# ARTICLE IN PRESS

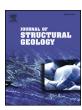
Journal of Structural Geology xxx (xxxx) xxx-xxx

ELSEVIER

Contents lists available at ScienceDirect

## Journal of Structural Geology

journal homepage: www.elsevier.com/locate/jsg



# Development of sigmoidal en-échelon microfractures in beryl and the role of strain localization evidenced by fluid inclusions

Graciela M. Sosa<sup>a,\*</sup>, Sebastián Oriolo<sup>b</sup>, Alfons van den Kerkhof<sup>a</sup>

- <sup>a</sup> Geoscience Center of the University of Goettingen, Goldschmidtstrasse 3, D-37077 Goettingen, Germany
- b CONICET Universidad de Buenos Aires. Instituto de Geociencias Básicas, Aplicadas y Ambientales de Buenos Aires (IGEBA), Intendente Güiraldes 2160, C1428EHA Buenos Aires, Argentina

#### ARTICLE INFO

## Keywords: Fluid-deformation interaction Fluid inclusions Hydrolytic weakening Crack initiation Healed microfractures Shear zone thickness

#### ABSTRACT

A combined fluid inclusion and microstructural study was carried out in beryl crystals from the San Cayetano Nb-Ta-bearing pegmatite (San Luis, Argentina). Primary aqueous-carbonic fluids ( $T_0$ ) were subsequently retrapped during shearing, resulting in en-échelon microfractures. The more brittle behaviour of beryl compared to quartz makes this mineral more suitable for the preservation of fluid inclusions and microstructures. The primary inclusions  $T_0$  are preserved in strain-free domains, whereas the pseudo-secondary  $T_1$ -to  $T_3$ -type inclusions occur in domains showing intracrystalline deformation.  $CO_2$  was relatively immobile or reacted to form carbonate in early  $T_1$ -type inclusions, whereas  $H_2O$  preferentially migrated along microfractures and was trapped as  $T_2$ -and  $T_3$ -type inclusions. Short intragranular trails of fluid inclusions, representing en-échelon healed microfractures, resulted from progressive strain localization. Contrary to previous proposals, this new model implies the progressive thickness reduction of intracrystalline micro-shear zones. Besides, hydrolytic weakening linked to dislocation glide is the most likely mechanism to explain the evolution of fluid inclusions, with intracrystalline deformation enhancing anisotropic diffusion. This study highlights the potential of combined fluid inclusion and microstructural studies in order to understand the interaction between fluid activity and deformation. In this way, valuable insights can be provided into the progressive development of overprinted fabrics and low-to medium-temperature deformation mechanisms of minerals.

## 1. Introduction

Sigmoidal en-échelon fractures and veins are key structures recording the coupling between fluid activity and rock deformation. They occur in a wide range of scales and have been extensively documented in natural examples, laboratory experiments and numerical simulations (e.g., Riedel, 1929; Closs, 1955; Beach, 1975; Pollard et al., 1982; Nicholson and Pollard, 1985; Ramsay and Huber, 1987; Olson and Pollard, 1991; Bons et al., 2012; Lisle, 2013; Thiele et al., 2015). These microfractures typically nucleate as tensional cracks parallel to the maximum compressive stress axis during simple shearing parallel to the vein array and rotate during progressive deformation, thus allowing the recognition of segments that underwent different amount of strain (e.g., Beach, 1975; Pollard et al., 1982; Bons et al., 2012; Lisle, 2013; Thiele et al., 2015). Several mechanisms have been proposed to explain the evolution of these structures (Beach, 1975; Nicholson and Pollard, 1985; Olson and Pollard, 1991; Thiele et al., 2015), though they may alternatively result from shearing of pre-existing veins (Bons et al., 2012 and references therein).

Although sigmoidal en-échelon fractures are common at macroscale, some investigations also refer to en-échelon microfractures, usually related to planar arrays (Bombolakis, 1973; Kranz, 1979, 1983; Anders et al., 2014; Cheng et al., 2015). These features comprise extensional fractures, which occur below the millimetre scale and allow fluid entrapment in related cavities (Anders et al., 2014 and references therein), providing important information about the interaction between fluid flow, rock strength, and fault zone nucleation and subsequent evolution (Kranz, 1979; Anders et al., 2014).

Over the last few years, fluid inclusions in beryl (e.g., Thomas et al., 2009) have been studied to characterize pegmatite-forming processes such as phase separation, liquid-liquid immiscibility and post-trapping crystallization and cooling rates, but so far the correlation between fluid inclusions and deformational features have not been considered. In the present study, sigmoidal en-échelon microfractures defined by intracrystalline fluid inclusion trails in beryl crystals are documented in detail. Based on microstructural analysis, microthermometry on fluid inclusions and Raman spectroscopy, a new mechanism for the evolution of intragranular sigmoidal en-échelon microfractures is proposed,

https://doi.org/10.1016/j.jsg.2018.04.005

Received 5 December 2017; Received in revised form 9 March 2018; Accepted 6 April 2018 0191-8141/ © 2018 Published by Elsevier Ltd.

<sup>\*</sup> Corresponding author.

E-mail address: gsosa@gwdg.de (G.M. Sosa).

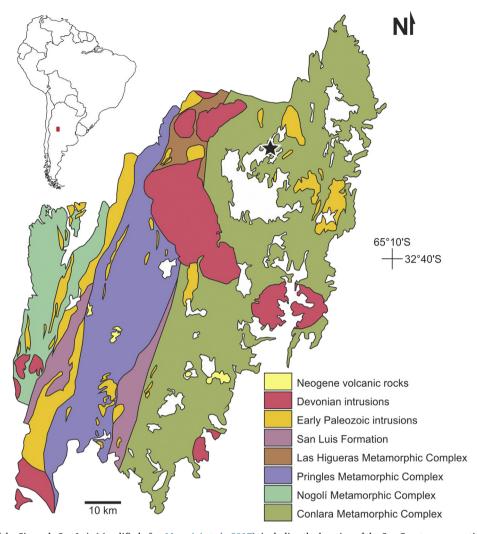


Fig. 1. Geological map of the Sierra de San Luis (simplified after Morosini et al., 2017), including the location of the San Cayetano pegmatite (black star). Inset shows the position of the Sierra de San Luis (red area) in South America. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

emphasizing the role of strain localization, and the coupling between deformation and fluid activity.

## 2. Materials and methods

The present study concentrates on fluid inclusions in beryl from the "San Cayetano" Nb-Ta-bearing pegmatite, located southwest of Las Aguadas, San Luis, Argentina (32° 23′ 7.6″S, 65° 31′ 11.7″W) (Fig. 1). The pegmatite is related to Ordovician synkinematic, meta-to peraluminous S-type granites (Ortiz Suárez and Sosa, 1991) and is concordantly emplaced parallel to the NNE-striking subvertical foliation of fine-grained quartz-mica schists, suggesting a late syn-kinematic emplacement during ductile deformation of the country rock (Sosa, 1992; Sosa et al., 2002). Based on similar field relationships, P-T conditions and the absence of metasomatism, Ortiz Suárez and Sosa (1991) concluded that the pegmatites were emplaced during the Ordovician, i.e. during or shortly after the peak of metamorphism and deformation. Magmatism, deformation and metamorphism can be attributed to the Ocloyic phase, which is part of the Famatinian orogenic cycle (e.g.; Ortiz Suárez et al., 1992; Sato et al., 2003; López de Luchi et al., 2007; Steenken et al., 2008).

The tabular pegmatitic body (ca.  $10 \times 100 \, \text{m}$ ) mainly consists of quartz, K-feldspar, plagioclase and muscovite. Beryl, tourmaline, garnet and apatite are present as accessory minerals, whereas columbite-

tantalite, pyrite, and bismutinite are the main ore minerals. The earliest pegmatite minerals crystallized at ca.  $500-400\,^{\circ}\text{C}$  and  $2-3\,\text{kbar}$  from an aqueous-carbonic fluid, preserved as primary inclusions in beryl and plagioclase (Sosa et al., 2014). These conditions are in agreement with regional metamorphism and the liquidus conditions of pegmatite crystallization.

Beryl in the San Cayetano pegmatite is pale green and occurs as randomly oriented euhedral to subhedral prismatic crystals. The crystals are normally ca. 1 cm in diameter, but larger crystals (more than 1 m in length) were found in other pegmatites.

Beryl usually contains clustered or isolated fluid inclusions, besides inclusions arranged along short trails or randomly oriented curved surfaces. The present sample contains disseminated regular inclusions as well as intragranular en-échelon fluid inclusion trails (Fig. 2). The latter are aligned along micro-shear zones that are perpendicular to the crystal length and record shearing approximately orthogonal to the crystallographic c-axis. In the adjacent quartz, these micro-shear zones can be traced as parallel bands of fluid inclusions but without en-échelon geometry (Fig. 3).

Phase transition temperatures of fluid inclusions in beryl were collected using a LINKAM THMS-600 heating-freezing stage. Temperature constraints were obtained for the melting of solid  $CO_2$  ( $T_m$   $CO_2$ ), ice melting ( $T_m$  ice),  $CO_2$  clathrate hydrate melting ( $T_m$   $CO_2$  hyd), partial homogenization of the carbonic phases ( $T_h$   $CO_2$ ), and total

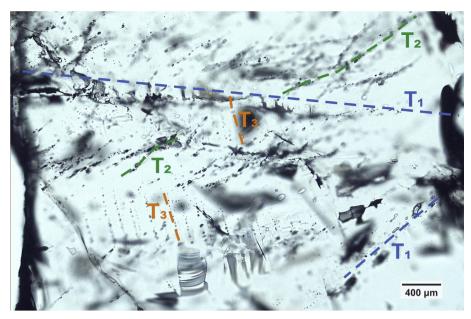


Fig. 2. Microphotographs showing spatial arrangement of T<sub>1</sub> to T<sub>3</sub>-type fluid inclusions.

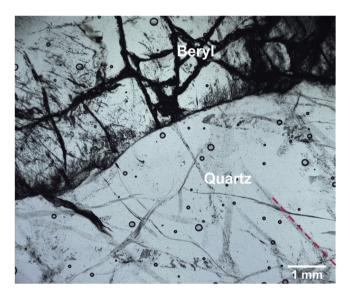


Fig. 3. Quartz crystal in contact with beryl showing bands of parallel fluid inclusions trails.

homogenization for aqueous ( $T_{ht}$   $H_2O$ ) and aqueous-carbonic inclusions ( $T_{ht}$   $H_2O$ - $CO_2$ ). The stage was calibrated with a set of synthetic fluid inclusion standards. The accuracy is better than 0.5 °C for temperatures below -56.6 °C, temperatures around 0 °C have accuracy of 0.2 °C, whereas for temperatures between 200 and 600 °C accuracy is better than 5 °C. Salinities were calculated from ice melting temperatures using the revised equations of Bodnar (2003).

Raman analyses were accomplished on selected fluid inclusions using a Horiba-Jobin-Yvon HR-Raman spectrometer provided with a 488 nm (blue) Laser and attached to an Olympus BX41 microscope. For quantitative measurements, spectra were acquired using a  $100 \times$  objective and spectra were collected between  $100 \, \mathrm{cm}^{-1}$  and  $4000 \, \mathrm{cm}^{-1}$ , integrating 3 cycles of  $30 \, \mathrm{s}$  for each measure.

## 3. Results

Four fluid inclusion types can be distinguished in beryl (Fig. 4), denoted by  $T_0$  to  $T_3$ . Primary aqueous-carbonic inclusions ( $T_0$ ) show

LLV or LV phase equilibria at room temperature and comprise ca. 14% of the total amount of fluid inclusions, whereas the rest corresponds to pseudo-secondary fluid inclusions with subsequently lower  $CO_2$  content. The latter fluid inclusions can be subdivided in  $(T_1)$  2-phase aqueous-carbonic inclusions (14%) with some  $CO_2$  as evident from solid  $CO_2$  and clathrate melting,  $(T_2)$  aqueous inclusions (29%) with minor  $CO_2$  (only clathrate melting observed), and  $(T_3)$  2-phase aqueous inclusions (43%) without demonstrable  $CO_2$ . Phase transition temperatures and fluid properties are summarized in Table 1.

Primary aqueous-carbonic fluid inclusions  $(T_0)$  show varying water volume fractions (0.6--0.9) and have an average size of  $23\,\mu\text{m}$ . They occur in clusters, along crystal growth zones, or sometimes as isolated inclusions (Fig. 5a). In inclusions showing a double meniscus at room temperature,  $CO_2$  always homogenizes into the vapour phase. Melting of solid  $CO_2$  occurs between -56.9 and  $-56.6\,^{\circ}\text{C}$ , whereas  $CO_2$  homogenization temperature ranges between 17.9 and  $28.2\,^{\circ}\text{C}$ . The bulk  $CO_2$  content can be estimated at ca. 6 mol %. Small amounts of  $N_2$  are present, as demonstrated by Raman spectroscopy. Clathrate hydrate melting varies between 8.9 and  $9.9\,^{\circ}\text{C}$ . Total homogenization of inclusions varies between 258 and 284 °C and the salinity of the inclusions corresponds to 3--6 wt. % NaCl.

Pseudo-secondary  $T_1$  inclusions are always two-phase at room temperature with varying water volume fractions between 0.80 and 0.95 (Fig. 5b), and arranged along intragranular trails. The inclusions are normally irregular, indicating late modification (decrepitation) (Fig. 6a). Some inclusions contain daughter crystals, i.e. siderite, goethite and cristobalite, as demonstrated by Raman spectroscopy (Fig. 6). Partial homogenization of the carbonic phase could not be observed, whereas final melting of solid  $CO_2$  could be only incidentally observed. Therefore, the  $CO_2$  content must be lower compared to  $T_0$  and estimated < 6 mol %. Small amounts of  $N_2$  are present as indicated by the lower  $CO_2$  melting temperatures and Raman spectroscopy. Clathrate hydrate melts between 8.1 and 9.6 °C. Total homogenization of  $T_1$  inclusions varies between 236 and 266 °C and the salinity corresponds to 4–5 wt. % NaCl.

Pseudo-secondary  $T_2$ -type inclusions are also two-phase at room temperature, showing water volume fractions of 0.85 (Fig. 5c). These inclusions are regularly shaped and have an average size of 15  $\mu$ m. The bulk  $CO_2$  content can be estimated as < 3 mol%. The presence of minor  $CO_2$  is demonstrable only from clathrate melting between 7.3 and 13.0 °C. Total homogenization temperatures of inclusions vary between

G.M. Sosa et al.

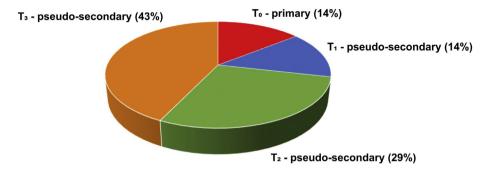


Fig. 4. Frequency of fluid inclusion types.

Table 1 Overview of fluid inclusion properties. Homogenization of carbonic inclusions ( $T_h$  CO<sub>2</sub>) always to vapour (V), melting of solid CO<sub>2</sub> ( $T_m$  CO<sub>2</sub>), CO<sub>2</sub> clathrate hydrate melting ( $T_m$  CO<sub>2</sub> hyd), salinity in NaCl wt. %, and total homogenization of inclusions (Tht).

Fluid inclusion type	$T_h$ $CO_2$ to vapour (°C)	$T_m CO_2$ (°C)	$T_m$ CO $_2$ hyd (°C)	Salinity (NaCl wt.%)	Bulk CO <sub>2</sub> (mol %)	T <sub>ht</sub> (°C)
T <sub>0</sub>	17.9 to 28.2	−56.9 to −56.6	8.9 to 9.9	3 to 6	ca. 6	258 to 284
$T_1$	n.o.	-57.3 to $-56.6$	8.1 to 9.6	4 to 5	< 6	236 to 266
$T_2$	n.o.	n.o.	7.3 to 13.0	2 to 6	< 3	236 to 277
$T_3$	n.o.	n.o.	n.o.	1 to 5	0	166 to 206

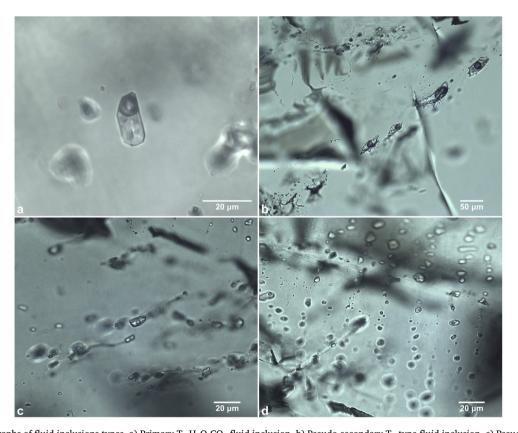


Fig. 5. Microphotographs of fluid inclusions types. a) Primary  $T_0$   $H_2O$ - $CO_2$  fluid inclusion. b) Pseudo-secondary  $T_1$ -type fluid inclusion. c) Pseudo-secondary  $T_2$ -type inclusions. d) Pseudo-secondary  $T_3$ -type aqueous inclusions.

236 and 277  $^{\circ}\text{C}$  and the salinity of the inclusions corresponds to 2–6 wt. % NaCl.

Aqueous  $T_3$ -type inclusions are two-phase at room temperature and typically occur along parallel trails (Fig. 5d). They show regular shapes and have an average size of ca. 8  $\mu$ m. Total homogenization temperatures of the inclusions vary between 166 and 206 °C and the salinity of the inclusions is 1–5 wt. % NaCl.

Representative fluid inclusions were used for estimating the P-T trapping conditions from isochores calculated with the ISOC software

package of Bakker (2003). The fluid-trapping conditions (Fig. 7) are constrained by the metamorphic conditions of 400–500 °C and 2–3 kbar, determined for the country rock (Ortiz Suárez and Sosa, 1991). The isochores for  $\rm H_2O\textsc{-NaCl-CO}_2$  corresponding to the primary inclusions ( $\rm T_0$ ) run at significantly higher P-T conditions compared to the later  $\rm T_1$ -  $\rm T_3$ . The trapping conditions largely remain above the  $\rm H_2O\textsc{-CO}_2$   $\pm$  NaCl solvus and therewith stating homogeneous fluid trapping. A lithostatic paleo-geothermal gradient of 30 °C/km delimits the minimal trapping conditions, whereas the actual geothermal gradient

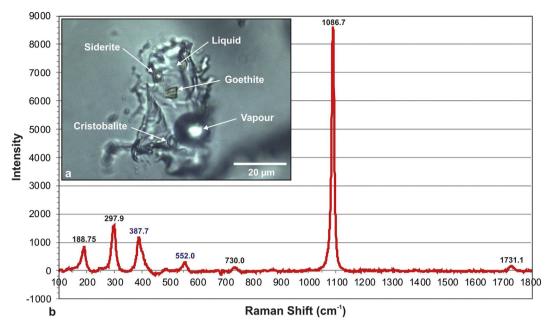


Fig. 6. a) Re-equilibrated T1-type fluid inclusion showing decrepitation textures with volume shrinkage and daughter crystals. Daughter crystals (siderite, cristobalite, goethite) and vapour  $(CO_2, N_2)$  compositions were determined by Raman spectroscopy. b) Raman spectrum of siderite daughter crystal, with subordinated peaks of goethite (blue). (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

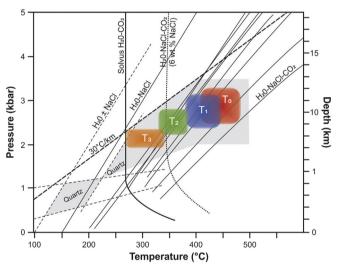


Fig. 7. Crystallization conditions for beryl (boxes marked  $T_0$  to  $T_3$ ) and quartz (shaded area) based on fluid inclusion data. Selected isochores for  $H_2O$ -NaCl-CO $_2$  and  $H_2O$ -NaCl for fluid inclusions in beryl (solid lines) and in quartz (dashed lines), calculated after Bakker (2003). Miscibility limits (solvus) are shown for salt-free  $H_2O$ -CO $_2$  and for  $H_2O$ -CO $_2$ -NaCl (6 wt% NaCl; Krüger and Diamond, 2001). P-T trapping conditions of primary fluid inclusions in beryl are constrained by the metamorphic conditions of the country rock. The minimal trapping conditions are delimited by a lithostatic geothermal gradient of 30 °C/km.

must have varied between ca. 50 and ca.  $30\,^{\circ}\text{C/km}$  for  $T_0$  towards  $T_3$ . Fig. 7 also shows that fluid inclusions in beryl did not form below ca.  $300\,^{\circ}\text{C}$ , whereas re-equilibration and fluid (re)trapping must have continued in quartz during cooling below ca.  $200\,^{\circ}\text{C}$ .

## 4. Discussion

Several models have been proposed for the genesis of en-échelon structures (Riedel, 1929; Closs, 1955; Beach, 1975; Pollard et al., 1982; Nicholson and Pollard, 1985; Ramsay and Huber, 1987; Olson and

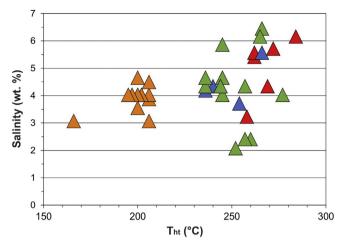
Pollard, 1991; Bons et al., 2012; Lisle, 2013; Thiele et al., 2015). Thiele et al. (2015) referred to strain localization as an active process in enéchelon vein formation, favouring the coalescence of several small veins into fewer larger ones with time. However, strain localization and strain hardening/softening, which may trigger changes of shear zone thickness with time (e.g., Vitale and Mazzoli, 2008), have been not considered so far, though they are key processes occurring in the lithosphere at all scales, supported by both natural and numerical data (e.g., Sibson, 1983; Vitale and Mazzoli, 2008; Platt and Behr, 2011; Montési, 2013; Oriolo et al., 2015).

In the studied beryl samples, pseudo-secondary fluid inclusion types  $T_1$  to  $T_3$  are arranged along intragranular trails representing healed microfractures (Fig. 2). The microstructures show multi-stage deformation, resulting in the transposition of fluid inclusion intragranular trails, and forming subparallel en-échelon short trails. Microthermometric properties of fluid inclusions within one trail are almost the same, but clear differences are observed for the type  $T_1$  -  $T_3$  pseudo-secondary fluid inclusions, i.e., decreasing  $CO_2$  content, together with lower total homogenization temperatures and slightly lower salinity (Table 1).

The primary fluid inclusions show highest total homogenization temperatures. Similar temperatures are recorded for the pseudo-secondary  $T_1$ -type inclusions, which were trapped after the opening and subsequent healing of microfractures. In addition, implosion-decrepitation textures of  $T_1$  fluid inclusions demonstrate volume shrinkage due to fluid underpressure (Fig. 6a), probably as a result of isobaric cooling (Van den Kerkhof and Hein, 2001). On the other hand,  $T_2$  and  $T_3$  inclusions show subsequent lower homogenization temperatures (Fig. 8). Besides, aqueous inclusions have somewhat lower salinity than the aqueous-carbonic ones (Table 1). Salinities of  $T_0$  primary aqueous-carbonic inclusions range from 3 to 6 wt % NaCl, whereas salinities of  $T_3$  aqueous inclusions range from 3 to 5 wt. % NaCl. The presence of  $T_3$  aqueous inclusions range from 3 to 5 wt. % NaCl. The presence of  $T_3$  aqueous inclusions range from 3 to 5 wt. % NaCl. The presence of  $T_3$  aqueous inclusions range from 3 to 5 wt. % NaCl. The presence of  $T_3$  aqueous inclusions range from 3 to 5 wt. % NaCl. The presence of  $T_3$  aqueous inclusions range from 3 to 5 wt. % NaCl. The presence of  $T_3$  aqueous inclusions range from 3 to 5 wt. % NaCl. The presence of  $T_3$  aqueous inclusions range from 3 to 5 wt. % NaCl. The presence of  $T_3$  aqueous inclusions range from 3 to 5 wt. % NaCl. The presence of  $T_3$  and  $T_3$  inclusions the presence of  $T_3$  and  $T_3$  inclusions range from 3 to 5 wt. % NaCl. The presence of  $T_3$  and  $T_3$  inclusions range from 3 to 5 wt. % NaCl. The presence of  $T_3$  and  $T_3$  inclusions range from 3 to 5 wt. % NaCl. The presence of  $T_3$  and  $T_3$  inclusions range from 3 to 5 wt. % NaCl. The presence of  $T_3$  and  $T_3$  inclusions range from 3 to 5 wt. % NaCl.

Instead of the classical hypothesis implying constant shear zone thickness, an alternative model of en-échelon structure development implying progressive strain localization and consequent shear zone thickness decrease is proposed (Fig. 9). It is inferred that  $T_1$  fluid inclusions resulted from the nucleation and early evolution of both microshear zones and en-échelon microfractures (Fig. 9a), whereas  $T_2$  and  $T_3$ 

G.M. Sosa et al.



**Fig. 8.** Total homogenization temperature vs. salinity of fluid inclusion types.  $T_0$ : red,  $T_1$ : blue,  $T_2$ : green,  $T_3$ : orange. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

inclusions reveal further deformation along progressively thinner microshear zones, leading to the local overprint of en-échelon microstructures (Fig. 9b and c). Although  $T_1$  en-échelon microfractures remain relatively undeformed during  $t_2$  and  $t_3$ , probably due to a lack of lateral propagation of  $t_2$  and  $t_3$  micro-shear zones, decrepitation microstructures typical for  $T_1$  inclusions may record post-entrapment reequilibration, due to internal underpressure with a subsequent entrapment of  $T_2$  and/or  $T_3$  inclusions. For the genesis of  $T_3$  inclusions, two alternative interpretations are considered (Fig. 9). In one case,  $T_3$  might result from further sinistral shearing, giving rise to opening and subsequent counterclockwise rotation of  $T_3$  microfractures ( $c_1$ ). Alternatively,  $T_3$  microfractures might arise from a change in the shearing sense ( $c_2$ ). In any case, however, strain localization might play a major role.

Consequently, the segments that were generated during early stages of deformation and were subsequently sheared do not contain primary fluid inclusions, but, instead, are largely overprinted by fracture healing and fluid trapping during later deformation, recorded by later fluid

inclusions (Fig. 9; Herwegh et al., 2008). Similarly, segments that are parallel to the maximum compressive stress represent low-strain domains that remained relatively undisturbed during the final stage of deformation and, thus, record the earlier fluid inclusion generation, instead of the expected last generation. In other words, shearing during a certain stage n gives rise to  $T_n$  fluid inclusion trails in high-strain domains, whereas the previous n-1 stage is recorded only by  $T_{n-1}$  inclusions in low-strain domains.

Fluid inclusion and related microstructures provide insights into coupled fluid-deformation processes. Primary inclusions are preserved in relatively strain-free domains, whereas pseudo-secondary fluid inclusions can be observed in segments affected by intracrystalline deformation, similarly to quartz inclusions reported by Kerrich (1976).  $\rm CO_2$  remained immobile or reacted to form siderite in  $\rm T_1$ -type inclusions. Water might migrate more easily and was trapped as  $\rm T_2$ -and  $\rm T_3$ -type inclusions, as experimentally demonstrated for quartz (e.g., Baumgartner et al., 2014), thus accounting for the progressive loss of  $\rm CO_2$  observed for type  $\rm T_1$  to  $\rm T_3$  inclusions. Therefore, hydrolytic weakening seems to be the most plausible mechanism to explain these results.

Hydrolytic weakening plays an important role for the deformation of quartz (Griggs, 1967, 1974), but may also play a role for other silicates which contain hydroxyl in their crystal structure (Kirby et al., 1990). Beryl normally contains water mostly in structural channels, even when the replacement of Si by OH cannot be excluded. Therefore, the effect of water in beryl is assumed to be minor, compared to minerals like quartz. The results of Kirby et al. (1990) indicate that beryl is compositionally, thermally, and mechanically unstable in the vicinity of crack surfaces at temperatures higher than 177 °C. Water promotes crack growth via processes that involve chemical interactions of hydrogen species and the stressed bonds at crack tips. The mobility of water, in turn, is particularly efficient along microcracks and, consequently, dislocation gliding related to development of micro-shear zones and associated en-échelon microfractures might provide ideal channels for water diffusion (Bakker and Jansen, 1990, 1994; Heggie, 1992; Boullier, 1999; Bakker, 2009). Hence, hydrolytic weakening is strongly linked to deformation mechanisms (i.e., dislocation glide), as the latter enhance anisotropic diffusion (Bakker, 2009).

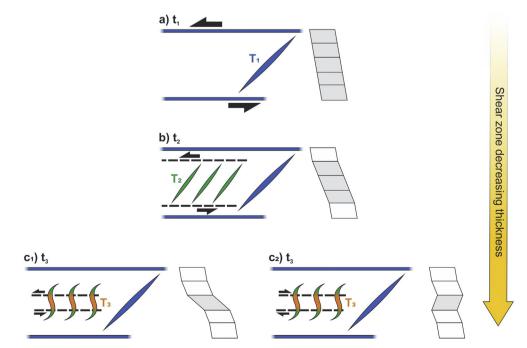


Fig. 9. Sketch showing generation of intragranular en-échelon structures involving progressive decrease of shear zone thickness and strain localization. Dotted lines indicate the overprinted high-strain area affected by shearing. To the right, the scheme shows overprinted areas (grey) and low-strain domains (white). Note that T1 remains relatively undeformed during t2 and t3, probably due to a lack of lateral continuity of to and t3 micro-shear zones. Nevertheless, T1 fluid inclusions are affected during these latter events, as recorded by decrepitation textures. For T3, two alternative models are presented (c1: further sinistral shearing, giving rise to opening and subsequent counterclockwise rotation of T3; c2: reverse shearing).

#### 5. Final remarks

Data obtained from fluid inclusions along intragranular trails show that fluids became progressively less saline, and the temperature gradually decreased with the progressive evolution of the deformation during beryl crystallization, contemporaneously with pegmatite emplacement under oxidizing conditions. Primary  $T_0$  inclusions are preserved in relatively strain-free domains, whereas pseudo-secondary ( $T_1$  to  $T_3$ ) fluid inclusions are observed in areas affected by intracrystalline deformation.  $CO_2$  is more or less immobile or reacted to form siderite in early  $T_1$ -type inclusions, whereas water migrated more easily along microfractures and was trapped as late  $T_3$ -type inclusions.

Short trails of en-échelon fluid inclusions represent microfractures that were repeatedly opened and sealed during deformation. These microstructures formed as a result of progressive strain localization and consequent decrease in the thickness of microshear zones. In contrast to previous proposals implying constant shear zone thickness, this model emphasizes the role of strain localization during the genesis of en-échelon healed microfractures.

The present synkinematic microstructures highlight the role of fluid inclusion studies in structural geology and show how fluid inclusions can be applied in tracing the nature and compositional evolution of geological fluids with changing physico-chemical conditions during deformation and fluid trapping. In turn, the microstructural characterization provides a robust framework to understand fluid evolution in "strain-dominated" settings. Hence, the combination of fluid inclusion and microstructural studies provide irreplaceable information to understand the coupling of fluids and deformation, as stated by Hollister (1990) and Anders et al. (2014). This approach might be powerful to evaluate overprinted fabrics (e.g., Herwegh et al., 2008) and, particularly, low-to medium-temperature deformation mechanisms, not only in quartz, but also in other silicates like beryl, which have been so far poorly explored (e.g., Derez et al., 2015).

## Acknowledgements

The authors would like to thank Dr. Axel Vollbrecht for his constructive comments. The critical and detailed reviews of Prof. J.L.R. Touret and Dr. B. Leiss significantly contributed to improve the manuscript.

## References

- Anders, M.H., Laubach, S.E., Scholz, C.H., 2014. Microfractures: a review. J. Struct. Geol. 69, 377–394.
- Bakker, R.J., 2003. Package FLUIDS 1. Computer programs for analysis of fluid inclusion data and for modelling bulk fluid properties. Chem. Geol. 194, 3–23.
- Bakker, R.J., 2009. Reequilibration of fluid inclusions: bulk-diffusion. Lithos 112, 277–288.
- Bakker, R.J., Jansen, J.B.H., 1990. Preferential water leakage from fluid inclusions by means of mobile dislocations. Nature 345, 58–60.
- Bakker, R.J., Jansen, J.H.B., 1994. A mechanism for preferential H<sub>2</sub>O leakage from fluid inclusions in quartz, based on TEM observations. Contrib. Mineral. Pet. 116, 7–20.Baumgartner, M., Bakker, R.J., Doppler, G., 2014. Re-equilibration of natural H2O-CO2-salt-rich fluid inclusions in quartz Part 1: experiments in pure water at constant pressures and differential pressures at 600°C. Contrib. Mineral. Pet. 168, 1017.
- Beach, A., 1975. The geometry of en-echelon vein arrays. Tectonophysics 28, 245–263.
   Bodnar, R.J., 2003. Introduction to aqueous electrolyte fluid inclusions. In: In: Samson, I.,
   Anderson, A., Marshall, D. (Eds.), Fluid Inclusions: Analysis and Interpretation, vol.
   32. Mineralogical Association of Canada Short Course, pp. 81–100.
- Bombolakis, E.G., 1973. Study of the brittle fracture process under uniaxial compression. Tectonophysics 18, 231–248.
- Bons, P.D., Elburg, M.A., Gómez-Rivas, E., 2012. A review of the formation of tectonic veins and their microstructures. J. Struct. Geol. 43, 33–62.
- Boullier, A.-M., 1999. Fluid inclusions: tectonic indicators. J. Struct. Geol. 21, 1229–1235
- Cheng, Y., Wong, L.N.Y., Zou, C., 2015. Experimental study on the formation of faults from en-echelon fractures in Carrara Marble. Eng. Geol. 195, 312–326.
- Closs, E., 1955. Experimental analysis of fracture patterns. Bull. Geol. Soc. Am. 66,

- 241-258
- Derez, T., Pennock, G., Drury, M., Sintubin, M., 2015. Low-temperature intracrystalline deformation microstructures in quartz. J. Struct. Geol. 71, 3–23.
- Griggs, D., 1967. Hydrolytic weakening of quartz and other silicates. Geophys. J. Int. 14, 19–31.
- Griggs, D., 1974. A model of hydrolytic weakening in quartz. J. Geophys. Res. 79, 1653–1661.
- Heggie, M., 1992. A molecular water pump in quartz dislocations. Nature 355, 337–339.
   Herwegh, M., Berger, A., Ebert, A., Brodhag, S., 2008. Discrimination of annealed and dynamic fabrics: consequences for strain localization and deformation episodes of large-scale shear zones. Earth Planet. Sci. Lett. 276, 52–61.
- Hollister, L.S., 1990. Enrichment of CO<sub>2</sub> in fluid inclusions in quartz by removal of H<sub>2</sub>O during crystal-plastic deformation. J. Struct. Geol. 12, 895–901.
- Kerrich, R., 1976. Some effects of tectonic recrystallization on fluid inclusions in vein quartz. Contrib. Mineral. Pet. 59, 195–202.
- Kirby, S., Hemingway, B.S., Lee, R., 1990. Anomalous fracture and thermal behavior of hydrous minerals. In: Duba, A.G., Durham, W.B., Handin, J.W., Wang, H.F. (Eds.), The Brittle-ductile Transition in Rocks. American Geophysical Union. http://dx.doi. org/10.1029/GM056p0119.
- Kranz, R.L., 1979. Crack growth and development during creep of Barre granite. Int. J. Rock Mech. Min. Sci Geomech. Abstr. 16, 23–35.
- Kranz, R.L., 1983. Microcracks in rocks: a review. Tectonophysics 100, 449-480.
- Krüger, Y., Diamond, L., 2001. P-V-T-X properties of two H<sub>2</sub>O-CO<sub>2</sub>-NaCl mixtures up to 850°C and 500 MPa: synthetic fluid inclusion study. In: In: Noronha, F., D'pria, A., Guedes, A. (Eds.), XVI ECROFI, Porto, 2001, Abstracts. Faculdade de Ciéncias de Porto, Departamento de Geologia, vol. 7. pp. 241–244 Memória.
- Lisle, R.J., 2013. Shear zone deformation determined from sigmoidal tension gashes. J. Struct. Geol. 50, 35–43.
- López de Luchi, M., Siegesmund, S., Wemmer, K., Steenken, A., Naumann, R., 2007. Geochemical constraints on the petrogenesis of the Paleozoic granitoids of the Sierra de San Luis, Sierras Pampeanas, Argentina. J. S. Am. Earth Sci. 24, 138–166.
- Montési, L.G.J., 2013. Fabric development as the key for forming ductile shear zones and enabling plate tectonics. J. Struct. Geol. 50, 254–266.
- Morosini, A.F., Ortiz Suárez, A.E., Otamendi, J.E., Pagano, D.S., Ramos, G.A., 2017. La Escalerilla pluton, San Luis Argentina: the orogenic and post-orogenic magmatic evolution of the famatinian cycle at Sierras de San Luis. J. S. Am. Earth Sci. 73, 100–118.
- Nicholson, R., Pollard, D.D., 1985. Dilation and linkage of echelon cracks. J. Struct. Geol. 7, 583–590.
- Olson, J.E., Pollard, D.D., 1991. The initiation and growth of en echelon veins. J. Struct. Geol. 13, 595–608.
- Oriolo, S., Oyhantçabal, P., Heidelbach, F., Wemmer, K., Siegesmund, S., 2015. Structural evolution of the Sarandí del Yí Shear Zone: kinematics, deformation conditions and tectonic significance. Int. J. Earth Sci. 104, 1759–1777.
- Ortiz Suárez, A., Sosa, G.M., 1991. Relaciones genéticas entre las pegmatitas portadoras de estaño y las metamorfitas asociadas en la zona de La Carolina/San Francisco del Monte de Oro. Prov. San Luis. Rev. Asoc. Geol. Arg 46, 339–343.
- Ortiz Suárez, A., Prozzi, C., Llambías, E., 1992. Geología de la parte Sur de la Sierra de San Luis y granitoides asociados, Argentina. Rev. Estud. Geol. 48, 269–277.
- Platt, J.P., Behr, W.M., 2011. Grainsize evolution in ductile shear zones: implications for strain localization and the strength of the lithosphere. J. Struct. Geol. 33, 537–550.
- Pollard, D.D., Segall, P., Delaney, P.T., 1982. Formation and interpretation of dilatant echelon cracks. Bull. Geol. Soc. Am. 93, 1291–1303.
- Ramsay, J.G., Huber, M.I., 1987. The techniques of modern structural geology. In: Folds and Fractures, vol. 2 Academic Press, London.
- Riedel, W., 1929. Zur Mechanik geologischer Brucherscheinungen. Zbl. Mineral. Geo. Pal. B 354–368.
- Sato, A.M., González, P., Llambías, E., 2003. Evolución del orógeno Famatiniano en la Sierra de San Luis: magmatismo de arco, deformación y metamorfísmo de bajo a alto grado. Rev. Asoc. Geol. Argent. 58, 487–504.
- Sibson, R.H., 1983. Continental fault structure and the shallow earthquake source. J. Geol. Soc. Lond. 140, 741–767.
- Sosa, G., 1992. Pegmatitas portadoras de niobio y tantalio, Sierra San Luis, Argentina. Congreso Geología Económica, Córdoba, Argentina.
- Sosa, G.M., Augsburger, M.S., Pedregosa, J.C., 2002. Columbite-group minerals from raremetal granitic pegmatites of the Sierra de San Luis, Argentina. Eur. J. Mineral. 14, 627–636.
- Sosa, G.M., van den Kerkhof, A.M., Wemmer, K., Lüders, V., Plessen, B., Montenegro, T., 2014. Fluid inclusions, isotopic fluid composition and new age datings of rare-metal pegmatites, Sierra de San Luis, Argentina. Latin American Colloquium, Heidelberg.
- Steenken, A., Siegesmund, S., Wemmer, K., Lopez de Luchi, M.G., 2008. Time constraints on the Famatinian and Achalian structural evolution of the basement of the Sierra de San Luis (Eastern Sierras Pampeanas, Argentina). J. S. Am. Earth Sci. 25, 336–358.
- Thiele, S.T., Micklethwaite, S., Bourke, P., Verrall, M., Kovesi, P., 2015. Insights into the mechanics of en-échelon sigmoidal vein formation using ultra-high resolution photogrammetry and computed tomography. J. Struct. Geol. 77, 27–44.
- Thomas, R., Davidson, P., Badanina, E., 2009. A melt and fluid inclusion assemblage in beryl from pegmatite in the Orlovka amazonite granite, East Transbaikalia, Russia: implications for pegmatite-forming melt systems. Mineral. Pet. 96, 129–140.
- Van den Kerkhof, A.M., Hein, U.F., 2001. Fluid inclusion petrography. Lithos 55, 27–47.
  Vitale, S., Mazzoli, S., 2008. Strain analysis of heterogeneous ductile shear zones based on the attitudes of planar markers. J. Struct. Geol. 32, 321–329.