

## Melt inclusions texture and thermal history in minerals from the Dej Tuff, Transylvania basin, Romania

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**Abstract:** This paper is based upon petrography and microthermometry and shows the presence of “foamy/bubbly” silicate melt inclusions in quartz, feldspar and zircon from the “Dej Tuff” that crystallized in magma chamber before and/or during the plinian- and plinian- phreatomagmatic explosive volcanic activity occurred during early Badenian (14.8-15.1 Ma) in the NW-Transylvania Basin. The “foam-like” silicate glass inclusions” were trapped as homogeneous and/or heterogeneous silicate melt, now consisting of a package of tiny bubbles occupying up to 75-80% in the silicate glass volume. The homogenization temperature determined in the heating stage at one atmosphere, by vapor bubble(s) disappearance (i.e. the minimum trapping temperature), in the silicate melt phase ranged between 710°-900°C ( $\pm 20^\circ\text{C}$ ), with maximum of frequency between 750° to 830°C (n=344). These values are in very good agreement with temperature calculated by common geothermometers such as Fe-Ti oxide, Zr saturation temperature,  $\Delta^{18}\text{O}$  (Qz-Mt) etc, applied in some classical rhyolitic magmas. The trapping pressure of the “foam-like” silicate glass inclusions” from “Dej Tuff’s phenoclasts and phenocrysts was estimated between 200 MPa and 20 MPa, based upon spontaneous homogeneous vs. heterogeneous bubble nucleation during heating in the microthermometric stage. It is suggested in this study that “foam-like” silicate glass inclusions is an adequate term for the silicate melt inclusions described in “Dej Tuff” phenoclasts, beside “hourglass inclusions” and common silicate melt ones. The silicate melt was probably trapped during crystals growing in a shallow foam layer before eruption in the top of a molten magma chamber that ultimately was erupted explosively as probably a result of a hotter mafic influx at its bottom region.

**Keywords:** “foam-like” glass inclusions”, microthermometry, Badenian, “Dej Tuff”

### 1 Introduction

The silicate melt inclusions containing more than one shrinkage bubble at the room temperature conditions were described early in the history of the subject since Sorby (1858), Vogelsang (1869) and Zirkel (1893). It should be noticed that the serial drawings no. 90 and 91 from the XIX<sup>th</sup> plate of Sorby’s mentioned paper could be taken as “the first microthermometric cycle” presented in a published paper worldwide about silicate melt inclusions. There was estimated a temperature range of 800° -1000°C, based upon the color of the experimentally-heated augite crystal from Vesuvius. More

recently Roedder (1979) presented a piece of feldspar from the “Icelandic tuff” in which a “foam-like” silicate melt inclusion assemblage was formed. The most important and convincing data on the topic were published on microthermometry and petrography of silicate glass inclusions trapped in volcanic quartz worldwide by Clocchiatti (1975). This author presented original serial drawings and pictures of sequential microthermometry on “ponce” silicate glass inclusions, and recorded their features during heating, including, in many cases, the final homogenization.

Kamenetsky and Danyushevsky (2005) studied quartz crystals from the Taupo Volcanic

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Zone (New Zealand) and revealed that bubbles nucleated in the melt inclusions at the  $\alpha$ - $\beta$  transition temperature (573°C) and dissolved completely at 820-850°C (homogenization). In this study we also observed at the quartz transition temperature an enhancement of bubble nucleation, but many bubbles were earlier nucleated coincidentally with  $T_g$  values around 500-530°C (glass transition temperature in rhyolites, e.g., Bagdassarov et al., 1996), and the microtexture of the silicate foam became visible even in the apparently “bubble-free” glass inclusions at room temperature. The “Bishop Tuff” formation in the western USA is another classic example of rhyolitic explosive products with a large amount of published data during the last decades by Anderson (1991, 2003), Skirius et al. (1990), Peppard et al. (2001), Wallace et al., (2003) on glass inclusions trapped in quartz phenocrysts and phenoclasts. The term phenoclast used in this study, mainly from the quartz and feldspar broken crystals, was taken from Best and Christiansen (1997) which stated that: “Rapidly erupted Plinian pyroclasts that form ash-fall deposits mix with cool atmosphere and thus generally quench before volatile dissolved in melt inclusions can nucleate bubbles and blow their host crystal apart”. Further information about phenoclasts generated by fragmentation of the magmatic crystals was taken from Bindeman (2005), who reported that during explosive volcanic eruptions phenocrysts fragmentation was caused mainly by decrepitation of silicate melt inclusions and the resulted crystal fragments were defined as phenoclasts. A volcanic rock sample containing fragments of feldspar and rarely quartz from Amiata volcano, Italy described firstly as “reignimbrite” by Rittman (1936), was used here for comparison. Data on melt inclusions in rhyolite or dacite worldwide were also given by Johnson et al. (1994), Manley (1996), Lowenstern (1995, 2003), Frezzotti (2001), Naumov et al. (1993), Cesner (1998) and many others.

The study of “foam-like” glass inclusions from “Dej Tuff” phenoclasts shown that they

could be used to estimate trapping temperature by homogenizing in the heating stage under the microscope, if we related them to an enclosed silicate foam rather than a common silicate melt, which normally shown only one contraction bubble at room temperature condition

## 2 Geological setting

The geological formation known as “Dej Tuff” was defined by Pošepny (e.g., Szakács, 2000) in the NW part of the Transylvanian Basin (Fig. 1) and was deposited during the climactic plinian or plinian-phreatomagmatic volcanic activity in the Badenian time (14.8-15.1 Ma - Szakács et al., 2012). There are three different acidic tuffs sequences spread in the Transylvanian Basin (NW-Romania). Their lithofacial features suggest that majority of the “Dej Tuff” tephra has been reworked and redeposited after their original deposition in seawater environment (Szakács, 2000). The caldera (s) that generated such large volume volcanoclastic material is localized probably in the NW-Transylvanian Basin between the Vihorlat Mountains (Ukraine) and Gutâi Mountains from Romania (Szakács, 2000; Fülöp, 2002). Data on petrochemistry mineralogy, litho-stratigraphy and timing, were presented by Mârza and Meszaros, 1991; Mârza et al., 1991a,b; Szakács, 2000, Szakács et al., 2012 and references therein.

The mineralogical composition of the “Dej Tuff” includes quartz, plagioclase as main rock forming minerals and minor components represented by K- feldspar and plagioclase, amphibole, pyroxene, and with Fe-Ti oxides, zircon, apatite and allanite as accessory minerals, rarely completed by monazite and xenotime. Various lithoclasts of magmatic or nonmagmatic origin (metamorphic, sedimentary) are frequent observed and described (Szakács, 2000, 2003).

For this study, quartz and feldspar phenoclasts and rarely phenocrysts were extracted simply by crushing of pumice fragments, ignimbrites and unwelded tephra collected from Șoimeni, Pâglișa, Jichișul de Sus,

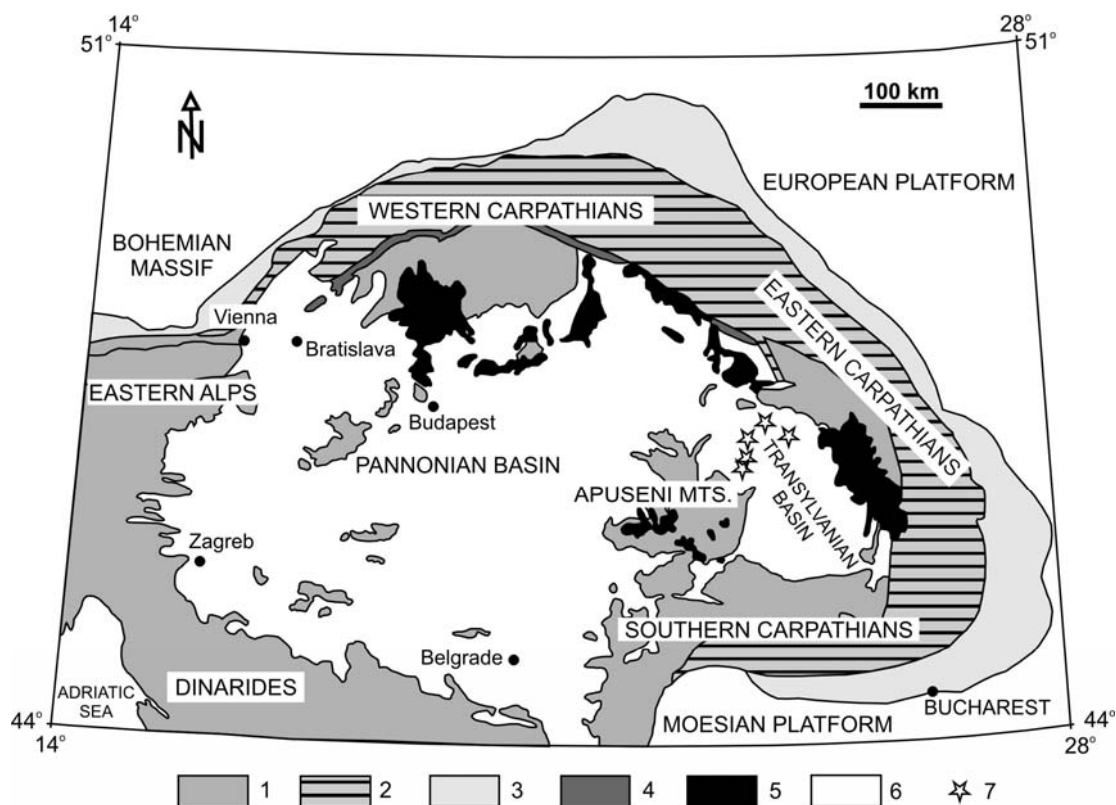


Fig. 1. Geological sketch map of Carpathian-Pannonian region (after Seghedi et al., 2004), showing the location of the samples used in this study. 1: Inner Alpine-Carpathian belt and Dinarides; 2: Alpine-Carpathian Flysch belt; 3: Molasse; 4: Pieniny Klippen belt; 5: Outcropping calc-alkaline volcanic rocks; 6: Neogene-Quaternary sedimentary deposits; 7: Sample location.

Măgura Ciceului and Ceparu localities. Zircon microcrysts were also collected from several ignimbrite and pumice samples. All these samples were selected to identify and describe melt inclusion microtexture and thermal history features by using petrography and microthermometry. Further silicate melt inclusion study is needed to elucidate the thermal history of each sequence in the “Dej Tuff” tephra.

### 3 Melt inclusion petrography

Doubly-polished thin sections were made on rock samples and/or phenocrasts. The crystals were simply hand-picked under the binocular microscope. They consist of quartz and/or feldspar grains (0.5 to 3 - 4 mm) and zircon microcrysts (200 – 500 microns in length).

Five types of melt and fluid inclusions were observed and described at the room temperature (phase assemblage), as follows: mono-, and biphasic glass inclusions, multiphase glass

inclusions, vapor-rich inclusions, opaque globular inclusions and microphenocrysts (microlites).

#### 3.1 Monophasic and biphasic glass inclusions

A common silicate glass inclusion type described in many papers (e.g., Anderson, 2003) is a glass inclusion apparently “bubble-free” at room temperature. In this study it is proved that this kind of glass inclusions contain homogeneous silicate foam trapped before eruption. I based this observation mainly upon the fact that during microthermometry the apparently “bubble-free” inclusions shown silicate “foam-like” microtexture. During the heating procedure in the stage, above 500°C the silicate glass inclusion has been transformed into a “foam-like” or “cloudy bubbly” microtexture. This behavior is robust being reproducible for several repeated cycles in the same inclusion.

The primary assemblages of “foam-like” glass inclusions trapped mainly in quartz and feldspar phenocrysts in the “Dej Tuff” display

cavities of different size (10 to more than 200 microns) and various shapes, from irregular to negative crystals forms. Generally, they are primary, distributed as zonal melt inclusions in feldspar, and as random clusters in quartz (Fig. 2 and Fig. 3). It should be noticed that the “foam-like” silicate melt inclusions described in this study are either monophasic (glass) or biphasic (glass + one or more bubbles), sometimes occurring in the same crystal zone, and both were frequently homogenized during microthermometry. After complete microthermometry analysis, it is concluded that the components of the inclusions studied acts as a compact homogeneous package made by bubbles and silicate melt. A contracting bubble is released during cooling, after trapping, and the package silicate + bubbles remained undisturbed in certain conditions (the ideal case). The same contraction bubble(s) ultimate disappeared during heating in the stage and allowed to measure the final homogenization temperature (e.g., *bc* in E, Fig. 2).

Severes et al., (2007) demonstrated experimentally that H<sub>2</sub>O-loss during heating from the glass inclusion from quartz in Bishop Tuff is important, e.g., from 4wt % to 1 wt %, so we can expect that glass-bubbles assemblages resulted from thermal decrepitation and/or water-loss, become a disrupted silicate foam. Although in our reliability experiments by repeated microthermometric runs (around 30) the homogenization temperature was almost the same for the same inclusion (Fig. 7), suggesting that water-loss was not so important in this case. We can emphasized that the ideal “foam-like” glass inclusion” are apparently “bubble-free” at room temperature, and any glass inclusions with variable amount of bubbles suggest that the content was disrupted by thermal decrepitation and losses variable amount of water. It is evident from this study that ‘foam-like” glass inclusions are thermosensitive and they decrepitated frequently during heating in the stage (Fig. 5 and Figs. 9 to 16). Moreover the darkness phenomena starting around T<sub>g</sub> (glass transition temperature) and enhanced at  $\alpha \rightarrow \beta$  quartz transition is probably just an optical feature of

the enclosed “foamy-like” silicate melt, under the microscope (Fig. 14). Microtexture features of melt inclusions (glass  $\pm$  bubbles) in quartz in the “Bishop Tuff” were interpreted by Wallace et al. (2003) to be related to water speciation in the silicate glass inclusions during cooling of the plinian-fall and pyroclastic flow deposits. In this study reheating episodes seem to be predominantly responsible for the generation of the observed microtexture features of the melt inclusions.

Pseudosecondary trails decorated with “foamy-like” glass inclusions were also observed in quartz and feldspar phenoclasts. In zircon they are elongated and seem to be mainly secondary melt inclusions trapped during overgrowth. Hourglass-shaped inclusions (Anderson 1991), are also frequent in quartz and feldspar phenoclasts in the pumiceous “Dej Tuff” pyroclastic deposits, and probably their initial content was also a “foam-like” silicate melt which was disrupted by depressurization on the narrow neck side of the hourglass inclusion.

### 3.2 Multiphase glass inclusions

This kind of melt inclusions contains one or more vapor bubbles and daughter minerals, or accidental trapped solid phases plus silicate glass. They are more frequently distributed in the inner core of the feldspar phenocrysts and are very rare in quartz. Sometimes the glass is a bubble-rich, “foam-like” silicate melt and the solid phases obviously did not melt during the heating cycles in the stage. The daughter minerals are represented by quartz or feldspar, sometimes biotite or amphibole. Their melting temperature is higher than our microthermometric capabilities (the used microthermometric device is working only up to 1064°C), and they remain partially melted in the silicate melt, together with the vapor bubble(s). Probably these silicate melt inclusion are no longer “foamy-like” glass inclusions showing just one contracted bubble at room temperature.

### 3.3 Vapor-rich inclusions

A vapor rich phase formed mainly by H<sub>2</sub>O, and perhaps some trace amount of CO<sub>2</sub>, was

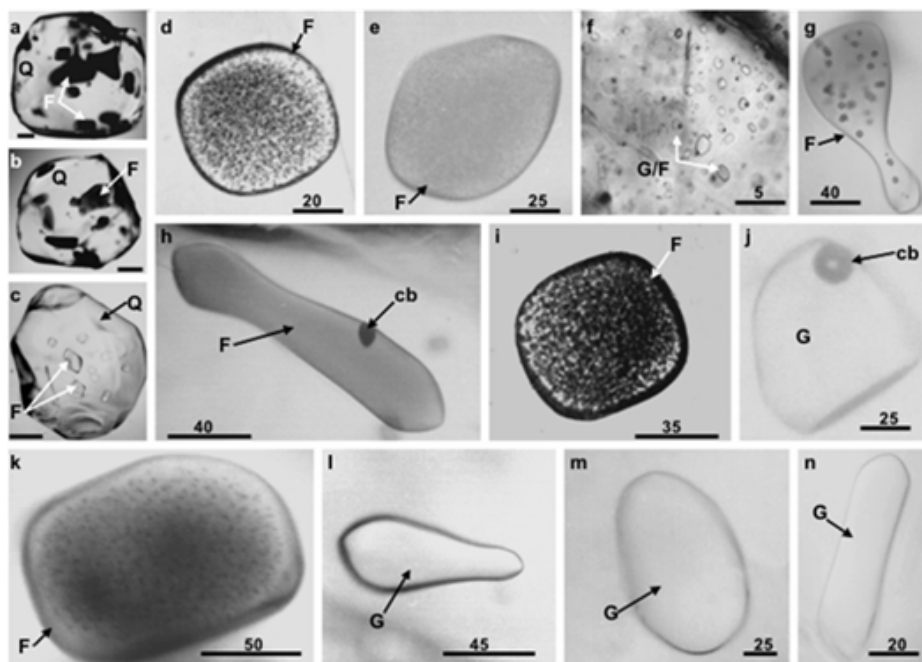


Fig. 2. “Foam-like” glass inclusions types in quartz phenocrysts from “Dej Tuff” formation, Transylvania basin, Romania. Quartz phenocrysts containing glass foam inclusions (F) partially decrepitated in **a.** and **b.**, **c.**-clear “bubble-free” glass inclusions; **d., e., i., k.**-natural reheated glass foam inclusions revealing silicate foam microtexture; **g.**-biphasic glass inclusions with glass+contraction bubble; **f.**-cluster of primary glass foam inclusions (F) in sanidine, some of them contain contraction bubble (G); **l., m., n.**-silicate glass inclusions bubble-free which shown obviously homogeneous foam microtexture above 500°C, and they homogenized on further heating in the stage (minimum trapping temperature). It should be noticed that during repeated microthermometric cycles some of them lost all bubbles and become “real” “bubble-free” silicate glass inclusions. Scale bar: a, b, c-250 μm; d, e, f, g, h, i, j, k, l, m, n-in μm.

trapped as monophasic gas inclusions. The CO<sub>2</sub> content was probably low and it is hard to be detected by microthermometry and/or crushing tests mainly because the abundance of tiny water vapor bubbles in the silicate glass. Sometime solid grains were observed inside the contraction bubble suggesting the presence of new crystallized solid phases. They are difficult to be distinguish from the false “vapor-like” microcavities formed by decrepitation of the former “foam-like” silicate melt inclusions which leaved behind empty voids that should be avoided for the study (e.g., Fig. 3g).

### 3.4 Opaque globular inclusions

This melt inclusion type is generally represented by Fe (-S-O) immiscible melt (Larocque et al. 2000; Pintea 2002), and suggest the presence of a mafic component input in the magma chambers. They could be recognized by their rounded shape and opacity under the microscope. A silicate blackish component

(obsidian) could be also envisaged and more sophisticated analytical facilities are needed to be described. In the “Dej Tuff” phenocrysts the globular inclusions were trapped frequently in feldspar and zircon, rarely in quartz crystals and originated probably in the mafic melt batches, introduced time to time in the lower part of the magma chamber as immiscible blebs. They were also observed inside the silicate glass inclusions as immiscible globules (e.g., Fig. 3d). Fe-Ti oxide show similar opaque appearance under the microscope but they have polygonal external shape being totally crystallized, probably by fractional crystallization rather by immiscibility. Sometime only the shape and optic features are not enough to separate between immiscible globule and crystallized opaque solid microinclusion.

### 3.5 Microphenocrysts

Apatite, zircon and feldspar microphenocrysts are the most frequent microcrysts

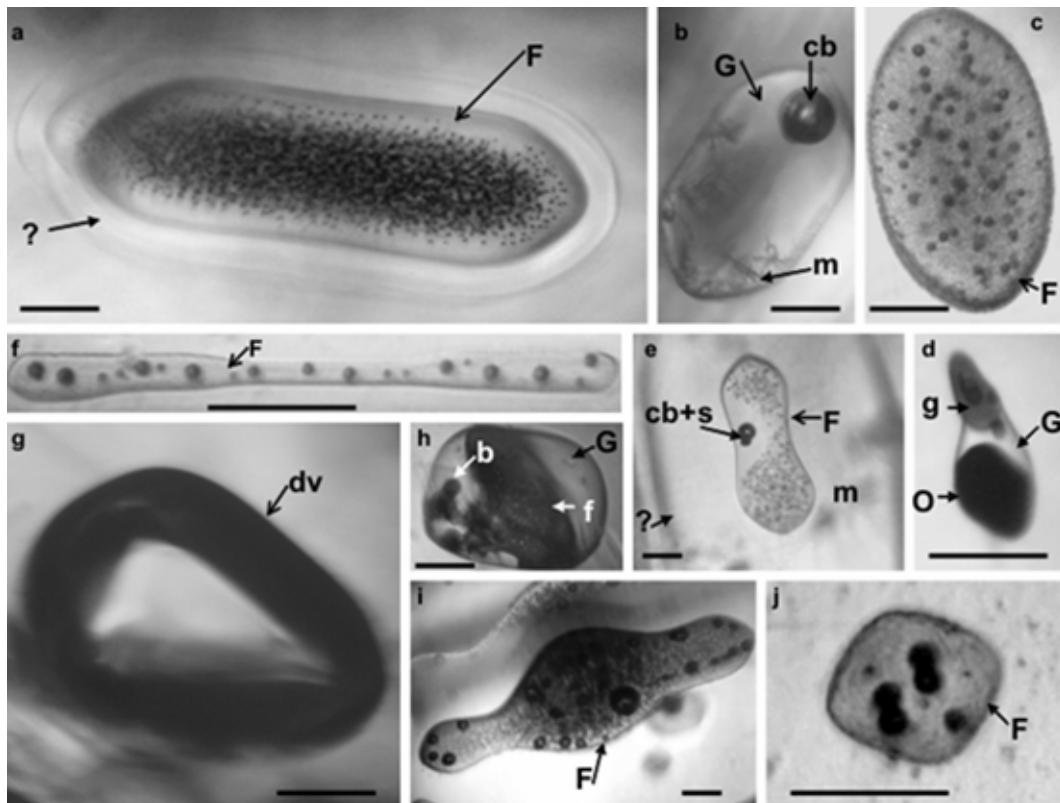


Fig. 3. More microtexture features of the “foam-like” glass inclusions from “Dej Tuff” trapped in quartz phenocrysts. **a.** Foam glass inclusion (F) surrounded by unknown halo(?), perhaps another silicate melt (?), **b.** Glass + contraction bubble (+ CO<sub>2</sub>-?) and microlites (m), **c.** Partly devitrified foam glass inclusion (F), **d.** Multiphase glass inclusion containing glass (G), opaque globule (O) and probably another droplet of immiscible silicate melt (mafic-g), **e.** Foam glass inclusion with one contraction bubble and an attached solid phase (cb+s) surrounded by an unknown ellipsoidal halo (silicate glass -?), **f.** Elongated secondary glass foam inclusion (F), partially decrepitated showing several ripened-? bubbles(b), **g.** Empty void(dv) left behind after complete decrepitation of a glass foam inclusion, **h.** Glass foam inclusion partially decrepitated containing glass(G), fissure (f) and bubbles (b), **i., j.** Typical partially decrepitated glass foam inclusion which never homogenized during the heating procedure in the stage.

trapped as solid inclusions beside the “foamy like” silicate glass inclusions”, mainly in zircon and feldspar, rarely in quartz. Some of them look-like tubular solid inclusions and contains silicate glass + bubbles which in several cases homogenized during heating procedure in the stage around 830-840°C (n=5), sometime up to 900°C in a plagioclase phenocryst from Şoimeni, for example. They were also described elsewhere by Davidson (2004) which pointed out some of their characteristics as such: microprobe analyses indicate an Ab<sub>75</sub>An<sub>20</sub>Or<sub>5</sub> component; euhedral form; uniform distribution in all samples; often decorating the growth zones; they are not daughter crystals; they have macrophenocrysts correspondent and can be used to distinguish between microphenocrysts and melt inclusion. The microphenocrysts

enclosed in magmatic mineral is a common feature and were described as very tiny microcrysts (<20µm), randomly distributed in macrophenocrysts, decorating growth zones and most often were trapped accidentally in silicate glass inclusion. The acicular feldspar microphenocryst were also described by Ştefan et al., (1982) in the upper Cretaceous quartz-monzodioritic porphyry in the Birtin dyke-like intrusion, and probably they are related to the pressure variation during melt decompression in ascending magma. Similar acicular product was observed by Keppler and Audetat (2005), by dissolving, in pure water, a piece of andesite in the diamond anvil cell at 854°C and 11.7 kb. Squeezing interstitial melt from a deep-seated batolith (granodioritic or andesite-dacite) and rapid ascension during decompression from high

pressure may be the origin of these microphenocrysts, probably flushed-up by the new hot melt batches in the crystallizing molten magma chamber (e.g., Bachmann and Bergantz, 2004).

#### 4 Microthermometry of glass foam inclusions

The microthermometric features of the “foam-like” silicate glass inclusion during the temperature variations as observed under the microscope are depicted in two examples in Fig. 4 and Fig. 5, respectively. In the first example (Fig. 4) a clear bubble-free glass inclusion is present at room temperature conditions, while in the second one (Fig. 5), the devitrified natural silicate foam was remelted. The spontaneous nucleation of a cloud of bubbles in the silicate melt, i.e. “foam-like” silicate melt, started usually at temperatures ranging between 500°C and 550°C with a maximum number of bubbles (up to 80% in the cavity volume) observed around 590°C and 600°C. It should be noted that the bubble cloud becomes clearly visible early around 500°C and 530°C, and in quartz crystals it is more enhanced during the alpha/beta transition around 573°C. In feldspar phenocrysts the behavior of melt inclusions during heating in the stage is almost the same with no change in the volume ratio between bubbles and melts. In this case bubble nucleation cannot be related to the transition above mentioned for quartz. It may be concluded from these observations that the “foam-like” silicate melt formation is influenced mostly by the temperature and pressure inside the melt inclusions. In addition, the cloud of bubbles seems to be, in many cases, the remaining silicate foam microtexture with darker appearance because the total reflexion under the optical microscope. In the open cavity under SEM microscopy, Clocchiatti (1975) evidenced a perfect “alveolaire” microtexture showing a channel network of very fine glass walls and named them “ponces”. A comprehensive physical description of the silicate foam texture was given by Cashman and Mangan (1994).

On further heating the “foam-like” microtexture becomes unstable and complete homogenization in the silicate melt state was recorded between 710°C and 900°C, with a maximum frequency between 750°C and 830°C, for  $n = 344$  measurements (Fig. 6). It is important to mention that the “foam-like” microtexture is generally stable between 500°C and 600°C, observed in the microthermometric stage, and the final homogenization is due by the disappearance of the contraction bubble which generally has the largest diameter above  $T_g$  (around 530°C, coincidentally with glass transition temperature for rhyolitic magma, i.e. Bagdassarov et al. 1996) and it remains frequently the only one bubble above 600°C. In the ideal case we suggest that the “foam-like” cell-microtexture would release by contraction a single vapor bubble, similar with the normal homogeneous silicate melt which shown only one bubble during contraction, as temperature decrease below the homogenization value. Correspondingly, we described here homogeneous silicate foam with one contracting bubble, which become homogeneous by heating up in the stage or heterogeneous (disrupted, decrepitated) foam when there are more than one bubble and they cannot be homogenized during microthermometry.

Each microthermometric measurement was replicated twice (sometime more) and generally the results obtained appear robust (Fig.7). Although it is observed that final homogenization temperature tends to decrease after multiple microthermometric cycles (on the contrary, if decrepitation occurred, the homogenization temperature increased in “normal” silicate melt or fluid inclusion). It is presumed that these phenomena could be related to the “foam-like” characteristics rather to the post-trapping event or thermal decrepitation in the heating stage. Anyhow the silicate “foam-like” seems to be fragile and sensitive to many external factors including P-T variations, or even during sample preparation procedure. This could be easily evidenced during microthermometry by heating quartz sample containing “foam-like”

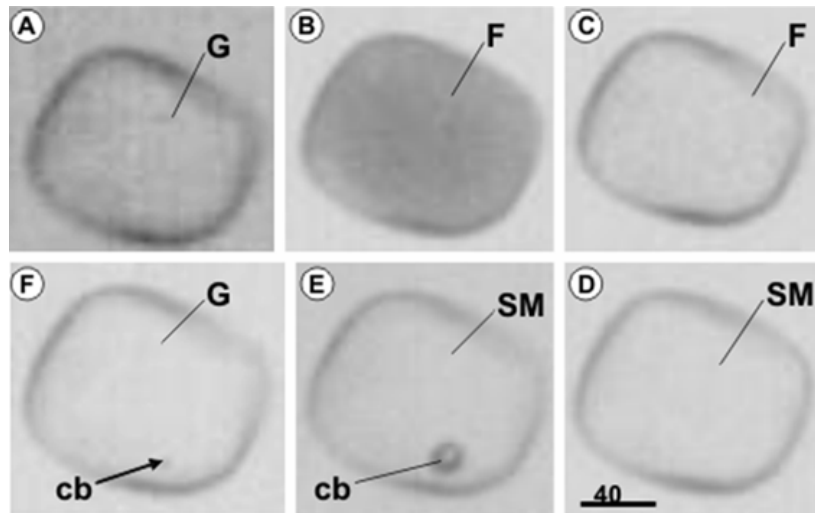


Fig. 4. Microphotographs sequence of a microthermometric cycle in a “foam-like” glass inclusion apparently monophasic (“bubble-free”) at room temperature from magmatic quartz phenocrysts from Dej tuff. **A.** Clear silicate monophasic glass inclusion, G- glass; **B.** Silicate foam revealed during the heating procedure in the stage at 580°C by spontaneous bubbles nucleation starting around 500°C, F - silicate foam; on further heating the bubbles vanished progressively showed in **C** at 770°C and final homogenization temperature was reached at 810°C in **D**, SM- silicate melt; **E.** On cooling back by cutting- off the power supply, a single contraction bubble (cb) nucleated in the silicate melt (SM) around 590°C, became more shrunken (cg) in the glass (G) at room temperature conditions as it shown in picture **F**. In the replicated cycle the same temperature values were recorded for the mentioned phase transitions. Scale bar in  $\mu\text{m}$ .

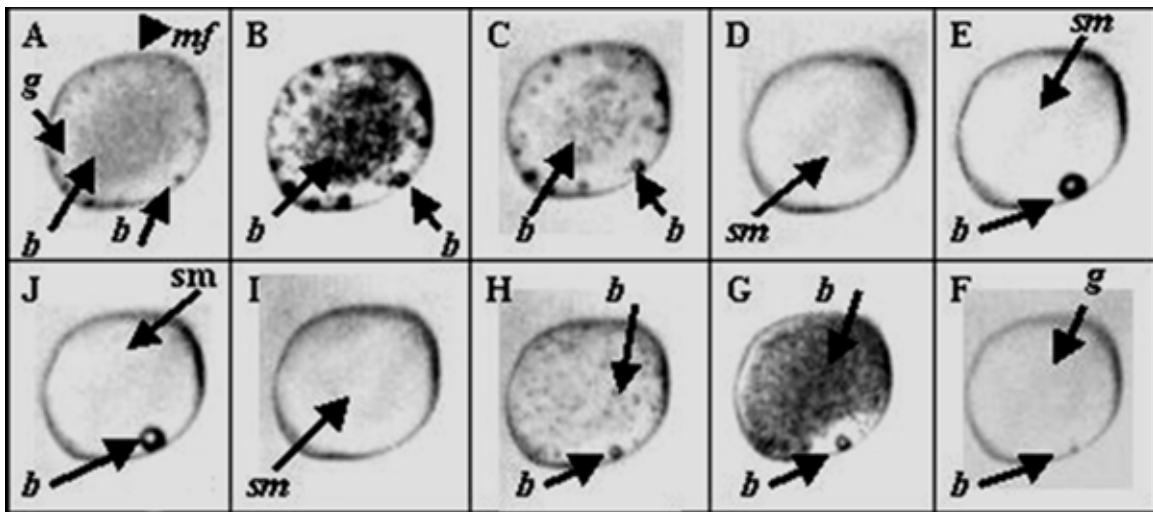


Fig. 5. Microthermometry sequences in a natural devitrified “foam-like” glass inclusion (FGI) from magmatic quartz phenocryst in ignimbrite from “Dej Tuff” formation.

**A.** At  $T=25^{\circ}\text{C}$ , the natural magmatic foam (*mf*) is composed by a large number of gas bubbles (*b*) as cloudy area and white glass (*g*); **B.** and **C.** - intermediate values at  $T=611^{\circ}\text{C}$  and  $T=681^{\circ}\text{C}$ , respectively; On further heating the foam is unstable and progressively the bubbles disappeared and the cavity contains at the final homogenization temperature a clear silicate melt showed in picture **D** at  $T_h=764^{\circ}\text{C}$  (*sm*- homogeneous silicate melt). During cooling back, at  $T=662^{\circ}\text{C}$  a single bubble was renucleated in cavity, and the foam became invisible as shown in picture **E** (*b* - gas bubbles, *sm* - silicate liquid). Back to the room temperature, the cavity contains now only one small shrunken bubble (*b*) and glass (*g*), showed in picture **F**.

In a replicated microthermometry cycle, after five days, the silicate foam renucleated spontaneous around 500°C with a maximum of bubbles number around 570°C; the pictures **G** and **H** were taken at 557°C and 696°C, respectively. The final homogenization in the silicate melt was measured now at  $T_h=735^{\circ}\text{C}$ , shown in picture **I**, *sm*- silicate melt; a shrinkage bubble was renucleated during decreasing of temperature in the stage, below 700°C showed in picture **J** (*b* - bubble, *sm*- silicate melt). *FGI* length = 60  $\mu\text{m}$ .



silicate glass inclusions” depicted in the serial microphotographs from Fig. 8 to Fig. 16.

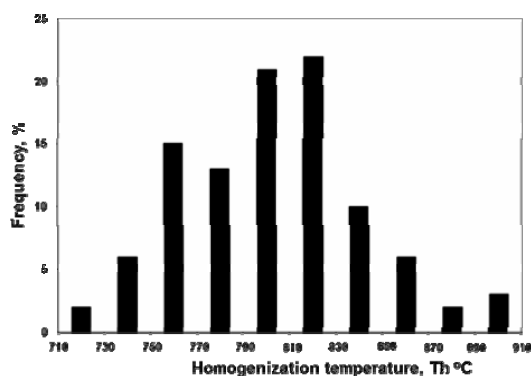


Fig. 6. Histogram of the homogenization temperature values from primary “foam-like” silicate glass inclusions in quartz, feldspar, and secondary in zircon, from the rhyolitic “Dej Tuff” formation from Transylvania basin. All measurements were done in a “home-made” microthermometry stage calibrated with potassium dichromate (398°C), halite (800°C), silver (961°C) and pure gold (1064°C), the precision of measurements ranged between  $\pm 20^\circ\text{C}$ .

## 5 Discussions

Theoretically the silicate foam was defined by Cashman and Mangan (1994) as containing a dispersed gas phase of  $> 74\%$  in volume, and more strictly the conceptual model of the foam structure-cells claims that rigid spheres attained maximum packing from 74.05% to 85% filled space. The bubble number density and nucleation rate depend on the supersaturating pressure and water content of the melt (Mangan and Sisson, 2000). For example, bubble number density is about  $9 \times 10^6$  at 175 MPa in a rhyolitic melt (Mangan et al., 2004). The same authors mentioned that the range in bubble number density observed in Plinian air-fall pumice ranged between  $10^8 - 10^{11}/\text{cm}^3$ . It is obvious that such data cannot be obtained by direct counting of bubbles number in the “foam-like” silicate glass inclusions by visual estimation under the microscope. Anyhow we estimated that up to 75 - 80% volume of the gas phase ( $\text{H}_2\text{O}$  vapor

bubbles) in the studied cavities was distributed in the necessary number of bubbles to be structured as foam (see Figs. 2, 3, 4). Consequently, it is more practical to name them “foam-like” silicate glass inclusions instead of normal silicate melt inclusions, because the silicate foam released a contracting bubble during decreasing temperature, suggesting a compact package of cell- microtexture between bubbles and silicate melt. Any change in this behavior would imply the presence of a heterogeneous or disrupted silicate foam. To be more convincing the reader is also advised to see Fig. 1c, 1g, as well as Fig. 2e, 2j in the papers of Skirius et al. (1990) and Fig. 1g, e, I, k, l, m, from Wallace et al. (2003) which shown similar feature on bubble – melt relation as resulted in our study during cycling microthermometry, but the mentioned authors have different explanation for the showed microtexture features. Similar pictures with “foam-like” silicate glass inclusions were presented from various volcanic samples e.g., by Clocchiatti (1975), Roedder (1979), Anderson (1991, 2003), Anderson et al. (2000), Frezzotti (2001), and Lowenstern (2003).

The first attempt to introduce bubbles dynamics of a viscous silicate melt (i.e. up to silicate foam) as a possible mechanism of volcanic eruption was mentioned long time ago (e.g., Rittmann, 1936). Since then a lot of data on silicate foam were accumulated mainly from glass technology (e.g., v deer Schaaf and Beerens, 2002), theoretical approaches (Proussevitch et al. 1993) and experimental study on bubbly silicate melts, presented by Hurwitz and Navon (1994), Sparks et al. (1994), Cashman and Mangan (1994), Mangan and Sisson (2000), Mangan et al. (2004), Martel and Bureau, (2001) and references therein.

Based on microthermometry data and petrographic observations presented in this study, such silicate foam would be formed in the upper part of the magma chamber, very close before eruptions, as it was suggested elsewhere by Stix (2007), as the main effect of the episodically hotter mafic magma influx in the

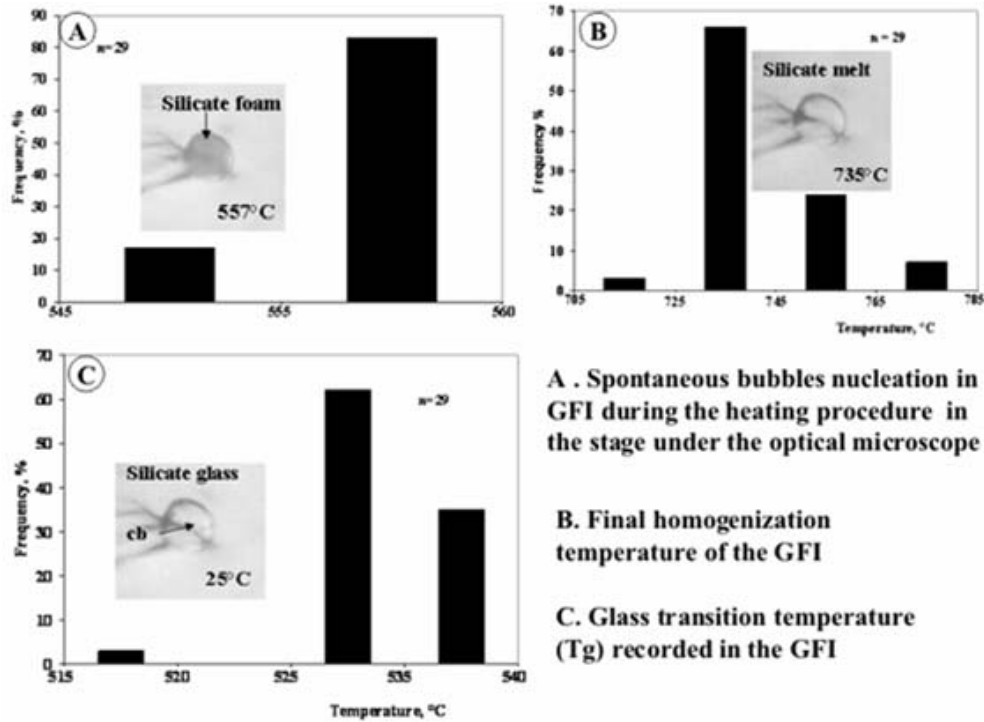


Fig. 7. Reliability data for a “foam-like” glass inclusion in quartz phenoclasts from the “Dej Tuff” formation. The same inclusion was repeatedly heated up until final homogenization temperature was achieved and then it was cooled- back to room temperature (n= 29). During heating, spontaneous bubble nucleation temperature in **A** and final homogenization temperature in **B** were recorded. Glass transition temperature (Tg) was recorded during cooling- back and was based upon the sudden collapsed bubble (s) in the silicate melt/glass around 530°C, depicted in **C**.

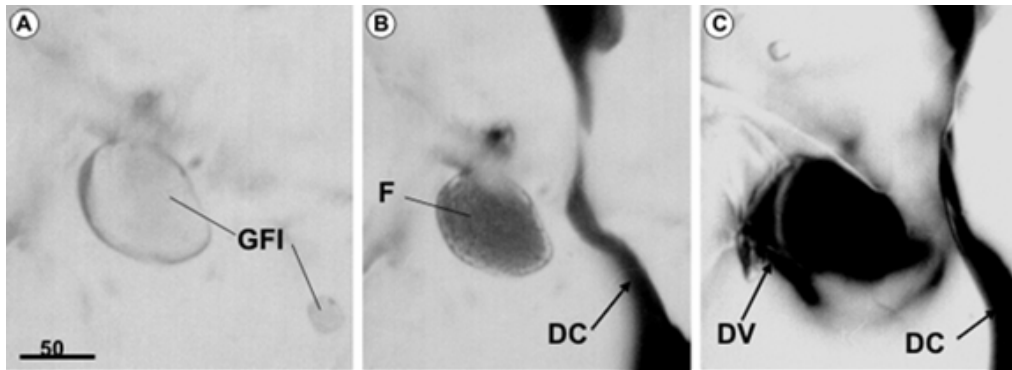


Fig. 8. Violent decrepitation of a “foam-like” silicate glass inclusion” (F) during heating procedure in the stage. A fissure (DC) was developed starting at 460°C and the silicate foam is completely revealed at 590°C (A, B). Increasing pressure and the proximity of the fissure caused explosion before homogenization temperature (C). A. 25°C, B. 590°C, C. 700°C.

rhyolitic magma chamber (Wark et al. 2007). In the Dej Tuff majority of the minerals were crystallized from this kind of rhyolitic silicate foam because they contain more than 95% of the melt inclusions in the form of “foam-like” silicate melt inclusions as primary assemblages. Many of them decrepitated partially or completely during rising temperature induced

probably by the new hotter vapor rich mafic magma influxes in the crystallizing rhyolite magma, as it was mentioned above. In fact this would be the principal mechanism of crystal fragmentation as it is documented in the literature (e.g., Bindeman, 2005) and demonstrated in this study by microthermometry (e.g., Fig. 14). The presence of microphenocrysts

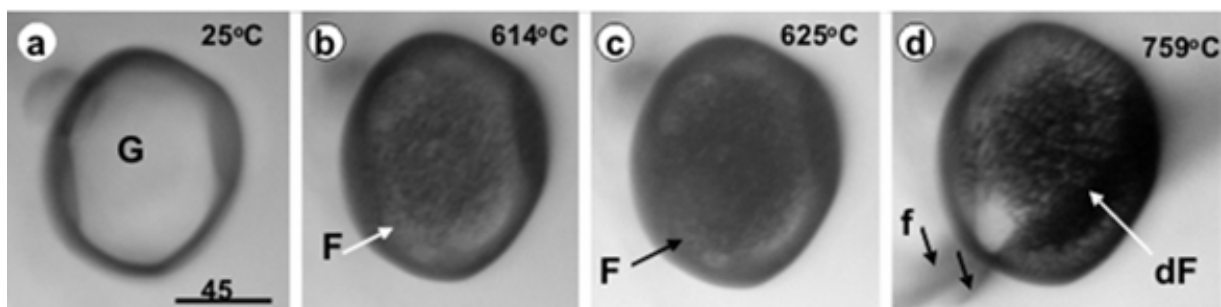


Fig. 9. “Foam-like” silicate glass inclusion “bubble-free” at room temperature (a) developed on heating a blackish microtexture, probably because total reflexion of the silicate foam microtexture (b and c). On further heating this inclusion decrepitated around 750°C (d) and a microfissure system developed (f). Cannot be homogenized on further heating and cooled-back below 500°C in this microtexture- feature as indicated in d.

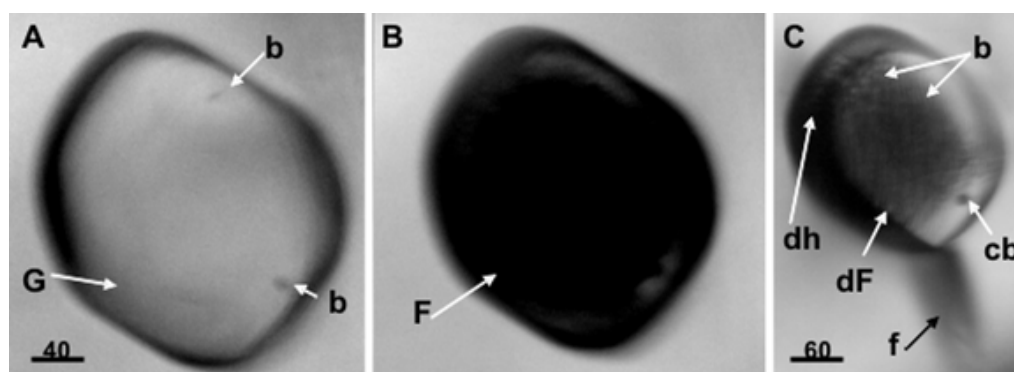


Fig. 10. Silicate glass inclusion with several bubbles (b) visible at room temperature conditions (A) revealing a dark foam microtexture at 625°C (B) decrepitating around 750°C and formed a characteristic halo(dh) and microfissure system (f) back to the room temperature (C). A separate contraction bubble (cb) and more bubbles (silicate globule? + vapor bubbles) - b in the heterogeneous silicate foam microtexture are shown in C. This remained unchanged back to room temperature.

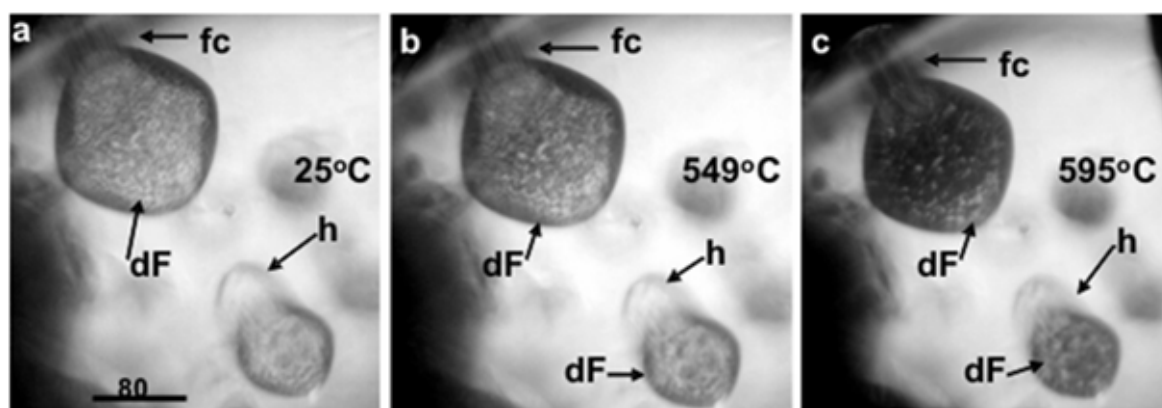


Fig. 11. Naturally decrepitated “foam-like” silicate glass inclusion showing a globular microtexture in (a) at room temperature. fc-microfissures, h-natural decrepitation halo. During re-heating in the stage the microtexture become darkness (dF) and globular microtexture was more evident after glass transition temperature ( $T_g$ ) and temperature ( $\alpha \rightarrow \beta$ ) of quartz transition (b and c). On further heating cannot be homogenized.

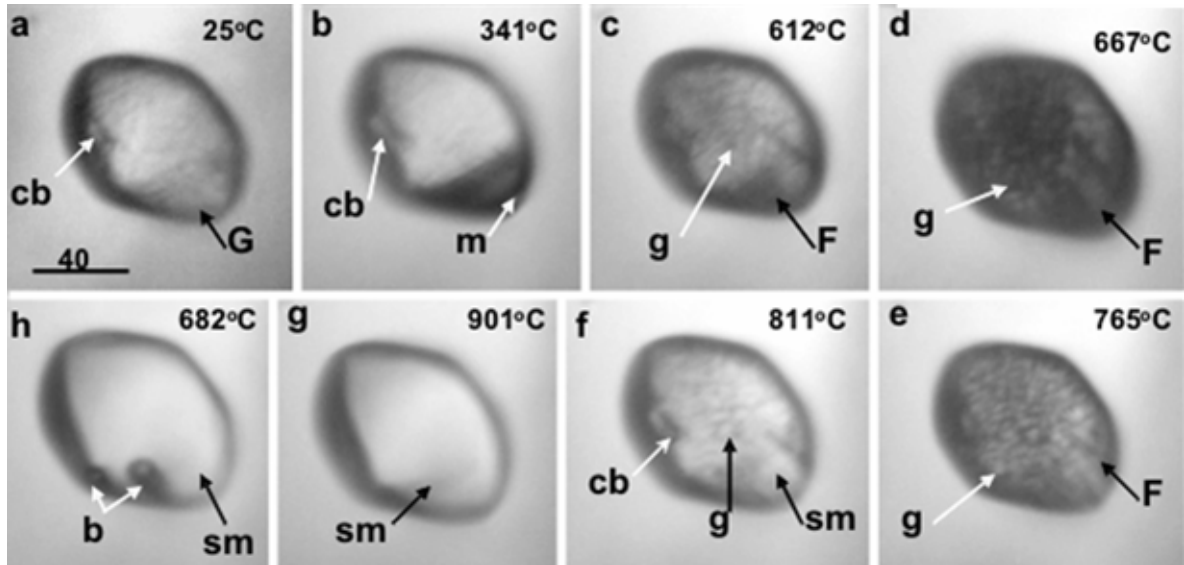


Fig.12a. Complete cycle of microthermometry in a “foam-like” silicate glass inclusion which is almost “bubble-free” at room temperature (a), but shown a microglobular microtexture on the surface and contained a contraction bubble-cb. The optic-dark silicate foam microtexture-F was revealed starting in (b) showing globular microtexture in (c), (d), (e), and (f). The contraction bubble-cb, homogenized at presumed trapping temperature of 901°C in (g). Back to room temperature, around 685°C two bubbles-b renucleated showed in (h). Other notations: m- meniscus between glass and wall cavity, g- silicate globule-?, sm- silicate melt.

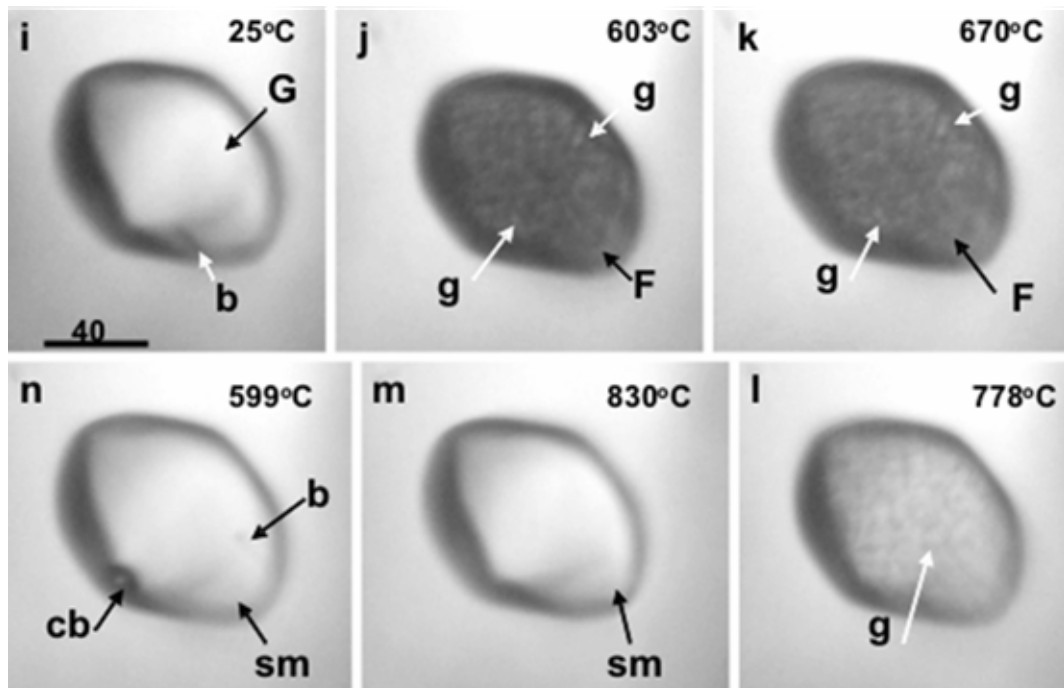


Fig. 12b. After one day the replicated microthermometric cycle showed the same behavior. At room temperature in (i) glass-G and a single contraction bubble-b (?) is present. The foam is revealed between 600°C and 700°C (j and k), and final homogenization temperature was recorded at 830°C (71 °C lower than in the first cycle. This is a characteristic feature during repeated microthermometry in “foam-like” silicate glass inclusions, that is the temperature become lesser probably because of the foam characteristic). Notations: F- silicate foam, S- silicate globule-?, silicate – melt, cb- contraction bubble, b- another bubble.

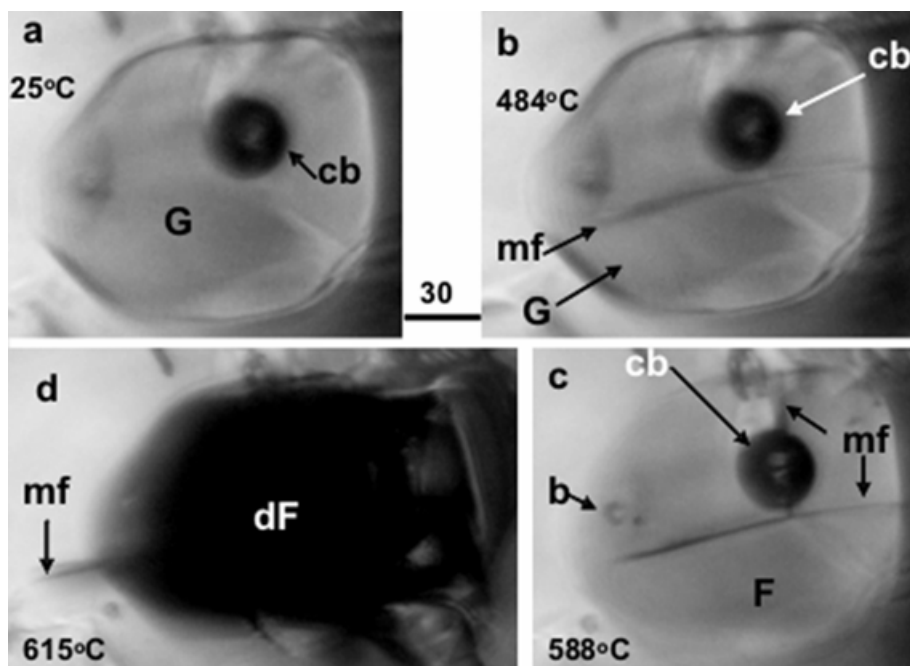


Fig.13. Experimental decrepitation of a “foam-like” glass foam inclusion during heating procedure in the stage. Notations: G-silicate glass, mf- microfissure, F- silicate foam, b- bubbles, dF- decrepitated silicate foam.

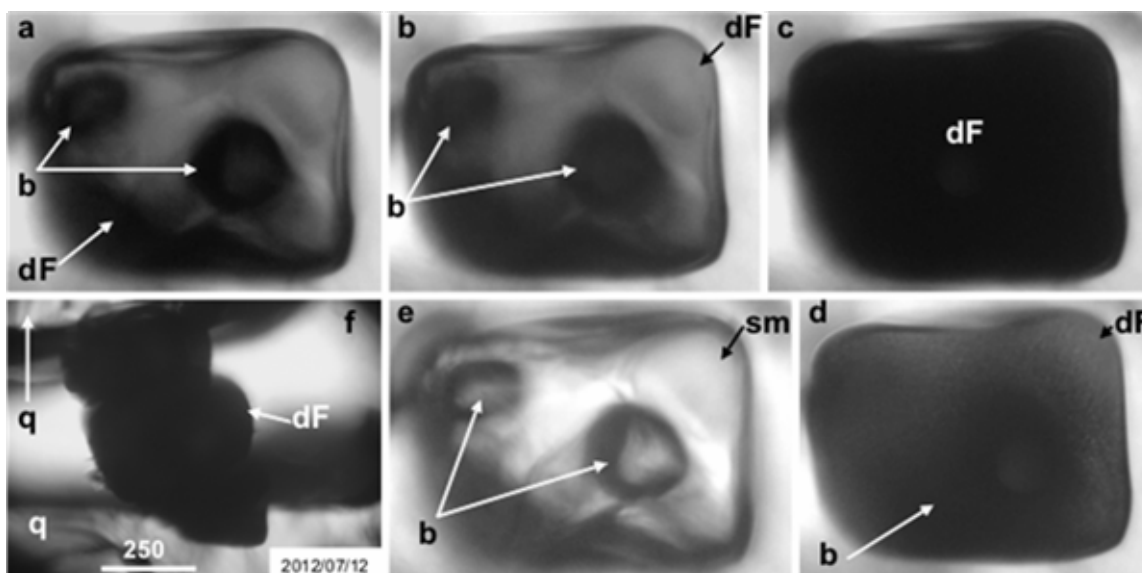


Fig. 14. Naturally decrepitated “foam-like” silicate glass foam inclusion containing two bubbles in a brownish glass microtexture at 25°C (a). During heating in the stage the silicate foam microtexture is reactivated between 625°C (b) and 686°C (c). This start clearing around 738°C (d) and become colorless at 837°C (e), and the two bubbles reduces slightly their size. At 841°C (e) the inclusion decrepitated so violently than the quartz (q) was broken in two pieces. The size of “foam-like” silicate glass inclusion did not change, it was open on the edge and the silicate foam (df) liberated and increased volume about *five times*, but all the stuff remained attached to the host quartz broken pieces in the stages, and looks like a “micro-/mini eruption”.

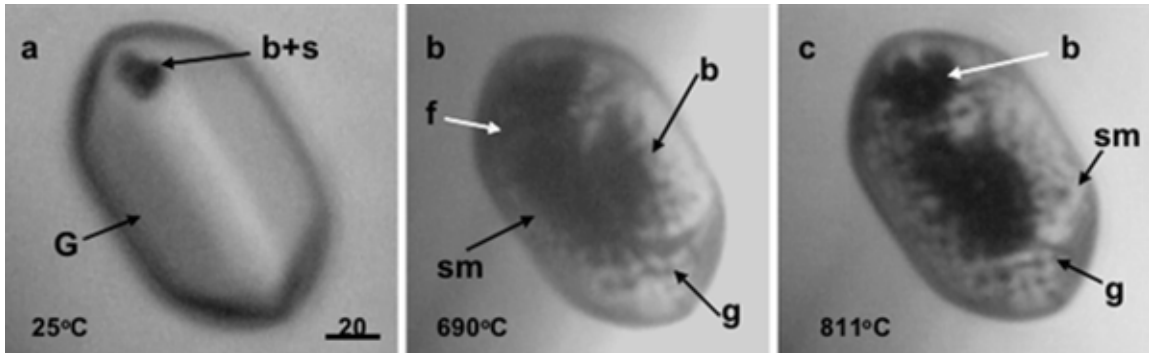


Fig. 15. "Foam-like" silicate glass inclusion contain glass and bubble plus attached solid microlites at room temperature (a). During heating in the stage a microfissure (f) was formed at 321°C and between 558° and 690°C (b) a mixture of bubbles (b), silicate melt (sm) and silicate globules (g) were released. Around 811°C (c) some vapor bubbles (b) coagulated and are coexistent in the heterogeneous silicate foam with silicate globules (g) and silicate melt (sm). It cannot be homogenized and on further heating decrepitated around 882°C along the c-axis and could not be relocated again.

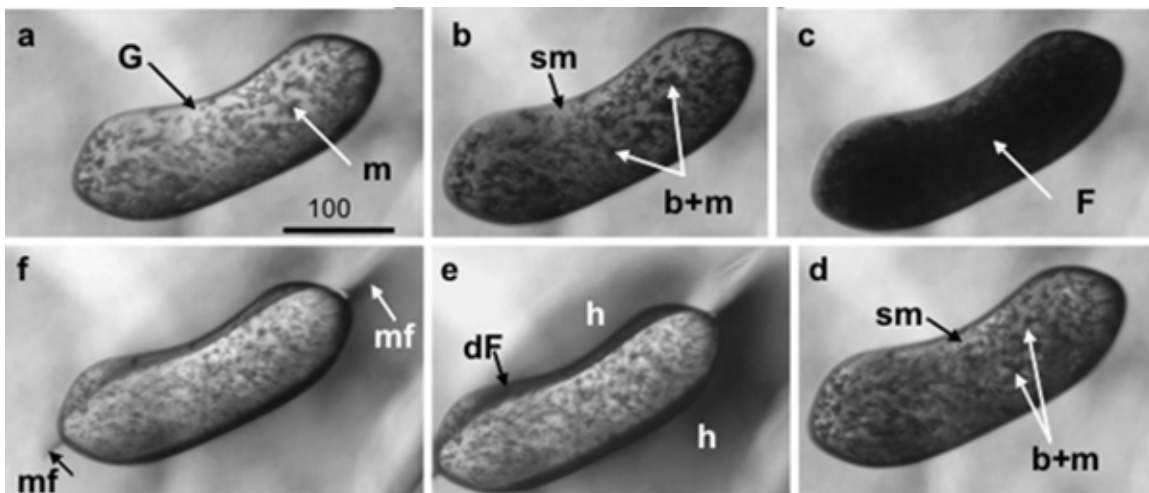


Fig. 16. Large glass "foam-like" silicate glass inclusion contains glass (G) and microlites (m) at room temperature (a). It start blackish around 597°C (b) and become complete dark around 611°C (c). On further heating, around 762°C (d) the silicate foam (F) is almost clear again and now contained silicate melt-sm, bubbles-b and microlites-m. The inclusion decrepitated violently at 776°C and a decrepitated halo (dF) was formed along a microfissure (mf), showed in (e). Back to room temperature the inclusion contains glass + microlites, but a lot of gas bubbles were lost. Microphotographs (e) and (f) were fixed at different size to see the fissure (mf) and the decrepitated halo (h).

as microinclusions in the same phenocrysts suggest rapid pressure changes during melt accessioned toward the shallow layers. The microthermometric experiments undertaken in this study show that the volatile-rich silicate melt "foam-like" was stable only up to 500° to 600°C in the microthermometric stage and by similarity at the same temperature in the magma chamber. Above these values under increased internal pressure in the cavities, during heating in the stage (or naturally heating in the pressurized

magma chamber by the hotter mafic magma) the volatile-rich "foam-like" silicate melt would be homogenized in a single melt phase, and decrepitated on further heating. As a consequence, we presume that a volatile supersaturated and homogeneous silicate melt was trapped in minerals growing in magma chamber prior eruption between 730° and 850°C, which is in good accord with other temperature estimation based on Fe-Ti oxide by Szakács (2000) in the Dej Tuff (more data for

## Melt inclusions in minerals from the Dej Tuff

Table 1. Comparative data between homogenization temperatures measured in “foam-like” silicate glass inclusions from the Dej Tuff and calculated temperatures by various geothermometers including melt inclusion microthermometry in several well known acidic tuff formations.

Acidic tuff formation	Fe-Ti oxide, T, °C	Zr sat. T, °C	$\Delta^{18}(\text{Qz-Mt})\text{T}$ , °C	Titan i-Q, °C	Melt Inclusions Th, °C	Author(s)
Dej Tuff, Romania	604-892	-	-	-	“foam-like” silicate glass inclusions” 710-900 (+/-20) (n=344)	Szakacs, 2000; Pintea, 2005, 2007 and this study
Toba Tuff, Indonezia	701-847 2.6-3.4 kb	-	-	-	~ 960 in Toba quartz, sanidine and plagioclase (Beddoe- Stephens et al., 1983)	Cesner, 1998
Bishop Tuff, USA	714-818	762 -801	714 -817	712-742 (cores) 700-810 (rims)	800-900, ~ 2kb, (quenching experiments)	Bindeman and Valley, 2002; Hildreth and Wilson, 2007; Wark et al., 2007
Taupo Volcanic Zone	690-990 (Shane, 1998)				820-850	Szakacs, 2000; Kamenetsky and Danyushevsky, 2005

comparison in Table 1). The trapping pressure was roughly estimated by comparing the microthermometric behavior of the included “foam-like” silicate melt during heating procedure in the stage with the experimentally results published by Mangan and Sisson (2000) on homogeneous and heterogeneous bubble nucleation processes. In this respect the pressure was estimated between 200 MPa and 20 MP, respectively. The depth of magma chamber where the melt inclusions were formed could be estimated around 7 Km in the case of homogeneous trapping.

In this study the presence of silicate foam was documented mainly on the microtexture changes observed during heating in the microthermometric stage. Another method such as SEM microscopy would be necessary to reveal the complete imaging on these microtextures inside the phenocrysts, as Clocchiatti (1975) have already done in many others quartz samples. At larger scale, relicts of foam-like microtexture could be observed in the

groundmass in thin sections of the rhyolitic ignimbrite and pumice lapilli, where the silicate glass is composed by different types of collapsed vesicles (see also Seghedi and Szakács, 1991). Such microtexture was described earlier by Istrate (1975) in the rhyolitic ignimbrites in the Laramian formation in the western part of the Vlădeasa massif from the North Apuseni Mountains (western Romania).

The breaking of phenocrysts by decrepitation of the “foam-like” silicate glass inclusions was another important feature, recorded during overheating in the microthermometric stage (Fig. 8 to Fig. 16), suggesting that this process enhanced the crystal fragmentation during the explosive volcanic eruptions. The accumulation mechanism of a silicate foam layer and their stability was pointed out at Masaya volcano (Nicaragua) by Stix (2007). Similarly, we suggest in the case of Dej Tuff, that the silicate foam is accumulated in the top of the magma chamber (trapped in the crystallizing minerals which were fragmented under the heating

episodes from below) at the base of the volcanic conduit(s), as an immiscible layer, and exploded when some critical parameters are reached. It was demonstrated experimentally by Cottrell et al. (1999) at Santorini that the episodic intrusion of magma, provided the necessary heat and perhaps contributed to the ascent of the volatile-rich magma to shallow crustal depth and probably the mafic injection continued until trigger the cataclysmic eruption by decompression. An analogue behavior is suggested in this study by the trapped “foam-like” silicate melt in quartz, feldspar and zircon (majority becoming phenoclasts after fragmentation because “foam-like” silicate foam decrepitation) by petrography and microthermometry at a “micrometric magma chamber scale”.

## 6 Conclusions

Based on petrography and microthermometry of “foam-like” glass inclusions” in minerals of the “Dej Tuff”, the following conclusions can be drawn:

- the studied minerals from the Dej Tuff contain more than 95% “foam-like” silicate glass inclusions as mainly primary assemblages in quartz and feldspar phenoclasts and secondary “foam-like” inclusions in zircon;
- the “foam-like” silicate glass inclusions are various in shape, size and distribution and they are “bubble-free” (glassy monophasic) or contained various ratio between glass and bubbles at room temperature conditions;
- their formation temperature ranged between 710-900°C ( $\pm 20^\circ\text{C}$ ) with a maximum of frequency between 750° to 830°C (n=344) in very good agreement with the Fe-Ti- oxide geothermometer used at Cepari by Szakács (2000) and calculated the formation temperature between 604°C and 892°C;
- the estimated trapping pressure ranged between 200 MPa and 20 MPa, suggesting a depth for the magma chamber around 7 Km based upon homogeneous bubble nucleation in “foam-like” silicate glass inclusions;
- the silicate foam was identified firstly in this study by visual observation during heating in the microthermometric stage of the apparently “bubble-free” silicate glass inclusions and named “foam-like” silicate glass inclusions;
- many of the “foam-like” glass inclusions decrepitated during the heating experiments, mainly the bigger ones (i.e. more than 150 microns) and coalesced bubbles were formed and the cavities never homogenized again. Frequently the empty decrepitated “foam-like” glass inclusions appearing opaque cavities (empty) under the optical microscope. The darkening phenomenon revealed during heating in the microthermometric stage seems to be the effect of the foam skeleton (microtexture) totally reflected the transmitted light (e.g., Fig.14);
- during reliability test the “foam-like” glass inclusions”, apparently “bubble-free” at room temperature revealed a compact package microtexture into silicate foam around 500-600°C during heating, and the recorded temperature were generally reproducible (Fig.7). No leakage was observed during replicated measurements, but the partial decrepitated cavities never homogenized again. It must be stressed that often during microthermometry, the repeated cycles in the same cavity shown homogenization temperature less than the precedent one, which is contrary in the normal silicate melt inclusions, and this is perhaps a specific characteristic to the trapped silicate foam or because the water-loss during experimentally (or naturally) heating and partial decrepitation (decompression);
- there are many reasons for decrepitation of “foam-like” silicate glass inclusions” including natural reheating episodes and partial decrepitation during the explosive events or artificially during sample handling and preparation; The larger “foam-like” silicate glass inclusions (more than 150 microns) decrepitated easily and this seems to be the main cause of mineral fragmentation (see also Bindeman, 2005);
- the entrapped silicate foam behave in a similar mode at a micrometric scale such as the silicate foam accumulated in the top of the magma



chamber at the base of the volcanic conduit as an immiscible layer, and would explode when some critical parameters are reached. The hotter volatile-rich mafic recharging generates more and more silicate foam in the upper level of the crystallizing rhyolitic magma chamber, perhaps triggering the cataclysmic volcanic eruption. In conclusion, the “foam-like” silicate glass inclusions behaved during the microthermometric experiments in this study as a micro-magma chamber analogue.

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