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Microwave processing of materials in metallurgy

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Мікрохвильова обробка матеріалів у металургії

Abstract. *The application of microwave technology in minerals processing and metallurgy is getting interest as it allows non-conventional treatment of depleted resources and has positive environmental and economic impact. Microwave processing provides rapid and selective heating with energy efficiency, in contrast to traditional methods. This work analysis some basic features of this technology and shows examples of modelling and experimental study of hybrid microwave treatment of oxide materials, highlighting differences in achievable temperatures and heating times. The model for experimental hybrid microwave furnace with cavity resonating in TM_{012} mode at 2.45 GHz with 2 kW power has been implemented in COMSOL software and tested on heating of zirconia samples with SiC based susceptor. Electric field and temperature distributions have been simulated and heating rate variations analysed in different positions of the cavity. Results of the analysis are discussed together with the potential use of microwave technology in ore treatment, mineral processing, smelting and carbothermic reduction. This technology has a very good potential in enhancing metal recovery, reducing energy consumption, and improving processing, but this requires understanding about how different materials reacts with microwaves and how the furnaces have to be optimized for a better sustainability.*
Key words: *microwave, mineral, applications, modelling, heating, reduction.*

Анотація. *Застосування мікрохвильових технологій у переробці мінералів та в металургії викликає інтерес, оскільки дозволяє нетрадиційну обробку виснажених ресурсів та має позитивний екологічний та економічний вплив. Мікрохвильова обробка забезпечує швидкий та селективний нагрів з високою енергоефективністю, на відміну від традиційних методів. У цій роботі проаналізовано деякі основні особливості цієї технології та наведено приклади моделювання та експериментального дослідження гібридної мікрохвильової обробки оксидних матеріалів, висвітлюючи відмінності в досяжних температурах та часі нагріву. Модель експериментальної гібридної мікрохвильової печі з резонатором, що резонує в режимі TM_{012} на частоті 2,45 ГГц з потужністю 2 кВт, була реалізована в програмному забезпеченні COMSOL та протестована на нагріванні зразків цирконію з використанням суцептора на основі SiC. Були змодельовані розподіли електричного поля та температури, а також проаналізовані зміни швидкості нагріву в різних положеннях резонатора. Результати аналізу обговорюються разом з потенційним використанням мікрохвильової технології в обробці руд, переробці мінералів, виплавці та карботермічному відновленні. Ця технологія має дуже хороший потенціал у підвищенні вилучення металів, зниженні споживання енергії та покращенні процесів, але це вимагає розуміння того, як різні матеріали реагують на мікрохвилі та як печі повинні бути оптимізовані для кращої стійкості.*
Ключові слова: *мікрохвилі, мінерал, застосування, моделювання, нагрів, відновлення.*

Introduction. Microwave technology operating in a frequency range of 300 MHz to 300 GHz, where frequencies of 915 MHz and 2.45 GHz being most commonly used [1]. In a high-frequency microwave field, polar molecules become polarized and oscillate at high frequencies along with the alternating electromagnetic field, resulting in the rapid oscillation of molecules and interactions between them. This converts the lost electromagnetic energy into thermal energy in the material [1, 2], so the dielectric loss factor is one of the key parameter in feasibility of this method for high temperature heating. In a simplified form, the power absorbed by a unit volume of medium from microwaves is proportional to frequency (f, Hz), real part of dielectric permittivity (ε'), loss factor ($\tan\delta$) and to squared electric-field strength (E, V/cm) inside the material:

$$P = 2\pi f \cdot \varepsilon_0 \cdot \varepsilon' \cdot \tan\delta \cdot |E|^2 \quad (1)$$

where $\varepsilon_0 = 8.854 \cdot 10^{-14}$ F/cm is the permittivity of the vacuum. The dielectric loss is dependent on material properties, frequency and temperature so the heating might become more accelerating (thermal runaway) or retarded with time when the composition of the material changes.

Literature analysis. Due to essential features of microwave processing, it has considerable potential in minerals processing, hydro- and pyrometallurgy, as it may present substantial energy savings [2]. Microwave heating differs fundamentally from conventional methods, quickly penetrating materials and interacting with their molecular and crystalline structures [1]. This interaction causes molecular vibration and frictional heating, making it more efficient than conduction heating [1-3]. This especially enables microwave processing in ore treatment, particularly for difficult to recovered minerals and high-value products (rare metals, precision metals) due their efficiency and environmental benefits [1].

In Table 1 it is seen that most of transition metals oxides and sulphides are very good microwave absorbers and can be heat very rapidly. Carbon can be heat very fast and this enables extremely rapid reduction to produce metals, as other oxides remain at lower

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temperatures. Carbothermic microwave reduction demonstrates that microwave heating can initiate chemical reactions at lower temperatures, impacting the reduction mechanism significantly. It was reported [3] that the highest temperatures were obtained with carbon and most of the metal oxides like NiO, MnO_2, Fe_3O_4, Co_2O_3, CuO and WO_3. Metal powders and some metal halides also heated well; gangue such as quartz, calcite and feldspar do not heat up [3].

Table 1. Microwave heating effect at 2.45 GHz on particulate solids with the time needed to reach the indicated temperatures [2-4].

Substance	Heating time, min	T °C	Substance	Heating time, min	T °C
Al	6	577	MgO	40	1300
Al_2O_3	24	1900	MnO	6	113
C	0.2	1000	MnO_2	6	1287
$CaCO_3$	7	61	MoO_3	0.46	750
CaO	40	200	MoS_2	0.1	900
Co	3	697	Ni	1	384
Co_3O_4	3	1290	Ni_2O_3	3	1300
Cr_2O_3	7	130	NiO	6.3	1305
CuO	4	800	PbO	13	900
CuS	5	600	TiO_2	8.5	79
Fe	7	768	UO_2	0.1	1100
Fe_2O_3	6	1000	V_2O_5	11	714
Fe_3O_4	0.5	500	Zr	6	462
FeS	6	800	ZrO_2	4	63

Yoshikawa et al. [5] have investigated the microwave carbo-thermal reduction reaction of nickel oxide. When NiO particles and graphite are used, 100% metallic Ni is produced after only a few minutes by microwave heating. The reduction reaction is highly effective under microwave magnetic field irradiation. Several studies have shown that the reduction of Fe, Sc and Mg oxides is effective under microwave irradiation, demonstrating that the energy efficiency of microwave irradiation is higher [6]. Another example is for manganese carbonate ores, which are becoming an increasingly important potential source of manganese, and the calcination and agglomeration of these ores by microwave irradiation has been investigated [7] where under optimal microwave conditions sintering temperatures of >1500 °C were achieved to product manganosite (MnO) and hausmannite (Mn_3O_4) with the sinter strengths comparable to those of conventionally processed materials.

In terms of industrial implementation [3] a metallurgical microwave reduction process could save 15-50% over a conventional operation. Microwave assisted carbothermic reduction on ilmenite concentrate mixed with lignite powder and $CaCO_3$ it was and confirmed that the reduction rate of metal oxide by microwave heating was faster than by conventional heating [3].

Rapid heating of ore minerals in a microwave transparent matrix generates thermal stress of sufficient magnitude to create micro-cracks along mineral boundaries, which improves reaction kinetics, grinding and leaching efficiency [3, 8]. The rapid volumetric heating by microwave treatment results in differential expansion and contraction of different mineral phases, creating internal stresses weakening the ore's structure which become more brittle and easier to grind, reducing the energy requirements for comminution [1]. In practice, it was demonstrated that microwave pretreatment of sulphide ores resulted in a reduction of up to 30% in grinding energy requirements [8]. The preferential heating of specific mineral phases can improve the liberation of valuable minerals from gangue components, increasing the overall recovery rate.

However, microwave processing in metallurgy requires proper investment in microwave generators and furnaces with specific design to ensure that the heat generation and distribution is optimal for the selected material. This dictates that more information about materials properties (especially dielectric permittivity and loss factor) vs. temperature, frequency and materials size need to be studied. This could be achieved in experimental facilities.

Objectives of this work. The goal of this work is to highlight potential of microwave applications in metallurgy and to demonstrate experimental and calculation methods for a better understanding how microwave power and heat are generated and distributed inside the materials.

Materials and methods. An experimental hybrid 2 kW, 2.45 GHz mono-mode (TM_{012}) microwave furnace was set up in Aalto University, Finland (Fig. 1).

The "hybrid" functionality of the microwave cavity comes from the use of SiC susceptor which can be placed around the sample in the case the material is weakly interacting with the microwave radiation at the beginning. Because SiC is a very good absorber, it starts heating first to provide radiative heating to the sample, which, upon increasing temperature, starts to couple with microwaves. In this case indirect heating share decreases, which also can lead to inverse temperature gradient (specimen becomes hotter inside than outside). For demonstration zirconia was used as test material which is known poorly coupled with microwaves at low temperatures but better at higher temperatures.

For calculation of the microwave power and heat distribution COMSOL Multiphysics software was used

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with two coupled modules, radiofrequency and heat transfer. First the magnitude of the electromagnetic field in the whole cavity must be evaluated and consecutively the heat generation by this field in the material can be calculated. This first step is based on Maxwell equations [9] where the cavity (item 1 in Fig. 1) is modelled in 3D for a harmonic propagation mode. The second step deploys standard Fourier heat transfer equation with added source due to absorption of the microwave power [4, 9]. This 3D model of the cavity and specimen is shown in Fig. 2.

Results and discussion. An example of calculated electrical field distribution in the cavity in shown in Fig. 3. The red zones correspond to maximal field strength (in this case 18 kV/m) and indicate the position where the specimen would be optimally located. As by the Eq. (1), this can lead to the maximum power which is proportional to square of the filed strength. In the case of zirconia, magnetic component is also present but its contribution to heating is much less [2, 3].

Temperature in the specimen as measured from its top depends on the input power and time. Figure 4 shows that temperatures over 1273K can be reached in less than 3 min when the input power exceeds 1.5 kW. In the SiC susceptor, very high heating rates (up to 2000 K/min) are spotted already after 1 min of heating, decreasing to 30-50 K/min after 5-10 min. However, at that point heating of the susceptor itself is almost zero because now zirconia sample starts to couple with the microwaves and the heating focus shifts into the sample [4].

The model developed allows proper simulation of the heating process and can be used to experimentally test the properties of different materials behavior in these conditions without explicit knowledge of their dielectric permittivity function. These results, as well as in earlier studies, show that in many cases temperature in the interior of the material could be much higher than at the surface and significant improvements in heat transfer can be achieved for materials, such as oxides which poor thermal conductivity limits conventional heat [2]. The energy densities in microwave systems can be relatively high and can lead to very high internal heating rates, especially at the beginning of the process which is difficult to achieve with conventional furnaces.

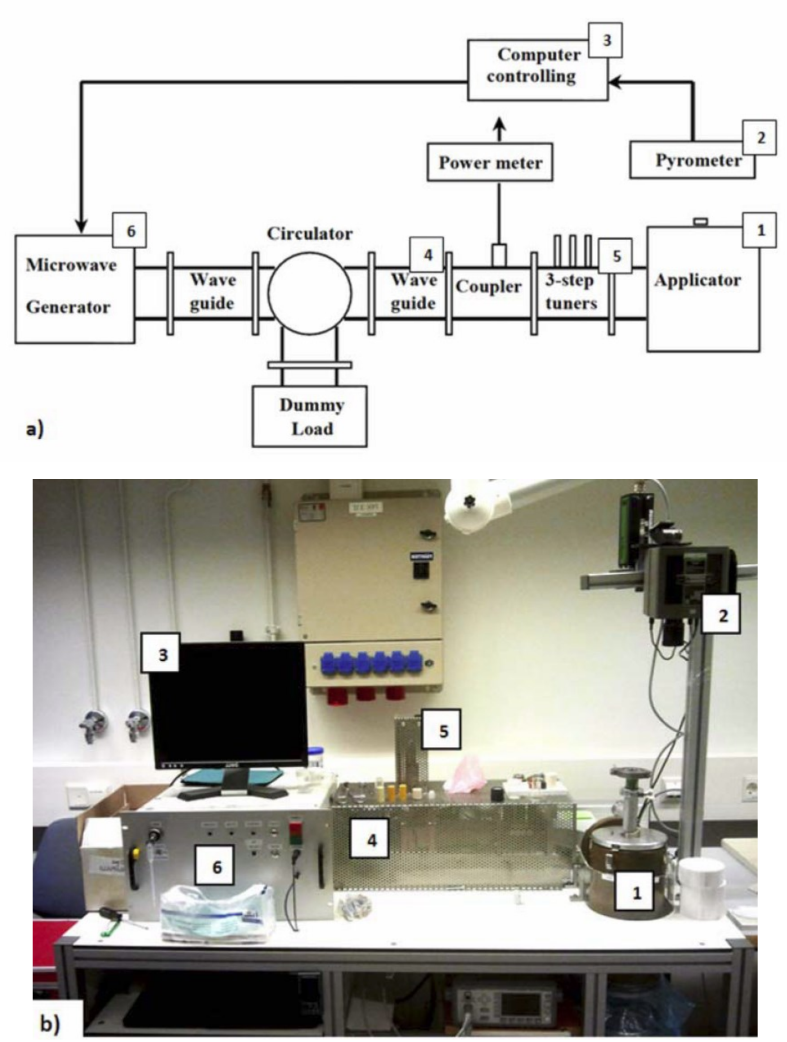

Fig. 1. The scheme (a) and overall view (b) of experimental 2 kW hybrid microwave furnace: 1 – the cavity with the sample, 2 – two-way optical pyrometer, 3 – control system, 4 – waveguide, 5 – microwave tuners, 6 – generator [4].

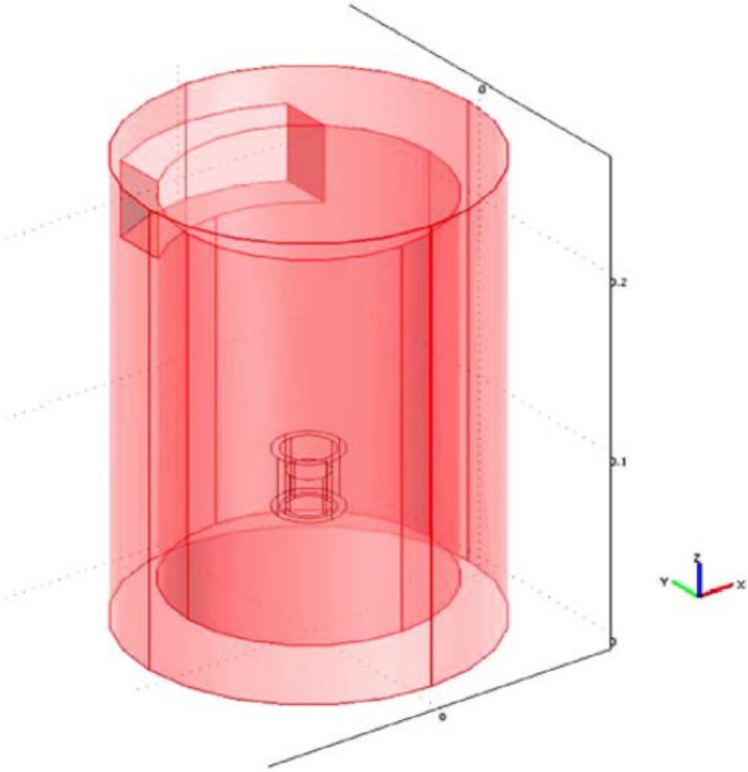

Fig. 2. The COMSOL model of the microwave furnace cavity (dimensions in m) and the specimen position.

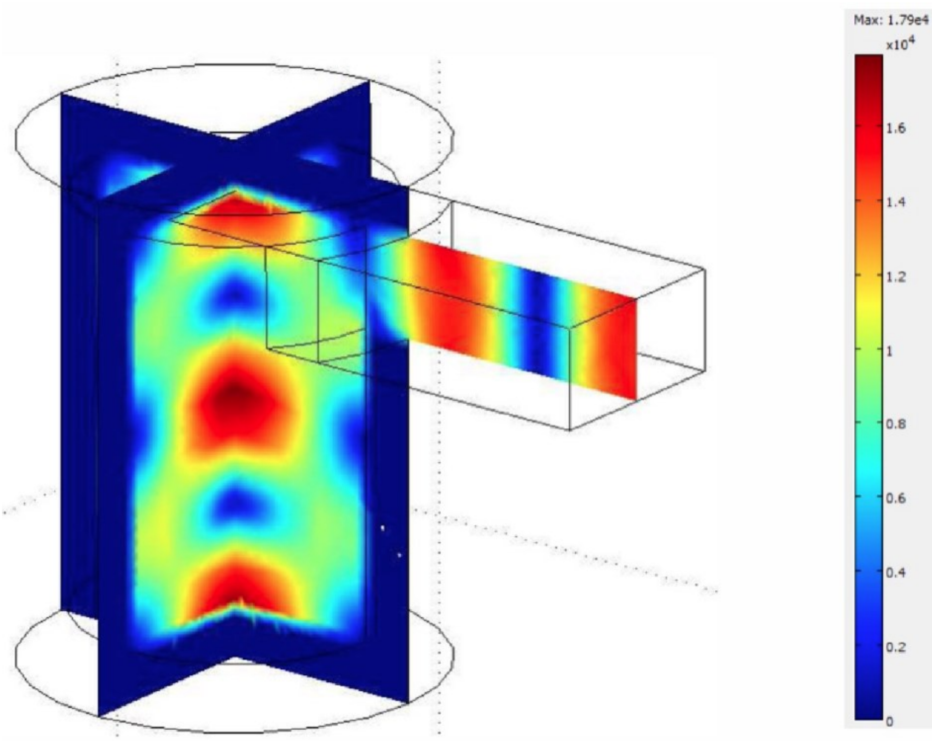

Fig. 3. Calculated electrical field strength (V/m) in the waveguide and cavity for 1.2 kW input at 2.45 GHz in TE_{10} mode.

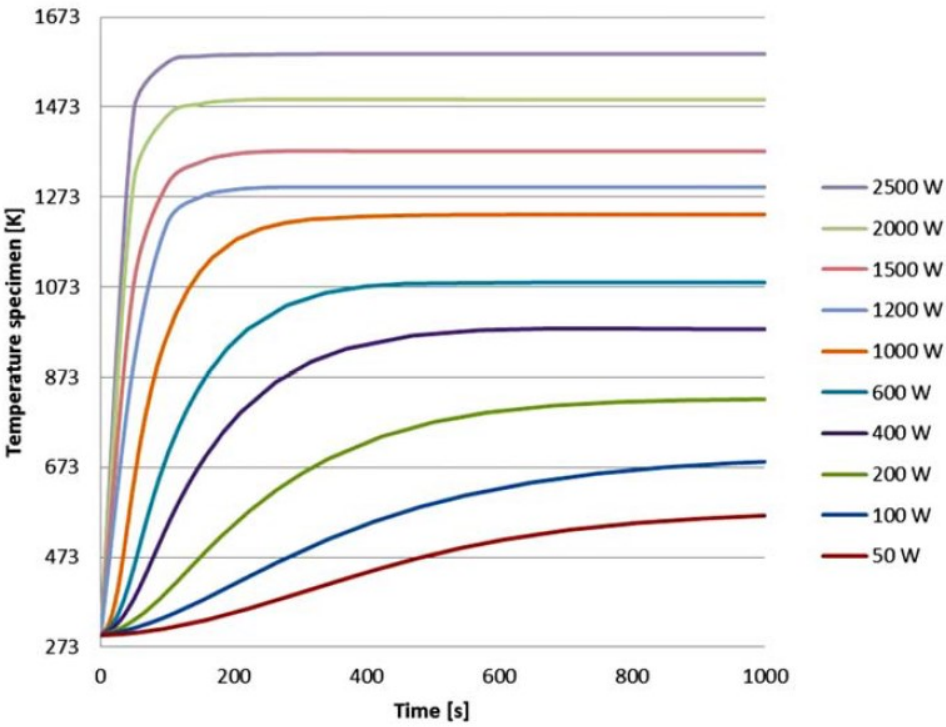

Fig. 4. Calculated temperatures of the specimen vs. time and input power.

The microwaves energy source is relatively clean and easily controlled, facilitating continuous processing, minimizing the amount of off-gas and also the amount of dust particles. This improves working conditions in microwave processes to be far more superior to those in conventional processes, promoting both endothermic and exothermic reactions as well as synthesis [2, 10]. Selective heating can be attained – for example when heating manganese carbonate ore, $CaCO_3$ component would remain much colder (Table 1) as major heat generation will be in the manganese phase.

Conclusions. Microwave technology in mineral metallurgy presents a progressive and efficient approach to ore processing, aligning with the industry's need for innovation and sustainability [1,2]. Despite that microwave energy is more expensive than electricity, the efficiency of microwave heating is often much higher than conventional heating and overcomes this costs differences, especially when high-value materials are being recovered. The practical applications of microwave treatment (ores processing, reduction, drying) have demonstrated significant improvements in process efficiency, energy consumption, and environmental impact [2, 3] and it is evident that microwaves could achieve effects such as inverse heating and rapid effect with a higher cost-efficiency. Among challenges required for successful implementation of the technique are still more fundamental understanding of microwaves interaction with different minerals as well as analysis of kinetics of the processes.

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