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Critical metal enrichment in crustal melts: the role of metamorphic mica

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25 depletion in Be, Sn and Cs with increasing metamorphic grade. These distinctive trends
26 appear independent of tectonic setting (i.e. continental collision and crustal thinning). Our
27 dataset highlights the importance of higher-temperature melting ($>750^{\circ}\text{C}$), in particular,
28 biotite breakdown reactions, for the release of Li, Be, Sn, Cs and W into crustal melts.

29

30 **INTRODUCTION**

31 Most trace metal-enriched granites and pegmatites (e.g. Li-Cs-Ta) are highly peraluminous,
32 suggesting they formed via mica-melting reactions from metasedimentary protoliths (e.g.,
33 Patiño Douce and Harris, 1998; Cerny et al., 2012). During these reactions, trace elements
34 that are hosted in micas may be released into the melt (Dahl et al., 1993). Along with
35 enriched protolith compositions (Clemens et al., 2009; Romer and Kroner 2016; Wolf et al.,
36 2018), melting reactions may play an important role in generating crustal magmas that are
37 sufficiently enriched to eventually fuel formation of critical metal deposits at shallower levels
38 in the crust (e.g., Linnen et al., 2012; London, 2018). Both fluid-present and fluid-absent
39 reactions involving micas will generate melt, with muscovite melting at lower temperatures
40 and biotite melting at higher temperatures (e.g., Weinberg and Hasalová, 2016).

41 The concentrations of different elements in these micas, and their behaviour during
42 melting reactions, will influence the composition of the melt produced at different
43 temperatures (Wolf et al., 2018). For example, residuum (melanosomes) in migmatites that
44 formed by muscovite-melting reactions at $\sim 750^{\circ}\text{C}$ in the Iberian Massif (Spain) are enriched
45 in metals such as Li, Cs, Sn and W compared to the crystallised partial melt (leucosome).
46 However both leucosomes and melanosomes in slightly higher temperature (800°C)
47 migmatites generated by biotite-melting reactions record similar concentrations of these
48 elements (Wolf et al., 2018). These data suggested that higher temperature melting reactions
49 release critical metals (previously stored in restitic mineral phases) into the melt.

50 It is unclear if these trends and observations can be applied more generally to crustal
51 melting over a larger range of temperatures, different tectonic settings and different protolith
52 compositions. Additionally, direct comparisons between the compositions of leucosomes and
53 melanosomes are compromised by magmatic dilution, crystallisation, segregation or transport
54 (Wolf et al., 2018). The role of sub- and supra-solidus metamorphic reactions in
55 concentrating and releasing critical elements is also poorly constrained. These shortcomings
56 make it difficult to assess why granites that are generated by crustal melting have such
57 variable critical element concentrations. Here we present a new approach for placing
58 constraints on the partitioning of critical elements with increasing temperatures during crustal
59 melting. By measuring the concentrations of 57 elements *in-situ* in muscovite and biotite
60 from metapelites at different metamorphic grades, we have found systematic changes in the
61 concentrations of different critical metals as temperatures rise and dehydration reactions
62 initiate. This provides a new opportunity to constrain the input of different critical metals into
63 crustal melts at different stages of the metamorphic-melting cycle.

64

65 **METHODS and SAMPLE MATERIAL**

66 We analysed trace element concentrations by LA-ICP-MS in muscovite and biotite in 22
67 samples from three different metamorphic cross-sections covering sub-greenschist to
68 granulite-facies conditions. Supplementary material S1–4 contains sample descriptions,
69 mineral assemblages, analytical methods and photomicrographs showing laser spot locations.
70 The full dataset of major and trace elements, secondary standards and all element plots are
71 presented in Table S5.

72 We selected samples from two different tectonic settings. The samples from the
73 Himalayas were metamorphosed during continent-continent collision, whereas the samples

74 from the Ivrea Zone received their metamorphic imprint during post-orogenic collapse and
75 extension.

76 The eight samples labelled SK-10-XX were collected from a cross-section through the
77 5–10 km-thick Main Central Thrust in the Sikkim Himalaya, India. An inverted prograde
78 metamorphic sequence is well-developed across this ductile structure, from low-greenschist
79 (chlorite and biotite grades) at the lowest levels through to upper-amphibolite (kyanite and
80 sillimanite grade) at the highest levels. The sampling localities, sample descriptions and
81 pressure-temperature results are documented in detail in Mottram et al. (2014). This sample
82 set covers the onset of muscovite melting.

83 The four samples labelled N-XX were collected from the hanging wall of the Main
84 Central Thrust in the Langtang Himalaya, Nepal, from higher structural levels and similar
85 metamorphic grades to the highest-grade Sikkim samples. These samples were
86 metamorphosed at kyanite to sillimanite grade, with coarser leucocratic streaks and patches
87 interpreted as leucosome in samples N5 and N11, across the muscovite-out isograd.

88 The ten IZ-4XX samples were collected from the Val Strona di Omega, in the Ivrea
89 Zone of the European Alps, Italy. They were metamorphosed at upper amphibolite to
90 granulite facies conditions, and show both muscovite and biotite melting reactions. Sampling
91 localities, sample descriptions and pressure-temperature results are documented in detail in
92 Kunz et al. (2018) and Kunz and White (2019).

93

94 **RESULTS**

95 The within-sample average concentrations of 26 elements in biotite and muscovite
96 shown in Fig. 1 are normalised to Bulk Silicate Earth concentrations (Palme and O'Neill,
97 2013) and plotted in order of increasing bulk partitioning behaviour (Jenner, 2017). Both
98 biotite and muscovite record concentration variability within and between samples, and

99 record enrichments and depletions in the concentrations of some critical elements (e.g., Sn,
100 In, Cs) compared to the composition of the bulk continental crust. We have not observed any
101 systematic patterns in element concentrations with petrographic location. The lowest-
102 temperature samples SK12 and SK15 were too fine grained to acquire “clean” analyses of
103 individual micas and the data are not presented.

104 Elemental concentrations in biotite vary more systematically than in muscovite
105 particularly in the high-temperature samples from the Ivrea Zone compared to the samples
106 from the lower grade Sikkim section (Figs. 2 and 3; Tab. S1). Specifically, there are
107 systematic changes of up to two orders-of-magnitude in the concentrations of Li, Be, F, V,
108 Co, Nb, In, Sn, Cs, Ta, and W with increasing metamorphic grade that are all greater than
109 analytical uncertainty; we have therefore focussed attention on these eleven elements.
110 Additionally, there are clear differences in concentration between the different field areas that
111 are most likely related to different bulk rock abundances of these elements.

112 Typically, biotite contains higher concentrations of Li, F, V, Co, Nb, Cs and Ta than
113 muscovite, whereas muscovite systematically hosts higher concentrations of Be, In, Sn and
114 W (Figs. 2 and 3). Concentrations of most elements in muscovite generally show little to no
115 systematic change with increasing metamorphic temperature except in the Langtang samples
116 which show increases in In and W concentrations and decreases in Li, Be and Cs
117 concentrations at supra-solidus temperatures.

118 Sub-solidus biotites in the Sikkim samples show no systematic changes in trace
119 element concentration. Above solidus temperatures, biotite in the Langtang and Ivrea Zone
120 samples show a gradual decrease in Li, Cs, Be and Sn concentrations and an increase in V
121 and Co concentrations with increasing temperature (Fig. 2). Concentrations of In and W
122 increase in biotite as temperatures increase to ~850 and 750°C respectively; concentrations
123 then decrease with increasing metamorphic grade (Fig. 2). Ta and Nb concentrations remain

124 fairly constant in biotite with increasing metamorphic temperature, except in the two highest-
125 grade samples that have abundant rutile (Fig. 3).

126

127 **DISCUSSION**

128 **Controls on trace element concentrations in sub-and supra-solidus phases**

129 Our data show that Li, Cs and Ta (commonly enriched together in LCT-pegmatites)
130 are predominantly hosted in biotite, while Be, Sn and W are predominantly hosted in
131 muscovite (c.f. Dutrow et al., 1986; Dahl et al., 1993; Bea et al., 1994; Yang and Rivers,
132 2000; Tischendorf et al., 2001; Neiva et al., 2002; Van Lichtervelde et al., 2008; Simons et
133 al., 2017). In the sub-solidus samples, concentrations of most critical metals measured in both
134 micas are variable and show no systematic changes. For example in the Sikkim samples
135 SK19–SK22 (which equilibrated at similar temperatures within uncertainty of 650–700°C)
136 we observe an order-of-magnitude difference in Li and Ta concentrations in both micas. We
137 therefore interpret this as either reflecting (i) differences in bulk composition and therefore
138 different initial trace element abundance (Romer and Kroner, 2016), or (ii) differences in the
139 continuity of this metamorphic sequence due to ductile deformation.

140 Partitioning of metals between biotite and muscovite can be explained by differences
141 in site and interlayer site preferences of these elements (Dahl et al., 1993). The apparent
142 increase in concentration of V, Co and In in biotite with increasing metamorphic grade could
143 be an effect of decreasing modal abundance of biotite. However, the concentration increase
144 starts at the onset of muscovite melting when the modal abundance of biotite is still
145 increasing. Therefore we interpret the observed trends for these elements as resulting from
146 differential partitioning in the presence of a melt phase. Conversely, the increase of W in
147 muscovite coincides with the decrease in modal abundance of muscovite, suggesting that this
148 might be a modal abundance effect.

149 The partitioning of critical metals between micas and other metamorphic minerals
150 may also be important at sub- and supra-solidus conditions. For example Li is hosted in
151 tourmaline and staurolite (e.g., Dutrow et al., 1986; London, 2011), Be in cordierite (Evensen
152 and London, 2002) and Sn and W in rutile, ilmenite and titanite (e.g., Zack et al., 2002;
153 Klemme et al., 2006). However, there are currently only sparse datasets for many critical
154 metals for solid/solid partitioning at high-temperature metamorphic conditions (e.g.,
155 Icenhower and London, 1995; Dutrow et al., 1986) making a quantification of partitioning
156 difficult to assess.

157

158 **Implications for the formation of enriched granites and pegmatites**

159 Our dataset demonstrates the importance of melting reactions involving biotite for the
160 concentration (in the melanosome) and release (into the leucosome) of a number of critical
161 metals, specifically Li, Be, Sn, Cs and W. Biotite is widely present in a variety of bulk-rock
162 compositions and is a known host of critical metals (e.g. Dahl et al., 1993; Evensen and
163 London, 2002). Our data show that concentrations of these elements in metapelitic biotite
164 remains approximately constant until the onset of biotite dehydration melting reactions, when
165 these elements are released into the melt. Our findings are in agreement with bulk rock data
166 from the Ivrea Zone (Bea and Montero, 1999) that show a similar depletion in bulk rock for
167 Li, Be, Cs and an increase in V with metamorphic grade.

168 This scenario of high-temperature biotite melting as the key for the enrichment of Li-
169 Cs-Ta in the melt is in conflict with previous hypothesis of LCT-enriched pegmatites being
170 generated by minimum-temperature melting of muscovite (e.g. Cerny, 1991; Romer and
171 Kroner, 2015, 2016). Instead, our data suggest that enrichment of LCT-enriched granitic
172 magmas most likely occurs during low-volume biotite melting of a mostly muscovite-absent
173 assemblage, a scenario that has also been argued for granitic Sn enrichment (Wolf et al.,

174 2018) and the enrichment patterns of successive granite suites of the Cornubian Batholith in
175 Cornwall, UK (Simons et al., 2017).

176 Biotite provides the source of these elements as well as a mechanism for allowing
177 more effective melt and metal transport. Crustal melts are viscous (Rutter and Neumann,
178 1995) and therefore difficult to mobilise without additional help, e.g. from pervasive ductile
179 deformation or the addition of fluxing elements such as F, which lower melt viscosity
180 (Dingwell et al., 1996). Additionally, F allows metals to be transported in the melt more
181 readily through complexing (London, 1987). High-temperature biotite can host F at weight-%
182 levels (Finch and Tomkins, 2016); in our samples the Sikkim and Ivrea Zone biotites contain
183 higher F concentrations than muscovite (0.13–1.6 wt% F compared to 0.04–0.16 wt% F
184 respectively; Fig 2).

185 Sn and W enrichments in granites have been linked to high-temperature melting of a
186 biotite-rich source (Romer and Kroner, 2016; Wolf et al., 2018). Our data show that, in
187 general, concentrations of Sn and W are higher in muscovite than in co-hosted biotite.
188 Concentrations of W in muscovite increase slightly with metamorphic grade; we do not
189 observe systematic changes of Sn concentrations in muscovite with metamorphic grade. In
190 contrast, biotite from the Ivrea Zone and Langtang samples show clear decreases in Sn and W
191 concentrations with increasing metamorphic grade. This observation supports the theory of
192 higher-temperature melting being critical for melt enrichment (Wolf et al., 2018; Michaud et
193 al., 2021). However we do not observe sequestering of Sn into biotite across the muscovite-
194 melting temperature interval, as previously suggested (Wolf et al., 2018).

195 Further investigation into different biotite melting reactions, including those which
196 are fluid-absent or fluid-present, are needed to understand the conditions/reactions under
197 which critical metal release is optimised and therefore provides the highest potential for melt
198 enrichment. Biotite is also a key constituent in non-pelitic metasediments (e.g. meta-

199 graywackes) that may also be source rocks for enriched granites and pegmatites. Focus on
200 biotite-melting reactions across a range of metasedimentary bulk compositions is therefore
201 key to constraining how and under which conditions critical metal enriched granites and
202 pegmatites form.

203

204 **CONCLUSIONS**

205 Micas are the main reactant during dehydration melting of metasediments. As micas host
206 significant concentrations of many critical metals and H₂O, their breakdown facilitates the
207 transfer of these metals from metamorphosed country rocks into melts. Our data show that
208 prograde sub-solidus metamorphic reactions do not lead to systematic changes in the
209 concentrations of different critical metals in micas. Upon crossing the muscovite dehydration
210 solidus, both muscovite and biotite concentration for Cs and Be start to decrease, while W
211 concentration increase. Once conditions for biotite dehydration melting are reached,
212 concentrations of Li, Be, Sn, Cs, and W in biotite decrease markedly as they are released into
213 the melt.

214

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218 sections from the Sikkim section. We thank three anonymous reviewers for their constructive
219 comments and Gerald Dickens for editorial handling.

220

221 **FIGURE CAPTIONS**

222 **Fig. 1** Average trace element concentration per sample of (a) muscovite (b) biotite.
223 Normalised to Bulk Silicate Earth (Palme and O'Neill, 2013); black line is the composition of

224 the bulk continental crust (Rudnick and Gao, 2003); element ordering is based on bulk
225 partitioning during differentiation of mid-ocean ridge basalts (Jenner, 2017).

226 **Fig. 2** Concentrations of Li, F, Cs, Be, Sn, W, V, Co and In vs. peak metamorphic
227 temperature. Grey shaded areas represent muscovite- (light grey) and biotite-melting (dark
228 grey) ranges. Trends in: biotite (solid arrow), muscovite (dashed arrow) data.

229 **Fig. 3** Concentration of Nb and Ta vs. peak metamorphic temperature. Grey shaded areas
230 represent muscovite- (light grey) and biotite-melting (dark grey) ranges. Trends in: biotite
231 (solid arrow), muscovite (dashed arrow) data.

232

233 SUPPLEMENTARY MATERIAL

234 **APPENDIX 1.** Sample description

235 **APPENDIX 2.** Table with mineral assemblages

236 **APPENDIX 3.** Detailed method description for LA-ICP-MS analysis

237 **APPENDIX 4.** LA-ICP-MS spot location

238 **APPENDIX 5.** Major and trace element data table for samples and secondary standard

239

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Figure 1

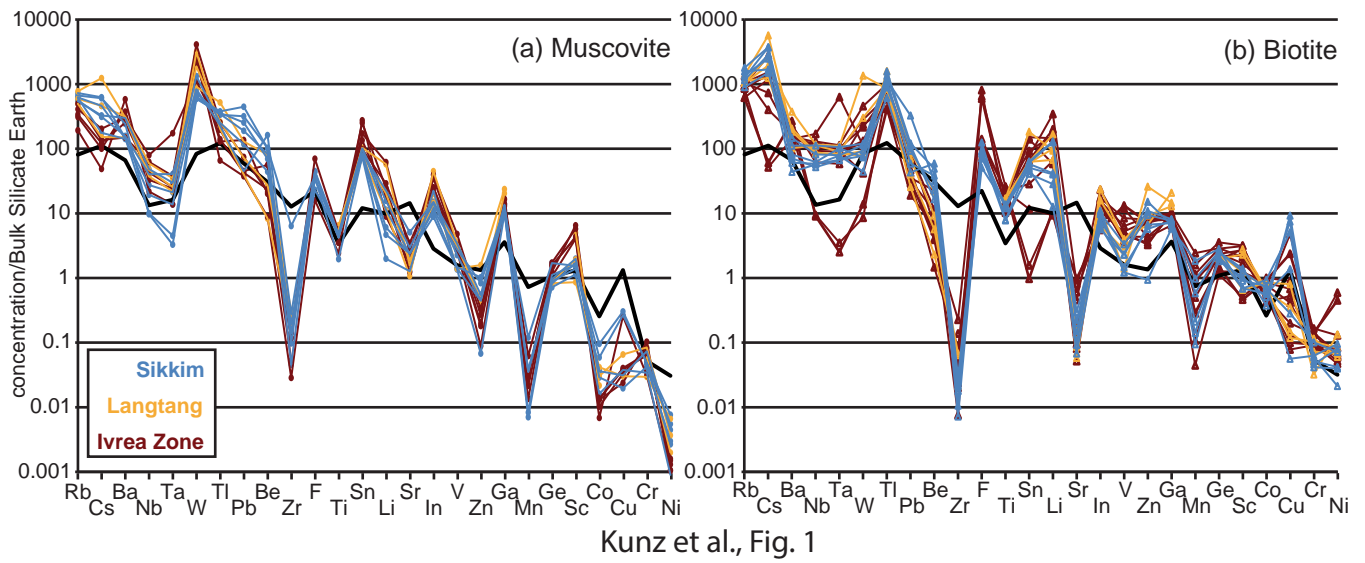
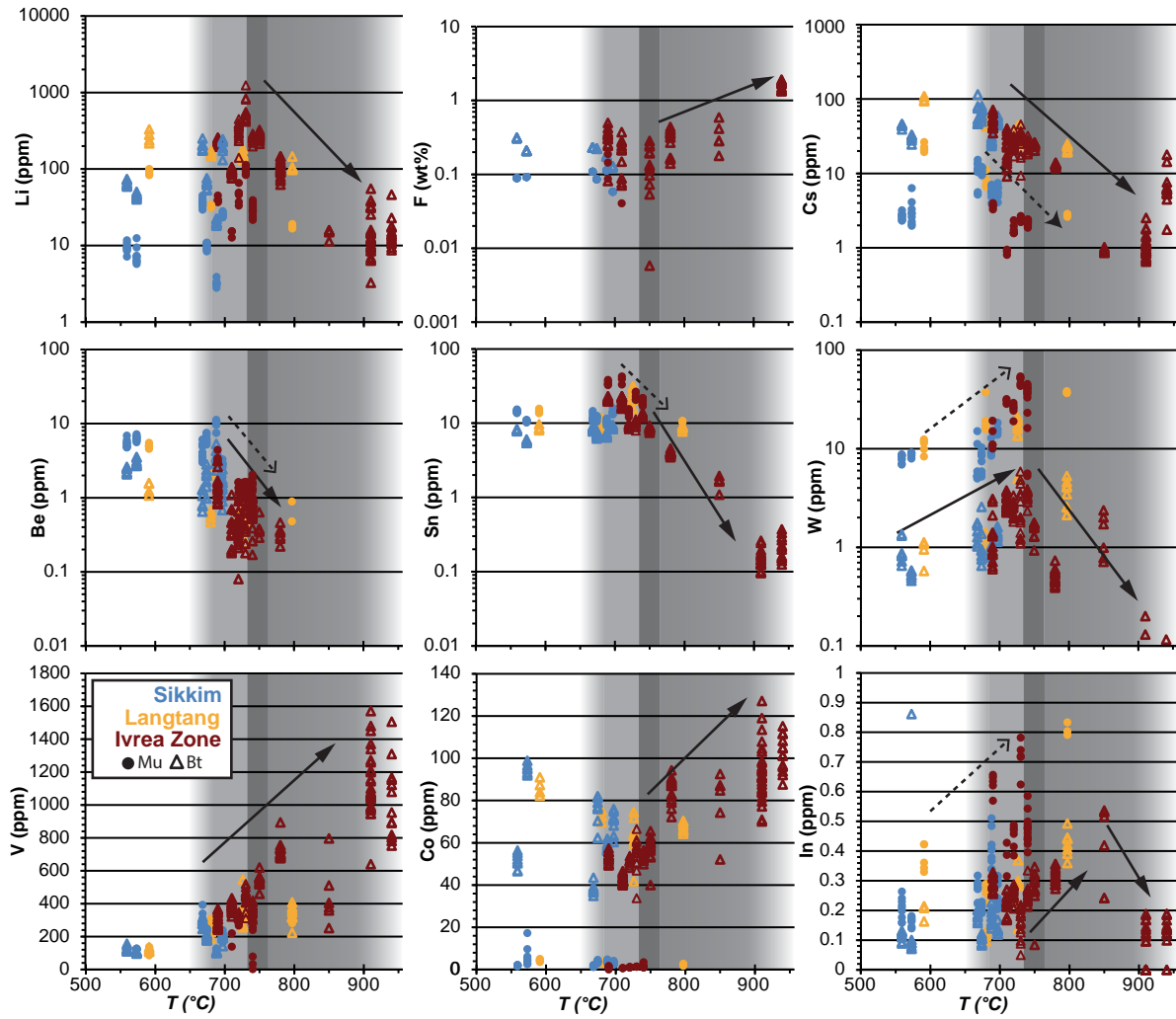
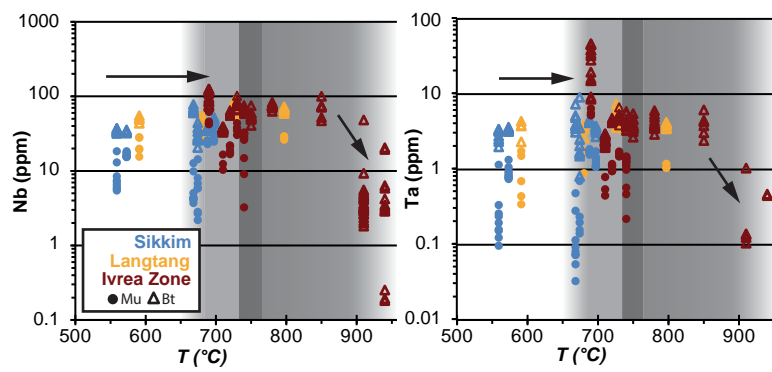


Figure 2



Kunz et al., Figure 2

Figure 3



Kunz et al., Figure 3