Micro/nanostructured bioactive titanium implant surface with sol-gel silicate glass nanoparticles

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ABSTRACT

Purpose: To develop a surface coating of sol-gel 70S30C bioactive glass (BAG) nanoparticles on titanium disks and dental implants and to characterize the results in terms of average surface roughness, adhesion strength, and coating stability upon implant insertion under clinical settings. Materials and Methods: BAG was prepared with the modified sol-gel technique, then milled into nanoparticles. The resultant powder was characterized in terms of phase structure, composition, and particle size. Titanium disks and dental implants were coated with BAG nanoparticles by...
electrophoretic deposition. Surface characterization of coated implants was conducted. Uncoated vs BAG-coated implants were examined for average surface roughness by a confocal laser scanning microscope. The pull-off test was conducted to measure the adhesion strength of BAG coating to the underlying disks. Coated implants were inserted under clinical settings into artificial and natural bone to measure the amount of coating loss and to evaluate the effect of insertion on coating thickness. **Results:** BAG nanoparticles had an amorphous structure, with a particle size of < 20 nm in diameter. Electrophoresis resulted in a continuous coating covering the whole implant surface. Microscopic analysis confirmed the porous nanostructure of BAG coating, which formed a homogenous surface with microcracks. BAG coating had a uniform thickness of 35.38 ± 4.67 μm. Average surface roughness was significantly lower for BAG-coated implants, with a reduction of surface irregularities (3.34 ± 0.45 μm for uncoated implants, 1.45 ± 0.23 μm for BAG-coated implants). Adhesion strength of 18.51 ± 3.37 MPa was recorded for BAG coating, and 66.23% ± 10.23% of the coating weight was found to remain on the implant surface after insertion into artificial bone. A reduction in BAG coating thickness occurred only in sites of high friction with bone after implant insertion into bovine bone. **Conclusion:** Coating titanium implants with 70S30C BAG nanoparticles is attainable through electrophoretic deposition and results in a homogenous coating layer with a moderately rough surface, considerable adhesion strength, and high coating stability observed during implant insertion. *Int J Prosthodont* 2022. doi: 10.11607/ijp.10272

**Keywords**

Bioactive glass, 70S30C, Titanium, Dental implants, Coating, Electrophoretic deposition.

**INTRODUCTION**

High success rates have been recorded for titanium dental implants owing to their corrosion resistance, mechanical strength, and biocompatibility, thus achieving direct contact with bone, a process known as osseointegration.¹, ² Osseointegration is mainly dependent on the interface between the dental implant and living bone, managed primarily by the surface oxide layer which also minimizes titanium ion release, inhibits corrosion, and passivates the metal.³, ⁴

However, bioinert titanium can hardly prevent infections or regulate bone cell processes.⁵ Moreover, negative influence on success of implants has been noticed in medically compromised and aged patients where the inflammatory phase gets prolonged with reduced new tissue formation during the regenerative phase.⁶ Diabetes mellitus, osteoporosis, using bisphosphonates, heavy smoking, and irradiation exposure before or after implant surgery
present a challenge to dental implant therapy by having the potential to disrupt tissue healing process and to poorly affect implant longevity.\(^7,8\)

Implant material properties, geometrical features, as well as surface topography affect osseointegration.\(^9\)

Surface topography, whether on the macro-, micro- and nanoscale, is mandatory for the initial stages of osseointegration, with the micro- and nano-geometry being recently the main point of research. Implant surface microtopography affects osseointegration on the cellular level, whereas implant-bone tissue interactions are greatly affected at both cellular and protein levels by the nano-topography.\(^8\) When implants contain nanostructures, specific protein interactions are promoted allowing for proliferation and adhesion of more bone cells and stronger integration of bone tissue.\(^8\) Rough surfaces help the survival of osteoblasts rather than fibroblasts, and induce better bone to implant contact. High anchorage to bone with implant long-term stability is guaranteed with rough surfaces more than smooth surfaces. Osteogenic cell differentiation is hindered in case of lack of surface roughness on the micro- and nanoscale.\(^4,10\) Therefore, titanium implant surface modifications have been focused on to increase their bioactivity, reduce the risk of infections, and enhance osseointegration after insertion.\(^5\)

Surface coating is a widely used approach to modify implant surface, resulting in wider range of surface properties and more improved biocompatibility and bioactivity.\(^11,12\) Coating degradation should be coordinated with bone regeneration to ensure bone maturation and interface stability. These materials must also shorten the duration between primary stability (achieved during surgery) and secondary stability (definitive bone integration).\(^13\) Due to the great importance and various advantages of coatings, they are developed to reduce the chance of dental implant failure. This is done by reversing the negative impact of osteoporosis, radiotherapy, smoking, etc. on the healing of implant-surrounding bone. Furthermore, owing to coatings, healing is accelerated and the risk of marginal bone resorption is reduced.\(^7\) Coating deposition is done by various techniques, including dip coating, electrophoretic deposition, pulsed laser deposition, sputtering, and plasma spraying.\(^12,14\) Electrophoretic deposition is one of the most applied techniques, being low-cost with short deposition time and room temperature processing. It also allows for film thickness control, where coatings can be deposited in a uniform manner and highly pure form on complex shaped substrates.\(^15-17\)

Since the introduction of bioactive glass (BAG) by Hench in 1969, different compositions and techniques of preparation have been tried for biomedical engineering.\(^18\) BAG has a gradual dissolution rate due to the low connectivity of its silicon dioxide (SiO\(_2\)) network, thus promoting the growth of a hydroxycarbonated apatite layer.
(HCA) on its surface by the action of its released ions. As the hydroxyapatite (HAP) layer continues to grow, BAG becomes substituted with developing bone tissue.\textsuperscript{(19, 20)} Sol-gel BAG is more used in biomedical applications than melt quenched BAG, for being produced at lower processing temperatures, leading to increased bioactivity and bioresorbability, more controlled microstructure, finer particle size, and higher porosity and purity.\textsuperscript{(19, 21, 22)} Moreover, there is higher level of bioactivity for 70S30C BAG (70 mol.% SiO2, 30 mol.% CaO) when compared to different gel glass compositions.\textsuperscript{(21)}

Bioactive glass coatings play a major role in protecting titanium implants against corrosion from body fluids, tissues, and alloy products.\textsuperscript{(23)} Titanium substrates coated with BAG favor cell growth and get more improved responses in contrast to uncoated ones. Regarding coating deposition, a strong bond is mandatory to be achieved between the coating and substrate.\textsuperscript{(24)} BAG composition, sintering temperature, surface roughness, coating thickness, and technique of coating application all influence the strength of adhesion between BAG coating and metallic substrate.\textsuperscript{(25, 26)} Maintaining such an implant-coating bond and resisting the pressure of load are two major criteria to consider a coating material functional, both of which have been shown to be fulfilled by bioactive glasses.\textsuperscript{(27, 28)}

Recently, titanium substrates have successfully been coated with 70S30C BAG nanoparticles. Coated substrates showed uniform coating thickness (32 – 39 µm), super hydrophilicity, and high cellular infiltration and vascularization at the interface upon subcutaneous implantation.\textsuperscript{(29)} The objective of the present study was to develop a surface coating of sol-gel 70S30C BAG nanoparticles on real titanium dental implants by means of electrophoretic deposition technique. BAG coated implants were studied regarding their microstructure, average surface roughness, and coating stability. The amount of coating loss upon implant insertion was calculated through measuring implant weights at different time points (before coating, after BAG coating, and after insertion and atraumatic retrieval from polyurethane). The effect of implant insertion on BAG coating thickness was also evaluated after implantation into bovine rib bone, imitating clinical settings. The null hypothesis was that there would be no significant difference, either in average surface roughness between uncoated and BAG coated implants, or among weights of implants before coating, after coating, and after insertion and retrieval from polyurethane, or in coating thickness between non-inserted and inserted BAG coated implants into bovine rib bone.

**MATERIALS AND METHODS**

**Bioactive glass preparation**
Bioactive glass 70S30C (70% SiO$_2$, 30% CaO) was prepared by modified sol-gel technique following the method published by Gadallah et al (2022)\(^{(29)}\). Polyethylene oxide, urea, tetramethyl orthosilicate, and calcium nitrate tetrahydrate were added to acetic acid aqueous solution and magnetically stirred (Hotplate and stirrer 1000, Jenway, United Kingdom) until completely dissolved. Deionized water with two to three drops of hydrofluoric acid, acting as a gelation catalyst, was added. The solution was then poured into multi-well plate and kept until complete gelation. The wet gel was dried and thermally stabilized at 700ºC. The resultant scaffolds were milled into nanoparticles by a planetary ball mill machine (PM 400, Retsch, Haan, Germany), at 350 rpm for 24 h with ZrO$_2$ balls in a ratio of 9:1 ball:glass.\(^{(30-32)}\)

**Characterization of BAG nanoparticles**

To ensure the amorphous structure of BAG nanoparticles, x-ray diffraction (XRD) analysis was performed in the 2θ range of 20 – 80º at a scan rate of 15º/min. The operation was conducted with copper K alpha (Cu K$_\alpha$) radiation at 30 kV and 10 mA with a wavelength of 1.5418 Å. The collected data was analyzed by Diffrac.Commander™ software. The diffraction database provided by the Joint Committee on Powder Diffraction Standards (JCPDS) was used to match the detected phase.

Field emission transmission electron microscope (FE-TEM, JEM-2100F JEOL, Tokyo, Japan) was also used to measure the particle size of the milled powder. The powder was added to ethanol producing a dispersed solution which was sonicated for 15 min, then 5 µL of the solution was dropped on a carbon coated copper grid and left for complete drying at room temperature. TEM characterization was conducted at 200 kV and particle size measurements were carried out using Gatan digital micrograph software.

Bioactive glass nanoparticles powder was sputter coated with platinum-palladium alloy by an auto fine coater (JEC-3000FC JEOL, Tokyo, Japan) for 75 sec. under 40 mA to have its surface morphology examined by scanning electron microscope (SEM, JSM-6010LV JEOL, Tokyo, Japan). Images were taken at 10 kV with working distance of 12 mm. Powder composition was analyzed using energy dispersive x-ray (EDX) to estimate the elemental peaks of BAG structure. Analysis was done at 20 kV with a magnification power of ×220. ZAF quantification method was employed.

**Preparation of titanium disks**

A titanium blank (grade V; Kera Ti 5-Disc, Eisenbacher Dentalwaren, Bavaria, Germany) was cut into disks of 10 mm in diameter and 1.5 mm in thickness (ISO 23317-2014)\(^{(33)}\) by a computer numerical control (CNC) wire...
cutting machine (EW-C850F, Ecowin, Dali, Taiwan). The disks were then subjected to airborne particle abrasion (Korostar D2800, Bego, Bremen, Germany) for 30 s at a blast pressure of 0.5-0.8 MPa with aluminum oxide particles of 250 µm. A custom-made aluminum holder was fabricated to keep a distance of 35 mm between the disk and the nozzle tip of the machine to standardize the sandblasting procedure. This was followed by ultrasonic cleaning (CD-4820, Codysion, Guangdong, China) for 10 min in absolute ethanol, then drying at room temperature.

**Coating of titanium disks and dental implants with BAG nanoparticles**

Electrophoretic deposition is a process where an electric field is used in a liquid medium to make suspended colloidal particles migrate and deposit onto a counter charged electrode.\(^{(15,34)}\) A glass suspension was prepared by magnetic stirring of 0.5 g BAG nanoparticles in 30 ml of absolute ethanol, then agitated in digital ultrasonic cleaner to achieve non-agglomerated particles.\(^{(35)}\) For the titanium disks, two disks were positioned within a custom-made copper holder to act as electrodes, separated by a fixed distance of 15 mm.\(^{(29)}\) Then, the assembly was immersed in the glass suspension. For the titanium implants, electrophoretic deposition setup with modifications was assembled (Fig. 1). 4.2 x 8 mm uncoated titanium (grade 23; Ti-6Al-4V ELI, with extra low interstitials) dental implants (OneQ-SL, Dentis, Daegu, Korea), with sandblasted, large grit, acid etched (SLA) surface were used. A copper cylinder 1 mm in thickness with inner diameter of 36 mm was used as the anode, while the implant was coupled as the cathode and placed in the center of the counter electrode.\(^{(36,37)}\) A direct current (DC) power supply (MS-605D, Maisheng, Hebei, China) was used as a source of constant voltage for a duration of 60 s at 30 V. After deposition, disks and implants with the resultant coating were dried at ambient temperature for 24 h, then sintered at 700°C for 2 h under argon in vacuum compact tube furnace (GSL-1500X-50LVS, MTI, Richmond, Virginia) with heating and cooling rates of 2°C/min.\(^{(29,38)}\) The coated disks and implants were isolated and stored in a desiccator.

**Surface characterization - Macroscopic and morphological assessment**

Coated implants were inspected macroscopically for coating uniformity, homogeneity, and implant coverage. They were examined, after sintering, by SEM for assessing coating microstructure, and compared qualitatively with uncoated implants and coated unsintered implants. All implants were sputter coated with platinum-palladium alloy by an auto fine coater for 75 sec. under 40 mA. Scanning was done at 10 kV with a working distance of 10 mm under 2 magnifications (x37 and x2000). Coating compositional elements were detected by EDX with an acceleration voltage of 20 kV.

**Coating thickness and implant-coating interface**
To determine coating thickness, coated implants were embedded in resin consisting of benzoyl peroxide (Alpha Chemika, Maharashtra, India) and methacrylate (Sigma-Aldrich, St. Louis, Missouri). Specimens were then longitudinally sectioned at the middle of implants by microtome (Micracut 150, Metkon, Bursa, Turkey) with a diamond wheel (Norton, Worcester, Massachusetts). Scanning electron microscope (JSM-IT200 JEOL, Tokyo, Japan) was used to study implant-coating interface. By ImageJ software\(^{39}\), thickness of BAG coating was measured on thread crests, thread flanks, thread roots, and antirotation grooves on each sectioned specimen (n=10).\(^{40}\)

**Topography and roughness analysis**

Since surface topography is dependent on surface roughness and orientation\(^{41}\), 2D and 3D topographies were observed, and average surface roughness (arithmetical mean height of a surface, \(S_a\))\(^{42}\) was measured for uncoated implants (control group; n=6) versus BAG coated implants (test group; n=6) by confocal laser scanning microscope (VK-X100 series, Keyence, Osaka, Japan). Sample size was estimated assuming 80% study power and 5% alpha error. Gadallah et al. (2022)\(^{29}\) reported mean (SD) surface roughness of 2.81 (0.32) for uncoated titanium substrates, and 1.74 (0.19) for bioactive glass coated ones. Based on comparison of means, the minimum sample size was calculated to be 5 implants, increased to 6 to make up for laboratory processing errors. Sample size was calculated using MedCalc Statistical Software version 19.0.5 (MedCalc Software bvba, Ostend, Belgium; 2019). Implants were placed on a 3D-printed custom-made base for stability on the microscope sample table. Laser was oriented to scan a spot on the flat antirotation groove of each implant under magnification power of \(\times50\). \(S_a\) values were determined with Keyence VK analyzer software.

**Adhesion strength of BAG coating**

A custom-made device was fabricated to conduct the adhesion strength test (Fig. 2). The device consisted of a copper holder with a slot of 6 mm in diameter in order to fit a copper cylindrical of the same diameter. Disks (n=6) were glued with epoxy adhesive (3M™ Scotch-Weld™ DP460, Saint Paul, Minnesota) to the copper cylinder so that 2 mm are left unglued all around each disk. A pressure of 0.2 MPa was applied to the glued assembly for 24 hours at room temperature until complete curing of the adhesive material.\(^{43}\) Then, it was transferred to the fabricated holder and placed in grips of the universal testing machine (Instron 3345, Norwood, Massachusetts). As recommended by ASTM C633\(^{44}\), the test was carried out at a constant crosshead speed of 1 mm/min at ambient temperature until complete separation of the assembly, which is the point of failure load. The adhesion strength (MPa) was then calculated by dividing the failure load (N) by the surface area of the tested coating (\(\text{mm}^2\)). Mode of failure was

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 qualitatively analyzed through stereomicroscopic (SZ-11, Olympus, Japan) images to study the remaining exposed surface after failure. The tested disks were also scanned by SEM and EDX to detect surface components after failure.

**Insertion of BAG coated implants into artificial bone**

Solid rigid polyurethane is used as an alternative test medium for human bone.\(^{(45)}\) It has similar mechanical properties to that of human cancellous bone, as described by the ASTM F-1839-08 standard\(^{(46)}\), making it a good material for procedural standardization with excluding anatomical and structural variations found in natural bone.\(^{(47,48)}\) Density of polyurethane (Nacional Ossos, São Paulo, Brazil) used in the current study was 20 pounds per cubic foot (PCF) corresponding to 0.32 g/cm\(^3\) (similar to (similar to thick porous compacta and coarse trabecular D2 type of bone)\(^{(50)}\) covered with a 2 mm cortical layer of polyurethane 40 PCF (0.64 g/cm\(^3\)) to mimic human alveolar bone.\(^{(49,50)}\) Two blocks were bound tightly together by a bar clamp (Irwin, Huntersville, North Carolina) to allow an atraumatic removal of implants after their insertion and hence, preserving their surface.\(^{(54-56)}\) A 3D printed surgical guide (Formlabs 2 desktop 3D printer, Formlabs, Somerville, Massachusetts) was fitted on the top such that sleeves were centered over the midline interface of the two blocks (Fig. 3a). Drilling was carried out by one calibrated, experienced clinician for intraexaminer reliability.\(^{(57)}\) Dentis Simple Guide Plus kit (Dentis, Daegu, Korea) was used for drilling with a surgical dental implant motor (X-Cube, Saeshin, Daegu, Korea) according to manufacturer’s instructions. Compressed air was applied between each drilling and at the end of drilling procedure to remove the resultant debris.\(^{(57)}\)

**Mechanical characterization - Measuring coating loss upon implant insertion**

Sample size was estimated assuming 5% alpha error and 80% study power. Based on a study performed by Hägi et al (2010)\(^{(58)}\), mean ±standard deviation of weight of coated implants before insertion into artificial bone was 1051.82 ±2.35 mg, while after insertion it was 1048.18 ±1.76 mg. Required sample size was calculated by G*power 3.0.10\(^{(59)}\) to be 5 implants, which increased to 8 to account for possible experimental errors. BAG coated implants (n=8), which were previously weighed before and after coating on a microbalance (XP26 Mettler Toledo, Greifensee, Switzerland), were inserted into polyurethane through the surgical guide, under a maximum torque of 30 Ncm, imitating the clinical scenario as accurately as possible (Fig. 3b). After complete insertion, the guide and bar clamps were removed to permit implant atraumatic retrieval from polyurethane blocks, to be weighed once more. Weight of
Implants before coating, after coating, and after their insertion and retrieval from polyurethane were used to calculate the amount of coating loss that occurred during implant placement.\(^{(58)}\)

**Qualitative assessment of retrieved implants**

After retrieval of implants from polyurethane, they were examined macroscopically and by SEM to inspect sites of coating loss, and to study implant-coating interface. Exposed titanium surface after coating delamination and polyurethane sockets existing after implants retrieval were scanned by EDX to estimate the presence of BAG on their surfaces.

**Implantation of BAG coated implants in bovine rib bone**

Bovine ribs were used because of their similarity with human edentulous jawbone in terms of macroscopic cortical and medullary composition, relationship and density.\(^{(60,61)}\) Bovine rib bones were brought from butcher’s shop and originated from the same animal which was not killed for the experiment.\(^{(62)}\) Fresh ribs were cleaned, and all soft tissue residues were removed.\(^{(63)}\) Blocks of 6 cm long were cut from the most proximal region of the ribs for their larger diameter, minimal cortical and greater cancellous bone, being similar to type-III/IV bone (D3, porous compacta and fine trabecular bone; and D4, fine trabecular bone)\(^{(50)}\), commonly found in human maxillary bone.\(^{(60,64)}\) The base of each bone block was levelled and made flat to be stable during osteotomy preparation and implant insertion.\(^{(65)}\) Cone-beam computed tomography was done for the bone blocks to accurately define the suitable position of implants and to allow guided insertion. Bone blocks were then stored in normal saline solution and frozen at -10°C till the time of experiment as described by Sedlin and Hirsch.\(^{(66)}\) On BlueSkyBio software, surgical guides were designed such that each bone block would receive two implants separated by 6 mm. Surgical guides were 3D-printed in clear resin by Formlabs 2 desktop 3D printer. Before implant site preparation, bone blocks were kept for 3 hours at room temperature while being hydrated through wrapping with saline-soaked gauze. Then, they received the surgical guides on their top (Fig. 3c).\(^{(61)}\) The bone-surgical guide assembly was fixed to the table using bar clamps to exclude any movement during osteotomy preparation or implant insertion. Osteotomies were prepared under saline irrigation through the surgical guide using Dentis Simple Guide Plus kit. BAG coated implants were then inserted in the pre-prepared osteotomies through the surgical guide, under a maximum torque of 30 Ncm according to the manufacturer’s instructions (Fig. 3d).

**Examination of BAG implants after insertion into bone by SEM**
After complete implant insertion, the assembly components were separated, and bone blocks containing the implants were embedded in resin (benzoyl peroxide and methacrylate). Embedding procedures started by cutting bone into smaller blocks, each containing one implant in the middle. These blocks were left overnight in phosphate buffered formalin for fixation before being immersed in tap water for 24 h. They were then dehydrated in ethyl alcohol with gradual increase in concentrations every 24 h until reaching the absolute alcohol. The specimens were finally placed in xylene for clearance before being embedded in resin to start polymerization. BAG coated implants, which were not inserted into bone, were also embedded in resin to serve as control. After resin was completely polymerized, microtome was used to grind each resin block at the middle of implants. The specimens were studied by SEM after being gold sputter coated at 10 mA by ion sputter evaporator (JFC-1100E - JEOL, Tokyo, Japan). SEM images were taken at 20 kV under different magnifications. BAG coating thickness was measured by ImageJ software, then compared for both non-inserted (control group; n=10) and inserted implants (test group; n=10) to evaluate the effect of implant insertion on the coating thickness in different locations (thread crests, thread flanks, thread roots and antirotation grooves).

Statistical Analysis

Data were analyzed using IBM SPSS for Windows (Version 23.0) and significance was inferred at P value <.05. Frequencies and percentages were calculated for categorical variables. Normality was checked for all quantitative variables using descriptive statistics, plots, and normality tests. All variables showed normal distribution, so means, standard deviation (SD), and 95% confidence interval (CI) were calculated, and parametric tests were used. Comparisons of surface roughness between uncoated and BAG coated implants were done using independent samples t-test. Comparison of implant weights at different timepoints (before coating, after coating, and after insertion) was done using repeated measures ANOVA, followed by multiple pairwise comparisons using Bonferroni adjusted significance level. Comparisons of BAG coating thickness between non-inserted and inserted implants were done using Chi-squared test with Monte Carlo corrected p value.

RESULTS

Characterization of BAG nanoparticles

X-ray diffraction analysis of BAG nanoparticles exhibited the broad amorphous halo at $2\theta = 15 - 35^\circ$ commonly observed for glasses, confirming the amorphous structure of the sample (Fig. 4a).
Through transmission electron microscopy, BAG particles were reported to be nanosized with particle diameter of less than 20 nm (4.87 to 18.55 nm; Fig. 4b). The nanoparticles were highly clustered into larger aggregates. SEM revealed the irregular morphology of BAG nanoparticles with different sizes and shapes (Fig. 4c). The powder consisted of a mixture of nano-sized particles and micro-sized agglomerates. EDX showed the dominant peaks of oxygen, silicon, and calcium, the main constituent elements of BAG (Fig. 4d).\(^{(29)}\)

**Surface characterization - Macroscopic and morphological assessment**

Macroscopically, electrophoresis resulted in uniform coating deposition. Implant surfaces were continuously covered with BAG, including implant thread crests (Fig. 5). SEM revealed homogenous coating with no obvious irregularities for both unsintered and sintered coated implants (Fig. 6). Organized microcracks were observed throughout the entire surface of the sintered coated implants, when compared to unsintered ones (Fig. 6c and 6e). Moreover, nanoparticles in the coating did not fuse or melt after sintering at 700ºC, instead preserved their amorphous irregular compact appearance among the cracks (Fig. 6f). EDX analysis of coated implants after sintering showed the typical components of BAG (Si, Ca, and O; Fig. 7).

**Coating thickness and implant-coating interface**

Upon examining the longitudinally sectioned specimens by means of SEM, BAG coating was found to have a uniform thickness, interrupted by the previously observed microcracks (Fig. 8a and 8b). BAG coating was in close contact to the titanium surface with no visible spacing between both. Thickness of BAG coating on different locations of coated implants is illustrated in Table 1, with an overall mean thickness of 35.38 ±4.67 μm (mean ±SD).

**Topography and roughness analysis**

Confocal laser scanning microscope analysis confirmed the foregoing observations of SEM, regarding topographies of uncoated and BAG coated implants. Uncoated titanium implants presented irregularities all over the surface (Fig. 9a and 9b), which were noticeably reduced in BAG coated implants resulting in a finer and more uniform surface microstructure (Fig. 9c and 9d). This surface planarization is accompanied with a statistically significant decrease in average surface roughness (\(S_a\)) for BAG coated implants, when compared to that of uncoated implants (3.34 ±0.45 μm for uncoated implants; 1.45 ±0.23 μm for BAG coated implants; \(P=.001\)) as shown in Table 2. The microcracks formed on BAG coating surface after sintering are demonstrated in Fig. 10, with their measured widths ranging between 1.47 and 3.66 μm.

**Adhesion strength of BAG coating**
Results of adhesion test for BAG coated titanium disks are shown in Table 3. The adhesion strength of the resultant coating was calculated to be 18.51 ±3.37 MPa (mean ±SD). Stereomicroscopic images illustrated mixed failure at the coating/glue interface, cohesive failure at the glue phase, and adhesive failure at the glue/copper interface, while the titanium/coating interface was intact (Fig. 11a and 11b). The mode of failure was confirmed by EDX analysis of exposed areas showed elemental peaks of Si and Ca, which indicate the presence of BAG. Carbon peak was related to the glue material (Fig. 12).

**Mechanical characterization - Measuring coating loss upon implant insertion**

Statistically significant difference was found among implant weights at different time points (before coating, after BAG coating, and after their insertion and retrieval from polyurethane test blocks) as demonstrated in Table 4. Measurements revealed that the mean weight of intact BAG coating is 4.28 mg, while the mean weight of coating that remained on implants after insertion and retrieval is 2.86 mg, with less than 40% coating loss occurred upon implant insertion.

**Qualitative assessment of retrieved implants**

On macroscopic examination of implants which were inserted and retrieved from polyurethane test blocks, BAG coating was only sheared off at points of primary stability (thread crests) due to their high friction with socket walls during insertion (Fig. 13a). The coating appeared almost intact without delamination both on thread roots and antirotation grooves. However, BAG coating underwent fragmentation on the implant neck due to its contact with denser polyurethane layer (40 PCF, 0.64 g/cm³) mimicking the cortical layer of human alveolar bone. EDX for exposed titanium surface indicated the presence of BAG compositional elements (Si, Ca, and O), confirming that titanium was still covered with BAG nanoparticles even after its delamination (Fig. 13b). Carbon peak was high due to implant contact with polyurethane test blocks, mainly composed of C, H, O, and N. (47)

On examining polyurethane after implant retrieval, the sockets contained BAG on their walls (Fig. 13c). In other words, BAG coating which was detached from the implant surface during insertion was not entirely lost outside the socket but retained within the peri-implant space. This was confirmed by EDX analysis for the polyurethane socket, which revealed the presence of silicon, calcium, and oxygen (BAG components) in high percentages on its surface (Fig. 13d). The high peak of carbon is related to the composition of the polyurethane polymer itself. (47) SEM of retrieved implants revealed that, after coating fragmentation, there was no clearly defined interface between titanium implant surface and BAG coating (Fig. 14a and 14b). The lack of distinguishable interface indicates that BAG coating
was not entirely removed through its whole thickness but remained attached to the titanium surface. BAG coating underwent cohesion failure within itself rather than adhesion failure with titanium surface.

**Examination of BAG coated implants after insertion into bone by SEM**

Figure 15a shows BAG coated implant, inserted into bovine rib bone. BAG coating has almost been intact throughout its thickness on the antirotation groove due to its passive, yet intimate, contact with bone during insertion (Fig. 15b). Reduction in BAG coating thickness was observed on the thread crests and flanks

A qualitative analysis was performed to evaluate the effect of implant insertion on the thickness of BAG coating (Table 5). Most readings of BAG coating thickness lied in the range of 30-40 µm for the non-inserted implants. After insertion, BAG coating thickness majorly decreased on the thread crests and flanks to lie in the 0-10 µm range, indicating the high friction with bone and shear stresses applied during insertion. On the thread roots, readings mostly constituted the range of 10-20 µm after insertion. However, there was no statistically significant difference between the non-inserted and inserted implants for BAG coating thickness on the antirotation grooves, supporting the results of SEM.

**DISCUSSION**

Dental implant therapy has long provided high rate of clinical success for restoring lost teeth in healthy patients. However, due to poor quality bone, impaired blood supply, and delayed tissue healing in medically compromised and aged patients, implant failure can occur presenting a critical challenge. Consequently, there is an urgent need for further evidence-based research on novel implant surfaces that can integrate more rapidly and effectively into host bone. Bioactive glasses are becoming widely attractive and greatly considered in bone regeneration and healing for their ability to adhere to bone and soft tissues without being rejected.

The present study aimed to develop a surface coating of sol-gel 70S30C BAG nanoparticles (< 20 nm) on titanium disks and real dental implants, to help reduce the time interval needed between implant installation and osseointegration, and to improve implant-bone cellular interactions. Bioinert titanium is unable to sustain early osseointegration, especially in patients with systemic diseases due to impaired blood supply and delayed bone remodeling, which may result in implant failure. Titanium dental implants with a sandblasted, large grit, acid-etched (SLA) surface were used in this study, where Khalili and Naji (2020) reported that mechanical adhesion between the metal and coating could be enhanced by pretreating the metal substrate with sandblasting followed by acid etching before being coated.
Sol-gel 70S30C BAG was chosen as the coating material for being the most bioactive among the binary calcium silicate glass system (CaO-SiO2) as reported by Sarvanapavan and Hench (2001)(21). This system is well known to have the potential to discharge ions that regulate many physiological activities. Dissolution of Si and Ca ions from BAG creates a slightly alkaline environment, suitable for adhesion and proliferation of cells, and production of matrix, leading to faster implant integration. This occurs because of the ionic exchange occurring between BAG and surrounding environment by the effect of the silicate network. Dental implants with BAG coatings become therapeutic implants with a multimodal local delivery system.(29) BAG was prepared following the modified sol-gel technique demonstrated by Gadallah et al (2022)(29), then were ball milled into nanoparticles. When compared to microscale particles, nanoparticles turn out to produce more homogenous coating and provide better bioactivity.(72,73)

Electrophoretic deposition was carried out following the technique mentioned by Braem et al. (2012)(36), where the implant was used as the cathode surrounded by a metal cylinder acting as the anode to ensure homogenous coating all around the implant. After electrophoretic deposition, BAG coated implants were sintered under argon in vacuum furnace to avoid titanium oxidation that may result during sintering.(36) Martinez et al (2000)(38) revealed that optimal sintering should be performed between 700ºC for complete elimination of nitrate groups, and 900ºC where crystallization of β-wollastonite occurs. Consequently, the BAG coated implants were sintered for 2 h at 700ºC in order to preserve its amorphous structure, by not exceeding the glass transition temperature (717ºC) and crystallization temperature (845ºC) of sol-gel derived BAGs.

The resultant coating was homogenous, possessing a porous network of BAG nanoparticles with fine and organized surface microcracks, as was documented for calcium silicate coatings with higher Si content.(29) Microcracks resulted from the mismatch in coefficients of thermal expansion between titanium and BAG during sintering phase, coupled with residual stresses at the interface between them.(11,14)

Coating mechanical properties are governed by its thickness.(13) The resultant BAG coating showed a uniform thickness with an average of 35.38 ±4.67 μm (mean ±SD), which came in accordance with results of Gadallah et al. (2022)(29). BAG coating showed lower thickness on the thread flanks, which can be attributed to their non-perpendicular orientation to the electrolytic flow at the time of electrophoretic deposition, in contrast to the thread crests and roots which were almost at a right angle. Moreover, thread crests had the greatest thickness due to their closest position and shortest distance to the cylindrical copper anode. Burtscher et al. (2019)(13) reported that layer thicknesses between 20 and 50 μm are optimal, for their high corrosion resistance and bonding strength.
and delamination occur with thicker coatings (>100 μm), due to receiving higher stresses of shear, tension, and compression. Dou et al. (2013) also showed that thinner coatings within the nano-range (>1 μm) resulted in metal substrate corrosion, together with coating cracking and peeling off upon insertion in simulated body fluid.

Results of the current study did not support the stated null hypothesis. BAG coated implants demonstrated $S_a$ of 1.45 ±0.23 μm, which agreed with results of Gadallah et al. (2022) for 70S30C BAG coated titanium substrates ($S_a$=1.737 ±0.193 μm). Braem et al. (2012) also reported the same range of values for melt quenched BAG, coated on titanium substrates by electrophoretic deposition ($S_a$=1.59 ±0.34 μm). Titanium implant surface was transformed in the current investigation into a micro/nanoscaled surface with moderate average surface roughness, according to the classification suggested by Albrektsson and Wennerberg (2004). Moderately rough surfaces result in better bone response and more enhanced clinical performance, when compared to smoother or rougher surfaces.

Adhesion strength of implant coatings is mandatory to withstand mechanical forces during implant placement. Results of the current study regarding the adhesion strength of 70S30C BAG (18.51 ±3.37 MPa) showed higher values than those of plasma sprayed 45S5 BAG (8.56 ±0.57 MPa). Moreover, they exceeded the reported required adhesion strength of at least 15 MPa for plasma sprayed hydroxyapatite coatings on titanium alloys. Thickness of coatings should be taken into consideration in order to maximize performance of the coating material. According to Baino (2019), thinner coatings (48 ±5 μm) showed better adhesion results than thicker ones (296 ±8 μm).

According to Mehdipour et al. (2012), voltage used in electrophoretic deposition also affects coating adhesion strength to substrate. It was reported that adhesion strength increased with the increase in voltage up to 60 V, after which it started to decline once more, due to the increase in coating thickness with higher voltages. Although quantitative data are mandatory for adhesion strength assessment, the qualitative mode of failure is important as well since it brings the quantitative results into reality. Absence of titanium elemental peak in EDX analysis indicated that the actual adhesion strength of BAG to titanium disks is relatively higher than the calculated value (18.5 MPa).

BAG coated implants were inserted into polyurethane mechanical test blocks to measure the amount of coating loss upon implant insertion. More than 60% of the coating weight remained on the implant surface after their insertion and atraumatic retrieval from polyurethane test blocks. This percentage of remaining BAG coating was more than that resulted by Hägi et al. (2010), who conducted the same experiment for calcium phosphate (CaP) coatings, which either lacked or bore an incorporated depot of the model protein bovine serum albumin (BSA). According to
Hägi et al, percentage of remaining CaP coating was 39.22% for coatings which lacked BSA, while 55.38% for those which bore BSA.

The barely disturbed BAG coating layer on implant thread roots and antirotation grooves could be translated into high coating stability, and low susceptibility to fragmentation. Delamination primarily occurred on vulnerable areas responsible for primary stability, which are the thread crests and thread flanks, due to high friction during implant insertion. Presence of BAG nanoparticles on exposed titanium surface after coating delamination, as demonstrated by EDX analysis, together with indistinct implant-coating interface reflects the strong mechanical bond between titanium implant and BAG coating.

BAG coated implants were inserted into bovine rib bone to assess the effect of insertion on the thickness of BAG coating. After insertion, reduction in BAG coating thickness was noticed in sites of primary stability, which normally undergo the highest friction with bone during implant insertion. Almost intact coating layer was observed, by SEM and measurements, along the thread roots and antirotation grooves. These results came in accordance with those of Hägi et al (2010) (58), where both types of calcium phosphate coatings, either bearing or lacking incorporated depot of BSA, showed a decrease in mean overall thickness after implant insertion into explanted porcine mandibles, with a progressive increase in coating thickness from the outer chamber (thread crest) to the inner chamber (thread roots).

Within the limits of the current investigation, the following could be concluded: Dental titanium implants can be surface coated with sol-gel derived 70S30C BAG nanoparticles, through economic and effective electrophoretic deposition technique. The optimized technique parameters resulted in a homogenous, uniform, amorphous, and porous BAG coating. Statistically significant lower average surface roughness was observed for BAG coated implants, when compared to uncoated ones, resulting in moderately rough surface ($S_a=1.45 \pm 0.23 \, \mu m$). BAG coating demonstrated an adhesion strength of 18.51 ±3.37 MPa to the underlying titanium substrates. Furthermore, more than 60% of the coating weight remained on implants after insertion and retrieval from polyurethane, indicating mechanically durable and stable BAG coating with strong bond to the implant surface. Coating thickness was reduced only in sites undergoing high friction with bone during implant insertion, yet remained barely disturbed elsewhere.

Findings of the present study may facilitate dental implant therapy in medically compromised patients. The mechanical characterization performed for BAG coated implants proves that the coating is durable enough to withstand implant insertion into bone. The study was mainly limited by the absence of in vivo or clinical studies, as it
mainly involved in vitro surface and mechanical characterization of BAG coating. The study’s limitations also included the use of homogenous and isotropic polyurethane test blocks with particular cortical thickness and bone density, which does not reflect the clinical reality. However, bovine rib bones were also used in the study which are more similar to human jawbone in terms of composition and density. Further investigations are required to evaluate the effect of BAG coating on the insertion torque and primary stability of implants, due to the resultant increased diameter of implants after coating. Moreover, in vivo and clinical studies are needed to analyze the integration of BAG coated implants with bone, together with its immunologic and biologic behavior.

**FUNDING**

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**CONFLICT OF INTEREST**

The authors have no conflict of interest related to the study.

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**TABLES**

**Table 1:** Thickness of BAG coating on different locations of titanium implant.

<table>
<thead>
<tr>
<th>Coating thickness (μm)</th>
<th>Thread crests</th>
<th>Thread flanks</th>
<th>Thread roots</th>
<th>Antirotation grooves</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Mean ±SD</strong></td>
<td>38.08 ±5.19</td>
<td>33.28 ±3.28</td>
<td>35.74 ±5.3</td>
<td>34.42 ±3.51</td>
</tr>
<tr>
<td><strong>Median (IQR)</strong></td>
<td>36.05 (7.72)</td>
<td>33.03 (2.85)</td>
<td>35.5 (7.87)</td>
<td>34.09 (3.83)</td>
</tr>
<tr>
<td><strong>Min – Max</strong></td>
<td>30.22 – 47.17</td>
<td>28.28 – 41.75</td>
<td>27.61 – 44.88</td>
<td>28.16 – 42.18</td>
</tr>
</tbody>
</table>

SD: standard deviation, IQR: interquartile range, Min: minimum, Max: maximum

**Table 2:** Comparison of average surface roughness ($S_a$) between uncoated and BAG coated titanium implants by confocal laser scanning microscope (μm).

<table>
<thead>
<tr>
<th></th>
<th>Uncoated implants</th>
<th>Coated implants</th>
<th>Mean difference</th>
<th>Percent change</th>
<th>P value</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Mean ±SD</strong></td>
<td>3.34 ±0.45</td>
<td>1.45 ±0.23</td>
<td>1.90 ±0.63</td>
<td>-55.67 ±11.12</td>
<td>.001*</td>
</tr>
<tr>
<td><strong>95% CI</strong></td>
<td>2.87, 3.82</td>
<td>1.20, 1.69</td>
<td>1.23, 2.56</td>
<td>-67.33, -44.00</td>
<td></td>
</tr>
</tbody>
</table>

SD: standard deviation, CI: confidence interval

T-test was used

*Statistically significant at P value <.05
Table 3: Results of adhesion bond strength test of BAG coating (n=6).

<table>
<thead>
<tr>
<th></th>
<th>Ultimate force (N)</th>
<th>Ultimate stress (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Disk A</td>
<td>519</td>
<td>18.36</td>
</tr>
<tr>
<td>Disk B</td>
<td>496</td>
<td>17.55</td>
</tr>
<tr>
<td>Disk C</td>
<td>429</td>
<td>15.18</td>
</tr>
<tr>
<td>Disk D</td>
<td>630</td>
<td>22.29</td>
</tr>
<tr>
<td>Disk E</td>
<td>423</td>
<td>14.96</td>
</tr>
<tr>
<td>Disk F</td>
<td>643</td>
<td>22.75</td>
</tr>
<tr>
<td>Mean ±SD</td>
<td>523.33 ± 95.31</td>
<td>18.51 ± 3.37</td>
</tr>
</tbody>
</table>

SD: standard deviation
**Table 4:** Comparison of implant weights (mg) at different timepoints (T<sub>0</sub>, T<sub>1</sub>, and T<sub>2</sub>).

<table>
<thead>
<tr>
<th>Implant</th>
<th>Weight before coating T&lt;sub&gt;0&lt;/sub&gt; (mg)</th>
<th>Weight after coating T&lt;sub&gt;1&lt;/sub&gt; (mg)</th>
<th>Weight after insertion T&lt;sub&gt;2&lt;/sub&gt; (mg)</th>
<th>Total weight of coating (mg)</th>
<th>Coating loss (mg)</th>
<th>Coating loss (%)</th>
<th>Remaining coating (mg)</th>
<th>Remaining coating (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Implant A</td>
<td>165.54</td>
<td>170.05</td>
<td>168.91</td>
<td>4.51</td>
<td>1.14</td>
<td>25.28</td>
<td>3.37</td>
<td>74.72</td>
</tr>
<tr>
<td>Implant B</td>
<td>163.91</td>
<td>168.21</td>
<td>166.75</td>
<td>4.3</td>
<td>1.46</td>
<td>33.95</td>
<td>2.84</td>
<td>66.05</td>
</tr>
<tr>
<td>Implant C</td>
<td>163.99</td>
<td>167.89</td>
<td>165.99</td>
<td>3.9</td>
<td>1.9</td>
<td>48.72</td>
<td>2</td>
<td>51.28</td>
</tr>
<tr>
<td>Implant D</td>
<td>163.71</td>
<td>168.3</td>
<td>167.14</td>
<td>4.59</td>
<td>1.16</td>
<td>25.27</td>
<td>3.43</td>
<td>74.73</td>
</tr>
<tr>
<td>Implant E</td>
<td>165.87</td>
<td>170.17</td>
<td>169.16</td>
<td>4.3</td>
<td>1.01</td>
<td>23.49</td>
<td>3.29</td>
<td>76.51</td>
</tr>
<tr>
<td>Implant F</td>
<td>164.54</td>
<td>169.16</td>
<td>167.9</td>
<td>4.62</td>
<td>1.26</td>
<td>27.27</td>
<td>3.36</td>
<td>72.73</td>
</tr>
<tr>
<td>Implant G</td>
<td>165.07</td>
<td>169.18</td>
<td>167.22</td>
<td>4.11</td>
<td>1.96</td>
<td>47.69</td>
<td>2.15</td>
<td>52.31</td>
</tr>
<tr>
<td>Implant H</td>
<td>163.77</td>
<td>167.69</td>
<td>166.18</td>
<td>3.92</td>
<td>1.51</td>
<td>38.52</td>
<td>2.41</td>
<td>61.48</td>
</tr>
<tr>
<td><strong>Mean</strong></td>
<td>164.55</td>
<td>168.83</td>
<td>167.41</td>
<td>4.28</td>
<td>1.43</td>
<td>33.77</td>
<td>2.86</td>
<td>66.23</td>
</tr>
<tr>
<td>±SD</td>
<td>±0.85</td>
<td>±0.95</td>
<td>±1.17</td>
<td>±0.28</td>
<td>±0.35</td>
<td>±10.23</td>
<td>±0.59</td>
<td>±10.23</td>
</tr>
<tr>
<td>SEM</td>
<td>0.30</td>
<td>0.34</td>
<td>0.42</td>
<td>0.10</td>
<td>0.13</td>
<td>3.62</td>
<td>0.21</td>
<td>3.62</td>
</tr>
<tr>
<td><strong>95% CI</strong></td>
<td>163.84, 168.03, 166.43, 165.26, 169.63, 168.39</td>
<td>4.04, 1.13, 25.22, 2.36, 57.68, 42.32, 3.35</td>
<td>74.78</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>RM-ANOVA P value</strong></td>
<td>T&lt;sub&gt;0&lt;/sub&gt;, T&lt;sub&gt;1&lt;/sub&gt;, and T&lt;sub&gt;2&lt;/sub&gt;</td>
<td></td>
<td></td>
<td>&lt;.001*</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Post-hoc</strong></td>
<td>T&lt;sub&gt;0&lt;/sub&gt; vs. T&lt;sub&gt;1&lt;/sub&gt;</td>
<td>&lt;.001*</td>
<td>T&lt;sub&gt;0&lt;/sub&gt; vs. T&lt;sub&gt;2&lt;/sub&gt;</td>
<td>&lt;.001*</td>
<td>T&lt;sub&gt;1&lt;/sub&gt; vs. T&lt;sub&gt;2&lt;/sub&gt;</td>
<td>&lt;.001*</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

RM-ANOVA: Repeated measures ANOVA was used.

T<sub>0</sub>: weight before coating, T<sub>1</sub>: weight after coating, and T<sub>2</sub>: weight after insertion; SD: standard deviation, SEM: standard error of the mean, and CI: confidence interval. Statistically significant at P value <.05.
Table 5: Qualitative analysis for the effect of implant insertion on the thickness of BAG coating.

<table>
<thead>
<tr>
<th>N (%)</th>
<th>Thread tips</th>
<th>Thread sides</th>
<th>Surfaces between threads</th>
<th>Vertical anti-rotation grooves</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Not inserted</td>
<td>Inserted</td>
<td>Not inserted</td>
<td>Inserted</td>
</tr>
<tr>
<td>40 – 50 μm</td>
<td>3 (30%)</td>
<td>0 (0%)</td>
<td>1 (10%)</td>
<td>0 (0%)</td>
</tr>
<tr>
<td>30 – 40 μm</td>
<td>7 (70%)</td>
<td>0 (0%)</td>
<td>8 (80%)</td>
<td>0 (0%)</td>
</tr>
<tr>
<td>20 – 30 μm</td>
<td>0 (0%)</td>
<td>0 (0%)</td>
<td>1 (10%)</td>
<td>0 (0%)</td>
</tr>
<tr>
<td>10 – 20 μm</td>
<td>0 (0%)</td>
<td>0 (0%)</td>
<td>0 (0%)</td>
<td>2 (20%)</td>
</tr>
<tr>
<td>0 – 10 μm</td>
<td>0 (0%)</td>
<td>10 (100%)</td>
<td>0 (0%)</td>
<td>8 (80%)</td>
</tr>
</tbody>
</table>

P_{MC} value: <.001* <.001* .002* 1.00

Values relating to the most important parameters are represented in bold.

N: number of readings out of 10

*Statistically significant at P value <.05
FIGURES

Fig. 1: Diagram showing modifications made in electrophoretic deposition technique to coat a 3D complex object/implant.

Fig. 2: Diagram to clarify the custom-made device used for adhesion bond strength testing.
Fig. 3: Implant insertion into artificial and natural bone. (a) Two polyurethane test blocks clamped together by a bar clamp (1) and fixed to the desk with another bar clamp (2) to ensure standardized experimental conditions and reduce movements during drilling procedure. (b) Guided implant insertion through the surgical guide into preprepared sockets. (c) Bovine rib bone block 6 cm in length with 3D-printed surgical guide on top. (d) Insertion of BAG coated implant into the bone block.
Fig. 4: Characterization of BAG nanoparticles. (a) XRD spectroscopy of 70S30C BAG nanoparticles powder. (b) Transmission electron microscopy revealing particle size of less than 20 nm after introduction of BAG powder into planetary ball mill machine (scale bar = 100 nm and 200 nm, respectively). (c) SEM image for the milled BAG nanoparticles (×5000; scale bar = 5 µm). It is noticed from TEM and SEM images that the nanoparticles have high tendency for agglomeration. (d) EDX analysis representing the main elemental peaks of Si, Ca, and O.
**Fig. 5:** Uncoated versus BAG coated titanium implant after sintering at 700°C for 2 h under argon.

**Fig. 6:** Scanning electron microscopy assessment of implant surfaces. (a and b) Uncoated titanium implant ([a]×37; scale bar=500 µm, [b] ×2000; scale bar=10 µm). (c and d) BAG coated titanium implant before sintering ([c]×37; scale bar=500 µm, [d] ×2000; scale bar=10 µm). (e and f) BAG coated titanium implant after sintering ([e]×37; scale bar=500 µm, [f] ×2000; scale bar=10 µm).
Fig. 7: EDX analysis representing the main elemental peaks of silicon, calcium, and oxygen.
**Fig. 8:** Titanium implant-BAG coating interface. (a and b) SEM images for longitudinal sections of (a) Uncoated versus (b) BAG coated titanium implants embedded in resin (×200; scale bar=100 µm).

**Fig. 9:** Topography and roughness analysis. (a) 2D and (b) 3D confocal laser scanning microscope image for the surface topography of uncoated titanium implants (×50; scale bar=100 µm) in (c) 2D and (d) 3D confocal laser scanning microscope for the surface topography of BAG coated titanium implants after sintering (×50; scale bar=100 µm).
**Fig. 10**: Confocal laser scanning microscope image for the coating microstructure with measured widths of cracks (×50; scale bar = 100 µm).
Fig. 11: Stereomicroscopic images for (a) The surface of BAG coated titanium disk after failure and (b) The opposing surface of copper cylinder.

Fig. 12: EDX analysis for the same surface indicating the presence of BAG on exposed titanium surface.
Fig. 13: Qualitative assessment of retrieved implants. (a) BAG coated titanium implant after insertion and atraumatic retrieval from polyurethane test blocks. (b) EDX analysis for the retrieved implants confirming the presence of BAG components on the exposed titanium surface even after coating delamination. (c) After separation of the two previously clamped blocks, note the BAG on the socket walls. (d) EDX analysis demonstrating the presence of BAG constituents (elemental peaks of Si, Ca, and O) on the socket walls of polyurethane test blocks.
**Fig. 14:** (a and b) Interface between the implant surface and BAG coating after insertion ([a] ×430, [b] ×500; scale bar = 50 µm). They confirm that, even after coating detachment, titanium surface is not bare and still contains BAG nanoparticles on its surface.
Fig. 15: Examination of BAG coated implants after insertion into bone. (a) An overall longitudinal SEM section for BAG coated titanium implant after insertion into bovine rib bone, showing BAG coating thickness on different implant sites. (×37; scale bar = 500 µm). (b) Minimally disturbed BAG coating on the anti-rotation groove (×800; scale bar = 20 µm).