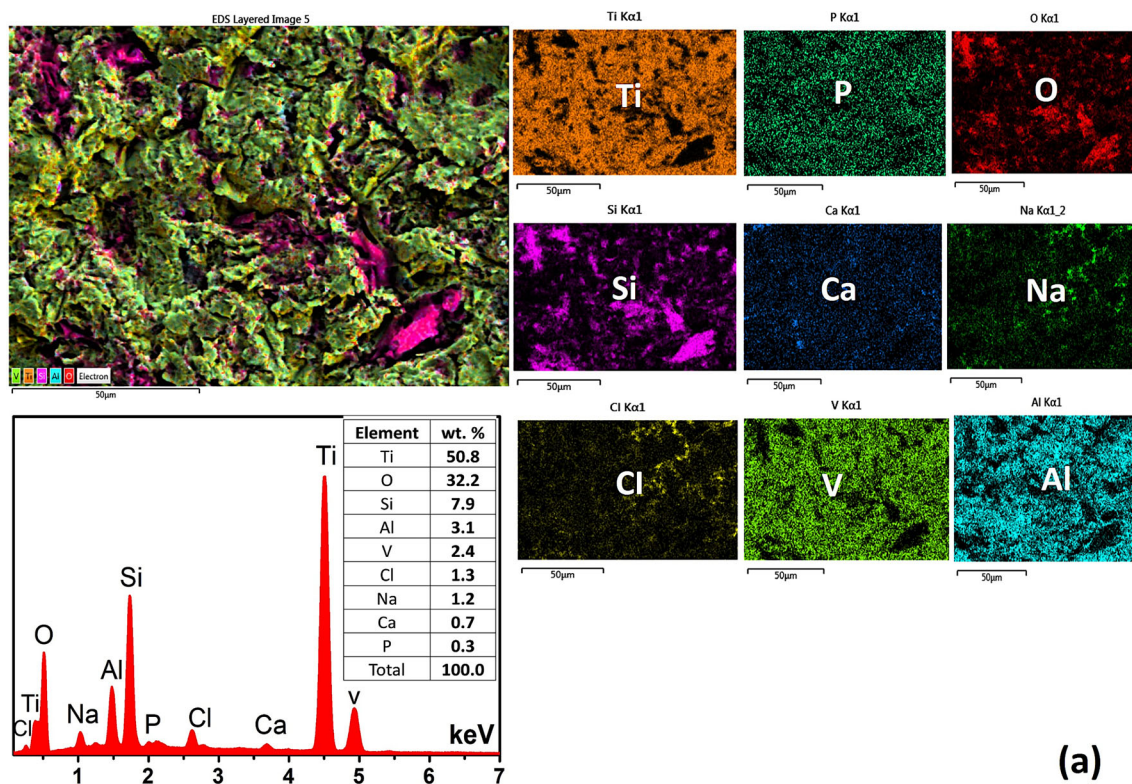


Fig. 5—EDS spectra and elemental mapping views of surfaces after incubation in SBF for 28 days; uncoated (a), single BG coated (b), and BG/10 wt pct Y_2O_3 coated (c).

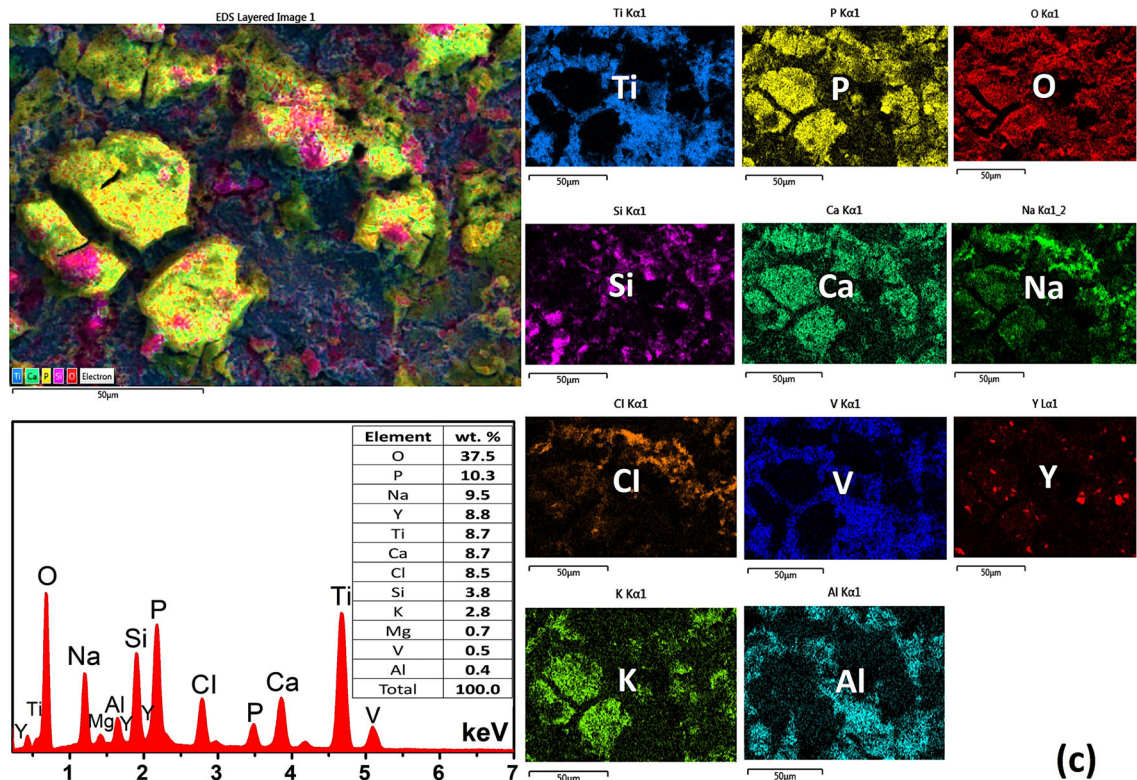


Fig. 5—continued.

C. Adhesion Strength

Ensuring sufficient adhesion strength is one of the critical parameter in modifying bio-ceramic surfaces.^[7,52,53] Insufficient adhesion strength remains a significant challenge in bioglass coatings as noted in the literature.^[25,54,55] Previous studies compared HA and BG-based bio-ceramic coatings on NiTi alloy surfaces. HA-based coatings had an adhesion strength of 23 MPa, while BG-based coatings had 13 MPa.^[21] In a similar study where surfaces of REX-734 alloy were modified, the adhesion strength of coatings based on hydroxyapatite (HA) was measured as 27.04 MPa, while it was 13.45 MPa for coatings based on bioactive glass (BG).^[33] This indicates that BG surface modifications exhibit weaker adhesion strength compared to hydroxyapatite; therefore, further studies are required to improve this property. Figure 8 displays the adhesion strength (in MPa) and elongation at break (in pct) characteristics of BG-based coatings.

The findings exhibit significant variations in the adhesion strength values of the tested coatings. The adhesion strength of single BG coatings was measured as 13.72 MPa, and it was observed that 1 wt pct Y_2O_3 addition had no significant effect on the adhesion strength. However, when the addition amount was increased to 5 wt pct, the adhesion strength of BG increased by 27 pct and was measured as 17.42 MPa. The most prominent improvement in adhesion strength was achieved with a 10 wt pct Y_2O_3 addition, which increased the adhesion strength of BG by 52 pct, measured as 20.81 MPa.

D. Corrosion Resistance

Corrosion resistance is a crucial factor that affects the performance of metallic implants. In the literature, different corrosion-rendering solutions have been studied and ordered according to their forming ions or compounds. The list starts with the least complex solution, NaCl, followed by phosphate-buffered saline (PBS), Ringer's solution, simulated body fluid (SBF), minimum essential medium (MEM), and finally Fetal bovine serum (FBS) or bovine serum albumin (BSA). As we go further down the list, the complexity of the corrosion analysis increases.^[56] Depending on the resistance of the metal, metal alloy, or composite, a solution of less than 1 wt pct weight of sodium chloride (NaCl) or a solution of 3.5 wt pct weight of NaCl can be used.^[57] When dealing with moderately or slightly strong corrosion-resistant substrates, a 3.5 wt pct NaCl solution will be a suitable medium to obtain more understandable results in corrosion investigation.^[58] Uncoated and bioceramic coated specimens were tested for corrosion resistance in 3.5 wt pct NaCl solution Tafel and OCP analysis results are presented in Figures 9(a) and (b).

During the corrosion analysis, after reaching a stable OCP potential, a Tafel scan was initiated. Leakage free measurement of TAFEL is important because any possible leakage results in excessive data distortion, especially in anodic section, and therefore proper stabilization and equilibrium of surface reactions with minimal deviation in OCP is maintained *via* a a time-dependent potential rate limit which ensures rapid attainment of equilibrium (with a drift rate < 15 mV/h).

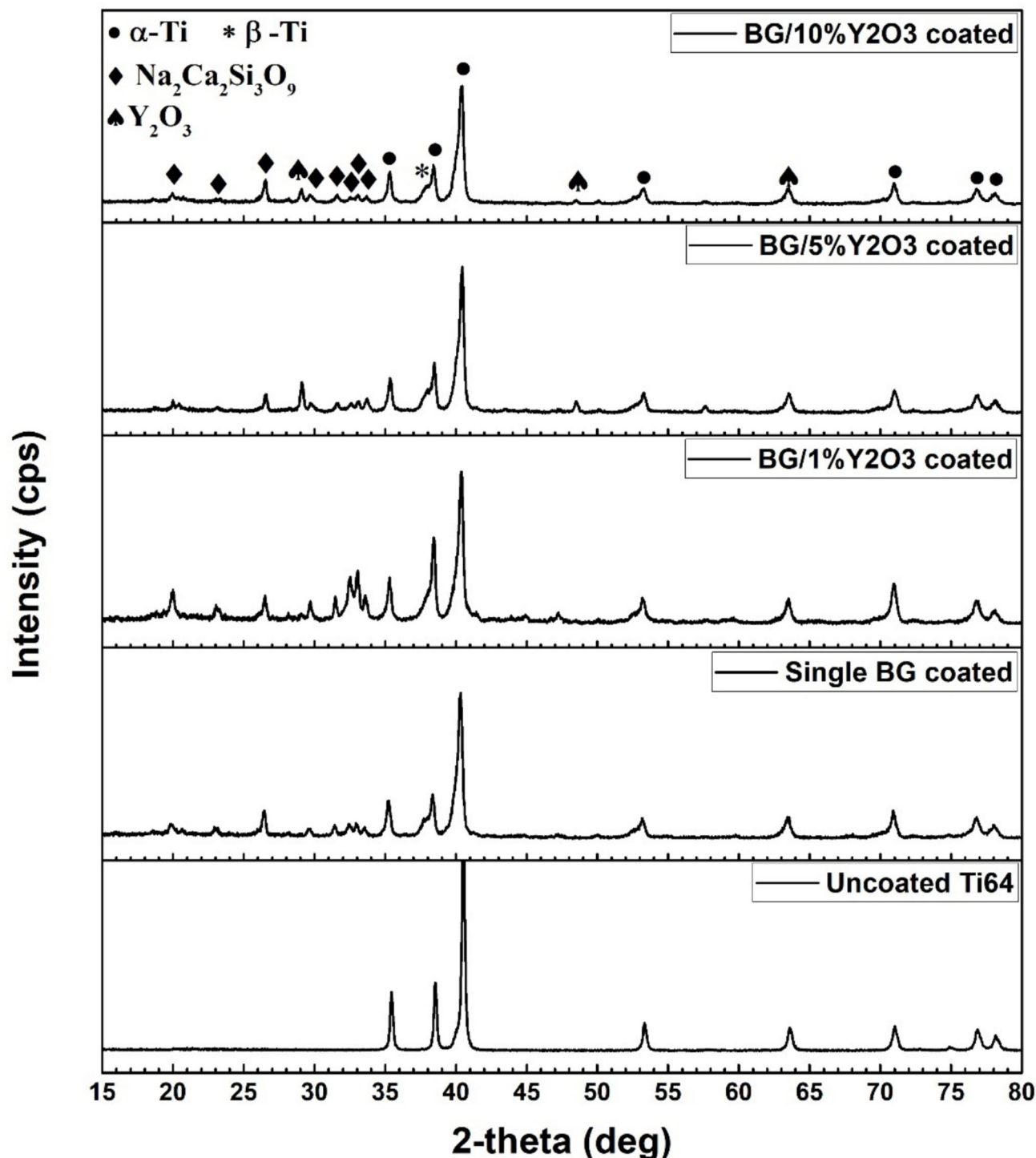


Fig. 6—XRD patterns of uncoated, and coated samples.

When condition is met, the OCP period terminates and the Tafel measurement starts. Initially, each sample was kept for 1000 seconds in the OCP period, then, as a function of time, the OCP potential change was monitored, and the OCP period was terminated when the potential deviation became less than 0.3 mV min^{-1} . The OCP behavior of each sample revealed different initial potentials (E_{initial}), which indicates the existence of

different surface compositions and behaviors as an expected outcome of successful surface modification.

Regarding uncoated Ti-6Al-4V substrates, the deviation in open circuit potential (E_{ocp}) was less than 0.3 mV min^{-1} at approximately 3500 seconds, with a measured stable potential value of 200 mV. For BG-coated Ti-6Al-4V substrates, the E_{ocp} deviation remained below 0.3 mV min^{-1} at around 2000 seconds,

with a measured final stable potential of 75 mV. For BG/5 wt pct Y₂O₃ substrate, the E_{ocp} deviation remained below 0.3 mV min⁻¹ at around 2000 seconds, with a stable potential value of 55 mV. Similarly, for the BG/10 wt pct Y₂O₃ substrate, the E_{ocp} deviation was less than 0.3 mV min⁻¹ around 1500 seconds, with a stable potential value of 75 mV. Only BG coated and 10 wt pct Y₂O₃/BG coated samples revealed less than 25 mV deviation from their E_{initial} to the final E_{ocp} values, while the rest exhibited significant deviation between their E_{initial} to the final E_{ocp} values. High deviation from initial potential to final open circuit potential is usually observed in unevenly deposited multiple-phase surface reactions. In other words, while

Table III. Results of Degradation Tests (as Percent Weight Loss)

Specimen	Degradation (Wt Pct)
Uncoated	0
BG Coated	0.34 ± 0.29
BG/1 Wt Pct Y ₂ O ₃ Coated	0.66 ± 0.30
BG/5 Wt Pct Y ₂ O ₃ Coated	0.33 ± 0.28
BG/10 Wt Pct Y ₂ O ₃ Coated	0.49 ± 0.50

BG and Y₂O₃ form their equilibrium potentials *via* the formation of passive layers, such as oxide, hydroxide, or chloride, available uncoated Ti sites also set their passivation reactions and, as a result of multiple passivations of equilibrium reactions, exceeding potential deviation through the OCP stage is expected to occur.

In the Tafel corrosion test, given in Figure 9(a), each substrate shows various degradations in NaCl solution. The 3.5 wt pct NaCl solution is mainly composed of Na⁺, Cl⁻, H⁺ and OH⁻ ions. In tested samples, two main constituents, Ti-6Al-4V and BG, are expected to undergo degradation in corrosion solution upon applied potential. In the cathode region of the Tafel, at vertical lines to the current axis, the samples produce hydrogen evolution as shown in reaction (I), predominantly expected in the Ti-6Al-4V sample.



While potential is scanning through the E_{corr}, the slope region, oxygen consumption reaction (II) is expected to initiate.^[59]

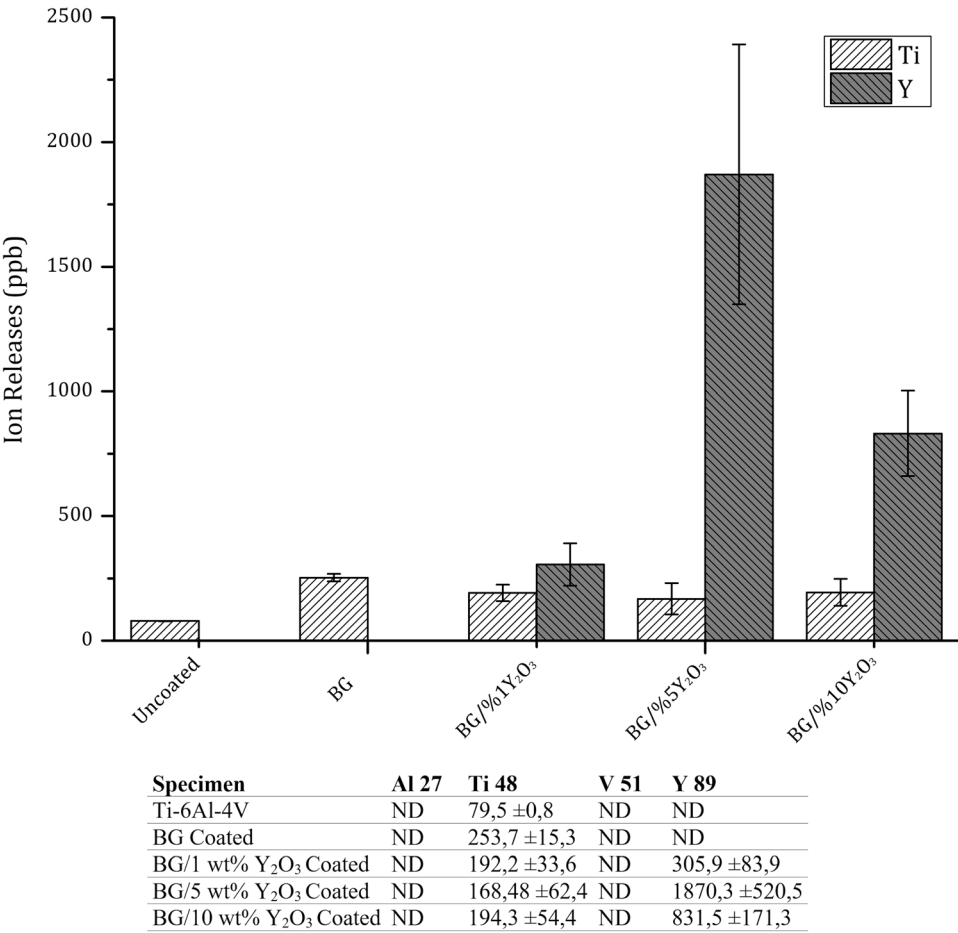


Fig. 7—Detected Ti, Al, V and Y Ion releases recorded by ICP-MS.

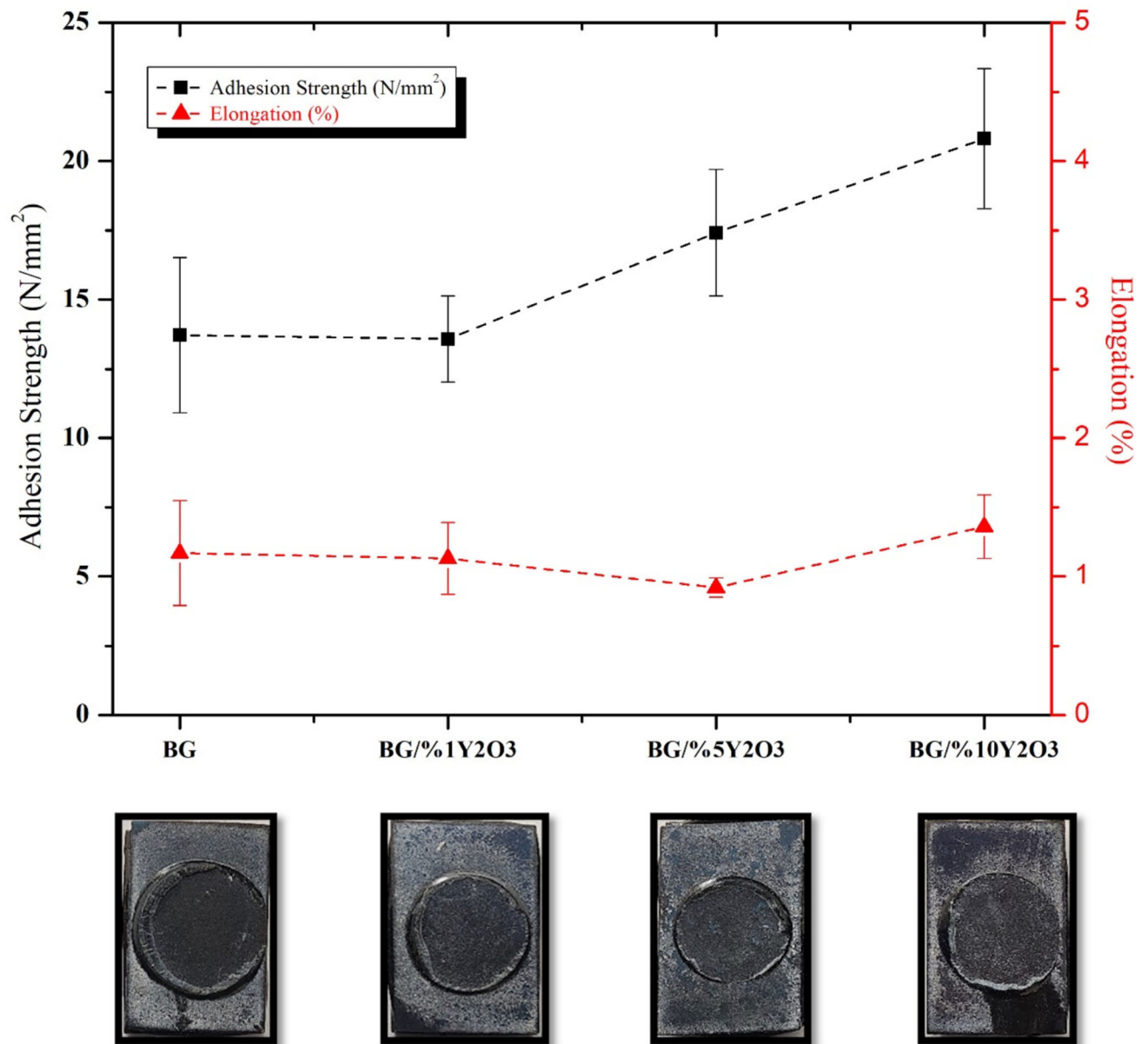
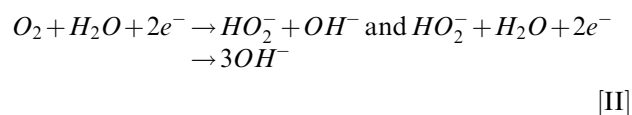


Fig. 8—Compression of Adhesion Strength and Elongation.



Al and Ti in Ti-6Al-4V alloy, oxides to Al^{3+} and Ti^{2+} upon immersion, as previously reported.^[58] Both metals tend to form protective and stable oxide films such as

$Al_2O_3 \cdot 3H_2O$ and TiO_2 ^[60–63] at anodic scan in Tafel. BG mainly comprises SiO_2 , CaO , Na_2O , and P_2O_5 , and it is already reported that SiO_2 dissolves in aqueous solutions containing less than 3.5 wt pct NaCl.^[64] Silica is generally stable, but it can dissolve to form silicic acid in certain conditions, with pH and temperature playing significant roles in the process. In a study, it is explained that the dissolution of silica tends to decrease in two cases. In the first case, if an aqueous medium contains a

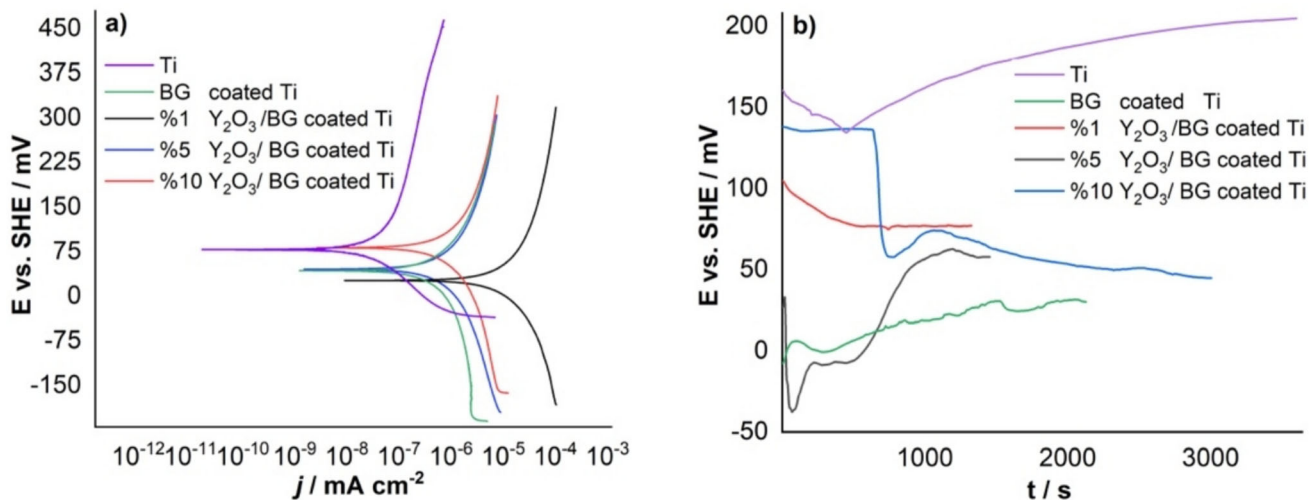


Fig. 9—Tafel curves are represented in a for Ti-6Al-4V, BG coated, and Y_2O_3 doped - BG coated Ti-6Al-4V substrates in (a), OCP curves obtained by set condition of < 15 mV/h of samples before Tafel are presented in (b).

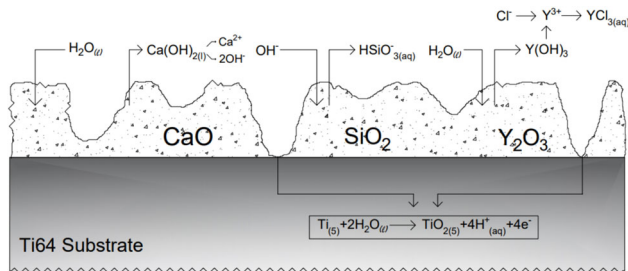


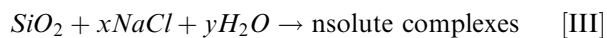
Fig. 10—Schematic representation of the corrosion mechanism in bioceramic-coated Ti-6Al-4V.

high number of monovalent ions, such as K^+ and Na^+ , the hydration number of cations increases, and the solubility of silica decreases. In the second case, if an aqueous medium comprises a high concentration of divalent cations, such as Ca^{2+} , a decrease in the activity coefficient of water occurs, resulting in a decrease in silica solubility. The performed study shows that the silica tends to dissolve more in saline media than the multi-monovalent/divalent ions-containing solutions, such as PBS, SBF, FBS, or BSA. Other studies report the high tendency of silica dissolution/complexing in NaCl at high pressure and temperature conditions and show SiO_2 dissolution as a function of temperature in Na^+ , including aqueous solutions with the rate reaction equation given in Eq. [1] (Figure 10).^[65]

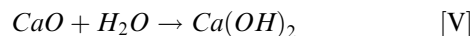
$$\text{rate reaction} = \left(k_1 e^{\left(-\frac{E_{a1}}{RT} \right)} + k_2 [Na^+] [OH^-] e^{\left(-\frac{E_{a2}}{RT} \right)} \right)^{1/2} \quad [1]$$

It is known that activation energy of a reaction rate ($k = A \times e^{-E_a/RT}$) related to free Gibbs energy, $-\Delta G^\ddagger$, and the free Gibbs energy can be converted into applied overpotential as an electromotor driving

force; $-xnF(E - E^\ominus)$. In other words, the effect of dissolution by temperature increase can also be driven by applied overpotential, $E^\ominus$, in the electrochemical sense. Upon the applied overpotential, possible nsolute complexes of SiO_2 in BG can be envisaged to be accelerated as given in reaction (III).^[65] If higher pH conditions sustained SiO_2 can react with OH^- groups according to the reaction (IV) to form $HSiO_3^-$ ions, but at positive anodic overpotentials the attack of OH^- groups in the relatively conductive media of Na^+ and Cl^- are also drive this reaction. In high pH conditions, SiO_2 can react with OH^- groups according to the reaction (IV) to form $HSiO_3^-$ ions, but at positive anodic overpotentials, the attack of OH^- groups in the relatively conductive media of Na^+ and Cl^- likely induces this reaction.



Both CaO and Na_2O are expected to react in aqueous media and producing hydroxide products by given reactions. Calcium hydroxide products are also tending to form calcium hydrogen phosphate products, given in reaction [V].



The coating also carries Y_2O_3 in the inner matrix and the electroactive surface. Y_2O_3 may undergo hydrolysis and then dissolve according to reactions (VI) and (VII) in the presence of water, especially under the influence of an applied overpotential in conductive media, resulting in the formation of yttrium hydroxide ($Y(OH)_3$). While $Y(OH)_3$ is relatively stable in neutral to basic pH conditions, which are typically found in a 3.5 pct NaCl solution, the presence of chloride ions and increased ionic strength can slightly increase the solubility of

$Y(OH)_3$ and form relatively soluble complexes like YCl_3 as presented in reaction (VIII).

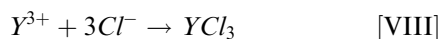
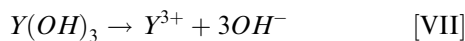
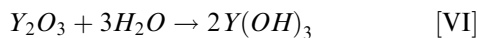


Table IV shows the calculated Tafel fitting elements of tested samples given with protection efficiency (P pct) in Figure 9(a). As expected, pure titanium exhibits the best corrosion resistance and reveals the slowest dissolution kinetic data, J_{corr} , and the thermodynamic data, E_{corr} .^[66] The corrosion resistance of titanium is followed by BG coating with a comparatively small J_{corr} value. Calculation of slopes is driven from Butler-Volmer equation, which defines the relationship between the applied overpotential and change in current density during both anodic and cathodic polarizations. According to the Butler-Volmer equation, slopes β_a and β_c are inversely proportional to the charge transfer coefficients α_a and α_c . Since the corrosion reactions occurring during anodic and cathodic scans involve multiple ions, various simultaneous reduction and oxidation reactions take place. Therefore, the sum of α_a and α_c should not be expected to equal '1', as would be the case for simple one-step, single-electron transfer reactions. When slopes β_a and β_c are evaluated for each sample, lower values of α_a and α_c indicate slower kinetics and thus correspond to higher slope values, and vice-versa.

In the J_{corr} calculation, I_{corr} values were divided by the exposed corrosion area of the samples. Before proceeding with further evaluation, it is important to verify the credibility of the I_{corr} values through linear polarization resistance (LPR) analysis. In the LPR analysis of each sample, known parameters as E_{corr} , I_{corr} , β_a , and β_c from Tafel experiment can be used to apply in Stern-Geary equation, presented in Equation [2].

$$I = I_{corr} \left[10^{\frac{E - E_{corr}}{\beta_a}} - 10^{-\frac{E - E_{corr}}{\beta_c}} \right] \quad [2]$$

Linear polarization typically involves a small potential perturbation around E_{corr} , usually ± 10 to ± 20 mV. This small range ensures the linearity and validity of the Stern-Geary approximation. As an example, the BG/1 wt pct Y_2O_3 sample is considered for evaluation. The related values for BG/1 wt pct Y_2O_3 are: $E_{corr} = -178$ mV, $I_{corr} = 3.220 \mu A$, $\beta_a = 493.6$ mV/decade, and $\beta_c = 298.9$ mV/decade. The calculated I values are presented in Table V.

The polarization resistance (R_p) can be calculated within a small potential range (e.g., ± 10 mV) around E_{corr} using Eq. [3]. Let us use the values from the ± 10 mV range in the Table V.

$$R_p = \frac{\Delta E}{\Delta I} \quad [3]$$

From Table V, at $E = -188$ mV (which is -10 mV from E_{corr}), $I = -0.406 \mu A$, and at $E = -168$ mV (which is $+10$ mV from E_{corr}), $I = 0.393 \mu A$. Therefore, ΔE is 20 mV $= 0.020$ V, and ΔI is $0.393 - (-0.406) = 0.799 \mu A = 0.799 \times 10^{-6}$ A. Substituting these values into Eq. [3], R_p is calculated to be approximately $25,032 \Omega$. The Stern-Geary constant B can then be calculated from Eq. [4] as approximately 80.83 mV.

$$B = \frac{\beta_a \times \beta_c}{2.303 \times (\beta_a + \beta_c)} \quad [4]$$

Finally, the I_{corr} calculated from the Stern-Geary approximation using Eq. [5] is approximately $3.23 \mu A$, which deviates by about 0.3 pct from the Tafel-calculated value of $3.23 \mu A$.

$$I_{corr} = \frac{B}{R_p} \quad [5]$$

The minimal deviation observed between the I_{corr} values from Tafel analysis and those from the Stern-Geary approximation ensures the validity of the calculated I_{corr} . When the same approximation is applied to all samples, the results are presented in Table VI.

The obtained I_{corr} values from the Tafel analysis and the Stern-Geary approximation are within an acceptable range. For uncoated Ti-6Al-4V, the calculated I_{corr} (8.83 nA) is quite close to the reported value (7.42 nA), with a small difference of 16 pct. This discrepancy may be due to measurement sensitivity at very low current values and is generally considered acceptable. Similarly, the calculated I_{corr} ($5.31 \mu A$) for the coated sample is close to the reported value ($4.87 \mu A$), showing a 9 pct difference. This small variation may arise from factors such as experimental measurement errors or surface roughness. Overall, the results are within an acceptable range.

When comparing Y_2O_3 -containing samples, kinetic data (J_{corr}) is considered a more reliable reference than thermodynamic data (E_{corr}), as the differences in E_{corr} values among the samples are not significant. Investigation of J_{corr} values revealed that the BG coating exhibited the highest corrosion resistance, although it was still inferior to the uncoated Ti-6Al-4V. Samples containing Y_2O_3 showed lower corrosion resistance with slight variations among them. While BG/1 wt pct Y_2O_3 and BG/10 wt pct Y_2O_3 exhibited similar corrosion resistance, BG/5 wt pct Y_2O_3 showed a slightly higher tendency toward corrosion in the corrosive medium. These slight differences may be attributed to the very low and potentially uneven deposition of yttrium oxides in the samples.

The overall corrosion resistance of the tested samples can be ranked as follows: uncoated Ti-6Al-4V > BG coating > BG/1 wt pct Y_2O_3 coating > BG/10 wt pct Y_2O_3 coating > BG/5 wt pct Y_2O_3 coating. For the calculation of corrosion rate, the ASTM G3 standard method was applied, as presented in Eq. [6].

Table IV. The Fitting Parameters Obtained from Tafel Curve Given in Fig. 9

Samples	E_{corr} vs SHE/mV	$j_{corr}/\text{nA cm}^{-2}$	β a/mV dec ⁻¹	β c/mV dec ⁻¹
Uncoated Ti-6Al-4V	73	7.42	417.1	125.2
BG	37	4.2	504	3×10^3
BG/ 1 Wt Pct Y ₂ O ₃	21	32.2	493.6	298.9
BG/ 5 Wt Pct Y ₂ O ₃	38	696	802.2	877.7
BG/ 10 Wt Pct Y ₂ O ₃	71	31.7	5×10^3	4×10^3

Table V. Calculated Values Using the Stern-Geary Approximation for the BG/1 Wt Pct Y₂O₃ Sample Are Presented

E/mV	$E - E_{corr} / \text{mV}$	$\frac{E-E_{corr}}{\beta_a}$	Anodic Part	$-\frac{E-E_{corr}}{\beta_c}$	Cathodic Part	$I/\mu\text{A}$
-198	-20	-0.04052	0.912	0.06691	1.166	-0.818
-193	-15	-0.03039	0.932	0.05018	1.123	-0.615
-188	-10	-0.02026	0.954	0.03346	1.080	-0.406
-183	-5	-0.01013	0.977	0.01673	1.039	-0.200
-178	0	0	1.000	0	1.000	0.000
-173	5	0.01013	1.023	-0.01673	0.962	0.197
-168	10	0.02026	1.048	-0.03346	0.926	0.393
-163	15	0.03039	1.073	-0.05018	0.891	0.586
-158	20	0.04052	1.098	-0.06691	0.857	0.776

Table VI. Calculated I_{corr} Values Using the Stern-Geary Approximation for the BG/1 Wt Pct Y₂O₃ Sample Are Presented

Samples	E_{corr}/mV	I_{corr}/TAFEL	$R_p/\text{k}\Omega$	B/mV	I_{corr}/calc	Deviation/Pct
Uncoated Ti-6Al-4V	-126	7.42 nA	2695.42	23.8	8.83 nA	16.00
BG	-162	416.80 nA	509.68	210.9	413.8 nA	0.72
BG/1 Wt Pct Y ₂ O ₃	-178	3.220 μA	25.03	80.8	3.23 μA	0.30
BG/5 Wt Pct Y ₂ O ₃	-159	4.87 μA	39.06	207.3	5.31 μA	9.00
BG/10 Wt Pct Y ₂ O ₃	-123	3.17 μA	315.46	1000	3.17 μA	0.03

$$CR_{mpy} = \frac{0.129 \times i_{corr} \times E_w}{\rho} \quad [6]$$

In the equation, 0.129 is a constant for mils per year (mpy), E_w is the equivalent weight, and ρ is the density. The E_w value was taken as 11.69, the same as for Ti-6Al-4V, for all tested samples. However, the ρ value was taken as 4.43 for Ti-6Al-4V and 2.6 for BG-containing samples. While BG has a specific density value, the calculation of E_w is not possible because Bioglass is a glass-like ceramic material generally composed of oxides such as SiO₂, Na₂O, CaO, and P₂O₅. When this material corrodes, chemical processes such as glass dissolution, ion exchange with the environment, and hydroxylation occur, rather than reactions involving known electron transfer processes. The calculated corrosion rate (CR) values for all samples are presented in Table VII.

A small addition of Y₂O₃ did not result in a significant difference, as evidenced by the similar corrosion rate (CR) values of BG and BG/1 wt pct Y₂O₃. However, the addition of 5 and 10 wt pct Y₂O₃ increased the corrosion rates by approximately 12 and 8-fold, respectively. The observed decrease in corrosion

Table VII. Calculated CR Values for all Samples Are Presented

Samples	CR /mpy
Uncoated Ti-6Al-4V	2.531×10^{-3}
BG	0.242
BG/1 Wt Pct Y ₂ O ₃	0.240
BG/5 Wt Pct Y ₂ O ₃	2.821
BG/10 Wt Pct Y ₂ O ₃	1.842

resistance upon doping the highly corrosion-resistant Y₂O₃ into the BG matrix is intriguing. This may be due to the formation of internal holes and cracks in localized areas within the BG matrix caused by the doped Y₂O₃. Nevertheless, as the amount of doped Y₂O₃ increases to a certain level, it demonstrates better protective efficiency compared to samples with lower Y₂O₃ content. Although the doped Y₂O₃ may accelerate the corrosion of BG due to potential internal matrix crack formation, an increased amount of Y₂O₃ up to 5 wt pct on the outer surface may provide improved corrosion resistance compared to samples doped with 10 wt pct. The lack of a linear relationship between the 5 and 10 wt pct

samples may arise from the porous nature of the coatings. However, one clear outcome is that the addition of Y_2O_3 accelerates the corrosion rate.

IV. CONCLUSIONS

The study focused on enhancing Ti-6Al-4V alloy surfaces with Y_2O_3 -doped BioGlass-based bioceramic coatings to improve functionality. Four different coatings were applied, and bioactivity, degradation, ion release, corrosion, and adhesion tests were conducted to evaluate their properties. The results revealed that all coatings exhibited advantageous surface morphologies conducive to osteointegration, with crack-free and porous structures. The coatings exhibited thicknesses ranging from 20 to $25\ \mu\text{m}$, with specific values of $21.4 \pm 3.2\ \mu\text{m}$ for single BioGlass coatings and $25.4 \pm 1.7\ \mu\text{m}$ for the 10 wt pct Y_2O_3 -doped BioGlass coatings. Single BioGlass coatings notably enhanced bioactivity, while the addition of Y_2O_3 negatively impacted bioactivity as its content increased. Moreover, the coatings increased the release of Ti ions and slightly reduced corrosion resistance. Interestingly, Y_2O_3 significantly improved adhesion strength in the bioceramic coatings. The addition of 10 wt pct Y_2O_3 increased the adhesion strength of single bioglass coatings from $13.72 \pm 2.80\ \text{MPa}$ to $20.81 \pm 2.53\ \text{MPa}$. Without compromising the contribution of Y_2O_3 addition to the adhesion strength of BG coatings, the concentration and distribution of Y_2O_3 can be adjusted to mitigate its negative effects on corrosion and bioactivity. The findings suggest that the developed coated implant holds promise for potential consideration in the design of orthopedic implant devices.

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DATA AVAILABILITY

Data is provided within the manuscript.

COMPETING INTERESTS

The authors have no competing interests as defined by Nature Research, or other interests that might be perceived to influence the results and/or discussion reported in this paper.

ETHICAL APPROVAL

Performed research did not involve any living organism in any part, and therefore, any informed consent was not applied.

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