Study of the zircon morphology and internal structures as a tool for constraining magma source: example from granitoid bodies in the northern Sanandaj Sirjan zone (SW Saqqez)

Narges Daneshvar1, Mohammad Maanijou1*, Hossein Azizi2, Yoshihiro Asahara3

1 Department of Geology, Faculty of Science, Bu-Ali Sina University, Hamedan, 65174-33391, Iran.  
2 Mining Department, Faculty of Engineering, University of Kurdistan, Sanandaj, Iran.  
3 Department of Earth and Environmental Sciences, Graduate School of Environmental studies, Nagoya University, Furo-cho, Chikusa, Nagoya 464-8601, Japan.  
*Corresponding author, e-mail: mohammad@basu.ac.ir  
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Abstract

The granitic intrusives in southwest Saqqez are located in the northern Sanandaj Sirjan zone. These granites can be divided into mesocratic and leucocratic granites. The external morphology and internal structures of zircon from these granites have been investigated employing the classic Pupin method supplemented by electron microscope analysis. The zircon crystallization is a function of temperature, chemical composition, water content of magma, velocity of crystallization and Zr saturation of magma. So, we have focused on the study of zircon to evaluate the chemical characteristic of these bodies. The minimum temperature of crystallization in mesocratic granite based on morphology of zircon crystal ranges from 784 to 788°C and for leucocratic granite ranges from 704 to 847°C, which show good correlation with calculation of saturation temperature of zircon (755 to 866°C for mesocratic granite; 755 to 832°C for leucocratic granite). Crystal growth {101} in most of zircon crystals in case study indicating I-type source of magma. Also, crystal growth {100} shows high temperature which confirm the result of thermometry. Also, absent of apatite and monazite inclusions in zircons and intergrowth hydrothermal zircons indicating dry I-type magma which correspondent with geochemical data, mineralogical survey and field observation.

Keywords: Zircon, Thermometry, Morphology, Saqqez, Iran.

Introduction

Zircon is one of main accessory mineral in igneous rocks (Deer et al., 1992) and due to its chemical and physical stability, it is useful tool for determining petrology, geochronology and formation history of magmatic rocks (Corfu et al., 2003; Sturm & Steyerer, 2003; Sturm, 2010). Morphology and internal texture of zircon can identify magmatic, metamorphic and recrystallization processes (Pupin and Centre national de la recherche scientifique (France), 1976; Pupin & Turco, 1972; Pupin, 1975; Corfu et al., 2003). Zircon crystallizes in the tetrahedral system and grows up as prismatic crystal with length to width ratio of one to five (Corfu et al., 2003). Less ratio of length to width (1:10) indicate high rate of crystallization in magma source (Corfu et al., 2003).

Due to resistance of zircon during the evolution of magma, in this research we tried to survey the morphology of zircon and also the chemical characteristic of granites in southwest Saqqez, to understand genetic classification of these magmas and comparing the result of morphology of zircon with geochemical data and mineralogical observation.

General geology

The study area is located between 36°06'00"-36°09'43"northern and 46°00'00"-46°14'02"eastern in southwest Saqqez. According to the structural map of Iran (Stocklin & Nabavi, 1973), is located in Sanandaj- Sirjan zone (SaSZ) (Fig. 1a). The western and southwestern Saqqez area consist of Precambrian metamorphic basement, which have an unconformable boundary with Permian carbonaceous to clastic deposits. The metamorphic basement with later magmatic activity in the study area are related to Pan-African orogenic phase in the Saqqez area (Babakhani et al., 2003). Based on geological map of Saqqez (Babakhani et al., 2003), the main units belong to the Precambrian basement and include schist, gneiss with some granitoid and marble which exposed in the southwest of Saqqez. A part of the granitoid bodies are highly metamorphosed, and some dynamic structures overprinted the rocks. These granites are the oldest igneous rocks on the map which intruded to metamorphosed rock and outcropped in west Miredeh village (Fig. 1b). These granites have a smooth rolling topography and are mesocratic in the west and leucocratic in the east (Fig. 2a and b). There is no obvious boundary
between them and are exposed over an area around 14 km² as some patches in the southwest of Saqqez city. These massives have concordant contacts with the surrounding Precambrian schist. These bodies are equivalent to the Doran granite (Babakhani et al., 2003) and was foliated due to dynamic deformation (Fig. 2b). The mesocratic granite is cut by some quartz veins (Fig. 2c), meanwhile diabasic dykes intruded in leucocratic granite (Fig. 2d). In this study, morphology and chemical analysis of these granites are discussed to indicate the magma source.

Sample preparation
Approximately 100 fresh samples were collected from the granitoid body. Forty-five thin sections were prepared from selected samples and observed by polarized-light microscope. Twelve samples are selected for chemical analysis. Around 500g of each rock sample was crushed in a steel jaw crusher and was pulverized in a tungsten carbide mill to 230 mesh. Analytical methods on the powdered rock samples include wavelength dispersive X-ray fluorescence spectrometry (WD-XRF) of Rigaku ZSX Primus II for determining major oxide and inductively coupled plasma-mass spectrometry (ICP-MS) of Aligent 7700x for determining amount of Zr at Nagoya University, Japan.

Two samples of mesocratic granites (SPM3 and SPM4) and three samples of the leucocratic granites (GKR2, GKR3 and GKR6) selected for surveying of morphology of Zircons.

Figure 1. (a) Simplified structural subdivision map of Iran (Stocklin & Nabavi, 1973). (b) Geological map of the study area in southwest Saqqez (modified after Babakhani et al., 2003). The samples locations are shown on the map.
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These samples were crushed and powered to 250 micrometers. After magnetite separation, heavy minerals were separated by heavy-liquid (diiodomethane) method. Zircon grains were selected from the heavy minerals by hand picking under a binocular microscope. The zircon grains were then mounted in an epoxy resin and polished until the zircons could be observed brightly in reflected light. The zircons mounted in the resin were observed by a scanning electron microscope (SEM) (Hitachi S-3400N) at Nagoya University, and cathodoluminescent (CL) and backscattered electron (BSE) images were taken to detect their internal structures.

Mineralogy and geochemistry
The mesocratic granites in the southwest Saqqez mainly include quartz (30–35%), alkali feldspar (15–20%), plagioclase (20–25%), biotite (5–10%), and hornblende (10-15%) (Fig. 3a and b) with accessory minerals of zircon, titanite, apatite, and iron oxide which are deformed by active tectonic in the study area. The hornblende seems to convert to biotite and tourmaline (Fig. 3a and b). The biotite display brown to yellowish pleochroism and shows local alteration into chlorite. The leucocratic granite in southwest Saqqez includes mainly quartz (30-35%), alkali feldspar (15-20%), plagioclase (20-25%), and biotite (5-10%) (Fig. 3c and d). This body has granular, granophyric and perthite textures which indicate crystallization has happened deep to surface. The quartz grains are anhedral with wavy extinctions. They sometimes showing serrated contacts with neighboring quartz grains, and some of them occur as lenticular poly-crystalline aggregates. Quartz ribbons suggest grain boundary migration. The alkali feldspars are mostly microcline and exhibit microperthitic to perthitic textures. The plagioclases with euhedral to subhedral form are affected by dynamic deformation, show lenticular kink band structure and are often characterized by altered cores.
In the diagram of total alkali oxides (Na$_2$O+K$_2$O) versus SiO$_2$ variation (Middelmost, 1985), the mesocratic granite samples plot in granodiorite with exception of two samples which plot in monzogranite and granite, and the leucocratic granite samples plot in the granite field, with exception of one sample which plots in the granodiorite field (Fig. 4a).

Figure 3. Photomicrographs of the granites in the southwest of Saqqez. (a and b) These photos show main minerals of the mesocratic granites: quartz, plagioclase, alkali feldspar, biotite, tourmaline, and hornblende. (c and d). These photos show main minerals of the leucocratic granites: quartz, plagioclase, alkali feldspar, and biotite. Kfs= K-feldspar, Pl= plagioclase, Hbl= hornblende, Bt= biotite, and Tur= tourmaline. Abbreviations are after Kretez (1983).

Figure 4. (a) Na$_2$O+K$_2$O versus SiO$_2$ diagram (Middelmost, 1985), the mesocratic granite samples plot in granodiorite expect two samples which plot in monzogranite and granite and leucocratic granite sample plot in granite field.
By utilizing the $\text{Al}_2\text{O}_3/(\text{Na}_2\text{O}+\text{K}_2\text{O})$ [A/NK] versus $\text{Al}_2\text{O}_3/(\text{CaO}+\text{Na}_2\text{O}+\text{K}_2\text{O})$ [A/CNK] diagram (Shand, 1927), the mesocratic and leucocratic granite samples plot in the peraluminous field, with exception of one sample (Fig. 4b).

Discussion

External morphology of zircon

Investigation of internal and external morphology of zircon crystal, can help determination of magma source, chemical composition (saturation degree of Al and alkalin elements) (Pupin, 1980) and temperature of melt (Pupin & Turco, 1972). Variation of internal structure can interpret chemical and physical changes. Altogether, the zircon external morphology combined with internal structure has the ability to record magma melting, contamination and mixing with different magma, fractional crystallization and differential and final emplacement an intrusion (Corfu et al., 2003; Benisek & Finger, 1993; Siebel et al., 2006).

Relationship between morphology of zircon, chemical composition and geodynamic setting of magma source were discussed by many researchers (Pupin & Turco, 1972; Pupin, 1975, 1980, 1985, 1988; Pupin and Centre national de la recherche scientifique (France), 1976; Krasnobaev, 1986; Wang & Kienast, 1999; Berezhnaya, 1999). For example, Pupin (1980) classified zircon crystal based on relative growth of prismatic shapes $\{100\}$ to $\{110\}$ and pyramidal $\{211\}$ to $\{101\}$. According to survey of morphology of zircon in igneous rocks, it can be determining three groups (1) granite with crustal source, (2) hybrid granite and (3) granite with mantle source (Pupin, 1980; Fig. 5). He showed that the relative growth of pyramidal shapes with a chemical composition (Al / (Na+K ratio) or index A) and relative growth of pyramidal shapes are directly related to the crystallization temperature. In other hand, in dry alkaline to tholeiitic magma, zircons are commonly in $\{100\}$ and $\{101\}$ form and also, I-type granite are characterized by a mainly of zircon crystal with flat pyramid $\{101\}$ whereas S-type granites mainly have zircon crystal with steep pyramids $\{211\}$ (Benisek & Finger, 1993; Sturm, 1999; Belousova et al., 2005; Köksal et al., 2008; Fig. 6). Pupin rules were discussed by many authors (e.g, Benisek & Finger, 1993; Vavra, 1990, 1993). For example, based on Vavra studies (1990, 1993) external morphology of zircon, due to kinetic factors like rate of distribution and absorption in every crystal during growth event can change times and times, so influence the rate of growth and morphology of zircon (Vavra, 1994). In other hand, relative growth pyramid of zircon is related mostly by supersaturation of zirconium in magma instead of magma temperature (Benisek & Finger, 1993).

Figure 5. Zircon typological classification and corresponding thermometric proposed by Pupin (1980). Index A is the ratio Al/(Na+K) which controlling the development of zircon pyramids, whereas temperature affects the development of zircon prism.
Based on Pupin idea (1980), temperature of magma is the most important factor for relative growth of various form of zircon crystal, so it can be used as geothermometer. But, as Vavra’s studies (1990) growth rate and size of zircon crystal are controlled by oversaturation of melt from ZrSiO$_4$ and the amount of trace element and these factors are more important than temperature. According to him, pyramids morphology {211} versus {101} is basically control external trace element, while the prism form is determined the supersaturation degree of ZrSiO$_4$. In addition of mentioned cases, zircon has a good tenacity for absorption of trace element, therefore, survey of its zoning (which indicate variation of element during magma evolution) increase the survey value of this mineral (Watson, 1996; Watson et al., 1997; Belousova et al., 2005; Shabanian et al., 2009).

The crystalline system of zircon is tetragonal and its crystal symmetry is tetragonal or ditetragonal and dipyramidal (Martins et al., 2014). Zircon crystals in granite of southwest Saqqez are honey yellow to colorless and transparent to translucent. The majority of them are euhedral and less subhedral and commonly between 100-200 μm in size (Fig. 7). They show high to regular birefringence. The main crystalline faces are observed in separated zircon include pyramids {101} and prisms {100}, {110} are the basis of zircon typology (Pupin and Turco, 1972). Various types of zircon are reported by Pupin (1980) on IA-IT diagram based on relative development of prismatic and pyramidal crystal faces. The parameter of A index (IA) is related the majority of Al/(Na+K) ratio and with the development of pyramids faces. The {211}, {101} and {301} pyramids are respectively well-developed in aluminous, alkaline and peralkaline medium, respectively (Martins et al., 2014). The parameter of T index (IT) is related with temperature of zircon crystallization, which is responsible for the development of the prismatic faces. A high T index {100} prism indicates a high temperature than a low T index (110) prism.

Three main parameters for reorganization of typology of zircon are included:
- typological distribution (frequency of each type and subtype)
- TET (Typological evolutionary trend)
- Mean point (A, T) calculation.

The calculation of TET is given by:

$$\text{as; } \text{IA}=\sum n_{\text{IA}A} \times A, \quad \text{IT}=\sum n_{\text{IT}T} \times T,$$

where $n_{\text{IA}A}$ and $n_{\text{IT}T}$ are the respective frequencies for each value of IA or IT. The TET represents the evolution of the different types during the magmatic stage and is defined as the mean points of IA calculated for each value of IT (Pupin, 1988), so, for calculation of typological evolution trend, a line with slope of $\text{ST/SA}$ (standard of deviation of index T/standard deviation of index A) has been drawn from intersection of IA and IT that is equals to tangent of angel of two axes T.E.T and I.A (Pupin, 1980).

Pupin (1980) suggested, one of the main application of the typologies study of zircon is a genetic classification, with three main divisions as:
- Granitoids of crustal origin.
• Hybrids (crustal and mantle origin) granitoids.
• Granitoids of mantle origin.

On the typology diagram, district TET crustal granitoids commonly include leucogranites and aluminous monzogranite which characterized by low A and T index. Hybrid granitoids have calc-alkaline property and mainly are monzogranites and granodiorites. They indicate large ranges of A (higher than those crust ones) and T (from very high to low). Finally, mantle origin source are alkaiine granitoids with high A and T index values (Pupin, 1980).

For the typological of zircon population (Pupin, 1980), at least 100 euhedral crystal were chosen randomly and identified for each sample. The zircon population from granites in southwest of Saqqez show a great variety of subtypes, which are more concentrated in the right-hand side of zircon typological diagram (Pupin, 1980). The zircon morphology in sample GKR2 are P4, S20, S19 and P3, in Sample GKR3 are P4, S19 and S20, in sample GKR6 are P4 and P3, in sample SPM3 are P4, P3, S20 and S19 and in sample P4 are mostly S20, S19, P4 and S15 which are mostly {110} and {100} prism and {101} pyramid (Fig. 8). The typological evolutionary trend measured for studies zircons for GKR2, GKR3, GKR6, SPM3 and SPM4 have angles of 25.6°, 18.3°, 27°, 22.3° and 25.6°, respectively (Fig. 9).

Figure 7. (a-e) photos of separated zircon crystals of samples GKR2, GKR3, GKR6, SPM3 and SPM4

Figure 8. Typological statistics of the investigation zircon population in separated zircon crystals of meta-granite in southwest Saqqez.
So, the typological evolutionary trends on studied zircons are similar to I-type granite source and based our plot on genetic classification IA-IT diagram Pupin (1980) indicated I-type granite with crustal- plus- mantle origin.

BSE image of separated zircon for samples CKR2, GKR3, GKR6, SPM3 and SPM4 have shown in Fig. 10. Also, zircon in S-type or in hybrid magma have inclusions of apatite and monazite but I-type granite is free or have less of those inclusions (Pupin, 1980). Separated crystals of zircon in meta-granite in southwest Saqqez are free of inclusion which indicating I-type granite (Fig. 10). Also, loss of hydrothermal zircon as intergrowth in these samples show they are formed from the dry I-type granite (Corfu et al., 2003; Pupin, 1980).

**Internal morphology of zircon**

The zoning show compositional variation of Zr and Si and more importantly in Hf, P, Y, REE, U and Th (Corfu et al., 2003). This feature is best seen in cathodoluminiscence (CL) image (Figs. 11-15). The growth history of zircons from metagranite in southwest Saqqez can be recognized by using the parallel and perpendicular crystal sections to crystallographic axis (c-axis) in Figs 11-15. The present of well-developed zoning is one of the main features for determining of magmatic zircon. Separated zircon crystals from the metagranites show well-developed magmatic oscillatory zoning and are free of visible inclusions in sections perpendicular to the crystallographic c-axis and \{101\} pyramids and \{100\} prisms are well developed (Figs. 11-15).

Some zircon crystals have well-rounded cores with a different zonation pattern compared to the rime (Fig. 12-6), which indicating a phase of zircon undersaturation of the melt. Loss of oscillatory zoning and low CL intensity rims are typical feature of recrystallization (Fig. 11-6, 13-19 and 15-20) indicating a possible depletion in heavy element (Hancher & Miller, 1993; Poller et al., 2001).

Xenocrystal of zircon is a common feature in magmatic rock which is seen as euhedral and subhedral. Always, interpretation of these xenocrystal cores are problematic, so the key for their sources is field observation. For example, xenocrystal cores in zircon of migmatite and in S-type granite are inherent zircons, but in the most cases, it is difficult to find the source. In Figs. 11-13, 12-6, 12-10, 13-15,14-8, 14-26,14-31, 15-3 and 15-4 some zircons have the primary cores with overgrowth ring, indicating the inherent zircons. Mostly, the separated zircons are inherent free zircon that show the I-type source and there are a few inherent zircons. There are only a few xenocrystal cores of old protolith in northern part of Sanandaj- Sirjan zone.
**Thermometry by crystal of zircon**
Based on morphology of zircon crystals or by using geochemical data can identify the temperature of magma. These maintained case is used for determining the minimum temperature of zircon crystallization in granites of southwest Saqqez.

**Thermometry based morphology of zircon**
Given that the prism-type in zircon is related to the temperature of melt, so zircon morphological features can be used as a geothermometer (Pupin & Turco, 1972). Zircons that crystallized from high-temperature melt are dominant by {100} prism and those that crystallized from low-temperature melts by {110} prism. Given temperatures according to the morphology of zircon grains in samples GKR2, GKR3, GKR6, SPM3 and SPM4 are 785, 847, 704, 784 and 788 °C, respectively (Fig. 9).

Figure 10. Back-scatter image of scattered zircon crystals separated of samples GKR2, GKR3, GKR6, SPM3 and SPM4 from meta-granite in southwest Saqqez, indicating loss of inclusion in zircon.
Thermometry based on saturation temperature of zirconium

Assessing temperature of magma is difficult. Because the appropriate mineral pairs with temperature sensitive exchange reaction are lack and re-equilibration may happen during the cooling. Thermometry with using of saturation zirconium by Watson & Harrison (1983) provides acceptable and robust means of estimating magma temperature. Zirconium solution is related to temperature. So, zircon as a common mineral in intermediate to acidic rocks, is used as geothermometer. Zircon in igneous rock either precipitated from the melt or may be accidental, inter from wall of host rock

Figure 11. CL image of zircon crystal section oriented parallel to the crystallographic axis in GKR2 sample.

Figure 12. CL image of zircon crystal section oriented parallel to the crystallographic axis in GKR3 sample.
through late magmatism, or inherited which derived at a deeper level from a contributing source material, which have thicker magmatic overgrowths.

Watson & Harrison (1983) confirmed the relationship between the solubility of zircon, temperature and composition of the melt base on the equation 1:

Equation 1 \( \ln(D_{zr})=\frac{12900}{T(K)}-0.85(M-1)-3.80 \)

In this equation, \( D_{zr} \) is rate of zirconium concentration in molten (ppm). \( T \) represents the temperature in kelvin and \( M \) is cationic ratio depending on \( \text{SiO}_2 \) and aluminous melt calculated through Equation 2:

Equation 2 \( M=\frac{(Na+K+2Ca)(Al/Si)}{} \)

With revising the equation 1 based on \( T \) parameter, we have:

Equation 3 \( T_{zr}=\frac{12900}{2.95+0.85+\ln(496000/Zr \text{ melt})} \)

The thermometer requires the following: (1) Proper calibration under the appropriate conditions. Watson and Harrison (1983) demonstrated that their empirical relationship applies over a wide range of conditions and melt compositions. Solubility is largely insensitive to pressure and appears to deviate from their equation only for dry (1.5 wt% \( H_2O \)) or peralkaline melts; thus, it should apply to most intermediate to felsic magmas in the crust. (2) Melt saturated in zircon.

Figure 13. CL image of zircon crystal section oriented parallel to the crystallographic axis in GKR6 sample.
Figure 14. CL image of zircon crystal section oriented parallel to the crystallographic axis in SPM3 sample.

Textural evidence can be used to identify early saturation. Inherited zircon suggests saturation throughout the parent magma’s history. (3) An adequate estimate of melt composition (major element and Zr concentrations).

Twelve samples of these granites from southwest Saqqez are selected for chemical analysis. According to equation provided after Watson & Harrison (1983), zircon crystallization temperatures of these granites were calculated 755 to 866°C for mesocratic and 755 to 832°C for leucocratic granites (Table 1).
Table 1. Representative major elements (wt%) and trace element zirconium (ppm) composition of granite samples in south west Saqqez (ICP-MS analysis) with determination of saturation temperature of zirconium by Watson & Harrison equation (1983) (W and H) and Bochnke et al (2013) equation (B).

<table>
<thead>
<tr>
<th>Sample</th>
<th>Si</th>
<th>Al</th>
<th>Ca</th>
<th>Na</th>
<th>K</th>
<th>Zr</th>
<th><em>T°C</em> (W and H)</th>
<th><em>T°C</em> (B)</th>
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<tr>
<td>ND-1</td>
<td>31.34</td>
<td>7.63</td>
<td>1.25</td>
<td>3.38</td>
<td>1.51</td>
<td>295</td>
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<td>ND-2</td>
<td>29.40</td>
<td>10.11</td>
<td>0.28</td>
<td>3.89</td>
<td>2.76</td>
<td>308</td>
<td>866</td>
<td>850</td>
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<td>7.99</td>
<td>0.46</td>
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<td>185</td>
<td>822</td>
<td>800</td>
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<td>840</td>
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<td>1.72</td>
<td>3.21</td>
<td>1.93</td>
<td>118</td>
<td>755</td>
<td>740</td>
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Leucocratic granite

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<tr>
<th>Sample</th>
<th>Si</th>
<th>Al</th>
<th>Ca</th>
<th>Na</th>
<th>K</th>
<th>Zr</th>
<th><em>T°C</em> (W and H)</th>
<th><em>T°C</em> (B)</th>
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<tr>
<td>ND-4</td>
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<td>171</td>
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<td>760</td>
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<td>312</td>
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<td>0.87</td>
<td>118</td>
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Figure 15. CL image of zircon crystal section oriented parallel to the crystallographic axis in SPM4 sample.
Calculation of temperature based on zircon saturation using equation of Boehnke et al. (2013) revised the relationship between the solubility of the molten provided from Watson & Harrison (1983) and provided equation 4 to estimate the saturation temperature of zircon (variables are like equation 1).

\[
\text{In}DZr = \left(\frac{1018 \pm 32}{T(K)}\right) - (1.16 \pm 0.15)(M-1) - (1.48 \pm 0.09)
\]

According to equation of Boehnke et al (2013), zircon crystallization temperature of granites bodies in southwest Saqqez are calculated 740°C to 850°C (mesocratic granite) and 740 to 823°C for leucocratic granite (Table. 1) which have good correlation with obtained temperature based on morphology of zircon.

**Zircon typology and initial \(^{87}\text{Sr}/^{86}\text{Sr} \) ratio**

There is good correlation between the genetic scheme derived from zircon typology studies and the \(^{87}\text{Sr}/^{86}\text{Sr} \) whole-rock ratios (Pupin, 1980). Initial \(^{87}\text{Sr}/^{86}\text{Sr} \) Sr isotope ratios of 12 samples of granites are available (submitting data) including 0.7077 to 0.7157, indicating I-type affinities.

**Conclusion**

Zircon external morphology and internal structure suggest that mesocratic and leucocratic granite are of I-type granites. Crystal growth \{101\} in most of zircon crystals in the case study, indicating I-type source of magma. Based on typology of zircon, minimum crystallization temperature in mesocratic and leucocratic granites in southwest Saqqez, are 704 to 847°C and 784 to 788°C, respectively, which show good correlation with calculation of saturation temperature of zircon (755-866°C, mesocratic granite; 755-832°C; leucocratic granite). Also, crystal growth \{100\} show high temperature which confirm the result of thermometry. Also, loss of apatite and monazite in zircons and intergrowth hydrothermal zircons indicating dry I-type magma. The mineralogical and isotopic data measured in these granites are consist with the typological study of their zircon population. There is good correlation between the genetic derived from zircon typology and the \(^{87}\text{Sr}/^{86}\text{Sr} \) whole-rock ratios, indicating survey of zircon morphology with good confidence level can use for determination magma source and evolution of magma as a simple and inexpensive method.

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