# ATLAS 5 large-area and nano-scale imaging of iron ore from the El Laco volcano, Chile

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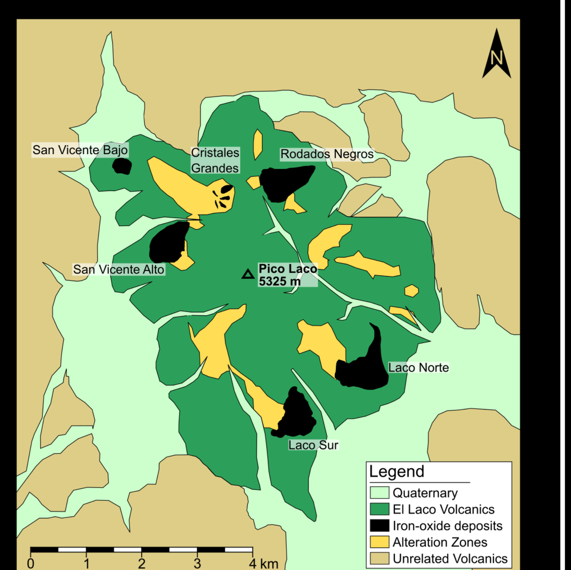
## Introduction

The El Laco iron deposit, located in the Chilean Andes, has more than 1.1 Gt (resources and reserves) of massive iron ore dominantly in the form of magnetite. It formed in the late Cenozoic (Pliocene-Pleistocene) at very shallow depths (<100 m) and relatively high temperatures, possibly above 1000°C. Texturally, the massive iron oxide rocks are vuggy and locally macrospherulitic and resemble extrusive rocks of magmatic origin. However, this interpretation has been challenged, and some investigators suggest a purely hydrothermal mode of origin.


General world location of the study area, the El Laco volcano, and a simplified geological map modified from Tornos, F., Velasco, F., & Hanchar, J. M. (2016). Iron-rich melts, magmatic magnetite, and superheated hydrothermal systems: The El Laco deposit, Chile. Geology, 44(6), 427–430.

"Flow contact" sample (EL-BM-01)

This sample, superficially resembling a flow contact, was collected in San Vicente Bajo, and has an approximate size of 13 x 7 x 5.5 cm. It is mostly composed of magnetite with apatite. The outer layer is massive magnetite with pervasive hexagonal euhedral to subhedral, fractured, apatite crystals ranging from 1 to 7 mm in diameter. The apatite crystals go up to 2.3 cm in length occurring perpendicularly to a sinuous contact with the inner layer. All growths of apatite seem to be rooted from this division.

## Purpose

The goal of this research is to shed light on the debate, by providing new observations in the form of a full suite of novel imaging datasets of El Laco ore samples. These include macro- to nano-scale Zeiss ATLAS 5 imaging datasets (LM, FIB-SEM, EDS, 3D Nanotomography and 3D EBSD) and tools to assist in the visualization of quantitative information. Samples of unconsolidated tephra and of coherent lava have been examined using a novel workflow of optical and electron microscope images in a correlative workspace. This suite of techniques allows a seamless correlation of large sample surfaces from the macro- to nano-scale, which subsequently enables the observation and interpretation of features that were previously inaccessible. These observations, at a higher resolution than previously available, will help to constrain whether the mineralizing fluid from which magnetite was deposited was a hydrothermal fluid or a melt.

## Atlas 5 Multi-Resolution Large Area SEM Imaging

ATLAS 5 is a combined system of hardware and software that allows for efficient sample navigation of correlated data from various sources (such as LM, SEM, FIB-SEM, EDS, etc.). Large area mosaics are scanned or rather imaged as the system acquires image tiles from simultaneous signals. Other sources may be imported later on within the correlative workspace where each source and various capture regions are aligned and appear as layers. What is obtained is a stacked set of images in which each layer can be leveled with transparency and all levels are navigated synchronously. ATLAS 5 has allowed us to quickly scan and identify different features, better assuring us of not missing tiny features that could have been missed otherwise.

### Spherulitic Texture (EL-BM-02)

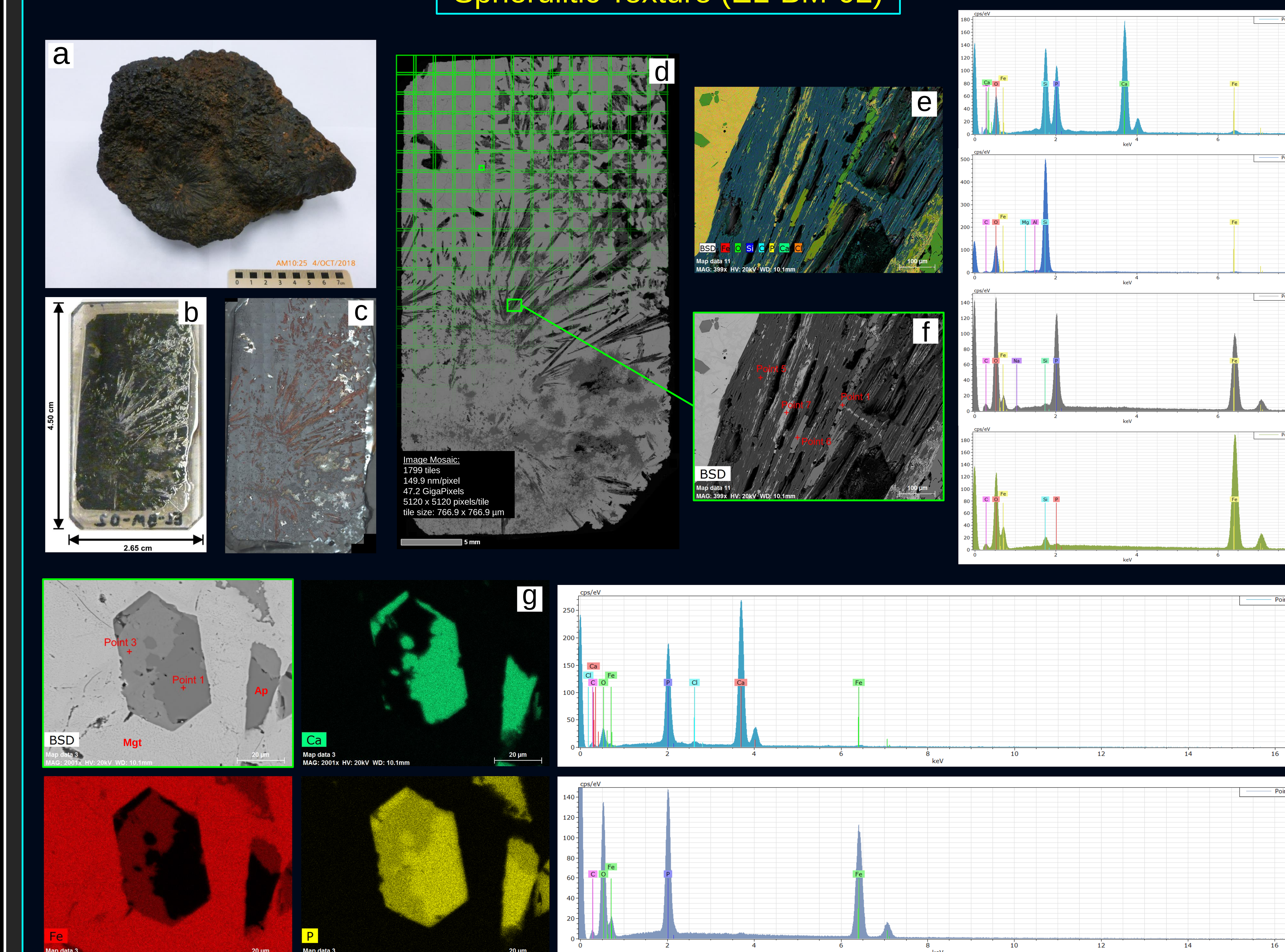



**Figure 1**: (a) The hand sample is from Laco Sur, where small magnetite spherules were found [1] near the open pit of "friable ore". Its approximate size is 17 x 16.5 x 10.5 cm with a metallic dark-grey lustre with most of the sample being orange-brown which is an indication of heavy alteration. The primary minerals are Magnetite, Hematite, possibly Maghemite or Martite (sample is magnetic with a few anomalous regions, repulsing a mag-pen; there are different magnetic domains, some naturally "magnetized" to attract and others to repulse, with no particular distribution observed), and a relatively soft (<5 Mohs scale) whitish mineral (only seen on a sawed off fresh surface, it fills small blob-like holes, most in orders of a few millimetres), later revealed to baryte (by EDS & EMPA). The "spherulitic magnetite" textures are radial or rather stellate growths of fine possibly magnetite and apatite needles ranging from 1 to 2.3 cm in length and no more than 1.5 mm in diameter with most being finer. These seem to be floating in massive magnetite matrix. The "cores" are somewhat vesicular as opposed to being plain massive. There are many small holes throughout the sample ranging from 1 to 4 mm in diameter, possibly from alteration. There are many pockets or vuggs with most being 1 cm or less in diameter. However, one of these is observed to be up to 5 cm in diameter with subhedral octahedrons of magnetite ranging from 1 to 2 mm in size. This is possibly formed from a gaseous or rather vaporous phase. Additionally, there are many smaller "incomplete spherulites" or rather fans of magnetite needles observed. (b & c) A thin-section was prepared from the sample (a) with a focus on the stellate, possibly rapid growth textures. (d) This is an ATLAS 5 produced multi-resolution large area SEM image mosaic of the (b & c) thin section. (e & f) Using EDS/EDX chemical maps, the composition of the stellate needles is revealed to be more complex. Point 5 is almost pure silica or rather a silicate, possibly diopside that was altered due to hydrothermal processes. Point 6 shows strong signals in all Fe-P-O, an unidentified phase. Point 7 is primarily Fe-oxide with minor amounts of Si and P, most likely Magnetite (Mgt). Point 1 has strong signals of Ca, Si, P and O, possibly altered diopside or apatite (Ap). (g) Another set of EDS maps shows a possible Fe alteration feature of a euhedral grain of what is chlorapatite (point 1), with a secondary (point 3) unidentified Fe-P-O phase.

### Porous Andesite (EL-JM-82)

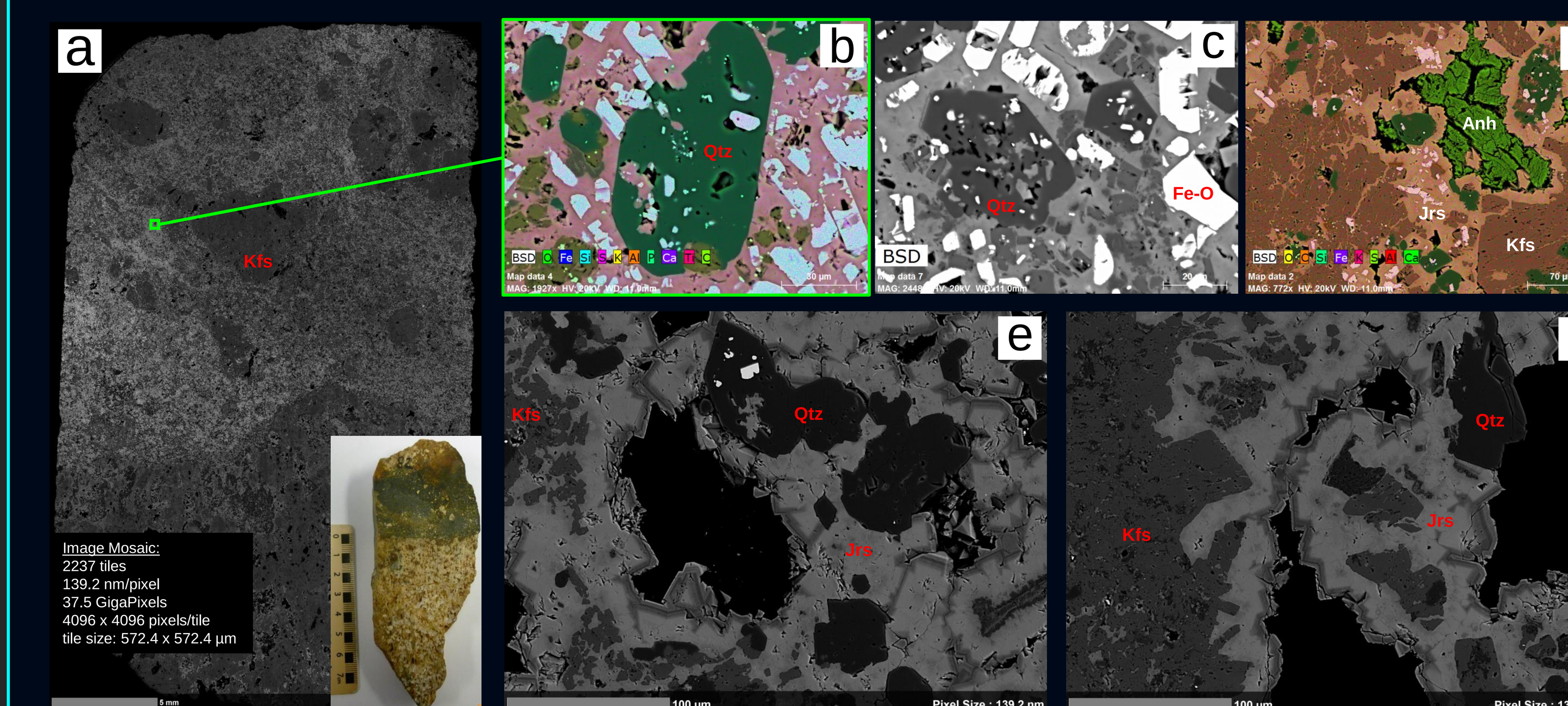



**Figure 2**: The hand sample EL-JM-82 (a, lower right), approximately 12.1 x 1.8 x 5.2 cm in size, accompanied with a thin-section (a) was obtained from a previous study [2] as sample EL-96-82. It is a slab cut of a porous andesite bomb from Laco Sur. The sample is porous or rather froth- or pumice-like beige andesite with ochrous material filling in numerous solution cavities. There is a magnetite vein that is from 3 to 6 cm wide cutting across the sample containing angular silicate rock fragments. Overall, the sample is significantly altered with darkened orange haloes around the cavities. (b & c) These are composite EDS maps showing a round silicate (dark grains) grains with many smaller inclusions. These small but bright inclusions are primarily Fe-oxide supposedly magnetite and in some cases, there is a strong silicon signal. That said, most of these magnetite grains are respectively euhedral but incomplete. These partially grown crystals along with the silicate grains are floating within a matrix of a relatively mixed composition. It has strong EDS signals of S, Si, Al, K, Fe and O, these are jarosite (Jrs) or possibly alunite (Alu) from hydrothermal alteration [3] and possibly some traces of mica. There are trace amounts of titanium throughout the sample. (d) The interstitial material or matrix shows similar composition on a larger scale with a significant amount of Fe-oxide. The upper right irregularly shaped grain is predominantly sulphur and calcium, likely a product of alteration. It is an unidentified phase, but its texture is relatively uncommon within the sample. (e & f) Sharply defined zoning or rather haloes are observed around the sample's cavities, as partly mentioned earlier. These are likely the result of hydrothermal fluids passing through.

### Unconsolidated Tephra (EL-JM-P4)

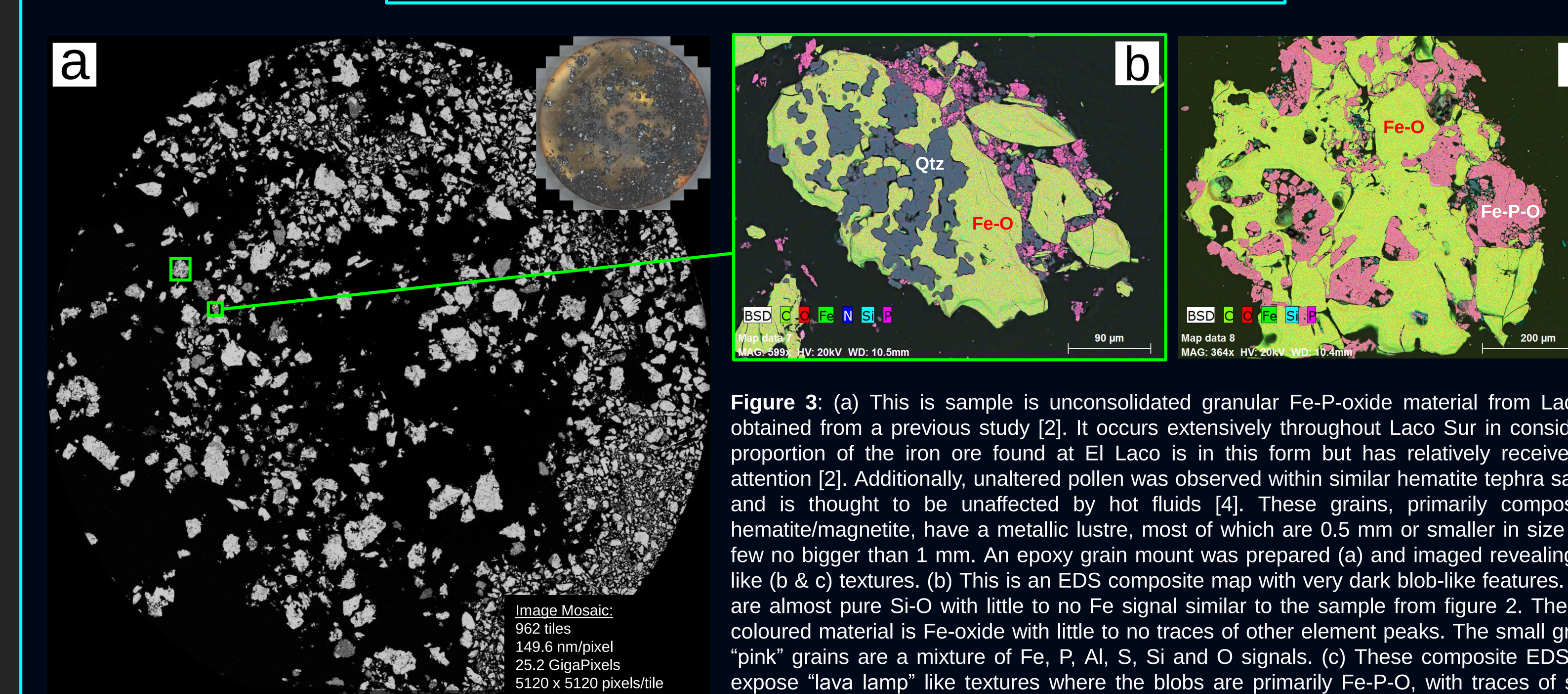
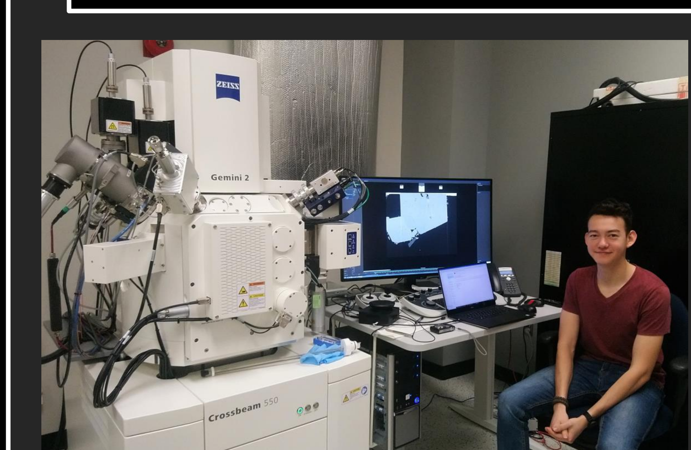
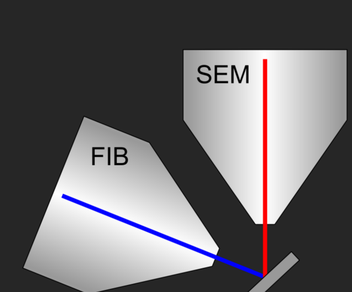



**Figure 3**: (a) This is sample is unconsolidated granular Fe-P-oxide material from Laco Sur obtained from a previous study [2]. It occurs extensively throughout Laco Sur in considerable proportion of the iron ore found at El Laco is in this form but has relatively received little attention [2]. Additionally, unaltered pollen was observed within similar hematite tephra samples and is thought to be unaffected by hot fluids [4]. These grains, primarily composed of hematite/magnetite, have a metallic lustre, most of which are 0.5 mm or smaller in size with a few no bigger than 1 mm. An epoxy grain mount was prepared (a) and imaged revealing melt-like (b & c) textures. (b) This is an EDS composite map with very dark blob-like features. These are almost pure Si-O with little to no Fe signal similar to the sample from figure 2. The lightly coloured material is Fe-oxide with little to no traces of other element peaks. The small granular "pink" grains are a mixture of Fe, P, Al, S, Si and O signals. (c) These composite EDS maps expose "lava lamp" like textures where the blobs are primarily Fe-P-O, with traces of silicate grains hosted in a Fe-oxide matrix.

## FIB-SEM and 3D Nanotomography






Targets have been selected for closer examination by FIB-SEM, a "dual beam" machine, to elucidate the composition and mineralogy of very small composite mineral inclusions suspected of being melt inclusions. A focused ion beam (FIB) is used to sequentially slice away cross-sections (thicknesses within the order of a few nanometres), where each slice is then aligned and extruded to reconstruct a 3D model. Simultaneously, EDS and EBSD imaging can also be performed to obtain compositional and crystallographic information in a 3D space. See sample EL-JM-11.

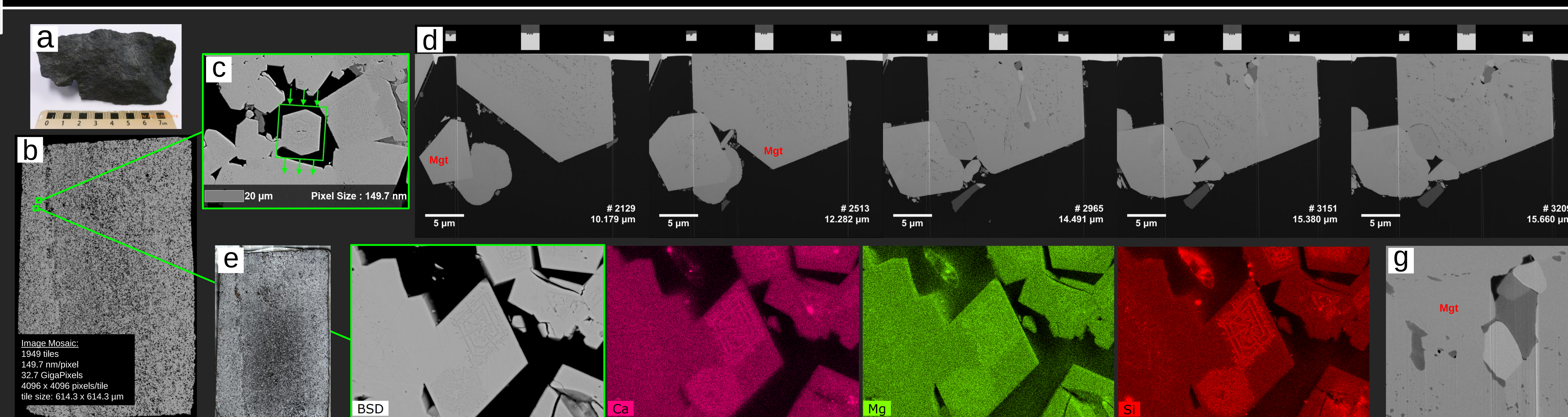



**Figure 4**: (a) The hand sample EL-JM-11, collected in Laco Sur, was obtained from a previous study [2] along with a prepared thin-section (b & e) that have been imaged by SEM & LM. The sample (a) is very fine-grained (grains less than 0.2 mm in size) magnetite with poorly defined layers resembling a welded tuff. (b) An image mosaic was produced with ATLAS 5 and has revealed many different features and phase such as Monazite-(Ce) with secondary cavity-filling structures (not shown here), possibly dendrites of Fe-oxide surrounded by a spheroidal overgrowth (not shown here) of Fe-P-O and many occurrences of well-defined zoning (shown above) in magnetite (Mgt) grains including what appears to be domains with a large number of inclusions. Some of which are distributed along growth zones making them very likely identifiable as primary inclusions. (c) A euhedral magnetite grain with well-defined zoning was chosen as a target for 3D nanotomography. The green box outlines the FIB milling target of about 34 µm by 32 µm. The arrows depict the FIB milling direction. (d) These are select slices from a preliminary ATLAS 3D run (on a ZEISS Crossbeam 550 L) which reveal that the zoning is in fact composed of very small inclusions and not voids. Further towards the middle of the grain, slices reveal polymineralic, possibly melt-, inclusions which would not have been visible with conventional SEM imaging. Particularly in slice #3151, the inclusions enclosed fit in a "jigsaw" like pattern, rounded grains wrapping around one another. There are as well other phases that have yet to be confirmed. (f) Prior to the ATLAS 3D run, a similar compositional EDS map was taken revealing that these inclusions are predominantly Ca-Si-Mg phases. However, further work is needed on a new grain in order to reconstruct a 3D model with compositional EDS/EDX (energy-dispersive x-ray spectroscopy) information, and possibly 3D EBSD (electron backscatter diffraction) crystallographic information. (g) This is a magnified view on the central inclusion cluster observed in slice #3151.

## Conclusion

The ATLAS 5 toolkit and workflow has provided efficient means to image and navigate through entire samples: this combined system of hardware, software and suite of techniques has allowed us to provide a seamless correlation of images from the macro-scale to the nano-scale and the tools to assist in the visualization of quantitative information. In addition to various observations in other samples, the target selected in sample EL-JM-11 for closer examination by FIB-SEM with ATLAS 3D has revealed new information. It has made visible the very small polymineralic inclusions exposing melt-like shapes that were previously not accessible. This reveals how important higher resolution (nano-scale) and large-area imaging really is.

## Acknowledgements

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## References

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