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LOCAL MICROCHEMICAL HETEROGENEITY AND IRON LOCALIZATION IN SELECTED PARTICLES OF TITANIUM SLAGS OF DIFFERENT ORIGIN: A SEM-EDS/WDS STUDY

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ABSTRACT

Titanium slags are chemically and microstructurally heterogeneous intermediates whose process behavior cannot be inferred from a single composition value alone. This study compares local iron localization and impurity-related microchemical architecture in selected particles of two titanium slags of different origin: a slag prepared from a 60/40 blend of Sierra Leone and Satpayev ilmenite feedstocks and an industrial TiZir slag. Powder particles were examined on a JEOL JXA-8230 electron probe microanalyzer at 20 kV using backscattered-electron imaging, SEM-EDS point/area analysis and mapping, and local WDS mapping. The dataset comprised multiscale images, six EDS mapping sets, 32 point analyses, two area analyses, and one WDS map. The novelty claimed here is not the already established existence of microchemical heterogeneity, but the side-by-side classification, under a common analytical workflow, of matrix-associated Fe, Fe-enriched associations, a documented Fe–S occurrence, and contrasting silicate architectures in the two available datasets. The values 6.04 and 6.36 wt.% Fe refer only to two selected mapped fields; they are neither bulk compositions nor statistically representative sample means. The 60/40 slag contained a Ti–Fe–Mn oxide matrix, Fe–Mn-enriched regions, a documented Fe–S-bearing point, silicate inclusions, and a rare multielement Si–Al–Ca–Ti–Zr–Nb–REE–Th domain. The TiZir slag showed more sharply separated Fe-rich regions, including one documented Fe–Cr–Mn-rich point, and elongated aluminosilicate inclusions depleted in Ti. Within the purposive dataset, the Fe–S signature was documented at one targeted point on one S1 particle, whereas the diagnostic Fe–Cr–Mn signature was documented at one targeted point on one S2 particle; these counts are occurrence records, not population frequencies. Because complete matched records of smelting history, degree of reduction, cooling conditions, and certified bulk composition are unavailable, the observed S1–S2 differences are interpreted descriptively and are not attributed causally to slag origin or production history. The data do not by themselves establish leaching kinetics or crystalline phase identity. Further work should combine polished-section quantitative EPMA/WDS, XRD/EBSD, statistically designed sampling, and controlled iron-removal tests.



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INTRODUCTION

Titanium feedstocks enter chloride, sulfate, pigment, and metal-production routes in several mineralogical and technological forms. Ilmenite is abundant but contains structurally bound iron, whereas natural rutile is richer in TiO_2 and generally requires less upgrading. Reductive smelting converts ilmenite concentrates into a Ti-rich slag and a metallic iron-bearing product, thereby concentrating titanium in an oxide phase. The resulting slag is often treated as a compositionally simple high- TiO_2 feed in process calculations, but its actual solidified structure is heterogeneous and reflects feed mineralogy, reduction intensity, liquidus relations, impurity partitioning, cooling history, and post-solidification transformations (Pistorius & Coetzee, 2003; Guéguin & Cardarelli, 2007; Gupta et al., 2021).

This heterogeneity is important because iron is not necessarily present in a single chemical or microstructural state. It can remain distributed through the Ti-rich oxide matrix, become concentrated in discrete Fe–Mn- or Fe–Cr-bearing domains, occur in residual metallic or sulfide-type regions, or be associated with multiphase boundaries. Silicon, aluminum, calcium, magnesium, manganese, chromium, and alkalis may segregate into distinct oxide or silicate domains and alter both comminution and subsequent hydrometallurgical response. Recent investigations confirm that non-titanium constituents in high-titania slags are partitioned non-uniformly and that local phase chemistry can differ substantially from the bulk composition (Han et al., 2025; Jing et al., 2026).

The technological consequence discussed in the literature is that slags with different iron-bearing microstructures may respond differently during oxidation, reductive transformation, magnetic separation, acid leaching, or preparation of synthetic-rutile and chlorination feedstocks. In the present dataset, however, no slag-wide Fe comparison is available: the Fe values compared later in the article are local values from selected mapped fields, not bulk compositions. Reaction accessibility can depend on particle-scale and microdomain-scale factors such as exposed surface area, continuity of Fe-rich regions, contact with the Ti-rich matrix, intergranular silicates, porosity, and crack development. Work on upgraded ilmenite and synthetic-rutile routes has repeatedly shown that pretreatment history and mineralogical properties affect the efficiency and selectivity of iron removal (Spencer et al., 2022; Daba et al., 2022; Kurniawan et al., 2022; Jing et al., 2023).

Kazakhstan has a technologically significant ilmenite resource base that includes the Satpayev deposit and other complex titaniferous ores. Recent studies have addressed beneficiation, electric smelting, fluxing, chromium removal, carbothermal transformation, and hydrothermal refining of Kazakhstan ilmenite concentrates (Myrzakulov et al., 2024a, 2024b; Kenzhaliyev et al., 2024; Akhmetova et al., 2024, 2025a, 2025b; Motovilov et al., 2026). These studies strengthen the case for a process-mineralogical description of Ti-rich products: the suitability of a slag for further purification or chlorination depends on how iron and other impurities are structurally and spatially distributed after smelting.

A previous study by Sarsembekov et al. (2026) focused on niobium speciation in natural ilmenite and synthetic Fe–Ti–Nb–O model systems. The present work addresses a separate, previously unpublished analytical dataset comprising two titanium slags and does not reuse the XRD, thermal-analysis, or model-system results of that publication as new data. The novelty claimed in the present study is not the general observation that titanium slags are heterogeneous or that Fe can be non-uniformly distributed—both are already established in the literature. Rather, the contribution is a side-by-side local classification of matrix-associated Fe, Fe-enriched associations, a documented Fe–S occurrence, and silicate architecture in two different-origin slags examined under the same SEM–EDS/WDS framework, with explicit separation of observed chemistry from crystallographic phase identity. This comparison is descriptive and does not establish that the observed differences were caused by slag provenance or production conditions.

The objective of this study was therefore to compare, at the level of the selected particles and analyzed microdomains, the Fe–Ti–O matrix, Fe-enriched, silicate, and complex impurity regions in a 60/40 Sierra Leone–Satpayev titanium slag and an industrial TiZir slag. Particular attention was paid to the distinction between observed local chemistry and inferred phase identity. The work is intended to provide a local process-mineralogical baseline for future controlled studies of reductive phase transformation and selective iron removal; it is not intended as a statistically representative characterization of either whole slag lot or as evidence of a leaching mechanism.

LITERATURE REVIEW

The mineralogy of high-titania slags has historically been described in terms of Ti-rich oxide solid solutions accompanied by rutile and minor silicate or glassy constituents. Classical studies demonstrated that the solidified product is governed by the FeO–TiO₂-related phase field and by the distribution of Mg, Mn, Al, Si, and other oxides (Toromanoff & Habashi, 1984; Pistorius & Coetzee, 2003; Guéguin & Cardarelli, 2007). Integrated XRD and electron-probe investigations further showed that phase-scale and grain-scale observations are needed to connect bulk chemistry with the actual carriers of impurities (Samal et al., 2009). Silicate phases are particularly important at grain boundaries and may influence cracking and comminution behavior (Pistorius & Kotzé, 2009).

Contemporary work extends this framework toward process design. Gupta et al. (2021) reproduced titania-slag formation in an induction furnace and demonstrated the sensitivity of slag constitution to smelting conditions. Vorobkalo et al. (2023) investigated production of titanium-rich slag and a Ti-bearing ferroalloy, highlighting the linkage between reduction, metal–slag separation, and final slag chemistry. Han et al. (2025) directly examined the distribution of non-titanium components in titanium-rich slag and emphasized that impurity behavior must be interpreted through spatially resolved analysis. At the slag–metal interface, Jing et al. (2026) showed pronounced elemental redistribution, illustrating why residual iron-rich regions can occur despite efficient bulk metal separation.

The Fe–Ti–O subsystem is itself compositionally complex. Updated equilibrium measurements for Fe₂O₃–TiO₂ quantify the stability fields and solid solubility of hematite, pseudobrookite, and rutile over a broad temperature range (Jiao et al., 2025). In practical smelting systems, oxygen potential and reduction degree determine whether iron remains in the oxide assemblage or enters the metallic phase. Alternative reduction routes—including thermal-plasma processing—also produce highly heterogeneous transformed products that require microstructural characterization before downstream separation can be evaluated (El Khalloufi et al., 2024).

For Kazakhstan feedstocks, the need to connect mineralogy and process conditions is particularly evident. Electric-smelting and fluxing studies of Satpayev concentrates have established that additives, temperature, and reduction conditions modify the Ti-bearing slag and the distribution of iron-bearing products (Myrzakulov et al., 2024a, 2024b). Chromium-bearing ilmenite concentrates pose an additional difficulty because Cr can persist in refractory spinel-type constituents and affect the quality of titanium products (Kenzhaliyev et al., 2024). Recent beneficiation work on Satpayev ore likewise confirms a complex association of ilmenite with zircon-bearing and silicate components (Motovilov et al., 2026).

The second technological context is downstream purification. The quality of synthetic rutile depends on initial ilmenite properties and on the ability to transform iron into a removable form while retaining titanium in the solid residue (Spencer et al., 2022). Reductive and acid-leaching studies show that accessible Fe-bearing regions can be removed efficiently, but selectivity depends on pretreatment, particle structure, solution chemistry, and the stability of Ti-

bearing phases (Daba et al., 2022; Kurniawan et al., 2022). Sequential leaching of titanium slag can raise TiO_2 grade, but the response is strongly conditioned by the distribution and chemistry of non-titanium constituents (Jing et al., 2023).

Taken together, the cited literature establishes two points that constrain the present contribution. First, microchemical heterogeneity and non-uniform partitioning of Fe and other impurities in titanium slags are already known and are therefore not claimed here as a new phenomenon. Second, most cited studies focus on a single slag system, phase equilibria, slag-metal interfaces, or downstream upgrading rather than a side-by-side local comparison performed under one SEM-EDS/WDS workflow. The specific gap addressed here is thus limited to comparative classification of matrix-associated Fe, discrete Fe-rich associations, an Fe-S occurrence, and contrasting silicate architectures in the two available datasets. Because the samples are not matched by feed, smelting history, degree of reduction, cooling history, or certified bulk composition, the present work does not attribute the observed local differences causally to provenance or production route. Table 1 summarizes both the relevance and the limitations of recent studies relative to this gap.

Table 1. Recent studies relevant to the interpretation of titanium-slag microstructure

Study	Object / approach	Relevance / limitation relative to the present work
Gupta et al. (2021)	Synthetic titania slag produced by induction smelting	Demonstrates sensitivity of slag constitution to smelting conditions; it treats a single synthetic slag rather than a matched cross-slag comparison of local Fe localization.
Spencer et al. (2022)	Ilmenite properties vs. synthetic-rutile quality	Links feed mineralogy to downstream upgrading response; it does not resolve Fe-bearing microdomains in different titanium slags under one common imaging protocol.
Vorobkalo et al. (2023)	Titanium-rich slag and Ti-bearing ferroalloy	Relates reduction and metal-slag separation to product chemistry; the emphasis is process performance rather than local comparative Fe architecture.
Jing et al. (2023)	Sequential leaching of titanium slag	Shows that impurity distribution affects sequential leaching; it measures process response but does not compare local Fe carriers across slags of different provenance.
Myrzakulov et al. (2024a,b)	Satpayev ilmenite electric smelting and fluxing	Provides Satpayev-specific smelting and fluxing context and, importantly, shows why process history is a confounder when S1 is compared with an unrelated industrial slag.
Han et al. (2025)	Distribution of non-titanium components in Ti-rich slag	Directly demonstrates non-uniform distribution of non-titanium components in Ti-rich slag; therefore heterogeneity itself is not a novel claim of the present work.

End of Table 1

Study	Object / approach	Relevance / limitation relative to the present work
Jiao et al. (2025)	Fe ₂ O ₃ -TiO ₂ phase equilibrium	Constrains possible Fe-Ti-O phase fields; equilibrium information cannot by itself identify crystallographic phases in rough-powder EDS measurements.
Jing et al. (2026)	Slag-iron interface and elemental distribution	Shows microscale redistribution at the slag-iron interface; it supports local partitioning but does not provide statistical prevalence of Fe-bearing microdomains across slag populations.
<i>Note – compiled by the authors from the cited literature.</i>		

MATERIALS AND METHODS

Materials and sample provenance

Two powdered titanium slags were investigated. Sample S1 was a titanium slag prepared from a feed blend consisting of 60% Sierra Leone ilmenite material and 40% ilmenite from the Satpayev deposit in East Kazakhstan. Sample S2 was an industrial titanium slag produced by TiZir Titanium, Norway. For the present comparison, the available project archive did not contain a matched set of records for smelting temperature and heat history, degree of reduction, cooling rate or cooling schedule, and certified bulk chemical composition for S1 and S2. These variables may materially affect phase development and the partitioning of Fe, Mn, Cr, Si, and other components. Accordingly, all cross-sample differences reported below are limited to the observed local SEM-EDS/WDS features and cannot be assigned to slag origin alone. The mapped-field compositions are not used as substitutes for whole-sample assays or material-balance data.

The samples were available as crushed powder. Individual particles were mounted on brass specimen holders using conductive double-sided carbon tape. The use of powder mounts enabled rapid screening of many exposed fracture surfaces, but it also introduced topographic effects that are absent from polished cross-sections. This limitation was treated explicitly when interpreting oxygen values, weak signals, and point-to-point differences. No quantitative phase abundance was calculated from the number of analyzed points.

Electron-probe microanalysis

Morphology and local chemistry were investigated using a JEOL JXA-8230 electron probe microanalyzer. All archived SEM/EDS spectrum reports used in this study record an accelerating voltage of 20.00 kV. Backscattered-electron compositional contrast was acquired in COMPO/BSE mode. Point and area compositions were obtained by energy-dispersive X-ray spectrometry and reported with the JEOL software ZAF correction. The archived point-spectrum reports for both samples consistently record process-time setting T3 and a live acquisition time of 60.00 s; real acquisition time varied with dead time. The exported point-spectrum reports display beam current as 0.00 nA at the report precision, so an exact point-by-point EDS probe current cannot be reconstructed from the surviving export. The previously stated range of 25 pA–5 nA was therefore removed rather than retained without documentary support.

For the WDS map of the complex S1 microdomain, the archived parameter file records an accelerating voltage of 20.0 kV, a probe current of 2.493×10^{-8} A (24.93 nA), beam-scan magnification $\times 4000$, a dwell time of 10.00 ms per point, a 352×264 measurement grid, and a step size of 0.0852 μm in both X and Y. The recorded WDS lines/crystals were O K α /LDE2, Fe K α /LIF,

Zr L α /TAP, Nb L α /PETJ, Si K α /TAP, Ce L α /LIF, Nd L α /LIF, and Th M α /PETJ; COMPO imaging was recorded simultaneously.

The surviving EDS archive does not document the reference standards or a separate calibration sequence. Accordingly, the EDS compositions are treated as archived software-corrected local analyses rather than standards-based quantitative EPMA. EDS is a full-spectrum method rather than a fixed preselected-line measurement; the JEOL reports identify the line family used for quantification for each reported element. A constituent was treated as detected when the JEOL export provided a quantified value supported by the corresponding spectrum or map, whereas entries explicitly marked 'nd' were retained as not detected. Trace values affected by potential peak overlap, holder contribution, or rough-surface geometry were not used for phase assignment without corroboration.

The interpretation followed established principles of SEM/EPMA microanalysis. BSE contrast was treated as a qualitative guide to compositional differences, while quantitative EDS values were interpreted as the composition of the excited interaction volume rather than as a direct mineral formula. Particular caution was applied to rough surfaces, minor constituents, and possible peak interferences, consistent with standard microanalysis practice (Newbury & Ritchie, 2015; Goldstein et al., 2018).

Analytical dataset and classification strategy

The dataset comprised BSE/COMPO images acquired from $\times 40$ to $\times 4000$, six composite EDS mapping sets, 32 point EDS analyses, two area EDS analyses, and one WDS map (Table 2). Nineteen point analyses and four mapping sets belonged to S1, whereas 13 point analyses and two mapping sets belonged to S2. The analyzed positions were selected deliberately to represent visually distinct microstructural domains rather than by a random or systematic probability-sampling design. The data therefore support local comparative microchemical classification but not modal quantification, slag-wide means, confidence intervals, or population statistics. Within this targeted archive, the Fe–S-bearing composition (49.55 wt.% Fe and 30.33 wt.% S) is documented at one EDS point on one S1 particle (1 of 19 S1 point analyses). The diagnostic Fe–Cr–Mn composition (53.69 wt.% Fe, 7.49 wt.% Cr, and 8.71 wt.% Mn) is documented at one EDS point on one S2 particle (1 of 13 S2 point analyses). Additional Fe-rich S2 points were analyzed, but they did not show the same reported Cr-rich association. These counts are occurrence records within purposive feature-targeted sampling and must not be interpreted as estimates of slag-wide frequency.

Microdomains were grouped by dominant chemical association into five descriptive classes: (1) Ti-rich Fe–Ti–O matrix; (2) Fe–Mn- or Fe–Cr–Mn-enriched regions; (3) an Fe–S-bearing region; (4) silicate and aluminosilicate inclusions or mixed silicate–oxide regions; and (5) a complex multielement domain containing Si, Al, Ca, Ti, Zr, Nb, and rare-earth/thorium signals. The class names are deliberately chemical rather than crystallographic. A specific mineral name was used only where the available evidence was sufficient; otherwise, the term “microdomain” or “region” was retained.

Table 2. Structure of the SEM–EDS/WDS analytical dataset

Material	BSE/COMPO	EDS mapping sets	Point EDS	Area EDS	WDS
60/40 slag (S1)	$\times 40$ – $\times 4000$	4	19	1	1 map
TiZir slag (S2)	$\times 40$ – $\times 3300$	2	13	1	—
Total	Multiscale	6	32	2	1 map
<i>Note – compiled by the authors</i>					

Quality control and interpretative boundaries

Several analytical boundaries were defined before comparison. First, the high oxygen values in some point and area analyses may be affected by particle inclination, surface relief, and the matrix-correction conditions; they are not used to infer exact oxide stoichiometry. Second, mapped-field averages represent only the selected fields and must not be interpreted as certified bulk slag composition. Third, EDS does not independently identify crystal structure, so Fe-rich and silicate regions are not assigned to a named phase without complementary diffraction or crystallographic evidence. Because the sampling was purposive, no statistical comparison of whole-sample Fe contents or microdomain frequencies is made from the 32 point analyses or the two area analyses.

Fourth, Nb values in the hundredths of a weight percent range were treated as near the practical interpretative limit under the archived conditions. In the Fe–S-rich S1 point, the software-reported Nb value was not accepted as confirmed because S K and Nb L signals can complicate deconvolution in the relevant energy range; WDS verification would be required. Fifth, simultaneous Cu and Zn signals observed in selected analyses were excluded from process interpretation because the particles were mounted on brass holders and secondary excitation or holder contribution cannot be ruled out. These rules reduce the risk of converting instrumental output into unsupported phase claims.

RESULTS AND DISCUSSION

Particle morphology and scale of heterogeneity

Both slags consisted predominantly of angular to subangular particles with brittle fracture surfaces and strong BSE contrast (Figure 1). At low magnification, neither material appeared texturally uniform. S1 contained numerous fine bright features and locally mottled regions within particles, whereas S2 displayed several large particles with elongated darker inclusions and discrete bright domains against a comparatively continuous Ti-rich background. The contrast persists over multiple scales, indicating that the heterogeneity is not restricted to isolated trace particles.

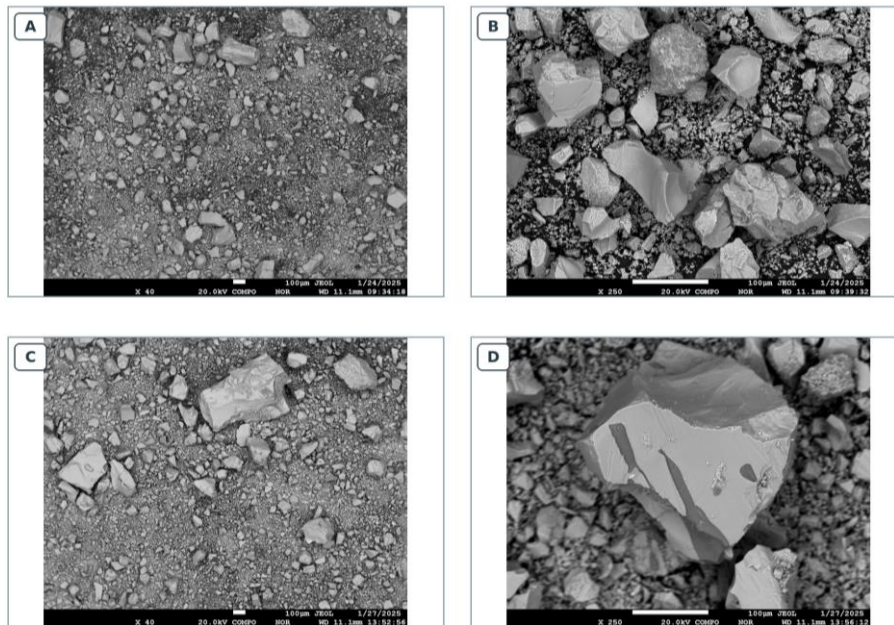


Figure 1. BSE/COMPO morphology of the investigated titanium slags:
(A, B) 60/40 slag at $\times 40$ and $\times 250$; (C, D) TiZir slag at $\times 40$ and $\times 250$.

Note – compiled by the authors

This morphology is consistent with the general behavior of titania-rich slags, in which high-temperature reduction produces a Ti-rich oxide melt that solidifies together with non-titanium constituents that have limited or composition-dependent solubility. Classical descriptions of titanium slag emphasize the coexistence of Ti-rich oxide solid solutions, rutile-related constituents, and intergranular silicate material (Pistorius & Coetzee, 2003; Guéguin & Cardarelli, 2007). The present images do not establish these phases crystallographically, but they demonstrate the type of compositional partitioning expected from such a multiphase solidification history.

Comparison of representative mapped fields

The area analysis of one selected mapped S1 field yielded 49.32 wt.% O, 40.48% Ti, 6.04% Fe, 1.54% Si, 1.34% Mn, 0.67% Al, 0.45% Mg, 0.12% Zr, and 0.03% Nb, as summarized in Table 3. The corresponding selected S2 field yielded 49.81% O, 38.74% Ti, 6.36% Fe, 3.05% Si, 0.95% Mn, 0.89% Al, and 0.19% Ca. The numerical proximity of 6.04 and 6.36 wt.% Fe applies only to these two local fields. It is not evidence that S1 and S2 have similar bulk Fe contents, and no inferential statistics are applied to this pair of purposively selected observations. Their juxtaposition is used only to illustrate that selected local fields with similar area-averaged Fe can display different spatial patterns.

The EDS maps show that the Ti signal broadly follows the principal matrix in both materials, whereas Fe forms a mixture of matrix-level distribution and localized maxima (Figure 2). Silicon differs more visibly. In S2, elongated Si-rich domains are sharply separated from much of the Ti-rich matrix and spatially correlate with Al and minor alkalis or Ca. In S1, Si is more variably distributed and participates in local and complex multielement regions. Such differences are important for later treatment because intergranular or elongated silicates can control fracture paths, expose reaction interfaces, or persist as insoluble residues. Pistorius and Kotzé (2009) showed that silicate constituents play a material role during titania-slag comminution, while recent work on non-titanium components emphasizes their non-uniform distribution (Han et al., 2025).

Table 3. Area compositions of representative mapped fields, wt.%

Material	O	Mg	Al	Si	Ca	Ti	Mn	Fe	Zr	Nb
60/40 slag	49.32	0.45	0.67	1.54	—	40.48	1.34	6.04	0.12	0.03
TiZir slag	49.81	—	0.89	3.05	0.19	38.74	0.95	6.36	—	—

Note – local area analyses only; values characterize one selected mapped field per material and are not bulk slag compositions, population means, or a statistical basis for comparing overall Fe content.

Iron localization in the 60/40 slag

Common S1 matrix points contained approximately 53.71–60.88 wt.% Ti, 8.82–11.96% Fe, and 1.18–1.56% Mn. One matrix point contained 0.72% Si, while Nb was not detected. At the analyzed locations, the recurring Ti–Fe–Mn association shows that Fe contributes to the principal Ti-rich EDS interaction volume rather than occurring exclusively as a separate Fe-rich particle. Whether such locally matrix-associated Fe is less amenable to physical separation is a working hypothesis for future controlled tests; no separation response was measured in the present study.

In addition to this matrix-level distribution, S1 contained distinct Fe–Mn-enriched regions. One point contained 39.92 wt.% Ti, 19.83% Fe, and 6.50% Mn. An area analysis of another heterogeneous grain gave 41.34% Ti, 19.13% Fe, and 5.90% Mn, while a local point in the same general class contained 29.69% Fe and 22.19% Ti. These data define a continuum between Ti-rich matrix chemistry and more clearly iron-enriched microdomains. The observation is consistent

with the broader principle that Fe and Mn can partition together in reduced or partially reduced titaniferous oxide systems, although a specific phase assignment would require XRD/EBSD or quantitative EPMA on polished cross-sections.

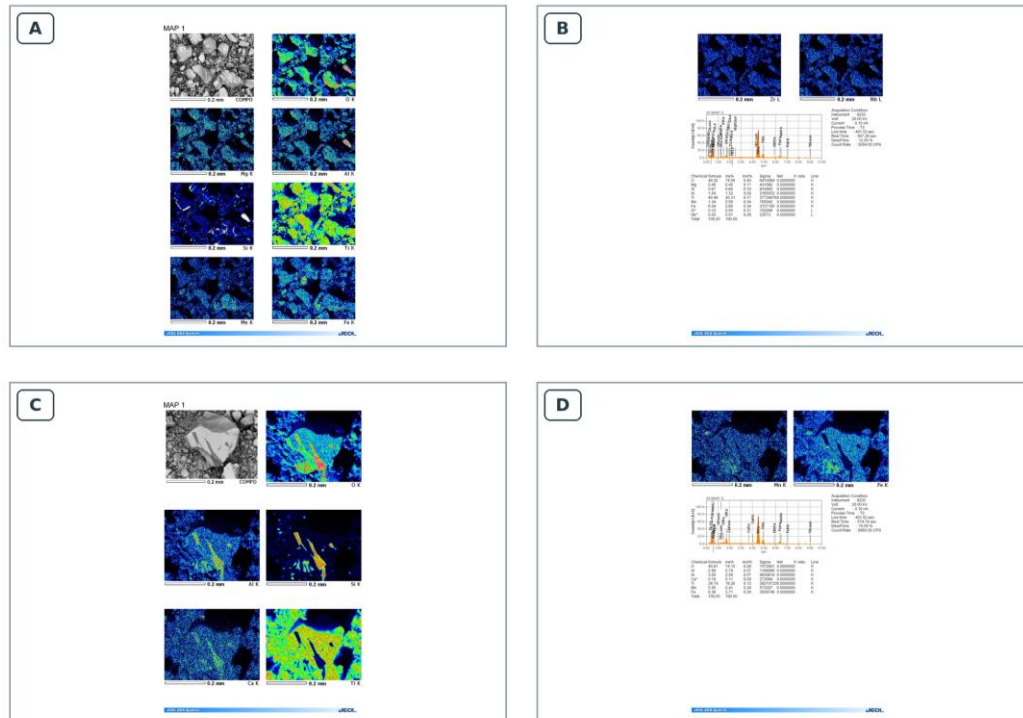


Figure 2. Composite EDS maps of representative fields: (A, B) 60/40 slag; (C, D) TiZir slag.

Map intensity is interpreted qualitatively as spatial localization

Note – compiled by the authors

A particularly bright S1 microdomain contained 49.55 wt.% Fe and 30.33% S, together with 3.54% Ti and 1.55% Si. This composition justifies describing the analyzed interaction volume as Fe–S-bearing and strongly differentiates it from the oxide-matrix analyses. The software also reported 0.69% Nb, but this value is not used in the interpretation because of the potential S K/Nb L spectral complication. The archived dataset contains one documented Fe–S-bearing EDS point on one S1 particle; no second independent S1 particle with the same Fe–S signature was documented in the available point-analysis archive. Accordingly, this feature is treated as a local occurrence requiring verification, not as a prevalent S1 constituent or as evidence of a specific sulfide phase. Its response during oxidation or leaching remains to be tested.

Iron localization in the TiZir slag

S2 likewise contained a Ti-rich matrix, with representative local compositions of approximately 39.46–50.19 wt.% Ti and 5.95–7.30% Fe (the values are detailed in Table 4). More strongly contrasted Fe-rich local analyses were also recorded. One point contained 53.69% Fe, 13.32% Ti, 8.71% Mn, and 7.49% Cr. Other Fe-rich points contained 39.58–57.32% Fe, 5.60–8.14% Ti, and 1.63–5.30% Mn. The combined high-Fe–Cr–Mn signature is documented at one targeted EDS point on one S2 particle; the additional Fe-rich points confirm Fe enrichment but do not reproduce the same reported Cr-rich association. The observations therefore establish local compositional contrast within the analyzed particles, not the prevalence of Fe–Cr–Mn regions throughout the TiZir slag lot.

The simultaneous enrichment of Fe, Mn, and Cr is a compositional observation that identifies a target for follow-up characterization. Chromium-bearing spinel-type impurities are

known to complicate ilmenite upgrading and titanium-product purification because they can persist through reduction and leaching stages (Kenzhaliyev et al., 2024). The present EDS data do not demonstrate that the S2 region is a spinel or any other specific crystallographic phase, nor do they demonstrate its dissolution behavior. Any suggested relation between the local Fe–Cr–Mn association and process refractoriness is therefore a working hypothesis to be tested by polished-section quantitative EPMA/WDS, XRD/EBSD, and controlled selective-dissolution experiments (Figure 3).

A useful comparison within the present dataset is therefore not a comparison of whole-slag average Fe, which was not measured, but a descriptive comparison between locally matrix-associated Fe and chemically separated Fe-rich interaction volumes observed in the selected particles. This distinction is consistent with modern process studies in which reduction efficiency and metal–slag partitioning are evaluated together with microstructure (Vorobkalo et al., 2023; Jing et al., 2026).

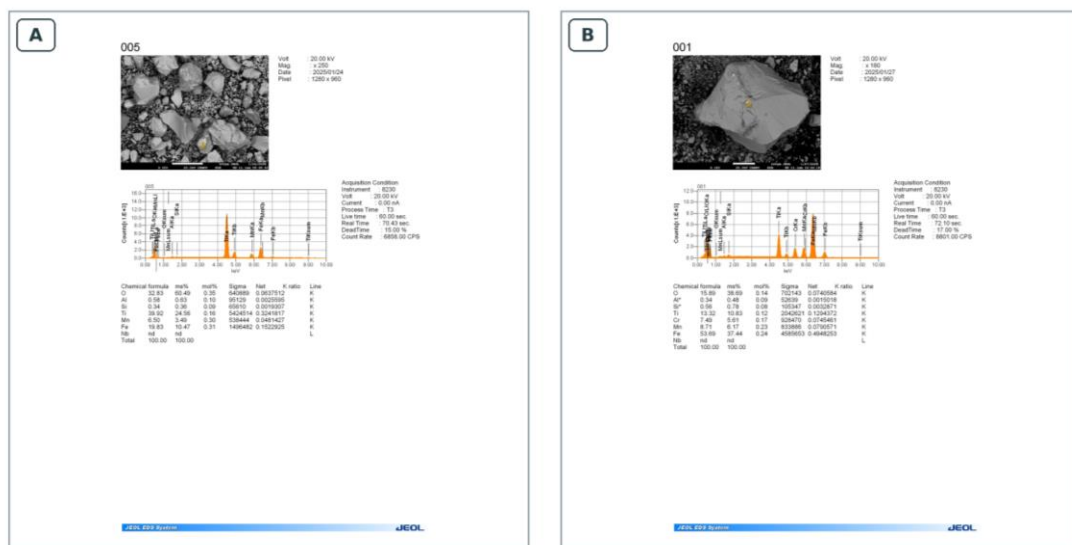


Figure 3. Representative Fe-enriched microdomains:
(A) Fe–Mn-rich region in the 60/40 slag; (B) Fe–Cr–Mn-rich region in the TiZir slag. Values shown in the source spectra refer to the local interaction volume
Note – compiled by the authors

Table 4. Representative matrix and Fe-enriched microdomains, wt. %

Material / type	Ti	Fe	Mn	Cr	Si	Interpretation
60/40, matrix 1	53.71	11.96	1.40	—	n.r.	Ti–Fe–Mn oxide matrix
60/40, matrix 2	60.88	8.82	1.56	—	0.72	Ti-rich matrix
60/40, Fe–Mn	39.92	19.83	6.50	—	n.r.	Fe–Mn-enriched region
TiZir, matrix	41.81	7.30	n.r.	n.r.	n.r.	Ti–Fe oxide matrix
TiZir, Fe–Cr–Mn	13.32	53.69	8.71	7.49	n.r.	Separated Fe-rich region
TiZir, Fe-rich	5.60	57.32	1.63	n.r.	n.r.	Separated Fe-rich region

Note – n.r. — not reported in the selected source spectrum; “interpretation” is descriptive and not a crystallographic phase assignment.

Silicate, aluminosilicate, and mixed microdomains

S1 contained a separate Si–Al–Ca-rich region with 62.04 wt.% O, 23.72% Si, 4.32% Al, 4.61% Ca, 2.78% Ti, and 0.88% Fe. The low Fe and Ti contents relative to the surrounding matrix support its classification as a silicate/aluminosilicate microdomain, but the chemical data are insufficient for a unique mineral name. Its presence demonstrates that at least part of the Si in S1 is hosted by a chemically separated region rather than being uniformly dissolved in the Ti-rich matrix.

Silicate segregation was more visually explicit in S2. Elongated dark inclusions contained 28.34–31.26 wt.% Si, 3.49–3.62% Al, 0.82–0.98% Na, 0.73–0.99% K, and 1.72–1.77% Ca, with only 2.03–3.43% Ti. This association is characteristic of a distinct local aluminosilicate-rich interaction volume and explains the strong Si-map contrast. The elongated geometry is recorded as a morphological observation. Its effect on fracture, reagent access, or liberation was not measured here and should be evaluated experimentally rather than inferred from shape alone.

S2 also contained intermediate Si–Fe–Mn–Ca-bearing regions. One point contained 16.39% Si, 18.45% Fe, 7.51% Mn, 10.67% Ti, and 3.38% Ca; another contained 21.85% Si, 11.40% Mn, 9.20% Ca, 6.29% Fe, and 5.46% Ti. These compositions may represent mixed interaction volumes, fine intergrowths, or reaction regions between oxide and silicate constituents. Their existence cautions against treating the oxide/silicate boundary as a perfectly sharp two-phase interface at the SEM interaction-volume scale.

From the standpoint of subsequent upgrading, silicate domains can influence both mechanical liberation and solution behavior. Their chemical stability, alkali content, and connectivity may control residue formation or the opening of reaction paths. The literature on titania slag comminution and on synthetic-rutile quality supports the need to include silicate architecture in process design rather than considering SiO₂ only as a bulk impurity number (Pistorius & Kotzé, 2009; Spencer et al., 2022).

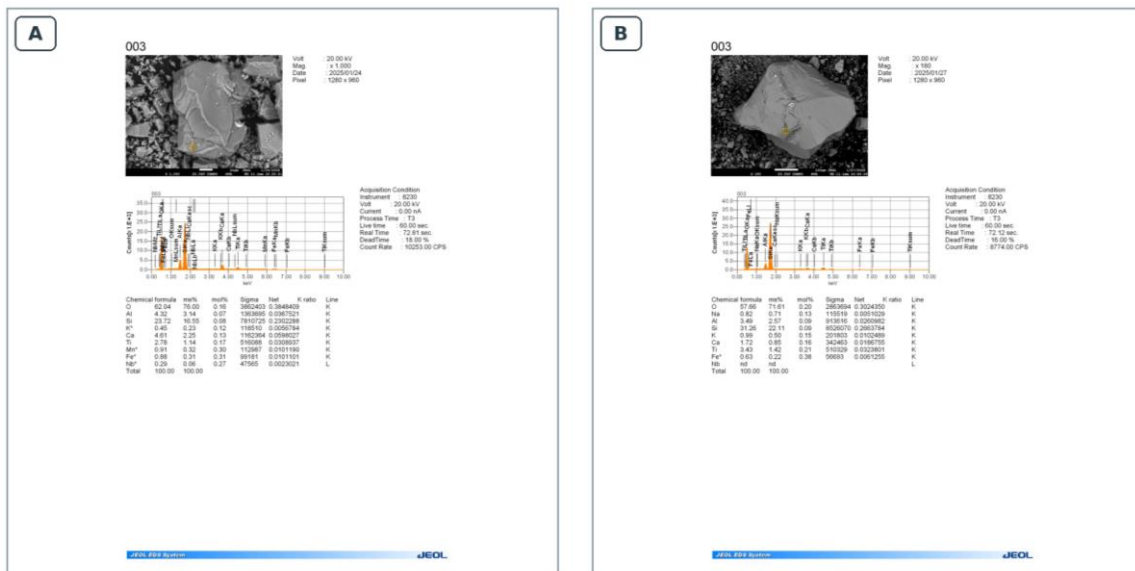


Figure 4. Representative silicate microdomains:

(A) Si–Al–Ca-rich region in the 60/40 slag; (B) elongated aluminosilicate inclusion (B) in the TiZir slag.

Note – compiled by the authors

Table 5. Representative silicate, mixed, and Fe–S-bearing microdomains, wt. %

Material / type	Si	Al	Ca	Fe	Mn	Cr / S	Feature
60/40, silicate	23.72	4.32	4.61	0.88	—	—	Low Ti and Fe
60/40, Fe–S	1.55	n.r.	n.r.	49.55	n.r.	S 30.33	Fe–S-bearing region
TiZir, aluminosilicate 1	28.34	3.62	1.77	n.r.	n.r.	—	Na 0.98; K 0.73
TiZir, aluminosilicate 2	31.26	3.49	1.72	0.63	n.r.	—	Na 0.82; K 0.99
TiZir, mixed 1	16.39	n.r.	3.38	18.45	7.51	—	Ti 10.67
TiZir, mixed 2	21.85	n.r.	9.20	6.29	11.40	—	Ti 5.46

Note – n.r. — not reported in the selected source spectrum. Values are local EDS measurements and are not normalized phase formulas.

Complex impurity-rich region in the 60/40 slag

A rare S1 domain showed a much more complex association than the principal matrix. Across the analyzed points, Si was approximately 16–23 wt.%, Al about 2.9–4.7%, Ca about 2.6–6.8%, Ti about 3–10%, and Zr reached approximately 1.7%. Nb was locally reported at roughly 0.6–2.2%, together with signals of Nd, Ce, Th, and in selected points Ba or K. WDS mapping confirmed that the heavy-element signals were spatially heterogeneous rather than uniformly distributed through the surrounding Ti-rich matrix.

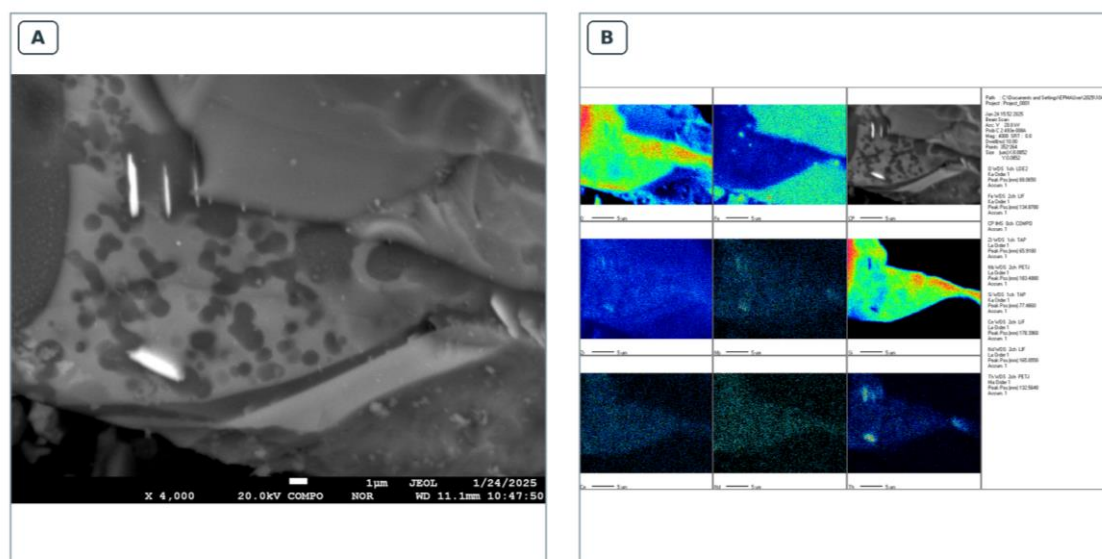


Figure 5. Complex impurity-rich microdomain in the 60/40 slag and associated local WDS/EDS distribution. The region is treated as a multiphase chemical association rather than a uniquely identified mineral

Note – compiled by the authors

The correct interpretation is a complex, fine-scale multielement microdomain rather than a single confidently identified mineral. Several explanations are plausible: inherited accessory-mineral fragments from the feed, reaction products between accessory and silicate/oxide components during smelting, or an interaction volume containing multiple submicrometre constituents. This ambiguity is analytically important. In process mineralogy, the temptation to assign a familiar mineral name from EDS chemistry alone can lead directly to incorrect

predictions of reagent response. The domain is therefore retained as an observational category requiring polished-section WDS/EPMA and, ideally, microdiffraction or EBSD for definitive characterization.

The rare-element aspect is secondary to the present article, which focuses on Fe localization. The observation is limited to a rare multielement domain in the selected S1 material and does not establish the prevalence or bulk inventory of accessory-element carriers in the slag. It nevertheless identifies a target for future carrier-tracing work and is consistent with the separate Nb-focused study of Sarsembekov et al. (2026).

Comparative process-mineralogical interpretation

The central comparison can be summarized at three local observational levels. First, the two selected mapped fields happen to yield close Fe values: 6.04 wt.% in S1 and 6.36 wt.% in S2; these are field-specific values, not slag averages. Second, the analyzed microdomains show different Fe associations: S1 includes Ti-Fe-Mn matrix analyses, Fe-Mn enrichment, and one documented Fe-S-bearing point, whereas S2 includes Ti-rich matrix analyses, several Fe-rich points, and one documented Fe-Cr-Mn-rich point. Third, the observed silicate architecture differs in the selected particles: S1 contains local and mixed silicate/multielement regions, while S2 displays conspicuous elongated aluminosilicate inclusions. These observations characterize the analyzed fields and particles only and cannot by themselves establish whole-lot composition or prevalence.

These observations motivate, but do not validate, several technological hypotheses. A discrete Fe-rich region exposed on a particle surface could differ in dissolution or oxidation behavior from Fe associated with a Ti-rich analytical volume; a Cr-bearing Fe-rich association could also differ from an Fe-Mn-rich association. Likewise, elongated silicate-rich regions could influence fracture or residue formation. None of these effects was measured in the present work, and no cause-and-effect relationship is assigned to the observed chemistry or morphology. Controlled experiments are required to determine whether any of these hypotheses is correct.

The hypothesis is consistent with recent upgrading studies. Daba et al. (2022) and Kurniawan et al. (2022) showed that acid-based iron removal is strongly conditioned by the initial state and pretreatment of ilmenite materials. Jing et al. (2023) demonstrated that sequential leaching of titanium slag can raise TiO₂ grade, while Spencer et al. (2022) linked feed properties to synthetic-rutile quality. These studies collectively indicate that reaction conditions cannot be optimized independently of mineralogical state.

For Kazakhstan ilmenite research, S1 is especially relevant because 40% of its feed originated from the Satpayev deposit. Recent Satpayev studies have addressed beneficiation, smelting, and the role of fluxes and aluminosilicate constituents (Myrzakulov et al., 2024a, 2024b; Akhmetova et al., 2025b; Motovilov et al., 2026). The present microchemical observations extend that context from process performance to impurity localization in a Ti-rich slag. They should not, however, be extrapolated directly back to the original ore without a tracked mass balance and matched feed/product sampling.

The practical next step is a controlled experimental matrix in which the two slags are characterized as polished cross-sections, subjected to identical reductive/oxidative pretreatments and iron-removal conditions, and then re-characterized by XRD-Rietveld, SEM/EPMA, and chemical mass balance. Such work would convert the present descriptive microchemical differences into testable cause-and-effect relationships between localization, phase transformation, and selective Fe removal.

Table 6. Comparative microchemical architecture and process implications

Feature	60/40 slag	TiZir slag	Working hypothesis for future testing
Main matrix	Ti-Fe-Mn oxide; finer dispersed heterogeneity	Ti-Fe oxide; distinct elongated inclusions	Test whether locally matrix-associated Fe changes accessibility after controlled redox treatment.
Fe-rich regions	Fe-Mn; local Fe-S	Fe-Cr-Mn, sharply contrasting	Test whether discrete Fe-rich associations respond differently from matrix-associated Fe and whether Cr-bearing regions persist preferentially.
Silicate component	Local/mixed; complex multielement regions	Elongated Al-Si-Na-K-Ca inclusions	Test whether silicate/oxide interfaces influence cracking, liberation, or residue formation under controlled conditions.
Rare-element association	Local Zr-Nb-REE-Th-rich region	Nb mainly trace in available analyses	Verify carrier identity crystallographically before assigning any recovery behavior.
Selected-field Fe	6.04 wt. %	6.36 wt. %	Use matched bulk assays and statistically designed mapping before relating local Fe values to whole-slag process response.
<i>Note – entries in the final column are prospective hypotheses derived from local observations. They are not demonstrated technological effects and require controlled experimental validation</i>			

Study limitations

The principal limitation is sample preparation. Rough powder particles are suitable for screening and locating strongly contrasting regions, but they are less suitable than polished sections for accurate quantitative EPMA because take-off geometry, surface orientation, and local relief vary. This limitation is particularly relevant for oxygen and for small differences between neighboring matrix points. The second limitation is representativeness: point and field analyses were deliberately selected from morphologically distinct regions rather than by random sampling. Therefore, the dataset cannot be converted into modal abundance, whole-slag concentration statistics, confidence intervals, or population frequencies. The values 6.04 and 6.36 wt. % Fe characterize two selected mapped fields only. The documented Fe-S signature in S1 and diagnostic Fe-Cr-Mn signature in S2 each occur at one targeted point on one analyzed particle in the present archive; these are occurrence counts, not prevalence estimates.

The third limitation is phase identification. Chemical association does not uniquely determine crystal structure, especially in Fe-Ti-O systems with broad solid solutions and in fine silicate intergrowths. Complementary XRD, EBSD, microdiffraction, or standards-based quantitative EPMA/WDS is necessary before naming specific oxide, sulfide, or silicate phases. The fourth limitation is process provenance and interpretation: a matched record of smelting conditions, degree of reduction, cooling history, and certified bulk composition was unavailable for the two samples, and no leaching, oxidation, magnetic-separation, or reduction experiments were performed on them as part of this dataset. Consequently, the article reports local microchemical observations and formulates hypotheses for future selective-iron-removal studies; it does not establish why S1 and S2 differ, a removal efficiency, or a kinetic mechanism.

CONCLUSIONS

A common SEM-EDS/WDS protocol revealed substantial local microchemical heterogeneity in the selected particles of two titanium slags of different origin. The two selected mapped fields yielded 6.04 and 6.36 wt. % Fe, respectively; these values are local field descriptors

and not bulk slag averages. Within the targeted point-analysis archive, S1 showed Ti–Fe–Mn matrix analyses, Fe–Mn-enriched regions, and one documented Fe–S-bearing point on one particle, while S2 showed Ti-rich matrix analyses, several Fe-rich regions, and one documented Fe–Cr–Mn-rich point on one particle. These occurrence counts do not establish slag-wide frequencies.

The silicate architecture also differed. The 60/40 slag contained local Si–Al–Ca-rich and complex multielement regions, including a rare Zr–Nb–REE–Th-bearing microdomain. The TiZir slag showed more conspicuous elongated aluminosilicate inclusions enriched in Si and Al with minor Na, K, and Ca and depleted in Ti. Transitional Si–Fe–Mn–Ca regions indicate fine-scale interaction or intergrowth between silicate and oxide components.

The results show that a single selected-field Fe value is insufficient to describe the local process-mineralogical architecture visible in the analyzed particles: spatial localization, associated Mn/Cr/S, and silicate geometry provide additional descriptive information. The specific contribution of the present study is a common-workflow comparative classification of these local microchemical associations; the general existence of heterogeneity in titanium slags is not claimed as novel. Because sampling was purposive and the samples lack matched production histories and bulk assays, the data cannot support whole-slag statistical comparison or causal attribution of the observed differences to provenance. Any proposed difference in accessibility of matrix-associated and chemically separated Fe remains a working hypothesis, and no leaching mechanism or phase identity is claimed from the present microanalysis alone.

Future work should use polished cross-sections, quantitative EPMA/WDS, XRD–Rietveld and/or EBSD, followed by controlled reduction, oxidation, magnetic separation, and iron-removal tests with full Fe–Ti material balances. This approach will allow the microchemical classes identified here to be linked quantitatively with transformation kinetics and TiO₂-rich product quality.

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DATA AVAILABILITY STATEMENT: The primary SEM–EDS/WDS data supporting the findings of this study are available from the corresponding author upon reasonable request. The data are not publicly archived because they form part of an ongoing research dataset.

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