### A novel protocol for resolving feldspar crystals in synchrotron X-ray microtomographic images of crystallized natural magmas and synthetic analogs

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#### ABSTRACT

X-ray computed microtomography is a non-destructive imaging technique recognized in the geosciences as a powerful tool to investigate rock textures directly in three dimensions (3D) at the micrometer and sub-micrometer scale. The quantitative morphological and textural analysis of images requires segmentation and characterization of phases in the reconstructed volume based upon their gray levels (related to their relative X-ray attenuation) and/or morphological aspects. Often the differences in X-ray attenuation of some phases are so small that no contrast is observed in the reconstructed slices or, although the human eye can discern the differences between these phases, it is difficult, or sometimes impossible, to reliably segment and separately analyze these phases. Facing this challenge, we propose an experimental and computational procedure that allows the segmentation of phases with small density variations in geomaterials. By using an experimental protocol based on phase-contrast synchrotron X-ray microtomography combined with a customized 3D image processing procedure, we successfully segmented feldspar from the glassy matrix in both a natural volcanic sample and a synthetic analog. Our results demonstrate that crystallized natural volcanic rocks and synthetic analogs can be characterized by synchrotron X-ray phase-contrast microtomography and that phase-retrieval processing is an invaluable tool for the reconstruction of 3D multiphase textures.

**Keywords:** Synchrotron X-ray microtomography, phase-contrast X-ray imaging, phase-retrieval, 3D rock textures, crystallization, feldspars

#### INTRODUCTION

## Overview on textural analysis of crystallized rocks with focus on feldspar crystals

The study of rock textures is important to understand the evolution of the conditions and processes that lead to their formation. Crystal abundances, sizes, shapes, and spatial distributions represent specific markers of the paragenesis of magmas during transport in the crust toward the surface and give us information on the timescales of magma ascent (Marsh 1988, 1998, 2007; Higgins 2000, 2002, 2006; Hersum and Marsh 2007; Baker et al. 2012a). Crystal and vesicle textures are conventionally studied using optical and scanning electron microscopy (SEM), which only allow two-dimensional (2D) imaging of samples. In recent years, three-dimensional (3D) analysis of rock textures, using X-ray and neutron computed microtomography (µCT), has become a fundamental tool to investigate their properties (e.g., porosity, crystallinity, crystal and vesicle size distributions, shapes, orientations, connectivity, etc.) through a non-destructive, volumetric characterization (Fig. 1). X-ray µCT allows us to image larger sample volumes

(from millimeter-sized volumes with spatial resolutions at the sub-micrometer and micrometer scale up to several tens of millimeter volumes working at spatial resolutions of the order of 40–50  $\mu$ m) than a thin section (with a typical size of 27 × 46  $\times$  0.030 mm<sup>3</sup>). X-ray  $\mu$ CT provides a realistic visualization of the 3D shapes and orientations of crystals that can be quantified through the extraction of parameters such as volume fraction, size distributions, orientation, connectivity, etc. A great advantage of 3D imaging and analysis is that it does not require any stereological corrections as in the 2D approach (Fig. 1). Moreover, no specific sample preparation is required before X-ray imaging analysis. However, like all techniques, X-ray µCT has limitations, and for some studies detailed analysis of 2D sections provides complementary data that cannot be easily obtained from using X-ray µ-CT only (Baker et al. 2012a). Highresolution SEM images can be used to segment and separate tiny crystals (<1  $\mu$ m) over large areas (typically ca. 0.5  $\times$  0.5 mm<sup>2</sup>) of the sample (Fig. 1). This approach is often complementary to X-ray µCT to characterize different aspects of the same process, such as the nucleation and twinning of crystals (e.g., Arzilli et al. 2015). Backscattered SEM images allow us to distinguish phases with similar average atomic number (e.g., feldspars and trachytic glass) and chemical information can be also obtained.

0003-004X/16/0010-2301\$05.00/DOI: http://dx.doi.org/10.2138/am-2016-5788

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2D v	s 3D
Advantages and	d special features
<ul> <li>Image mapping of large area of the sample at high resolution (pixel size &lt;1 µm).</li> <li>Phase abundance can be determined</li> <li>Latitoe orientation of crystals is available</li> <li>Chemical compositions of major and trace elements can be determined</li> </ul>	Large volume of sample can be imaged     Non-destructive 3D imaging and analysis     Textural features of the sample can be visualized in 3D     Volumetric analysis; phase abundance and size can be directly measured in 3D     Preferred orientation and connectivity of objects can be measured in 3D     Stereological conversion are not necessary     Experimental apparatus are available to perform 4D experiments
Limit	ations
Destructive procedures are needed to prepare thin sections     Stereological conversion is needed Population of crystals are described statistically	Not always possible to identify and separate easily different minerals, due to similar density contrast and crystal sizes smaller than spatial resolution - Image processing and analysis are time consuming

FIGURE 1. Comparison of 2D and 3D textural analysis approaches.

Furthermore, the ease of access to an SEM is advantageous when compared to synchrotron facilities (Baker et al. 2012a).

X-ray µCT has been successfully employed for the 3D study of the textures of igneous, metamorphic, and sedimentary rocks (e.g., Carlson 2006; Polacci et al. 2009; Baker et al. 2012a; Madonna et al. 2013; Cnudde and Boone 2013; Cilona et al. 2014; Fusseis et al. 2014; Zucali et al. 2014a, 2014b). This technique has been used to characterize the occurrence and evolution of vesiculation and crystallization recorded in different types of natural igneous and metamorphic rocks (e.g., Carlson and Denison 1992; Zandomeneghi et al. 2010; Degruyter et al. 2010a, 2010b; Voltolini et al. 2011; Baker et al. 2012b; Pamukcu et al. 2012; Polacci et al. 2012). In particular, through this technique, several authors have investigated: (1) the crystallization and fabric of metamorphic rocks (Carlson and Denison 1992; Carlson et al. 1995; Denison and Carlson 1997; Brown et al. 1999; Ketcham and Carlson 2001; Carlson 2002; Ketcham 2005; Ketcham et al. 2005; Zucali et al. 2014; Sayab et al. 2014); (2) silicate and oxide crystals dispersed in a glassy matrix of volcanic pumices and scoriae (e.g., Gualda 2006; Gualda and Rivers 2006; Pamukcu and Gualda 2010; Gualda et al. 2010; Zandomeneghi et al. 2010; Voltolini et al. 2011; Pamukcu et al. 2012); (3) kimberlite textures, by segmenting olivine phenocrysts from an altered clayrich matrix (Jerram et al. 2009); and (4) oxide and sulfide minerals associated with magmatic ore deposits (Godel et al. 2010, 2012, 2013 2014; Barnes et al. 2011); the texture of meteorites, regolith particles and chondrules, by segmenting Fe-Ni alloy and sulfide grains dispersed in a silicate matrix (Benedix et al. 2008; Ebel et al. 2008; Friedrich et al. 2008; Uesugi et al. 2010, 2013; Tsuchiyama et al. 2011, 2013).

This comprehensive, although non-exhaustive, list demonstrates that in the last 20 yr a growing use of X-ray  $\mu$ CT has occurred in the analysis of crystallized rock textures resulting in scientific and technological advancements in the capability of visualizing and characterizing rock-forming phases in 3D. However, distinguishing different crystalline phases with small variations of the attenuation coefficients still represent a challenge in X-ray imaging, both from an analytical and computational point of view.

In volcanic rock samples, vesicles are more easily resolved from the other phases in the rock (crystals and glass) because of their significantly lower X-ray attenuation. On the other hand, distinguishing among different types of ferromagnesian crystals (e.g., pyroxenes and olivine) and types of feldspars (plagioclase and alkali feldspars) is challenging because their X-ray attenuation coefficients are similar. Furthermore, in X-ray image processing feldspar is one of the most difficult phases to segment from the glassy matrix (e.g., Giachetti et al. 2011) (Fig. 2), and only a few studies have addressed the separation of feldspar from the matrix using a 3D approach (e.g., Zandomeneghi et al. 2010; Voltolini et al. 2011; Arzilli et al. 2015).

In recent years, a growing literature devoted to the establishment of sophisticated strategies to solve the problem of separation of mineral phases with similar densities in igneous rocks has been published (Uesugi et al. 1999; Tsuchiyama et al. 2000; Ketcham and Carlson 2001; Gualda and Rivers 2006; Pamukcu and Gualda 2010; Zandomeneghi et al. 2010; Voltolini et al. 2011; Pamukcu et al. 2012). One solution to this challenge is the "dual-energy X-ray microtomography" technique, which exploits the different absorption of materials at two different X-ray energies. With this approach, an X-ray µCT scan of a given sample is performed at energies above and below the X-ray absorption edge of an element characteristic of the phase to be investigated. These two images are then logarithmically subtracted to provide more X-ray contrast between two phases with similar absorption properties (e.g., Gualda et al. 2010; Tsuchiyama et al. 2013). However, data collection requires twice as much time as a single tomogram, and there are limitations in the application of this technique to major minerals of volcanic rocks, even though it is suitable for accessory minerals such as magnetite, titanite, zircon, and allanite (Gualda et al. 2010). This method is potentially applicable to rock samples crystallized under equilibrium conditions (Gualda et al. 2010; Tsuchiyama et al. 2013). However, a further limitation of such method is related to rocks that are not in equilibrium in the system, such as volcanic rocks or experimental samples. In



FIGURE 2. Plot showing the linear attenuation coefficient ( $\mu$ ) of basaltic and trachytic glasses and anorthite, albite, labradorite and sanidine crystals as a function of Energy (keV). Linear attenuation coefficients ( $\mu$ ) of each phase were obtained from the National Institute of Standards and Technology (NIST) database (http://physics.nist.gov/PhysRefData/FFast/html/form.html).

these samples, in fact, there is a great risk for overlapping X-ray attenuation coefficients among phases because the chemical zonation present in these minerals expands the range of X-ray attenuation coefficients.

#### Motivation for the study

We developed an experimental and computational procedure that can be applied to X-ray µCT images to segment and analyze phases with similar X-ray attenuation coefficients in geological specimen. We illustrate the proposed procedure using feldspar crystals and silicate glass as example of geomaterials because of the ubiquity of feldspar crystals in igneous rocks and the challenge represented by segmentation and separation of this phase in X-ray image processing.

Two samples were selected as test cases (Table 1), each containing two phases with similar X-ray attenuation coefficients that makes phase separation and segmentation via commonly used experimental protocols impossible (Zandomeneghi et al. 2010; Baker al. 2012a). The first sample was a natural pumice (sample ST164241198B) from Stromboli volcano erupted during a paroxysmal explosion on November 24, 1998. This highly vesiculated pumice contains plagioclase crystals from several hundred micrometers to a few millimeters in size, set in a basaltic glassy matrix, and surrounded by a thin layer of glass and often directly in contact with vesicles (Bertagnini et al. 1999). In this case, labradoritic plagioclase crystals and basaltic glass have similar X-ray attenuation coefficients. The second sample was a synthetic trachyte (sample D1) obtained through high-temperature, high-pressure crystallization experiments (Arzilli and Carroll 2013), resembling the composition of the magma erupted during the Campanian Ignimbrite eruption (Campi Flegrei, Italy), and containing alkali feldspar crystals in the glass. The sample texture is characterized by spherulitic alkali feldspar grown in a poorly vesiculated trachytic melt, with sizes ranging between a few and hundreds of micrometers. Spherulitic crystal shapes are characterized by interstitial glass between each lamella in spherulite. Such a complex texture makes the separation of alkali feldspars from the surrounding rock matrix particularly difficult. In this case, the similarity of alkali feldspar (sanidine) and trachytic glass X-ray attenuation coefficients (Fig. 2) makes segmentation of these crystals a challenge in X-ray µCT imaging.

#### METHOD

#### Phase-contrast µCT technique and phase-retrieval method

Transmission (or absorption) X-ray µCT yields a 3D map of the linear X-ray attenuation coefficient, which is a function of the atomic number and volumetric mass density of the material. Based on the compositional contrasts and X-ray properties of different phases, various minerals can be identified in a rock. The absorption contrast between phases in a sample is determined by variations in mass density and chemical composition for a given mineral and is based exclusively on the detection of amplitude variations of the transmitted X-rays. The use of phase-sensitive techniques allow us to increase the dynamic range measurable by detecting contrast related to the X-ray phase shifts produced by the sample in the transmitted X-ray beam. In this case, the contrast will be related to the refraction of X-rays in the regions of the sample where contact between two phases occurs. The contrast on the images will be a combination of absorption and refraction effects; the use of phase-contrast enhances the visibility of objects with similar linear attenuation coefficients by increasing the visibility of their phase boundaries (edge enhancement) (Snigirev et al. 1995; Cloetens et al. 1996). The use of phase-contrast µCT requires a high spatial coherence X-ray beam that is available at third generation synchrotron X-ray imaging beamlines due to the large source-to-sample distance and small angular source size (Cloetens et al. 1997; Fitzgerald 2000). In this case, the implementation of the technique, known as free-space propagation (FSP), is very simple from an experimental point of view: to record phase effects it is sufficient to move the detector to a specific distance from the sample (Cloetens et al. 1996). This distance gives the phase introduced by the sample the possibility to develop into intensity variations (phase contrast). If the propagation distance is chosen properly, phase-contrast will result in edge enhancement. Limited phase-contrast effects can also be observed and successfully exploited using a conventional microfocus X-ray source in FSP mode (Wilkins et al. 1996). Phase-contrast X-ray µCT has been successfully applied not only to the study of light materials (e.g., biological tissues, polymers, wood) but also to the characterization of heavy materials as metallic alloys, magnetic materials (e.g., Cloetens et al. 1997; Mancini et al. 1998, Baruchel et al. 2000), geomaterials (e.g., Polacci et al. 2006; Marinoni et al. 2009; Zandomeneghi et al. 2010; Mayo et al. 2012: Baker et al. 2012a: Fusseis et al. 2014), and to archaeological and paleonthological studies (e.g., Tafforeau et al. 2006; Bernardini et al. 2012; Zanolli et al. 2014). Synchrotron X-ray phase-contrast µCT technique based on free-space propagation was used in this study to discriminate among different phases in the investigated rock samples.

Often the segmentation of phases cannot be obtained directly from the reconstructed microtomographic images because of their low contrast even though they are perceptible by the human eve. The cases where no contrast can be observed are even worse. As a consequence, the application of phase-retrieval methods (Gureyev et al. 2006, 2008; Pfeiffer et al. 2006; Guigay et al. 2007; Beltran et al. 2010; Weitkamp et al. 2011) is needed for extracting these phases.

Phase retrieval is a technique for extracting quantitative phase information from X-ray propagation-based, phase-contrast tomographic images (Cloetens et al. 1997). Phase-retrieval procedures are widely used in biomedical applications (e.g., Langer et al. 2010; Mohammadi et al. 2014; Maire and Withers 2014), materials science (e.g., Cloetens et al. 1999; Buffière et al. 1999; Mayo et al. 2012; Maire and Withers 2014) and paleontological studies (e.g., Tafforeau et al. 2006; Smith et al. 2010), whereas their use in petrology and mineralogy is still very limited. Different procedures have been developed to extract phase information from phase-contrast X-ray images. Some approaches require recording multiple CT scans at different sample-to-detector distances [e.g., holotomography; Cloetens et al. (1997)], while others use phase-retrieval algorithms that are based on intensityonly measurements (Teague 1983; Gureyev and Nugent 1996, 1997; Paganin and Nugent 1998; Cloetens et al. 1999; Guigay et al. 2007).

Recently, rather than imaging samples at multiple distances from the detector, which is time consuming from an experimental and computational point of view, single-distance phase-retrieval algorithms have been developed. The most widely used is based on the transfer of intensity equation (TIE). It was introduced by Paganin et al. (2002) and it only requires the acquisition of one X-ray µCT data set at a single sample-to-detector distance. The method requires a priori knowledge of the complex refractive index (n) for each material present in the sample (Paganin et al. 2002; Beltran et al. 2010). The refractive index of any given material can be expressed as:

$$n = 1 - \delta + i\beta$$

where  $\delta$  is the refractive index (speed of light of a given wavelength in the material divided by speed of that light in vacuum or air) decrement and  $\beta$  is the absorption index. Both the real part,  $\delta$ , and the imaginary part,  $\beta$ , are positive and dimensionless real numbers, the imaginary part  $\beta$  describing the absorption while the real

TABLE 1. Summary of acquisition conditions and image processing details of the analyzed volumes of interest (vol) of the investigated samples
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Sample	Instrument	lsotropic voxel size (µm)	Imaged volume (pixels)	Imaged volume (mm <sup>3</sup> )	Analyzed VOI (pixels)	Analyzed VOI (mm <sup>3</sup> )
ST164241198B	SYRMEP	2.2	$1200 \times 1400 \times 1200$	21.47	$1056 \times 1398 \times 1000$	15.72
D1	SYRMEP	2.0	-	3.95	-	3.95
Notes: ST164241	198B is the nut	ural pumice of Stromboli. D1	l is the trachytic synthetic s	ample.		

part  $\delta$  describes the phase shift introduced by the material (Born and Wolf 1959; Wilkins et al. 1996; Snigirev et al. 1995; Cloetens et al. 1996). In the following, we will illustrate the application of the Paganin's phase-retrieval method to the X-ray images of our samples and discuss the results obtained from crystal segmentation.

We specify that this application of the Paganin's method did not allow us to obtain a quantitative reconstruction of the mass and electron densities of the different phases present in the rock sample; however, it allowed us to properly segment and characterize the phases of interest from a morphological and textural point of view. Paganin's method is formally limited to homogeneous objects, e.g., samples that consist of one material and air, but, in practice, it is used for multimaterial samples as well. Therefore, in these materials artifacts may appear. The artifacts consist of blurring if the ratio  $\gamma = \delta/\beta$  is higher than the value chosen in the reconstruction procedure. The method is used despite these artifacts, because multi-phase objects are described by a more-complicated relationship between absorption and phase. In fact, to properly characterize multi-material objects requires more than one image at each tomographic angle for proper phase retrieval (Burvall et al. 2011), a method too time consuming and expensive for routine X-ray  $\mu CT$ .

#### Synchrotron phase-contrast X-ray µCT measurements

Synchrotron phase-contrast X-ray  $\mu$ CT measurements were performed at the SYRMEP beamline (Tromba et al. 2010) of the Elettra-Sincrotrone Trieste laboratory (Basovizza, Italy). Elettra is a third generation synchrotron facility, and the SYRMEP beamline, devoted to the application of hard X-ray imaging techniques, operates in an energy range between 8.5 and 38 keV. The experiments were performed in white-beam mode, which at SYRMEP provides a nearly parallel, laminar-shaped X-ray beam with a maximum area of 100 mm (horizontal) × 6 mm (vertical) at 15 m from the source. An air-cooled, 16 bit microscope CCD camera with a 2048 × 2048 pixel chip (KAI 4022M CCD, Photonic Science) was used to acquire the  $\mu$ CT scans (Figs. 3a and 4a). The optical system is based on the indirect detection of X-rays: a 25 µm thick single-crystal LuAG:Ce scintillator screen, used to convert X-rays into visible light, was lens-coupled to the CCD camera. The sample-to-detector distance was set to 150 mm. For each sample 1800 radio-graphic images (or projections) were acquired by the detector with equiangular steps over a full rotation angle of 180° and an exposure time/projection of 2.1 s.

#### IMAGE PROCESSING AND RESULTS

#### Slice reconstruction and segmentation

The Syrmep\_tomo\_project 4.0 software, custom-developed at the SYRMEP beamline, and the GRIDREC algorithm (Dowd et al. 1999) were used to reconstruct the 2D axial slices from the sample projections. These slices were then converted to the 8-bit raw format and stacked by using the Fiji software (Schneider et al. 2012) to obtain volumes in which the isotropic voxel has an edge size of 2.2  $\mu$ m for the Stromboli pumice and 2  $\mu$ m for the synthetic trachytic sample. Three-dimensional visualization (volume rendering) of the reconstructed volumes was obtained with the commercial software VGStudio MAX 2.0 (Volume Graphics).

Prior to segmentation, a volume of interest (VOI) was selected for both investigated samples. For the Stromboli pumice, the VOI corresponds roughly to the whole imaged volume (Table 1), the external part of which was cropped close to the outer boundaries of the sample. The synthetic trachyte VOI coincided with the whole sample (Table 1) (Arzilli et al. 2015).

The reconstructed slices of the Stromboli pumice (Fig. 3a) show a low contrast between plagioclase, vesicles, and basaltic glass, which is why after the application of manual thresholding the Stromboli pumice binary images appear very noisy (Fig. 3b). The segmentation of alkali feldspars in the synthetic sample was impossible because the contrast of this mineral is too close to that of the glass (Fig. 4a). Therefore, in both samples the segmentation process required a pre-processing step, which





FIGURE 3. (a) Reconstructed axial slice of Stromboli pumice before phase-retrieval processing. (b) Thresholded binary image, illustrating segmentation of plagioclase crystals from basaltic glass and vesicles.



**FIGURE 4.** Reconstructed axial slice of the synthetic trachyte samples before (**a**) and after (**b**) phase-retrieval processing. The image in (**b**) shows alkali feldspar (light gray), trachytic glass (dark gray), vesicles (black), clinopyroxenes and oxides (white).

consisted in applying phase-retrieval processing to enhance the contrast between plagioclase and alkali feldspar and the glassy matrix (Figs. 3 and 4).

To achieve this objective the single-distance phase-retrieval algorithm developed by Paganin et al. (2002) was applied to the acquired radiographic images to allow segmentation of crystals from silicate glass. This algorithm combined with the Filtered Back-Projection algorithm (Herman 1980) was used to reconstruct the 3D distribution of the complex X-ray refractive index within the sample. This processing was performed using the commercial software package X-TRACT (Paganin et al. 2002; Mohammadi et al. 2014), version 5.8, developed by the CSIRO group (http://xrsi.cmit.csiro.au/WebHelp/X-TRACT/webframe. html, Canberra, Australia) (Fig. 5a), although alternative freeware solutions to the application of the Paganin's algorithm and synchrotron X-ray CT reconstruction could have been used (Weitkamp et al. 2011; Chen et al. 2012; Rivers 2012; Gursoy et al. 2014; Brun et al. 2015).

The ratio y between the real and imaginary part of the refractive index is constant at a given wavelength for a homogeneous material. We calculated the  $\delta$  and  $\beta$  values using the freeware from the Center for X-ray Optics database (http://henke.lbl.gov/ optical constants/getdb2.html) (Henke et al. 1993). The refractive index can be obtained from the chemical formula of the phase of interest, its mass density (g/cm3) and the photon energy (keV) used during the X-ray µCT acquisition (Figs. 5 and 6). In white beam mode, the energy selection was done considering the medium energy of the spectrum employed during the experiment (22 keV). The mass density of feldspar was obtained from the Mindat web site (http://www.mindat.org; Ralph and Chau 2014): the density of labradorite (2.69 g/cm3) was used for the Stromboli plagioclase crystals, and the density of sanidine (2.52 g/cm<sup>3</sup>) was used for the synthetic trachyte alkali feldspar. Figure 6 shows the variation of  $\gamma$  as a function of the photon energy. The calculated  $\gamma$  value for plagioclase (labradorite) in the natural Stromboli pumice is 353, whereas, for alkali feldspar (sanidine) in the synthetic sample,  $\gamma$  is 362.

The initially calculated  $\gamma$  values (Fig. 6) did not sufficiently



FIGURE 5. Scheme of the proposed phase-retrieval procedure. # Commercial software; \* open-source software.



**FIGURE 6.** Plot of  $\gamma = \delta/\beta$  for basaltic and trachytic glasses and labradorite, anorthite, anorthoclase, and sanidine crystals as a function of Energy (keV). The refractive index was obtained using the CXRO X-ray database (http://henke.lbl.gov/optical\_constants/) (Henke et al. 1993).

increase the contrast among phases. To enhance contrast these values were tuned by visual inspection to achieve two results: (1) reduction of phase-contrast "artifacts" at the edges of the crystals and (2) enhancement of contrast between feldspars and glass. Unfortunately, blurring of the images is generally a consequence of the application of the TIE phase-retrieval algorithm. Therefore, for each data set a few iterations were needed to optimize the  $\gamma$  parameter to obtain the best contrast while minimizing blurring effects. The initial  $\gamma$  value for plagioclase (labradorite) in



FIGURE 7. Comparison of edge effects between phase-contrast and phase-retrieved images. (a) Raw image obtained in phase-contrast mode for Stromboli basaltic pumice. (b) Result from the TIE phase-retrieval algorithm with a  $\delta$ -to- $\beta$  ratio optimized for plagioclase of the Stromboli basaltic pumice. (c) Raw image obtained in phase-contrast mode for the synthetic trachyte. (b) Result from the TIE phase-retrieval algorithm with a  $\delta$ -to- $\beta$  ratio optimized for sanidine of the trachyte.

the natural Stromboli pumice was then tuned to  $\gamma = 120$ . The same procedure was applied to alkali feldspar (sanidine) in the synthetic sample, tuning the  $\gamma$  value to 50 (Arzilli et al. 2015). The final results of this procedure strongly enhance the contrast between plagioclase crystals, vesicles, and glass in the natural pumice (Figs. 7b and 8a). These images can be compared to the results of reconstruction without phase-retrieval processing presented in Figures 3a and 7a. A similar increase in contrast was obtained for the alkali feldspars and trachytic glass in the

synthetic sample (Figs. 4b and 7c-7d).

After phase retrieval, the reconstructed slices of the Stromboli pumice sample were corrupted by artifacts with the appearance of concentric rings and named "ring artifacts" in CT imaging (Fig. 7b). These artifacts may arise in synchrotron radiation  $\mu$ CT from dead pixels in CCD detectors, damaged scintillator screens, and instabilities of the synchrotron beam (Rivers 1998; Titarenko et al. 2010; Van Nieuwenhove et al. 2015). The version of the X-TRACT software we used in this work allowed us to only apply a ring-removing filter developed by Rivers (1998) that is based on sinogram-processing. In our study, the Rivers's filter was insufficient to remove all ring artifacts in the data set. However, because the majority of plagioclase crystals were not corrupted by rings this allowed us to correctly perform their segmentation. In more complicated cases, other filters may need to be applied to reduce ring artifacts during the reconstruction procedure (e.g., Sijbers and Postnov 2004; Münch et al. 2009). Some freeware for CT reconstruction (Marone et al. 2010; Chen et al. 2012; Gursoy et al. 2014; Brun et al. 2015) allows the application of these filters for ring artifact reduction.

### Segmentation of plagioclase crystals in the natural Stromboli pumice

The next step in image processing was segmentation. This process allows the separation of objects from the background to obtain binary volumes containing only the feature of interest. Several segmentation techniques are available, but there is no single method successfully applicable to all cases and the procedure is sample-dependent. Segmentation was performed with the Pore3D software library (Brun et al. 2010). To this purpose, we applied a manual bi-level thresholding in 3D that allowed us to segment the features of interest from the background by selecting a threshold value (Pal and Pal 1993) based on the analysis of the histogram of gray levels in the VOI (selecting the threshold in the region near two peaks). The oscillatory compositional zoning of the plagioclase phenocrysts in the Stromboli samples was not resolvable using phase-contrast X-ray  $\mu$ CT. Therefore, using the phase-retrieval procedure we assumed a homogeneous plagioclase crystal density. Plagioclase crystals were separated from vesicles and glass through segmentation combined with a masking procedure based on plagioclase shape (Faessel and Jeulin 2010; Lin et al. 2015).

The image background of our Stromboli pumice was affected by Poisson noise, which consists of random variations in brightness levels that can corrupt the image quality (Lev et al. 1977; Le et al. 2007). Because of the inherent resolution limitations of the X-ray  $\mu$ CT technique, images were also affected by







partial-volume effects (Ketcham 2005), which consist of variations in attenuation shown by each pixel and result in a blurring of material boundaries (the material in any one voxel can affect X-ray attenuation values of surrounding voxels). Variations in attenuation are due to chemical zonation in plagioclase, chemical heterogeneities in the glass, and limited spatial resolution. Poisson noise and partial-volume effects made the segmentation of plagioclase difficult. As an example, Figures 8b–8c shows pixel outliers belonging to both vesicles and glassy matrix that were not segmented after thresholding. We know there are no plagioclase crystals in vesicles, and so we have an internal gauge of signal, which we then used to assess the accuracy of the rest of the image.

Following this approach, the masking procedure consisted in segmenting plagioclase via two different steps:

(1) The first step consisted of manual bi-level thresholding, using the Pore3D software, to segment the realistic shape of plagioclase crystals taking into account a significant amount of pixel outliers left in the binary images (Figs. 8b and 9).

(2) The second step consisted in the approximate isolation of plagioclase trying to take into account the minimum possible amount of pixel outliers (Fig. 8c) with the aim of obtaining a mask of the plagioclase shapes (Fig. 8d). The amount of pixel outliers was partially removed using a sequence of filters on the segmented, binary image. First, we used a 3D filter removing outliers with the Pore3D software library and, subsequently, a similar 2D filter with the Fiji software (Fig. 9). Through these filters we were able to delete connected components with volume sizes below a specified threshold value based upon visual inspection (Figs. 8c-8d and 9). The combined use of 3D and 2D filters applied to binary images allowed us to remove most of the pixel outliers without changing crystal shapes. The shapes of plagioclase crystals were isolated using 4 cycles of 2D erosion and dilation filters in Fiji. To avoid excessive plagioclase shape change when applying the erosion filter, the internal voids of plagioclase crystals were filled during the masking process, which also produced crystals that are more compact. Through the second step of segmentation, a mask of plagioclase crystals

was obtained (Figs. 8d and 9).

The masking operation was combined with the first segmentation by applying the logic "AND" operator in Fiji (Figs. 8b–8d) to complete the separation of the phases of interest (Figs. 8e and 9). The plagioclase crystals were completely segmented preserving their shapes and internal structures (Figs. 8e and 10a). This method had the additional benefit of reducing the effect of the ring artifacts on the segmentation of plagioclase crystals. The procedure used for the Stromboli's sample, including phase retrieval and segmentation, can take about 3 h per each sample.

### Segmentation of alkali feldspar crystals in the trachytic synthetic sample

In the synthetic trachyte, semi-automatic volume segmentation (Zanolli et al. 2014; Arzilli et al. 2015) with manual corrections was performed to separate crystals of alkali feldspar from the glassy matrix by using the AMIRA software v.4.1.2 (Mercury Computer Systems). Semi-automatic volume segmentation with manual corrections can also be performed with the Avizo 3D software (FEI Visualization Sciences Group). This segmentation consists of manually drawing the edges of the crystals on the 2D slices. This is repeated every 5-10 slices (this interval depends on the size of the crystal and the complexity of their shape) from the beginning to the end of the crystal, while in the intermediate slices the crystal shape will be reconstructed automatically by the software (via an interpolation procedure). The advantage of this technique is that the operator can obtain the real morphology of the object of interest by visual inspection (Fig. 10b). This technique can be time consuming for a large numbers of crystals, but it is a valuable approach in specific cases related to the study of crystal nucleation, twinning, and crystal lattice orientation (Arzilli et al. 2015).

#### IMPLICATIONS

Recently, several authors have discussed the effect of X-rays on attenuation and transmission to distinguish between different minerals and glass compositions using X-ray microtomography (e.g., Tsuchiyama et al. 2005, 2013; Gualda and Rivers 2006; Gualda et al. 2010; Baker et al. 2012a). The X-ray attenuation coefficient for magnetite is larger than that of quartz, feldspar, or glass, making dense minerals easily distinguishable from silicic glasses and felsic minerals (Gualda and Rivers 2006; Gualda et al. 2010), whereas the X-ray attenuation coefficients for quartz and feldspar are very close to that of several silicate glasses and segmentation is very challenging (e.g., Gualda and Rivers 2006; Pamukcu et al. 2010; Voltolini et al. 2011; Arzilli et al. 2015). Therefore, the main contribution of this study to petrology is to propose a novel application of the phase-retrieval procedure in 3D image processing to resolve feldspar crystals in multiphase crystallized rocks. This procedure is important for the volcanological and petrological community because it opens new avenues in the 3D study of crystallization kinetics of silicate melts, which, together with degassing, is the most important process driving magma and eruption dynamics.

Despite the observation that the phase-retrieval method employed here has limitations, such as a slight blurring of images, which in our work was kept to a minimum to avoid





FIGURE 10. Volume renderings of crystals. (a) Volume renderings of the segmented plagioclases in Stromboli pumice sample; the maximum length of the largest crystal is  $\sim 1$  mm. (b) Volume rendering of the segmented alkali feldspars in the synthetic trachyte sample; the average length of each lamella within spherulites is 150 µm.

significant changes in crystal shape, it represents a fundamental step in image processing to increase the contrast among minerals displaying only slightly different X-ray attenuations. This means that small differences in density and chemical composition can be resolved through phase-contrast imaging, as already shown in paleontological specimens, materials science, and biomedical applications (e.g., Tafforeau et al. 2006; Smith et al. 2010; Langer et al. 2010; Mayo et al. 2012; Mohammadi et al. 2014). Another limitation is the need to use a suite of software tools to execute the protocol; we expect that in the near future a single software package will contain all of the algorithms necessary, which will accelerate the data processing. The techniques shown here can

be applied to a wide range of natural rocks and synthetic samples characterized by the presence of crystals with sizes between a few micrometers to a few millimeters and phases with similar X-ray attenuation coefficients.

The results of this study highlight that careful application of a phase-retrieval algorithm to synchrotron X-ray microtomographic data sets of volcanic rocks can provide fundamental information for 3D quantitative analysis of magmatic crystallization processes. We envisage that future work will expand the range of application of this procedure to different geomaterials. It is worth noting that our imaging protocol can be further refined [for instance using a phase-retrieval approach for multi-material objects as proposed by Beltran et al. (2010)] and also applied to older data sets to extract data that could not be extracted before.

#### **ACKNOWLEDGMENTS**

We thank M.R. Carroll for providing the trachytic sample, S. Mohammadi for her precious help with the X-TRACT software, and D. Dreossi, M. Voltolini, and F. Brun for helpful discussions. We are grateful to C. Zanolli for useful advice on the Amira software. M. Pankhurst, T. Giachetti, an anonymous reviewer, Editor K. Putirka, and Associate Editor T. Shea are acknowledged for helpful suggestions and comments that have greatly improved this manuscript. Elettra is kindly acknowledged for providing in-kind fundings through proposal no. 20120015 (PI Margherita Polacci).

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MANUSCRIPT RECEIVED MARCH 30, 2016 MANUSCRIPT ACCEPTED JUNE 7, 2016 MANUSCRIPT HANDLED BY THOMAS SHEA