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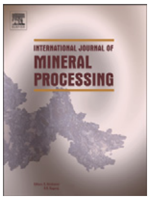

High-resolution dielectric characterization of minerals: A step towards understanding the basic interactions between microwaves and rocks☆

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ABSTRACT

Microwave energy was demonstrated to be potentially beneficial for reducing the cost of several steps of the mining process. Significant literature was developed about this topic but few studies are focused on understanding the interaction between microwaves and minerals at a fundamental level in order to elucidate the underlying physical processes that control the observed phenomena. This is ascribed to the complexity of such phenomena, related to chemical and physical transformations, where electrical, thermal and mechanical forces play concurrent roles. In this work a new characterization method for the dielectric properties of mineral samples at microwave frequencies is presented. The method is based upon the scanning microwave microscopy technique that enables measurement of the dielectric constant, loss factor and conductivity with extremely high spatial resolution and accuracy. As opposed to conventional dielectric techniques, the scanning microwave microscope can then access and measure the dielectric properties of micrometric-sized mineral inclusions within a complex structure of natural rock. In this work two micrometric hematite inclusions were characterized at a microwave frequency of 3 GHz. Scanning electron microscopy/energy-dispersive x-ray spectroscopy and confocal micro-Raman spectroscopy were used to determine the structural details and chemical and elemental composition of mineral sample on similar scale.

1. Introduction

Microwave heating was demonstrated to be potentially beneficial in reducing the cost of various mineral processing unit operations (Haque, 1999), thereby making the mining process more efficient and sustainable (Kingman, 2006). For example, the effectiveness of the microwave-induced thermally assisted liberation was extensively demonstrated (Kingman et al., 2004; Walkiewicz et al., 1989). Microwave fields induce volumetric and selective heating; in a heterogeneous material only the 'lossy' phases absorb electromagnetic energy, avoiding the waste through the 'bulk-heating' of the surrounding rock (Jones et al., 2005). Due to the different expansion rates of the minerals in the rock, micro-fractures are induced, theoretically making the grinding process less energy-consuming (Kingman and Rowson, 1998).

In spite of its potential, the application of microwave energy in the mining process has not delivered the outcomes required in order to become a commonly used technology in this field. The reasons are multiple but certainly can be ascribed to a lack of fundamental understanding of the interacting mechanisms between minerals and the microwave field itself. In particular, few pioneering studies were proposed for correlating physical effects (mechanical, thermal) to the electromagnetic energy absorbed (Jones et al., 2005; Whittles et al., 2006; Ali and Bradshaw, 2009, 2010, 2011), and a sparse literature base was developed so far for correlating these effects to the electromagnetic properties that are the most important for understanding the microwave-matter interactions.

A very first example of study of the combined effects of thermo-physical, mechanical and electromagnetic properties involved in the microwave-assisted fragmentation and comminution has been presented in the recent literature (Hartlieb, et al., 2012, 2016; Meisels et al., 2015; Toifl, et al., in press).

The heating mechanism of minerals in electromagnetic fields is correlated to complex physical phenomena, related to dielectric and magnetic losses:

$$P_{abs} = \omega\varepsilon_0\varepsilon''_{eff}E^2_{rms}V + \omega\mu_0\mu''_{eff}H^2_{rms}V \qquad (1)$$

where P_{abs} is the average power deposited within the sample in watts [W], $\omega = 2\mu f$ is the wave angular frequency in hertz [Hz], ε_0 and

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μ_0 are the vacuum permittivity and permeability, respectively in farads per metre [F/m] and henry per metre [H/m], ε''_{eff} and μ''_{eff} are the electric and magnetic effective loss factors, E_{rms} and H_{rms} are the average intensities of the electric and magnetic field, respectively in volts per metre [V/m] and amperes per metre [A/m], V is the volume of the heated sample in [m^3].

The deposited power is therefore dependent upon the material characteristics which are variable with frequency (Metaxas and Meredith, 1983):

$$\begin{array}{r}\varepsilon''_{eff} = \varepsilon'' + \sigma/\omega\varepsilon_0 \\ \mu''_{eff} = \mu''\end{array}. \qquad (2)$$

It is then essential to evaluate the complex dielectric permittivity $\varepsilon^* = \varepsilon' \text{-} j\ \varepsilon''_{eff}$ and magnetic permeability $\mu^* = \mu' \text{-} j\mu''_{eff}$ of the sample under analysis. The first quantity, at microwave frequencies, is mainly determined by the polarization effect, quantified by ε''_{eff}. The only exception is for highly conducting materials where the conduction phenomenon, quantified by σ, is dominant. μ''_{eff} is usually negligible for non-magnetic samples.

The heterogeneity of ores makes the interaction with the microwave field highly complex. Selective heating of certain mineral inclusions with respect to the surrounding matrix typically occurs (Kingman et al., 2000; Walkiewicz et al., 1988). This is because such mineral inclusions have significantly higher dielectric properties than the rest of the rock forming material, namely a higher ε''. From Eq. (1), the power absorbed by the mineral inclusions is therefore higher than the surrounding gangue material, so the microwave energy 'focuses' selectively on such inclusions, giving rise to a temperature differential which is the basis of many applications of microwave energy in mineral processing.

The dielectric properties of each phase are required to describe such a complicated selective heating phenomenon from an electromagnetic point of view and then potentially relate it to a multi-physics description of the process. This need of a comprehensive description of the multi-phase constitution of rocks was highlighted by Meisels et al. (2015). Unfortunately, conventional methods for the dielectric characterization of materials are not suited to this as they allow only the quantification of the properties of the material at centimetre scale.

Different techniques were applied for mineral characterization in the Ultra High Frequency (UHF) microwave range (300 MHz–3 GHz), where the Industrial-Scientific-Medical (ISM) spectra are located. For example, the short circuited waveguide method, based on the previously proposed standing wave perturbation method by Roberts and Von Hippel (1946), was used for the analysis of minerals between 1 and 22 GHz (Nelson et al., 1989). In Tinga (1989), a bridge configuration, with samples mounted in the central section of a waveguide, was used for testing the dielectric properties of some metal oxides at 2.45 GHz over a wide range of temperatures. An open-ended coaxial line technique was widely exploited as well: in reference Salsman and Holderfield (1994) chalcopyrite, chalcocite and cobaltite were analysed across a limited temperature range [20–300 °C]. The cavity perturbation method was used extensively for even higher temperature range [20–800 °C] (Pickles et al., 2005).

The main limitation of these techniques is that they cannot quantify the properties of the individual minerals in rocks. Additionally, the majority of conventional dielectric measurement techniques are highly dependent on the density of the sample under study (Nelson et al., 1989). Application of complex mixture theories is then needed for calculating the dielectric properties of single minerals from a conventional dielectric measurement (Shivola, 1999). These require additional information about particle size, shape and distribution and they represent just a simplified approximation of the real scenario. In this way, 'controllable' synthetic compounds can be measured accurately (Salsman et al., 1996).

In this paper a novel method for measuring the dielectric properties of mineral inclusions embedded in a natural rock at microwave frequencies is presented. Such a method is based upon Scanning Microwave Microscopy (SMM) that is able to achieve extremely high spatial resolution and accurately measure the dielectric constant and losses of the sample under analysis.

The SMM technique is introduced and exemplified for the first time to hematite inclusions embedded in a gangue matrix. A comprehensive chemical and dielectric characterization of such micrometric inclusions is shown. The SMM capability of mapping the dielectric properties with sub-micrometric resolution is highlighted.

2. Materials and methods

A set of ore samples were core drilled to produce sample mounts of 25 mm diameter and 15 mm height. These samples were from the batch of rocks used for a microwave heating test work, which results were recently reported by John et al. (2015). The heated phase selected for that investigation was a copper concentrate sample. All the rocks originate from a North American mine.

The mounts were cleaned with distilled water and dried using compressed air. The cleaned mounts were polished using a Struers Rotopol polishing machine to a surface roughness of 10 nm, necessary for

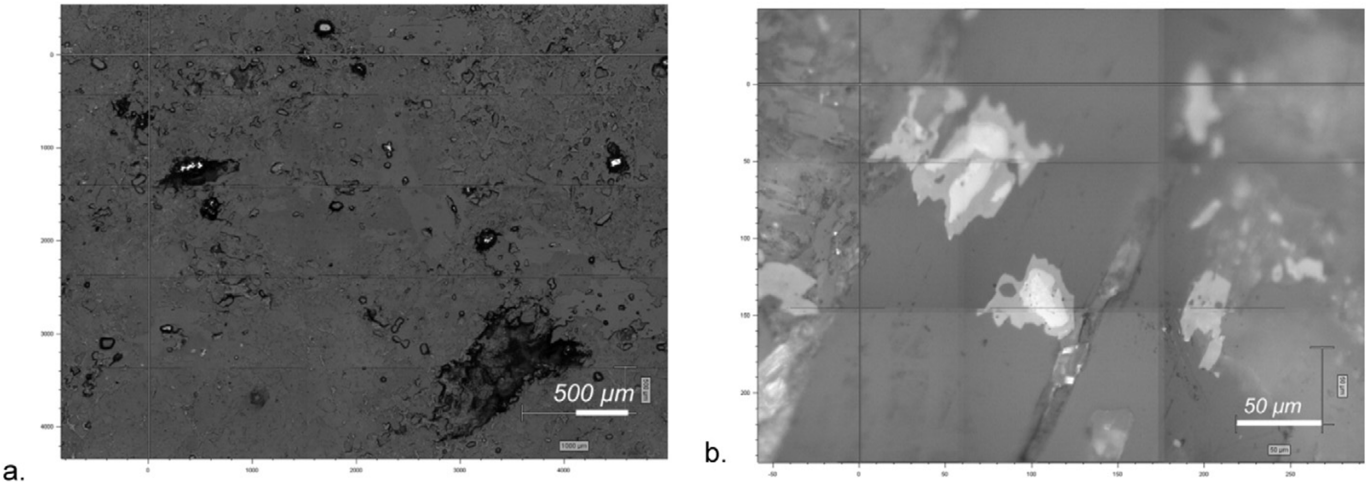

Fig. 1. Optical images of a natural rock sample surface. The images are composed of several sub images stitched to form the final one ('montage' map). Fig. 1a. shows a 6 × 5 mm area (5× magnification) and Fig. 1b shows a 100× magnification of a 0.35 × 0.3 mm area. A high heterogeneity of the surface is visible at different scales. Different grey tones are ascribable to different materials. Phases are clearly recognizable at very different scales.