



Slovak Dolomite as a Raw Material Source for Metal Magnesium Production

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ABSTRACT

In order to ensure self-sufficiency and a stable supply of essential mineral raw materials within the EU, the European Raw Materials Alliance (ERMA) was established. One of its key objectives is to secure access to sustainable raw materials and support the exploration and mining of these materials within the EU. Metallic magnesium has been included on the list of critical minerals for EU countries since 2011. The most suitable raw materials for Mg production by the silicothermic reduction method are dolomite or magnesite, and the Slovak Republic has considerable resources of these carbonate raw materials. For technological research, six samples of dolomite from different deposits were selected. The samples were annealed at selected temperatures and characterized by differential thermal analysis (DTA), X-ray diffraction (XRD), and chemical analyses. Results published in the conference paper by Danková et al. (2025) showed that for the silicothermic reduction of magnesium, it is necessary to verify the calcination conditions for each sample individually and determine the influence of hydration activity or active sites in their structure to increase magnesium reduction. The selected calcined dolomite samples were subjected to repeated DTA/TG analysis after a two-month interval to determine their hydration. Based on these results, the dolomite sample designated as ST-1, calcined under specified conditions, was used for the laboratory experiment of silicothermic reduction of magnesium. The resulting product was analyzed by SEM/EDX, which detected a high ratio of metallic magnesium (in at. %).

1. Introduction

Magnesium metal is characterized by easy heat dissipation, certain ductility, and high chemical reactivity and has a special status between 26 elements and minerals defined as “critical raw materials for the EU” (Report on Critical Raw Materials for the EU, 2014). For many decades, magnesium has been used as an

“industrial metal” for light weighting in structural applications, in addition to other traditional applications such as aluminum alloying, steel desulfurization and protective anodes. In the last decade, magnesium has shown significant potential to become a “technology metal” in a variety of new applications from biomedical devices to energy storage/battery products (Luo, 2025).

Metal magnesium is produced commercially through

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the electrolytic process or the silicothermic process, also known as the Pidgeon process.

The electrolytic process extracts molten magnesium from molten magnesium chloride, while the silicothermic process involves heating calcined magnesium ores (like dolomite or magnesite) with a reducing agent, such as ferrosilicon, in a vacuum chamber. The raw materials for magnesium production include seawater, salt brines, and solid minerals like dolomite and magnesite, which contain magnesium in various forms.

Among the Mg resources, dolomite is the most important resource for primary Mg metal production. At present, approximately 85 % of the global primary Mg metal is produced from dolomite using the Pidgeon process in China (Wu et al., 2021; Zhang et al., 2024).

The Pidgeon process took root in China in the late 1990s and has undergone 30 years of prosperous development and technological innovations. Addressing the deficiencies of the Pidgeon process has prompted the gradual emergence of innovative principles and processes. Several studies have been conducted to resolve the draw-backs of the commercial Mg production processes.

Most of them focused on the aspects of an eco-friendly and efficient Mg production. How to economically produce metallic magnesium using low-grade magnesite was described by Huang et al. (2025).

In their work, Li et al. provided fundamental data support and theoretical guidance for achieving energy efficiency, carbon reduction in magnesium smelting, and the industrial adoption of innovative processes (Li et al., 2025).

Lee et al. considered the solid oxide membrane (SOM) process to be promising due to its environmental-friendly Mg production and their study investigated a novel electrolytic process using a liquid tin cathode (Lee et al., 2021). Liquid-metal-electrode-assisted electrolysis for the production of sodium and magnesium was also studied by Guo et al. (2025).

Jeoung et al. realized the preparation of high-purity Mg metal from calcined dolomite using molten salt electrolysis and vacuum distillation (Jeoung et al., 2023).

The conference paper by Danková et al. presented at the XVI IMPRC 2025 deals with the procedures and conditions to prepare intermediate products from domestic dolomite suitable for magnesium metal preparation using silicothermic process (Danková et al., 2025). The individual procedures and analyses leading to these results are supplemented and discussed. Also, the stability of calcined intermediates and their hydration was studied. Finally, experiment of magnesium metal reduction in laboratory conditions was realized and

obtained product was characterized by SEM/EDX analyses.

2. Experimental

2.1. Raw samples

The dolomite in Slovakia occurs in several Middle and Upper Triassic formations thick up to several 100 meters, or forming intercalations, interbeds, lenses in beds irregularly alternating with surrounding limestones. They are present in numerous geological units, their cover sequences and tectonic nappes. The most significant are the Middle- and Upper-Triassic dolomites of the Hronic unit, bearing the important dolomite deposits in the Choč nappe of the Strážovská Highlands.

Dolomite raw samples from the Slovak localities: Sedlice (SED-1, SED-2), Trebejov (TR-1), Stráňavy (ST-1, ST-2) and Kľačany (KRA-1) were used for experimental purposes, Figure 1.

Bulk samples of dolomites were freely air-dried, then subjected to preparatory work consisting of crushing in three stages in jaw crushers and sorting on sieves of different sizes: + 8.0; 4.0 - 8.0; 2.0 - 4.0; 1.0 - 2.0; - 1.0 mm. All samples, or the grain fractions prepared from them were subsequently homogenized and quartered. From each raw sample after the preparatory works, as well as from the grain size classes, individual homogeneous parts were prepared for further laboratory processing, including their grain size characteristics (Danková et al., 2025).

2.2. Experimental procedures

Annealing tests of dolomite fractions were carried out in an electric laboratory furnace ELOP-1,200/15 at temperatures of 1,000 °C and 1,050 °C with a holding time of 0.5, 1, 2 and 2.5 hours. Optimized annealing tests of samples of fraction below 8.0 mm were carried out in an electric laboratory furnace KSL1600X-A2 at temperatures of 1,150, 1,200 and 1,300 °C with a duration of 1.5 hours.

The crucibles were placed in a muffle furnace and then the furnace was heated up continuously from room temperature to selected temperature (the heating rate was 10 °C/min) with selected holding time. The products of calcination obtained at different temperatures were taken out and stored in a desiccator before DTA/TG analyses were carried out (Danková et al., 2025).

2.3. Methods of characterization

Qualitative mineralogical analysis of input samples was

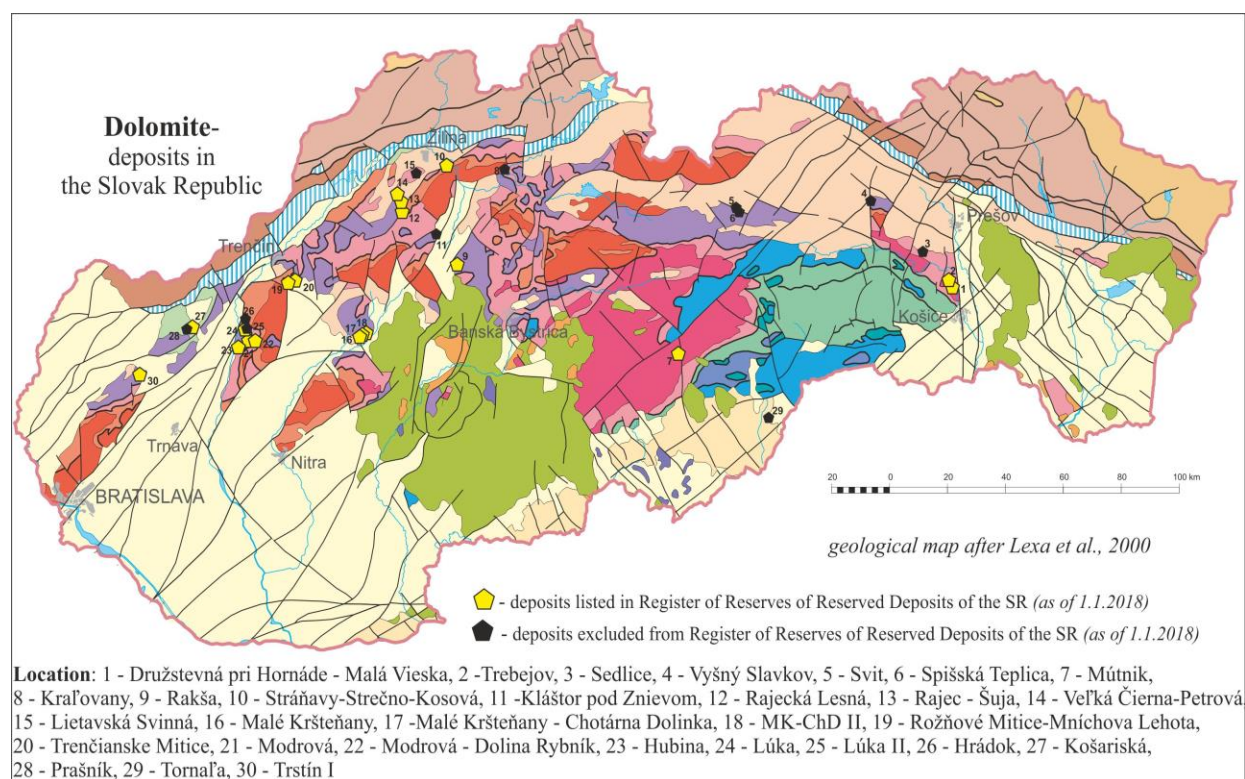


Figure 1. Significant dolomite deposits in individual geological units of the Western Carpathians. Yellow pentagon and designations in bold in location indicate the deposits with reserves stated in the Balance of Reserves in Reserved Deposits with the state to 1.1.2018 (Bačo et al., 2022). Locations of technological samples collection: points 2, 3, 8, 10

carried out by the X-ray diffraction (XRD) method on the BRUKER D2 Phaser device: $\text{CuK}\alpha$ radiation, monochromatic Ni filter, accelerating voltage of the X-ray radiation generator 30 kV, current intensity 10 mA, range of detected angles $5 - 70^\circ 2\theta$, step 0.01° , time 0.3 sec/step. Processing and evaluation of measured data were realized using DIFFRAC.EVA V3.1. Software equipped with the PDF-2/2013 database.

NETZSCH STA 449 F3 Jupiter derivatograph (NETZSCH Gerätebau GmbH., Selb, Germany) equipped with a Std SiC furnace and an Autovac MF Cs rotary pump was used for Differential thermal analyses/Thermogravimetric (DTA/TG) analyses. Measurements were made under the following conditions: heating range: $20 - 1,400^\circ\text{C}$, heating rate $10^\circ\text{C}\cdot\text{min}^{-1}$, reference material: powdered Al_2O_3 , crucibles: ceramic Al_2O_3 , furnace atmosphere: N_2 circulation: $20\text{ ml}\cdot\text{min}^{-1}$.

The morphology of the prepared intermediate products was studied by scanning electron microscopy FE MIRA 3 (Tescan, Czech Republic) equipped by XRD energy-dispersive (EDX) analyzer of chemical composition (Oxford Instruments) (Danková et al., 2025). Raw material samples and processed intermediates/products were subjected to chemical analysis in the Geoanalytical laboratories of the SGUDS in Spišská Nová Ves.

2.4. Laboratory experiment of magnesium reduction

The main raw materials that are used in magnesium smelting include calcined dolomite ($\text{CaO}\cdot\text{MgO}$), ferrosilicon (FeSi), and fluorite (CaF_2).

Two laboratory test of magnesium reduction were performed. A dolomite sample ST-1 of grain fraction below 8 mm was milled and then annealed at a temperature of $1,150^\circ\text{C}$ for 1.5 hours. The calcined sample was mixed with powdered FeSi and CaF_2 in a ratio of 77.5:17.5:5, fine pulverized and then pressed into a pellet with a diameter of 13 mm and thickness of 5 mm.

The experiments were carried out in an electrically heated horizontal tube furnace with flowing argon. The pellet was placed into the furnace in ceramic-glass tube. In the first experiment the pellet was heated at 800°C for 1 h with the aim to ensure the inert atmosphere in the furnace.

The reduction was carried out at $1,100^\circ\text{C}$ for 4 hours. The heated rate of the laboratory furnace was $10^\circ\text{C}/\text{min}^{-1}$. In the second experiment dolomite sample ST-1 of grain fraction below 1 mm was annealed at the temperature of $1,200^\circ\text{C}$ for 1.5 hours. The pellet was prepared same as in the first experiment. The reduction temperature was increased up to $1,150^\circ\text{C}$ with the same reaction time of 4 hours.

3. Results and Discussion

According to the XRD analysis selected dolomite raw materials were characterized by high purity, the CaO content varied from 30.3 to 30.7 % and MgO from 20.9 to 21.4 %. Only for two samples an accessory proportion of quartz and calcite was detected.

The detailed XRD analyses and chemical analyses of particular grain fractions of studied samples were published in our previous study by Danková et al. (2023). These small amounts of impurities did not affect the quality of studied dolomites. Laboratory technological processing and research on Slovak dolomites was carried out according to the scheme presented in Figure 2.

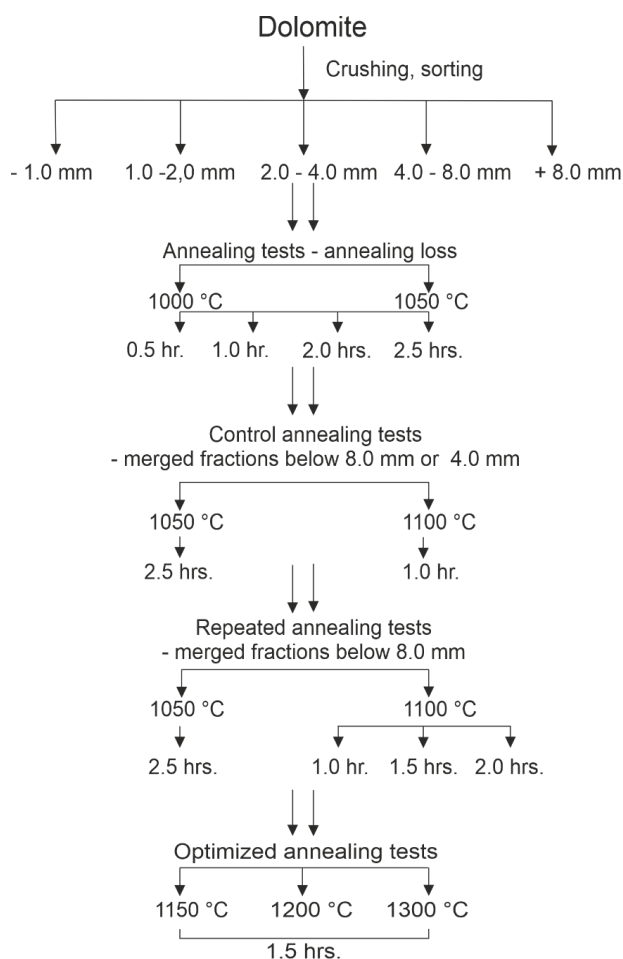


Figure 2. Scheme of the technological processing of dolomite samples

Initial annealing test of dolomite fractions were performed with the aim to determine the value of annealing loss of investigated raw materials. For the samples KRA-1 and ST-1, the required thermal decomposition (above 98 %) was reached for all studied grain fractions at both temperatures (1,000 and 1,050 °C), as well as for the sample ST-2 except the fraction below 1.0 mm annealed at 1,050 °C for 0.5 hour. For the samples SED-1 and SED-2 the decomposition occurs in

the coarse-grained fraction (4 - 8 mm) at both temperatures with holding time of minimal 2 hours. For sample TR-1, only annealing of fractions over 2 mm at the temperature of 1,050 °C with a holding time of over 2 hours was effective (Danková et al, 2023; Danková et al., 2025).

On the basis of these results the control annealing tests were performed with samples of merged grain fractions below 4.0 and 8.0 mm at temperature of 1,050 °C for 2.5 h and at 1,100 °C for 1 h, Figure 2. Obtained products were evaluated with regard to the required conditions for calcined dolomite according to the Blahút et al. (1994).

From the chemical analyses the prepared products contained higher values of CO₂ than is required (maximal 0.3 %), what probably should relates with some facts: Calcined dolomite readily absorbs moisture from the air to form hydroxides Ca(OH)₂ and Mg(OH)₂, where the formation of calcium hydroxide is more pronounced. The Ca(OH)₂ particles are larger (1-5 microns) than brucite, Mg(OH)₂, particles (0.5-1 microns).

Hydration of the MgO and CaO phases of the calcined product proceeds differently. CaO generally tends to hydrate faster than MgO, but at the same time, higher calcination temperatures (above 900 °C) reduce the rate of CaO hydration. Hydration is rapid and can also occur during the sample preparation for chemical analysis (sample grinding, pellets preparation for elemental X-ray analysis).

The XRD analysis of prepared intermediates performed with a longer time lag pointed at the presence of calcite and periclase, but also smaller amounts of portlandite, vaterite, and aragonite were detected (Danková et al., 2023; Bekényiová et al, 2025).

For this reason, in the next experiment, the prepared calcination products were evaluated only using DTA/TG analysis to verify their stability. Repeated annealing tests were realized with samples of merged grain fractions below 8.0 mm at temperature of 1,050 °C for 2.5 h and at 1,100 °C for 1 and 1.5 h, Figure 2.

From the DTA/TG analysis performed immediately after the annealing non weight loss was observed for all studied samples, indicating the required decomposition of carbonates. Also the analysis of the samples annealed at 1,100 °C for 2 h realized with the time lag of two months pointed at their good stability. While small endothermic peaks in the temperature range 365 – 413 °C were detected on measured DTA curves of studied samples, minimal or no weight loss was observed on their TG curves.

An indirect method of verifying stability was scanning electron microscopy (SEM), where dolomitic lime particles were observed for annealed samples with the time lag (of two months, the samples were left loose in bags in the laboratory). Annealing at higher temperature led to better decomposition of the sample and formation of a larger number of small dolomitic lime particles, Figure 3.

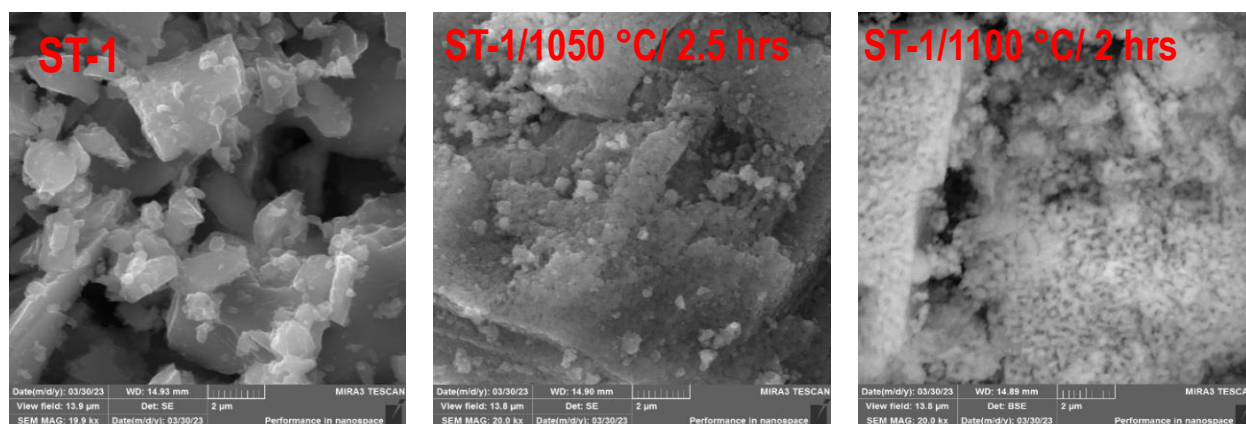


Figure 3. Morphology of the surface of dolomite sample ST-1; raw dolomite and annealed sample at various temperatures and times observed with time lag

The next optimization of the annealing process consisted in the annealing of merged grain fractions at temperatures of 1,150, 1,200 and 1,300 °C for 1.5 hours in order to determine the appropriate temperature for the thermal decomposition of the investigated samples with respect to the theoretical loss by annealing. For samples SED-1, SED-2, TR-1 and KRA-1, the required thermal decomposition (above 98 %) occurred at temperatures of 1,150 and 1,200 °C. For samples ST-1 and ST-2, the required decomposition occurred only at a temperature of 1,200 °C, Table 1 (Danková et al., 2025).

In spite the shape of the DTA curves for each sample annealed at different temperatures was similar, Figure 4, the most optimal annealing losses were achieved, except for the sample KRA-1, at a temperature of 1,200 °C with

a holding time of 1.5 hours. The annealing temperature of 1,300 °C was already unsatisfactory for all samples. Low annealing loss values were determined for individual samples, probably due to reverse chemical reactions. Both DTA curves of sample TR-1 were smooth without expressive endothermic peaks.

The same shape was observed also for the SED-1 and SED-2 samples annealed at 1,200 °C, Figure 4. For SED-1 sample calcined at 1,150 °C only a weak hint of a peak at a temperature of around 400 °C was observed from the measured DTA curve, Figure 4. For ST-1, ST-2 and KRA-1 samples more pronounced endothermic peaks were detected at temperatures of 400 °C and 600 °C for both annealing temperatures of dolomites. (Danková et al., 2025)

Table 1

Parameters of calcined dolomites after annealing at selected temperatures (Danková et al., 2025)

		Sample											
Temperature (°C)	Time (hrs)	SED-1		SED-2		TR-1		ST-1		ST-2		KRA-1	
		Annealing loss (%)	TD* (%)	Annealing loss (%)	TD (%)	Annealing loss (%)	TD (%)	Annealing loss (%)	TD (%)	Annealing loss (%)	TD (%)	Annealing loss (%)	TD (%)
1,150	1.5	46.94	98.34	46.92	98.30	46.89	98.24	46.70	97.84	46.64	97.72	47.09	98.66
1,200	1.5	47.11	98.70	47.22	98.93	47.01	98.49	46.98	98.43	47.02	98.51	47.01	98.49
1,300	1.5	44.38	92.98	44.02	92.23	42.40	88.83	44.32	92.86	44.52	93.27	43.76	91.68

*TD – theoretical decomposition of dolomite to loss of annealing

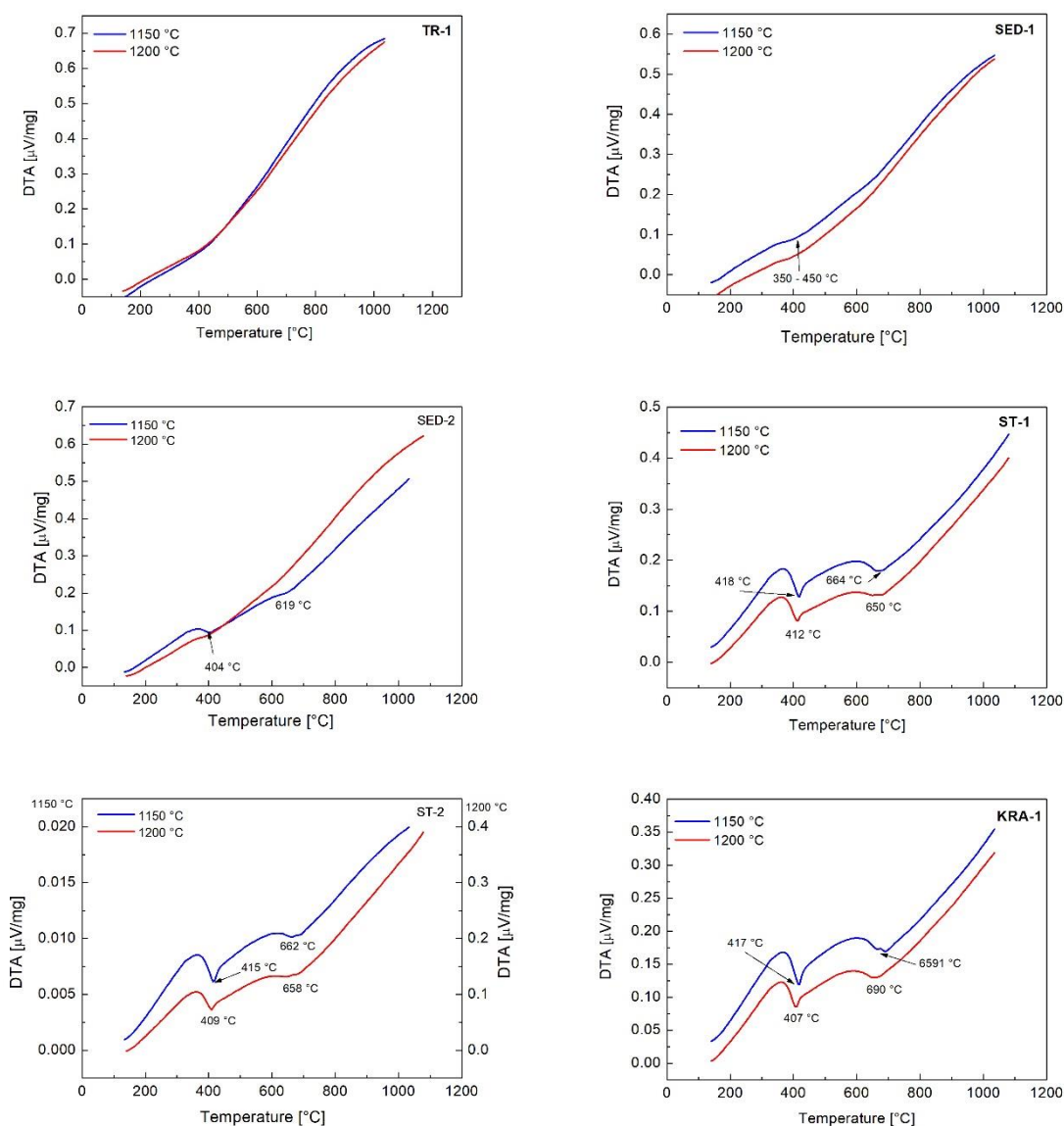


Figure 4. Comparison of DTA curves of selected dolomite samples annealed at temperatures of 1,150 °C and 1,200 °C for 1.5 hours (Danková et al., 2025)

In order to determine the degree of hydration of the samples, or stability of the prepared intermediates, samples TR-1, SED-1, ST-1, KRA-1 annealed at temperature of 1150 °C with holding time of 1.5 hours were again subjected to repeated DTA/TG analysis with a time lag of two months. The analyses resulted in the weight loss in the range of 0.34 - 1.55 % for the studied samples. The DTA record of the sample TR-1 was almost identical, for SED-1 a small peak at a temperature of 400 °C was evident on the DTA curve, corresponding to the hydration of the sample. Higher weight losses were detected for ST-1 and KRA-1 samples, 1.55 and 1.54 %, respectively. The endothermic peaks in the temperature range of 350 - 450 °C (corresponding to the $\text{Ca}(\text{OH})_2$ phase) and 600 – 700 °C (reaction of $\text{Ca}(\text{OH})_2$ with atmospheric CO_2 to form CaCO_3) present on the DTA

curves measured immediately after the calcination, Figure 3, were more pronounced when measured after two months lag, which caused slightly shift of DTA curve to the right side, that means reactions occur at a higher temperature than expected, Figure 5.

Individual weight losses correspond to the hydration activity of calcined dolomite and represent only its hygroscopicity, i.e. the ability to bind water molecules from the surrounding environment (air humidity) and do not fully represent its reducing activity (Che et al., 2020; Danková et al., 2025). In general, when the hydration activity is higher, the reducing activity is also higher. However, with the same hydration activity, the reduction activity may not be the same, due to the different structure of dolomite (Danková et al., 2025). Negligible weight loss detected on TG curves pointed at good

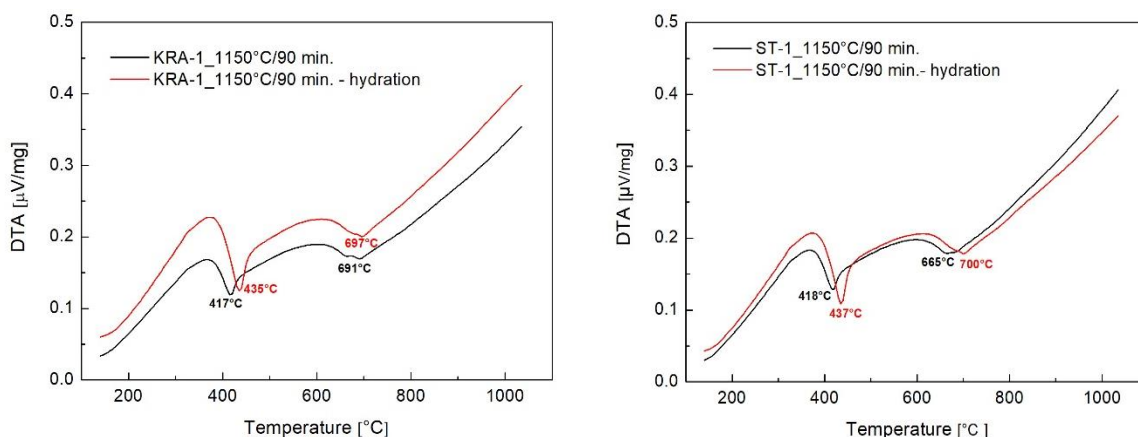


Figure 5. DTA analysis of calcined samples KRA-1 and ST-1 compare to their analyses performed after two months lag

stability of prepared intermediates as well as on suitable process of their calcination. From these results based on hydration activity, the magnesium reduction of calcined dolomite samples, especially of ST-1 and KRA-1, in laboratory experimental conditions should be successful.

3.1. Laboratory experiment of magnesium reduction

Based on the results of DTA/TG analyses of studied dolomites, simple experimental tests of magnesium metal reduction were performed using the calcined sample ST-1.

First, the pellet contained calcined dolomite sample was subjected to DTA/TG analysis in the temperature range of 20 – 1,400 °C in inert atmosphere with the aim to simulate the silicothermic reduction. The pellet represents the input mixture into the furnace ($\text{CaO} \cdot \text{MgO} + \text{FeSi} + \text{CaF}_2$) for the production of metallic magnesium. Under these experimental conditions, the pellet was continuously heated up to 1,400 °C.

Four temperature peaks at 412, 1090, 1,140 and 1,218 °C were observed from the measured DTA curve, Figure 6. The first endothermic peak corresponded to the hydration and decomposition of portlandite phase $\text{Ca}(\text{OH})_2$, what is also observable as a weight loss on the TG curve. The second significant endothermic peak at 1,090 °C was connected with the silicothermic reaction of MgO with ferrosilicon forming a magnesium vapor. The area between the first two peaks indicated that no reactions had occurred.

Above the temperature of 1,100 °C a weight increase was evident on the measured TG curve and two exothermic peaks at temperatures of 1,140 °C and 1,218 °C are obvious and related to reverse oxidation of Mg to MgO. Also phase changes of CaO with SiO to form Ca_2SiO_4 during the heating process took place, Figure 5. However, this product was likely oxidized to MgO, because the measurement was performed in N_2 atmosphere, not in vacuum or under inert conditions.

On the basis of these results, the laboratory experiments in the horizontal furnace with flowing argon was

performed. The first laboratory experiment was not so successful due to low operating temperature of the laboratory furnace (for the first time of the new furnace using, maximal temperature of 1,100 °C was achieved). Despite that the powder product with approximately 91 atm. % of Mg was prepared, but crystalline structure of prepared metal magnesium was not observed by SEM (Bekényiová et al., 2025).

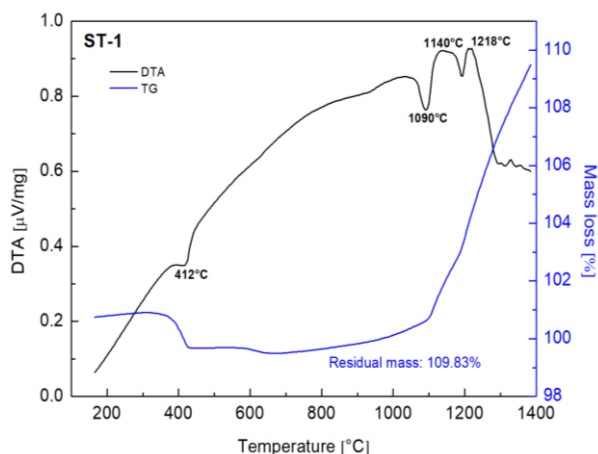


Figure 6. DTA/TG curve of pellet ($\text{CaO} \cdot \text{MgO} + \text{FeSi} + \text{CaF}_2$) prepared for silicothermic reduction

In the second laboratory test the pellet with calcined sample ST-1 was heated in argon atmosphere at 1,150 °C for 4 hours. This temperature was sufficient for silicothermic reduction, what was confirmed by observing of tree-structured particles of metal magnesium by SEM, Figure 7.

Subsequent EDX analysis confirmed that approximately 91 atm. % of metal magnesium was prepared, Figure 8.

In the future, it is planned to repeatedly perform laboratory tests of magnesium metal reduction to ensure a homogeneous argon flow in the tube and stable reduction conditions, which could lead to the complete reduction of calcined dolomite.

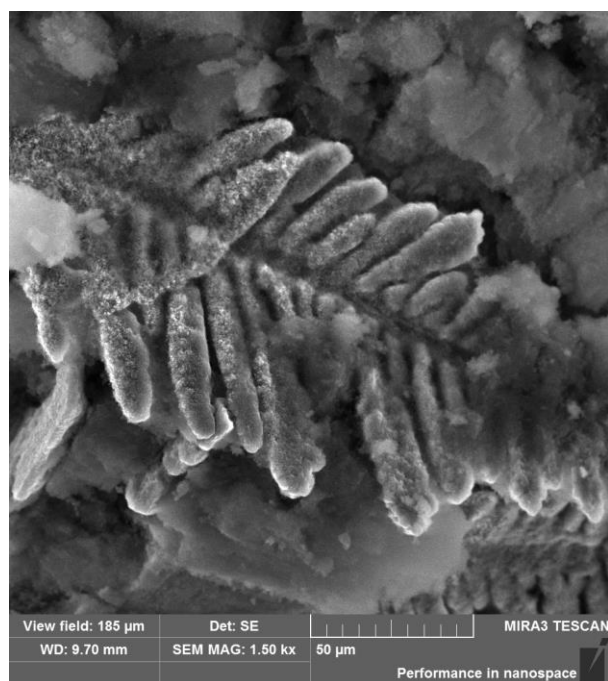


Figure 7. Product of reduction observed by SEM

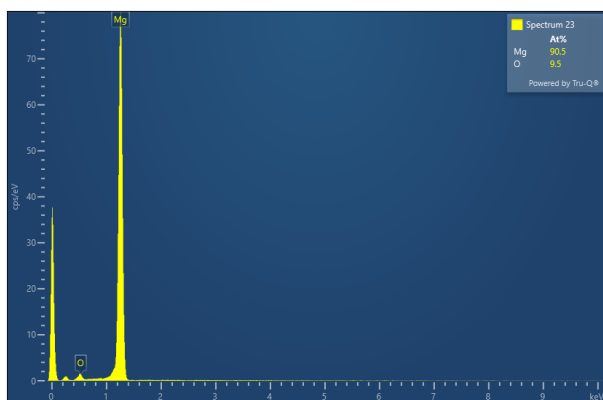
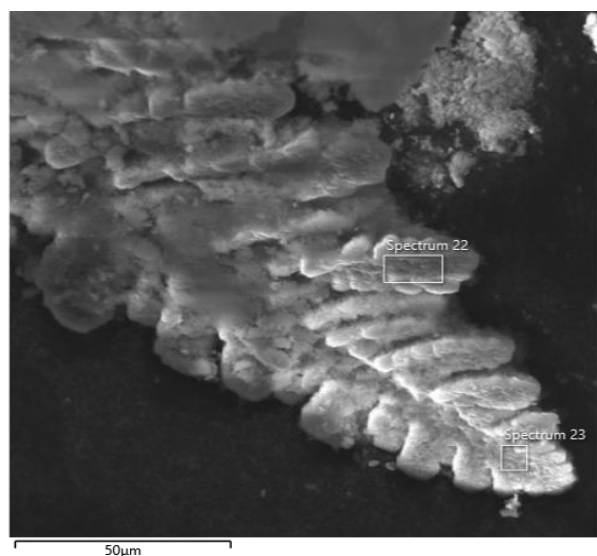


Figure 8. EDX analysis of laboratory prepared metal magnesium product

4. Conclusions

This study confirmed the hypothesis that, for the silicothermic reduction of magnesium, it is necessary to verify the calcination conditions for each sample individually and to determine the influence of hydration activity and active sites in their structure to increase the reduction of Mg.

Optimizing the annealing process led to the determination of suitable conditions for preparing intermediates for silicothermic reduction. In general, all studied samples of Slovak dolomites can be used for metal magnesium production; however, considering hydration activity, samples ST-1 and KRA-1 proved to be the most suitable. DTA analysis simulating the silicothermic reduction, as well as initial laboratory experiments, resulted in a product with a high ratio of metal magnesium.

The results obtained from laboratory technological processing of dolomites will enable the suggestions of the methods for magnesium intermediates preparation applicable to semi-operational and operational conditions of metal magnesium production. Given the current crisis caused by the shortage of critical raw materials, this laboratory technological research is current and important, especially regarding the use of high-quality domestic raw material resources, which can be particularly interesting and beneficial for manufacturers operating in the Slovak Republic.

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Slovački dolomit kao izvor sirovina za proizvodnju metalnog magnezijuma

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INFORMACIJE O RADU

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Dolomit

Kalcinacija

Silikotermička redukcija

Metalni magnezijum

Proširena i recenzirana verzija rada - rad prezentovan na simpozijumu XVI Međunarodna konferencija pripreme mineralnih sirovina i reciklaže (IMPRC 2025)

I Z V O D

U cilju obezbeđivanja samodovoljnosti i stabilnog snabdevanja ključnim mineralnim sirovinama unutar EU, uspostavljena je Evropska alijansa za kritične sirovine (ERMA). Jedan od njenih ključnih ciljeva je da osigura pristup održivim sirovinama i podrži istraživanje i eksploataciju ovih materijala unutar EU. Metalni magnezijum je uvršten na listu kritičnih minerala za zemlje EU od 2011. godine. Najpogodnije sirovine za proizvodnju Mg metodom silikotermičke redukcije su dolomit i magnezit, a Republika Slovačka poseduje značajne resurse ovih karbonatnih sirovina. Za tehnološko istraživanje, odabrano je šest uzoraka dolomita iz različitih ležišta. Uzorci su žareni na odabranim temperaturama i karakterisani korišćenjem diferencijalne termičke analize (DTA), rendgenske difrakcije (XRD) i hemijskih analiza. Rezultati objavljeni u radu sa konferencije od strane Danková et al. (2025) pokazali su da je za silikotermičku redukciju magnezijuma neophodno verifikovati uslove kalcinacije za svaki uzorak pojedinačno i odrediti uticaj aktivnosti hidratacije ili aktivnih mesta u njihovoj strukturi radi povećanja redukcije magnezijuma. Odabrani kalcinirani uzorci dolomita podvrgnuti su ponovljenoj DTA/TG analizi nakon perioda od dva meseca kako bi se utvrdila njihova hidratacija. Na osnovu ovih rezultata, uzorak dolomita označen kao ST-1, kalcinisan pod određenim uslovima, korišćen je za laboratorijski eksperiment silikotermičke redukcije magnezijuma. Dobijeni proizvod je analiziran pomoću SEM/EDX, čime je detektovan visok udeo metalnog magnezijuma (u at. %). minuta.