**RESEARCH PAPER** 

## **Environmental scanning electron microscopy (ESEM)** and nanoindentation investigation of the crack tip process zone in marble

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Abstract This study explores the interaction between crack initiation and nanomechanical properties in the crack-tip fracture process zone of Carrara marble. Specimens with preexisting cracks were loaded in a uniaxial testing machine until the process zone appeared at the tips of the preexisting cracks. ESEM analysis reveals an increase in microcrack density in the process zone with increased loading of the specimen. Nanoindentation testing comprised of lines and grids of single nanoindentations located both near and far from the process zone shows a decrease in both indentation modulus and indentation hardness near grain boundaries in intact material, and with closeness to the process zone. Ultimately, the study confirms that the crack-tip process zone manifests itself as an area of reduced indentation hardness and indentation modulus in marble.

**Keywords** Carrara marble · Environmental scanning electron microscopy (ESEM) · Fracture process zone · Nanoindentation

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## 1 Introduction

The determination of the mechanisms at play during crack propagation is one of the key challenges in the field of fracture mechanics of rocks. The principles of linear-elastic fracture mechanics (LEFM) dictate that fracture propagation criteria (such as either local stress-intensity criteria or global energy release criteria) should be used to consider the otherwise infinite stresses at the tip of an ideal crack [16, 19, 20]. Another way to reflect these conditions is through the fracture process zone (FPZ) as suggested by Irwin, Barenblatt, and Dugdale [7, 14, 20, 21]. The FPZ develops in the near-crack-tip region when the stress reaches the value of the material strength. At this point, the material yields to form a damage zone termed the FPZ [18]. Researchers have found significant interest in the detailed experimental investigation of the FPZ in order to determine its precise effect on the material behavior. In metals, the FPZ is often known as the "plastic zone." Strain-hardening metals produce an increase in hardness within the plastic zone, and strain-softening metals produce a decrease [32]. The development of strength and deformability in the FPZ is not clearly established for brittle and quasi-brittle materials and is the subject of this investigation. By specifically studying microstructure, such a finding might allow one to relate material property changes to the FPZ in such material.

Microscale experimental investigation of the FPZ in brittle and quasi-brittle materials chiefly uses four techniques: microscopy [3, 19, 29], interferometry [10–12, 14, 28], acoustic emission [6, 18, 21, 25, 27, 40], and ultrasonic pulses [20, 30, 38]. Although these techniques provide important information on the microstructure of the FPZ—in particular, the exponential increase in microcrack density within the FPZ with closeness to the main crack or fault—they fail to directly measure material properties within the