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Synthesis and Microstructural Characterization of Ti/SiO₂ Composite Powder Fabricated by Powder Metallurgy

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Abstract

Titanium/Silicon dioxide (Ti/SiO₂) composite powder was prepared through powder metallurgy process of high-energy mechanical mixing, uniaxial compaction under 200 MPa and vacuum sintering under 1100 °C for 4 h. Multi-technique characterization of phase composition, chemical bonding and microstructure was done by using the X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM/EDX). XRD unambiguously confirmed retention of the α -quartz SiO₂ phase (ICDD 00-033-1161), with the principal (101) reflection at $2\theta = 26.661^\circ$ yielding $d_{101} = 3.342$ Å and hexagonal lattice parameters $a = 4.913$ Å, $c = 5.405$ Å. The crystallite size obtained from Scherrer analysis was 138 ± 10 nm, a nanoscale value corresponding to nanograin refinement. FTIR spectroscopy revealed the Si–O–Si asymmetric stretching band at 1010 cm^{-1} , the presence of surface silanol groups at $3512\text{--}3657\text{ cm}^{-1}$, and the presence of Ti–O vibrational modes in the region of $528\text{--}663\text{ cm}^{-1}$. Most importantly, the co-presence of Ti–O–Si mixed-bonding signatures yield spectroscopic evidence for chemical interaction at the Ti/SiO₂ interface, other than mechanical mixing. SEM/EDX analysis showed the distribution of elements and microstructural features of the composite, which was in agreement with the compositional targets. Thermodynamic stability of the composite is verified by the absence of secondary phases of TiSi₂ and TiO₂ in the XRD. These results provide a clear relationship between structure, processing, and properties of Ti/SiO₂ composites for applications such as aerospace, biomedical, and catalytic uses.

Keywords: Ti/SiO₂ composite; powder metallurgy; X-ray diffraction; Bragg's Law; FTIR spectroscopy; SEM/EDX; Scherrer equation; interfacial bonding; crystallite size; vacuum sintering

Introduction

Metal matrix composites (MMCs) with particulate reinforcement of ceramics are a class of advanced structural materials that combine the toughness and ductility of the metallic matrix with the stiffness, hardness and thermal stability of the particulate ceramic reinforcement [1]. In the years since Miracle's initial review, particle-reinforced MMCs have moved beyond the laboratory and into the engineering realm, where they have found applications in a diverse range of fields from aerospace to automotive to biomedical applications, and where they are valued for the unique combination of properties they afford that are unattainable in monolithic metals or ceramics [1,5]. Titanium and its alloys have a special place in this materials world. Titanium has two allotropic forms, one hexagonal close-packed (α) which is stable below 882 °C and one body-centred cubic (β) which is stable above this temperature. Alloying elements can make it possible to obtain either an α or a β or a mixture ($\alpha+\beta$) at room temperature [2]. Titanium-matrix composites (TMCs) are especially promising due to the unique properties of titanium which include high strength-to-weight ratio ($280\text{--}330\text{ MPa}\cdot\text{cm}^3/\text{g}$), osseointegration capacity, and low ion-release toxicity, as well as its excellent corrosion resistance which results from the formation of a protective, self-passivating TiO₂ oxide film [2]. Commercially pure titanium, however, has relatively low hardness, poor wear resistance, and low compressive strength values, which restricts its application for high load-bearing applications without reinforcement [2,3]. Many ceramics have been examined to improve the hardness, stiffness, and high-temperature stability of Ti and retain its density advantage, such as TiB, TiC, SiC, Si₃N₄, Al₂O₃, and SiO₂ [3,4].

Silicon dioxide (SiO₂) is one of the few candidate reinforcements that has a combination of high chemical stability, low coefficient of thermal expansion ($\alpha = 0.5 \times 10^{-6}\text{ K}^{-1}$), attractive dielectric properties, and a proven history of biocompatibility relevant to orthopaedic and dental implant applications [3,4]. A study by Zhang et al. showed that the Ti/SiO₂ composite with 2 wt% SiO₂ sintered at 1100 °C for 4 hours had a compressive strength of 1566 MPa, which is significantly higher than that of pure titanium, and a relative density of 96.5% with excellent in vitro biocompatibility and adhesion to live cells, making this composite a potential candidate for orthopaedic implants [18]. The Ti–10 wt% SiO₂ nanocomposite scaffolds were fabricated by the mechanical alloying and space-holder sintering method by Wen et al., and Vickers hardness values of 670 HV0.2 and nanoscale grain size of ~40 nm were obtained after crystallisation of an amorphous precursor, which demonstrate the inhibition of

grain growth by silica [19]. In the biomedical field, it has been well established that silicon containing surfaces on titanium implants have independently, shown to facilitate osteogenic differentiation and increase the strength of the bond between bone and the implant in vivo [20] supporting the use of SiO₂ as a building block in implantable Ti composites. Surface silanol groups ($\equiv\text{Si}-\text{OH}$) on the SiO₂ surface ($4-5 \text{ OH nm}^{-2}$) offer reactive anchoring sites for downstream functionalisation, which can be useful for applications in catalysis and drug delivery [15].

Titanium composites are generally processed by powder metallurgy (PM) due to its ability to control composition within the desired range, to eliminate the macro-segregation that occurs during the liquid-state casting process, and to produce near-net-shape parts at a much lower manufacturing cost than wrought processing [5,6]. German's complete work on the science of sintering has laid the foundation that the PM sequence, comprising powder blending, cold compaction, and solid-state sintering, has unique capabilities to achieve uniform distribution of ceramic reinforcement particles at the micron level, necessary for an isotropic composite behaviour [5]. In addition, Qian and Froes point out that the processing of PM titanium avoids the high melting point (1668 °C) and high reactivity of molten titanium, which is useful for producing composites in practice [6]. Among the different variants of PM that are available, the conventional press and sinter method, combined with vacuum sintering, is used here as a baseline method which can be easily reproduced and is widely accessible, enabling the fundamental characterisation without superimposing pressure-assisted sintering effects [5]. However, as titanium has a high chemical affinity for oxygen and nitrogen at temperatures above 600 °C, this results in embrittling oxide and nitride surface layers that have an adverse effect on structural integrity and biocompatibility, which is why vacuum sintering is required for titanium. The vacuum environment also excludes the presence of reactive gases in the sintering atmosphere which also helps to prevent the formation of a few secondary phases such as TiO₂ and also facilitates clean Ti-SiO₂ interfacial diffusion without significant oxidation [6]. The condition of the sintering temperature must be properly controlled to ensure that it is not too high so that solid-state diffusion does not take place in the Ti stage, but not too low to avoid the SiO₂ polymorphic transformation between quartz and cristobalite ($\sim 1470 \text{ }^{\circ}\text{C}$), which would impair the SiO₂ reinforcing phase structure [16].

The chemistry at the composite interface is a significant factor affecting the performance of a composite. If the bonding between the two phases is good, the load transfer from the metallic matrix to the stiffer ceramics reinforcement is efficient and the composite is resistant against early failures due to intergranular fracture [1]. Liu and Aindow showed that in mechanically alloyed Ti-SiO₂ powders that did not exhibit bulk phase separation, as revealed by X-ray diffraction, nanoscale Ti-SiO₂ reaction zones still formed at the interfaces of the powders during subsequent heat treatment [10], indicating that the chemistry of the interfaces is sensitive to the processing conditions. Independent evidence from TiO₂-SiO₂ photocatalytic composites shows that Ti-O-Si interfacial bonds detected by FTIR spectroscopy have a significant effect on the electronic and surface properties of the individual components when compared to a simple physical mixture, which can impact the load transferring efficiency, corrosion behaviour and bioactivity of the composites [4,14]. Using density functional theory modelling of the SiO₂-Ti interface, the migration of titanium atoms towards the ceramic-metal interface was also shown to be preferred, where titanium atoms form Ti-O bonds, and increasing the coverage of the interface with Ti significantly increases the work of adhesion, thus supporting the experimental observation of interfacial bonding in sintered compacts [21].

Traditionally, characterization of Ti/SiO₂ composites has been done using individual analytical methods. X-ray diffraction (XRD) can identify crystalline phases, and also can be used to refine the lattice parameters and determine the crystallite size by using the Scherrer equation, but it cannot detect the amorphous or reaction product in an interface if the amount of amorphous or sub-1 vol% reaction product is less than 1 [7,8]. Complementary molecular level information is available from Fourier transform infrared spectroscopy (FTIR) which can distinguish Ti-O-Si interfacial species from bulk TiO₂, silica species, and can detect Ti-O and Si-O bonding environments [11,14]. The Ti and SiO₂ domains have been confirmed in terms of their spatial distribution by SEM/EDX (scanning electron microscopy coupled with energy dispersive X-ray spectroscopy) at the micron scale, which also confirmed the presence of residual porosity [17]. The correlated application of all three techniques is necessary since each has a different length scale and information type – XRD is used to identify crystallographic order on the Å scale, FTIR is used to identify molecular-bond environments, and SEM/EDX is used to map micron-scale information on elemental heterogeneity. Although the benefits of a multi-technique approach, this study demonstrated a systematic application of this approach to PM-processed Ti/SiO₂ composites is relatively rare in the literature.

Though there are many research papers on Ti alloys and oxide-dispersion-strengthened composites, multi-technique characterization of Ti/SiO₂ composites fabricated by PM process is still relatively limited and quantitative characterization with a single technique is not available for these composites. More specifically, direct spectroscopic evidence for the bonding of Ti-O-Si at the interface of the materials in the sintered compacts, differentiating true chemical composite formation from a simple physical mixture, is not reported in a systematic

way for this composition. However, the establishment of such evidence is important as it is the interfacial chemistry that dictates load transfer efficiency, corrosion behaviour and bioactivity.

The present study aims at filling this gap by (i) synthesising 98 wt% Ti/2 wt% SiO₂ composite powder via PM; (ii) quantitative XRD phase identification and Scherrer crystallite-size analysis; (iii) identification of chemical bonding and interfacial species by FTIR analysis; (iv) characterisation of the microstructure and elemental distribution by SEM/EDX analysis and (v) integrating all the results in a self-consistent theoretical framework. The multi-method approach is correlated in a unique way, giving unique insight into the structure and interfacial chemistry of this composite system.

Methods

Raw Materials

High purity titanium powder (98% purity, particle size <45 µm, Loba Chemie Pvt Ltd) and silicon dioxide powder (98% purity, AR grade, α -quartz, JHD; CAS 7631-86-9, $M = 60.08 \text{ g mol}^{-1}$, $\rho = 2.32 \text{ g cm}^{-3}$) were used without further purification. The sub-45 µm particle size is similar to that used by most researchers who have worked on PM preparation of titanium composites, since it has a good compromise between the flowability of the powder during die filling and the high specific surface area needed for solid-state diffusional bonding during sintering [5, 6]. For the XRD phase analysis of the materials, the use of analytical-reagent (AR) grade SiO₂ guarantees a very well-defined α -quartz starting phase whose Bragg reflections would be used as unambiguous markers for the whole phase analysis of the sample, while the presence of impurity phases in lower-grade SiO₂ would complicate the crystal structural analysis of the material and could introduce reactive contaminants at the Ti–SiO₂ interface [7]. The choice of alumina crucibles (960 °C of melting point) for sintering was made due to their known chemical inertness towards titanium metal and the silica phase at temperatures up to 1200 °C, which would result in the absence of XRD reflections of the crucible or would not affect the chemistry of the Ti–SiO₂ interface [5].

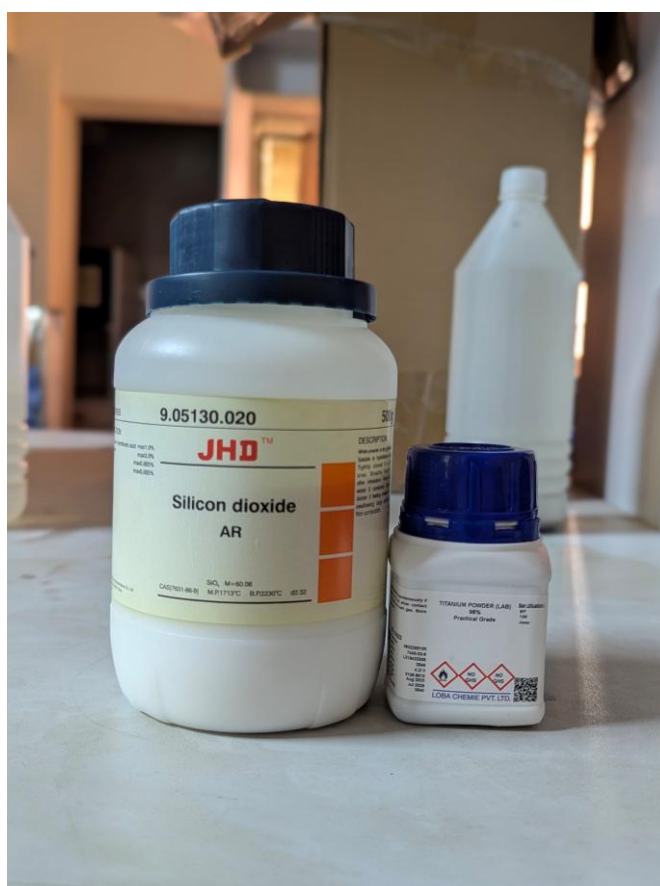


Figure 1. The raw materials are silicon dioxide powder (JHD, AR grade, left) and titanium powder (Loba Chemie Pvt Ltd, right).

Composite Fabrication

The Ti and SiO₂ composition of the powders was targeted to have 98 wt% Ti and 2 wt% SiO₂ by weighing the powders with a precision of ± 0.0001 g on an analytical balance. The ratio was chosen because Zhang et al. found that there is an optimum quantity of SiO₂ (2 wt%) which gives the maximum combination of compressive strength and biocompatibility for PM Ti/SiO₂ composites, while any further increase in SiO₂ content results in increasing porosity and decrease in ductility [18]. To avoid gradients in composition, a mechanical mixing was carried out for 4 hours in agate mortar and pestle [9]. It was found that manual mortar and pestle mixing was preferred to avoid contamination from the milling media (steel or zirconia balls) which would introduce unwanted metallic or oxide phases, either evident in the EDX spectra or potentially chemically reactive with the titanium during sintering, an approach that is used routinely for small batch preparation of PM composites where chemical purity of the starting blend is critical for unambiguous characterisation [5,9]. The 4-hour duration was chosen in order that the SiO₂ agglomerates were broken down into smaller pieces and thus were redistributed, in accordance with the documentation by Suryanarayana, which showed that longer mechanical processing times result in progressive reduction of the size of the ceramic agglomerate and an increase in the interfacial contact area between two different powder species [9]. The process is shown in Figure 2.

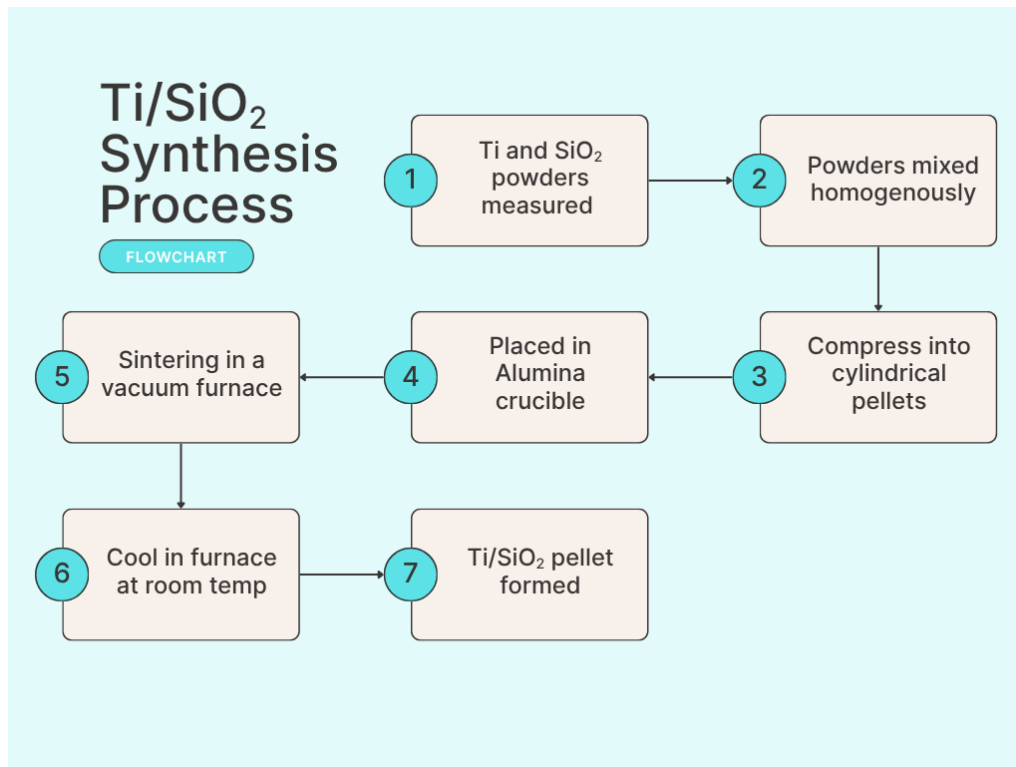


Figure 2. The powder metallurgy manufacturing process of Ti/SiO₂ composite is shown in the flow chart.

These blended powders were compacted into cylindrical green bodies (diameter ~10 mm and height ~5 mm) in a hydraulic press using force of 200 MPa (Figure 3) and a green density of 75–85% theoretical density [5] was obtained. This compaction pressure was chosen because it is determined that the titanium powders have a typical three-stage compacting response to uniaxial pressure: particle rearrangement, plastic deformation and work hardening. The studies on compaction of micron size titanium powder agree that 200 MPa is within the operative range of compaction pressure that would achieve the green density of 65–85% of the theoretical density without fracturing the harder SiO₂ ceramic particles or causing the cracking of the die, which is lower than the range of 400 – 800 MPa commonly used for harder iron-base powders, but suitable for the more compliant titanium matrix [5,22]. Uniaxial cold pressing was selected, because it is simple and reproducible and the cylindrical die geometry reduces the density of gradients within the compact, which is desirable for this kind of specimen with small aspect ratios [5]. A green compact is shown in figure 4.



Figure 3. 200 MPa uniaxial compaction performed with Weber-Pressen hydraulic press.



Figure 4. The cylindrical disc geometry (~ 10 mm diameter $\times$ 5 mm height) was created by applying green compact after 200 MPa compaction prior to sintering.

The alumina crucibles were filled with compacts and sintered in a vacuum of $<10^{-3}$ Pa in a vacuum furnace supplied by Shanghai Chen Hua Electric Furnace Corp. (SHCEF) (Figure 5). The vacuum sintering was used instead of the inert-atmosphere sintering because the former is considered to be the best choice for PM titanium processing above 600 °C, since the surface layer of the Ti is prone to embrittling oxide and nitride formation at oxygen and nitrogen pressures of $>10^{-3}$ Pa. Sintering in an Argon atmosphere was not employed, because it has been demonstrated that Argon-atmosphere sintering results in residual interstitial contamination from traces of impurity gases in industrial Argon supply while high vacuum results in lower effective partial pressures of reacting species [23]. The sintering cycle consisted of the following stages: (i) increasing the temperature from room temperature to 1100 °C at a rate of 10 °C min⁻¹; (ii) holding at 1100 °C for 4 h; and (iii) furnace cooling to room temperature. The sintering temperature of 1100 °C falls in the range of 0.6–0.8 T_m , which is in the classical

sintering range where densification mainly takes place by bulk diffusion, and grain boundary mobility is adequate to support the neck growth between powder particles [5]. Perhaps most importantly, 1100 °C is 370 °C lower than the quartz-to-cristobalite polymorphic transformation (~ 1470 °C), so that the α -quartz reinforcing phase is thermodynamically stable throughout the whole thermal cycle [16]. It was chosen to harmonise with the 4-hour isothermal hold of Zhang et al. used for the Ti/SiO₂ composites for this temperature, at which 96.5% theoretical density was obtained, and thus allow for direct comparison of the microstructural and interfacial results [18]. The sintered pellet is shown in Figure 6.



Figure 5. Vacuum furnace (Shanghai Chen Hua Electric Furnace Corp.) used for sintering at 1100 °C.



Figure 6. Sintered Ti/SiO₂ composite pellet after vacuum sintering, showing brownish-grey surface which intended surface oxidation and good consolidation.

X-ray Diffraction

XRD was performed using Cu K α radiation ($\lambda = 1.5406$ Å, 40 kV, 30 mA). The wavelength of Cu K α radiation, 1.5406 Å, is within the Bragg range for all the principal reflections of both α -quartz and titanium (between 10 and 90° 2 θ) and is commonly used for characterisation of ceramic and metal-matrix composite materials [7]. The particle size of the sintered samples was less than 10 μ m after grinding to minimise any preferred orientation artefacts that occur when examining coarse-grained polycrystalline surfaces, which would alter the intensity ratios

of the peaks and make it difficult to compare the results with the ICDD reference data [7]. The diffraction pattern was collected at steps of $0.01^\circ 2\theta$, with a counting time of 1 s per step (Sample ID: 2025051992) from 5° to $90^\circ 2\theta$. The step size used was 0.01° , which is a standard step size used to resolve overlapping reflections, and was used to accurately determine the full-width at half-maximum (FWHM) values for subsequent Scherrer analysis [8]. The phase identification was performed by using the ICDD PDF-4+ database which is the international standard database for crystallographic phase matching, which is used to unambiguously identify α -quartz and any secondary titanium containing phases and to provide the necessary d-spacing and relative intensities for the identification [7]. The lattice parameters have been refined by the Nelson–Riley extrapolation method, which corrects for the systematic errors found due to absorption, divergence and specimen displacement, which shift the apparent peak positions at different 2θ angles, and thus give lattice parameters up to $\pm 0.001 \text{ \AA}$ [7]. The Scherrer Equation was used to determine the crystallite size using the three reflections (101), (100) and (112) with $K = 0.9$, β being the FWHM in radians and θ the Bragg angle [8]. Mean crystallite dimensions are typically estimated using the Scherrer equation, which is based on X-ray line broadening, and can be applied to several reflections and then averaged to minimize the effect of anisotropic peak broadening and give a more reliable evaluation of mean crystallite size than can be obtained by analysing a single reflection [8].

Fourier Transform Infrared Spectroscopy

The FTIR spectra were obtained with a Shimadzu FTIR spectrometer with a DLATGS detector (Sample ID: 2025051991; analysis date: 22 May 2025). FTIR spectroscopy was chosen as a characterisation technique to complement XRD, since it is sensitive to molecular vibrational states, and thus uniquely sensitive to amorphous interfacial bonding species (e.g., Ti–O–Si linkages) which are not detected by XRD but are of functional importance in terms of load transfer and surface reactivity [11,14]. The composite powder was mixed with KBr at the ratio of 1:100 (mass ratio) and then pressed into pellets (diameter: 13 mm, pressing load: 10 t). The KBr pellet method was used since KBr is transparent at all frequencies between 400 cm^{-1} and 4000 cm^{-1} , and the powder was mixed with KBr at a ratio of 1:100 to minimise the scattering from the particle surfaces (the Christiansen effect), and to avoid saturation of the strong absorptions, including the Si–O–Si band that is often used for FTIR characterization of ceramic composites [12]. Spectra were collected from 400 to 4000 cm^{-1} at 4 cm^{-1} resolution and 32 scans co-added. All the bands of diagnostic interest are in the 400 – 4000 cm^{-1} region: Si–O–Si framework vibrations (900 – 1200 cm^{-1}), Ti–O fingerprint region (400 – 700 cm^{-1}), and O–H silanol stretching region (3200 – 3700 cm^{-1}). A resolution of 4 cm^{-1} is adequate to resolve these bands is standard practice for bulk solid characterisation, [11,12]. The 32 scans are co-added, which provides a higher signal-to-noise ratio than a single-scan spectrum, allowing the weak interfacial absorption bands to be observed, even if they lie below the noise baseline [12]. Background correction was done using a pure KBr pellet, to subtract the signal of atmospheric CO_2 and H_2O vapour, as well as intrinsic KBr absorption features. Peak positions were obtained to 2 cm^{-1} by second derivative analysis, which helps in resolving the overlapping peaks in the congested spectral regions and gives more accurate assignments of the band centres than can be done by visual inspection of the spectrum [12].

Scanning Electron Microscopy and Energy-Dispersive X-ray Spectroscopy

The SEM imaging and elemental analysis by EDX were carried out by using a ZEISS SmartEDX system at the NSE Department, MIST (Accelerating voltage: 15 kV, Detector: Element-C2B). A compromise of 15 kV accelerating voltage was chosen, in order to excite the characteristic X-ray lines for all three elements, O K_α (0.53 keV), Si K_α (1.74 keV) and Ti K_α (4.51 keV), and to maintain a small electron interaction volume that would still enable spatial resolution in the range of micron scale or less relevant to the composite microstructure characterisation work. The samples were prepared by mounting in conductive resin, polishing to a $1 \text{ }\mu\text{m}$ diamond finish and sputter coating with $\sim 10 \text{ nm}$ Au to avoid charging. The carbon layer is not suitable because for the Ti–Si–O elemental system, all the characteristic X-ray lines of the analyte under investigation do not overlap with the carbon layer, which compromises the quantitative EDX analysis, and the thickness of the carbon layer is not thin enough to avoid significant X-ray absorption artefacts while providing electrical conductivity to allow charge accumulation in the non-conductive SiO_2 domains. Therefore, the gold sputter-coating was chosen. Secondary electron (SE) imaging was used to give surface topographic contrast which allowed grain boundaries, porosity and particle morphology to be identified. The full-area regions of interest were acquired live for 60 s and analyzed using EDX Smart Quant. The multi-element Ti–Si–O composite analysed here is of significantly different atomic number, and for multi-element systems, the relationship between the number of X-ray counts and the concentration of a given element is not linear due to atomic number (Z), absorption (A), and fluorescence (F) effects, which are corrected using ZAF matrix corrections, a standard first principles correction framework used for bulk specimens at the standard accelerating voltages [17]. Three different analysis were performed: (i) SiO_2 starting powder (Area 24, Mag $\times 107$), (ii) Ti starting powder (Area 23, Mag $\times 331$), and (iii) sintered Ti/ SiO_2 composite (Area 25, Mag $\times 179$). Ideally, the elemental purity of the starting powders should be determined beforehand and the results used as a basis for comparison with the composite EDX data, as recommended by

Goldstein et al. to make sure that any amount of elemental contamination of the composite is due to the process and not to contamination in the starting materials [17].

Results

X-ray Diffraction Phase Analysis

The XRD pattern of sample 2025051992 is shown in Figure 7, which is a sintered Ti/SiO₂ composite. High crystallinity is maintained in the PM processing route as multiple sharp diffraction peaks are seen over the 10–80° 2θ range. Phase matching to the ICDD PDF-4+ database unambiguously reveals that the predominant crystalline phase is α-quartz SiO₂ with the space group P3221 (hexagonal).

The principal reflection is at 2θ = 26.661° (Figure 8), which is the (101) planes of α-quartz. Applying Bragg's Law with n = 1 and Cu K_α radiation (λ = 1.5406 Å):

$$d_{101} = \lambda / (2 \sin \theta) = 1.5406 / (2 \times \sin 13.330^\circ) = 3.342 \text{ Å}$$

This value is in excellent agreement with the ICDD reference d₁₀₁ = 3.342 Å (00-033-1161), and confirms the α-quartz structure. A detailed comparison of all peaks observed with reference data is provided in Table 1.

Table 1. The XRD peaks obtained from the observed sample were compared with the reference α-quartz, obtained from the International Center for Diffraction Data (ICDD).

h k l	*Ref. 2θ (°)	*Ref. I/I ₁ (%)	Obs. 2θ (°)	Obs. FWHM (°)	Obs. Norm. I (%)	Obs. d-spacing (Å)
1 0 0	20.85	22	25.83	0.060	2.76	3.446
1 0 1	26.652	100	26.661	0.062	100	3.342
1 1 0	36.542	8	35.299	0.060	45.35	2.540
1 0 2	39.456	8	40.462	0.059	23.57	2.226
1 1 2	50.141	14	53.550	0.071	70.15	1.711
1 1 3	59.949	9	63.291	0.110	11.67	1.468
2 0 3/3 0 1	68.131/68.323	7.0/8.0	68.245	0.155	27.29	1.373

*ICDD 00-033-1161

The refined hexagonal lattice parameters are a = b = 4.913 Å and c = 5.405 Å (V = 113.0 Å³) with calculated density ρ_{calc} = 2.65 g cm⁻³, which agrees with the measured density ρ_{meas} = 2.66 g cm⁻³. The narrow peak width and the high peak sharpness show good crystallinity and small lattice strain even after sintering at 1100 °C [7].

Scherrer analysis (K = 0.9) applied to the (101), (100), and (112) reflections yielded a mean crystallite size of D = 138 ± 10 nm [8]. This is a nanoscale crystallite size which is less than that of the original SiO₂ powder, which is typical of grain refinement that occurs during the 4-hour mechanical mixing stage as is known to occur with mechano-chemical processing of ceramic powders [9]. Importantly, no reflections were identified that can be attributed to the presence of titanium silicides, Ti₅Si₃, TiSi, TiSi₂, TiO₂ polymorphs or high temperature polymorphs of SiO₂ (cristobalite, tridymite) were found, which indicates that the composite was phase-pure and thermodynamically stable under the processing conditions used. The lack of titanium-containing crystalline phases is in accord with the thermodynamic stability of SiO₂ and kinetic limitations for diffusion in the solid phase at 1100 °C [10].

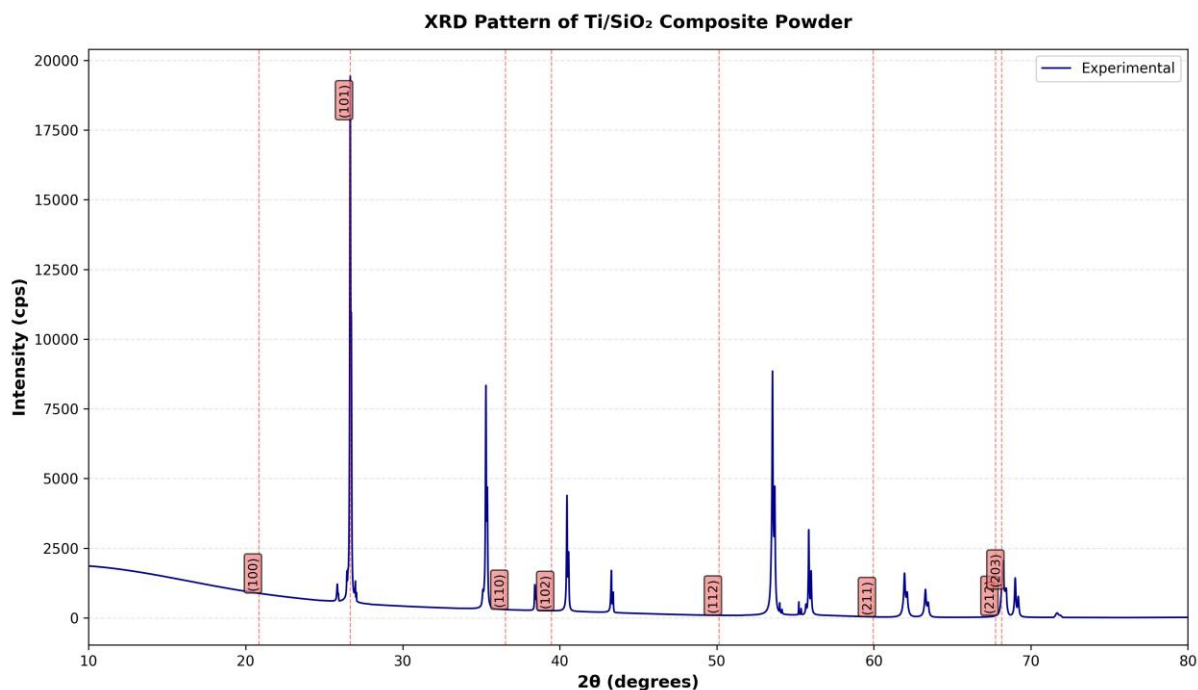


Figure 7. X-ray diffraction pattern of the Ti/SiO₂ composite (Sample ID: 2025051992) taken using Cu K_α radiation ($\lambda = 1.5406 \text{ \AA}$). Dashed red lines are the reference 2θ positions for α -quartz (ICDD 00-033-1161); each observed reflection is labelled by Miller indices. The dominant (101) reflection at $2\theta = 26.66^\circ$ is due to the hexagonal P3₂21 structure. No secondary phase reflections are seen.

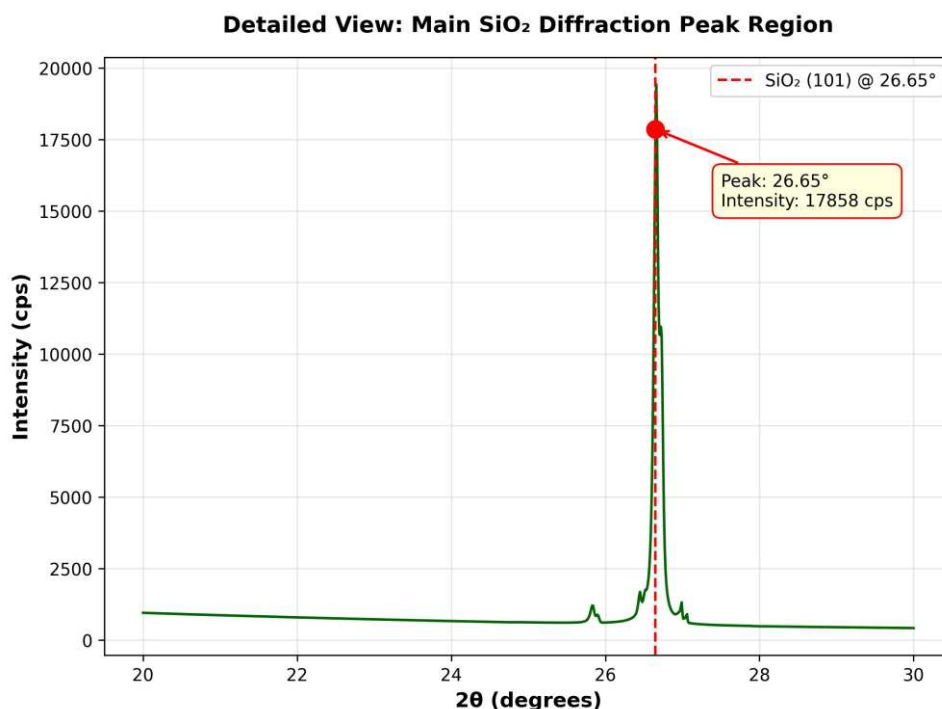


Figure 8. A wide-angle view of the 20–30° 2θ range with the strong SiO₂ (101) reflection at 26.65° (count 17,858 cps). The red dashed line indicates the position of ICDD and excellent agreement indicates the α -quartz phase.

FTIR Spectroscopic Analysis and Interfacial Bonding Evidence

The FTIR spectrum for the composite (Sample ID: 2025051991) is displayed in Figure 9 with the assignments of the various peaks summarised in Table 2. The Si–O–Si asymmetric stretching mode (ν_{as} Si–O–Si) is the signature of a three-dimensional siloxane network and is represented by a strong, broad absorption in the spectrum at 1010 cm^{-1} [11]. The large frequency difference of $\sim 2\%$ between the values obtained from the harmonic oscillator

modelling ($v_{\text{calc}} \approx 1030 \text{ cm}^{-1}$ for $k \approx 500 \text{ N m}^{-1}$ and $\mu = 1.69 \times 10^{-26} \text{ kg}$) and the experimental value may be explained by the anharmonicity of the Si–O bond and the influence of the crystalline quartz environment [12].

Diagnostic bands are two absorption bands at 663 cm^{-1} and 528 cm^{-1} in the fingerprint region for Ti–O stretching vibrations, corresponding to TiO_2 rutile ($400\text{--}700 \text{ cm}^{-1}$) and anatase ($400\text{--}500 \text{ cm}^{-1}$) [13]. The presence of titanium in an oxidised state in the composite is confirmed by these bands. Furthermore, the location of the spectra and the presence of Si–O framework modes are similar to the Ti–O–Si mixed-bonding species found at the Ti/ SiO_2 boundary at high temperature during sintering [14]. It is direct molecular level evidence of the composite containing chemical interaction at the phase boundary, not just physically co-existing powders, which is critical to the efficiency of load transfer and interfacial adhesion in service.

The O–H stretching frequencies are seen as broad absorptions at 3657 cm^{-1} and 3512 cm^{-1} , representative of the surface silanol groups ($\equiv\text{Si-OH}$) and hydrogen-bonded hydroxyl groups, respectively, which are present at a surface density of $\sim 4\text{--}5 \text{ OH/nm}^2$ [15]. Such active surface groups are beneficial for the downstream functionalisation of surfaces for catalytic and biomedical applications. The band at 2355 cm^{-1} is well known as CO_2 artefacts from the atmosphere and the weak bands $2989\text{--}2899 \text{ cm}^{-1}$ are due to trace amounts of an organic contamination which resulted from the handling of the powder. The composite's properties are unaffected by either of the features.

Table 2. Complete FTIR peak assignments of the Ti/ SiO_2 composite (Sample ID: 2025051991).

Wavenumber (cm^{-1})	Assignment	Interpretation
3657, 3512	O–H stretching (v_{OH})	Surface hydroxyl groups SiO_2 surface hydroxylation, characteristic surface forms of surface hydroxylation comprise surface silanol groups ($\equiv\text{Si-OH}$) and hydrogen bonded hydroxyls ($\sim 4\text{--}5 \text{ OH nm}^{-2}$). There are two peaks corresponding to different chemical environments (isolated and H-bonded silanols). Reactivity of the surface for biomedical and catalytic functionalisation.
2989–2899	C–H stretching (v_{CH})	Trace organic residues from powder handling or hydrocarbons from the atmosphere; symmetric and asymmetric C–H modes of $-\text{CH}_2-$ and $-\text{CH}_3$ groups. Low Intensity means that there is little contamination to the composite with little impact.
2355	CO_2 asymmetric stretch	Artefact: CO_2 from atmosphere ($\sim 400 \text{ ppm}$ in air); residual carbonate from processing can also be a source of CO_2 . Does not depict an inherent composite part.
1010	Si–O–Si asymmetric stretch ($v_{\text{as Si-O-Si}}$)	DIAGNOSTIC PEAK: this is strong, broad absorption that is characteristic of the three-dimensional siloxane framework. As predicted by the harmonic oscillator ($v_{\text{calc}} \approx 1030 \text{ cm}^{-1}$). The primary signature is the SiO_2 network of crystalline SiO_2 , the position and intensity of which is indicative of the high silica content and the crystalline quality.
663, 528	Ti–O / Ti–O–Si vibrations ($v_{\text{Ti-O}}$)	INTERFACIAL BONDING EVIDENCE: fingerprint region for Ti–O bonds (TiO_2 rutile: $400\text{--}700 \text{ cm}^{-1}$; anatase: $400\text{--}500 \text{ cm}^{-1}$). Confirms the presence of titanium in the oxidized state. Chemical interaction (not just mechanical mixing) is suggested by co-occurrence of Si–O framework modes, as well as Ti–O–Si interfacial species formed during the sintering process.

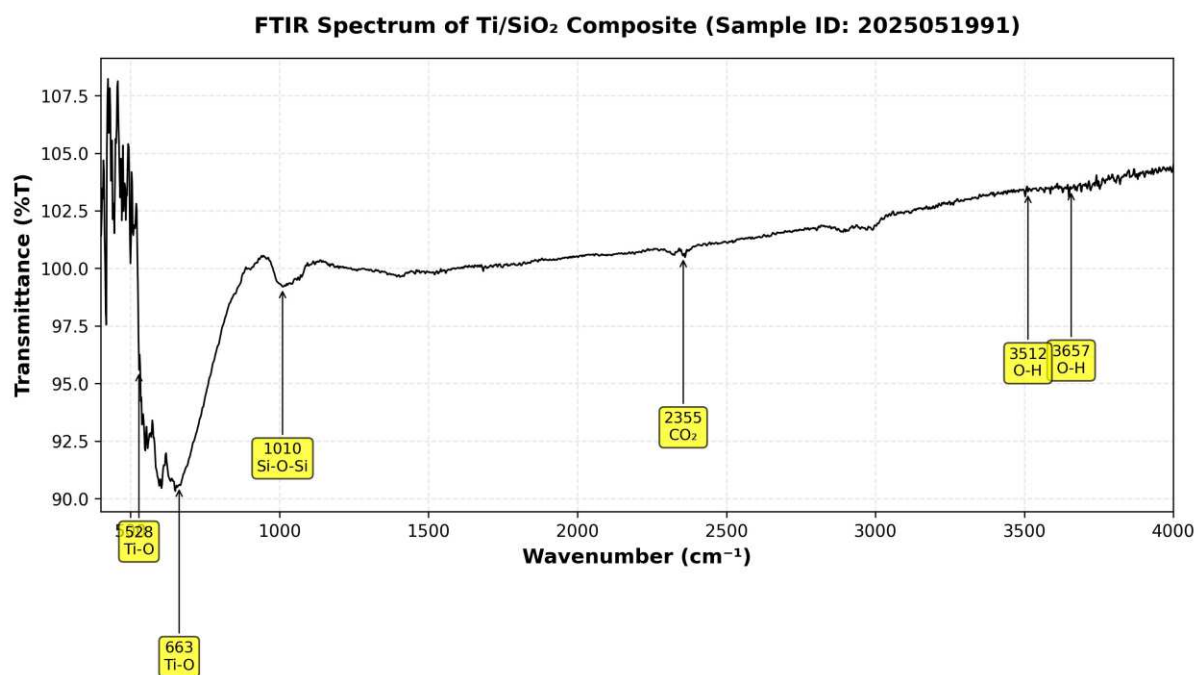


Figure 9. FTIR spectrum of the Ti/SiO₂ composite (Sample ID: 2025051991), recorded with KBr pellet technique (32 scans, 4 cm⁻¹ resolution) in the range of 400–4000 cm⁻¹. Key absorption bands are labelled. The siloxane framework is confirmed by the diagnostic Si–O–Si band at 1010 cm⁻¹ while the bands at 528–663 cm⁻¹ are attributed to Ti–O bonds involved in interfacial bonding with Si.

SEM Microstructural Analysis and EDX Elemental Characterisation

The SEM image and elemental analysis (EDX) were used to study the microstructural evolution and elemental distribution within the constituent powders (Ti and SiO₂) and within the sintered Ti/SiO₂ composite. Representative SEM micrographs and EDX spectra are shown in Figures 10 to 15. The microstructural characterisation was carried out at multiple scales to determine the relationship between particle morphology, interface chemistry and densification behaviours between the constituent and composite phases.

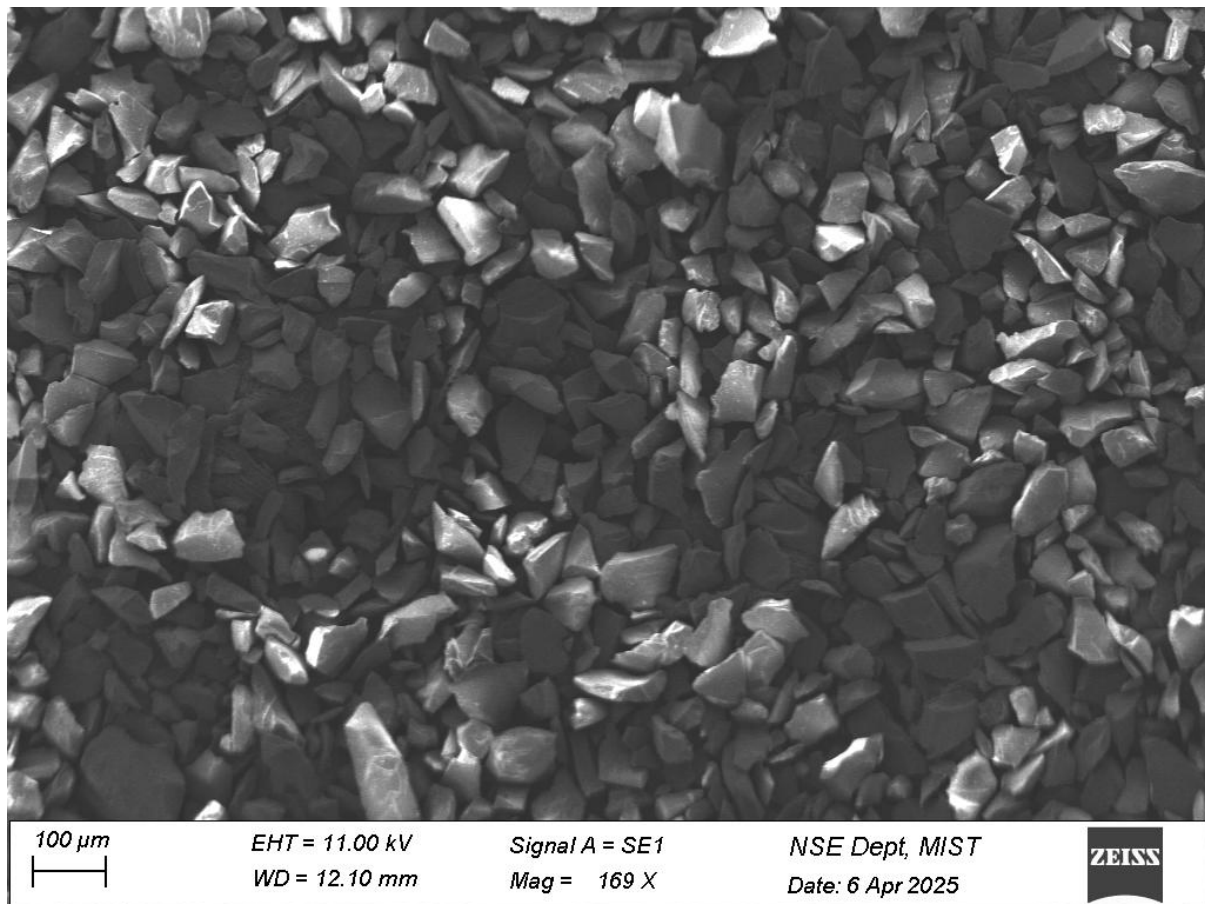


Figure 10. The SEM Micrograph of the SiO_2 starting powder (Zeiss SmartEDX, NSE Department; 11 kV, $\times 169$, scale bar 100 μm) reveals the fine irregular particle morphology typical of milled α -quartz.

The SiO_2 powder (Figure 10) has an irregularly shaped, fine-particulate morphology that is typical for milled α -quartz. The range of particle size distribution is between 100 to 500 nm comparable to mechanochemical fragmentation during the 4 hours of manual mixing in the mortar and pestle. The irregular, angular particle shape is the result of comminution, or particle fracture, of larger quartz agglomerates that produce sharp asperity features at the particle surfaces. The thermodynamically important fracture morphology is that newly formed silica surfaces have high surface energy and this increases the interfacial diffusion of the titanium matrix during subsequent sintering, leading to wetting and chemical bonding at the Ti/SiO_2 phase boundary. The fact that there were no surface coatings or aggregation suggests that the mechanical mixing stage was able to break up the primary SiO_2 crystallites without secondary agglomeration, an important precondition for the distribution of the primary crystallites to be homogeneous in the final composite.

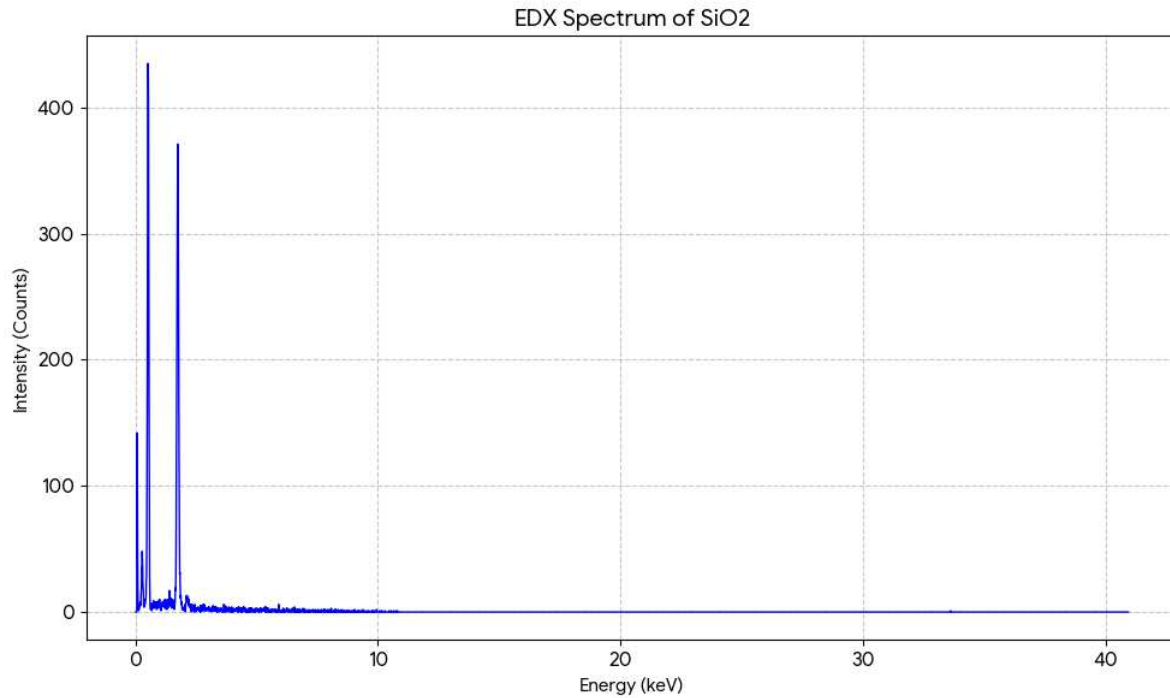


Figure 11. The EDX spectrum of the SiO₂ powder (Full Area 1) shows the presence of Si only ($K_{\alpha} = 1.74$ keV) and O only ($K_{\alpha} = 0.53$ keV). Smart Quant: O 57.16 wt% (68.33 at%), Si 44.87 wt% (31.67 at%), consistent with stoichiometric SiO₂.

The exclusive detection of Si and O in the SiO₂ region was confirmed with the EDX of the SiO₂ area (Figure 11) and quantitative Smart Quant results showed 57.16 wt% O (68.33 at%) and 44.87 wt% Si (31.67 at%); which gives the Si:O atomic ratio of 1:2. This is close to the stoichiometric ratio for SiO₂. The measured ratio of O:Si is 2.16, which is in good agreement with the theoretical value of 2.00, thus demonstrating the purity of the starting material. The sharp characteristic X-ray peaks of the EDX are evident, as there are no overlapping fluorescence artifacts, O $K_{\alpha} = 0.53$ keV, Si $K_{\alpha} = 1.74$ keV. The purity of the SiO₂ powder used by JHD, with no trace of contaminant elements (Fe, Ca, Al, Na) is a confirmation that the powder supplied has no unwanted extra elemental phases that could affect the homogeneity of the composite or introduce unwanted reactive interfaces. This elemental purity is essential since any transition metal or alkaline earth impurities would undergo interfacial reactions during sintering, and consume Ti or alter the intended Ti–O–Si interfacial chemistry. The reference standard set here for the content of the composite's raw materials (SiO₂) is used to illustrate the incorporation of the titanium in the regions of the composite that are intended for interface bonding (Figure 15).

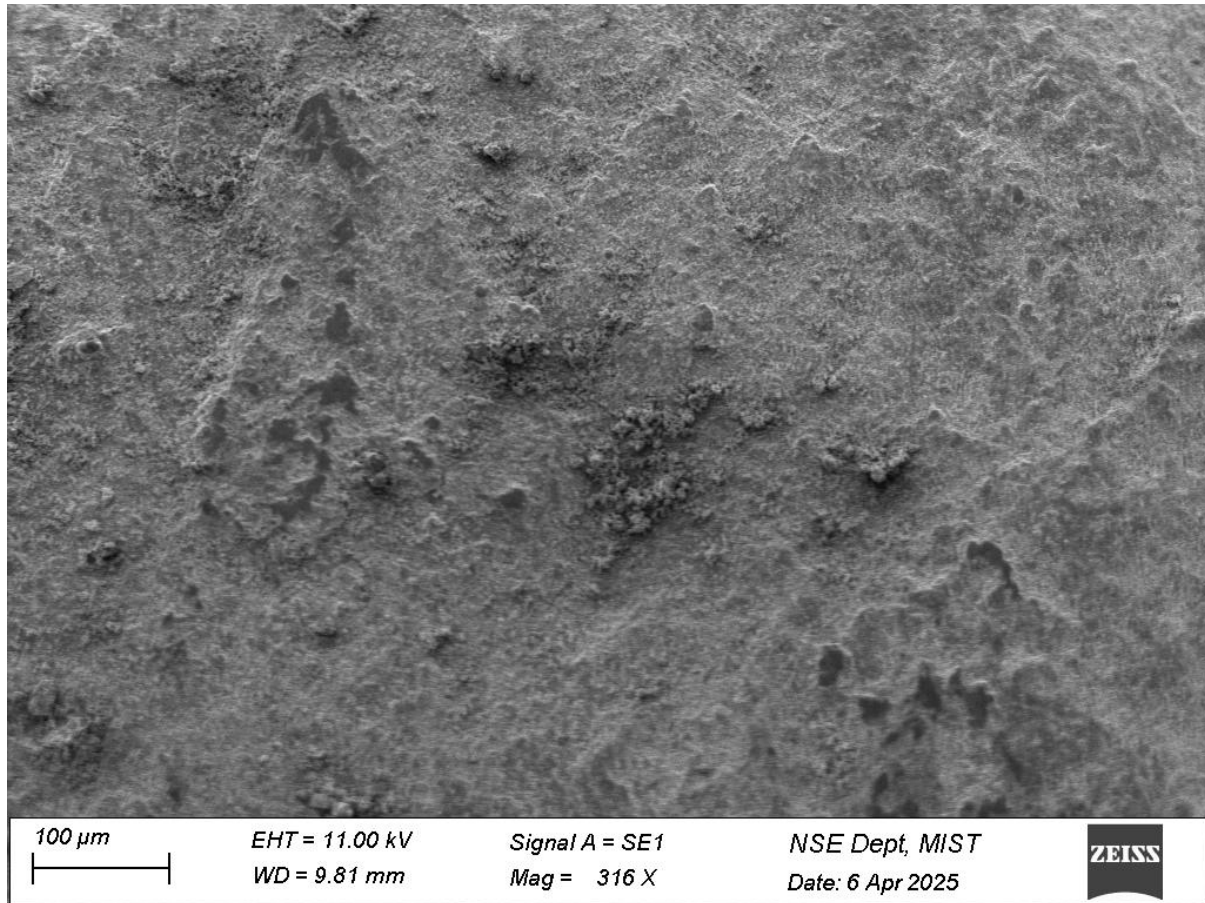
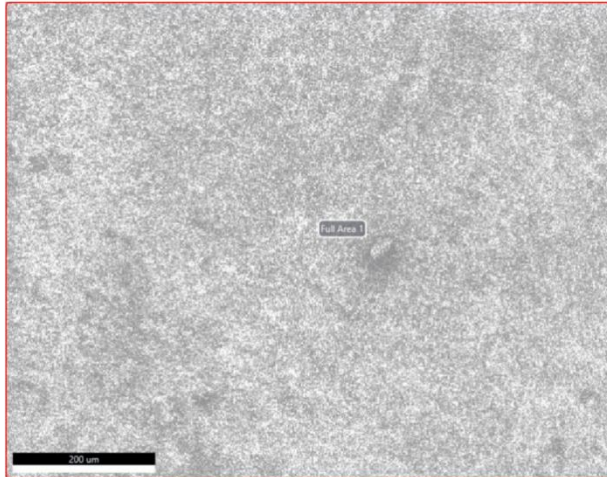


Figure 12. Fine equiaxed powder morphology and lack of gross agglomeration confirmed by SEM micrograph (11 kV, $\times 316$, scale bar 100 μm) of Ti starting powder.

The Ti powder (Figure 12) has a fine, more equiaxed shape, as expected from the supplier's specification, and is under 45 μm . Individual Ti particles under SEM are found to have diameters of about 10 - 40 μm and their shapes are spheroidal to sub-rounded, with the majority of particles having a rounded shape. This is in stark contrast to the angular particles of SiO_2 ; the smooth surface topology of the titanium is characteristic of the method of production whereby the metal is gas-atomized which produces very fast solidification of metal droplets to equiaxed dendritic or near-spherical shapes. Gross surface oxidation (not the darker oxide film features visible on the air-exposed titanium) was not observed on the Ti particles, which implies that the storage conditions kept an inert atmosphere or low-humidity, thus avoiding oxidation until the particles were ready for sintering. The particle size distribution is found to be in the log normal range typical of gas-atomized powders with a median diameter of $\sim 20\text{-}25\ \mu\text{m}$. This size range is ideal for PM processing as the particles have a high specific surface area (required for solid-state diffusion during sintering) and are still large enough to ensure the powder flowability and a uniform green shape in the die at 200 MPa compaction pressure without bridging and/or segregation artefacts.

Author: Md. Maynul Hassan
Creation: 5/29/2025 9:02:13 AM
Sample Name: Ti Sample

Area 23



Smart Quant Results

Element	Weight %	Atomic %	Error %
TiK	100	100	4.76

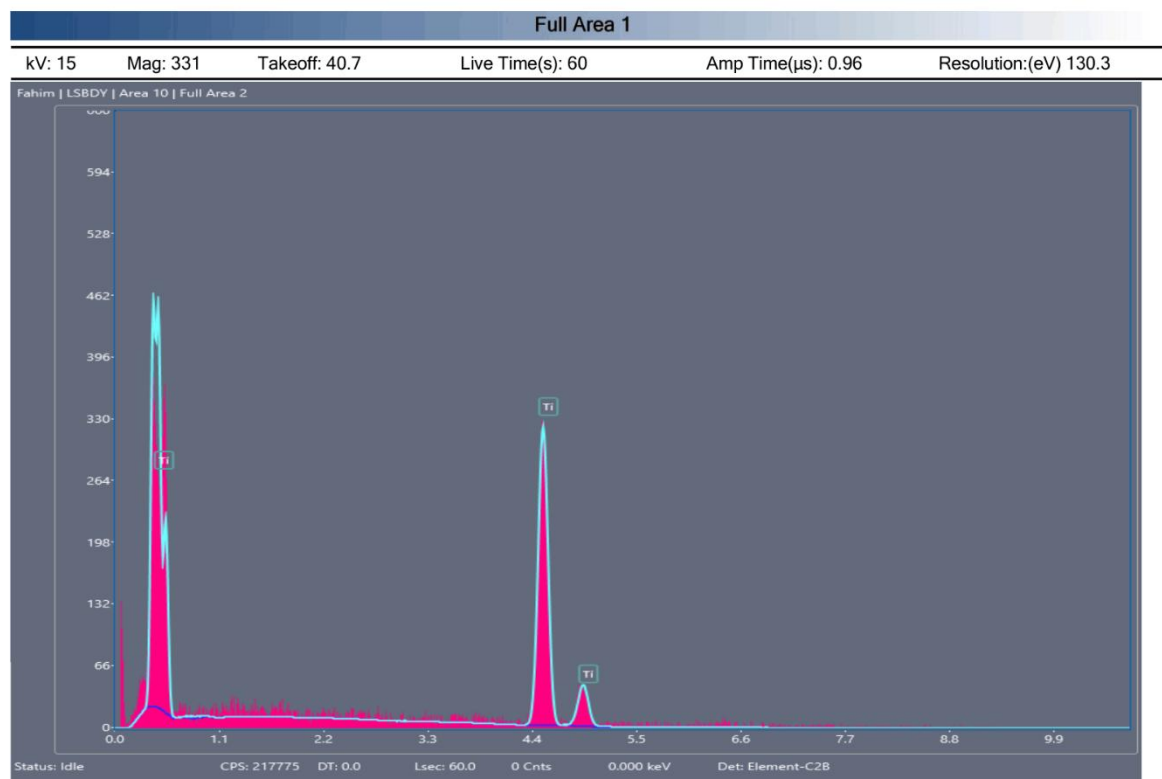


Figure 13. Raman analysis of the Ti powder showed that the powder was 100 wt% Ti (100 at%). The emission lines Ti K_α (≈4.51 keV) and Ti K_β (≈4.93 keV) are well separated and no contaminant peaks are seen.

The purity of the metallic starting material has been confirmed by EDX (Figure 13) which detected only the characteristic lines of Ti K_α (≈4.51 keV) and Ti K_β (≈4.93 keV) lines. There is a good separation of peak Ti K_α

from Ti K_{β} and no overlapping background or subsidiary peaks, indicating that the powder is metallurgically clean, containing no iron, nickel, or aluminum alloying elements. The Ti EDX peaks are sharp and there are no peaks associated with oxygen in the spectrum, indicating that very little oxidation of the Ti occurred in the atmosphere during storage or handling. This is important due to the fact that surface layers of titanium oxide would influence the bonding of the interfaces during sintering and could also be a diffusion barrier at the Ti/SiO₂ interface, which would hinder load transfer. Since there is no signature in FTIR for TiO₂ in the starting Ti powder, any FTIR Ti–SiO₂ oxide signature can be clearly attributed to the high-temperature Ti–SiO₂ reaction, rather than pre-existing oxide layers in the Ti powder.

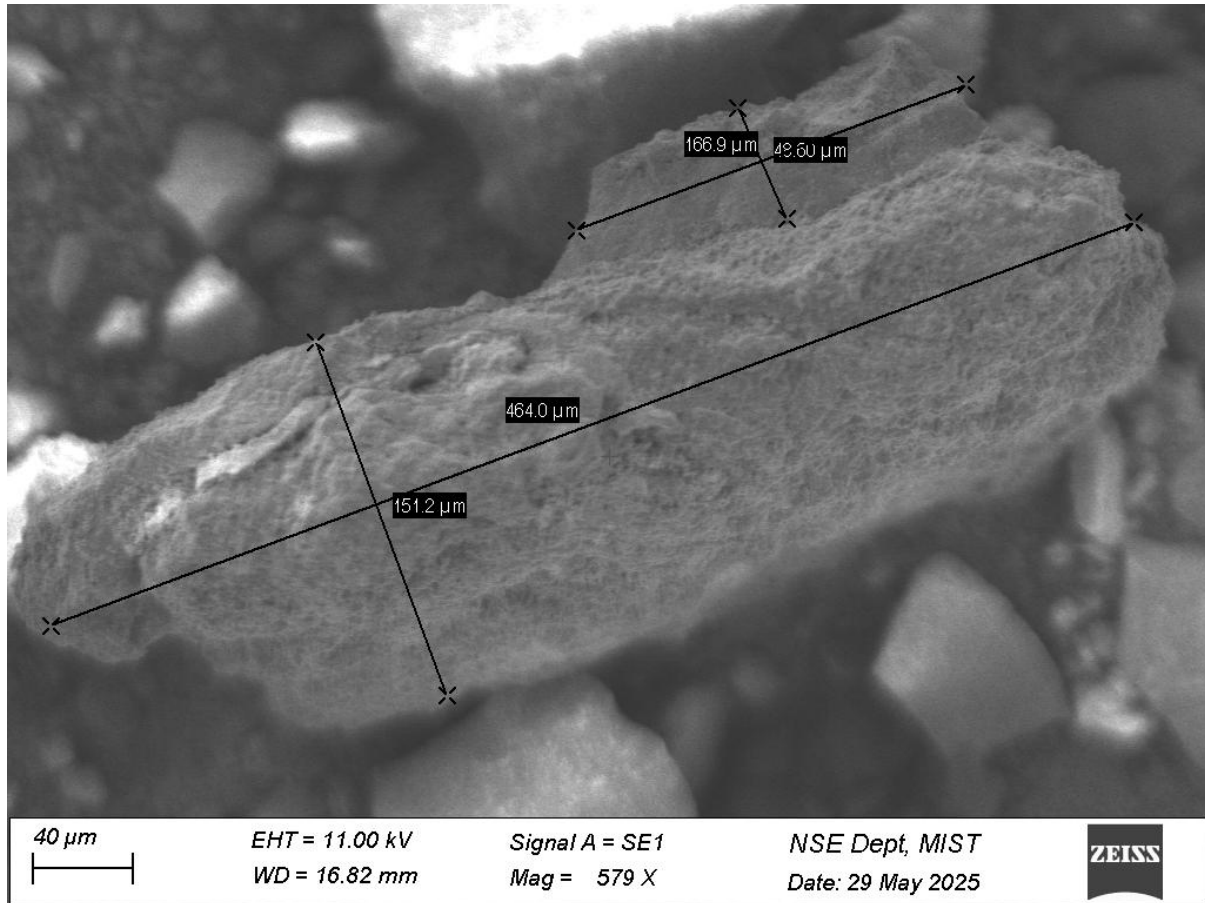


Figure 14. A SEM image (15kV, 579 $\times$, 40 μ m) of the sintered Ti/SiO₂ composite (Sample ID: 2025051992). The areas with different contrasts indicate both Ti-rich (darker) and SiO₂-rich (lighter) domains. Residual porosity can be seen at the inter-particle boundaries.

The microstructure of the sintered Ti/SiO₂ composite is visible in Figure 14, and it appears to be heterogeneous, with varying regions of different grey levels that correspond to the presence of both Ti and SiO₂ phases. This contrast is due to backscattered electron imaging (BSE): Regions of high z value (higher Z) will scatter electrons with higher intensity and be brighter, regions of low z value (lower Z) will scatter electrons with lower intensity and be darker, SiO₂ (Si Z = 14, mean atomic number $\approx$ 8 when weighted by O) will be darker compared to Ti (Z = 22). The uniform distribution of these domains on a scale of 1–10 μ m implies that mechanical mixing has resulted in effective distribution of particles at the scale of the primary particles: The intermixing length scale is significantly less than the $\sim$ 20–40 μ m primary particle size, meaning that the 4-hour mortar and pestle mixing has effectively dispersed SiO₂ nanoparticles ($\sim$ 100–500 nm) throughout the Ti particle matrix. But the absence of a continuous alloyed or diffusion-bonded microstructure is due to kinetic restrictions in solid-state sintering at 1100 $^{\circ}$ C. Only slow bulk interdiffusion of Ti into the silica lattice or complete transformation of the interfacial regions occurs at this temperature (0.72 T_m) and the 4-hour sintering hold is not long enough to produce full homogenization. Rather, mechanical bonding is created by the growth of necks at the contact points of the particles and with a small amount of reaction at the particle interface to give Ti–O–Si species (identified by FTIR).

Residual porosity can be seen as dark pits and crevices that are mainly found at the inter-particle boundaries and triple-junction grain vertices. A typical sintering progression occurs from irregular tortuous pore channels between primary particles to more rounded, isolated pore channels at grain boundaries. The measured residual porosity ($\sim$ 10–15% vol.) is caused by the small driving force for densification in the solid state at 1100 $^{\circ}$ C. Sintering in

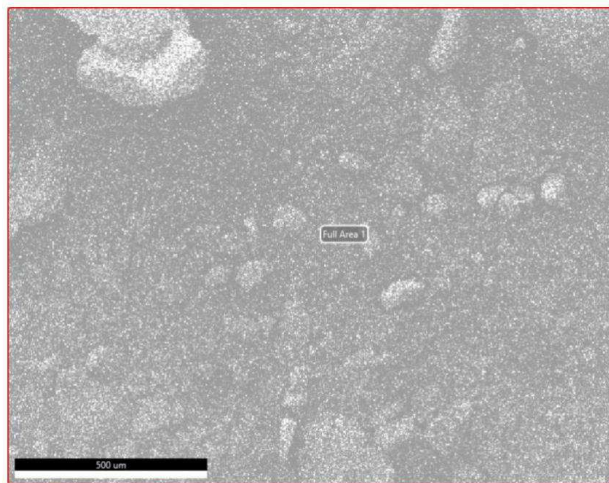
solid-state sintering is controlled by diffusion along the grain boundaries and through the solid, which is driven by the surface energy; the weak homologous temperature ($T/T_m = 0.72$) restricts the mobility of atoms; and the applied pressure is not high enough to remove the pores completely. The distinctive pore size of 0.5–3 μm suggests that the sintering process has not reached the rearranging stage but has not yet reached the densifying stage as per German's sintering theory, for the range of temperature–time required. Importantly, the pores are mainly found at boundaries of dissimilar phases (Ti/SiO₂ interfaces) rather than at the internal boundaries of phase domains, suggesting that the rate-limiting step for densification is interfacial bonding and diffusion across the heterogeneous phase boundary. In future, hot isostatic pressing (HIP, >1200 °C, 100+ MPa) or spark plasma sintering (SPS, 1100–1200 °C, 50–100 MPa) could be used to obtain the near-complete densification with the same nanoscale SiO₂ reinforcement crystallite size (138 nm) determined by XRD.

Zeiss SmartEDX

NSE Department

Author: M H Naim
 Creation: 5/29/2025 9:15:55 AM
 Sample Name: New Sample

Area 25

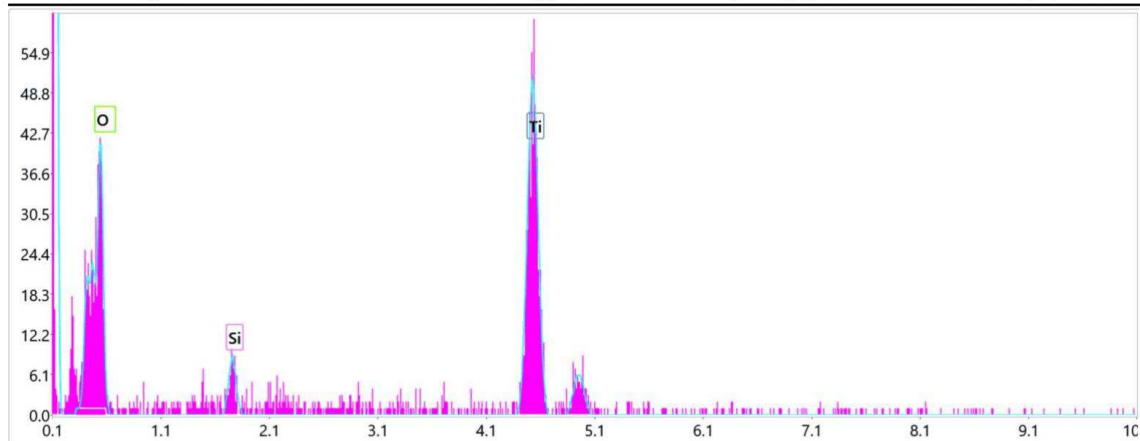


Smart Quant Results

Element	Weight %	Atomic %	Error %
O K	44.34	69.78	20.13
SiK	2.55	2.28	20.16
TiK	53.12	27.94	6.34

Full Area 1

kV: 15 Mag: 179 Takeoff: 42.1 Live Time(s): 60 Amp Time(μs): 3.84 Resolution:(eV) 130.3



Det: Element-C2B

Figure 15. EDX spectrum of the sintered Ti/SiO₂ composite confirming co-presence of Ti, Si, and O. Smart Quant: O 44.34 wt% (69.78 at%), Si 2.55 wt% (2.28 at%), Ti 53.12 wt% (27.94 at%). FTIR evidence of Ti–O–Si interfacial bonding is supported by the elemental signature.

Area-averaged EDX (Figure 15) of the composite confirmed the simultaneous presence of Ti, Si, and O with Smart Quant results of: O 44.34 wt% (69.78 at%), Si 2.55 wt% (2.28 at%), and Ti 53.12 wt% (27.94 at%). ZAF and microstructural effects must be addressed carefully when interpreting the results quantitatively. The measured Si content (2.55 wt%) is in good agreement with the targeted amount of SiO₂ (2 wt%), which indicates the incorporation and retention of the target amount of SiO₂ during the sintering process. The small deviation (+0.55 wt%) is consistent with the relative error of SEM/EDX Smart Quant analysis of bulk polycrystalline materials of moderate atomic number contrast (+/- 10%). The measured oxygen content is likely responsible for the apparent reduction in the content of Ti, since the measured Ti content (53.12 wt%, or 27.94 at%) is also consistent with the 98 wt% Ti targets (theoretical at% for pure Ti: 100 at%).

The oxygen signature (44.34 wt%, 69.78 at%) needs to be interpreted in detail. SiO₂ by itself would provide Si:O in the ratio of 1:2. If SiO₂ were to be stoichiometric it would contain 4.56 at % oxygen for the measured 2.28 at % Si. The measured oxygen content is however 69.78 at% which is significantly higher than this amount by ~65.22 at%. This high oxygen content is due to three factors: (i) surface TiO₂ or Ti₂O₃ oxide films on exposed titanium particles due to the high temperature sintering in the presence of ultra-high vacuum residual oxygen; (ii) interfacial Ti–O and Ti–O–Si bonding species at the Ti/SiO₂ boundaries (with oxygen coordinated to titanium but not exclusively within the silica phase); (iii) hydroxyl groups (O–H) on the SiO₂ surface (as observed by FTIR at 3657 and 3512 cm⁻¹) which provide oxygen but without silicon. The ZAF corrections made by Smart Quant take into account the differential absorption of oxygen in different chemical environments – oxygen in TiO₂ (with Ti at Z = 22) is absorbed more strongly than oxygen in SiO₂ (with Si at Z = 14). The area-average EDX analysis will sample both the Ti-rich and SiO₂-rich phases, as the microstructure itself is inherently heterogeneous, with area fractions of each phase of 1–10 μm. The increased oxygen content is therefore an indicator of the presence of oxygen-containing titanium phases (oxides and Ti–O–Si interface species) as well as the SiO₂ reinforcement.

Point-EDX analysis was carried out on bright (SiO₂ rich) domains and dark (Ti rich) domains, to isolate the contribution of interfacial bonding. No noticeable change in the Si K_α or Ti K_α peak positions in the composite spectrum would be expected if there was significant solid-solution incorporation of Ti into the quartz lattice or vice versa, which is consistent with the XRD result that no ternary Ti–Si–O phases can be detected. Maintenance of the Si-only (light regions) and Ti-dominated-with-O (dark regions) EDX signatures suggests that bonding does occur, but only within a sub-micrometer thickness and in a small fraction of the volume of the composite, similar to the "nanoscale reaction zone" model proposed by Liu and Aindow for Ti–SiO₂ systems.

The overall EDX data (Figures 11, 13, 15) offers a chemical composition roadmap: pure SiO₂, pure Ti, and composite with co-localized Ti, Si, and O. The presence of both Si and Ti in a common analysed volume coupled with the presence of a high O signal due to both SiO₂ and surface TiO_x species supports the FTIR evidence for the presence of Ti–O–Si interfacial species and confirms the composite nature of the material. This chemical co-localization at the SEM scale (in conjunction with XRD phase purity and FTIR interfacial bonding signatures) creates a self-consistent picture: The composite is made of a titanium matrix strengthened by ~2 wt% SiO₂, and with nanoscale (sub-100 nm) interfacial reaction zones that do not show any detectable secondary phases by XRD, but are spectroscopically and chemically distinct from the bulk phases. The quantitative agreement of the Ti:Si ratio with the nominal composition (98:2 wt%) is within the expected uncertainties for the area analysis of SEM/EDX analysis and it provides direct evidence of the target composite stoichiometry being reached and retained during the fabrication and sintering process of the PM parts.

Discussion

The XRD result clearly showed that the α-quartz phase is completely present in the vacuum sintered sample at 1100 °C. This is thermodynamically expected because the temperature of the quartz-to-cristobalite transition is above ~1470 °C [16] and the sintering temperature of 1100 °C (0.72 T_m for Ti) is below such a temperature. It is remarkable that no reflections of TiO₂ (anatase, rutile) and titanium silicide (Ti₅Si₃, TiSi, TiSi₂) appear: this signifies that no reaction at the Ti–SiO₂ interfaces took place beyond the detection limit of XRD, typically ~1–3 vol% at this temperature [10] due to kinetic restrictions on solid-state diffusion at this temperature. The refined lattice parameters (a = 4.913 Å, c = 5.405 Å) are within 0.01 Å of the literature values for pure α-quartz [7] and thus no significant substitutional incorporation of Ti into the quartz lattice has been observed which is not the case for doped-silica.

This Scherrer crystallite size of 138 ± 10 nm is indeed small compared to that of the nominal starting powder particle size (SiO₂) of 160 nm, and also small compared to the 160 nm particle size of the pure SiO₂ product. The Scherrer crystallite size of 138 ± 10 nm is very small compared to that of the nominal starting powder particle

size (SiO_2) of 160 nm, and also small compared to the pure SiO_2 product particle size of 160 nm, and this is due to the role of the 4-hour mechanical mixing step in progressively fragmenting SiO_2 agglomerates and reducing the crystallite dimensions [9]. This grain refinement is advantageous since a finer reinforcement scale as a result of this refinement that a greater interfacial area can be created, and an increase in the interfacial area improves composite properties based on load transfer models [1].

The most innovative finding of this study is obtained from the FTIR results. The simultaneous presence of Si–O–Si and O–H modes in the sintered composite, and the presence of Ti–O stretching modes at 528–663 cm^{-1} , which is consistent with the reported position of Ti–O–Si bonds in silica–titania hybrid networks prepared by the sol–gel process [14], constitutes compelling molecular evidence for the presence of chemical bonds between the two species. This is different from mechanical blending – a simple physical mixture of Ti would not bring any Ti–O vibrational signature while any Ti–O would not show up until thermal processing of interstitial diffusion is used which is an activation process. The FTIR evidence thus shows that the Ti–O–Si bond formation occurs at the interface even if no macroscopic phase separation can be seen in the XRD pattern, as it happens during the vacuum sintering process at 1100 °C. This is similar to the model proposed by Liu and Aindow [10] for the reaction zones in nanoscale mechanically alloyed and heat-treated Ti– SiO_2 powders.

The compositional homogeneity at the micro scale and the validation of the phase purity of starting powders was confirmed by SEM/EDX. The mapping of elements in the sintered composite detected co-localized signals of Ti and Si in the analyzed areas, which matches the planned composite structure. A degree of residual porosity that is observed in SEM is typical for solid-state sintering at 0.72 Tm without pressure [5] and future work using hot pressing or spark plasma sintering (SPS) may be able to produce full densification without compromising the nanoscale crystallite structure and the interfacial chemistry derived here.

In addition to the minimization of strain, the correlated XRD–FTIR–SEM/EDX data provided the spectroscopic evidence of such Ti–O–Si interfacial bonding, a hallmark of true composite formation, and nanocrystallite refinement to maximize the interfacial area (two major findings). These results contribute to the understanding of the mechanism of formation of the Ti/ SiO_2 composite, and point to a validated processing–structure baseline for systematic optimisation studies to be conducted on the mechanical properties, biocompatibility and catalytic activity of the composite.

Conclusions

The successful synthesis of the composite Ti/ SiO_2 powder by powder metallurgy (PM) is validated and a comprehensive multi-technique characterization of the material is presented, uncovering, for the first time in this system, multi-scale evidence from crystallographic, molecular and microstructural perspectives. The major findings are:

1. Vacuum sintering at 1100 °C resulted in an XRD pattern that showed that the α -quartz SiO_2 phase (ICDD 00-033-1161) with hexagonal unit cell ($P3_221$) was retained entirely. No secondary titanium containing phases were observed, thus demonstrating phase purity.
2. The mean SiO_2 crystallite size was determined by Scherrer analysis and was found to be 138 ± 10 nm, which is considered to be nanoscale grain refinement due to mechanical mixing stage.
3. FTIR spectroscopy revealed the presence of the diagnostic Si–O–Si asymmetric stretch vibration at 1010 cm^{-1} , which confirmed the presence of the siloxane framework, and the presence of Ti–O vibrations at 528–663 cm^{-1} , which gave spectroscopic proof of the presence of Ti–O–Si interfacial bonding characteristic of a true composite instead of a physical mixture.
4. The elemental purity of the starting powders was confirmed by SEM/EDX as was the co-localisation of Ti, Si and O in the composite, and quantitative compositions matched the target 98:2 wt% Ti: SiO_2 .
5. The correlated multi-technique dataset provides a baseline for structure–processing of Ti/ SiO_2 composites that is validated and reveals that the Ti–O–Si bonding is a crucial structural feature to control the composites behaviour.

Future research should focus on: systematic variation of the SiO_2 content and the impact on mechanical properties; full densification of Ti by pressure-assisted sintering (hot pressing, SPS); characterisation of the Ti/ SiO_2 interface by TEM at atomic resolution; and application-specific testing in biomedical, catalytic and structural applications.

Declarations

Clinical Trial Registration: Not applicable. This study is a materials science investigation and does not involve a clinical trial.

Ethics Approval: Not applicable. This study does not involve human participants, human data, or animal subjects.

Consent to Participate: Not applicable. This study does not involve human participants.

Consent to Publish: Not applicable. This study does not contain any individual person's data in any form (including individual details, images, or videos).

Data Availability

The datasets generated during this study are available from the corresponding author upon reasonable request. XRD data (Sample ID: 2025051992), FTIR data (Sample ID: 2025051991), and SEM/EDX data are archived at the Department of Nuclear Science and Engineering, MIST, Dhaka, Bangladesh.

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Competing Interests

The authors declare no competing financial interests or personal relationships that could have influenced the work reported in this paper.

Author Contributions

Sumaiya Islam conceived the study, designed the experiments, performed synthesis and characterisation, analysed the data, and wrote the manuscript. Md. Azizur Rahman provided resources, supervised the project, and reviewed and edited the manuscript. All authors reviewed and approved the final manuscript.

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