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Gamma mark: an ingenuity to ease the aiming of melt inclusions in phenocrysts with NanoSIMS

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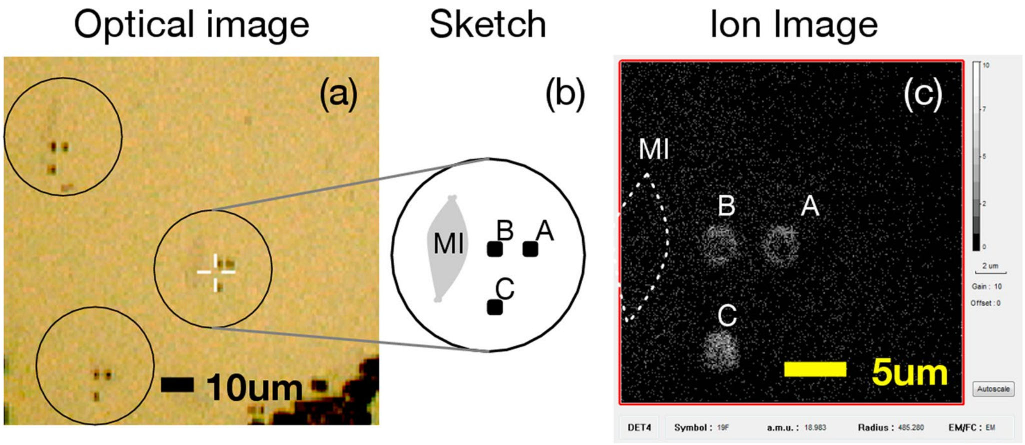
Abstract

This short report introduces one of the ways to quickly and accurately aim melt inclusions in phenocryst using NanoSIMS.

Keywords NanoSIMS, Melt inclusions, Volatile elements, Volcanic gas, Magma

Graphical Abstract



Introduction

Melt inclusions in phenocrysts (patches of glass a few to few tens μm in diameter) from volcanic rocks are the important analysis target for knowing the concentrations of volatile components in the magma before the eruption (many references, e.g., Miyagi et al. 2023). A secondary ion mass spectrometer (especially NanoSIMS) is a device that can quantify water and carbon dioxide in such small targets.

Problem

The size of the melt inclusions that are the subject of analysis in this paper is only 5 to several tens of μm on the sample surface, and a precision of about 1 μm is

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required to apply the beam to prevent them from protruding. The first step is to move the sample holder so that the target is within the irradiation areas of the primary ion beam.

However, melt inclusion does not enter the field of view of the ion image in one shot because the electric field differs depending on the location of the sample holder, which deflects the trajectory of the ion beam from the aiming. This problem appears with other SIMS instruments such as Cameca ims-3f, -4f, -6f, -7f, ims-1270, and -1280. The ion beam's position is sometimes 20 to 50 microns away from the intended location in the optical image. Nevertheless, with these SIMS, the misalignment of the primary ion can be immediately confirmed with an optical image, so it is easy to correct the stage position. However, this method is impossible with NanoSIMS because NanoSIMS cannot provide optical images and ion images simultaneously.

Furthermore, even if the aiming were accurate enough in the optical image mode position, it would shift several microns while moving to the ion image mode position of NanoSIMS. The deviation between the optical image and the ion analysis is more than 5 μm (sometimes about 100 μm), and the degree of the deviation is not constant, so it is impossible to correct it with simple arithmetic calculations.

Melt inclusions are not visible until the electric conductive coating, e.g., Au, is sputtered off. To look for offset melt inclusions, a relatively strong beam is needed to remove coatings of a wider area and ion images for about 5 to 10 min, and then about 1 min is needed to acquire an ion image with a weaker beam. The problem here is that melt inclusions are often not visible in the ion image in the first trial, and therefore, repeating the above operation with different sample positions is necessary. As a result, it sometimes takes 30 to 1 h to search for a single melt inclusion. It is extremely time-consuming to search for lost melt inclusions using ion imaging.

The procedure, called Γ mark method, solves two problems of NanoSIMS: the problem that the optical image aiming and the ion beam aiming do not match (common with other SIMS), and the problem that the stage position does not match between the optical image mode and the ion image mode (specific to NanoSIMS).

Solution

The procedure, Γ mark method, for quick and accurate targeting melt inclusions is described in (1)–(6).

(1) In optical image mode, preset the coordinates of the melt inclusions to be analyzed.

(2) In SIMS mode, make three marks with the ion beam at each preset coordinate in the following ways. Move the stage to the melt inclusion. Set the raster 1 μm, the Stage Navigator's X-Y step to 5 μm, and the ion current to 50 pA. Sputter for 10 s (Fig. 1, point A). Move the stage 5 μm to the left and sputter 10 s (Point B). Move the stage down 5 μm twice and sputter for 10 s (Point C). As a result of the above operations, three beam marks are formed like a Γ-shaped constellation; thus, it is the Γ mark. Repeat marking as much as the number of melt inclusions to analyze. The Γ mark is an asymmetrical geometric shape that can distinguish mirror images easily, which can be made by the minimum number of spattering. The vertical length of

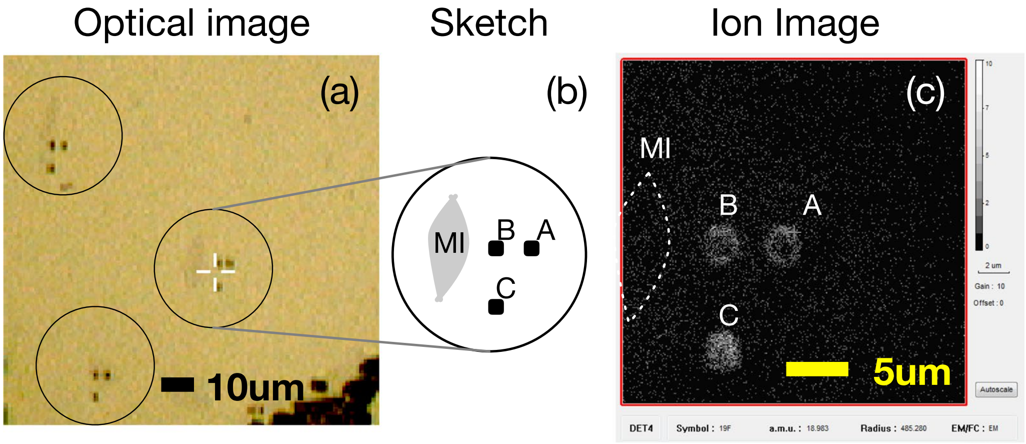

Fig. 1 Example of using the Γ mark. **a** Optical image. Circles surround the melt inclusion and the Γ mark. **b** Sketch of a melt inclusion and the Γ mark. MI is melt inclusion. A, B, and C are Points A, B, and C, respectively. **c** Ion image of ^{19}F. The Γ mark is visible, while the melt inclusion is invisible. The area surrounded by the dashed line indicates the position of the melt inclusion, as imagined from the information in the sketch

the Γ mark can be used as a 10-μm scale on the sample surface. The Γ mark resembles a face (eyes and mouth) and is easier for humans to distinguish it from other patterns on a polished surface.

(3) In optical image mode, check the location of the Γ mark (Fig. 1a). Sketch every Γ mark and the melt inclusion (Fig. 1b).

(4) In SIMS mode, observe the Γ mark with a ^{12}C and ^{19}F ion image (Fig. 1c). Recommended conditions are: raster is 25 μm, ion current 2 to 5 pA, dwell-time 500 to 700 μs/pixel. Freeze the image as soon as the first scan is complete.

(5) Correct the stage position. Based on the position of the Γ mark observed in the ion image and the sketch information in (3), adjust the stage position so that the melt inclusion is in the center of the field of view of the ion image. Utilize the NanoSIMS RTI program's function to center the stage to the location by clicking on the ion image.

(6) Confirmation of aiming. To increase the sputtering speed, narrow down the raster size of the ion image to about 6 μm and increase the current value to 50 pA. In addition to ^{12}C and ^{19}F,^{16}OH, ^{18}O, ^{30}Si, ^{32}S, and ^{35}Cl are also monitored. Since phenocrysts contain almost no S nor Cl, it is easy to confirm whether the outer shell of the glass inclusion is in the field of view of the ion image.

With this method, you will never miss the target if the sketch is correct. Although the Γ mark method requires additional operations: (2) marking with an ion beam (about 1 min; three 10-s irradiations and stage movement), (3) confirming the discrepancy between the Γ mark and the melt inclusion (it takes about 1 min per point, as multiple items are checked at once), and (4) scanning the surface using a minute current (about 1 min, the total time required for the analysis of melt inclusions is reduced by 5 to 10 times.

Other researchers have also tried to improve SIMS's analytical location accuracy. For example, there are methods in which marking by a focused electron beam using a SEM device and observing by oxygen ion images with SIMS device (Nagashima et al. 2015), marking using a FIB device and observing by oxygen images with SIMS device (Nakashima et al. 2012), or marking using a SIMS ion beam and observing using a SEM device (Kawasaki et al. 2022). Alternatively, a laser device attached to an ordinal light reflectance microscope can mark the sample surface (e.g., Photonic Instruments Co., Ltd). These methods sequentially process the sample with different equipment, requiring extra vacuuming waiting time, especially longer for water analysis. On the other hand, this report's method completes marking and observation using only the NanoSIMS device.

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Author contributions
The author installed NanoSIMS to AIST in 2016 and developed the idea described in this paper while using NanoSIMS to analyze melt inclusions.

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Availability of data and materials
Not applicable.

Declarations

Competing interests
The author has no competing financial or personal interests which may have impacted the interpretation of the presentation of this information.

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