Compositional Variation of Amphiboles During Magma Mixing: A Case Study of Huangyangshan A-Type Granite in Kalamaili Metallogenic Belt, East Junggar, China

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The Huangyangshan A-type granitic pluton, distributed along the thrust fault in the Kalamaili region of East Junggar, Xinjiang, China, consists of alkaline granite containing abundant dioritic enclaves that formed via magma mixing. Both the host granite and the enclaves contain sodic amphiboles. The textural evidence indicates that amphiboles crystallized as a magmatic phase in both units. We determined major and trace element contents of amphiboles from both units to investigate the compositional variation of the amphiboles during the magma mixing process. The results show that cations of W- and C-site are influenced by chemical compositions of the magma whereas cations of A-, B- and T-site and Al^{3+} are controlled by crystal structure. Therefore, the variations of W- and C-site cations can reflect magma evolution. The core and rim of the amphiboles show similar trace element patterns, which also suggests that the amphiboles are late-stage phases. Furthermore, the amphibole-only thermometers yield reasonable estimates that are consistent with petrographic evidence. However, thermometers based on partition coefficients and all the currently available amphibole-based barometers that rely on Al contents or D_{Al} cannot be applied to Fe-rich and Al-poor amphiboles.

Keywords: amphibole, A-type granite, dioritic enclave, magma mixing, Sn mineralization, Huangyangshan

INTRODUCTION

Magma mixing is a common magmatic process during formation of granitoids (Kumar et al., 2004; Liu et al., 2013; Xiong et al., 2020). Previous studies suggest that magma mixing can be important for formation of critical metal deposits (e.g., Ma et al., 2013). If mafic magma injects into felsic magma during magma mixing, microgranular mafic enclaves (MMEs) would likely form. During this process, amphiboles are usually the major rock-forming minerals.
The amphibole-group are complex in chemical composition but share a general chemical formula expressed as AB\(_2\)C\(_6\)T\(_4\)O\(_{22}\)W\(_2\), where A, B, C, and T represent different crystal sites (Leake et al., 1997). The T site is commonly occupied by Si\(^{4+}\), Al\(^{3+}\), Ti\(^{4+}\), and the like and is tetrahedrally coordinated, forming a double chain of tetrahedra. The A site occurs in the center of space between the double-chains of the structure. The C site is octahedrally coordinated and occupied by cations including Mg\(^{2+}\), Fe\(^{2+}\), Mn\(^{2+}\), Al\(^{3+}\), Fe\(^{3+}\), Mn\(^{3+}\), Ti\(^{4+}\), etc. Cations on the B-site situate at the periphery of the strip of the octahedra C-cations occupy. Anions including OH\(^-\), F\(^-\), Cl\(^-\), and O\(^{2-}\) dominate the W-site. This complexity gives rise to a complicated classification for amphiboles (Hawthorne et al., 2012). Owing to the complexity, amphiboles are capable of accommodating a variety of elements with variable ionic charges and radii, including alkali elements, rare earth elements (REE), high field strength elements (HFSE), and large ion lithophile elements (LILE) (Siegel et al., 2017). Therefore, amphiboles are sensitive to chemical compositions of the crystallizing melts and may record some processes during magma evolution.

The Huangyangshan A-type granite located in the Kalamaili metallogenic belt, Xinjiang, is alkaline and contains abundant mafic microgranular enclaves (MMEs) (Yang et al., 2011). The reported ages for both units were 311–305 Ma and 300 ± 6 Ma, respectively (Su et al., 2008; Yang et al., 2011). The consistent ages along with the gradational contact between the host granite and MMEs and the presence of acicular apatite indicate magma mixing during the formation of the Huangyangshan A-type granite (Yang et al., 2009, 2010, 2011). In both units, Na-rich amphiboles are a common mineral phase. Therefore, in this paper, we choose amphiboles from the MMEs and their host granite to explore the compositional variations during magma mixing. Also, due to their sensitivity, amphiboles are potential geo-thermometer and barometer. For this reason, we tested whether they could yield proper estimates on the crystallization temperature and pressure of the amphiboles in the Huangyangshan A-type granite.

**GEOLOGICAL SETTING**

The Huangyangshan A-type granite, distributed along the Kalamaili region in East Junggar, Xinjiang (Figure 1). This region represents the northern margin of East Junggar, part of the Junggar terrane that belongs to the Central Asian Orogenic Belt (CAOB) (Sengor et al., 1993; Xiao et al., 2003). The NW-SE trending Huangyangshan A-type granite covers an area of ~230 km\(^2\) and is intruded into the early Carboniferous strata, showing sharp contact (Yang et al., 2011). Contact metamorphism has also been observed, which implies shallow depth for emplacement (Yang et al., 2011). The granites consist of 40–55% alkali-feldspar, 5–15% plagioclase, 20–30% quartz, and 5–10% mafic minerals (biotite and/or amphibole) and accessory minerals (<1%) including apatite, zircon, and fluorite. Based on the content of mafic minerals, four main phases can be distinguished. A great number of MMEs with variable sizes are present in the granite. The MMEs exhibit sharp to diffuse contact with their host granite and chilled margins or biotite-rich reaction rims are locally present (Figure 2), indicating hybridization between MMEs and host granites (e.g., Yang et al., 2010). These MMEs are dioritic in composition and composed of 5–15% amphibole, 20–30% alkali-feldspar, 30–40% plagioclase, 5% biotite, and <1% acicular apatite.

The Kalamaili region defines an important metallogenic belt in the East Junggar terrane. According to previous studies, Au and Cu mineralization are mainly distributed at the margins of calc-alkaline granitoids, whereas tin mineralization mainly occurs at the margins of alkaline granitoids including the Huangyangshan granite (Yang et al., 2011).

**ANALYTICAL METHODS**

**Petrographic and Electron-Microprobe Analyses**

Doubly polished thin sections were made for rock samples collected from the Huangyangshan granite and the hosted MMEs. Petrographic observations were performed using optical microscopy and scanning electron microscope (SEM). All polished thin sections were carbon-coated and major element contents of amphiboles were determined using a JEOL JXA-8100 electron microprobe (Tokyo, Japan) at the Laboratory of Mineralization and Dynamics, Chang’an University. During EPMA analysis, the accelerating voltage, sample current, and beam diameter were 15 kV, 10 nA, and 5 µm, respectively. Fourteen elements in total were analyzed. The standards for calibrating element contents include: synthetic MnTiO\(_3\) (for Mn K\(^α\)), plagioclase (Na K\(^α\), Ca K\(^α\), and Al K\(^α\)), magnetite (Fe K\(^α\)), sandine (K K\(^α\)), zirconium metal (Zr K\(^α\)), olivine (Mg K\(^α\) and Si K\(^α\)), synthetic Nb-Ta alloy (Nb L\(_\alpha\) and Ta L\(_\alpha\)), synthetic rutile (Ti K\(^α\)), and fluorite (F K\(^α\)). The analytical precision (expressed as relative standard deviation) for major element oxide contents >1 wt.% are better than 3%. Summary of EPMA data for amphiboles is given in Table 1 and the complete EPMA dataset for amphiboles is presented in Supplementary Material S1.

**Laser-Ablation ICP-MS Analysis**

Trace elements of amphiboles were analyzed using a Photon Machines 193 nm excimer laser ablation system coupled with an Agilent 7700 ICP-MS (Santa Clara, CA, United States) at the Laboratory of Mineralization and Dynamics, Chang’an University. The analyzed elemental isotopes included \(^{7}\)Li, \(^{27}\)Al, \(^{29}\)Si, \(^{35}\)Cl, \(^{45}\)Sc, \(^{49}\)Ti, \(^{55}\)Mn, \(^{63}\)Cu, \(^{66}\)Zn, \(^{69}\)Ga, \(^{85}\)Rb, \(^{88}\)Sr, \(^{89}\)Y, \(^{90}\)Zr, \(^{93}\)Nb, \(^{118}\)Sn, \(^{137}\)Ba, \(^{152}\)La, \(^{146}\)Ce, \(^{141}\)Pr, \(^{146}\)Nd, \(^{147}\)Sm, \(^{153}\)Eu, \(^{157}\)Gd, \(^{159}\)Tb, \(^{161}\)Dy, \(^{165}\)Ho, \(^{166}\)Er, \(^{165}\)Tm, \(^{172}\)Yb, \(^{175}\)Lu, \(^{177}\)Hf, \(^{181}\)Ta, \(^{206}\)Pb, \(^{232}\)Th, and \(^{238}\)U. Helium gas was used as the carrier gas. The diameter, energy density, and repetition rate of the laser beam were 35 \(\mu\)m, 5.9 J/cm\(^2\), and 8 Hz, respectively. For each analysis, signals on the gas background and samples or standards were collected for 10 s and 40 s, respectively. NIST SRM 610 and NIST SRM 612 were used as...
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FIGURE 1 | Geological map of the Huangyangshan pluton (adapted from Yang et al., 2011). 1-Heshanshitou Group; 2-plagioclase granite; 3-medium grained biotite alkali feldspar granite; 4-medium grained hornblende alkali feldspar granite; 5-medium grained arfvedsonite alkali feldspar granite; 6-medium to fine grained arfvedsonite alkali feldspar granite; 7-fine grained biotite alkali feldspar granite; 8-fine grained mixed granite; 9-diabase dyke; 10-diorite dyke; 11-granite porphyry dyke; 12-pulsating contact; 13-beveling contact; 14-intrusive contact; 15-structural belt boundary; 16-fault and occurrence; 17 and 18-number of structure belt; I1-Altai structure belt; I2-Almantai structure belt; II1-Kalamali structure belt; II2-Jiangjunmiao structure belt; I1y-Irtysh Fault; IIy-Almantai Fault; IIIy-Kalamali Fault.

the primary and secondary external standards, respectively. The LA-ICP-MS data were processed using Iolite v.3.71 (Paton et al., 2011) and the Si concentrations of the amphiboles obtained by EMPA was the internal standard for calculating trace element concentrations. The accuracy based on the test on NIST SRM 612 (expressed as relative difference between measured and reference values) was < 15% for most analyzed elements. The precision test on NIST SRM 612 indicated that the precision (expressed as relative standard deviation) was better than 10% for most analyzed elements except for Ti that showed analytical precision of 15.6%. The results are summarized in Table 2 and the complete dataset of trace element concentrations is provided in Supplementary Material S2.

RESULTS

Petrography

The granite mainly exhibits medium- to fine-grained texture and vermicular texture and mantling texture are locally present. The major rock-forming minerals are alkaline feldspar (mainly perthite) and quartz, with minor plagioclase (An23 to An29,
FIGURE 2 | Field photos of the enclaves in the Huangyangshan alkaline granite. (a–c) Both sharp and transitional boundaries exist indicating the rheology of magma during magma mixing. (d) Alkali feldspar of the host granites crosscuts the boundaries of the MME.

TABLE 1 | Major element contents of amphiboles from the host granite and enclaves.

| Amphibole | Granites Mean (n = 13) | Enclaves Mean (n = 2) | Amphibole | Granites Mean (n = 13) | Enclaves Mean (n = 2) |
|-----------|------------------------|----------------------|-----------|------------------------|----------------------|
| Na$_2$O   | 7.00                   | 9.17                 | Al$^{3+}$ | 0.16                   | 0.05                 |
| FeO       | 31.11                  | 27.07                | Fe$^{2+}$ | 0.14                   | 0.03                 |
| K$_2$O    | 1.44                   | 0.38                 | Mn$^{2+}$ | 0.05                   | 0.04                 |
| F         | 0.79                   | 0.27                 | Fe$^{2+}$ | 4.17                   | 3.64                 |
| MnO       | 0.35                   | 0.30                 | Mg$^{2+}$ | 0.04                   | 0.16                 |
| CaO       | 3.00                   | 4.86                 | C-site    | 4.75                   | 4.11                 |
| SiO$_2$   | 48.83                  | 50.90                | Mn$^{2+}$ | 0.00                   | -                    |
| Ti$_2$O$_5$| 0.04                  | 0.04                 | Fe$^{2+}$ | 0.00                   | -                    |
| ZrO$_2$   | 0.07                   | 0.57                 | Ca$^{2+}$ | 0.54                   | 0.84                 |
| Al$_2$O$_3$| 1.11                  | 0.25                 | Na$^+$    | 1.45                   | 1.16                 |
| SnO$_2$   | 0.00                   | 0.01                 | B-site    | 2.00                   | 2.00                 |
| MgO       | 0.16                   | 0.65                 | Na$^+$    | 0.79                   | 1.72                 |
| Nb$_2$O$_5$| 0.01                  | 0.03                 | K$^+$     | 0.30                   | 0.08                 |
| TiO$_2$   | 1.63                   | 1.28                 | A-site    | 1.09                   | 1.80                 |
| Total     | 95.47                  | 95.76                | A-site    | 1.09                   | 1.56                 |
| Si        | 8.08                   | 8.24                 | F$^-$     | 0.53                   | 0.14                 |
| Al        | 0.05                   | –                    | O$_2^-$   | 0.37                   | 0.30                 |
| T-site    | 8.15                   | 8.24                 | W-site    | 2.00                   | 2.00                 |
| Ti        | 0.18                   | 0.15                 | Fe$^+$    | 0.97                   | 0.99                 |
| Zr        | 0.01                   | 0.05                 |           |                        |                      |

Fe$^+$ = Fe$^{2+}$/(Fe$^{2+}$ + Fe$^{3+}$). The apfu values are calculated based on 24 oxygen atoms.
oligoclase), biotite, and arfvedsonite (Figure 3A). Most aegirine occurs along the boundaries of amphibole grains in the host granite, indicating that arfvedsonite crystallizes earlier than aegirine (Figure 3A), which may also imply the magma evolved toward more oxidized trend (Scaillet and MacDonald, 2001). The accessory minerals mainly include zircon, fluorite, and apatite. The presence of fluorite indicates that the granitic magma is likely F-saturated.

The MMEs mainly show subhedral granular texture and porphyritic texture and mantling texture locally occurs. Compared with the granite, the MMEs contain relatively abundant plagioclase and mafic minerals (mainly alkaline amphibole and aegirine), whereas the amounts of quartz and alkali feldspar are less than those of the host granite. The accessory minerals in the MMEs include titanite, apatite, and zircon. Apatite shows an acicular habit (Figure 3B), indicating a quench process. The amphibole crystallizes in a shape with excessively long c-axis and is semi-oriented (Figures 3B,C), suggesting that MMEs remained their rheology to some extent during formation. Typical transitional zone (diffuse contact) between the MMEs and the host granite can be observed under microscope (Figure 3D). It is also noted that quartz is mantled by a mineral assemblage consisting of aegirine and titanite (Figures 3C, 4a,b). This texture is similar to quartz ocellus texture described in previous studies (e.g., Baxter and Feely, 2002; Renna et al., 2006). Back-scattered electron imaging shows that the early arfvedsonite is enclosed by the aegirine rimming the quartz xenocryst (Figure 4b). The replacement of arfvedsonite by aegirine can also be observed (Figure 4c), indicating that the aegirine postdates the arfvedsonite. Other mafic minerals including biotite are only present in minor quantities. Zircon as an accessory phase is often subject to hydrothermal alteration (Figure 4d).

### Major Element Compositions of Amphiboles and Formula Assignments

The average major and trace element compositions of amphiboles are given in Tables 1, 2, respectively. Major and trace element compositions of the host granite and MMEs from Su et al. (2008) and Yang et al. (2011) are also listed in Table 3 for comparison (complete dataset of bulk rock is given in Supplementary Materials S3, S4).

The K content of amphiboles from the host granites ranges from 0.275 to 0.371 apfu and shows a dichotomy that one group exhibits higher value (0.352–0.371 apfu) whereas the other lies...
below the median value (~ 0.3125 apfu), ranging from 0.215 to 0.304 apfu. The K content of amphiboles from the MMEs lies in the above-mentioned two ranges respectively. The Na content of amphiboles from both the granite and MMEs vary in a narrow range (2.185–2.488 apfu) except two analyses showing abnormally high values (0.215–0.3125 apfu). The Na content of amphiboles from the host granite ranges from 0.235–0.607 apfu except one analysis showing abnormally high value (1.039 apfu), whereas the Ca content of amphibole from the MMEs has higher Ti and Al than MME-host amphiboles (0.017–0.620 apfu, 0.044–0.084 apfu, respectively). Ti and Al show opposite patterns that amphiboles from granites have higher Mg, Si and Mn content than MME-host amphiboles. The Mg, Si and Mn content of amphibole from the MMEs is relatively low (~ 0.004–0.120 apfu, 7.81–8.351 apfu, 0.043–0.25 apfu). The Mg, Si and Mn content of amphibole from granites have constrained Mg, Si and Mn content to be lower than MME-host amphiboles (0.017–0.211 apfu, 0.044–0.084 apfu, respectively). Fe<sup>3+</sup> is calculated based on the algorithm provided by Locock (2014) and both granite-host and MME-host amphiboles share a similar range of Fe<sup>3+</sup> (0.0–0.620 apfu) and Fe<sup>2+</sup> (3.669–4.423 apfu). Fluorine content ranges from 0.2–0.9 apfu and Cl content is below 0.1 apfu except HY5–9 which has 1.4 apfu Cl. The abnormally high value of HY5–9 may be ascribed to Cl-rich inclusions.

After analyzing the major elements of amphiboles, the chemical formula of amphibole can be calculated. Generally, the amphiboles-group minerals have a structure formula as AB₂C₅T₈O₂₂W₂, and the specific formula can be calculated using the spreadsheet provided by Locock (2014). The results of the formula assignment are listed in Table 4 (the detailed formulae assignment is given in Supplementary Material S5). In our case, the A-site of amphiboles is occupied by Na<sup>+</sup>, K<sup>+</sup> and vacancies; Ca<sup>++</sup> and vacancies; Mg<sup>++</sup> and vacancies; Fe<sup>3+</sup> and Fe<sup>2+</sup> and vacancies; Al and vacancies; Ti and vacancies; W<sup>6+</sup> and vacancies; T<sup>4+</sup> and vacancies; Si and vacancies; O<sup>-</sup> and vacancies; Cl and vacancies; F and vacancies; Fe<sup>2+</sup> and Fe<sup>3+</sup> and vacancies; Mg<sup>++</sup> and vacancies; Ca<sup>++</sup> and vacancies; Na<sup>+</sup> and vacancies; K<sup>+</sup> and vacancies; Sr and vacancies; Y and vacancies; La and vacancies; Ce and vacancies; Pr and vacancies; Nd and vacancies; Sm and vacancies; Eu and vacancies; Gd and vacancies; Tb and vacancies; Dy and vacancies; Ho and vacancies; Er and vacancies; Tm and vacancies; Yb and vacancies; Lu and vacancies; Hf and vacancies; Ta and vacancies; Pb and vacancies; Bi and vacancies; Th and vacancies; and U and vacancies.

### Table 3: Average whole-rock major and trace element contents of the host granite and MMEs.

| Unit       | Host granite (n = 18) | MMEs (n = 2) |
|------------|-----------------------|--------------|
| SiO₂ wt%   | 75.9                  | 58.8         |
| TiO₂       | 0.3                   | 1.0          |
| Al₂O₃      | 11.0                  | 16.2         |
| Fe₂O₃      | 2.6                   | 7.2          |
| MnO        | 0.1                   | 0.1          |
| MgO        | 0.1                   | 2.7          |
| CaO        | 0.5                   | 3.7          |
| Na₂O       | 3.8                   | 6.0          |
| K₂O        | 4.3                   | 3.2          |
| P₂O₅       | 0.1                   | 0.2          |
| LOI        | 0.5                   | 0.5          |
| Total      | 98.8                  | 99.2         |
| Li (ppm)   | 107.36                | 114.01       |
| Be         | 5.08                  | 1.96         |
| Sc         | 3.13                  | 14.88        |
| V          | 1.05                  | 141.43       |
| Cr         | 21.03                 | 22.59        |
| Co         | 0.20                  | 18.11        |
| Ni         | 8.32                  | 11.41        |
| Cu         | 2.25                  | 5.57         |
| Zn         | 73.26                 | 137.40       |
| Ga         | 24.22                 | 21.34        |
| Rb         | 173.44                | 92.28        |
| Sr         | 34.33                 | 246.80       |
| Y          | 68.16                 | 48.80        |
| Zr         | 341.49                | 166.07       |
| Nb         | 14.50                 | 10.46        |
| Mo         | 0.60                  | 0.28         |
| Cd         | 0.17                  | 0.14         |
| In         | 0.22                  | 0.17         |
| Cs         | 13.05                 | 12.68        |
| Ba         | 100.73                | 314.72       |
| La         | 31.15                 | 23.04        |
| Ce         | 69.13                 | 53.50        |
| Pr         | 9.46                  | 7.54         |
| Nd         | 38.21                 | 31.40        |
| Sm         | 9.56                  | 7.43         |
| Eu         | 0.21                  | 1.04         |
| Gd         | 10.29                 | 7.45         |
| Tb         | 1.92                  | 1.34         |
| Dy         | 11.64                 | 8.26         |
| Ho         | 2.49                  | 1.70         |
| Er         | 7.27                  | 5.07         |
| Tm         | 1.20                  | 0.80         |
| Yb         | 7.86                  | 5.28         |
| Lu         | 1.14                  | 0.74         |
| Hf         | 11.25                 | 5.17         |
| Ta         | 1.49                  | 1.39         |
| Pb         | 24.51                 | 16.69        |
| Bi         | 0.70                  | 0.29         |
| Th         | 13.78                 | 9.49         |
| U          | 4.60                  | 2.92         |

The whole-rock geochemical data are cited from Tang et al. (2007), Su et al. (2008), and Yang et al. (2010).
TABLE 4 | Average calculated formulae and cation assignments (in apfu) of amphiboles based on (Locock, 2014).

| Unit       | Samples | Amphibole/granite | Amphibole/MMEs |
|------------|---------|-------------------|----------------|
|            | (n = 12) | (n = 2)           |                |
| T-site (8) | 8.17    | 8.32              |                |
| Si         | 8.14    | 8.32              |                |
| Al\(^{3+}\) | 0.03    | 0.00              |                |
| C-site (5) | 4.79    | 4.68              |                |
| Ti\(^{2+}\) | 0.20    | 0.11              |                |
| Al\(^{3+}\) | 0.18    | 0.06              |                |
| Zr         | 0.01    | 0.00              |                |
| Mn\(^{2+}\) | 0.05    | 0.07              |                |
| Fe\(^{3+}\) | 0.15    | 0.12              |                |
| Fe\(^{2+}\) | 4.17    | 3.96              |                |
| Mg         | 0.04    | 0.34              |                |
| B-site (2) | 2.00    | 2.00              |                |
| Na\(^{+}\) | 1.56    | 1.75              |                |
| Ca         | 0.44    | 0.25              |                |
| A-site (1) | 1.00    | 0.92              |                |
| Na\(^{+}\) | 0.70    | 0.60              |                |
| K          | 0.30    | 0.32              |                |
| W-site (2) | 0.41    | 0.56              |                |

The ideal number of each site is given in the brackets.

B-site is occupied by Na\(^{+}\) and Ca\(^{2+}\). In both sites, Na\(^{+}\) is the dominant cation. Therefore, these amphiboles belong to sodic amphiboles enriched in alkali. The octahedral C-site is occupied by various cations Ti\(^{4+}\), Al\(^{3+}\), Zr\(^{4+}\), REE\(^{3+}\), Mg\(^{2+}\), Fe\(^{2+}\), Fe\(^{3+}\), Mn\(^{2+}\), etc. The tetrahedral T-site is dominated by Si\(^{4+}\) with less Al\(^{3+}\). The W-site mainly contains anions F\(^{-}\), Cl\(^{-}\) and OH\(^{-}\), indicating (OH, F, Cl)-dominant amphiboles. According to the formula, W-site also contains O\(^{2-}\), the amount of which is calculated by Ti in apfu (Oberti et al., 1992).

In Table 4, The C-site often shows low occupancy (< 5 apfu), whereas the A-site has an over-occupancy (> 1 apfu), although the average value seems to be in the normal range. The low occupancy of the C-site and over-occupancy of the A-site cannot be reduced, no matter how we changed calculation procedures and normalization schemes or set up different initial Fe\(^{3+}\)/Fe\(^{2+}\) ratios and oxo-components. However, this issue seems to be common for alkali amphiboles in alkali and peralkaline rocks, as reported by Hawthorne et al. (1993). The reason may be related to the misfit of B-site cations in C-site that is followed by A-site cations shifting to B-site (Siegel et al., 2017). Although cation misfit may exist, B-site cations, which determine the sub-group of amphiboles, do not show such shift in formula and still point out that most amphiboles from the Huangyangshan granite and MMEs are sodic amphiboles.

Trace Element Chemistry of Amphiboles

The chondrite-normalized amphiboles REE pattern of both MMEs and granites are generally similar (Figures 5E,F), characterized by relatively flat light and middle REE, marked negative Eu-anomaly and an enrichment in HREE (Figures 5B,D). The (La/Sm)_N, Eu/Eu^∗ and (La/Yb)_N of amphiboles range from 0.42–1.74, 0.004–0.028, 0.03–0.13 in MMEs and 0.45–1.43, 0.002–0.039, 0.03–0.73 in granites. Amphiboles REE pattern of MMEs shows a weakly positive Ce-anomaly (1.19–1.63) with slightly lower total REE concentration than that of amphiboles hosted in granites (Figure 5D). The abundance of REE in each amphibole varies significantly, nearly an order of magnitude.

The primitive mantle-normalized trace elements pattern of amphiboles shows a negative Ba, Eu, Sr anomalies and positive Pb anomaly (Figures 5A,C). The Ba, Sr, Pb contents of amphiboles range from 1.61–6.40 ppm, 18.45–90.10 ppm, 9.06–25.60 ppm in MMEs and 0.45–11.50 ppm, 3.43–150.60 ppm, 7.54–58.60 ppm in granites. In amphiboles of MMEs, Nb and Ta show a positive anomaly compared with adjacent U, Th and La, Ce (Figures 5A,C). Nb, Ta, U and Th contents of amphiboles range from 8.62–150.10 ppm, 0.68–5.94 ppm, 0.03–1.22 ppm and 0.08–2.69 ppm, respectively. Zr and Hf of amphiboles do not show noticeably anomaly both in MMEs and granites but with an enrichment of Hf compared with Zr. The Zr and Hf concentrations of amphiboles range from 19.4–494.00 ppm, 1.19–49.30 ppm in MMEs and 161.00–728.00 ppm, 5.50–27.10 ppm in granites. These trace elements plotted in the spidergram also vary significantly.

Compared with the bulk compositions of the granite and MMEs, the Nb and HREE concentrations of the amphiboles are higher than the whole-rock Nb and REE concentrations (Tables 2, 3), whereas the LILE, Th, U and LREE concentrations of the amphiboles are lower, implying that HREE and Nb are more compatible in amphiboles than LILE, Th, U and LREE.

DISCUSSION

Magma Mixing and Formation of MMEs in the Huangyangshan Granite

Petrogenesis of MMEs and A-type granites are still controversial because of their complex geochemical characteristics (Bonin, 2007). MMEs are commonly found in granitoids and are more mafic than their host rocks in chemical compositions. Due to the compositional difference and the occurrence of MMEs, the MMEs was first regarded as refractory residues of partial melting and entrained by granitoids (White and Chappell, 1977). To date, four hypotheses have been proposed to explain the genesis of MMEs:

1) Refractory residues of partial melting source (e.g., White and Chappell, 1977; Barbarin and Didier, 1992; White et al., 1999; Chappell et al., 2000);
2) Early cumulus from host magma (e.g., Dodge and Kistler, 1990; Chen et al., 2009; Shellnutt et al., 2010; Huang et al., 2014);
3) Xenolith of wall rocks captured by magma (e.g., Yang et al., 2004, 2006);
4) Magma mixing through mafic magma injected into chambers of less mafic magma (e.g., Wiebe et al., 1997; Kumar and Rino, 2006; Su et al., 2008; Guo et al., 2010; Yang et al., 2010, 2011).
5) Different crust-derived melts mixing (e.g., Roberts and Clemens, 1993; Domanska-Siuda et al., 2019).

Although the petrogenesis of MMEs is still open to debate, the magma mixing model is the most widely accepted. The mixing processes provide constraints on the petrogenesis and sources of granitoids and information about the tectonics settings (Yang et al., 2006, 2011; Kazemi et al., 2019).

In our case, major and trace elements of the MMEs and granites prove that the MMEs formed via magma mixing (Figure 6). If we assume mafic magma similar to the average oceanic crust injected into the average host granites that are little influenced by the mafic magma, this two end-members mixing in Figure 6B shows that nearly 40-70% of felsic magma added to the mafic magma. Besides, zircon U-Pb ages support magma mixing because both MMEs and host granites are contemporary and the acicular apatites resulted...
from quenching hot mafic magma within cold felsic magma (Yang et al., 2011).

Voluminous felsic magma contaminated the mafic magma rendering the trace elements pattern of the MMEs resembles that of host granites. Therefore, as a late phase, amphiboles from both units are depleted in Ba, Eu, Sr while enriched in Pb. The similar patterns of REE and other trace elements reflect homogeneous mixing in trace elements which is also supported by the absence of zonation of amphiboles. In contrast, the major element contents are not only under control of the magma composition but are also influenced by other factors like crystal structure and valence balance. Therefore, the major elements of both amphiboles should record the chemical variation during magma mixing.

**Compositional Variation of Amphiboles**

**Halogens in W-Site of Amphiboles**

A-type granites and their sodic amphiboles are usually enriched in halogens such as F and Cl. As W-site anions, F and Cl can substitute for OH$^-$ and O$^{2-}$ (Hawthorne et al., 2012). Substitution between (F$^-$, OH$^-$, Cl$^-$) and O$^{2-}$ require involvement of Ti$^{4+}$ to maintain charge balance, i.e. (Mg, Fe$^{2+}$ + 2 (OH, F, Cl)$^-$ → Ti$^{4+}$ + 2 O$^{2-}$ (Oberti et al., 1992). Therefore, if F and Cl contents are controlled by crystal
chemistry, they must show a negative correlation with Ti. However, Figure 7F shows that Ti in the amphiboles has no clear correlation with F and Cl, implying that F in the amphiboles is mainly influenced by chemical compositions of host rocks, other than crystal chemistry. Similarly, F and Cl do not show correlation with Fe (Figure 7E), implying that the influence of Fe-F avoidance principle (e.g., Morrison, 1991) is negligible and also supporting the conclusion that halogens are mainly controlled by chemical compositions of melts.

Although amphiboles in our case crystallize as later phases, the variation of F still records the evolution of itself during
magma mixing. Because the chemical variation of magma is the only factor that influences F in the amphiboles, lower F content of amphiboles from F-saturated granites reflects that F decreased during evolution before magma mixing, which could be the result of fluorite crystallization. Subsequent mixing that the lowered the F content of the MMEs is recorded by the amphiboles from the MMEs.

Although crystal chemistry has little influence on F content, conversely, abundance of F can control partitioning of major and trace elements that occupies other sites and further change cause in crystal chemistry (Adam et al., 1993; Iveson et al., 2017, 2018). In aluminosilicate melts, elevated silica and alumina contents normally enhance polymerization whereas addition of F would form F-Si and F-Al complexes and depolymerize the melts (Iveson et al., 2017). In addition, polymerization of melts is a key factor that controls the partition coefficients of HFSE, REE and some other economic metals (e.g., Mo, W, Nb). Generally, the lower polymerization degree of the melt is, the lower $D_{\text{amp/melt}}$ of these elements will become (Iveson et al., 2018). On the contrary, Cl prefers to form alkali-Cl complexes, resulting in higher polymerization that facilitates incorporation of trace element into amphibole. In our case, both amphiboles from the host granites and MMEs are F-rich and Cl-poor, which is consistent with the conclusion that F is compatible in amphiboles and Cl is incompatible in amphiboles (e.g., Van den Bleeken and Koga, 2015). Although lacking reliable partition coefficients that are applicable for using amphibole halogen contents to estimate the halogen contents of the melts, the influence of halogens on major and trace elements partitioning cannot be ignored.

**Can Cations of A-, B-, and C-Sites Trace Magma Mixing?**

A- and B-sites are mainly occupied by Na with considerable K and Ca. On B-site, Na$^+$ and Ca$^{2+}$ can substitute for each other, followed by T-site substitution between Si$^{4+}$ and Al$^{3+}$. This coupled substitution can be expressed using the following reaction:

$$^{\text{B}}\text{Na}^+ + ^\text{T}\text{Si}^{4+} \leftrightarrow ^\text{B}\text{Ca}^{2+} + ^\text{T}\text{Al}^{3+}$$

Similarly, K$^+$ and Na$^+$ can also substitute for each other on A-site without any change in valence. For fluoro- sodic-amphiboles, elevated Na and F can constitute local configuration of $^\text{A}\text{Na}^3\text{F}^\text{W}\text{F}^\text{W}\text{Na}^5$ to maintain the stability of amphiboles (Hawthorne et al., 1996).

In **Figures 7A, B**, negative correlations of Ca-Si and Na-Al reflect Ca and Na are under the control of substitution and thus B-site cations cannot trace chemical variation of magma. The substitution of B-site also influences the A-site and the sum of cations of A-site often exceed 1 apfu, indicating crystal chemistry dominates the behavior of K and Na$^+$ (Supplementary Table 5).

Although the bulk CaO content of the MMEs is higher than the host granite (Table 1), the amphibole from the MMEs contain lower CaO content than that from the host granite. The reason for such discrepancy may be ascribed to MMEs containing more plagioclase than the granite. Therefore, more Ca partitions into plagioclase other than amphibole. Different from the CaO content, the Na$_2$O and K$_2$O contents of the amphiboles from both units are comparable, although the K$_2$O contents of the amphiboles are much lower than the bulk K$_2$O contents of both rock units. This phenomenon may be ascribed to Na having higher diffusivity than K and further imply the melt mixing cannot be fully traced by major elements of A-site either (Hawthorne et al., 2005).

As trace elements, Pb, U and Th can be incorporated into A- and B-site (Tiepolo et al., 2007; Hawthorne et al., 2012). Generally, Pb occupies the A-site, whereas U and Th occupy the B-site. Although they are expected to occur mostly as tetravalent cations which are unsuitable for A- and B-site from the perspective of charge balance, due to their small ionic radii, they prefer to occupy these sites in contrast (Tiepolo et al., 2007). Partitioning of Pb, U and Th is mainly controlled by melt compositions (i.e., polymerization of magmas). In addition, partitioning of U is also influenced by the redox state, because U$^{6+}$ is difficult to incorporate into amphibole. In this study, the Pb contents of amphiboles is similar to their hosts, indicating Pb is not so incompatible. This is consistent with the results of Brenan et al. (1995) and Tiepolo et al. (2007). Uranium and Th also have higher partition coefficients in sodic amphiboles than calcic amphiboles (Tiepolo et al., 2007).

More interestingly, Li is commonly expected to substitute for Na. It can also replace cations such as Mg or Fe$^{2+}$ via coupled substitution on C-site (e.g., Hawthorne et al., 1993, 1994; Oberti et al., 2003), which brings complexity in discerning the occupancy of Li.

Incorporation mechanism of Li in amphiboles includes (e.g., Hawthorne et al., 1994):

- $\text{Li}^+ \leftrightarrow \text{Na}^+$
- $\text{Li}^+ + \text{Fe}^{3+} \leftrightarrow 2\text{Fe}^{2+} + 2\text{Mg}^{2+}$
- $\text{Si}^{4+} + \text{Li}^+ \leftrightarrow \text{Al}^{3+} + \text{Mg}^{2+}$
- $\text{Ca}^{2+} + \text{Li}^+ \leftrightarrow \text{Na}^+ + \text{Mg}^{2+}$

In our case, Si and Mg show a negative correlation, which indicates that C-site occupied by Li is coupled with Si incorporated in T-site (Figure 7C). This process may further explain why T-site shows excess Si (> 8 apfu).

Besides Li, the C-site of amphiboles is mainly occupied by Fe$^{3+}$ and minor Mg$^{2+}$, Mn$^{2+}$, Ti$^{4+}$ and Fe$^{3+}$ (e.g., Hawthorne et al., 2012). Because Fe$^{3+}$ and Fe$^{2+}$ cannot be directly determined by EMPA, the content of Fe$^{3+}$ is estimated through algorithm based on charge balance (Locock, 2014). Therefore, Fe$^{3+}$/Fe$^{2+}$ cannot represent the redox state since no Mn$^{3+}$ exists in the amphiboles. No correlation between Fe$^{3+}$ and Mg$^{2+}$ also supports this (Figure 7D). In our case, C-site is only partly filled (< 5 apfu), the effect of crystal chemistry is insignificant except for Al, which is allocated to both C- and T-site, the latter where Si constrain the behavior of Al. Although Si negatively correlates with Mg, implying the Li-substitution (Figure 7C), the
Li content of amphiboles is too low to significantly influence the variation of Mg. Also, considering amphibole is the major carrier of Mg in both MMEs and host granites, Mg in amphiboles records the evolution of magma. Therefore, the amphiboles of the MMEs and host granites prove that high MgO magma mixed with low MgO magma.

The variations of Fe and Ti of amphiboles are different from their hosts. As suggested by the mixing model, the unmixed mafic magma is higher in Fe and Ti than granites but Fe and Ti of amphiboles from the MMEs are lower than those of granites. If we assume the high MgO amphibole crystallized earlier than low MgO amphiboles, the former has lower Fe and higher Ti while the latter is opposite depicts such a scenario: when high-MgO amphibole crystallized, the crystallization of titanite and Fe-oxide had already decreased the Fe and Ti content of the mixed magma, as evidenced by the similar range of Ti content among high-MgO amphibole and amphiboles from granites; subsequent injection of felsic magma replenish the Fe to increase the Fe of low-MgO amphibole while consistently crystallizing titanite further lower the Ti of low-MgO amphibole.

Similar to Ti, other HFSE (including Nb, Ta, Zr, Hf) also occupy C-site but behave diversely. Zr$^{4+}$ and Hf$^{4+}$ have the same valence as Ti$^{4+}$ but larger ionic radii (Shannon, 1976), Zr and Hf are thus sensitive to compositional change of amphiboles or melts (Tiepolo et al., 2007). In contrast, although Nb$^{5+}$ and Ta$^{5+}$ have higher valence and the high valence can be balanced by oxo-component introduced during Ti incorporation ((Mg, Fe)$_2$O + 2 (OH, F, Cl)$^-$ → Ti$^{4+}$ + 2 O$^2$) (Tiepolo et al., 2001, 2007). Hence, Nb and Ta in amphiboles could behave as slightly incompatible or even compatible elements with increasing Ti (Tiepolo et al., 2007). In both the Huangyangshan granite and its MMEs, Zr and Hf show different partitioning behavior between the amphibole and melt. Niobium is relatively compatible compared to Ta in the amphibole from the granite whereas both Nb and Ta behave compatibly in the amphiboles from the MMEs (Figures 5A,C).

Distribution of REE
Distribution of REE in amphibole-group minerals is mainly influenced by crystal structure and melt compositions. The effect of melt compositions relates to major elements that are involved during incorporation of REE into amphiboles. The difference in substitution mechanism can be justified by different site preference between LREE and HREE (Bottazzi et al., 1999; Shimizu et al., 2017). Similarly, the size of different sites also controls the site preference of REE (Blundy and Wood, 1994). Many studies have demonstrated that LREE prefer to substitute for Ca$^{2+}$ whereas HREE prefer to substitute for Fe$^{3+}$ corresponding to M4-site (B-site) and M4’-site (B-site) or M2-site (C-site) respectively (e.g., Bottazzi et al., 1999; Tiepolo et al., 2007).

Due to the lower Ca content and higher Fe content of sodic amphibole, HREE are much more compatible than LREE in sodic amphibole and the $D_{\text{amph-melt}}$ of REE in our case are higher than the values reported in other studies (Table 5; e.g., Marks et al., 2004; Siegel et al., 2017). The REE partition coefficients of the amphibole from the host granites show a pattern that from La to Td and from Dy to Lu, each group fits a specific parabolic trend that can be calculated using the model of Blundy and Wood (1994). However, REE partition coefficients of the amphibole from the MMEs do not show the same trend but monotonically increase from La to Lu instead (Figure 8). This phenomenon may indicate that in melts with different compositions, site preference of REE could be different. In addition, the partition coefficients decrease from core to rim (Table 5, complete partition coefficients of REE between amphiboles and their hosts are given in Supplementary Material S6), regardless of the hosts of amphiboles, implying that most REE are distributed into accessory minerals, such as titanite and/or zircon.

Temperature and Pressure Estimate of Sodic Amphiboles
According to major and trace elements, zircon U-Pb ages, texture, and rheology, the petrogenesis of the MMEs hosted by the Huangyangshan A-type granite requires mafic magma injection (Figure 9). If the amphiboles are the later phase during magma mixing, they may record similar temperature. Notably, most thermometers and barometers for amphibole are based on chemical compositions of calcic amphiboles. However, in our case, amphiboles are mostly sodic amphiboles that may not be suitable for these thermometers and barometers (e.g., Ridolfi et al., 2010; Ridolfi and Renzulli, 2012; Putirka, 2016). Therefore, the prerequisite is to test whether these amphiboles have intrinsic flaws that prevent them from estimating temperature or pressure and seek for a reference value to test the estimates of various thermometers.

Before testing and estimating the crystallization temperatures of the amphiboles, an “amphibole-saturation temperature” for a given liquid composition can be estimated using liquid-only thermometer provided by Molina et al. (2015). Because the chemical compositions of amphiboles are not needed for calculation, liquid-only thermometer can yield reasonable values of 1024 ± 42°C for MMEs and 843 ± 42°C for granites, regardless of the species of amphiboles.
FIGURE 8 | Diagrams showing the relationship between ionic radii and apparent partition coefficient $D_{\text{amph-mel}}$ (average REE content of amphiboles/average REE content of the bulk rock) for amphiboles from the host granite (A) and from the enclaves (B). Blue dash lines represent Lattice-Strain Theory fit through these data. Black dash lines reflect potential site preference of REE.
We then tested and estimated the crystallization temperatures of the amphiboles from the Huangyangshan granite and its MMEs using amphibole-only thermometers and thermometers based on partition coefficients. Amphibole-only thermometers are based on the correlation between temperature and chemical compositions of amphiboles that are either experimentally synthesized or natural samples (e.g., Ridolfi et al., 2010; Ridolfi and Renzulli, 2012; Putirka, 2016). Through these algorithms, the temperature estimates for the amphiboles in the MMEs and host granite are consistent within errors (~650°C), implying that the amphiboles crystallize as late phase, consistent with the fluorite, titanite, and Fe-oxide crystallized earlier than amphiboles (Price et al., 1999; Scaillet and MacDonald, 2001). As for thermometers based on partition coefficients, Molina et al. (2015) and Putirka (2016) recommend thermometers derived from correlation between temperatures and D_{Mg} or D_{Na}. Although Molina et al. (2015) emphasized that their dataset only contained amphiboles with Ca^B/(Ca^B + Na^B) > 0.75, Mg in amphiboles does not show any correlation with Ca or Na in our case (Figures 7G,H), therefore the thermometer provided by Molina et al. (2015) may be applicable to our samples. The temperature estimates are 946 ± 45°C for amphiboles in the MMEs and 1028 ± 45°C for amphiboles in the granite, respectively. However, the estimates are higher than the “amphibole saturation temperature” and the temperature of the amphibole from the host granite deviates from the amphibole stability field under most conditions (e.g., Xiong et al., 2005). On the other hand, the amphibole from the host granite crystallized earlier than that from the MMEs is unreasonable. This may be ascribed to that the Mg or Na exchange between the amphiboles and melts do not reach equilibrium, especially the Na in sodic amphiboles is under control of crystal chemistry. Thus, the temperature is more likely the closure temperature for element exchange to reach equilibrium other than the crystallization temperatures.

In summary, amphibole-only thermometers yield good results because the estimates are under “saturation temperature” but the thermometers also prove that the amphiboles crystallized as a late phase consistent with petrographic evidence. However, thermometers based on partition coefficients produce erroneous estimates.

The basis of amphiboles barometer is that Al-Tschermak exchange, which mainly involves in Al incorporation, is pressure-sensitive, so it seems to be proper to use Al to estimate pressure of crystallization (e.g., Hammarstrom and Zen, 1986; Shane and Smith, 2013; Mutch et al., 2016). However, most researchers have questioned the reliability of Al-in-amphiboles barometer, because the exchange of Al is also influenced by temperature and crystal compositions, which means that variation of Al content of amphiboles is too complex to use for estimating pressure directly (e.g., Kiss et al., 2014). In order to acquire accurate pressure estimates, Anderson and Smith (1995) recommend two filters: T < 800°C and Fe^{Amp}/(Fe^{Amp} + Mg^{Amp}) < 0.65. If amphiboles fit these filters, they are suitable for Al-in-amphiboles barometer. Unfortunately, in our case, amphiboles in both the granite and MMEs are Fe-rich, far beyond the values required by the filters. On the other hand, Al content of amphiboles in our case are too low, which results in an unreasonably low pressure. In addition to Al-in-amphiboles barometer, Putirka (2016) recommended D_{Al} barometer to lower the uncertainty of pressure estimates, but no reasonable estimates can be acquired (10.5 kbar for amphiboles of MMEs; 3.5 kbar for amphiboles of granites). The outstanding pressure difference between the amphiboles from the MMEs and granites may also indicate that the estimates do not reflect depths but more related to temperature variations, which is consistent with the conclusion of Erdmann et al. (2014). Therefore, sodic amphiboles cannot be used to reflect the pressure change during magma mixing.

**CONCLUSION**

We have shown major and trace elements variation of amphiboles of granites and MMEs during magma mixing and tentatively estimate the crystallization temperature and pressure of these amphiboles. In sodic amphiboles from both the MMEs and host granite, halogens in W-site and Mg, Fe, and Ti in C-site are only controlled by chemical compositions of magma and can potentially reflect the evolution of magma. By contrast, A-, B- and T-site cations are influenced by crystal chemistry and magma composition limiting their ability as tracers. Although F can depolymerize the melts, resulting in lower partition coefficients of trace elements, HFSE and REE behave more compatibly in sodic amphiboles than in calcic amphiboles. Also, taking “saturation temperature” as reference, amphiboles-only thermometers yield reasonable results and further prove amphiboles are later phase during magma mixing, but partition coefficient-based
thermometers show considerable deviation. All of the barometers now available rely on Al content or D$_{Al}$ of amphiboles cannot be applied to Fe-rich and Al-poor amphiboles.

**DATA AVAILABILITY STATEMENT**

The original contributions presented in the study are included in the article/Supplementary Material, further inquiries can be directed to the corresponding author/s.

**AUTHOR CONTRIBUTIONS**

YF and CY together conceived the idea for this study. CY conducted petrographic observation, EPMA and LA-ICP-MS analyses on amphiboles, and wrote the manuscript. YF revised and finalized the manuscript. YF and RL conducted the fieldwork and collected the samples. CYF provided assistance with the fieldwork. All authors contributed to the article and approved the submitted version.

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**FUNDING**

This study is funded by the National Natural Science Foundation of China (grant numbers: 91962214 and 41902073) and Department of Science and Technology of Shaanxi Province (grant number: 2020JM-215).

**ACKNOWLEDGMENTS**

We are very grateful for the insightful suggestions from HL, FY, and ES that helped improve this manuscript. We would also like to thank XD for handling this manuscript.

**SUPPLEMENTARY MATERIAL**

The Supplementary Material for this article can be found online at: https://www.frontiersin.org/articles/10.3389/feart.2021.650014/full#supplementary-material
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Conflict of Interest: The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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