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Multi-technique study of He^{+} micro-irradiation effects on natural quartz crystals contained in archaeological pottery

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ABSTRACT

The effect of α irradiation on natural quartz is commonly known in geology, resulting in the formation of luminescent halos around radioactive inclusions. In archaeological ceramics quartz grains are present and can be surrounded by radioisotopes contained in the clay. The aim of this study is a first attempt to detect similar halos on the outer rims of quartz grains isolated from archaeological pottery. Such identification could be of interest in luminescence dating and as an alternative method for ceramic artworks authentication. Isolated grains were characterized with cathodoluminescence imaging, SEM-EDX and Raman spectroscopy. Some of the crystals were irradiated for reference with a microbeam of He^{+} accelerated ions and investigated by means of real-time ionoluminescence. Obtained results seem to confirm that the natural effect on archaeological quartz might be too weak to be detected with these techniques and underline the importance of the multi-technique approach to avoid misinterpretations.

1. Introduction

Quartz is one of the most abundant minerals in the Earth's crust and it has nowadays massive employment for technological applications, such as in semiconductor industry and optoelectronics. For the monitoring and tailoring of specific properties, the structure of SiO_2 has been widely studied in the last few decades, together with the effects of lattice defects and extrinsic impurities [1–5]. These defects affect greatly the luminescent and electronical behavior of SiO_2, both in its crystal and amorphous phase, and can arise, for example, from the damage induced by different types of radiation [6–9].

The effect of α particles on natural quartz is well known in geological studies as the consequence of constant irradiation, for millions of years, of the portion of crystal surrounding radioactive inclusions, in particular U- and Th-bearing phases. α particles are capable of inducing lattice damage, that results in the formation of a visible luminescent halo easily detectable with cathodoluminescence (CL) imaging [10,11]. For a better understanding of this phenomenon, studies by means of He^{2+} or He^{+} artificial implantation at large facilities on both natural and synthetic quartz have been performed and can be found in the literature [12–14]. CL is the most common technique used in this case to detect the halos [10–15], but also Raman spectroscopy [15] and transmission electron microscopy with selected area electron diffraction (TEM-SAED) [13] have been reported. In these works the width of the halo, namely the penetration range of the particles in quartz, varies between 14 and 45 µm depending on the energy of the implanted particle (Table 1). Very often, imaging is correlated with an electron paramagnetic resonance (EPR) analysis, aiming to identify the nature of produced defects: these are generally acknowledged as unpaired electrons at oxygen vacancies (E'_1 centers) or Non-Bridging Oxygen Hole Centers (NBOHC) [13,16], as well as hole centers due to silicon vacancies [11]. Experiments with the same techniques have also been performed on other types of crystals, for example on zircon [17], albite [18] or $CePO_4$ [19]. It is worth noting that the lattice damage in quartz

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