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

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1 Revision 1

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3 **A novel protocol for resolving feldspar crystals in synchrotron X-ray microtomographic**  
4 **images of crystallized natural magmas and synthetic analogues**

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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 micron and sub-micron 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 analogue. Our results demonstrate that crystallized natural volcanic rocks and synthetic analogues 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

51

## INTRODUCTION

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

53 The study of rock textures is important to understand the evolution of the conditions and  
54 processes that lead to their formation. Crystal abundances, sizes, shapes and spatial distributions  
55 represent specific markers of the paragenesis of magmas during transport in the crust towards the  
56 surface and give us information on the time scales of magma ascent (Marsh 1988, 1998; Higgins  
57 2000, 2002, 2006; Hersum and Marsh 2007; Marsh 2007; Baker et al. 2012a). Crystal and vesicle  
58 textures are conventionally studied using optical and Scanning Electron Microscopy (SEM), which  
59 only allow two-dimensional (2D) imaging of samples. In recent years, three-dimensional (3D)  
60 analysis of rock textures, using X-ray and neutron computed microtomography ( $\mu$ CT), has become  
61 a fundamental tool to investigate their properties (e.g. porosity, crystallinity, crystal and vesicle size  
62 distributions, shapes, orientations, connectivity, etc.) through a non-destructive, volumetric  
63 characterization (Fig. 1). X-ray  $\mu$ CT allows us to image larger sample volumes (from mm-sized  
64 volumes with spatial resolutions at the sub-micron and micron scale up to several tens of mm  
65 volumes working at spatial resolutions of the order of 40-50 microns) than a thin section (with a  
66 typical size of 27 x 46 x 0.030 mm<sup>3</sup>). X-ray  $\mu$ CT provides a realistic visualization of the 3D shapes  
67 and orientations of crystals that can be quantified through the extraction of parameters such as  
68 volume fraction, size distributions, orientation, connectivity, etc. A great advantage of 3D imaging  
69 and analysis is that it does not require any stereological corrections as in the 2D approach (Fig. 1).  
70 Moreover, no specific sample preparation is required before X-ray imaging analysis. However, like  
71 all techniques, X-ray  $\mu$ CT has limitations, and for some studies detailed analysis of 2D sections  
72 provides complementary data that cannot be easily obtained from using X-ray  $\mu$ -CT only (Baker et  
73 al. 2012a). High resolution SEM images can be used to segment and separate tiny crystals (<1  $\mu$ m)  
74 over large areas (typically ca. 0.5 mm x 0.5 mm<sup>2</sup>) of the sample (Fig. 1). This approach is often  
75 complementary to X-ray  $\mu$ CT to characterize different aspects of the same process, such as the

76 nucleation and twinning of crystals (e.g., Arzilli et al. 2015). Back-scattered SEM images allow us  
77 to distinguish phases with similar average atomic number (e.g., feldspars and trachytic glass) and  
78 chemical information can be also obtained. Furthermore, the ease of access to an SEM is  
79 advantageous when compared to synchrotron facilities (Baker et al. 2012a).

80 X-ray  $\mu$ CT has been successfully employed for the 3D study of the textures of igneous,  
81 metamorphic and sedimentary rocks (e.g., Carlson 2006; Polacci et al. 2009; Baker et al. 2012a;  
82 Madonna et al. 2013; Cnudde and Boone 2013; Cilona et al. 2014; Fuisseis et al. 2014; Zucali et al.  
83 2014a,b). This technique has been used to characterize the occurrence and evolution of vesiculation  
84 and crystallization recorded in different types of natural igneous and metamorphic rocks (e.g.,  
85 Carlson and Denison 1992; Zandomeneghi et al. 2010; Degruyter et al. 2010a,b; Voltolini et al.  
86 2011; Baker et al. 2012b; Pamukcu et al. 2012; Polacci et al. 2012). In particular, through this  
87 technique, several authors have investigated i) the crystallization and fabric of metamorphic rocks  
88 (Carlson and Denison 1992; Carlson et al. 1995; Denison and Carlson 1997; Brown et al. 1999;  
89 Ketcham and Carlson 2001; Carlson 2002; Ketcham 2005; Ketcham et al. 2005; Zucali et al. 2014;  
90 Sayab et al. 2014); ii) silicate and oxide crystals dispersed in a glassy matrix of volcanic pumices  
91 and scoriae (e.g., Gualda 2006; Gualda and Rivers 2006; Pamukcu and Gualda 2010; Gualda et al.  
92 2010; Zandomeneghi et al. 2010; Voltolini et al. 2011; Pamukcu et al. 2012); iii) kimberlite  
93 textures, by segmenting olivine phenocrysts from an altered clay-rich matrix (Jerram et al. 2009);  
94 iv) oxide and sulfide minerals associated with magmatic ore deposits (Godel et al. 2010; Barnes et  
95 al. 2011; Godel et al. 2012; Godel et al. 2013, 2014); the texture of meteorites, regolith particles and  
96 chondrules, by segmenting Fe–Ni alloy and sulfide grains dispersed in a silicate matrix (Benedix et  
97 al. 2008; Ebel et al. 2008; Friedrich et al. 2008; Uesugi et al. 2010; Tsuchiyama et al. 2011; Uesugi  
98 et al. 2013; Tsuchiyama et al. 2013).

99 This comprehensive, although non-exhaustive, list demonstrates that in the last 20 years a  
100 growing use of X-ray  $\mu$ CT has occurred in the analysis of crystallized rock textures resulting in

101 scientific and technological advancements in the capability of visualizing and characterizing rock-  
102 forming phases in 3D. However, distinguishing different crystalline phases with small variations of  
103 the attenuation coefficients still represent a challenge in X-ray imaging, both from an analytical and  
104 computational point of view.

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

113 In recent years, a growing literature devoted to the establishment of sophisticated strategies to  
114 solve the problem of separation of mineral phases with similar densities in igneous rocks has been  
115 published (Uesugi et al. 1999; Tsuchiyama et al. 2000; Ketcham and Carlson 2001; Gualda and  
116 Rivers 2006; Pamukcu and Gualda 2010; Zandomenegi et al. 2010; Voltolini et al. 2011; Pamukcu  
117 et al. 2012). One solution to this challenge is the “dual-energy X-ray microtomography” technique,  
118 which exploits the different absorption of materials at two different X-ray energies. With this  
119 approach, an X-ray  $\mu$ CT scan of a given sample is performed at energies above and below the X-ray  
120 absorption edge of an element characteristic of the phase to be investigated. These two images are  
121 then logarithmically subtracted to provide more X-ray contrast between two phases with similar  
122 absorption properties (e.g., Gualda et al. 2010; Tsuchiyama et al. 2013). However, data collection  
123 requires twice as much time as a single tomogram, and there are limitations in the application of this  
124 technique to major minerals of volcanic rocks, even though it is suitable for accessory minerals such  
125 as magnetite, titanite, zircon and allanite (Gualda et al. 2010). This method is potentially applicable

126 to rock samples crystallized under equilibrium conditions (Gualda et al. 2010; Tsuchiyama et al.  
127 2013). However, a further limitation of such method is related to rocks that are not in equilibrium in  
128 the system, such as volcanic rocks or experimental samples. In these samples, in fact, there is a  
129 great risk for overlapping X-ray attenuation coefficients among phases because the chemical  
130 zonation present in these minerals expands the range of X-ray attenuation coefficients.

131

### 132 **Motivation for the study**

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

138 Two samples were selected as test cases (Table 1), each containing two phases with similar X-  
139 ray attenuation coefficients that makes phase separation and segmentation via commonly used  
140 experimental protocols impossible (Zandomenighi et al. 2010; Baker al. 2012a). The first sample  
141 was a natural pumice (sample ST164241198B) from Stromboli volcano erupted during a  
142 paroxysmal explosion on the 24th of November 1998. This highly vesiculated pumice contains  
143 plagioclase crystals from several hundred  $\mu$ m to a few mm in size, set in a basaltic glassy matrix,  
144 and surrounded by a thin layer of glass and often directly in contact with vesicles (Bertagnini et al.  
145 1999). In this case, labradoritic plagioclase crystals and basaltic glass have similar X-ray  
146 attenuation coefficients. The second sample was a synthetic trachyte (sample D1) obtained through  
147 high temperature, high pressure crystallization experiments (Arzilli and Carroll 2013), resembling  
148 the composition of the magma erupted during the Campanian Ignimbrite eruption (Campi Flegrei,  
149 Italy), and containing alkali feldspar crystals in the glass. The sample texture is characterized by  
150 spherulitic alkali feldspar grown in a poorly vesiculated trachytic melt, with sizes ranging between a

151 few and hundred  $\mu\text{m}$ . Spherulitic crystal shapes are characterized by interstitial glass between each  
152 lamella in spherulite. Such a complex texture makes the separation of alkali feldspars from the  
153 surrounding rock matrix particularly difficult. In this case, the similarity of alkali feldspar (sanidine)  
154 and trachytic glass X-ray attenuation coefficients (Fig. 2) makes segmentation of these crystals a  
155 challenge in X-ray  $\mu\text{CT}$  imaging.

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157

## METHODS

### 158 **Phase-contrast $\mu\text{CT}$ technique and phase-retrieval method**

159 Transmission (or absorption) X-ray  $\mu\text{CT}$  yields a 3D map of the linear X-ray attenuation  
160 coefficient, which is a function of the atomic number and volumetric mass density of the material.  
161 Based on the compositional contrasts and X-ray properties of different phases, various minerals can  
162 be identified in a rock. The absorption contrast between phases in a sample is determined by  
163 variations in mass density and chemical composition for a given mineral and is based exclusively  
164 on the detection of amplitude variations of the transmitted X-rays. The use of phase-sensitive  
165 techniques allow us to increase the dynamic range measurable by detecting contrast related to the  
166 X-ray phase shifts produced by the sample in the transmitted X-ray beam. In this case, the contrast  
167 will be related to the refraction of X-rays in the regions of the sample where contact between two  
168 phases occurs. The contrast on the images will be a combination of absorption and refraction  
169 effects; the use of phase-contrast enhances the visibility of objects with similar linear attenuation  
170 coefficients by increasing the visibility of their phase boundaries (edge enhancement) (Snigerev et  
171 al. 1995; Cloetens et al. 1996). The use of phase-contrast  $\mu\text{CT}$  requires a high spatial coherence X-  
172 ray beam that is available at third generation synchrotron X-ray imaging beamlines due to the large  
173 source-to-sample distance and small angular source size (Cloetens et al. 1997; Fitzgerald 2000). In  
174 this case, the implementation of the technique, known as Free-Space Propagation (FSP), is very  
175 simple from an experimental point of view: in order to record phase effects it is sufficient to move



176 the detector to a specific distance from the sample (Cloetens et al., 1996). This distance gives the  
177 phase introduced by the sample the possibility to develop into intensity variations (phase contrast).  
178 If the propagation distance is chosen properly, phase-contrast will result in edge enhancement.  
179 Limited phase-contrast effects can also be observed and successfully exploited using a conventional  
180 microfocus X-ray source in FSP mode (Wilkins et al. 1996). Phase-contrast X-ray  $\mu$ CT has been  
181 successfully applied not only to the study of light materials (e.g., biological tissues, polymers,  
182 wood) but also to the characterization of heavy materials as metallic alloys, magnetic materials  
183 (e.g., Cloetens et al. 1997; Mancini et al. 1998, Baruchel et al. 2000), geomaterials (e.g., Polacci et  
184 al. 2006; Marinoni et al., 2009; Zandomenighi et al. 2010; Mayo et al. 2012; Baker et al. 2012a;  
185 Fuisseis et al. 2014) and to archaeological and paleontological studies (e.g., Tafforeau et al. 2006;  
186 Bernardini et al., 2012; Zanolli et al., 2014). Synchrotron X-ray phase-contrast  $\mu$ CT technique  
187 based on free-space propagation was used in this study to discriminate among different phases in  
188 the investigated rock samples.

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

195 Phase retrieval is a technique for extracting quantitative phase information from X-ray  
196 propagation-based, phase-contrast tomographic images (Cloetens et al., 1997). Phase-retrieval  
197 procedures are widely used in biomedical applications (e.g., Langer et al. 2010; Mohammadi et al.  
198 2014; Maire and Withers 2014), materials science (e.g. Cloetens et al., 1999; Buffière et al. 1999;  
199 Mayo et al. 2012; Maire and Withers 2014) and paleontological studies (e.g., Tafforeau et al. 2006;  
200 Smith et al. 2010), whereas their use in petrology and mineralogy is still very limited. Different

201 procedures have been developed to extract phase information from phase-contrast X-ray images.  
202 Some approaches require recording multiple CT scans at different sample-to-detector distances  
203 (e.g., holotomography; Cloetens et al., 1997), while others use phase-retrieval algorithms that are  
204 based on intensity-only measurements (Teague 1983; Gureyev and Nugent 1996, 1997; Paganin and  
205 Nugent 1998; Cloetens et al. 1999; Guigay et al. 2007).

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

$$213 \quad n = 1 - \delta + i\beta$$

214 where  $\delta$  is the refractive index (speed of light of a given wavelength in the material divided by  
215 speed of that light in vacuum or air) decrement and  $\beta$  is the absorption index. Both the real part,  $\delta$ ,  
216 and the imaginary part,  $\beta$ , are positive and dimensionless real numbers, the imaginary part  $\beta$   
217 describing the absorption while the real part  $\delta$  describes the phase shift introduced by the material  
218 (Born and Wolf, 1959; Wilkins et al., 1995; Snigerev et al., 1995; Cloetens et al., 1996). In the  
219 following, we will illustrate the application of the Paganin's phase-retrieval method to the X-ray  
220 images of our samples and discuss the results obtained from crystal segmentation.

221 We specify that this application of the Paganin's method did not allow us to obtain a quantitative  
222 reconstruction of the mass and electron densities of the different phases present in the rock sample;  
223 however, it allowed us to properly segment and characterize the phases of interest from a  
224 morphological and textural point of view. Paganin's method is formally limited to homogeneous  
225 objects, e.g., samples that consist of one material and air, but, in practice, it is used for multi-

226 material samples as well. Therefore, in these materials artifacts may appear. The artifacts consist of  
227 blurring if the ratio  $\gamma = \delta/\beta$  is higher than the value chosen in the reconstruction procedure. The  
228 method is used despite these artifacts, because multi-phase objects are described by a more-  
229 complicated relationship between absorption and phase. In fact, to properly characterize multi-  
230 material objects requires more than one image at each tomographic angle for proper phase retrieval  
231 (Burvall et al. 2011), a method too time-consuming and expensive for routine X-ray  $\mu$ CT.

232

### 233 **Synchrotron phase-contrast X-ray $\mu$ CT measurements**

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

247

## 248 **IMAGE PROCESSING AND RESULTS**

### 249 **Slice reconstruction and segmentation**

250 The Syrmep\_tomo\_project 4.0 software, custom-developed at the SYRMEP beamline, and the  
251 GRIDREC algorithm (Dowd et al. 1987) were used to reconstruct the 2D axial slices from the  
252 sample projections. These slices were then converted to the 8-bit raw format and stacked by using  
253 the *Fiji* software (Schneider et al. 2012) to obtain volumes in which the isotropic voxel has an edge  
254 size of 2.2  $\mu\text{m}$  for the Stromboli pumice and 2  $\mu\text{m}$  for the synthetic trachytic sample. 3D  
255 visualization (volume rendering) of the reconstructed volumes was obtained with the commercial  
256 software VGStudio MAX 2.0 (Volume Graphics).

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

261 The reconstructed slices of the Stromboli pumice (Fig. 3a) show a low contrast between  
262 plagioclase, vesicles and basaltic glass, which is why after the application of manual thresholding  
263 the Stromboli pumice binary images appear very noisy (Fig. 3b). The segmentation of alkali  
264 feldspars in the synthetic sample was impossible because the contrast of this mineral is too close to  
265 that of the glass (Fig. 4a). Therefore, in both samples the segmentation process required a pre-  
266 processing step, which consisted in applying phase-retrieval processing in order to enhance the  
267 contrast between plagioclase and alkali feldspar and the glassy matrix (Figs 3 and 4).

268 To achieve this objective the single-distance phase-retrieval algorithm developed by Paganin et  
269 al. (2002) was applied to the acquired radiographic images to allow segmentation of crystals from  
270 silicate glass. This algorithm combined with the Filtered Back-Projection algorithm (Herman 1980)  
271 was used to reconstruct the 3D distribution of the complex X-ray refractive index within the sample.  
272 This processing was performed using the commercial software package X-TRACT (Paganin et al.  
273 2002; Mohammadi et al. 2014), version 5.8, developed by the CSIRO group  
274 (<http://xrsi.cmit.csiro.au/WebHelp/X-TRACT/webframe.html>, Canberra, Australia) (Fig. 5a),

275 although alternative freeware solutions to the application of the Paganin's algorithm and  
276 synchrotron X-ray CT reconstruction could have been used (Weitkamp et al. 2011; Chen et al.  
277 2012; Rivers 2012; Gursoy et al. 2014; Brun et al. 2015).

278 The ratio  $\gamma$  between the real and imaginary part of the refractive index is constant at a given  
279 wavelength for a homogeneous material. We calculated the  $\delta$  and  $\beta$  values using the freeware from  
280 the Center for X-ray Optics database ([http://henke.lbl.gov/optical\\_constants/getdb2.html](http://henke.lbl.gov/optical_constants/getdb2.html)) (Henke et  
281 al. 1993). The refractive index can be obtained from the chemical formula of the phase of interest,  
282 its mass density ( $\text{g/cm}^3$ ) and the photon energy (keV) used during the X-ray  $\mu$ CT acquisition (Figs.  
283 5 and 6). In white beam mode, the energy selection was done considering the medium energy of the  
284 spectrum employed during the experiment (22 keV). The mass density of feldspar was obtained  
285 from the Mindat website (<http://www.mindat.org>; Ralph and Chau, 2014): the density of labradorite  
286 ( $2.69 \text{ g/cm}^3$ ) was used for the Stromboli plagioclase crystals, and the density of sanidine ( $2.52$   
287  $\text{g/cm}^3$ ) was used for the synthetic trachyte alkali feldspar. Figure 6 shows the variation of  $\gamma$  as a  
288 function of the photon energy. The calculated  $\gamma$  value for plagioclase (labradorite) in the natural  
289 Stromboli pumice is 353, whereas, for alkali feldspar (sanidine) in the synthetic sample  $\gamma$  is 362.

290 The initially calculated  $\gamma$  values (Fig. 6) did not sufficiently increase the contrast among phases.  
291 In order to enhance contrast these values were tuned by visual inspection to achieve two results: i)  
292 reduction of phase-contrast "artifacts" at the edges of the crystals and ii) enhancement of contrast  
293 between feldspars and glass. Unfortunately, blurring of the images is generally a consequence of the  
294 application of the TIE phase-retrieval algorithm. Therefore, for each data set a few iterations were  
295 needed in order to optimize the  $\gamma$  parameter to obtain the best contrast while minimizing blurring  
296 effects. The initial  $\gamma$  value for plagioclase (labradorite) in the natural Stromboli pumice was then  
297 tuned to  $\gamma = 120$ . The same procedure was applied to alkali feldspar (sanidine) in the synthetic  
298 sample, tuning the  $\gamma$  value to 50 (Arzilli et al. 2015). The final results of this procedure strongly

299 enhance the contrast between plagioclase crystals, vesicles and glass in the natural pumice (Figs 7b  
300 and 8a). These images can be compared to the results of reconstruction without phase-retrieval  
301 processing presented in Figs 3a and 7a. A similar increase in contrast was obtained for the alkali  
302 feldspars and trachytic glass in the synthetic sample (Figs 4b and 7c-d).

303 After phase retrieval, the reconstructed slices of the Stromboli pumice sample were corrupted by  
304 artifacts with the appearance of concentric rings and named ‘ring artifacts’ in CT imaging (Fig. 7b).  
305 These artifacts may arise in synchrotron radiation  $\mu$ CT from dead pixels in CCD detectors,  
306 damaged scintillator screens and instabilities of the synchrotron beam (Rivers 1998; Titarenko et al.  
307 2010; Van Nieuwenhove 2015). The version of the X-TRACT software we used in this work  
308 allowed us to only apply a ring-removing filter developed by Rivers (1998) that is based on  
309 sinogram-processing. In our study, the Rivers’s filter was insufficient to remove all ring artifacts in  
310 the data set. However, because the majority of plagioclase crystals were not corrupted by rings this  
311 allowed us to correctly perform their segmentation. In more complicated cases, other filters may  
312 need to be applied to reduce ring artifacts during the reconstruction procedure (e.g. Sijbers and  
313 Postonov 2004; Münch et al. 2009). Some freeware for CT reconstruction (Marone et al. 2010;  
314 Chen et al. 2012; Gursoy et al. 2014, Brun et al. 2015) allows the application of these filters for ring  
315 artifact reduction.

316

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

318 The next step in image processing was segmentation. This process allows the separation of  
319 objects from the background to obtain binary volumes containing only the feature of interest.  
320 Several segmentation techniques are available, but there is no single method successfully applicable  
321 to all cases and the procedure is sample-dependent. Segmentation was performed with the *Pore3D*  
322 software library (Brun et al. 2010). To this purpose, we applied a manual bi-level thresholding in  
323 3D that allowed us to segment the features of interest from the background by selecting a threshold

324 value (Pal and Pal 1993) based on the analysis of the histogram of gray levels in the VOI (selecting  
325 the threshold in the region near two peaks). The oscillatory compositional zoning of the plagioclase  
326 phenocrysts in the Stromboli samples was not resolvable using phase-contrast X-ray  $\mu$ CT.  
327 Therefore, using the phase-retrieval procedure we assumed a homogeneous plagioclase crystal  
328 density. Plagioclase crystals were separated from vesicles and glass through segmentation combined  
329 with a masking procedure based on plagioclase shape (Faessel and Jeulin 2010; Lin et al. 2015).

330 The image background of our Stromboli pumice was affected by Poisson noise, which consists  
331 of random variations in brightness levels that can corrupt the image quality (Lev et al. 1977; Le et  
332 al. 2007). Because of the inherent resolution limitations of the X-ray  $\mu$ CT technique, images were  
333 also affected by partial-volume effects (Ketcham 2005), which consist of variations in attenuation  
334 shown by each pixel and result in a blurring of material boundaries (the material in any one voxel  
335 can affect X-ray attenuation values of surrounding voxels). Variations in attenuation are due to  
336 chemical zonation in plagioclase, chemical heterogeneities in the glass, and limited spatial  
337 resolution. Poisson noise and partial-volume effects made the segmentation of plagioclase difficult.  
338 As an example, Fig. 8b-c shows pixel outliers belonging to both vesicles and glassy matrix that  
339 were not segmented after thresholding. We know there are no plagioclase crystals in vesicles, and  
340 so we have an internal gauge of signal, which we then used to assess the accuracy of the rest of the  
341 image.

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

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

347 b) The second step consisted in the approximate isolation of plagioclase trying to take into  
348 account the minimum possible amount of pixel outliers (Fig. 8c) with the aim of obtaining a mask

349 of the plagioclase shapes (Fig. 8d). The amount of pixel outliers was partially removed using a  
350 sequence of filters on the segmented, binary image. First, we used a 3D filter removing outliers with  
351 the *Pore3D* software library and, subsequently, a similar 2D filter with the *Fiji* software (Fig. 9).  
352 Through these filters we were able to delete connected components with volume sizes below a  
353 specified threshold value based upon visual inspection (Figs. 8c-d and 9). The combined use of 3D  
354 and 2D filters applied to binary images allowed us to remove most of the pixel outliers without  
355 changing crystal shapes. The shapes of plagioclase crystals were isolated using 4 cycles of 2D  
356 erosion and dilation filters in *Fiji*. In order to avoid excessive plagioclase shape change when  
357 applying the erosion filter, the internal voids of plagioclase crystals were filled during the masking  
358 process, which also produced crystals that are more compact. Through the second step of  
359 segmentation, a mask of plagioclase crystals was obtained (Figs. 8d and 9).

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

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

368 In the synthetic trachyte, semi-automatic volume segmentation (Zanolli et al. 2014; Arzilli et al.  
369 2015) with manual corrections was performed to separate crystals of alkali feldspar from the glassy  
370 matrix by using the *AMIRA*<sup>®</sup> software v.4.1.2 (Mercury Computer Systems). Semi-automatic  
371 volume segmentation with manual corrections can also be performed with the *Avizo*<sup>®</sup> 3D software  
372 (FEI Visualization Sciences Group). This segmentation consists of manually drawing the edges of  
373 the crystals on the 2D slices. This is repeated every 5-10 slices (this interval depends on the size of



374 the crystal and the complexity of their shape) from the beginning to the end of the crystal, while in  
375 the intermediate slices the crystal shape will be reconstructed automatically by the software (via an  
376 interpolation procedure). The advantage of this technique is that the operator can obtain the real  
377 morphology of the object of interest by visual inspection (Fig. 10b). This technique can be time  
378 consuming for a large numbers of crystals, but it is a valuable approach in specific cases related to  
379 the study of crystal nucleation, twinning and crystal lattice orientation (Arzilli et al. 2015).

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## IMPLICATIONS

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### **Success of the procedure**

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In this work, we used for the first time a single-distance phase-retrieval method to successfully resolve feldspar crystals in a glassy rock matrix. More specifically, this method allowed us to display the habits and spatial distribution of plagioclase crystals in a natural pumice of Stromboli (Fig. 10a) and to display shapes and orientation of alkali feldspar in a synthetic trachytic glass (Fig. 10b). Both these results would have been impossible to obtain if not for the application of our newly developed protocol.

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The segmentation approach adopted for the Stromboli pumice is an invaluable tool for petrologists and volcanologists to quantify, for instance, crystal volume fractions and crystal number densities. On the other hand, the combination of phase-retrieval procedures and semi-automatic volume segmentation used for the synthetic sample represents a powerful tool for mineralogists and petrologists to investigate crystallographic orientations and crystal nucleation processes.

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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; Gualda and Rivers 2006; Gualda et al. 2010; Baker et al. 2012a; Tsuchiyama et al. 2013). The X-ray attenuation coefficient for magnetite is larger than that of

399 quartz, feldspar or glass, making dense minerals easily distinguishable from silicic glasses and  
400 felsic minerals (Gualda and Rivers 2006; Gualda et al. 2010). Whereas the X-ray attenuation  
401 coefficients for quartz and feldspar are very close to that of several silicate glasses, and  
402 segmentation is very challenging (e.g., Gualda and Rivers 2006; Pamukcu et al. 2010; Voltolini et  
403 al. 2011; Arzilli et al. 2015). Therefore, the main contribution of this study to petrology is to  
404 propose a novel application of the phase-retrieval procedure in 3D image processing in order to  
405 resolve feldspar crystals in multiphase crystallized rocks. This procedure is significantly important  
406 for the volcanological and petrological community because it opens new avenues in the 3D study of  
407 crystallization kinetics of silicate melts, which, together with degassing, is the most important  
408 process driving magma and eruption dynamics.

409 Despite the observation that the phase-retrieval method employed here has limitations, such as a  
410 slight blurring of images, which in our work was kept to a minimum to avoid significant changes in  
411 crystal shape, it represents a fundamental step in image processing in order to increase the contrast  
412 among minerals displaying only slightly different X-ray attenuations. This means that small  
413 differences in density and chemical composition can be resolved through phase-contrast imaging, as  
414 already shown in paleontological specimens, materials science and biomedical applications (e.g.,  
415 Tafforeau et al. 2006; Smith et al. 2010; Langer et al. 2010; Mayo et al. 2012; Mohammadi et al.  
416 2014). Another limitation is the need to use a suite of software tools to execute the protocol; we  
417 expect that in the near future a single software package will contain all of the algorithms necessary,  
418 which will accelerate the data processing. The techniques shown here can be applied to a wide  
419 range of natural rocks and synthetic samples characterized by the presence of crystals with sizes  
420 between a few  $\mu\text{m}$  to a few mm and phases with similar X-ray attenuation coefficients.

421 The results of this study highlight that careful application of a phase-retrieval algorithm to  
422 synchrotron X-ray microtomographic data sets of volcanic rocks can provide fundamental  
423 information for 3D quantitative analysis of magmatic crystallization processes. We envisage that

424 future work will expand the range of application of this procedure to different geomaterials. It is  
425 worth noting that our imaging protocol can be further refined (for instance using a phase-retrieval  
426 approach for multi-material objects as proposed by Beltran et al. (2010)) and also applied to older  
427 data sets in order to extract data that could not be extracted before.

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### 442 **References**

443 Arzilli, F., and Carroll, M.R. (2013) Crystallization kinetics of alkali feldspars in cooling and  
444 decompression-induced crystallization experiments in trachytic melt. *Contribution to Mineralogy  
445 and Petrology*, 166, 1011-1027.

446 Arzilli, F., Mancini, L., Voltolini, M., Cicconi, M.R., Mohammadi, S., Giuli, G., Mainprice, D.,  
447 Paris, E., Barou, F., and Carroll M.R. (2015) Near-liquidus growth of feldspar spherulites in

448 trachytic melts: 3D morphologies and implications in crystallization mechanisms. *Lithos*, 216–217,  
449 93–105.

450 Baker, D.R., Mancini, L., Polacci, M., Higgins, M.D., Gualda, G.A.R., Hill, R.J., and Rivers  
451 M.L. (2012a) An introduction to the application of X-ray microtomography to the three-  
452 dimensional study of igneous rocks. *Lithos*, 148, 262-276.

453 Baker, D.R., Brun, F., O'Shaughnessy, C., Mancini, L., Fife, J.L., and Rivers, M. (2012b) A  
454 four-dimensional X-ray tomographic microscopy study of bubble growth in basaltic foam. *Nature*  
455 *Communications*, 3, 1135.

456 Barnes, S.J., Osborne, G., Cook, D., Barnes, L., Maier, W.D., and Godel B. (2011) The Santa  
457 Rita nickel sulfide deposit in the Fazenda Mirabela Intrusion, Bahia, Brazil: geology, sulfide  
458 geochemistry and genesis. *Economic Geology*, 106, 1083–1110.

459 Baruchel, J., Cloetens, P., Härtwig, J., Ludwig, W., Mancini, L., Pernot, P., and Schlenker M.  
460 (2000) Phase imaging using highly coherent X-rays: Radiography, tomography, diffraction  
461 topography. *Journal of Synchrotron Radiation*, 7, 196–201.

462 Beltran, M.A., Paganin, D.M., Uesugi, K., and Kitchen M.J. (2010) 2D and 3D X-ray phase  
463 retrieval of multi-material objects using a single defocus distance. *Optics Express*, 18, 6423-6436.

464 Benedix, G.K., Ketcham, R.A., Wilson, L., McCoy, T.J., Bogard, D.D., Garrison, D.H., Herzog,  
465 G.F., Xue, S., Klein, J., and Middleton R. (2008) The formation and chronology of the PAT 91501  
466 impact-melt L chondrite with vesicle–metal-sulfide assemblages. *Geochimica et Cosmochimica*  
467 *Acta*, 72, 2417–2428.

468 Bernardini, F., Tuniz, C., Coppa, A., Mancini, L., Dreossi, D., Eichert, D., Turco, G., Biasotto,  
469 M., Terrasi, F., De Cesare, N., Hua, Q., and Levchenko V. (2012) Beeswax as Dental Filling on a  
470 Neolithic Human Tooth. *PLoS ONE*, 7 - 9, e44904.

471 Bertagnini, A., Coltelli, M., Landi, P., Pompilio, M., and Rosi M. (1999) Violent explosions  
472 yield new insights into dynamics of Stromboli volcano. *Eos Transactions AGU*, 80, 633–636.

473 Brun, F., Mancini, L., Kasae, P., Favretto, S., Dreossi, D., and Tromba G. (2010) Pore3D: A  
474 software library for quantitative analysis of porous media. Nuclear Instruments and Methods in  
475 Physics Research A, 615, 326–332.

476 Brun, F., Pacilè, S., Accardo, A., Kourousias, G., Dreossi, D., Mancini, L., Tromba, G., and  
477 Pugliese, R. (2015) Enhanced and Flexible Software Tools for X-ray Computed Tomography at the  
478 Italian Synchrotron Radiation Facility Elettra. Fundamenta Informaticae, 141, 233-243.

479 Brown, M.A., Brown, M., Carlson, W.D., and Denison, D. (1999) Topology of syntectonic melt-  
480 flow networks in the deep crust: Inferences from three- dimensional images of leucosome geometry  
481 in migmatites. American Mineralogist, 84, 1793–1818.

482 Buffière, J-Y., Maire, E., Cloetens, P., Lormand, G., and Fougères, R. (1999) Characterization of  
483 internal damage in a MMCp using X-ray synchrotron phase contrast microtomography. Acta  
484 Materials, 47, 1613-1625.

485 Burvall, A., Lundstrom, U., Takman, P.A.C., Larsson, D.H., and Hertz H.M. (2011) Phase  
486 retrieval in X-ray phase-contrast imaging suitable for tomography. Optics Express, 19, 10359.

487 Carlson, W.D. (2002) Scales of disequilibrium and rates of equilibration during metamorphism.  
488 American Mineralogist, 87, 185–204.

489 Carlson, W.D. (2006) Three-dimensional imaging of earth and planetary materials. Earth and  
490 Planetary Science Letters, 249, 133–147.

491 Carlson, W.D., and Denison, C. (1992) Mechanisms of porphyroblast crystallization: results  
492 from high-resolution computed X-ray tomography. Science, 257, 1236-1239.

493 Carlson, W.D., Denison, C., and Ketcham, R.A. (1995) Controls on the nucleation and growth of  
494 porphyroblasts: kinetics from natural textures and numerical models. Geological Journal, 30, 207-  
495 225.

- 496 Chen, R.C., Dreossi, D., Mancini, L., Menk, R., Rigon, L., Xiao, T.Q., and Longo, R. (2012)  
497 PITRE: software for phase-sensitive X-ray image processing and tomography reconstruction.  
498 *Journal of synchrotron radiation*, 19, 836-845.
- 499 Cilona, A., Faulkner, D.R., Tondi, E., Agosta, F., Mancini, L., Rustichelli, A., Baud, P., and  
500 Vinciguerra S. (2014) The effects of rock heterogeneity on compaction localization in porous  
501 carbonates. *Journal of Structural Geology*, 67, 75-93.
- 502 Cloetens, P., Barrett, R., Baruchel, J., Guigay, J.P., and Schlenker, M. (1996) Phase objects in  
503 synchrotron radiation hard X-ray imaging. *Journal of Physics D*, 29, 133-146.
- 504 Cloetens, P., Ludwig, W., Baruchel, J., Van Dyck, D., Van Landuyt, J., Guigay, J.P., and  
505 Schlenker, M. (1999) Holotomography: Quantitative phase tomography with micrometer resolution  
506 using hard synchrotron radiation X rays. *Applied Physics Letters*, 75, 2912-2914.
- 507 Cloetens, P., Pateyron-Salome, M., Buffiere, J.Y., Peix, G., Baruchel, J., Peyrin, F., and  
508 Schlenker, M. (1997) Observation of microstructure and damage in materials by phase sensitive  
509 radiography and tomography. *Journal of Applied Physics*, 81, 5878–5886.
- 510 Cnudde, V., and Boone, M.N. (2013) High-resolution X-ray computed tomography in  
511 geosciences: A review of the current technology and applications. *Earth-Science Reviews*, 123, 1-  
512 17.
- 513 Degruyter, W., Bachmann, O., and Burgisser A. (2010a) Controls on magma permeability in the  
514 volcanic conduit during the climactic phase of the Kos Plateau Tuff eruption (Aegean Arc). *Bulletin*  
515 *of Volcanology*, 72, 63–74.
- 516 Degruyter, W., Burgisser, A., Bachmann, O., and Malaspinas, O. (2010b) Synchrotron X-ray  
517 microtomography and lattice Boltzmann simulations of gas flow through volcanic pumices.  
518 *Geosphere*, 6, 470–481.

- 519 Denison, C., and Carlson, W.D. (1997) Three-dimensional quantitative textural analysis of  
520 metamorphic rocks using high-resolution computed X-ray tomography: Part II. Application to  
521 natural samples. *Journal Metamorphic Geology*, 15, 45–57.
- 522 Dowd, B.A., Campbell, G.H., Marr, R.B., Nagarkar, V., Tipnis, S., Axe, L., and Siddons, D.P.  
523 (1999) Developments in synchrotron x-ray computed microtomography at the National Synchrotron  
524 Light Source. *Proceedings of SPIE 3772, Developments in X-Ray Tomography II*, 224.
- 525 Ebel, D.S., Weisberg, M.K., Hertz, J., and Campbell, A.J. (2008) Shape, metal abundance,  
526 chemistry, and origin of chondrules in the Renazzo (CR) chondrite. *Meteoritics and Planetary  
527 Science*, 43, 1725–1740.
- 528 Faessel, M., and Jeulin, D. (2010) Segmentation of 3D microtomographic images of granular  
529 materials with the stochastic watershed. *Journal of Microscopy*, 239, 17-31.
- 530 Fitzgerald, R. (2000) Phase sensitive X-ray imaging. *Physics Today*, 53, 23.
- 531 Friedrich, J.M., Wignarajah, D.P., Chaudhary, S., Rivers, M.L., Nehru, C.E., and Ebel D.S.  
532 (2008) Three-dimensional petrography of metal phases in equilibrated L chondrites - Effects of  
533 shock loading and dynamic compaction. *Earth and Planetary Science Letters*, 275, 172–180.
- 534 Fusseis, F., Xiao, X., Schrank, C., and De Carlo, F. (2014) A brief guide to synchrotron  
535 radiation-based microtomography in (structural) geology and rock mechanics. *Journal of Structural  
536 Geology*, 65, 1-16.
- 537 Giachetti, T., Burgisser, A., Arbaret, L., Druitt, T. H., and Kelfoun, K. (2011) Quantitative  
538 textural analysis of Vulcanian pyroclasts (Montserrat) using multi-scale X-ray computed  
539 microtomography: comparison with results from 2D image analysis. *Bulletin of volcanology*, 73,  
540 1295-1309.
- 541 Godel, B., Barnes, S.J., Barnes, S.-J., and Maier, W.D. (2010) Platinum ore in 3D: insights from  
542 high-resolution X-ray computed tomography. *Geology*, 38, 1127–1130.

543 Godel, B., Gonzalez-Alvarez, I., Barnes, S.J., Barnes, S.-J., Parker, P., and Day, J. (2012)  
544 Sulfides and sulfarsenides from the Rosie Nickel Prospect, Duketon Greenstone Belt, Western  
545 Australia. *Economic Geology*, 107, 275–294.

546 Godel, B., Barnes, S.J., Gürer, D., Austin, P., and Fiorentini, M.L. (2013) Chromite in  
547 komatiites: 3D morphologies with implications for crystallization mechanisms. *Contribution to*  
548 *Mineralogy and Petrology*, 165, 173–189.

549 Godel, B., Rudashevsky, N.S., Nielsen, T.F.D., Barnes, S.J., and Rudashevsky, V.N. (2014) New  
550 constraints on the origin of the Skaergaard intrusion Cu–Pd–Au mineralization: Insights from high-  
551 resolution X-ray computed tomography. *Lithos*, 190–191, 27–36.

552 Gualda, G.A.R. (2006), Crystal size distributions derived from 3D datasets: sample size versus  
553 uncertainties. *Journal of Petrology*, 47, 1245–1254.

554 Gualda, G.A.R., and Rivers, M. (2006) Quantitative 3D petrography using X-ray tomography:  
555 application to Bishop Tuff pumice clasts. *Journal of Volcanology and Geothermal Research*, 154,  
556 48–62.

557 Gualda, G., Pamukcu, A., Claiborne, L., and Rivers, M. (2010) Quantitative 3D petrography  
558 using X-ray tomography 3: documenting accessory phases with differential absorption tomography.  
559 *Geosphere*, 6, 782–792.

560 Guigay, J.P., Langer, M., Boistel, R., and Cloetens, P. (2007) Mixed transfer function and  
561 transport of intensity approach for phase retrieval in the Fresnel region. *Optics Letters*, 32, 1617–  
562 1619.

563 Gureyev, T.E., and Nugent, K.A. (1996) Phase retrieval with the transport-of-intensity equation.  
564 II. Orthogonal series solution for nonuniform illumination. *Journal of the Optical Society of*  
565 *America A*, 13, 1670-1682.

566 Gureyev, T.E., and Nugent, K.A. (1997) Rapid quantitative phase imaging using the transport of  
567 intensity equation. *Optics Communication*, 133, 339–346.



568 Gureyev, T.E., Nesterets, Y., Paganin, D., Pogany, A., and Wilkins, S. (2006) Linear algorithms  
569 for phase retrieval in the Fresnel region. 2. partially coherent illumination. Optics Communications,  
570 259, 569–580.

571 Gureyev, T.E., Nesterets, Y.I., Stevenson, A.W., Miller, P.R., Pogany, A., and Wilkins, S.W.  
572 (2008) Some simple rules for contrast, signal-to-noise and resolution in in-line x-ray phase-contrast  
573 imaging. Optics Express, 16, 3223-3241.

574 Henke, B.L., Gullikson, E.M., and Davis, J.C. (1993) X-ray interactions: photoabsorption,  
575 scattering, transmission, and reflection at E=50-30000 eV, Z=1-92. Atomic Data and Nuclear Data  
576 Tables, 54, 181-342.

577 Herman, G.T. (1980) Image reconstruction from projections: the fundamentals of computerized  
578 tomography. Academic Press, New York.

579 Hersum, T.G., and Marsh, B.D. (2007) Igneous textures: on the kinetics behind the words.  
580 Elements, 3, 247–252.

581 Higgins, M.D. (2000) Measurement of crystal size distributions. American Mineralogist, 85,  
582 1105–1116.

583 Higgins, M.D. (2002) A crystal size-distribution study of the Kiglapait layered mafic intrusion,  
584 Labrador, Canada: evidence for textural coarsening. Contribution to Mineralogy and Petrology,  
585 144, 314–330.

586 Higgins, M.D. (2006) Quantitative textural measurements in igneous and metamorphic  
587 petrology. Cambridge University Press, Cambridge, UK.

588 Jerram, D.A., Mock, A., Davis, G.R., Field, M., and Brown, R.J. (2009) 3D crystal size  
589 distributions: a case study on quantifying olivine populations in kimberlites. Lithos, 112, 223–235.

590 Ketcham, R.A. (2005) Three dimensional textural measurements using high resolution  
591 X ray computed tomography. Journal of Structural Geology, 27, 1217–1228.

592 Ketcham, R.A., and Carlson, W.D. (2001). Acquisition, optimization and interpretation of X-ray  
593 computed tomographic imagery: applications to the geosciences. *Computers and Geosciences*, 27,  
594 381–400.

595 Ketcham, R.A., Meth, C., Hirsch, D.M., and Carlson, W.D. (2005) Improved methods for  
596 quantitative analysis of three-dimensional porphyroblastic textures. *Geosphere*, 1, 42–59.

597 Langer, M., Boistel, R., Pagot, E., Cloetens, P., and Peyrin, F. (2010) X-ray in-line phase  
598 microtomography for biomedical applications. *Microscopy: Science, Technology, Applications and*  
599 *Education*.

600 Lev, A., Zucker, S.W., and Rosenfeld, A. (1977) Iterative Enhancement of Noisy Images.  
601 *Systems, Man and Cybernetics, IEEE Transactions*, 7, 435-442.

602 Lin, Q., Neethling, S. J., Dobson, K. J., Courtois, L., and Lee, P. D. (2015) Quantifying and  
603 minimising systematic and random errors in X-ray micro-tomography based volume measurements.  
604 *Computers and Geosciences*, 77, 1-7.

605 Madonna, C., Quintal, B., Frehner, M., Almqvist, B.S.G., Tisato, N., Pistone, M., Marone, F.,  
606 and Saenger, E.H. (2013) Synchrotron-based X-ray tomographic microscopy for rock physics  
607 investigations. *Geophysics*, 78, D53-D64.

608 Maire E. and Withers P.J. (2014) Quantitative X-ray tomography. *International Materials*  
609 *Reviews*; 59, 1-43.

610 Mancini, L., Reiner, E., Cloetens, P., Gastaldi, J., Härtwig, J., Schlenker, M., and Baruchel J.  
611 (1998) Investigation of defects in AlPdMn icosahedral quasicrystals by combined synchrotron X-  
612 ray topography and phase radiography. *Philosophical Magazine A*, 78, 1175–1194.

613 Marinoni, N., Voltolini, M., Mancini, L., Vignola, P., Pagani, A., and Pavese, A., (2009) An  
614 investigation of mortars affected by alkali-silica reaction by X-ray synchrotron microtomography: a  
615 preliminary study. *Journal of Materials Science*, 44 – 21.

- 616 Marsh, B.D. (1988) Crystal size distribution (CSD) in rocks and the kinetics and dynamics of  
617 crystallization. 1. Theory. *Contribution to Mineralogy and Petrology*, 99, 277–291.
- 618 Marsh, B.D. (1998) On the interpretation of crystal size distributions in magmatic systems,  
619 *Journal of Petrology*, 39, 553–599.
- 620 Marsh, B.D. (2007) Crystallization of silicate magmas deciphered using crystal size  
621 distributions. *Journal of the American Ceramic Society*, 90, 746–757.
- 622 Mayo, S.C., Stevenson, A.W., and Wilkins, S.W. (2012) In-line phase-contrast X-ray imaging  
623 and tomography for materials science. *Materials*, 5, 937-965.
- 624 Mohammadi, S., Larsson, E., Alves, F., Dal Monego, S., Biffi, S., Garrovo, C., Lorenzon, A.,  
625 Tromba, G., and Dullin, C. (2014) Quantitative evaluation of a single-distance phase-retrieval  
626 method applied on in-line phase-contrast images of a mouse lung. *Journal of synchrotron radiation*,  
627 21, 784-789.
- 628 Münch, B., Trtik, P., Marone, F., and Stampanoni, M. (2009) Stripe and ring artifact removal  
629 with combined wavelet-Fourier filtering. *Optics Express* 17, 8567–8591.
- 630 Paganin, D., and Nugent, K.A. (1998) Noninterferometric phase imaging with partially coherent  
631 light. *Physical Review Letters*, 80, 2586–2589.
- 632 Paganin, D., Mayo, S.C., Gureyev, T.E., Miller, P.R., and Wilkins, S.W. (2002) Simultaneous  
633 phase and amplitude extraction from a single defocused image of a homogeneous object. *Journal of*  
634 *Microscopy*, 206, 33-40.
- 635 Pal, N.R, and Pal S.K. (1993) A review on image segmentation techniques. *Pattern Recognition*,  
636 26: 1277–1294.
- 637 Pamukcu, A.S., and Gualda G.A.R. (2010) Quantitative 3D petrography using X-ray tomography  
638 2: combining information at various resolutions. *Geosphere*, 6, 775–781.
- 639 Pamukcu, A.S., Gualda, G.A.R., and Anderson, A.T. (2012) Crystallization stages of the Bishop  
640 Tuff magma body recorded in crystal textures in pumice clasts. *Journal of Petrology*, 63, 589–609.

- 641 Pfeiffer, F., Weitkamp, T., Bunk, O., and David, C. (2006) Phase retrieval and differential phase-  
642 contrast imaging with low-brilliance x-ray sources. *Nature Physics*, 2, 258–61.
- 643 Polacci, M., Baker, D.R., Mancini, L., Tromba, G., and Zanini, F. (2006) Three-dimensional  
644 investigation of volcanic textures by X-ray microtomography and implications for conduit  
645 processes. *Geophysical Research Letters*, 33, L13312.
- 646 Polacci, M., Baker, D.R., Mancini, L., Favretto, S., and Hill, R.J. (2009) Vesiculation in magmas  
647 from Stromboli and implications for normal Strombolian activity and paroxysmal explosions in  
648 basaltic systems. *Journal of Geophysical Research*, 114, B01206.
- 649 Polacci, M., Baker, D.R., LaRue, A., Mancini, L., and Allard, P. (2012) Degassing behaviour of  
650 vesiculated basaltic magmas: an example from Ambrym volcano, Vanuatu Arc. *Journal of*  
651 *Volcanology and Geothermal Research*, 233-234, 55-64.
- 652 Ralph, J., and Chau, I. (2014) Mindat.org.
- 653 Rivers, M. (1998). Tutorial introduction to X-ray computed microtomography data processing.  
654 University of Chicago.
- 655 Sayab, M., Suuronen, J.-P., Hölttä, P., Aerden, D., Lahtinen, R., and Petteri Kallonen, A. (2014)  
656 High-resolution X-ray computed microtomography: A holistic approach to metamorphic fabric  
657 analyses. *Geology*, 43, 55–58.
- 658 Schneider, C.A., Rasband, W.S., and Eliceiri, K.W. (2012) NIH Image to ImageJ: 25 years of  
659 image analysis. *Nature Methods*, 9, 671–675.
- 660 Sijbers, J., and Postnov, A. (2004) Reduction of ring artefacts in high resolution micro-CT  
661 reconstructions. *Physics in Medicine and Biology*, 49, N247–N253.
- 662 Smith, T.M., Tafforeau, P., Reid, D. J., Pouech, J., Lazzari, V., Zermeno, J.P., Guatelli-Steinberg  
663 D., Olejniczakb, A.J., Hoffmang, A., Radovčić, J., Makaremii, M., Toussaintj, M., Stringer C., and  
664 Hublin, J.J. (2010). Dental evidence for ontogenetic differences between modern humans and  
665 Neanderthals. *Proceedings of the National Academy of Sciences*, 107, 20923-20928.

666 Snigirev, A., Snigireva, I., Kohn, V., Kuznetsov, S., and Schelokov, I. (1995) On the possibilities  
667 of X-ray phase contrast imaging by coherent high energy synchrotron radiation. Review of  
668 Scientific Instruments, 66, 5486–5492.

669 Tafforeau, P., Boistel, R., Boller, E., Bravin, A., Brunet, M., Chaimanee, Y., Cloetens, P., Feist,  
670 M., Hoszowska, J., Jaeger, J.-J., Kay, R. F., Lazzari, V., Marivaux, L., Nel, A., Nemoz, C.,  
671 Thibault, X., Vignaud, P., and Zabler, S. (2006) Applications of X-ray synchrotron  
672 microtomography for non-destructive 3D studies of paleontological specimens. Applied Physics A,  
673 83, 195-202.

674 Teague, M.R. (1983) Deterministic phase retrieval: a Green's function solution, Journal of the  
675 Optical Society of America, 73, 1434–1441.

676 Titarenko, V., Titarenko, S., Withers, P. J., De Carlo, F., and Xiao, X. (2010) Improved  
677 tomographic reconstructions using adaptive time-dependent intensity normalization. Journal of  
678 Synchrotron Radiation 17, 689–699.

679 Tromba, G., Longo, R., Abrami, A., Arfelli, F., Astolfo, A., Bregant, P., Brun, F., Casarin, K.,  
680 Chenda, V., Dreossi, D., Hola, M., Kaiser, J., Mancini, L., Menk, R.H., Quai, E., Quaia, E., Rigon,  
681 L., Rokvic, T., Sodini, N., Sanabor, D., Schultke, E., Tonutti, M., Vascotto, A., Zanconati, F., Cova,  
682 M., and Castelli, E. (2010) The SYRMEP Beamline of Elettra: Clinical Mammography and Bio-  
683 medical Applications. AIP Conference Proceedings, 1266, 18-23.

684 Tsuchiyama, A., Hanamoto, T., Nakashima, Y., and Nakano, T. (2000) Quantitative evaluation  
685 of attenuation contrast of minerals by using a medical X-ray CT scanner. Journal of Mineralogical  
686 and Petrological Sciences, 95, 125-137.

687 Tsuchiyama, A., Uesugi, K., Nakano, T., and Ikeda, S. (2005) Quantitative evaluation of  
688 attenuation contrast of X-ray computed tomography images using monochromatized beams.  
689 American Mineralogist, 90, 132–142.

690 Tsuchiyama, A., Uesugi, M., Matsushima, T., Michikami, T., Kadono, T., Nakamura, T., Uesugi,  
691 K., Nakano, T., Sandford, S.A., Noguchi, R., Matsumoto, T., Matsuno, J., Nagano, T., Imai, Y.,  
692 Takeuchi, A., Suzuki, Y., Ogami, T., Katagiri, J., Ebihara, M., Ireland, T.R., Kitajima, F., Nagao,  
693 K., Naraoka, H., Noguchi, T., Okazaki, R., Yurimoto, H., Zolensky, M.E., Mukai, T., Abe, M.,  
694 Yada, T., Fujimura, A., Yoshikawa, M., and Kawaguchi, J. (2011) Three-dimensional structure of  
695 Hayabusa samples: origin and evolution of Itokawa Regolith. *Science*, 333, 1125–1128.

696 Tsuchiyama, A., Nakano, T., Uesugi, K., Uesugi, M., Takeuchi, A., Suzuki, Y., Noguchi, R.,  
697 Matsumoto, T., Matsuno, J., Nagano, T., Imai, Y., Nakamura, T., Ogami, T., Noguchi, T., Abe, M.,  
698 Yada, T., and Fujimura, A. (2013) Analytical dual- energy microtomography: A new method for  
699 obtaining three-dimensional phase images and its application to Hayabusa samples. *Geochimica et*  
700 *Cosmochimica Acta*, 116, 5–16.

701 Uesugi, K., Tsuchiyama, A., Nakano, T., Suzuki, Y., Yagi, N., Umetani, K., and Kohmura, Y.  
702 (1999) Development of microtomography imaging system for rock and mineral samples.  
703 *Proceedings SPIE 3772, Developments in X-Ray Tomography II*, 214.

704 Uesugi, M., Uesugi, K., and Oka, M. (2010) Non-destructive observation of meteorite chips  
705 using quantitative analysis of optimized X-ray micro-computed tomography. *Earth and Planetary*  
706 *Science Letters*, 299, 359–367.

707 Uesugi, M., Uesugi, K., Takeuchi, A., Suzuki, Y., Hoshino, M., and Tsuchiyama, A. (2013)  
708 Three-dimensional observation of carbonaceous chondrites by Synchrotron radiation X-ray CT -  
709 quantitative analysis and developments for the future sample return missions. *Geochimica et*  
710 *Cosmochimica Acta.*, 116, 17–32.

711 Van Nieuwenhove, V., De Beenhouwer, J., De Carlo, F., Mancini, L., Marone, F., Sijbers, J.,  
712 (2015) Dynamic intensity normalization using eigen flat fields in X-ray imaging, *Optics Express*,  
713 23-21, 27975-27989.

714 Voltolini, M., Zandomeneghi, D., Mancini, L., and Polacci, M. (2011) Texture analysis of  
715 volcanic rock samples: quantitative study of crystals and vesicles shape preferred orientation from  
716 X-ray microtomography data. *Journal of Volcanology and Geothermal Research*, 202, 83-95.

717 Weitkamp, T., Haas, D., Wegrzynek, D., and Rack, A. (2011) ANKAphase: software for single-  
718 distance phase retrieval from inline X-ray phase-contrast radiographs. *Journal of Synchrotron*  
719 *Radiation*, 18, 617–629.

720 Zandomeneghi, D., Voltolini, M., Mancini, L., Brun, F., Dreossi, D., and Polacci, M. (2010)  
721 Quantitative analysis of X-ray microtomography images of geomaterials: Application to volcanic  
722 rock. *Geosphere*, 6, 793-804.

723 Zanolli, C., Bondioli, L., Coppa, A., Dean, C.M., Bayle, P., Candilio, F., Capuani, S., Dreossi,  
724 D., Fiore, I., Frayer, D.W., Libsekal, Y., Mancini, L., Rook, L., Medin Tekle, T., Tuniz, C., and  
725 Macchiarelli, R. (2014) The late Early Pleistocene human dental remains from Uadi Aalad and  
726 Mulhuli-Amo (Buia), Eritrean Danakil: Macromorphology and microstructure. *Journal of Human*  
727 *Evolution*, 74, 96–113.

728 Zucali, M., Voltolini, M., Ouladdiaf, B., Mancini, L., and Chateigner, D. (2014a) The 3D  
729 quantitative lattice and shape preferred orientation of a mylonitised metagranite from Monte Rosa  
730 (Western Alps): combining neutron diffraction texture analysis and synchrotron X-ray  
731 microtomography. *Journal of Structural Geology*, 63, 91–105.

732 Zucali, M., Barberini, V., Voltolini, M., Ouladdiaf, B., Chateigner, D., and Mancini, L. (2014b)  
733 Quantitative 3D microstructural analysis of naturally deformed amphibolite from the Southern Alps  
734 (Italy): Microstructures, LPO, and seismic anisotropy from a fossil extensional margin, in *Rock*  
735 *Deformation From Field, Experiments and Theory: A Volume in Honour of Ernie Rutter*.  
736 *Geological Society Special Publication*, 409, 201-222.

737

738 **Figure captions**

739 Figure 1. Comparison of 2D and 3D textural analysis approaches.

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

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

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

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

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

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

760 Figure 8. (a) Reconstructed axial slice of the Stromboli pumice after phase-retrieval processing.  
761 (b) Thresholded image after phase-retrieval illustrating segmentation of plagioclases from basaltic  
762 glass and vesicles. Note that noise is still present after segmentation. (c) Second segmentation  
763 consisting in the approximate isolation of the shape of plagioclase, and in deleting background



764 noise as much as possible. (d) Mask of plagioclase crystals. (e) After (d), the AND operator in *Fiji*  
765 was applied to combine both the first segmentation (b) and the mask (d) in order to segment  
766 plagioclase crystals from basaltic glass and vesicles.

767 Figure 9. Scheme of the image processing protocol that has been used to segment plagioclase  
768 from the background matrix in the Stromboli pumice.

769 Figure 10. Volume renderings of crystals. (a) Volume renderings of the segmented plagioclases  
770 in Stromboli pumice sample; the maximum length of the largest crystal is ~ 1 mm. (b) Volume  
771 rendering of the segmented alkali feldspars in the synthetic trachyte sample; the average length of  
772 each lamella within spherulites is 150  $\mu\text{m}$ .

Figure 1

## 2D vs 3D

### Advantages and special features

- Image mapping of large area of the sample at high resolution (pixel size  $<1 \mu\text{m}$ ).
- Phase abundance can be determined
- Lattice orientation of crystals is available
- Chemical compositions of major and trace elements can be determined

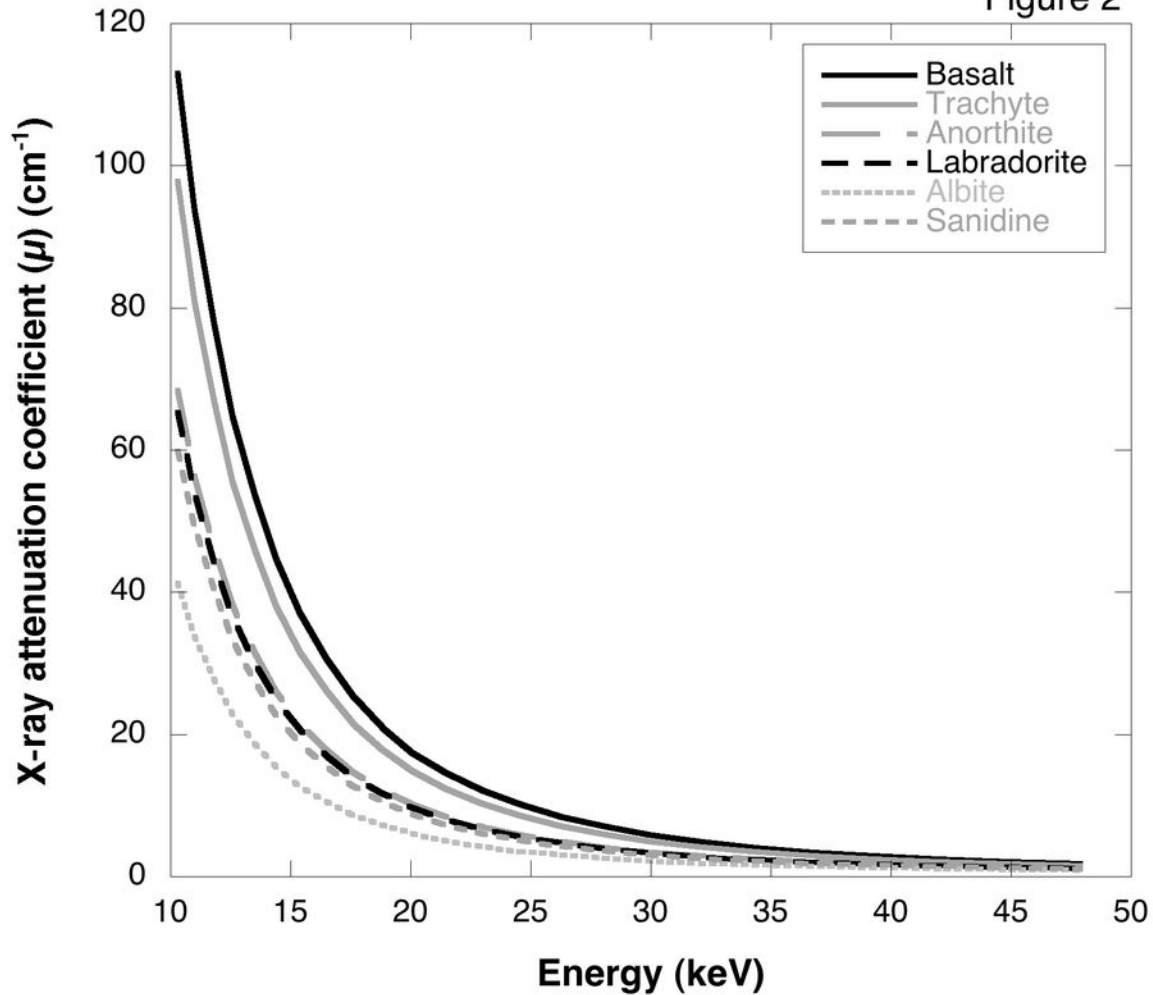
- Large volume of sample can be imaged
- Non-destructive 3D imaging and analysis
- Textural features of the sample can be visualized in 3D
- Volumetric analysis: phase abundance and size can be directly measured in 3D
- Preferred orientation and connectivity of objects can be measured in 3D
- Stereological conversion are not necessary
- Experimental apparatus are available to perform 4D experiments

### Limitations

- Destructive procedures are needed to prepare thin sections
- Stereological conversion is needed
- Population of crystals are described statistically

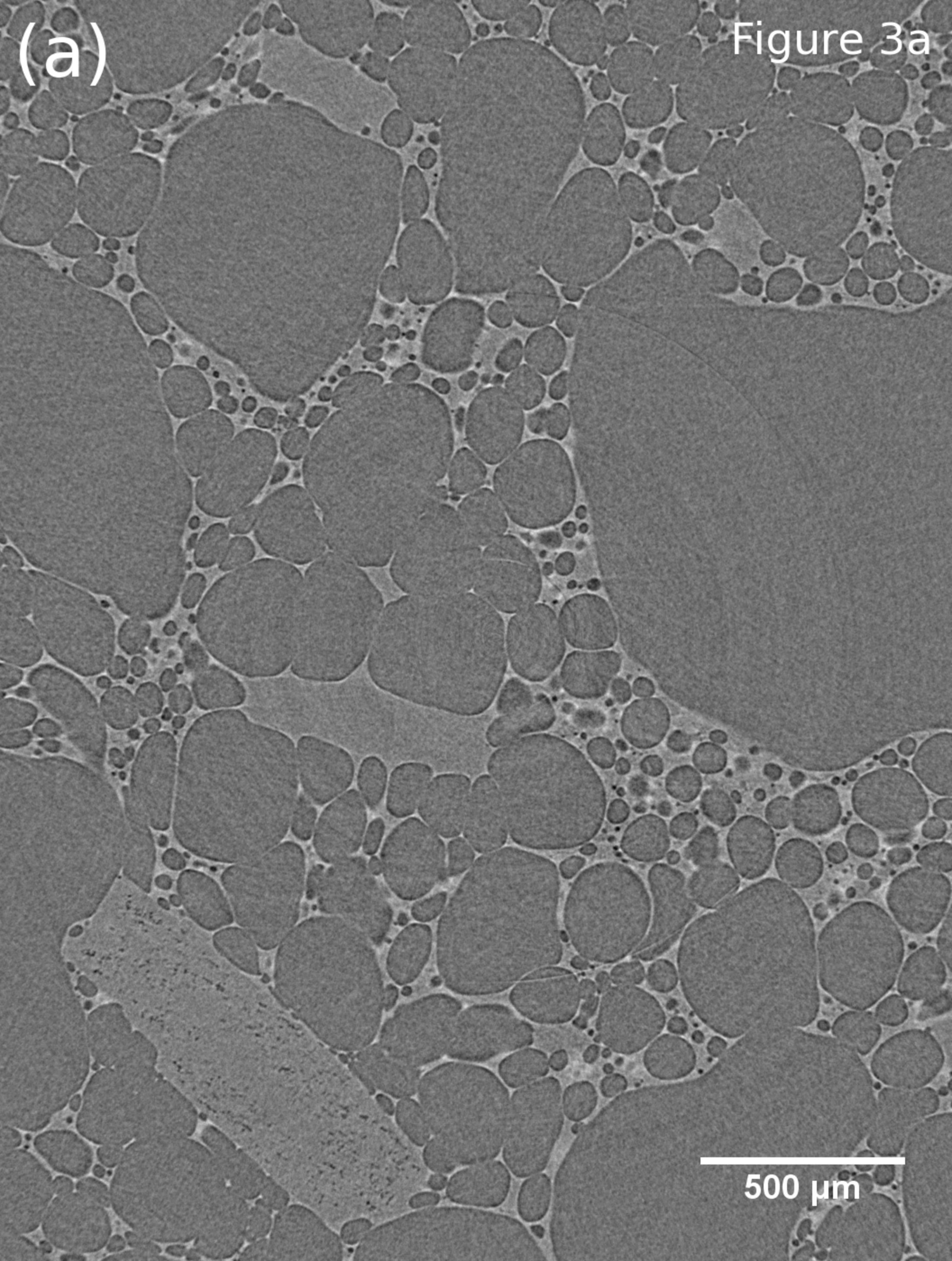
- Not always possible to identify and separate easily different minerals, due to similar density contrast and crystal sizes smaller than spatial resolution
- Image processing and analysis are time consuming

Figure 2



(a)

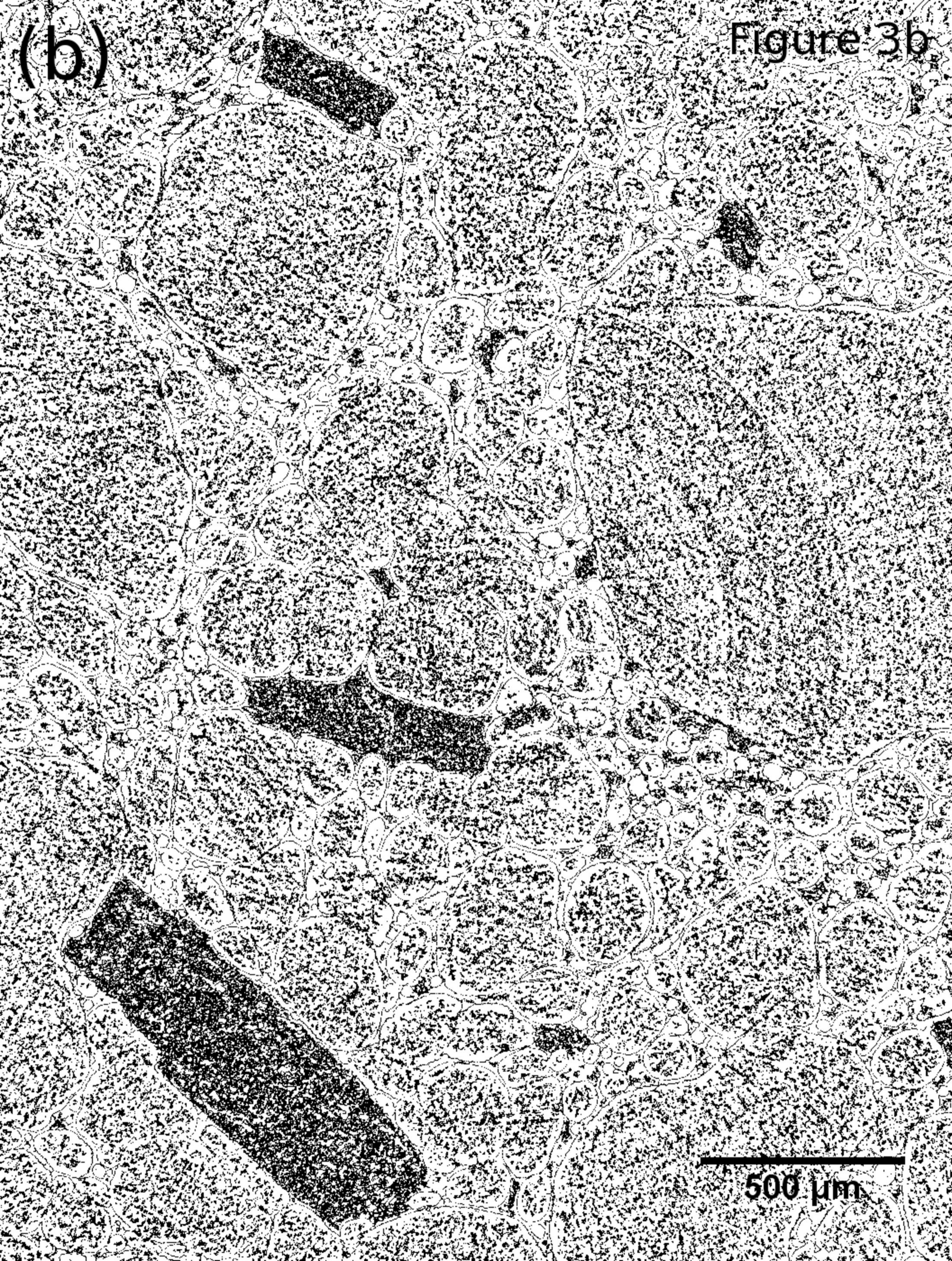
Figure 3a



500 μm

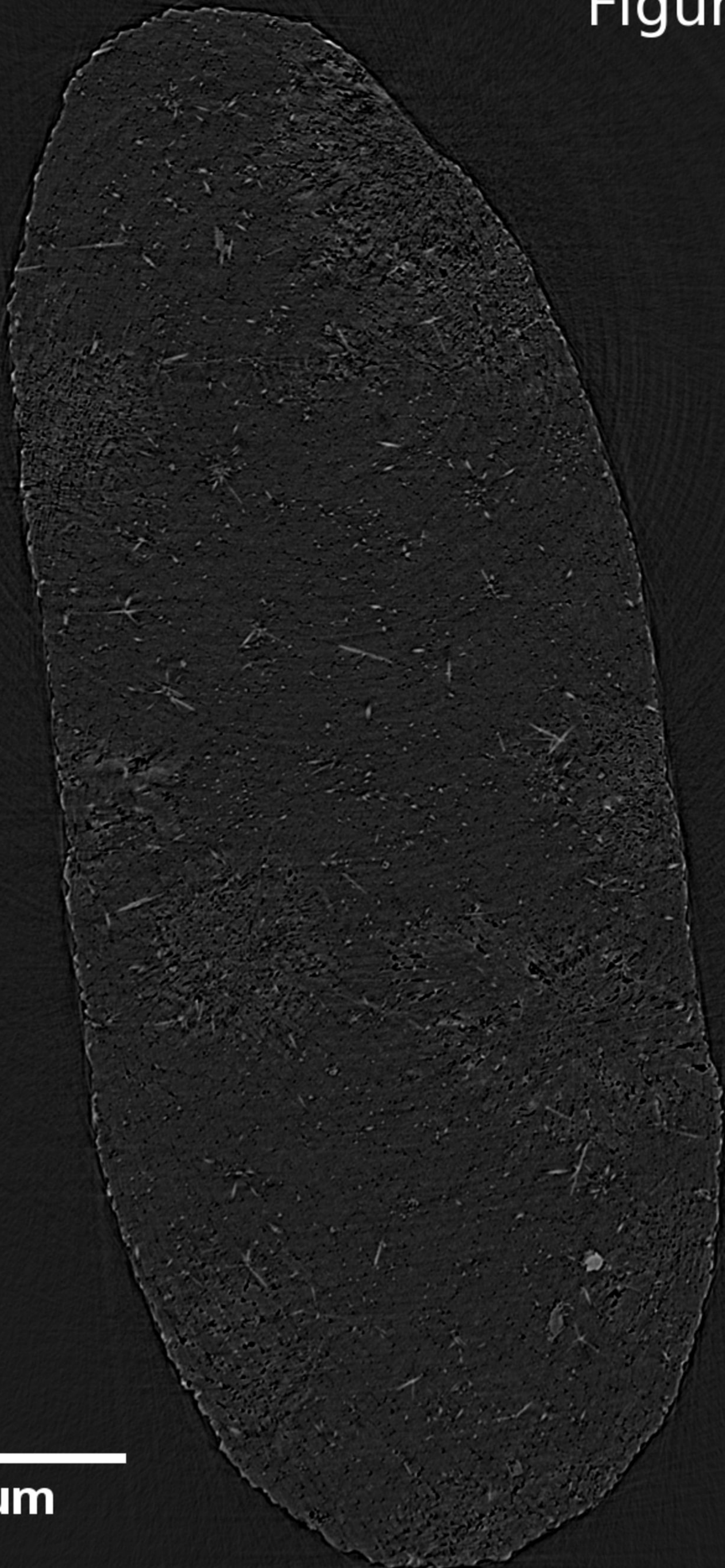
(b)

Figure 3b



500  $\mu\text{m}$

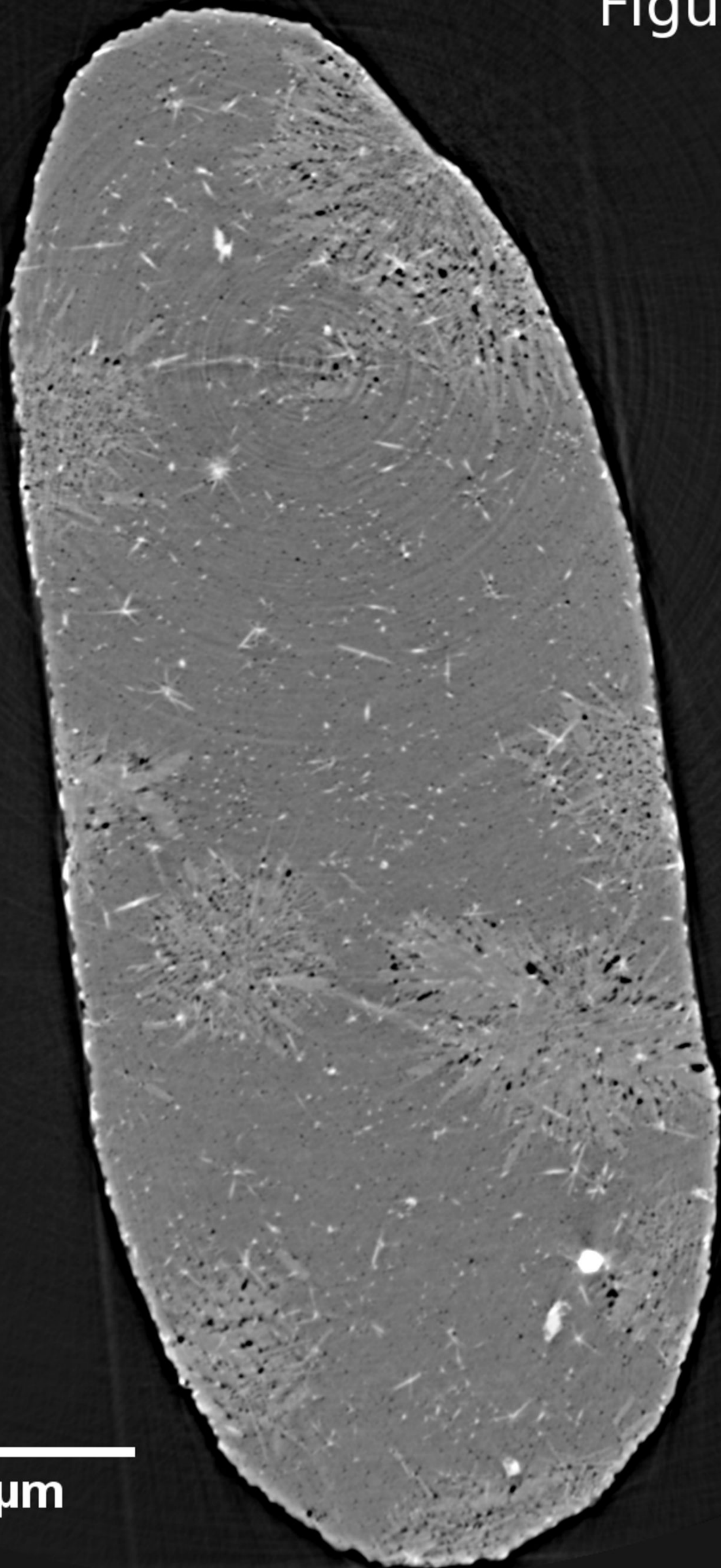
(a)



500  $\mu\text{m}$

(b)

Figure 4b



500  $\mu\text{m}$

# Pre-segmentation

## - Phase-retrieval processing -

### Calculation of Refractive Index

The calculation of the refractive index can be obtained using the CXRO X-ray database (Henke et al. 1993) ([http://henke.lbl.gov/optical\\_constants/getdb2.html](http://henke.lbl.gov/optical_constants/getdb2.html))

$\delta$  = refractive index decrement  
 $\beta$  = absorption index

To obtain  $\delta$  and  $\beta$  several parameters are needed:

- Chemical formula of the phase of interest
- Density ( $\text{g/cm}^3$ ) of the phase of interest
- Photon energy (keV) used during the X-ray  $\mu\text{CT}$  acquisition



### Single-distance phase-retrieval TIE algorithm

Available software

**X-TRACT software** (CSIRO group) #  
**ANKA phase software** (Weitkamp et al. 2011) \*  
**PITRE software** (Pfeiffer et al. 2006) \*  
**Tomopy software** (Gürsoy et al. 2014) \*  
**SYRMEP Tomo Project** (Brun et al. 2015) \*

**X-TRACT software**  
(used in this study)



For slices reconstruction several parameters are requested:

Photon energy (KeV)

Sample-detector distance

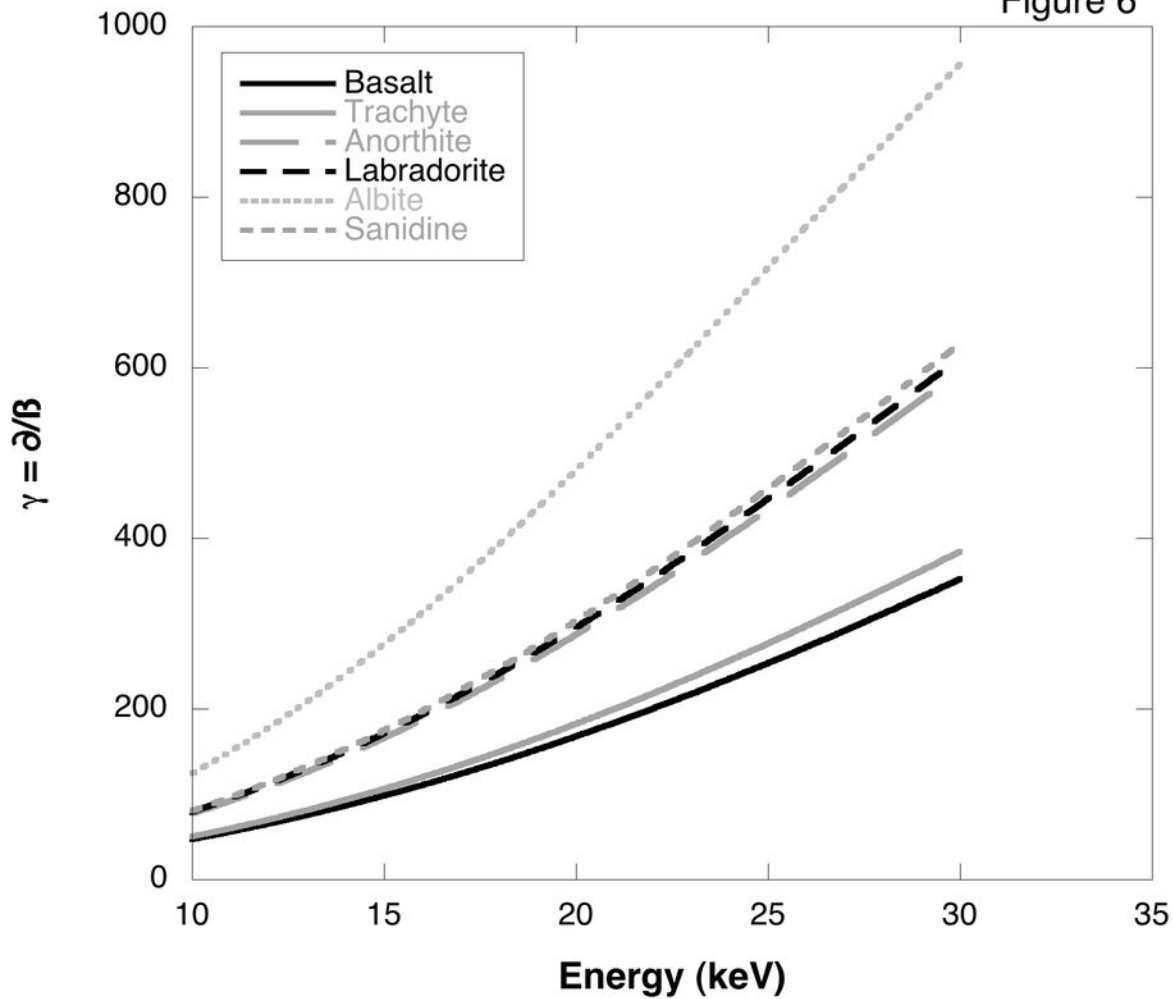
$\delta/\beta = \gamma$

The calculated value of  $\gamma$  (Fig. 6) may be tuned to obtain:

- Cancellation of phase-contrast artifacts
- Better contrast between phases avoiding blurring



Figure 6



(a)

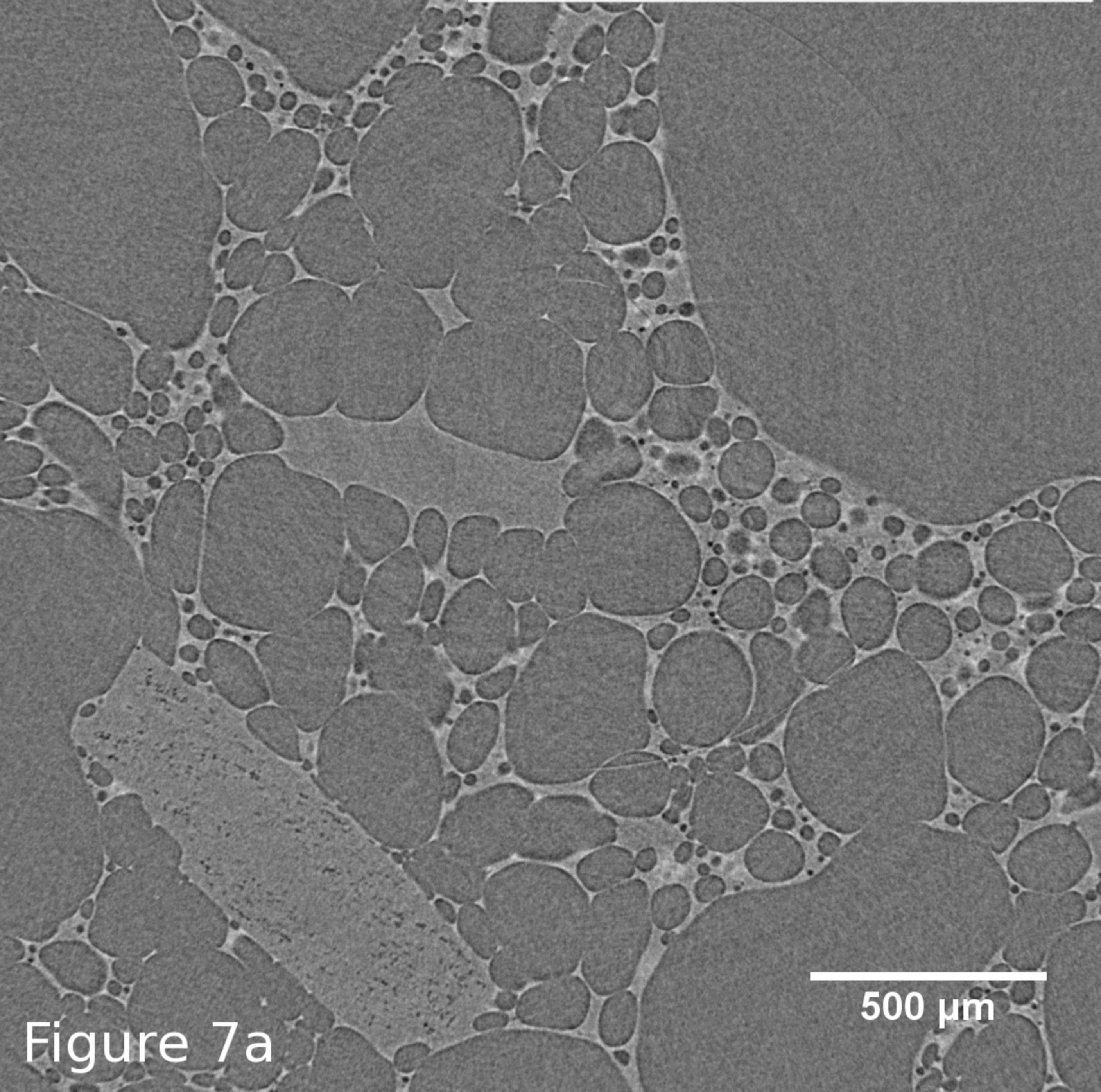
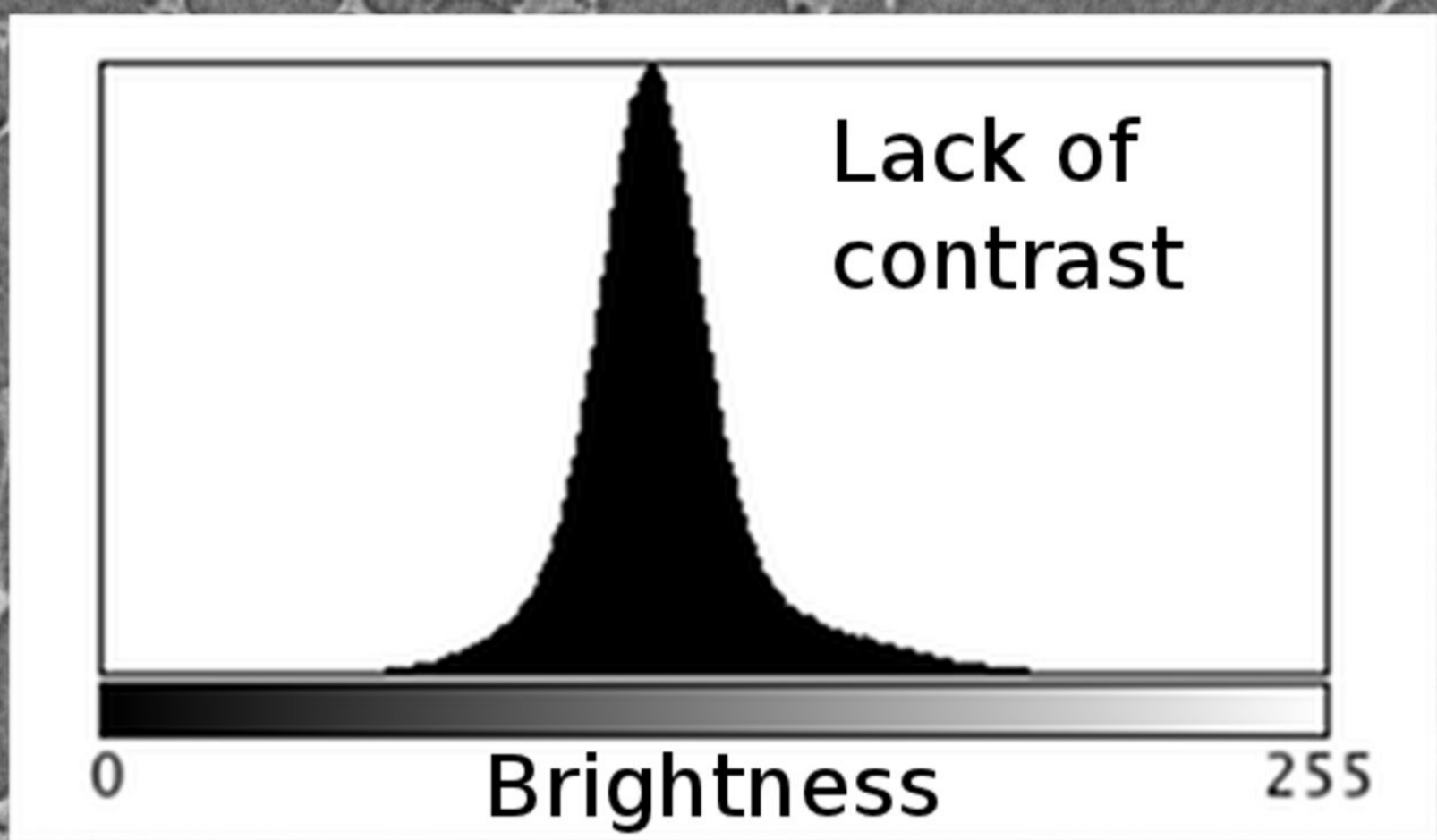


Figure 7a

(b)

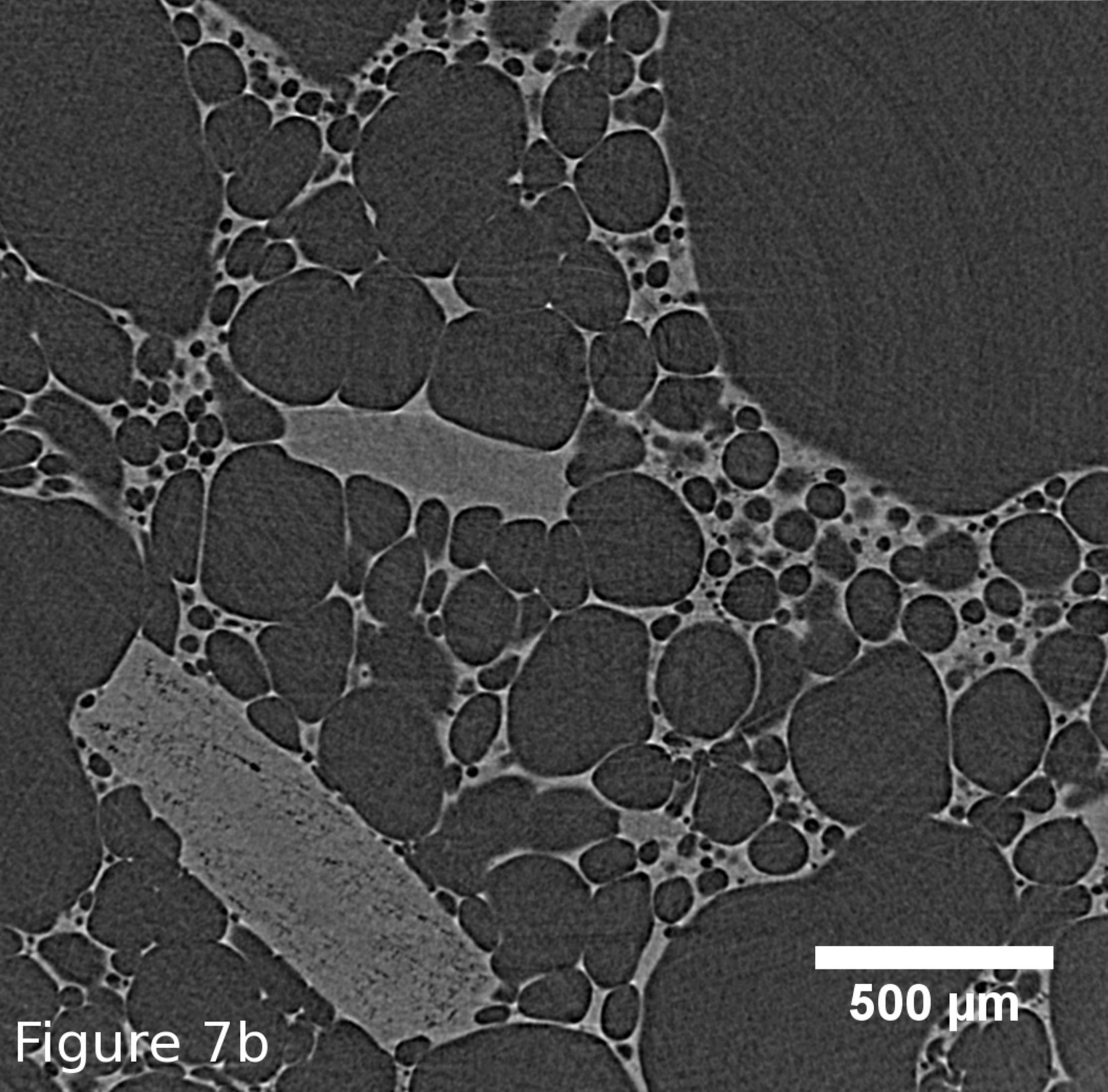
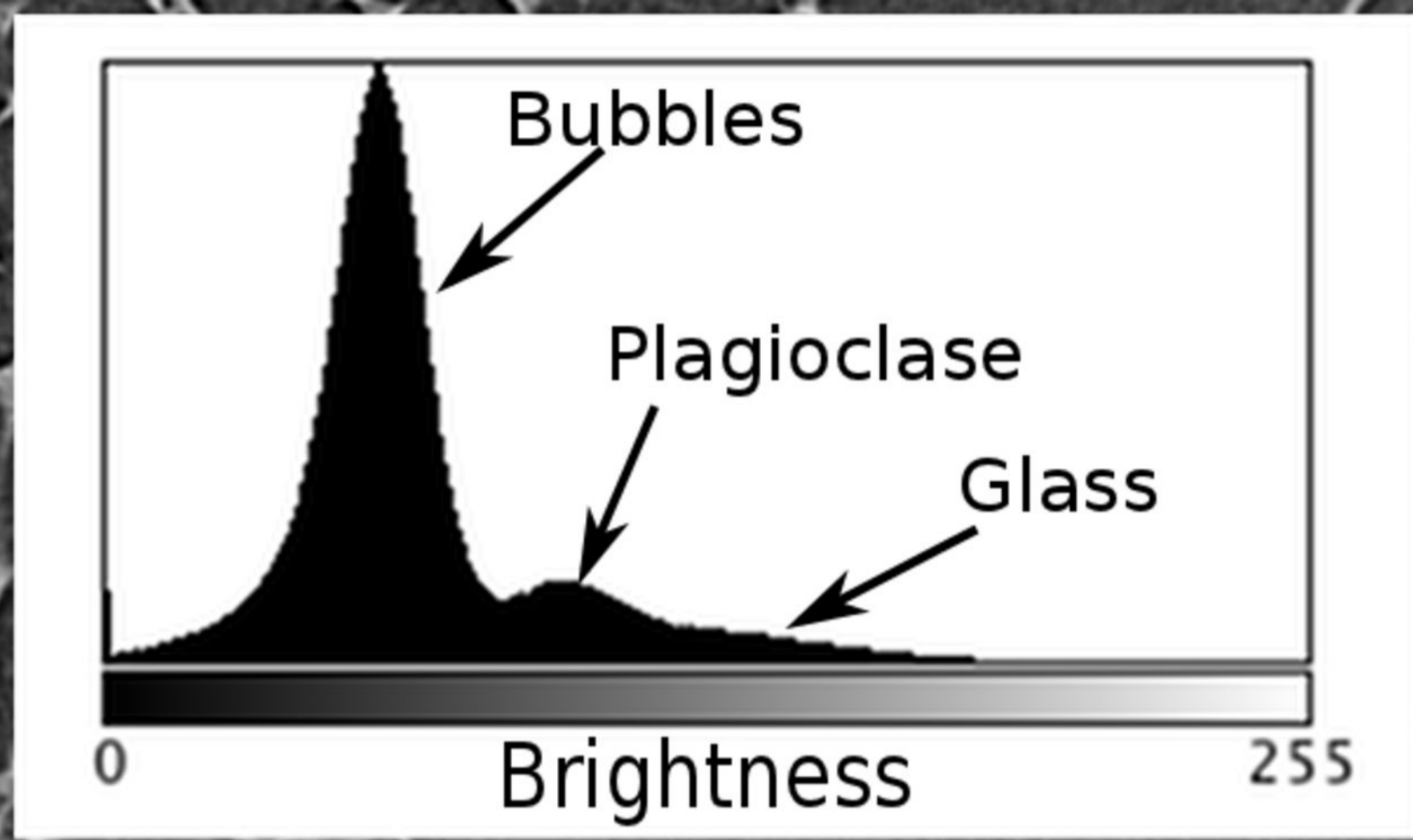
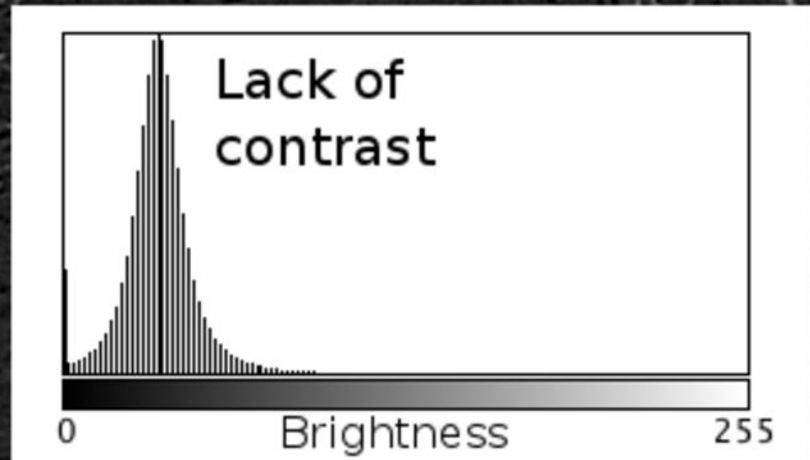


Figure 7b

(c)



100  $\mu\text{m}$

Figure 7c

(d)

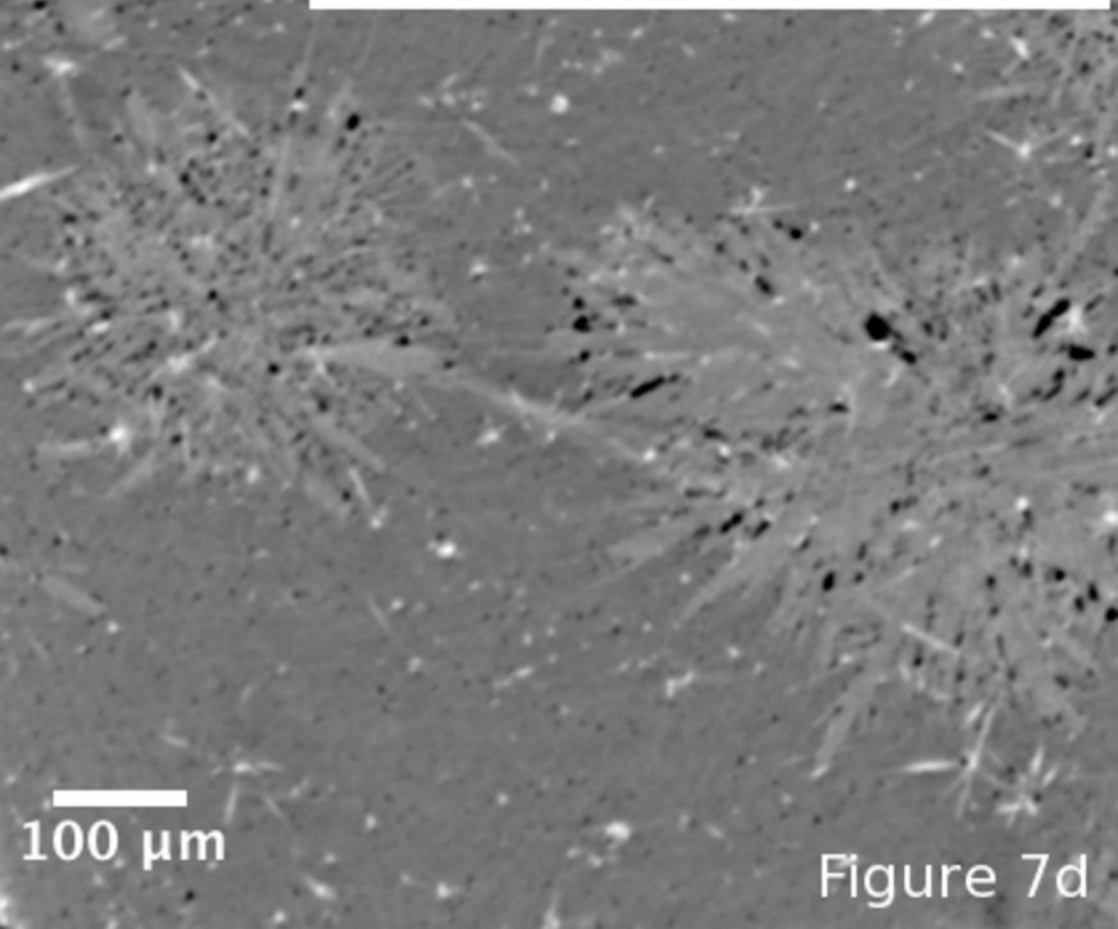
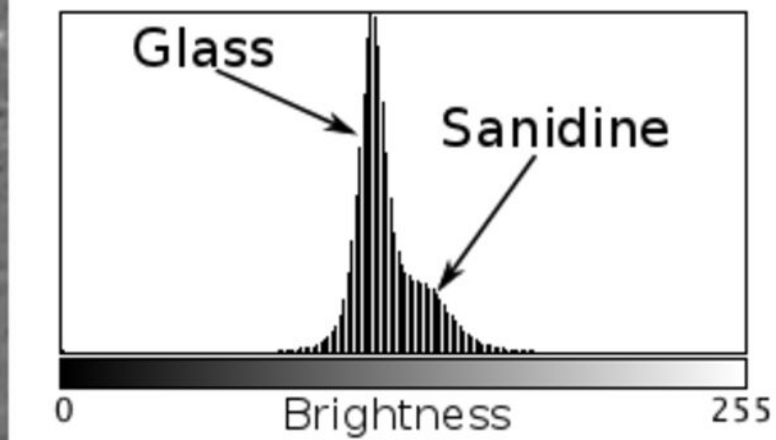
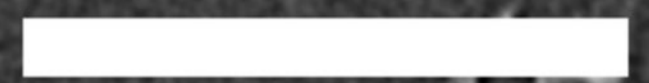
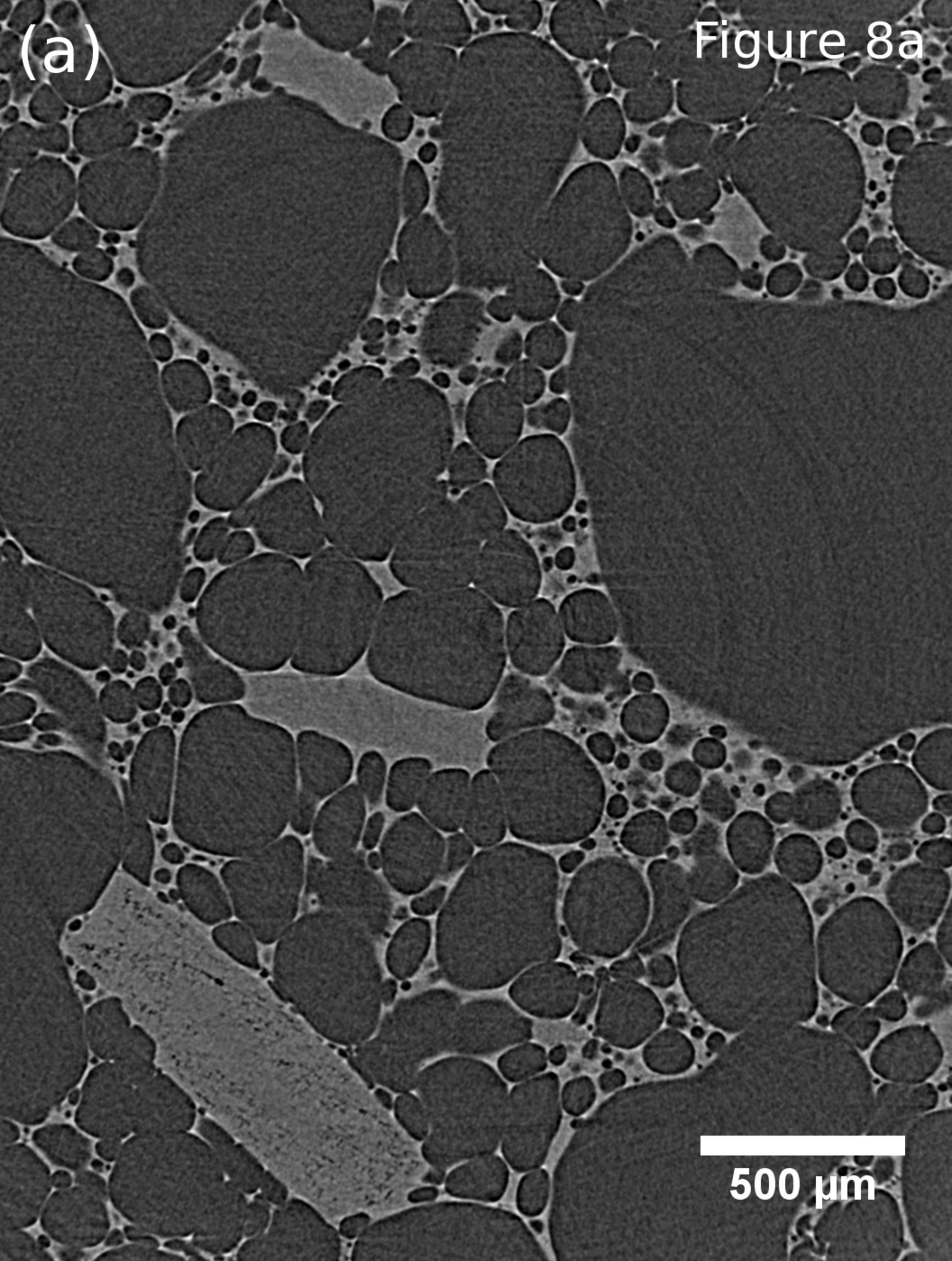


Figure 7d

(a)

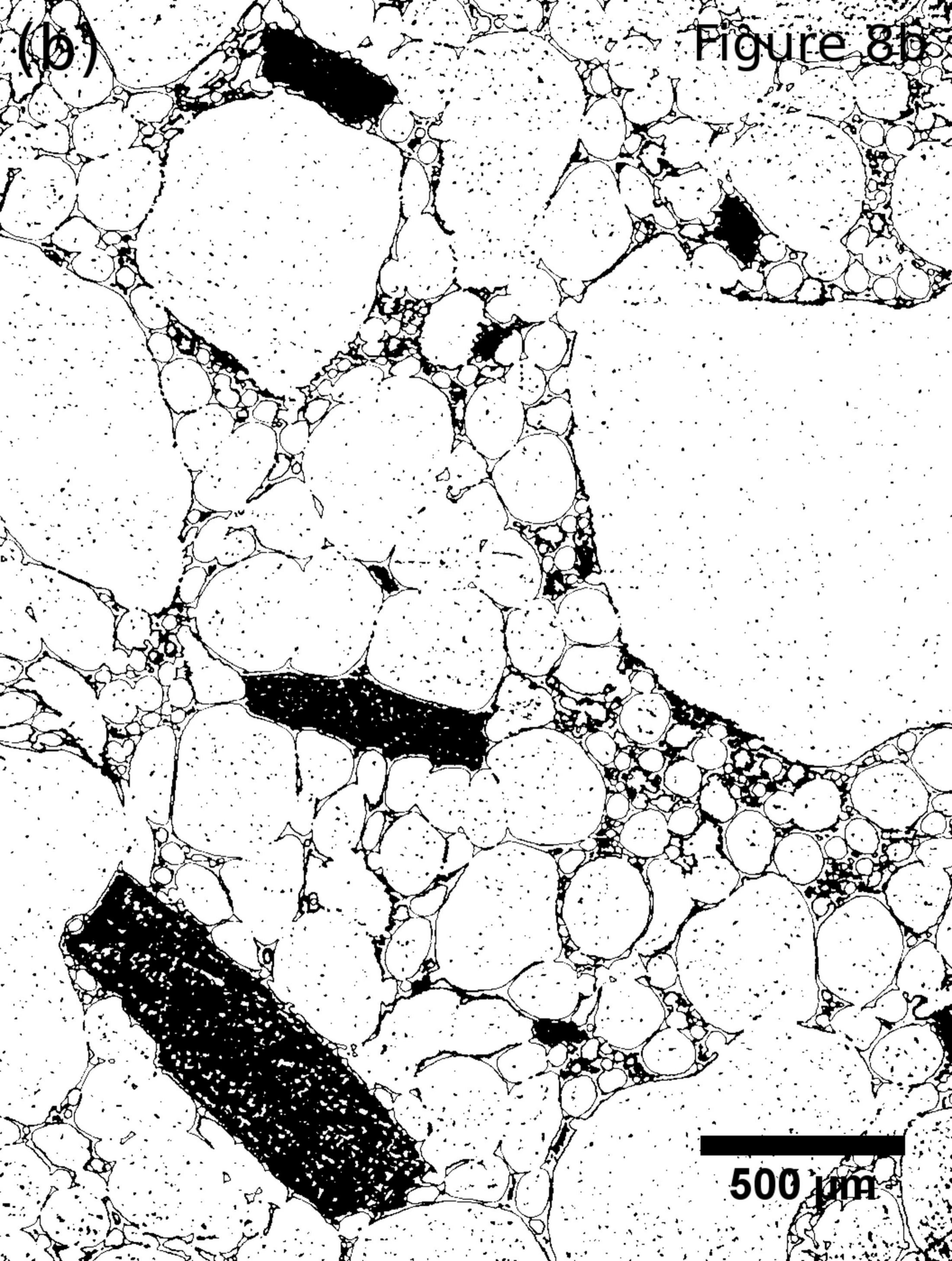
Figure 8a



500  $\mu\text{m}$

Figure 8b

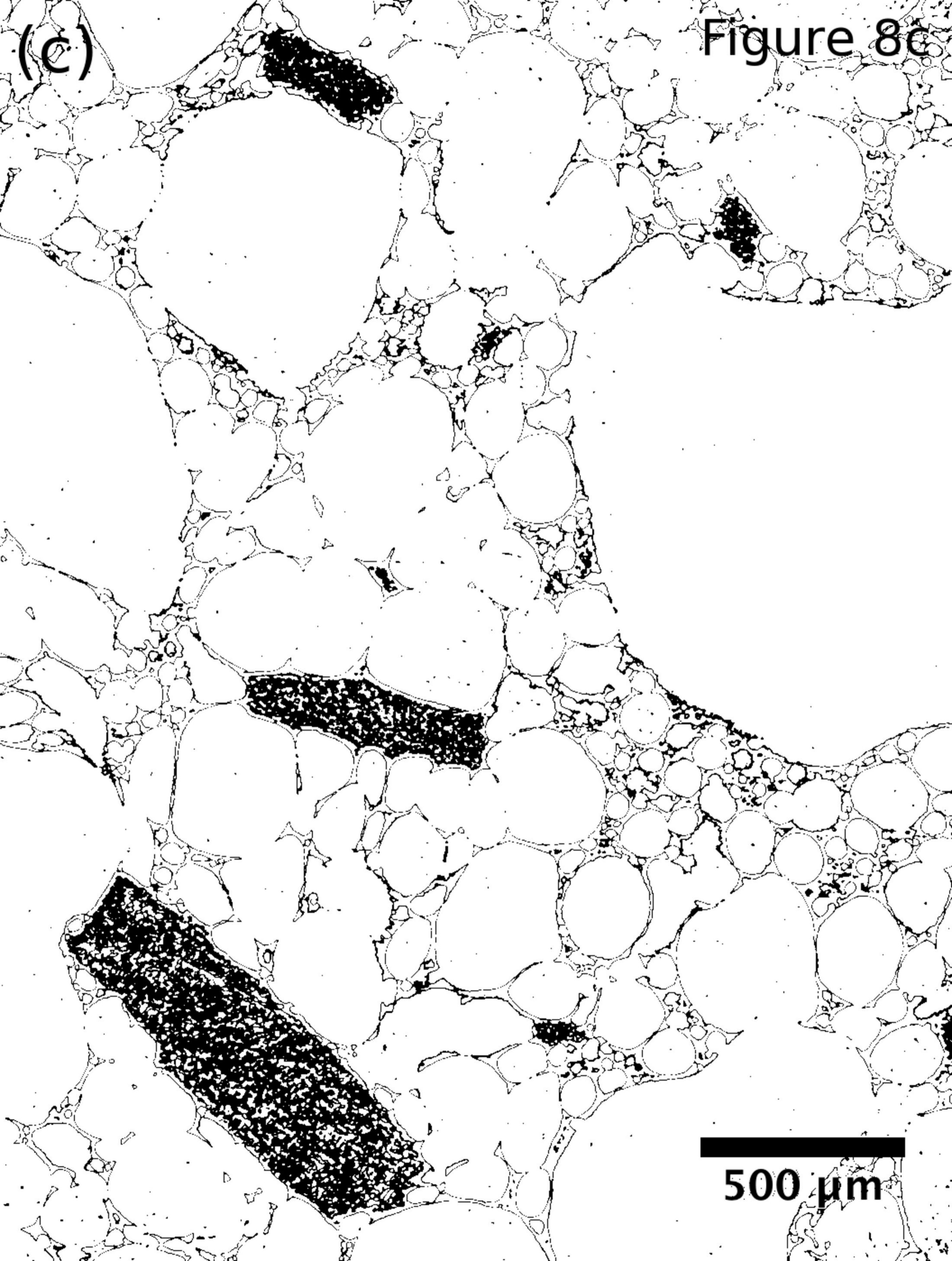
(b)



500  $\mu\text{m}$

Figure 8c

(c)



500  $\mu\text{m}$



(d)

Figure 8d



500 μm

(e)

Figure 8e



Figure 9

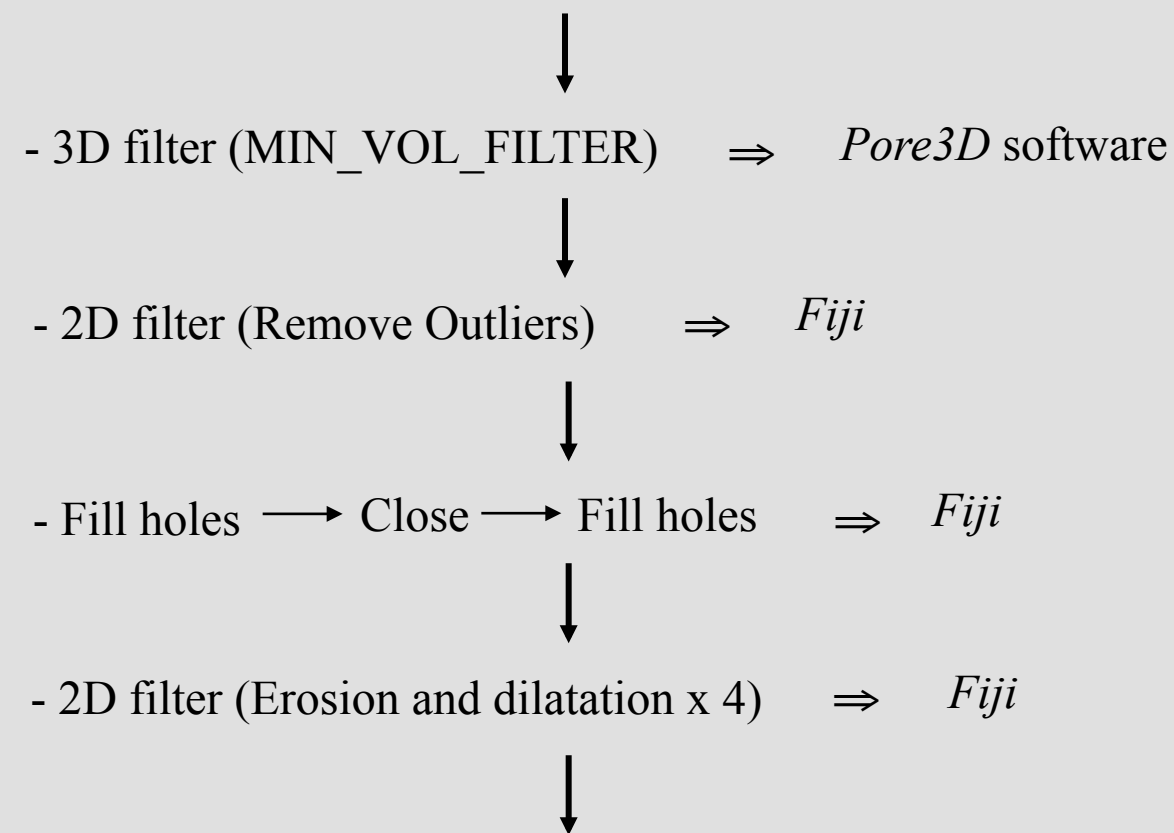
# Segmentation with masking procedure

## Manual thresholding

- **segmentation** of the real shape of plagioclase crystals taking into account a significant amount of pixel outliers, belonging to both vesicles and glass matrix that were not segmented after thresholding (Fig. 8b). We used the phase-retrieved raw volume (Fig. 8a) to segment plagioclase crystals. *Pore3D* software was used for the manual thresholding.

## Mask preparation

- **segmentation** consisted in the approximate isolation of the shape of plagioclase (not necessarily the real shape) trying to take into account the smallest amount of pixel outliers possible (Fig. 8c). *Pore3D* software was used for the manual thresholding. We used the phase-retrieved raw volume (Fig. 8a) for mask preparation.

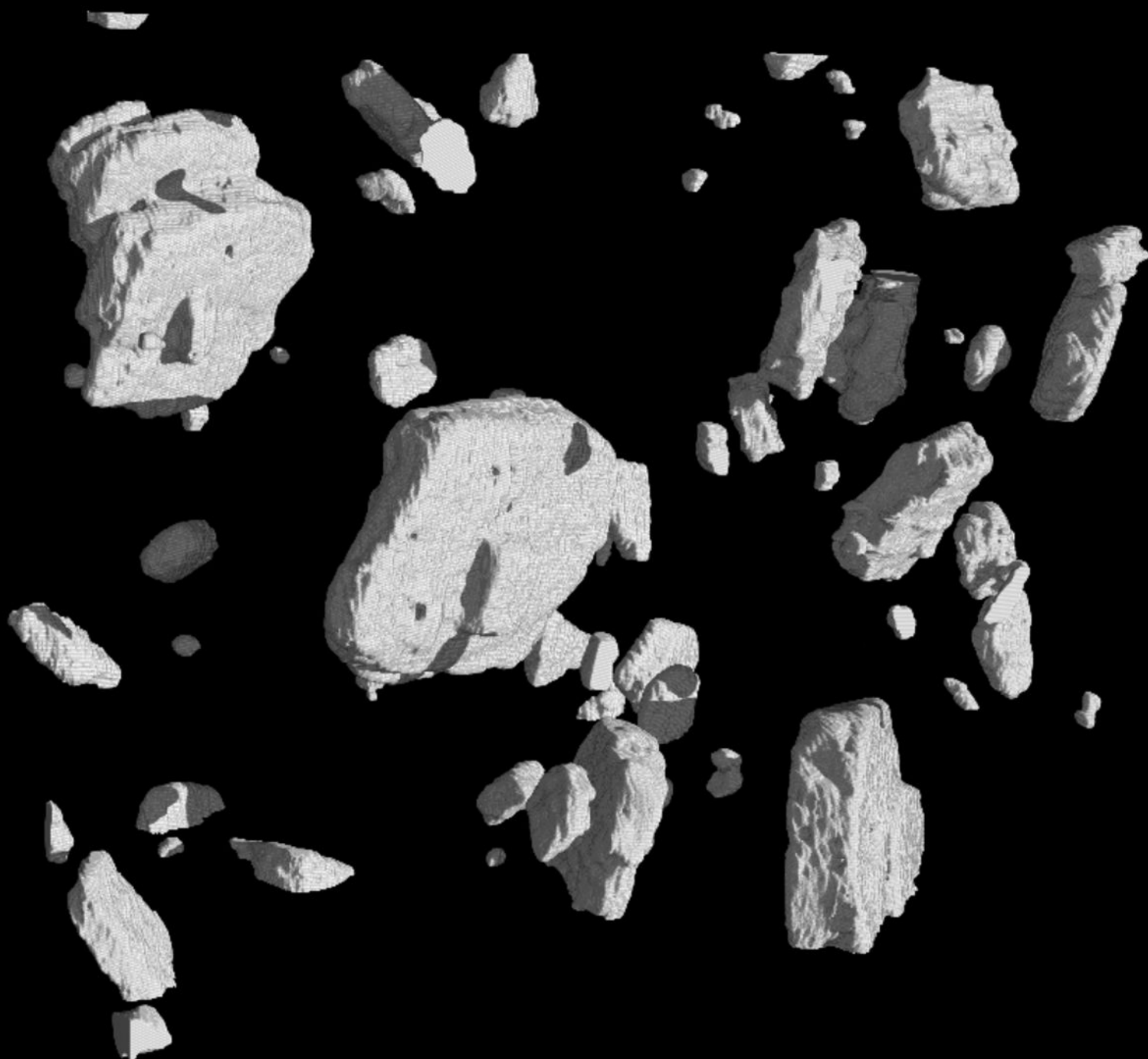


**AND** operator in *Fiji*

- Combine both the first segmentation (Fig. 8b) and the mask of plagioclase crystals (Fig. 8d) in order to complete the separation of the phase of interest.
- Plagioclase crystals were completely segmented preserving their shapes and internal structures (Fig. 8e)

Figure 10a

(a)



(b)



- 1 Table 1. Summary of acquisition conditions and image processing details of the analysed
- 2 Volumes of Interest (VOI) for the investigated samples.

---

Sample	Instrument	Isotropic voxel size ( $\mu\text{m}$ )	Imaged volume (pixels)	Imaged volume ( $\text{mm}^3$ )	Analysed VOI (pixels)	Analysed VOI ( $\text{mm}^3$ )
ST16424119						
8B	SYRMEP	2.2	1200x1400x1200	21.47	1056x1398x1000	15.72
D1	SYRMEP	2.0	-	3.95	-	3.95

---

- 3 note: ST164241198B is the natural pumice of Stromboli. D1 is the trachytic synthetic sample.