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FELLOWSHIP FINAL REPORT

Experiments and observations bearing on the origin of highly-fractionated granites and their critical metal endowment

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REPORT INFO

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ABSTRACT

An important paradigm guiding the exploration of granite-hosted critical metal deposits is the association between highly evolved rock compositions (i.e., aplites and pegmatites) and critical metal enrichment. To better understand this association, we have conducted melt extraction experiments at 400 and 1000 MPa and 750-900ºC to assess models of critical metal enrichment by extreme crystal fractionation. Two-stage experiments involved first melting a natural granodiorite starting material, then extracting the melt in the second stage using porous vitreous carbon. Results demonstrate crystal-melt separation, and therefore the feasibility of the method. Experiment glass compositions show significant chemical fractionation, and reveal insights into the origin of the compositional variation in natural granites.

1- Introduction

Mineral deposits of some of the critical metals essential to society are associated with the end stages of crystallization of the felsic magmas produced by melting of Earth's sedimentary crust. A unique attribute of these felsic magmatic systems is their extreme level of chemical fractionation. Key to determining how these deposits form, and where to find new ones, lies in understanding the processes by which such high levels of element enrichment can be achieved. To address this issue, laboratory experiments at controlled pressure, temperature and oxygen fugacity were done to simulate the crystallization process, in which felsic melts are segregated from their crystalline residue, and their compositions measured for major and trace elements.

2- Experimental details

The experimental method involves first partially melting a sample of natural peraluminous granodiorite in a graphite-lined Au, Au-Pd or Ag capsule at elevated P and T using a piston-cylinder (P-C) apparatus or internally-heated pressure vessel (IHPV). The premelted sample is then recovered, and placed along with vitreous carbon powder into a similar capsule arrangement as the initial step and subject to identical P-T conditions. Owing to its exceptional hardness, the vitreous carbon powder resists compaction, inducing a large pressure gradient for melt to flow from the partially-melted sample into the available pore space. The melt is thus trapped, and surrounded by a chemically-inert matrix, allowing for more accurate chemical analyses compared to experiments in which glass is adjacent to

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silicate minerals on which quench crystallization and microbeam impingement can occur.

Starting materials are derived from a natural granodiorite from the South Mountain Batholith (Nova Scotia, Canada) that was powdered in an agate ball mill, then seived to < 45 microns. This material was then fused at 1200ºC for 24 hours at 400 MPa, resulting in a homogeneous glass. Experiments employed both powdered rock and fused glass as starting materials. A total of eight two-stage experiments have been successfully completed: 1000 MPa, 750, 800 (x2), 850 and 900 ºC and 400 MPa, 800, 850 and 900 ºC. Most experiments were run for 72-120 hours in each step, for a total of 144-240 hours duration. Experimental run-products were extracted from the surrounding metal capsule, mounted in epoxy, then ground and polished. Run-product textural development and phase compositions were determined at ISTO using a Merlin compact ZEISS scanning electron microscope (SEM) equipped with a Bruker QUANTAX-XFlash 6 system for energy dispersive spectroscopy analysis. Following this, samples were extracted from the epoxy, and remounted in aluminum disks using indium for subsequent measurements of F, Cl and S by secondary ion mass spectrometry (SIMS). SIMS measurements were done at the Centre de Recherches Pétrographiques et Géochimiques (Vandoeuvre-lès-Nancy) using a Cameca IMS 1280-HR2 instrument employing methods described in Rose-Koga et al. (2020).

3- Results and discussion

Experimental results at 900, 850 and 800ºC (and both 400 and 1000 MPa) demonstrate that melt viscosity is sufficiently low, due to the presence of naturally-present fluxing agents (H_2O, F, alkalies), to drive melt penetration into the glassy carbon aggregate over the experiment timescale (Figure 1). The single experiment at 750ºC and 1000 MPa did not show any measureable melt migration, either because of low melt volume, or high viscosity (or both). Qualitatively, the proportion of glass in the melt trap increased with increasing experiment temperature. Glass (= quenched melt) compositions produced in experiments have higher SiO_2 concentrations than the granodiorite starting material (73-76 wt% vs 67 wt%, respectively), much lower Na_2O, CaO, TiO_2 and FeO, similar to slightly lower Al_2O_3 and F, and higher K_2O. These concentrations broadly plot along the major element compositional array defined by whole-rock compositions of more-evolved SMB samples. However, the experiment glasses have lower Na_2O and higher K_2O than the natural

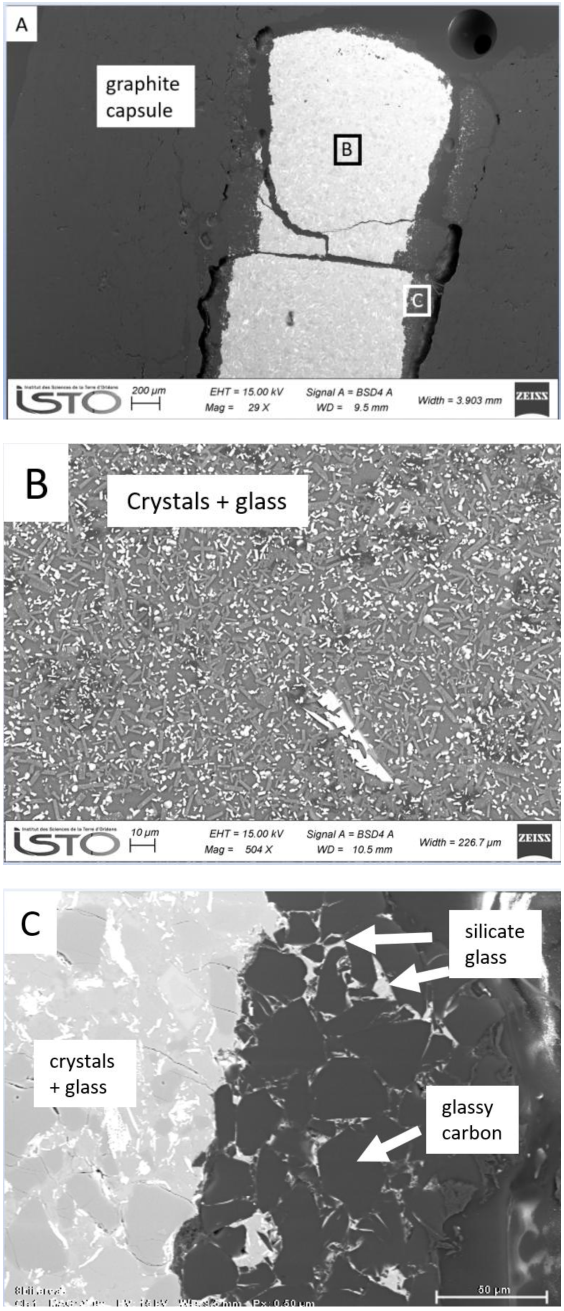

Figure 1. *Backscatter electron images of sectioned and polished sample Gran-in 8bii, which was equilbrated at 800ºC and 400 MPa for a total run time of 239 hours. In these images, the brightness is proportional to average atomic mass. A) Cross section of the whole sample, showing the sectioned "rock" and graphite capsule, with adhering vitreaous graphite rind. B) Close-up of area in B showing crystals (in order from dark to bright: quartz, glass, feldspar, biotite, orthopyroxene). C) Close-up of area in C with the presence of silicate glass (bright phase) within the interstitial space of the glassy carbon (dark phase).*

sample array. Mass balance for these elements indicates that the natural samples lie on a mixing array between experimental melt and coexisting feldspar, suggesting that the natural samples do not represent melt compositions, but instead a mixture of melt and accumulated feldspar. The F concentrations in run-product glasses range from 400-800 µg/g, with values increasing with temperature, approaching the initial concentration of ~760 µg/g. The shift in F concentrations likely reflect buffering by residual biotite, which maintains relatively low F in both the experimental glasses and during crystallization of the natural samples from the SMB. Planned trace element analyses of the trapped glass will provide additional constraints to support the crystal accumulation hypothesis, and also to provide estimates of the melt fraction in experiments.

4- Conclusions

The principal conclusions of this study are:

1) The feasibility of extracting high SiO_2 silicate melts from a granitic matrix is demonstrated in experiments done at 400 and 1000 MPa at temperature as low as 800ºC.

2) The success of this method allows for analysis of relatively low melt fractions to be measured for major and trace elements within an "inert" matrix, avoiding contamination from surrounding residual minerals.

3) The revelation that the natural sample whole-rock compositions do not correspond to melt compositions produced in experiments is perhaps not surprising. However, it will require some reconsideration of the experiment approach and interpretation of the natural data. It may be more reasonable to conduct experiments with progressively more evolved rock compositions (similar to the experiment melts), and monitor melt evolution in an iterative fashion.

5- Perspectives of future collaborations with the host laboratory

Given the initial success of the experiments and analyses performed during the research stay, further investigations will be continued by a PhD student under Brenan's supervision. The ISTO oversees an experimental capability with the IHPV apparatus to access moderate pressures that will be ideal for the ongoing work. Moreover, ISTO collaborators Koga and Rose-Koga have developed the analytical methods for volatile element analysis by SIMS that will be essential for characterizing the run-products from future experiments. Funding will be sought to support travel costs for the student to participate in the SIMS analysis, and perform additional experiments at ISTO.

6- Articles published in the framework of the fellowship

Brenan, J.M., Powell, M, Mihailoff, I., Rose-Koga, E.F, Koga, K.T. Experiments and observations bearing on the origin of highly-fractionated granites and their critical metal endowment. V.M. Goldschmidt Conference, July 2026.

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8- References

Rose-Koga, E.F., Koga, K.T., Devidal, J-L, Shimizu, N., Le Voyer, M., Dalou, C, and Dobeli, M. (2020) In-situ measurements of magmatic volatile elements, F, S, and Cl, by electron microprobe, secondary ion mass spectrometry, and heavy ion elastic recoil detection analysis. American Mineralogist, vol 105, pages 616–626.