

Extended abstract: Advanced processing methods for efficient flotation of Ti- and V- bearing ores

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1. Introduction

Titanium and Vanadium are listed as critical raw materials in the EU Critical Raw Materials Act (European Parliament and Council, 2024). Both metals are used in many high-performance applications, including wind turbines, airplanes, and nuclear applications. Regardless of their strategic importance in advanced technologies, they have received less attention compared to, e.g., battery minerals or rare-earth elements. Additionally, there is no Ti metal or V production currently in Europe. Although many untapped V-bearing titanomagnetite deposits in Europe, almost all remain unexploited due to their complex mineralogy and the consequent techno-economic feasibility.

To enhance the feasibility of extraction for such ore bodies in Europe, this study investigates advanced processing methods applied to two different Ti- and V-bearing ores. The selected methods were inert atmosphere grinding (N or CO₂) and combined microflotation (CMF). These methods were chosen because both have demonstrated improved flotation performance in other ore types. Maksimainen et al. (2010) reported improved PGM grades and recoveries for a low sulfur-containing ore, where the proposed mechanism for improved PGM flotation performance was depression of silicate gangue minerals. Similarly, Rulyov (2024) demonstrated significant improvements in Cu recovery through the use of combined microflotation in the flotation of Cu containing tailings.

These advanced methods were applied for two different ores in a flotation column set-up, both independently and together. Their performance was compared against a general benchmark procedure with conventional grinding, and flotation.

2. Materials and Methods

The experimental aspects of the study consisted of two separate campaigns, where two different Ti- and V-bearing ores were processed. A detailed description of the processed ore samples, reagents, and other methods is presented in the following chapters.

2.1 Ore samples and preparation

Applied mineralogical studies were conducted on the two ore types, and details are provided in Table 1. Notably, the Ti in Ore 1 is mostly found in fine ilmenite exsolution lamellas (typically 1-20 µm) within magnetite (Fig 1). In contrast, Ore 2 contains not only lamellae but also larger, granular inclusions..

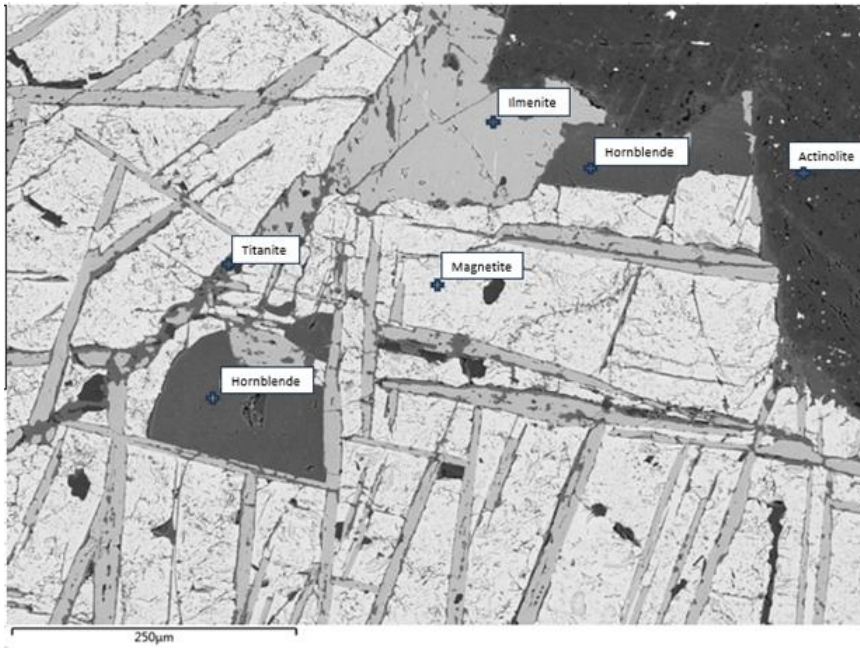


Figure 1. Backscattered image showing the complex exsolution texture of ilmenite in magnetite in Ore 1.

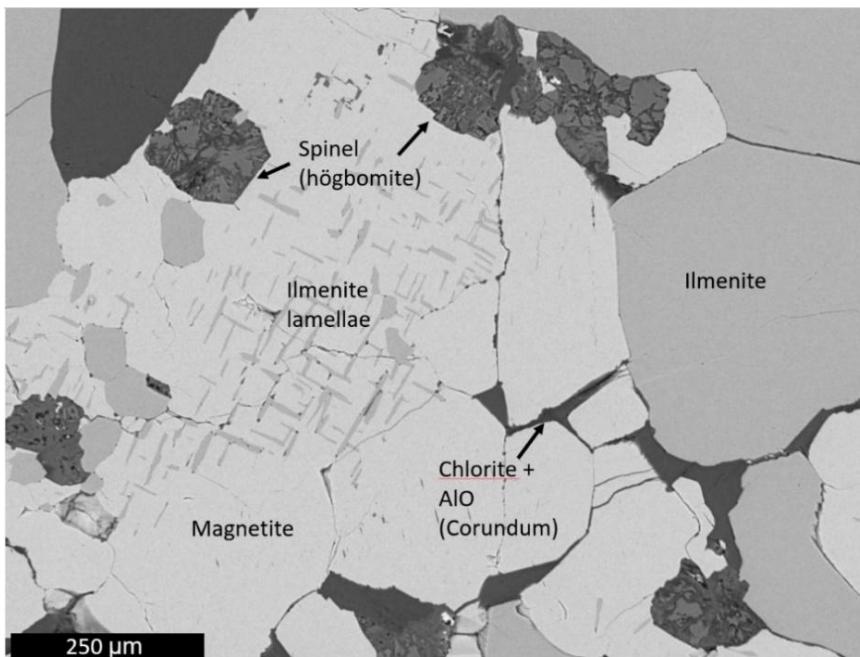


Figure 2. Backscattered image showing both ilmenite lamellae in magnetite and granular grains of ilmenite in Ore 2.

Table 1. Properties of the ore samples

Ore name	Ti (%)	V (%)	Major minerals
Ore 1	2.03	0.22	Amphiboles, Pyroxenes, Magnetite, Hematite, Ilmenite, Epidote, Chlorite, Titanite
Ore 2	6.15	0.31	Amphiboles, Pyroxenes, Plagioclase, Epidote, Magnetite, Ilmenite, Hematite, Quartz, Chlorite, Spinel

Both ore samples were received as drill cores crushed to a nominal size of <2 mm using a laboratory-scale crushing circuit as illustrated in Figure 3.

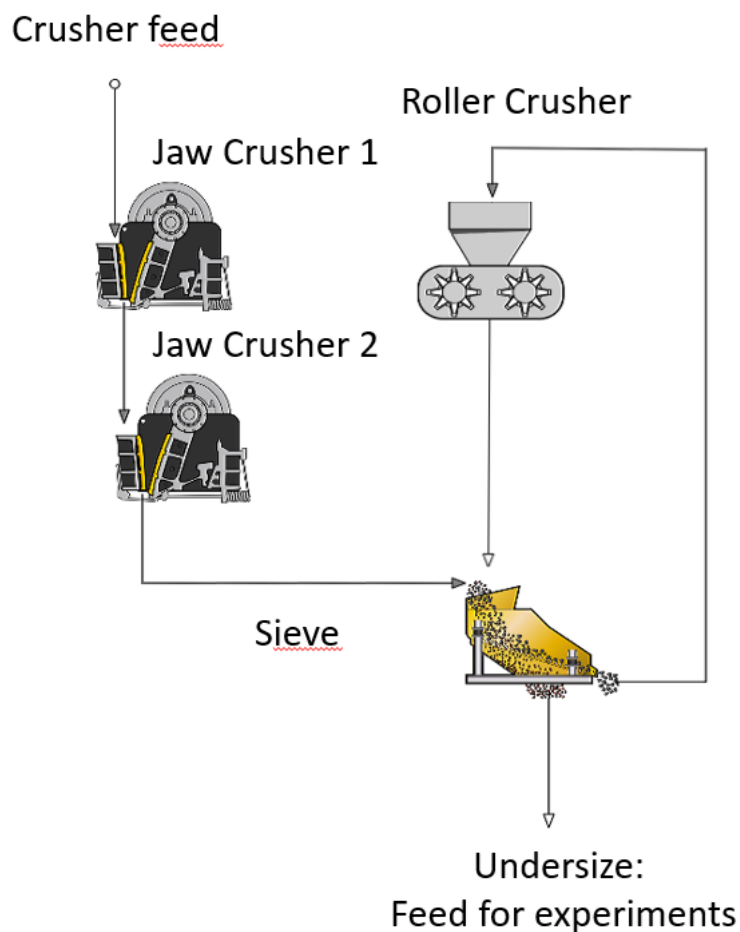


Figure 3: Crushing circuit used in the experiments

After crushing, the samples were divided into representative samples of 2.89 kg (Ore 1) and 2.48 kg (Ore 2). The sample splitting procedure was conducted using a Retsch PT600 rotary sampling device. The small variance in the representative sample mass between Ore 1 and Ore 2 was due to different total mass of the received drill core samples.

2.2 Grinding

The samples were ground in a Mergan ball mill using a ball charge of 22.5 kg.

The grinding was conducted either conventionally, in N, or in CO₂ atmosphere. If applied, N and CO₂ gas flow was initially applied to a bucket containing the water that would be added to the grinding mill. The gas tube was inserted to the bottom of the bucket while keeping a lid on the bucket. The water was treated for 15 minutes in the bucket, after which the oxygen levels observed reached a plateau at around 1.3-1.5 mg/l, while the initial oxygen levels were around 8.2-8.5 mg/l. After this initial 15-minute gas flushing, the water was promptly added to the grinding mill, already filled with the ore sample and grinding media. A final 5-minute gas flushing was done directly to the grinding mill by inserting a gas tube into the slurry and keeping the lid as closed as possible. After the final flushing, the gas tube was removed, and the lid was immediately sealed.

The grinding time and target d80 for Ore 1 were 87 minutes and 32 µm respectively. For Ore 2, two different grinding times and target d80 values were tested: 87 min with d80 of 41 µm and 60 minutes with d80 of 52 µm. The difference in grinding times was attributed to different mineral structures as described in Section 2.1.

2.3 Microbubble generator

The microbubbles were produced using a MBGen-001 microbubble generator (MBG), developed by TURBOFLOTSERVICE company (Kyiv, Ukraine). The nominal microdispersion flow rate was set at 0.9 ml/s.

2.4 Chemical reagents

The chemical reagents, their concentrations, and other parameters used for processing Ore 1 and Ore 2 are presented in Tables 2 and 3, respectively.

Table 2. Conditions used for Ore 1

Variable	Concentration / Level
pH	5.5
Collector 1: Sodium oleate (NaOL)	321 g/t
Collector 2: 1-Dodecyl phosphinate acid (1-DPA)	264 g/t
Depressant: Acidified water glass (AWG)	42 g/t
Frother: NasFroth 240 (NF240)	20 ppm

Table 3. Conditions used for Ore 2

Variable	Concentration / Level
pH	4.5 5.5
Collector 1: Tall Oil (TO)	1070 g/t
Collector 2: Light Fuel Oil (LFO)	1556 g/t
Collector 3: Berol 050 (Berol)	101 g/t
Activator: Sodium persulfate (SPS)	22
Depressant: Acidified water glass (AWG)	42 g/t
Frother Mix1: NasFroth 240 (NF240) + DowFroth 250 (DF250)	50 + 20 ppm
Frother 2: NasFroth 350 (NF350)	40 ppm

For the microbubble generator (MBG), NF350 was used at a concentration of 400 ppm to minimize the coalescence of the microbubbles. It is important to note that only a small amount of additional frother was added to the column system along with the microbubbles.

2.5 Flotation

The flotation experiments were conducted in a self-built laboratory-scale flotation column ($d = 0.5$ dm, $h = 23.25$ dm, $V_{\text{nominal}} = 4.57$ dm³). Each experiment followed the same procedure, and flotation was conducted in two phases (illustrated in Figures 4a and 4b). The experimental procedure followed these steps:

1. Grinding of mineral sample (conventional/N-/CO₂-Atmosphere)
2. Preparation of ground mineral sample for column flotation experiments
3. Conditioning
 - a. Add depressant, wait 3 minutes
 - b. Add activator (if applicable), wait 3 minutes
 - c. Add collectors, wait 3 minutes
 - d. Add frother(s), wait 3 minutes
4. Flotation Phase 1, conventional column flotation
5. Wait for steady state
6. Sampling of flotation Phase 1
7. Starting flotation Phase 2 by connecting and turning on the MBG
8. Stopping recirculation of fractions to avoid accumulation of microbubbles
9. Wait for steady state
10. Sampling of flotation Phase 2
11. Drying and splitting samples for analyses

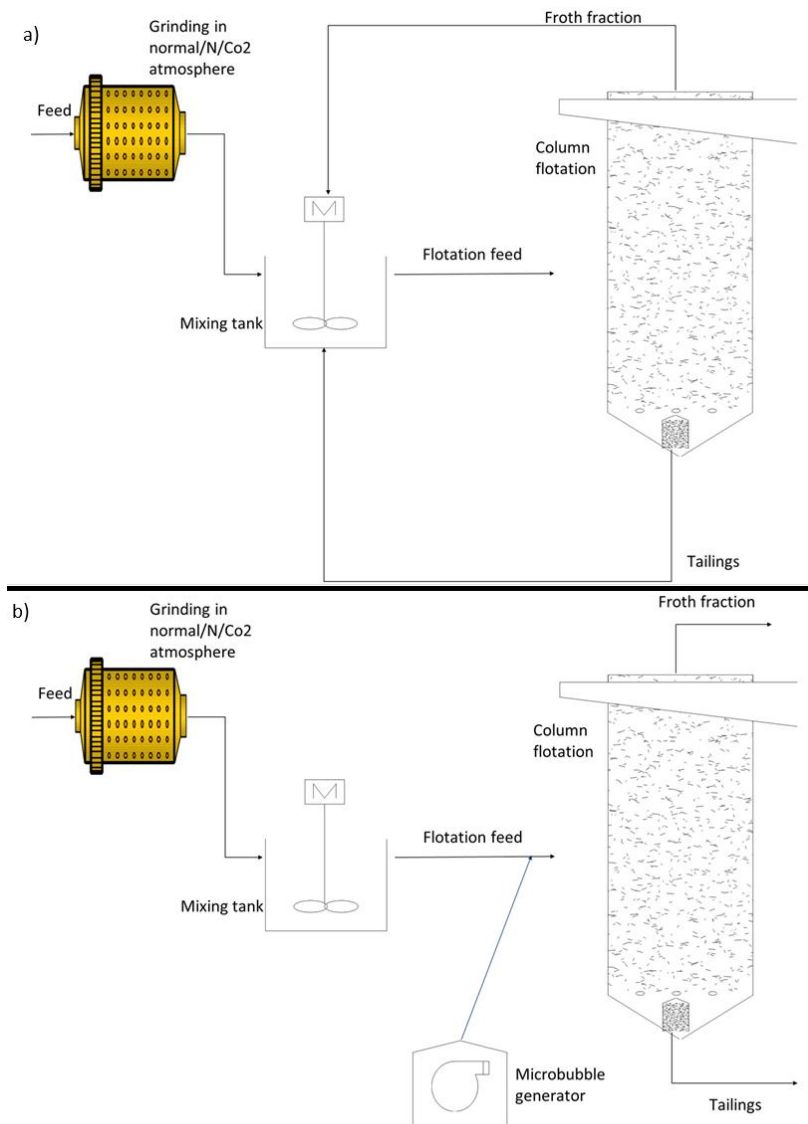


Figure 4. Flow-sheet illustration of column flotation a) phase 1 and b) phase 2

The sampling was conducted after allowing an apparent steady state: 30-40 minutes for phase 1 and 20-30 minutes for phase 2. Both the froth and tailings fractions were sampled simultaneously for the same duration, which enabled calculating flow rates and ensured that the samples were comparable for analyses. After sampling, all samples were initially dried through filtration. The resulting filtration cakes were completely dried by placing the samples on top of a hot plate and under a heat lamp, while occasionally mixing to prevent the formation of agglomerates. A representative sub-sample of each fraction was then sent to elemental analysis, which was performed using Energy Dispersive XRF (ED-XRF).

3. Results and discussion

The results of the column flotation experiments are presented in Table 4 (Ore 1) and Table 5 (Ore 2). These tables include data on mass pull, Ti recovery, and grade. It is noteworthy that V is usually

separated along with magnetite through magnetic separation; therefore, V results are not discussed in this study.

Table 4. Flotation results for Ore 1

Experiment	Mass Pull	Ti Recovery	Ti Grade
1.1 Conventional grinding, No MBG	59.75 %	65.83 %	2.25 %
1.2 Conventional, MBG enabled	60.74 %	68.45 %	2.19 %
2.1 Grinding in N, No MBG	56.88 %	63.78 %	2.68 %
2.2 Grinding in N, MBG enabled	49.00 %	57.84 %	2.71 %
3.1 Grinding in CO ₂ , No MBG	48.58 %	65.27 %	2.65 %
3.2 Grinding in CO ₂ , MBG enabled	48.29 %	65.84 %	2.92 %

Table 5. Flotation results for Ore 2

Experiment	Mass Pull	Ti Recovery	Ti Grade
1.1 Grinding in CO ₂ (87 min), NF240+DF250 (50+20 ppm), No MBG	13.21 %	39.69 %	16.05 %
1.2 Grinding in CO ₂ (87 min), NF240+DF250 (50+20 ppm), MBG enabled	26.15 %	58.35 %	16.70 %
2.1 Conventional grinding (60 min), NF350 (40 ppm), No MBG	62.11 %	79.39 %	10.76 %
2.2 Conventional grinding (60 min), NF350 (40 ppm), MBG enabled	63.75 %	78.77 %	11.71 %
3.1 Grinding in CO ₂ (60 min), NF350 (40 ppm), No MBG	32.74 %	54.29 %	15.13 %
3.2 Grinding in CO ₂ (60 min), NF350 (40 ppm), MBG enabled	38.29 %	52.23 %	12.42 %

The results show that flotation of such complex Ti-V ores is quite challenging. As mentioned in Section 2.1, the fine exsolution lamellae in magnetite range from 1 μm to some dozens of μm , making their liberation through conventional comminution methods difficult. This is particularly evident in the results of Ore 1, where only minimal improvement in Ti grade was obtained. In contrast, Ore 2 demonstrated a more favorable mineral structure, resulting in a more significant increase in Ti grade was obtained with alternating results for Ti recovery.

The advanced processing methods applied for Ti flotation demonstrated varying levels of improved performance. In all cases, grinding in inert atmosphere led to a better selectivity for Ti. This was reflected as better Ti grade, and especially a smaller mass pull. However, in some cases with Ore 2, the recovery of Ti was also significantly decreased.

The addition of the microbubbles improved recovery, grade, or both in nearly all experiments. At best, there was a significant increase in recovery along with a slight improvement in grade (Ore 2, 1.1 vs. 1.2). However, in the worst case, both grade and recovery decreased, when microbubbles were applied (Ore 2, 3.1 vs. 3.2). According to Rulyov (2024), the optimum microbubble dosage is dependent on both particle size and density. Considering that only particle size varies between Ore 2 experiments 1 and 3, it is likely that better results could be obtained by modifying the microbubble flow.

4. Conclusions

This study confirmed that advanced processing methods, which have shown success with other mineral types, are also applicable for Ti- and V- bearing ores. The initial results showed positive outcomes for both inert atmosphere grinding and combined microflotation. Inert atmosphere grinding consistently demonstrated higher Ti grade, though its impact on Ti recovery varied. The use of combined microflotation generally enhanced recovery rates, with significant improvements observed in the best-performing experiments. However, the variability in results implies that the microbubble dosage may not have been optimal in all experiments.

Although the results presented in this study are promising, further optimization is required to fully harness the positive effects of these methods for processing Ti- and V-bearing ores.

Acknowledgements

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