Crossref
DOI: 10.31643/2028/6445.08
Metallurgy and Metallurgical Engineering

Development of a methodology for microstructural and thermal verification of the quality of an industrial Ti-10V-2Fe-3Al triple vacuum arc remelted ingot

[1]Mamutova A.T., [2*]Chepushtanova T.A., [3]Mishra B.

[1] Ust-Kamenogorsk Titanium and Magnesium Plant JSC, Kazakhstan
[2]Satbayev University, Almaty, Kazakhstan
[3]Worcester Polytechnic Institute, USA, Worcester

** Corresponding author email: t.chepushtanova@satbayev.university*

Received: *March 5, 2026*
Peer-reviewed: *March 16, 2026*
Accepted: *May 6, 2026*

ABSTRACT

The article is devoted to the development of a methodology for microstructural and thermal verification of the quality of an industrial Ti-10V-2Fe-3Al triple vacuum arc remelted ingot produced by UK TMP JSC. It was established that all zones of the ingot demonstrate a two-stage thermal evolution characteristic of the β-metastable Ti-10V-2Fe-3Al alloy: decomposition of the metastable β phase (≈520–570 °C) and an endothermic α→β phase transformation (≈950–1120 °C). It was found that the enthalpy of the exothermic decomposition of the β matrix increases by approximately 60–80% in the lower zone of the ingot. The width of the phase transition (ΔT) correlates with an increase in microsegregation. It was also determined that the enthalpy of the endothermic α→β transformation decreases from the bottom part of the ingot toward the steady-state crystallization zone (Middle-1), which correlates with a reduction in the microsegregation parameters obtained from SEM–EDS profiles (ΔC_max, σC, L_{corr}). Thus, thermal analysis confirms the absence of a pronounced vertical gradient of structural stability and can be used as a validating criterion for the integral electrode quality index. For the first time, a quantitative correlation between SEM–EDS profiles and DSC–DTG characteristics has been proposed. Thermal analysis is suggested as an independent validator of microsegregation. An approach to the quantitative evaluation of microsegregation based on SEM–EDS profiles using the parameters ΔC_max, σC, L_{corr}, and $\Delta CO_{(local)}$ has been developed. Additionally, the use of an integral chemical index I_{chem}, and a critical threshold I^{crit}_{chem} is proposed for electrode quality control using thermal analysis results.

Keywords: Ti-10V-2Fe-3Al triple-remelt alloy, vacuum arc melting, DSC–DTG and SEM–EDS analyses, α/β phase transformations, thermal decomposition analysis.

Information about authors:

Mamutova Assem Tlekovna — *President of Titanium and Magnesium Plant JSC, Ust-Kamenogorsk, Kazakhstan, PhD student of the Mining and Metallurgical Institute, Satbayev University, Almaty, Kazakhstan. Email: a.mamutova@satbayev.university*

Chepushtanova Tatyana Aexandrovna — *Candidate of Technical Sciences, PhD, Professor, Head of Laboratory 'Metallurgical processes, heat engineering and powder metallurgy, Mining and Metallurgical Institute, Satbayev University, Almaty, Kazakhstan. Email: T.Chepushtanova@satbayev.university*

Mishra Brajendra — *Professor and MPI Director of Mechanical and Materials Engineering, Worcester Polytechnic Institute, USA, Worcester. Email: bmishra@wpi.edu*

Introduction

In industrial titanium metallurgy, vacuum arc remelting (VAR) remains the fundamental technology for producing large ingots, while triple VAR is used as a method to reduce inclusions, stabilize the molten pool, and improve chemical homogeneity [1]. However, even with multiple remelting cycles, risks of crystallization defects persist: macrosegregation, local segregation channels, "hot top" defects, inhomogeneity of the solidification zone, and variations in molten pool depth—factors that cannot be completely eliminated solely by increasing the number of remelts. Studies on VAR defects emphasize that the evolution of macrosegregation during different stages of triple VAR remains a distinct scientific problem requiring a combination of experimental and modeling approaches [[2], [3]].

A modern trend is the control of structure and segregation through melting regimes and pool management: melt depth, mushy zone width, thermal gradients, and solidification rate. These aspects are actively developed in numerical VAR

models, including multilevel and multiscale approaches [4].

The quality of a VAR ingot is determined not only by chemical composition but also by the trajectory of heat and mass transfer and arc/gap stability, which influence the pool profile and solidification conditions. Therefore, methods that "read" quality through structural-chemical markers (microsegregation, oxygen-enriched zones, α/β morphology) and link them to thermal signals of phase transformations and melting are becoming increasingly important [[5], [6]].

Control of the VAR process and ingot quality today relies on electrode gap control, melt pool modeling, and structure prediction. Arc gap control and melting stability are widely discussed. For Ti-10V-2Fe-3Al (Ti-1023), it has been shown that automatic electrode gap regulation (via maintaining average voltage and using specific pulse/polarity modes) significantly affects remelting stability and indirectly influences homogeneity and defect formation. This research direction is important as it links VAR "process maps" with industrial ingot quality [[7], [8], [9]].

VAR modeling as a tool for segregation prevention is considered a source of critical data. Models have evolved from axisymmetric to 3D/multiscale (arc → pool → solidification → grain structure). Key references include multiscale 3D VAR models (process-oriented coupling of arc/pool/solidification), multiscale modeling of microstructure formation in Ti-6Al-4V, and models validated for Ti-10V-2Fe-3Al aiming to reproduce pool profiles and solidification behavior [[4], [10]].

Additionally, modern solidification simulation approaches (e.g., CAFE methods) demonstrate that modeling is becoming a practical tool for selecting process parameters, although experimental validation remains necessary to confirm real homogeneity.

Macrosegregation and its persistence remain among the most critical defects, as they can survive subsequent heat treatment and degrade properties. For triple VAR, it is emphasized that repeated remelting does not always guarantee elimination of macrosegregation without process optimization [[11], [12], [13]].

Channel segregation and its relation to processing are also highly relevant. In industrial ingots, channel segregation typically appears in characteristic zones (often 1/4–3/4 of the radius), with morphology correlated to pool profile and steady/transient remelting regions [[14], [15], [16]]. These defects may be associated with local oxide particles, directly linking to oxygen enrichment and α_2-phase risks.

From a technological perspective, microsegregation of β-stabilizers (Fe, V) and oxygen should be considered as "hidden" quality markers. For Ti-1023, Fe microsegregation is critical due to the risk of β-flecks and property degradation. SEM–EDS is widely used to quantify these effects, including diffusion and homogenization behavior of Fe. This confirms that SEM–EDS profiles are not merely illustrative but serve as a quantitative quality control tool. Oxygen plays a separate role, affecting phase equilibria (β-transus), promoting local strengthening or embrittlement zones, and being difficult to accurately assess without spatial resolution beyond bulk chemical analysis [[17], 18], [19]].

Thermal analysis using high-temperature analyzers is also a universal tool for quality control of Ti alloys, particularly for determining β-transus and phase transformations via DSC/STA. DSC is widely used for evaluating β-transus, composition effects, oxygen influence, and thermokinetics. Literature emphasizes that DSC can be faster and more objective than purely metallographic methods. Thermal analysis allows evaluation of melting intervals, phase transitions, and heating rate effects. For Ti alloys, phase transformations and melting behavior are sensitive to processing conditions and material state (including heating rate effects). Some studies demonstrate the determination of liquidus/solidus temperatures using DTA as a classical approach for obtaining technologically significant temperatures [[20], [21], [22]].

This work proposes the development of a methodology for microstructural and thermal verification of the quality of an industrial Ti-10V-2Fe-3Al triple VAR ingot through metrological coupling of spatial chemical heterogeneity (SEM–EDS profiles) and thermokinetic analysis (STA/DSC/TG), as well as the introduction of the Ichemcrit criterion as a decision-making tool for rejection or correction of triple remelting parameters and charge preparation.

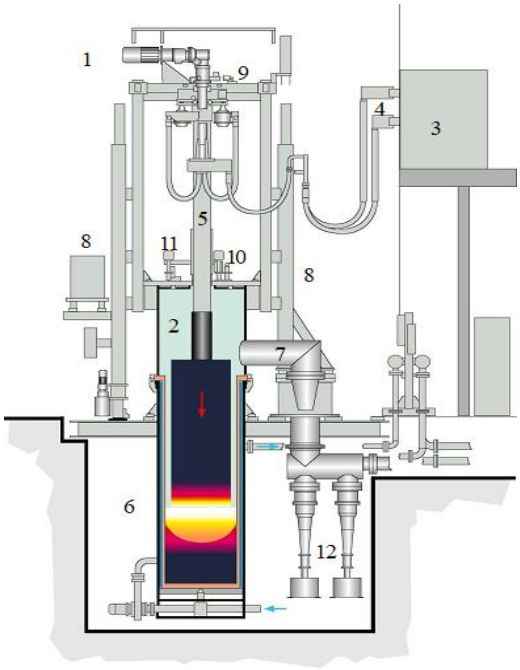
Initial materials

The object of the study was a triple vacuum arc remelted (VAR) ingot of Ti-10V-2Fe-3Al, produced by vacuum arc melting in accordance with the customer's specification: Al — 2.60–3.40%, V — 9.00–11.00%, Fe — 1.60–2.20%, O — max. 0.13%.

The objects of investigation for SEM–EDS and DSC/DTG analyses were three electrode zones: samples taken from the “top”, “middle 1”, “middle 2”, and “bottom” regions of the ingot.

Technology for the production of titanium ingots in a vacuum arc furnace (VAR)

The triple vacuum arc remelted Ti-10V-2Fe-3Al alloy was produced on an industrial scale at *Ust-Kamenogorsk Titanium and Magnesium Plant JSC*. The VAR furnace configuration used for producing the Ti-10V-2Fe-3Al alloy is shown in Figure 1.



Legend for the schematic: 1 – electrode feed drive; 2 – furnace chamber; 3 – melting power supply; 4 – busbars and cables; 5 – plunger-type electrode holder; 6 – water jacket with mold (crystallizer); 7 – vacuum duct; 8 – rotating column; 9 – coordinate adjustment system; 10 – load cell system; 11 – TV camera system; 12 – oil booster pumps.

Figure 1 – Schematic of the VAR furnace

The industrial Ti-10V-2Fe-3Al alloy ingot was produced by triple vacuum arc remelting (VAR). Multiple remelting was applied to minimize macrosegregation, improve chemical homogeneity, and stabilize the molten pool depth.

The main technological parameters for each remelting stage are presented below:

1st remelting: mass of melted metal – 4.5 t; melting time – 7 h 40 min; electrode area – 500 mm²; 2nd remelting: mass – 4.4 t; time – 10 h; electrode area – 600 mm²; 3rd remelting: mass – 4.4 t; time – 21 h 40 min; electrode area – 680 mm².

The density of titanium was taken in the range of 4.67–4.75 g/cm³.

The first remelting was carried out in a mold with a diameter of 620 mm. The mass melting rate was 9.90 kg/min (1st electrode), 7.49 kg/min (2nd electrode), and 7.96 kg/min (3rd electrode). The formation of a residual disk with a height of at least 40 mm ensured melt pool stability and controlled solidification.

A key technological parameter was cooling under residual pressure in the VAR chamber: 10 hours after the first and second remelting stages and 8 hours after the third. The average voltage in the steady-state phase was 29.8–33.3 V with a solenoid coil operation periodicity of ±3 s, ensuring arc stability and thermal regime control.

The calculated electrode composition was identical for all remelting stages: Al – 3.25%, V – 9.7%, Fe – 1.95%, O – 0.105%, C – 0.011%.

The selected processing conditions ensured process reproducibility and minimization of macrosegregation.

Research methodology, sampling, and homogeneity control strategy

To assess structural and chemical homogeneity, samples were taken from four characteristic zones along the ingot height: “top”, “middle 1”, “middle 2”, and “bottom”. Additionally, each ingot was divided into three sections with transverse cuts performed: at a distance of 40 mm from the top, from the central steady-state zone, and at a distance of 40 mm from the bottom.

This approach made it possible to analyze the vertical distribution of elements and identify potential zones of non-steady crystallization.

The chemical composition of all studied samples meets the specification requirements for Ti-10V-2Fe-3Al, confirming the effectiveness of triple VAR in terms of macrochemical homogeneity. Further analysis of structure and elemental distribution was carried out using SEM–EDS and DSC/DTG methods to identify microsegregation, local oxygen enrichment, and to perform thermal verification.

The study employed scanning electron microscopy (SEM) and energy-dispersive spectroscopy (EDS) using a JEOL JSM-7000 system.

Thermal analysis was performed using a STA 449 Jupiter NETZSCH thermal analyzer with heating up to 2000 °C.

For SEM–EDS investigations, key magnifications were selected to reflect the technological relationship of structure formation within the ingot:

– 500× — general structure, primary phase distribution;
– 1000–2000× — morphology of α plates;
– 5000× — local features, dispersed particles, segregation zones.

Thus, the study focused on identifying: vertical chemical gradients (increase of V or Fe upward or downward); distribution of β-stabilizers during remelting; presence of oxygen or carbon segregation; oxygen enrichment in the upper part due to interaction with residual gas in VAR; and oxygen accumulation at α-phase boundaries as an early stage of α_2–Ti_3Al formation.

SEM–EDS Analysis Results

As a result of analyzing the morphological evolution of the α/β structure, the following features along the ingot height were identified.

"Top" zone. At magnifications of ×3000–5000, thin parallel stepped layers, individual loose fragments, and isolated fine particles are observed. According to EDS data, Ti dominates (TiKα peak ~4.5 keV), with pronounced V and Al peaks, as well as elevated carbon content. The surface is generally smooth with minor defects; the increased carbon content is likely associated with the final stage of remelting and surface-related processes.

"Bottom" zone. Clusters of rounded and irregularly shaped particles are identified between the matrix steps, morphologically corresponding to non-metallic inclusions (oxide or slag fragments). Pronounced layering and the presence of large defects are observed, indicating structure formation under unstable initial remelting conditions and possible dendritic segregation. The Ti content is ~83.5–84 wt.%, while V and Al concentrations are close to nominal values, and no pronounced vertical gradient of β-stabilizers is detected. The carbon content is lower than in the top zone, confirming its predominantly surface-related nature in the upper section.

"Middle-1" zone. The structure is characterized by the most uniform lamellar morphology: α plates are well-bonded and aligned, the surface is relatively smooth, and the number of inclusions is minimal. The chemical composition is stable and closest to the nominal composition of Ti-10V-2Fe-3Al. Low carbon content and the absence of pronounced oxygen peaks indicate minimal gas saturation and oxide defects. This zone corresponds to a steady-state remelting regime and uniform formation of the α/β structure.

"Middle-2" zone. A layered and split structure is observed, with the presence of oxide phases and film-like inclusions. EDS data confirm the presence of local structural defects. Since the "middle-1" zone reflects a stable crystallization regime and the highest structural homogeneity, the SEM–EDS results for this sample are presented in Figures 2–4.

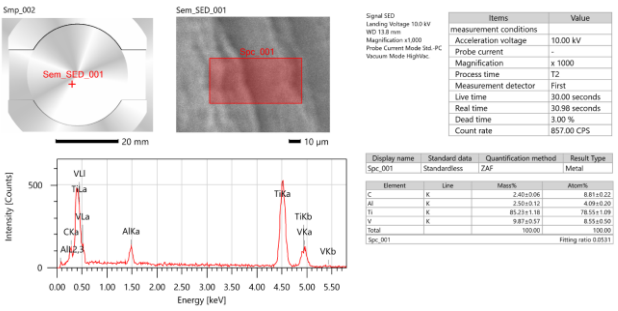


Figure 2 – SEM elemental analysis results over the surface area of the "middle 1" zone

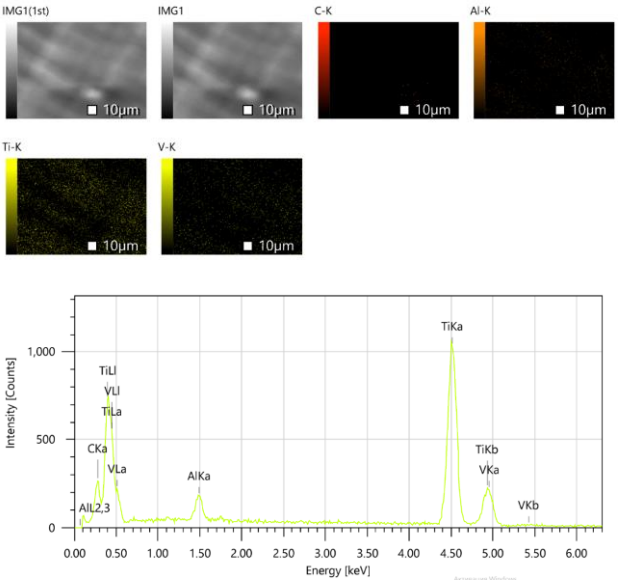


Figure 3 – EDS mapping results of the "middle 1" zone

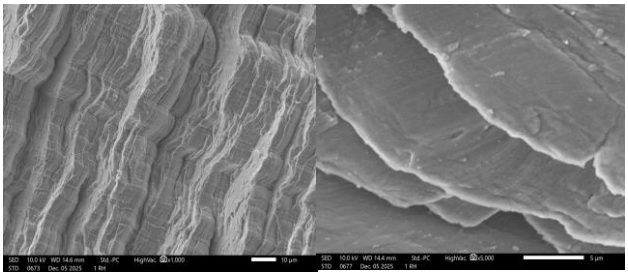


Figure 4 – SEM micrographs of the "middle 1" zone

As a result of analyzing the vertical distribution of Al–V–Ti, no pronounced chemical gradient along

the ingot height was identified. This confirms the high efficiency of triple VAR, which ensures melt homogenization and suppression of segregation, positively affecting phase stability, uniformity of the α/β structure, and reproducibility of mechanical properties.

SEM–EDS data show that the V content in all studied zones (top, bottom, middle-1) is within ~9.3–9.9 wt.%, Al — 2.5–3.1 wt.%, while the slight increase in Ti from the top to middle-1 falls within the measurement error. No significant vertical segregation of β-stabilizers was detected.

Thus, the Ti-10V-2Fe-3Al ingot is characterized by complete chemical uniformity along its height. The variation in the concentrations of the main alloying elements does not exceed 1–1.5% (Al and V — within ±0.5–1.0%, Ti — ~83–86%), which, for an industrial ingot weighing 4.5 t, indicates the high quality of the performed triple VAR process.

Macrohomogeneity in V and Al is confirmed by SEM–EDS: triple VAR effectively suppresses macrosegregation of β-stabilizers along the ingot height. Microdefects are distributed according to technological zones: bottom — non-metallic inclusions/layering (initial remelting stage, interaction with the baseplate/crucible); middle-1 — minimal defects (steady-state regime); middle-2 — film-like/oxide inclusions (local disturbances in charge preparation/purity); top — surface reactivity and elevated carbon content (final stage/surface processes).

Development of a methodology for electrode quality control based on SEM–EDS profiles

The obtained SEM–EDS profiles of V, Fe, Al, and O distribution along scan lines in the “top”, “middle 1”, “middle 2”, and “bottom” zones make it possible to transition from qualitative assessment to a quantitative electrode quality criterion based on microsegregation parameters.

The following diagnostic indicators are proposed: the maximum deviation of β-stabilizer concentrations (V, Fe) from the mean value along the scan line, $\Delta C_{max} = \max |C_i - \bar{C}|$; the standard deviation of concentration:

$$\delta C = \sqrt{\frac{1}{n}\sum_{i=1}^{n}(Ci - \bar{C})^2} \quad (1)$$

the correlation length L_{cor} – the characteristic size of chemically heterogeneous regions, determined from the autocorrelation function of the profile (the distance at which the correlation coefficient decreases to 1/e); local oxygen enrichment:

$$\Delta C_0^{(loc)} = C_{0,\,loc} - \overline{C_0} \quad (2)$$

For the central zone of the ingot (“middle-1”), the values of ΔC_{max} and σC are minimal, and L_{corr} is small, corresponding to fine-scale, statistically averaged microsegregation. In the lower part of the ingot, these parameters increase, reflecting the formation of more extended chemically heterogeneous regions. Based on these quantities, an integral index of chemical heterogeneity is introduced:

$$Ichem = a_1\frac{\Delta C_{max}}{\bar{C}} + a_2\frac{\delta C}{\bar{C}} + a_3\frac{\Delta Lcorr}{Lref} + a_4\frac{\Delta C_O}{\overline{C_{O,ref}}} \quad (3)$$

where a_i are weighting coefficients (determined empirically), and L_{ref} и $C_{O,ref}$ – are normalization parameters.

A threshold value I_{chem}^{crit} (critical chemical inhomogeneity index) is defined. If the condition $I_{chem} \le I^{crit}_{chem}$ is satisfied, the electrode is considered compliant with homogeneity requirements; if the threshold is exceeded, adjustment of the triple VAR regime or rejection of the billet is required.

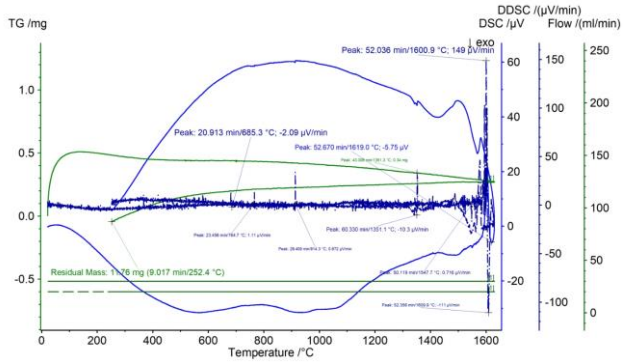
Thus, the proposed index transforms SEM–EDS analysis from a descriptive tool into a quantitative method of incoming quality control. Incorporation of this criterion into the charge preparation protocol enables closure of the technological chain “briquette → electrode → ingot” and ensures reproducible alloy structure under industrial conditions.

Results of thermal analysis (differential scanning calorimetry) DSC/DTG

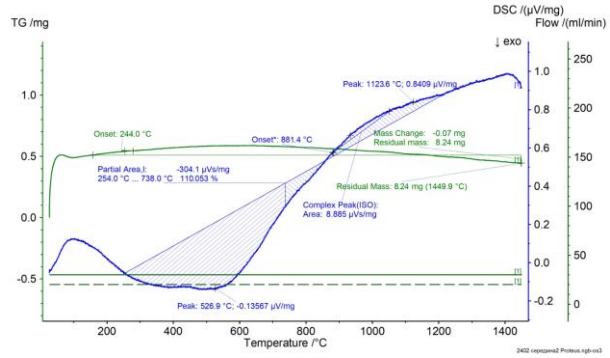
Thermal analysis by DSC/DTG provides information that is not accessible through SEM/XRD analysis, namely: the temperatures at which structural transformations begin, thermal markers of phase rearrangements, high-temperature anomalies, temperature indicators of local microzones/defects that manifest only at elevated temperatures, as well as the solidus–liquidus interval of alloys.

The aim of the thermal analysis using differential scanning calorimetry (DSC/DTG) was to determine the thermo-microstructural properties of the triple VAR Ti-10V-2Fe-3Al ingot, including the identification of temperature transformation gradients along the ingot height.

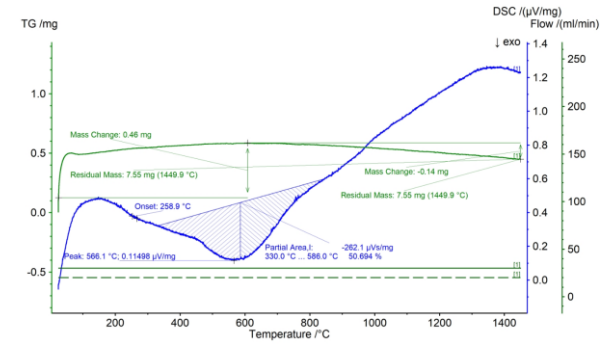
Figure 5 presents the results of the thermal analysis, including DSC/DTG thermograms of the Ti-10V-2Fe-3Al ingot.



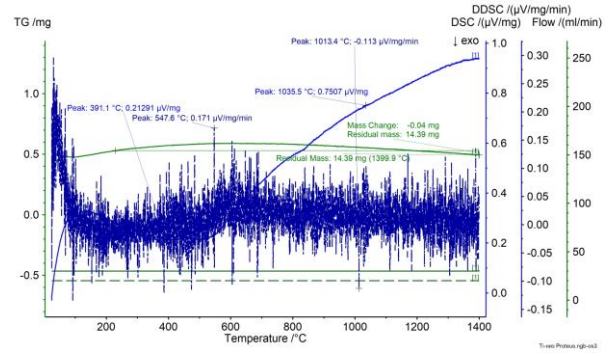
a - “middle 1” zone



b - “middle 2” zone



c – “top” zone



d – “bottom” zone

Figure 5 – DSC/DTG thermograms along the height of the Ti-10V-2Fe-3Al ingot for different zones

Table 1 presents the results of the thermal analysis of the zones of the industrial Ti-10V-2Fe-3Al ingot.

Table 1 – Summary thermal analysis of the zones of the industrial Ti-10V-2Fe-3Al ingot (triple VAR)

Zone	T, peak (°C)	Phase interpretation	Structural mechanism and diagnostic significance
Middle-1	685.3	Stage-wise decomposition of metastable β	Precipitation of dispersed α/ω phases; reflects initial microsegregation of β-stabilizers
	764.7	α coarsening	Redistribution of V and Fe; indicator of β-matrix homogeneity
	914.3	Onset of α→β transformation	Dissolution of secondary α; marker of transition toward the β-region
	1352.2	β stabilization	Completion of solid-state homogenization; indicator of steady-state remelting
	1495.8	Solidus	Onset of partial melting of segregated domains; sensitive indicator of microsegregation
	1547.7	Main melting stage	Increase in liquid phase fraction; reflects compositional distribution
	1610	Liquidus	Completion of melting; melting interval ΔT characterizes chemical homogeneity
Middle-2	526.9	Decomposition of metastable β	Precipitation of ω/early α; indicator of local heterogeneity
	975	Onset of precipitate dissolution	α→β transition; degree of phase stability
	1123	Peak of α→β transformation	Stabilization of β-matrix; reflects structural energy rearrangement
Top	566.1	Solid-state β decomposition	Dispersed α precipitation; influence of final remelting stage
	975	α→β transition	Dissolution of precipitates; uniformity of phase structure
	1449	Upper limit of solid-state region	Stabilization before melting; absence of premature partial melting
Bottom	547.6	Enhanced β decomposition	Intensive α/ω precipitation
	975	Onset of α→β	Phase dissolution; broader transformation range
	1013.4	Multi-stage α→β transformation	Non-uniform dissolution; presence of local compositional domains
	1399.9	End of solid-state region	β stabilization; absence of melting up to 1400 °C

It was established that all zones of the ingot exhibit a characteristic two-stage thermal evolution typical of the β-metastable Ti-10V-2Fe-3Al alloy: decomposition of the metastable β phase (≈520–570 °C) and the endothermic α→β transformation (≈950–1120 °C). The analysis of the zones revealed the following:

The “middle-1” zone is characterized by the smoothest transitions and minimal staging, confirming a steady-state crystallization regime during triple VAR.

The “bottom” zone exhibits additional staging (≈1013 °C) and more pronounced exothermic effects, indicating increased microsegregation formed during the initial stage of remelting.

Only in the “middle-1” zone, the solidus (≈1496 °C) and liquidus (≈1610 °C) temperatures were identified, which made it possible to evaluate the melting interval and confirm the absence of macrosegregation along the ingot height.

Figure 6 shows the dependence of the relative enthalpy of phase transformations on the ingot height.

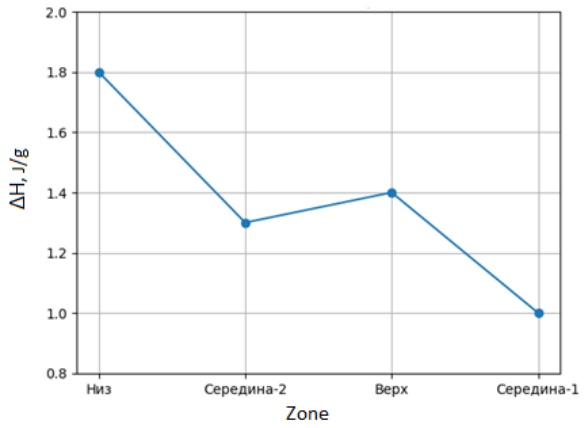


Figure 6 – Dependence of the relative enthalpy of phase transformations on the ingot height

It was established that the enthalpy of the exothermic decomposition of the β-matrix increases by approximately 60–80% in the lower zone of the ingot. The width of the phase transformation interval ΔT correlates with increasing microsegregation. The minimum values of ΔH and ΔT in the “middle-1” zone confirm a steady-state crystallization regime.

The thermograms demonstrate a clear dependence of the energetics of phase transformations on the position along the ingot height: the middle-1 zone shows minimum enthalpy and narrow transitions → maximum homogeneity; the bottom zone shows increased ΔH and broadened transitions → macro- and microsegregation, ΔH values for endothermic transformations along the ingot height Table 2.

Table 2 – ΔH values for endothermic transformations along the ingot height

Zone	ΔH, J/g	T_{α→β} onset, °C	T_{max α→β}, °C	$T_{solidus}$, °C	$T_{liquidus}$, °C
Bottom	1.7	~975	1013.4	—	—
Middle-2	1.4	~975	1123	—	—
Top	1.3	~975	—	—	—
Middle-1	1.0	914.3	1352.2	1495.8	1610

The “bottom” zone exhibits the maximum relative enthalpy of the endothermic transition ($\Delta H_{en}d_o$ ≈ 1.7 J/g), which is associated with a more pronounced staging of the α→β transformation and increased microsegregation of β-stabilizers.

The “middle-2” zone is characterized by a pronounced α→β transition peak at 1123 °C ($\Delta H_{en}d_o$ ≈ 1.4 J/g), reflecting active dissolution of precipitates and redistribution of alloying elements.

The “top” zone shows a less pronounced endothermic effect ($\Delta H_{en}d_o$ ≈ 1.3 J/g), corresponding to a more uniform phase structure after the final stage of remelting.

The “middle-1” zone demonstrates the minimum $\Delta H_{en}d_o$ ≈ 1.0 J/g and a smooth transformation behavior. It is also the only zone where the melting interval (1495.8–1610 °C) is recorded, allowing evaluation of the solidus–liquidus range and confirming high chemical homogeneity.

The enthalpy of the endothermic α→β transition decreases from the lower part of the ingot toward the steady-state crystallization zone (middle-1), correlating with a reduction in microsegregation parameters obtained from SEM–EDS (ΔC_{max}, σC, Lcorr).

An increase in microsegregation directly correlates with a rise in the enthalpy of phase transformations and a widening of temperature

intervals, confirming the consistency between SEM–EDS and DSC/DTG approaches.

Thus, thermal analysis confirms the absence of a pronounced vertical gradient in structural stability and can be used as a validation criterion for the integral electrode quality index.

Thermal verification (DSC)

To validate the I_{chem} index (which integrates the contribution of β-stabilizers and oxygen), a thermal component is introduced:

– presence/intensity of the β-phase decomposition transition (≈520–570 °C), staging/broadening of the α→β transformation (≈950–1123 °C), and the appearance of an additional marker (~1013.4 °C) as an indicator of large-scale heterogeneity (L_{corr}↑);

– for the "middle-1" zone — control of the solidus–liquidus interval as an integral indicator of homogeneity;

– confirmation of SEM–EDS results using DSC–DTG data: the higher the macro- and microsegregation (ΔC_{max}, σC, Lcorr), the wider the melting interval $\Delta T\_melt = T_{liquidus} - T_{solidus}$ due to the presence of local compositions with different melting temperatures. The observation of a well-defined solidus–liquidus pair and the absence of premature melting in other zones within 1400–1450 °C confirm the absence of severe macrosegregation and the correctness of the triple VAR regime.

A "thermal indicator" has been developed: the number of stages/peaks in the α→β transition range, the transition width ΔT, and the presence of early melting or expansion of ΔT_{melt}.

Thus, SEM–EDS determines the cause (spatial profile of chemical inhomogeneity), while DSC–DTG captures the consequence (thermodynamic and kinetic response of the structure). The integration of these methods enables the transition from qualitative assessment to a verifiable quality control protocol for industrial electrodes and ingots.

Conclusion

Thus, for the first time, a quantitative correlation between SEM–EDS profiles and DSC characteristics has been proposed. Thermal analysis is suggested as an independent validator of microsegregation.

Triple vacuum arc remelting ensures high chemical homogeneity of the main alloying elements (Ti, V, Al, Fe), as confirmed by SEM–EDS through the absence of a pronounced vertical gradient.

The results of thermal analysis (DSC/TG) confirm the SEM–EDS findings regarding the absence of a significant gradient along the ingot height. It has been established that the intensification and staging of β-phase decomposition and α→β transformation effects are characteristic of the lower zone of the ingot, which is consistent with increased microsegregation parameters (ΔC_{max}, σC, Lcorr).

A methodology for microstructural and thermal verification of the quality of an industrial Ti-10V-2Fe-3Al triple VAR ingot has been developed using DSC–DTG and SEM–EDS analyses. This approach is based on quantitative evaluation of microsegregation through SEM–EDS profiles using the parameters ΔC_{max}, σC, Lcorr, ΔC_o (local), as well as the application of the integral index I_{chem} and the threshold I^{crit}_{chem} for electrode quality control.

Conflict of interest. On behalf of all the authors, the corresponding author declares that there is no conflict of interest.

CRediT author statement: **A. Mamutova**: Conceptualization, Methodology, experiments, Funding; **T. Chepushtanova**: Data curation, Writing draft preparation, Investigation; **B. Mishra**: Supervision, Reviewing and Editing.

Acknowledgement. The research was carried out with the financial support of the agreement № 1090-ТМК from 02.11.2025 / 11-168 from 19.11.2025 г. of Ust-Kamenogorsk Titanium and Magnesium Plant JSC.

Cite this article as: Mamutova AT, Chepushtanova TA, Mishra B. Development of a methodology for microstructural and thermal verification of the quality of an industrial Ti-10V-2Fe-3Al triple vacuum arc remelted ingot. Kompleksnoe Ispolzovanie Mineralnogo Syra = Complex Use of Mineral Resources. 2028; 344(1):79-89. https://doi.org/10.31643/2028/6445.08

Ti-10V-2Fe-3Al титан қорытпасының үш мәрте вакуумды-доғалық қайта балқыту арқылы алынған өнеркәсіптік құймасының сапасын микрокұрылымдық және термиялық верификациялау әдістемесін әзірлеу

[1]Мамутова А.Т., [2]Чепуштанова Т.А., [3]Mishra B.

[1] Өскемен титан-магний комбинаты АҚ, Қазақстан
[2] Сәтбаев университеті, Алматы, Қазақстан
[3] Вустер политехникалық институты, Вустер, АҚШ

Мақала келді: *5 наурыз 2026*
Сараптамадан өтті: *16 наурыз 2026*
Қабылданды*: 6 мамыр 2026*

ТҮЙІНДЕМЕ

Мақала «Өскемен титан-магний комбинаты» АҚ-да алынған Ti-10V-2Fe-3Al қорытпасының үш мәрте вакуумды-доғалық қайта балқытудан кейінгі өнеркәсіптік құймасының сапасын микрокұрылымдық және термиялық верификациялау әдістемесін әзірлеуге арналған. Құйманың барлық аймақтары β-метатұрақты Ti-10V-2Fe-3Al қорытпасына тән екі сатылы термиялық эволюция сипаттамасын көрсететіні анықталды: метастабильді β фазаның ыдырауы (≈520–570 °C) және эндотермиялық α→β фазалық қайта құрылуы (≈950–1120 °C). β-матрицаның экзотермиялық ыдырау энтальпиясы құйманың төменгі аймағында шамамен 60–80%-ға артатыны анықталды. Фазалық ауысу ені ΔT микросегрегацияның артуымен корреляцияланады. Эндотермиялық α→β ауысуының энтальпиясы құйманың түбінен тұрақты күйдегі кристалдану аймағына (Middle-1) дейін төмендейтіні анықталды, бұл SEM-EDS профильдеріне сәйкес микросегрегация параметрлерінің төмендеуімен корреляцияланады (ΔC_max, σC, Lcorr). Бұл SEM–EDS профильдері бойынша алынған микросегрегация параметрлерінің (ΔC_max, σC, Lcorr) төмендеуімен сәйкес келеді. Қортындылай келе, алғаш рет SEM–EDS профильдері мен DSC–DTG сипаттамалары арасында сандық корреляция ұсынылды, ал термиялық талдауды микросегрегацияның тәуелсіз валидаторы ретінде пайдалану ұсынылды SEM–EDS профильдері негізінде микросегрегацияны ΔCmax, σC, L_{corr} және ΔCO(лок.) параметрлері арқылы сандық бағалау тәсілі әзірленді. Сонымен қатар, термиялық талдау нәтижелері негізінде электродтардың сапасын бақылау үшін интегралдық көрсеткіш $I_{che}m$ және шекті мән I^{crit}_{chem} қолдану ұсынылды.

Түйін сөздер: Ti-10V-2Fe-3Al үш мәрте қайта балқытылған қорытпасы, вакуумды-доғалық балқыту, DSC–DTG және SEM–EDS талдаулары, α/β фазалық ауысулар, термиялық ыдырауды талдау.

Информация об авторах:

Мамутова Асем Тлековна — *Өскемен титан-магний комбинаты АҚ президенті, Өскемен; докторант, Тау-кен металлургия институты, Сәтбаев университеті, Алматы, Қазақстан. Email: a.mamutova@satbayev.university*

Чепуштанова Татьяна Александровна — *Профессор, техника ғылымдарының кандидаты, PhD докторы, Металлургиялық процестер, жылутехника және ұнтақты металлургия» зертханасының меңгерушісі, МиОПИ кафедрасы, Тау-кен металлургия институты, Satbayev University, Алматы, Қазақстан. Email: t.chepushtanova@satbayev.university*

Mishra Brajendra — *Профессор, PhD, NSF ресурстарды алу және қайта өңдеу орталығының директоры, Вустер политехникалық институты, Вустер, АҚШ. Email: bmishra@wpi.edu*

Разработка методики микроструктурной и термической верификации качества промышленного слитка Ti-10V-2Fe-3Al тройного вакуумно-дугового переплава

[1]Мамутова А.Т., [2]Чепуштанова Т.А., [3]Mishra B.

[1] АО Усть-Каменогорский титано-магниевый комбинат, Казахстан
[2]Satbayev University, Алматы, Казахстан
[3] Вустерский политехнический институт, Вустер, США

Поступила: *5 марта 2026*
Рецензирование: *16 марта 2026*
Принята в печать: *6 мая 2026*

АННОТАЦИЯ

Статья посвящена разработке методики микроструктурной и термической верификации качества промышленного слитка Ti-10V-2Fe-3Al тройного вакуумно-дугового переплава АО УК ТМК. Установлено, что все зоны слитка демонстрируют характерную для β-метастабильного сплава Ti-10V-2Fe-3Al двухстадийную термическую эволюцию: распад метастабильной фазы β (≈520–570 °C), эндотермическая α→β перестройка (≈950–1120 °C). Установлено, что энтальпия экзотермического распада β-матрицы увеличивается на ~60–80% в нижней зоне слитка. Ширина фазового перехода ΔT коррелирует с увеличением

	микросегрегации. Установлено, что значение энтальпии эндотермического α→β перехода уменьшается от нижней части слитка к зоне установившейся кристаллизации (Середина-1), что коррелирует со снижением параметров микросегрегации по SEM-EDS профилям (ΔC_max, σC, L_{corr}). Таким образом, термический анализ подтверждает отсутствие выраженного вертикального градиента структурной стабильности и может использоваться как валидирующий критерий интегрального индекса качества электрода. Таким образом, впервые предложена количественная корреляция SEM-EDS профилей с DSC-DTG характеристиками, предложено термический анализ использовать как независимый валидатор микросегрегации. Разработан подход к количественной оценке микросегрегации по профилям SEM–EDS через параметры ΔCmax, σC, L_{corr} и ΔCO(лок.) и предложено использовать интегральный показатель I_{chem} и порог I^{crit}_{chem} для контроля качества электродов с помощью результатов термического анализа.
	Ключевые слова: сплав тройного переплава Ti-10V-2Fe-3Al, вакуумно-дуговая плавка, DSC-DTG и SEM–EDS анализы, α/β- фазовые переходы, анализ термического разложения.
Мамутова Асем Тлековна	***Информация об авторах:*** *Президент АО Усть-Каменогорского титано-магниевого комбината, Усть-Каменогорск; докторант, Горно-металлургический институт, Satbayev University, Алматы, Казахстан. Email: a.mamutova@satbayev.university*
Чепуштанова Татьяна Александровна	*Профессор, кандидат технических наук, доктор PhD, заведующая лабораторией Металлургические процессы, теплотехника и порошковая металлургия, кафедра МиОПИ, Горно-металлургический институт, Сәтбаев университеті, Алматы, Қазақстан. Email: t.chepushtanova@satbayev.university*
Mishra Brajendra	*Профессор, PhD, директор Центра извлечения и переработки ресурсов NSF, Вустерский политехнический институт, Вустер, США. Email: bmishra@wpi.edu*

References

[1] Banerjee D, Williams JC. Perspectives on titanium science and technology. Acta Materialia. 2013; 61(3): 844-879. https://doi.org/10.1016/j.actamat.2012.10.043

[2] Lutjering G, Williams JC. Titanium. Springer. 2007. https://doi.org/10.1007/978-3-540-73036-1

[3] Peters M, Kumpfert J, Ward CH, and Leyens C. Titanium Alloys for Aerospace Applications. Adv. Eng. Mater. 2003; 5:419-427. https://doi.org/10.1002/adem.200310095

[4] Kolli RP, Devaraj A. A Review of Metastable Beta Titanium Alloys. Metals (MDPI). 2018; 8(7):506. https://doi.org/10.3390/met8070506

[5] Banerjee D, Williams JC. Perspectives on Titanium Science and Technology. Acta Mater. 2013; 61:844–879. https://doi.org/10.1016/j.actamat.2012.10.043

[6] Mamutova AT, Chepushtanova TA, Mishra B. Thermodynamic prediction of the technological conditions for producing the Ti-10V-2Fe-3Al alloy by vacuum arc remelting. The Mining Journal of Kazakhstan. 2026; 1(249):55-60. https://doi.org/10.48498/minmag.2026.249.1.006

[7] Jing Z, Liu R, Geng N, Wang Y, Sun Y. Simulation of Solidification Structure in the Vacuum Arc Remelting Process of Titanium Alloy TC4 Based on 3D CAFE Method. Processes. 2024; 12(4):802. https://doi.org/10.3390/pr12040802

[8] Liu X, Zhang Y, Li H, Wang J, Chen L. The morphology features, formation mechanism and elimination of channel segregation in industrial-scale Ti–Nb ingots produced by vacuum arc remelting. Journal of Materials Research and Technology. 2023; 23:2781–2793. https://doi.org/10.1016/j.jmrt.2023.11.009

[9] Liu H, Wang L, Zhang Z. Effects of homogenization heat treatment on Fe microsegregation in Ti-1023 titanium alloy. Materials. 2023; 16(14):4892.

[10] TA Instruments. Determination of beta transus temperature in titanium alloys by DSC. TA Application Note TA131. 2018.

[11] Xumei Yang, Hongzhen Guo, Zhanglong Zhao, Yongquan Ning, Shichong Yuan, Shewei Xin. Quantitative analysis of the effect of deformation temperature on microstructure evolution and mechanical property of isothermally forged BT25y titanium alloy. Procedia Engineering. 2017; 207:2167-2172. https://doi.org/10.1016/j.proeng.2017.10.976

[12] Boyer RR. An Overview on the Use of Titanium in the Aerospace Industry. Materials Science and Engineering: A. 1996; 213:103-114. https://doi.org/10.1016/0921-5093(96)10233-1

[13] Zhao Q, et al. High-strength titanium alloys for aerospace engineering applications: A review on melting-forging process. Materials Science and Engineering A. 2022; 845:143260. https://doi.org/10.1016/j.msea.2022.143260

[14] Guo Jie, Huang Liqing, Wu Jingyang, LI Junjie, Wang Jincheng, Fan Kai. Evolution of Macrosegregation During Three-Stage Vacuum Arc Remelting of Titanium Alloys. Acta Metall Sin. 2024; 60(11):1531-1544. https://doi.org/10.11900/0412.1961.2022.00544

[15] Semiatin SL, Bieler TR. Effect of texture and slip mode on the anisotropy of plastic flow and flow softening during hot working of Ti-6Al-4V. Metallurgical and Materials Transactions A. 2001; 32:1787–1799. https://doi.org/10.1007/s11661-001-0155-1

[16] Semiatin SL, Seetharaman V, and Weiss I. in Advances in the Science and Technology of Titanium Alloy Processing, I Weiss, R Srinivasan, PJ Bania, D Eylon, and SL Semiatin, eds, TMS, Warrendale, PA. 1997, 3–73.

[17] Lütjering G. Influence of processing on microstructure and mechanical properties of (α+β) titanium alloys. Materials Science and Engineering A. 1998; 243:32–45. https://doi.org/10.1016/S0921-5093(97)00778-8

[18] Qinyang Zhao, Qiaoyan Sun, Shewei Xin, Yongnan Chen, Cong Wu, Huan Wang, Jianwei Xu, Mingpan Wan, Weidong Zeng, Yongqing Zhao. High-strength titanium alloys for aerospace engineering applications: A review on melting-forging process. Materials Science and Engineering A. 2022; 845:143260. https://doi.org/10.1016/j.msea.2022.143260

[19] Xiaoli Zhao, Shujun Li, Man Zhang, Yandong Liu, Timothy Sercombe, Shaogang Wang, Yulin Hao, Rui Yang, Lawrence E. Murr. Comparison of the microstructures and mechanical properties of Ti–6Al–4V fabricated by selective laser melting and electron beam melting. Materials & Design. 2016; 95:21-31. https://doi.org/10.1016/j.matdes.2015.12.135

[20] Qiu C, Adkins NJE, Attallah MM. Microstructure and tensile properties of selectively laser-melted and HIPed Ti-6Al-4V. Materials Science and Engineering A. 2013. https://doi.org/10.1016/j.msea.2013.04.099

[21] Boivineau M, Cagran C, Doytier D, Eyraud V, Nadal M-H, Wilthan B, Pottlacher G. Thermophysical Properties of Solid and Liquid Ti-6Al-4V (TA6V) Alloy. International Journal of Thermophysics. 2006; 27(2):507–529. https://doi.org/10.1007/PL00021868

[22] Joakim Karlsson, Anders Snis, Håkan Engqvist, Jukka Lausmaa. Characterization and comparison of materials produced by Electron Beam Melting (EBM) of two different Ti–6Al–4V powder fractions. Journal of Materials Processing Technology. 2013; 213(12):2109-2118. https://doi.org/10.1016/j.jmatprotec.2013.06.010