

Supplementary Materials

Methodology

Data processing of SEM-EDS

In our experiments, the small sizes of mineral phases posed challenges to accurately determining concentrations of major elements using SEM-EDS and LA-ICP-MS. This was due to the overlap of analytical spots with the surrounding glass matrix. To address this issue, we utilized a linear regression approach for Cr-spinel, magnetite, plagioclase, and olivine. Our strategy involved using an element with low abundance in these minerals but higher concentrations in the glass matrix as an internal standard. This calibration technique aimed to correct for some elements that may have been contaminated by the glass composition during analysis. For Cr-spinel and magnetite, we selected SiO₂ as the internal standard due to its entire incompatibility in spinel-group minerals. For plagioclase, TiO₂ served as the calibration element owing to its extremely low concentration in plagioclase. The strong correlation (R-square values > 0.8) between SiO₂ and oxides such as Al₂O₃, FeO_T, CaO, and TiO₂ can be observed in the magnetite grains. However, weaker correlations were observed between SiO₂ and Cr₂O₃, as well as between SiO₂ and MgO. These weaker correlations may be attributed to variations in Cr₂O₃ and MgO concentrations across different magnetite grains, likely due to Cr-Fe³⁺ and Mg-Fe²⁺ substitutions rather than the influence of the glass matrix. This assumption is supported by a better correlation for Cr-spinel grains. Thus, the calibration of major element concentrations in magnetite and spinel-group minerals is considered reliable. However, the R² values for many elements in plagioclase below 0.90 and sometimes under 0.70. This could be due to inherent chemical variations within the plagioclase crystals, SEM-EDS analytical errors, or insignificant correlations among these elements. The calculation of SiO₂, Al₂O₃, CaO and K₂O concentrations in plagioclase assumed a TiO₂ concentration of zero. For elements like MgO and MnO, which lacked high R² values, the minimum measured concentration was directly used. For Na₂O, we chose the maximum concentration. For olivine compositions, the significant correlations between Ni and both Al and Na suggested that decreases in Ni content must be attributed to the contamination of glass matrix.

Extended results and discussion

Mineral compositions of natural high-Mg andesite samples

All the spinel group minerals belong to the “Oxyspinel” group (A₂₊₃₊(B₃₊)₂O₄) (Bosi, et al. 2019) and can be subdivided based on the projections from magnetite or ulvöspinel prism (Fig. SI 2a). For simplicity, we adopted the classification in Figure SI 3a in terms of the relative proportions of the three trivalent cations, Cr, Al, and Fe³⁺. All the spinel group minerals contain moderate to large extent of MgO (up to 15 wt.%; Fig. SI 4e), which are more or less composed of pleonast (MgAl₂O₄) and Fe-pleonast (Fig. SI 3b). The primitive spinel group minerals consist mainly of chromite and hercynite, which are dominated by Cr and Al in their lattices, respectively. Natural magnetite usually contains less than 4 wt.% TiO₂

accompanied by variable Al_2O_3 concentrations ranging from 0.64 to 6.25 wt.%. In contrast, natural ilmenite contains less MgO and V_2O_3 (Fig. SI 4a).

Natural olivine compositions can be categorized into two distinct groups: Group 1 features olivine with higher forsterite (Fo) Group 2 consists of olivine with lower Fo values (Fig. SI 5). The highest Fo value found in the natural olivine is 92.94. Additionally, the CaO concentrations in both natural olivine groups are indistinguishable. Meanwhile, natural clinopyroxene and orthopyroxene show less variation in composition (Fig. 3), identified as augite and enstatite, respectively (after Morimoto (1989)). The pyroxene end-member compositions were calculated using the classification diagrams of Lindsley and Andersen (1983) to observe compositional shifts and verify equilibrium conditions (Fig. 3). Natural clinopyroxene spans a broad temperature range from 900 to 1200°C, while natural orthopyroxene mainly falls between 800 and 1100°C. Plagioclase, for the most part, shows a limited composition range, with anorthite values spanning 62.98–74.01, regardless of whether in rim or core areas. The range of $K_d(\text{Ca-Na})$ values (where $K_d(\text{Ca-Na}) = [\text{X}_{\text{Ca}}^{\text{plag}} * \text{X}_{\text{Na}}^{\text{liquid}}] / [\text{X}_{\text{Ca}}^{\text{liquid}} * \text{X}_{\text{Na}}^{\text{plag}}]$) and anorthite values for natural plagioclase are between 1.90 and 2.97 as well as 62.98 and 74.01, respectively (Fig. 4). The temperatures derived for plagioclase formation span a narrow window from 1018°C to 1059°C (Putirka 2008). A subtle distinction among the three high-Mg andesite samples reveals that the plagioclase in the 2000 high-Mg andesite formed at a relatively lower temperature compared to the plagioclase in the high-Mg andesites that were erupted at 1977 and 1989.

Chemistry and evolution of spinel group minerals

Spinel group minerals have long been acknowledged for their broad crystallisation range, rendering them key indicators of petrogenesis in variable settings (Irvine 1965; Irvine 1967). Chromite ($(\text{Fe}, \text{Mg})\text{Cr}_2\text{O}_4$) is typically the first crystalline phase in a variety of ultramafic or mafic rocks (Barnes and Roeder 2001) and its use has been instrumental in distinguishing rocks of different tectonic origins (Cookenboo, et al. 1997; Kamenetsky, et al. 2001), pinpointing the sources of volcanic rocks (Arai 1992) and mantle peridotites (Arai 1994; Dick and Bullen 1984). Cr-spinel also acts as a significant reservoir for platinum group elements (Kamenetsky, et al. 2015) and serves as an oxybarometer in the upper mantle (Ballhaus, et al. 1991; Mattioli and Wood 1986; O'Neill and Wall 1987; Wood and Virgo 1989; Woodland and Wood 1990). Magnetite can crystallise from both high-temperature silicate melts and low-temperature hydrothermal fluids, and has been extensively utilised to trace the origins of sedimentary and volcanic rocks (Grigsby 1990) and to identify the sources of materials in diverse deposits (Dare, et al. 2014; Pisiak, et al. 2017). Its prevalence in various magma types is thought to influence oxygen fugacity and differentiation in arc magmas (Jenner, et al. 2010; Lee, et al. 2010), potentially playing a role in the genesis of Cu–Au deposits (Sun, et al. 2004).

Spinel group minerals, though not abundant like pyroxenes and plagioclase, are frequently seen in the natural high-Mg andesite rocks (< 1%) (Cole, et al. 2000; Kilgour, et al. 2021; Rapien, et al. 2003). The compositions of spinel group minerals from the primitive chromite to the more evolved magnetite. Besides, intermediate phases like ferrian-chromite, indicative of Fe^{3+} and Cr^{3+} exchange during the crystallisation process (Fig. SI 2), suggest that the evolution of spinel group minerals records the magma evolution of the high-Mg andesites. The significant compositional divergence observed among spinel group minerals formed at various crystallisation

stages reflects the peritectic reaction of Cr-spinel (+ olivine) with the melt leading to clinopyroxene production (Irvine 1965; Irvine 1967). Our experiments at 200 MPa and 1100°C successfully reproduced the cotectic relationship between chromite and olivine (Fig. SI 3), which further demonstrates their reliability. The progression from Cr-spinel to magnetite is partially consistent with the “Fe–Ti” evolutionary trend identified by Barnes and Roeder (2001). The TiO₂ concentrations in these minerals also show a tendency to rise as the magma undergoes crystallisation (Fig. SI 4c). This process is accompanied by an increase in the $\text{Fe}^{2+}/(\text{Mg} + \text{Fe}^{2+})$ ratio in the spinels, a consequence of the crystallisation of olivine and clinopyroxene (Barnes and Roeder 2001). Globally, the “Fe–Ti” trend is indicative of low-pressure magma differentiation (Barnes and Roeder 2001), which concurs with the findings of our studies on liquidus equilibria.

Our high-Mg andesite samples contain hercynite phenocrysts with exceptionally low Cr₂O₃ (generally < 0.15 wt.%) (Fig. SI 3b). It is unlikely that these formed through the substitution of Al for Cr cations—a process indicating high-pressure or polybaric crystallisation (Barnes and Roeder 2001), due to Cr–Al exchange cannot entirely eliminating Cr cations from chromite. Moreover, the observed phenocryst-like hercynite grains are unlikely to grow larger after a prolonged period of cation exchange at high levels of crystallinity. Hercynite is stable across a broad spectrum of temperatures and pressures (up to 8.5 GPa and 1720°C) and is present in Earth’s metamorphic and igneous rocks as well as chondrites (BOHLEN, et al. 1986; Itoh, et al. 2004; Puziewicz and Johannes 1988; Schollenbruch, et al. 2010; Waters 1991; Woodland and Wood 1990). Experimental studies have shown that hercynite is more stable at 1 atmospheric pressure than under high-pressure conditions, due to the dissolution of excess Al₂O₃ at lower pressures (Woodland and Wood 1990). In addition, Mg and Ferric iron can help stabilizing hercynite ($\text{Fe}^{2+}\text{Al}_2\text{O}_4$) at low pressures (1 atm) and high temperatures (up to 1200°C) (Jastrzębska, et al. 2023; Woodland and Wood 1990). The coexistence of hercynite with augite, fayalite, plagioclase, and quartz at low pressures has been reported (Treiman 1985). Hercynite is also unstable at low oxygen fugacities (about one log unit below Quartz-Ilmenite-Fayalite) between 900 and 1100°C (Woodland and Wood 1990). For the Whakaari hercynites, Mg and ferric iron are always present (Fig. SI 3b). Given the absence of mantle xenoliths and the lack of evidence for metamorphic processes during the magmatic history at Whakaari, we infer that these hercynites formed in a relatively oxidized, recharged mafic magma characterized by low Cr₂O₃ and high Al₂O₃ and MgO concentrations in an upper-crustal magma chamber. This may also explain the absence of hercynite in our experiments as a static environment is unable to stabilize hercynite during magma differentiation. Additionally, the co-production of Cr-spinel, hercynite and magnetite in the high-Mg andesites suggests a mixing between different generations of magmas and crystals.

The relationship between magnetite and ilmenite

In our 200 MPa experiments, ilmenite is stable at $\Delta\text{NNO} +1$ but is replaced by magnetite at $\Delta\text{NNO} +2$ when the magma crystallinity reaches 30 to 60%. This aligns with prior studies indicating that ilmenite forms under conditions of lower oxygen fugacity compared to magnetite (Toplis and Carroll 1995). However, magnetite can be found in either 1 atm experiments at 1120°C at $\Delta\text{NNO} +1$ (anhydrous) or 500 MPa experiments at 950°C (water-saturated) under much more reduced conditions. At the same pressure, the water-saturated experiments (R2A4) on the bulk rock powder

starting material crystallized magnetite, whereas the water-undersaturated experiments (R2A5) led to the formation of ilmenite only. These results imply that magnetite saturation is influenced by a combination of factors including water content, oxygen fugacity and pressure. It is noteworthy that magnetite and ilmenite co-exist in a relatively evolved natural high-Mg andesite (WI05). The titanium concentrations in magnetite are generally low (Figs. SI 3, 4), suggesting that the liquid can enrich titanium during crystallisation permitting subsequent ilmenite saturation. MELTS simulations consistently result in the crystallisation of ilmenite (rhombohedral oxide) after magnetite (Jiang et al., 2024). However, our experimentally produced ilmenite consistently has higher Mg (up to 8.18 wt.%) and V (up to 1.18 wt.%) compared to ilmenite from the natural high-Mg andesites (Fig. SI 4). Since ilmenite formation is only observed at 200 MPa and NNO +1 in our experiments, this suggests that the experimental conditions may not be optimal for replicating natural ilmenite compositions.

Supplementary Figures

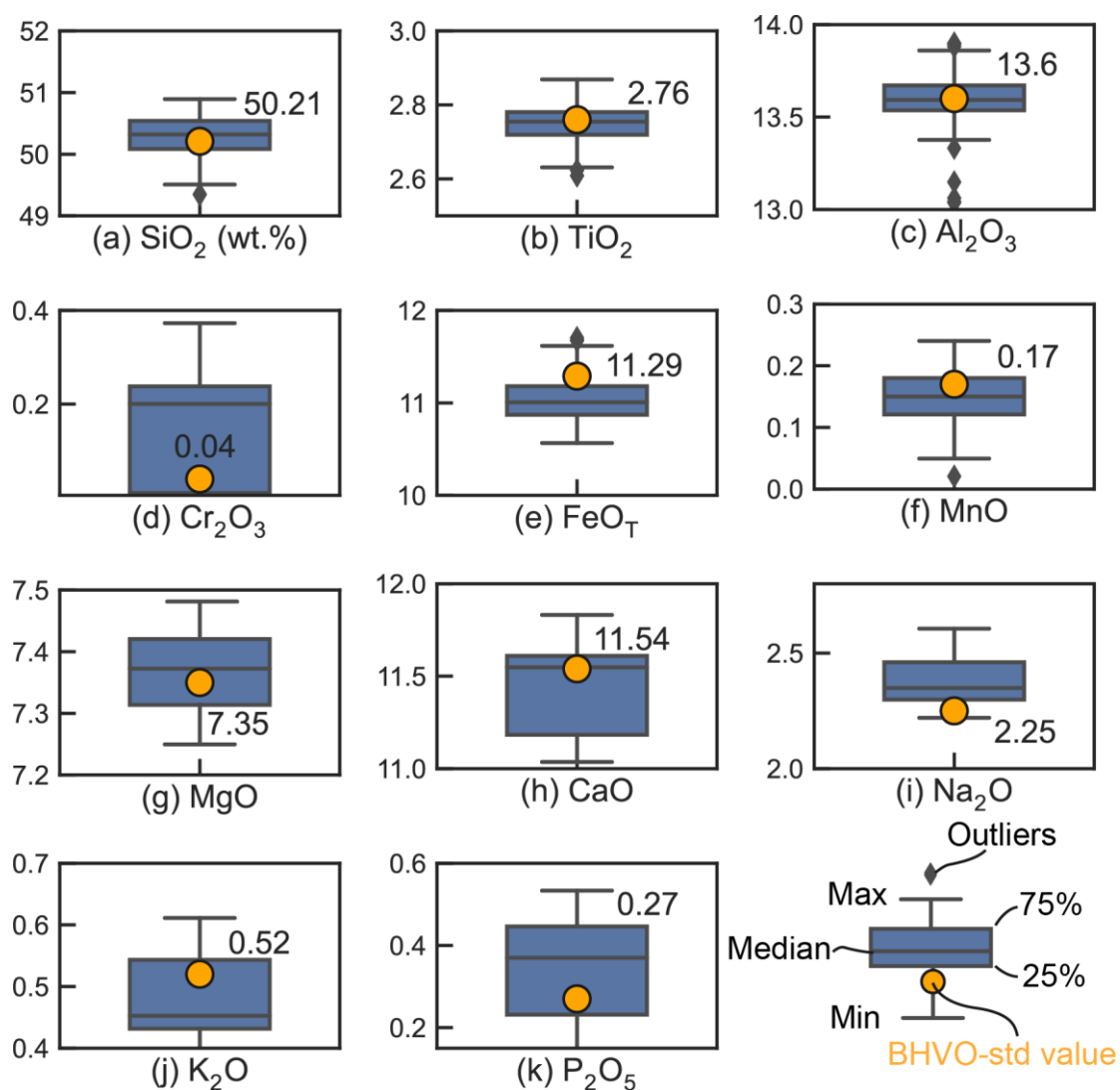


Figure SI 1. Compositions of BHVO-2g standard sample determined using SEM-EDS. The orange dots represent the recommended value from United States Geological Survey (USGS).

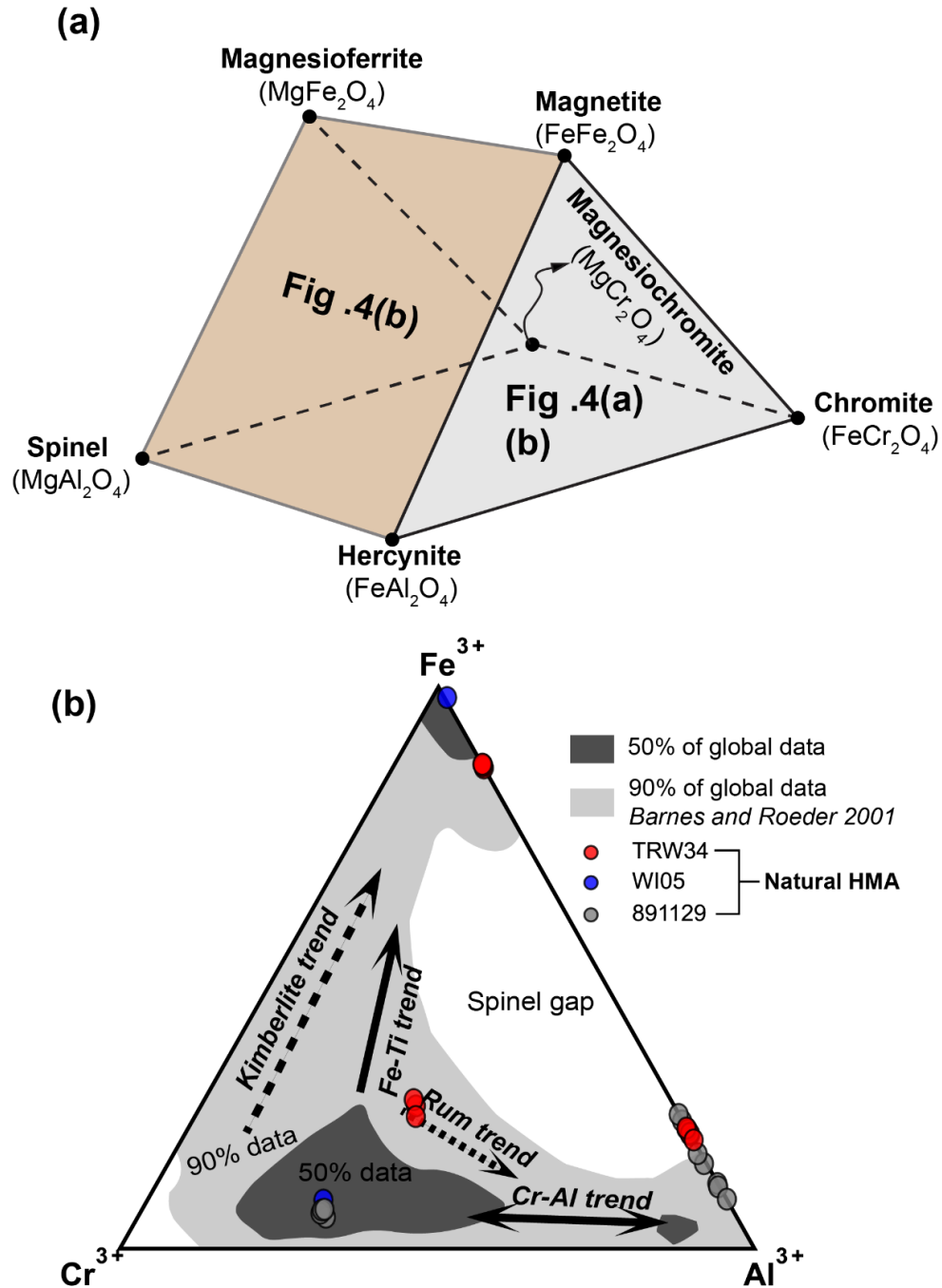


Figure SI 2. Chemical classification diagrams for the spinel group minerals. (a)

Magnetite or Ulvöspinel prism consists of 6 endmembers of spinel-hercynite-chromite-magnesiochromite-magnesioferrite-magnetite (Deer, et al. 2013; Stevens 1944). (b) Fe^{3+} - Cr^{3+} - Al^{3+} ternary projection onto the triangular face “b” in (a), where $\text{Cr}^{3+} = \text{Cr}/(\text{Cr} + \text{Fe}^{3+} + \text{Al})$; $\text{Fe}^{3+} = \text{Fe}^{3+}/(\text{Cr} + \text{Fe}^{3+} + \text{Al})$; $\text{Al}^{3+} = \text{Al}/(\text{Cr} + \text{Fe}^{3+} + \text{Al})$. The “Spinel gap” field originated from Barnes and Roeder (2001).

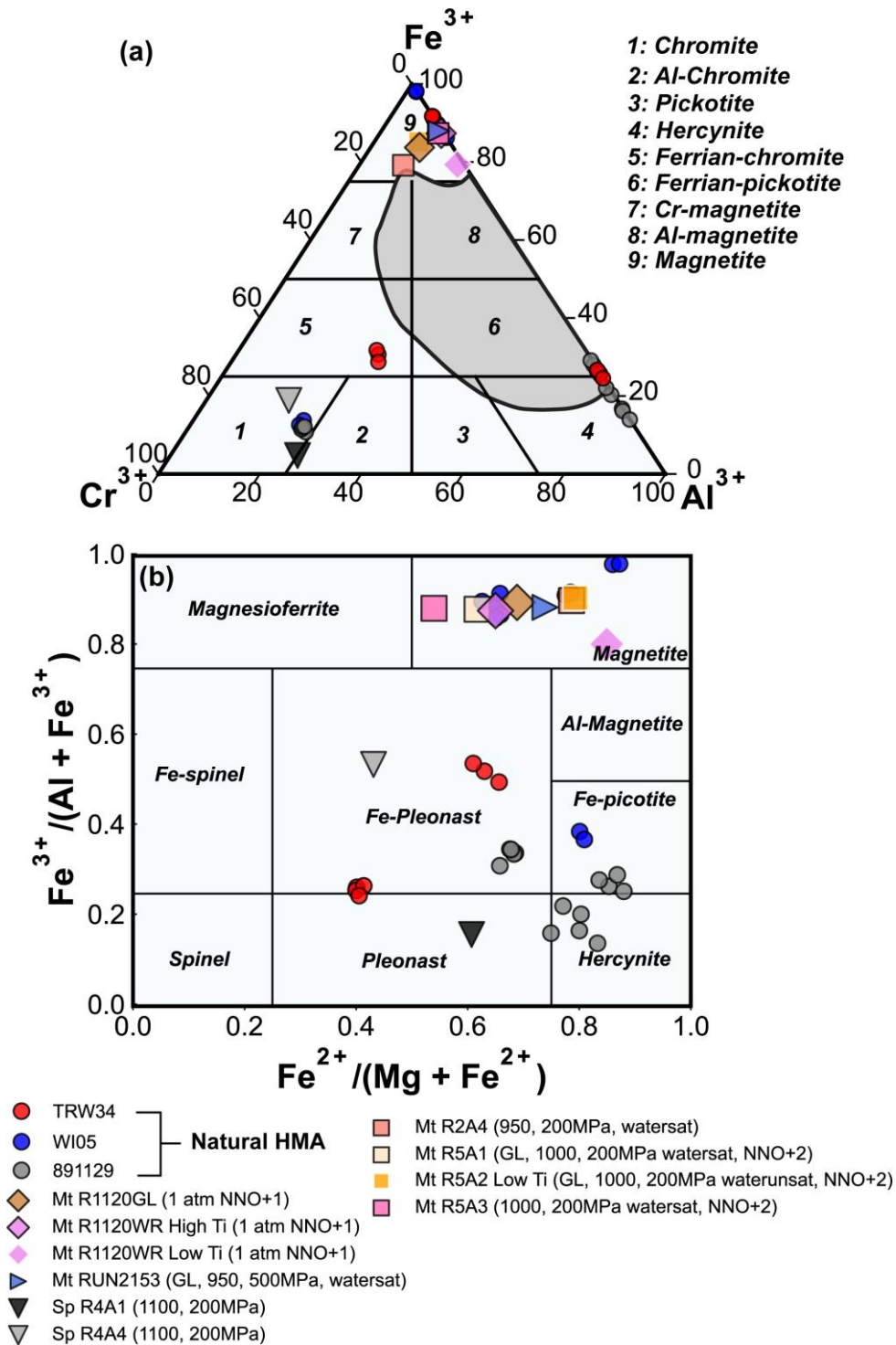


Figure SI 3. Chemical classification diagrams for the spinel group minerals. (a) Fe³⁺-Cr³⁺-Al³⁺ ternary projection onto the triangular face “b” in (a), where Cr³⁺=Cr/(Cr + Fe³⁺ + Al); Fe³⁺=Fe³⁺/(Cr + Fe³⁺ + Al); Al³⁺=Al/(Cr + Fe³⁺ + Al). The “Spinel gap” field originated from Barnes and Roeder (2001). (b) Binary classification diagram showing exchange of Fe²⁺-Mg²⁺ and Fe³⁺-Al³⁺ cations in the structural site “X” (Deer, et al. 2013; Stevens 1944).

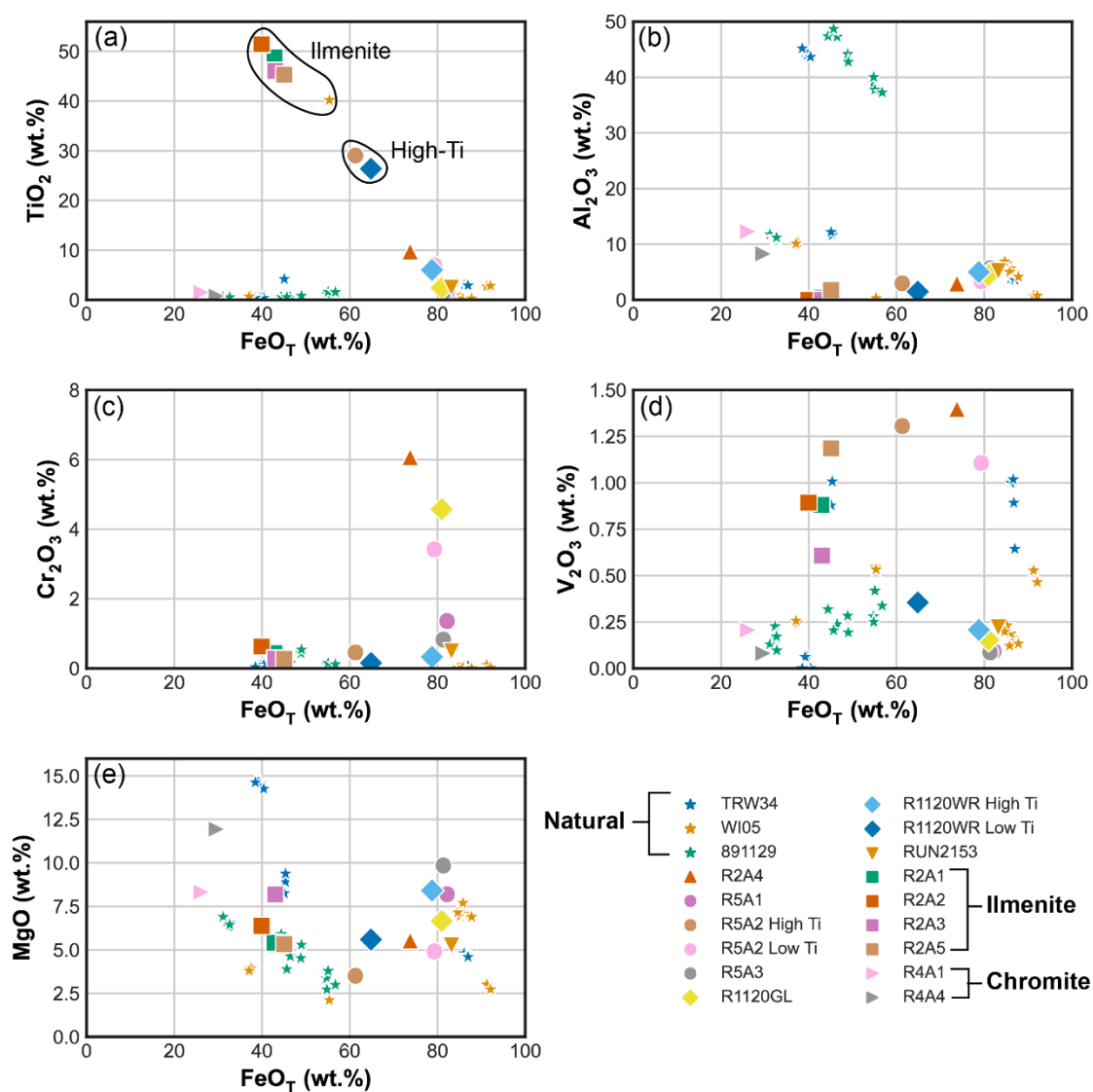


Figure SI 4. Chemical compositions of ilmenite and magnetite produced in our experiments. (a) TiO_2 (wt.%) versus FeO_T (wt.%). (b) Al_2O_3 versus FeO_T (wt.%). (c) Cr_2O_3 versus FeO_T (wt.%). (d) V_2O_3 versus FeO_T (wt.%). (e) MgO versus FeO_T (wt.%).

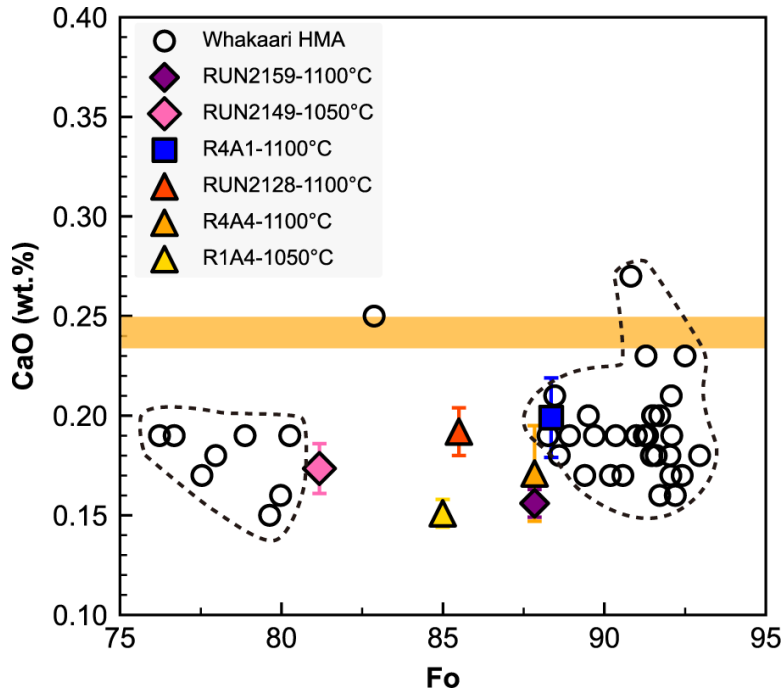
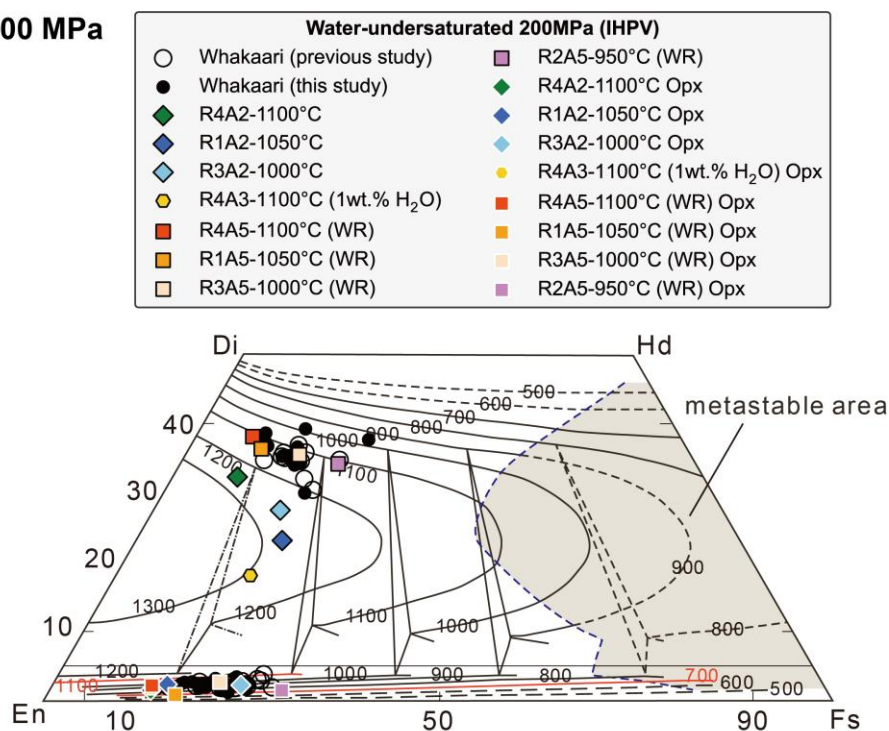


Figure SI 5. Composition of experimental and olivine compositions at Whakaari. (a) CaO (wt.%) versus Fo ($Fo = Mg / (Fe + Mg) * 100$ in molar fraction) The published olivine compositions are collected from Cole and Graham (1989); Heyworth, et al. (2007); Mandon, et al. (2021); and Shiraki, et al. (1994). CaO contents in these olivine grains (Table 3) were corrected for secondary fluorescence effects during SEM analysis. Error bars reflect the maximum and minimum values calculated from grain size variations, following the correction procedures of Gavrilenko et al. (2023).

(a) 200 MPa



(b) 500 MPa

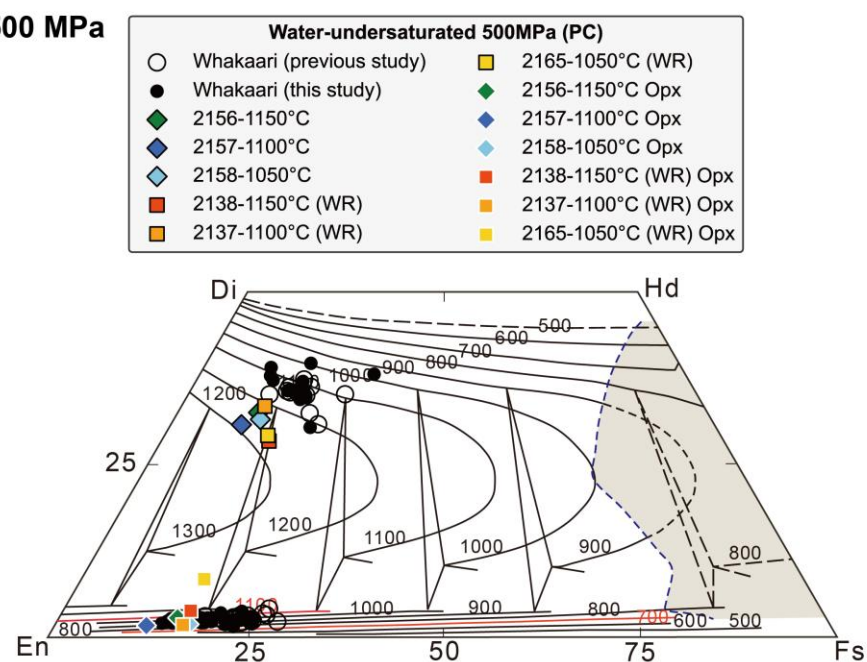


Figure SI 6. Wo–En–Fs ternary diagrams for pyroxenes thermobarometer (modified after (Lindsley & Andersen, 1983)) showing the results from water-undersaturated experiments and natural samples. (a) 1 atm and (b) 200 MPa. The natural pyroxenes were collected from Cole and Graham (1989); Heyworth, et al. (2007); Mandon, et al. (2021); and Shiraki, et al. (1994).

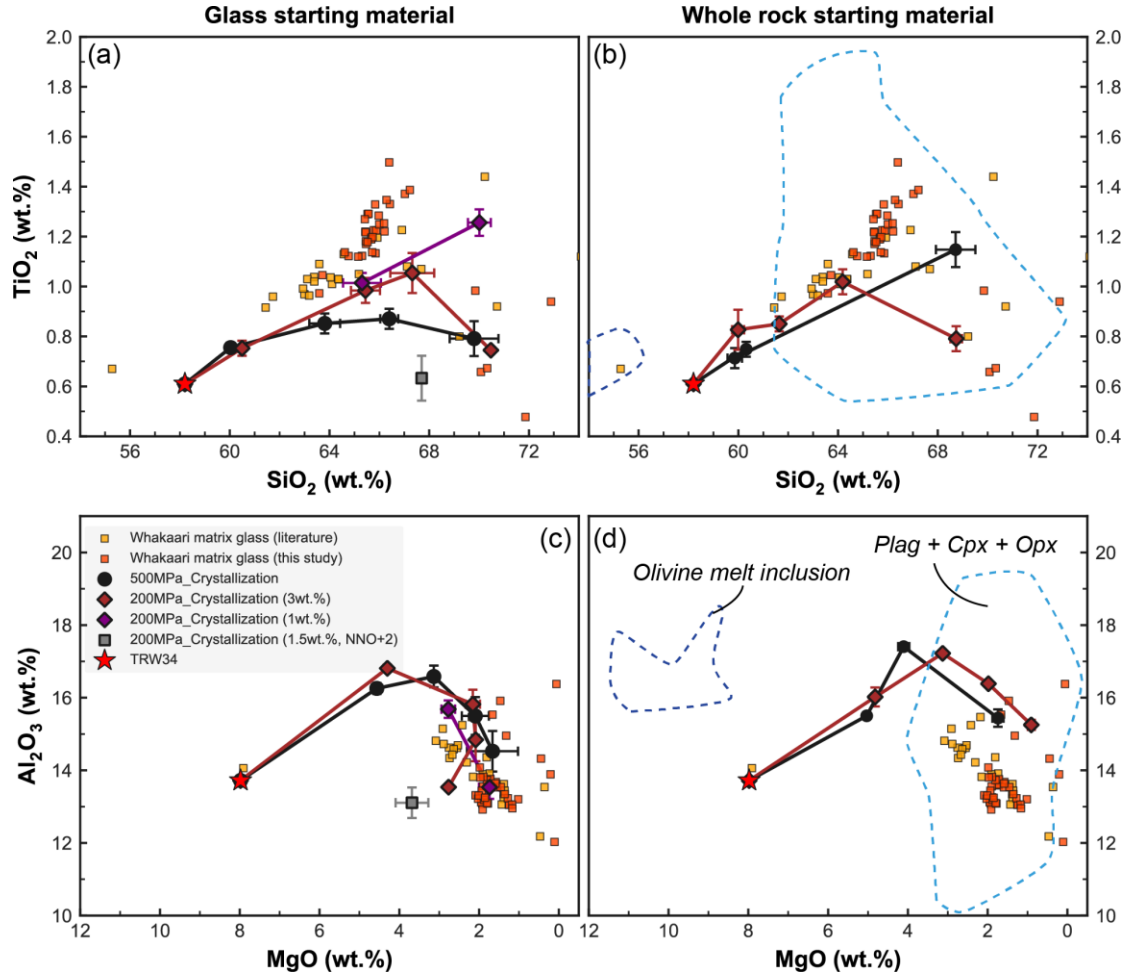


Figure SI 7. LLD results of water-undersaturated experiments using glass starting material (a and c) and unfused rock powder material (b and d). The large, yellow marker symbolize the “appropriate” experiments containing a phase assemblage of plagioclase + clinopyroxene + orthopyroxene. The natural matrix glass compositions and melt inclusions were sourced from previous studies (Heyworth, et al. 2007; Kilgour, et al. 2021; Mandon, et al. 2021) and this study.

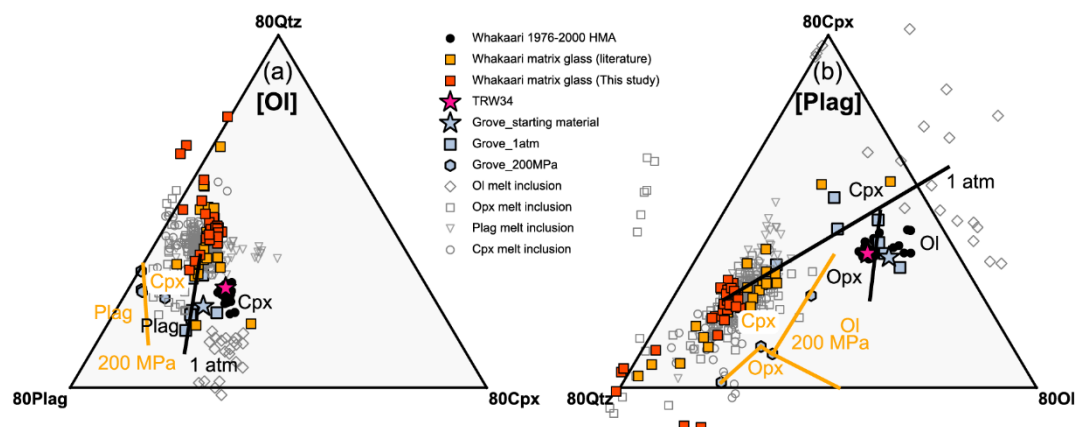


Figure SI 8. Compositions of water-undersaturated glasses on the quartz-plagioclase-clinopyroxene pseudoternary projections from olivine (a) and plagioclase (b). The experimental results were collected from Grove, et al. (2003).

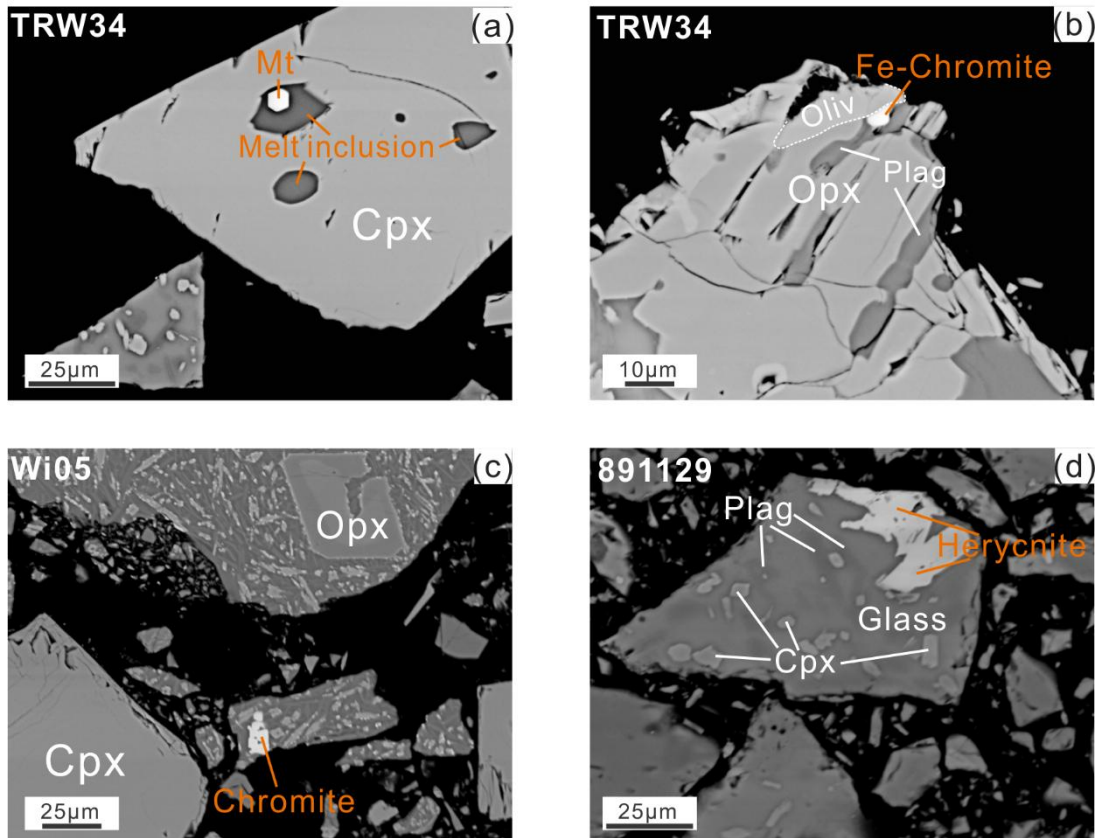


Figure SI 9. BSE images showing the appearance of magnetite (a), Fe-chromite (b), chromite (c), and hercynite (d). These minerals are found as small inclusions within melt inclusions, as larger phenocrysts, or as individual phenocrysts and glomerocrysts.

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