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Comparison of thermoluminescence (TL) and cathodoluminescence (ESEM-CL) properties between hydrothermal and metamorphic quartzes

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ABSTRACT

This paper reports on the Thermoluminescence (TL) and Cathodoluminescence (CL) emission of well-characterized hydrothermal milky quartz specimens from Hakkari in Turkey, labeled THQ, and Madrid in Spain, labeled SHQ, and metamorphic quartz from Madrid, in Spain, labeled SMQ. Both hydrothermal and metamorphic quartz samples display similar UV–IR CL spectra consisting of five groups of components centered at 330 nm and 380 nm linked to $[\text{AlO}_4]^\ominus$ centers, 420 nm due to intrinsic defects such as oxygen vacancies, lattice defects, and impurities which modify the crystal structure, 480 nm associated with $[\text{AlO}_4]^\ominus$ centers of substitutional Al^{3+} , and a red broad band related to the hydroxyl defects in the quartz lattice as precursors of non-bridging oxygen hole centers (NBOHC) and substitutional point defects. The Turkish quartz specimen exhibits higher CL intensity in the UV region (up to 330 nm) than the Spanish specimens probably linked to the presence of Ca (0.95% in THQ and less than 0.1% in SHQ and SMQ). At wavelengths greater than 330 nm, SMQ (formed at high pressure 6000 bars and temperatures over 500–600 °C) shows higher intensity than the hydrothermal (growth at 2000 bars and temperatures 200–300 °C) samples associated with the formation process. The natural blue TL glow curves of both THQ and SHQ display a weaker TL intensity than the SMQ, attributable to the Al (0.32%), Ti (0.14%), K (0.01%) and Zr (76 ppm) content. It is shown that mineralogical formation, crystallinity index and the content of the impurities seem to be the main parameters of influence in the shape intensity of the CL and TL glow curve emission.

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1. Introduction

Quartz is one of the most abundant minerals in nature being formed in igneous, metamorphic and sedimentary geological environments. It can be used to obtain information of specific properties of quartz-bearing materials for geological, mineralogical studies (Maarten and Broekmans, 2004; Okamoto et al., 2010) and, due to the luminescence properties, quartz has been often employed for retrospective dosimetry and dating purposes (Botter-Jensen et al., 2000; Bailiff et al., 2004; Bailiff et al., 2005; Pagonis et al., 2002; Yazici and Topaksu, 2003; Adamiec, 2005). Chemically, quartz is considered almost pure SiO_2 having accessory ions such as Ti^{4+} , Fe^{3+} , Na^+ , Li^+ , H^+ and Al^{3+} included in the crystal lattice at concentrations $> 10 \mu\text{g g}^{-1}$ (Lehmann et al., 2009). Thermodynamic conditions affect the type and frequency of lattice defects during the mineralization. There are some issues

which are caused by variations in the structure of quartz during the hydrothermal and metamorphic processes such as changes of pressure (P)–temperature (T) conditions, natural irradiation or alteration (Gotze, 2009). Hydrothermal quartz has idiomorphic and zoned textures and may show transient internal structure, episodically changing growth conditions; hydrothermal quartz grows at pressures of 2000 bars and temperatures in the range of 200–300 °C while metamorphic quartz is formed at higher pressures, up to 6000 bars and temperatures over 500–600 °C (Gotte et al., 2011).

Composition and structure of minerals and their genetic characteristics and typomorphic properties can be determined by luminescence techniques, cathodoluminescence (CL) and thermoluminescence (TL), among others (Remond et al., 1992; Pagel et al., 2000; Gotze et al., 2001). Intrinsic and extrinsic defects are potentially responsible for different luminescence emissions. They are generally observed in insulator materials during excitation with temperature, electrons, ions, UV or ionizing radiation. The defects determine the presence of both TL and CL trapping and recombination sites. Classical luminescence studies have

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been focused on synthetic crystals doped with different cations, and for experimental reasons, the use of TL and CL has frequently been limited to the UV–IR region of the spectrum. TL is a method based on the emission of light from a solid sample such as insulator or semiconductor when it is heated after being irradiated by some kind of radiation such as x-rays, gamma rays, beam of electrons, cosmic rays, etc. (McKeever, 1985). TL provides information about the trapped charge recombination sites related to metastable defects inside the lattice if the detrapping process is due to heat. All the factors involved in the luminescence phenomena (i.e. lifetime, efficiency, emission spectra, etc.) depend on the crystalline phase which is mainly induced by pressure and temperature. Thus, small variations in the lattice structure due to the presence of inclusions, impurities, substituted ions or surface defects in ppm concentrations reveal changes in the intensity and wavelength position of the emission spectra (Correcher et al., 2004). The emphasis on single crystals has resulted in theoretical modeling of the TL based on very simple defect models with well-defined phase site descriptions involving just a few atoms. This has led to the development of simple kinetic models. However, there is an increasing body of data pointing out that simple first-order kinetic description of TL is not always appropriate for several natural materials and different glow peaks may variously be described in terms of mixed-order kinetics or even continuous trap distribution that are usually linked to (i) large defect complexes, (ii) phase transitions, (iii) ionic self-diffusion or/and (iv) bond breaking induced also by dehydration–de-hydroxylation mechanisms (Garcia-Guinea et al., 2009). CL was used to find out growth zoning in quartz crystals (Ramseyer and Mullis, 1990; Gotze et al., 2001). CL is a process whereby light is created from an energetic electron beam. CL supplies data about transient defects after irradiation on the surface of the lattice. CL is used in the identification of the migration and diffusion of some luminescent centers from the emission bands (Kalceff and Phillips, 1995). It has also been used to distinguish between volcanic and hydrothermal mineralizations (Ramseyer et al., 1988; Evans et al., 1994). This paper is focused on the study of the luminescence emission (CL and TL) of hydrothermal and metamorphic quartzes previously characterized by means of x-ray diffraction (XRD), x-ray fluorescence (XRF), and differential thermal analysis-thermogravimetry (DTA–TG).

2. Materials and methods

Two natural milky hydrothermal quartzes from Turkey (THQ) and Spain (SHQ), and one natural metamorphic specimen from Spain (SMQ), have been used in this study. The samples were characterized by (i) XRF, using a Philips Spectrometer PW 1410/20 with a PW 1730 generator; (ii) XRD using a Phillips PW1710/00 diffractometer with a $\text{CuK}\alpha$ radiation source, equipped with a graphite monochromator. Patterns were obtained by step scanning from 2° to 64° (2θ in steps of 0.020° ; 4 s per step) and compared with the XRD card files of the Joint Committee on Powder Diffraction Standards. (iii) DTA–TG analyses recorded with a thermal analyzer Model 851e Mettler Toledo in N_2 atmosphere. Thermal treatments were performed at a heating rate of $10^\circ\text{C min}^{-1}$ from room temperature to 700°C . The samples were held in an alumina crucible and the reference material was also alumina.

CL spectra were measured on a polished slab in low vacuum mode without coating, using a Gatan MonoCL3 detector and PA-3 photomultiplier attached to the ESEM (Environmental Scanning Electron Microscope). The PMT covers a spectral range of 185–850 nm, and is most sensitive in the blue parts of the spectrum. A retractable parabolic diamond mirror and a photomultiplier tube

are used to collect and amplify luminescence. The sample was positioned 16.2 mm beneath the bottom of the CL mirror assembly. The excitation for CL measurements was made with a 25 kV electron beam. TL measurements were obtained using an automated Risø TL system model DA-12 (Botter-Jensen and Duller, 1992) provided with an EMI 9635 QA photomultiplier and a blue filter peaked at 320–480 nm, with 80 ± 16 nm FWHM and 60% minimum of peak transmittance. All the TL measurements were performed using a linear heating rate of 5°C/s from RT up to 500°C , in a N_2 atmosphere. The samples were carefully powdered with an agate pestle and mortar to avoid triboluminescence (Garcia-Guinea and Correcher, 2000). The weighed 5.0 ± 0.1 mg powder samples and three aliquots of powdered quartz samples were used for each measurement. The incandescent background was directly subtracted from the TL data.

3. Results and discussion

3.1. Sample characterization

Hydrothermal and metamorphic quartz specimens sliced at $40\ \mu\text{m}$ show different grain textures under the polarizing microscope (Fig. 1). Such variation is of great interest since the poly grained texture of the SMQ section exhibits deformed and welded

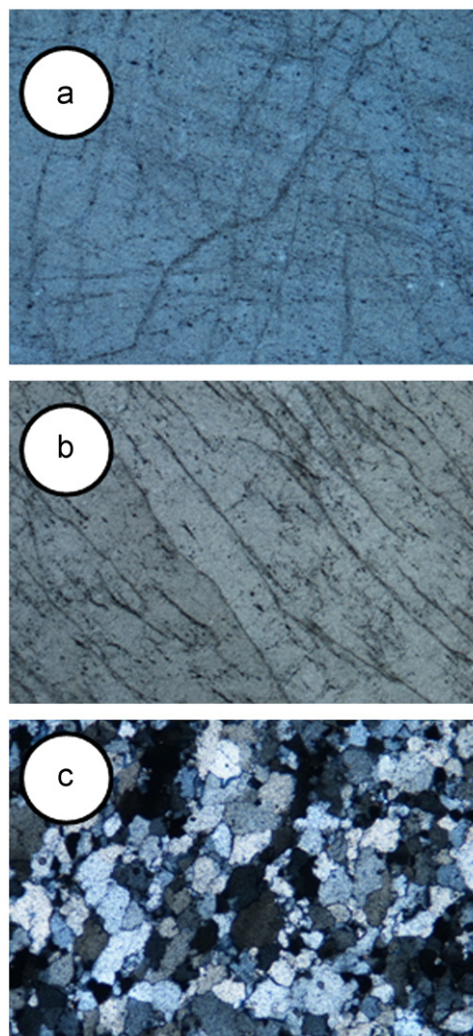


Fig. 1. Thin section photos observed under crossed polarizers: (a) Hydrothermal Spain, (b) Hydrothermal Turkey, (c) Metamorphic Spain.

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