A brief history of solid inclusion piezobarometry

Ross J. Angel[1]**, Matteo Alvaro**[2]**, and Silvio Ferrero**[3]

[1]IGG CNR, Via Giovanni Gradenigo 6, 35131 Padua, Italy
[2]Department of Earth and Environmental Sciences, University of Pavia, Via A. Ferrata, 1, 27100 Pavia, Italy
[3]Dipartimento di Scienze Chimiche e Geologiche, University of Cagliari, 09042 Monserrato, Italy

Correspondence: Ross J. Angel (ross.angel@igg.cnr.it)

Abstract. Solid inclusion piezobarometry is the determination of the entrapment conditions of solid inclusions in a host by measurement and interpretation of the residual pressure of the inclusion. The development over the past two centuries of the concepts, analytical tools and measurement techniques of inclusion piezobarometry is reviewed, and potential future developments are outlined for the special issue of the *European Journal of Mineralogy* devoted to the study of mineral and melt inclusions.

1 A brief history

Inclusions in natural rocks are an invaluable asset for geoscientists because they provide information about processes in the Earth's past that are otherwise hidden or subsequently overprinted. In particular, these "impurities" that are remnants of geological processes frozen in the rock record have the potential to provide quantitative data necessary for quantifying a wealth of processes of great impact for all humankind.

Solid inclusion piezobarometry is the determination of the entrapment conditions of inclusions in a host by the measurement and interpretation of the residual pressure of the inclusion. Several of the papers in this special issue (Tuttle and Henry, 2023; Wang et al., 2023; Pummell and Thomas, 2024) and a recent exhaustive review (Kohn et al., 2023) illustrate that it is a rapidly developing method within petrology and mineralogy that offers an additional constraint on rock histories beyond that of conventional thermodynamic analysis of mineral phase equilibria. The recent widespread use of inclusion piezobarometry is, however, based upon concepts, analytical tools and measurement techniques that have been developed over a period of two centuries. Certainly, the recognition of inclusions in crystals, and the possibility that the nature of the inclusions could shed light on the conditions of growth of the host crystal, is far older (e.g. Steno, 1669). But, as far as we can determine, Brewster (1820, 1835) was the first to report birefringence haloes around inclusions

(he called them cavities at the time, believing them to be filled with gas) when viewed under polarised light. From the changes in birefringence colours when viewed through tint plates, and by comparison with the birefringence he observed in glass when under the force of an indenter, Brewster correctly concluded that the inclusions were exerting an outwards force on their diamond host.

Although he was studying fluid and melt inclusions, containing vapour bubbles, it was Sorby (1858) who showed that the properties of inclusions (in this case the temperature at which the vapour bubble disappeared) could be used to indicate the pressure and temperature conditions of original entrapment. In a subsequent examination of inclusions in diamonds Sorby and Butler (1869) were able to demonstrate that the inclusions themselves were birefringent and thus crystalline. They interpreted the surrounding birefringent haloes in the diamond as "proving that the inclosed crystals have exerted a pressure on the surrounding diamond" but then drew the wrong conclusion that "We, however, do not imagine that the crystals have increased in size, but that probably they have prevented the uniform contraction of the diamond". This last statement is wrong. Harris et al. (1970) credit Sutton (1918) as the first to recognise correctly that the strain birefringence observed in the host arises from strain, and by implication stress or pressure, in the inclusion. However, that is not at all clear because the discussion by Sutton (1918) concerns heating the diamond after entrapment of the inclusion. He correctly deduced that the lower thermal expansion

coefficient of the diamond prevents the inclusion from expanding as much as it should on heating, and thus stress and strain would be created in the diamond around the inclusion and could lead to fracture of the diamond host. Therefore, Sutton must also have realised that cooling has the opposite effect to heating; it would lead to a reduction of inclusion stress in diamond. Therefore, cooling cannot be responsible for the birefringent haloes seen in diamonds when observed at room temperature. This was confirmed by the analysis (Goodier, 1933) of the stress developed in and around inclusions when the host is subject to an external stress, which include changes in external hydrostatic pressure and the effective stress generated as the result of cooling. Goodier (1933) showed that, starting from a system in uniform stress and strain, the final stress state is determined solely by the elastic properties of the system and the volume strain imposed on the host. And after a change in pressure or temperature a spherical inclusion has a uniform internal pressure while the host develops a deviatoric stress field, and hence the strain birefringence, while the pressure (equated to the mean stress) everywhere in the host remains equal to the external pressure. These results were confirmed and generalised by Eshelby (1957) for elliptically shaped inclusions. These analyses of Goodier and Eshelby showed that the final inclusion pressure, denoted P_{inc} in modern work (Angel et al., 2015), arises from two contributions to the volume change of the inclusion. The first is the pressure of the inclusion arising from it being constrained to the ideal volume change of its host subject to the change in pressure and temperature from entrapment. In the absence of plastic flow, cracking or other deformation of the host mineral, this can be calculated from thermodynamics, or specifically the equations of state of the host and inclusion phases if they are both isotropic. Hence it has been called "P_{thermo}". But if P_{thermo} is not equal to the external pressure applied to the host, there would be a force imbalance at the wall of the inclusion. If P_{thermo} is greater than the final external pressure on the host, the excess pressure in the inclusion would force the inclusion to expand, and thus the inclusion pressure would decrease until it is balanced by the radial stress in the host that builds up from compression of the host by the inclusion. It is this mutual relaxation that deforms the host anisotropically and thus creates the observed optical birefringence in cubic host minerals such as diamond.

With the determination of the components of the photoelastic tensor of diamond (Poindexter, 1955), all of the concepts and tools were apparently available to infer the pressures of inclusions from measurement of the birefringence in their diamond hosts. But it is difficult to measure and then interpret the birefringence because what is actually observed is the total birefringence integrated through the thickness of the specimen, so this approach remains an experimental challenge to this day (e.g. Howell et al., 2010; Howell and Nasdala, 2008). Prior to 1960 it therefore appears that the only methodology available to constrain the entrapment condi-

tions of solid inclusions was to determine the temperature at which inclusions fractured the host (Smith, 1952).

The key conceptual breakthrough that avoided the problem of measuring the birefringence (or strains in the host) was due to Rosenfeld and Chase (1961). They noted that after entrapment of the inclusion there is a path in $P - T$ space along which the inclusion crystal has exactly the same fractional volume change as the host. The slope of this line, subsequently called an isomeke (Adams et al., 1975b), depends on the contrast between the coefficients of volume expansion, α_H and α_I, and compressibility, β_H and β_I, of the two minerals:

$$\left(\frac{\partial P}{\partial T}\right)_{isomeke} = \frac{\alpha_H - \alpha_I}{\beta_H - \beta_I}. \tag{1}$$

Along this $P - T$ path a cubic inclusion such as garnet or spinel continues (without further compression or expansion) to completely fill the cavity in a cubic host mineral such as diamond. Therefore, no stress difference is developed between the host and inclusion along an isomeke, the inclusion pressure remains equal to the external pressure on the host and, consequently, there is no strain field in the host.

The insight of Rosenfeld and Chase (1961) therefore reduced the determination of possible entrapment conditions to finding a point on the entrapment isomeke and then either calculating the isomeke in $P - T$ space via Eq. (1) from the known properties of the minerals, or measuring isomekes directly by comparison dilatometry of the two minerals (Adams et al., 1975a; Cohen and Rosenfeld, 1979). The entrapment isomeke could often be found by either heating or cooling the host-inclusion system and finding the temperature at which the strain birefringence disappeared (e.g. Rosenfeld and Chase, 1961; Hutchison, 1967). Or the thermal expansion of the trapped and freed inclusion crystal could be measured separately by diffraction and the temperature of the entrapment isomeke at room pressure found by calculation (e.g. Harris et al., 1970).

Finding the entrapment isomeke experimentally avoids two problems. First, there is the problem of how to measure the pressure or stress state in the inclusion while still entrapped in its host and, second, how to allow for the mutual elastic relaxation of a host-inclusion system starting from a measurement of the final inclusion stress state, P_{inc}. The solution of this second step was long delayed. Instead, the forward problem of calculating the stress state of host-inclusion systems along a presumed exhumation path starting from known entrapment conditions was addressed by Gillet et al. (1984) and then Van Der Molen and Van Roermund (1986), both groups motivated by calculating under what conditions cracking would be initiated around inclusions during exhumation. These analyses avoided the problems of calculations over large P and T changes, and hence large changes in volume that take materials out of the linear elastic regime, by calculating the relaxation over a series of small decrements in P and T. If the calculated final

P_{inc} matched the measured one, then the proposed entrapment conditions could be the actual entrapment conditions of the inclusion. However, while this approach allows the effects of phase transitions such as from coesite to quartz within the inclusion to be calculated, it is not an elegant or practical approach to finding possible entrapment conditions from a measured final P_{inc}. That was enabled by Zhang (1998) showing that the relaxation term can be calculated directly from the elastic properties of the host and inclusion and the actual measured inclusion pressure. A $P - T$ point on the entrapment isomeke can then be calculated, usually at room temperature. And the isomeke through this point is calculated with Eq. (1) that represents (unlike the forward calculation method) all possible entrapment conditions from one calculation. First applications included estimates of entrapment isomekes of olivines in diamond from their P_{inc} determined from the up-shift of a Raman band of the olivine (Izraeli et al., 1999) and of coesite inclusions in diamond whose P_{inc} was determined by both Raman spectroscopy and in situ X-ray diffraction (Sobolev et al., 2000).

In the subsequent two decades the methodology has been improved and become widely used primarily because of three developments: first, the widespread availability of micro-Raman spectrometers to determine inclusion pressures (e.g. Enami et al., 2007, for quartz in garnet, QuiG) from the pressure-induced shift in their Raman lines; second, by the introduction of more precise and more realistic equations of state into the calculations, as summarised by Angel et al. (2014); and third, and perhaps even more importantly, by the provision of user-friendly software to perform the calculations of entrapment conditions from the measurements of either the Raman shifts of inclusions (e.g. Kohn, 2014) or the pressures or strain states inferred from them or X-ray measurements (e.g. Angel et al., 2017; Mazzucchelli et al., 2021).

In contrast, measurements of the stress fields in the host are not commonly used to determine inclusion pressures or their entrapment conditions. Pressure determination, for example by EBSD, is not possible because specimen preparation to allow the stress field in the host close to the inclusion to be measured will always result in relaxation of that stress (Campomenosi et al., 2018; Zhang, 1998; Zhong et al., 2019). But modern developments in mineral physics have allowed the study of inclusions to return full circle to the effects in the host represented by the birefringent haloes first reported by Brewster (1820) two centuries ago. Quantitative measurements of the birefringence around undisturbed inclusions in diamond are now possible (Howell et al., 2010), and the stresses that they imply agree with independent determinations from mapping by Raman spectroscopy (Nasdala et al., 2003; Howell and Nasdala, 2008).

Minerals are not elastically isotropic. This means that even a spherical inclusion will develop anisotropic stress (if the inclusion is anisotropic) or anisotropic strain (if the host is anisotropic). Much recent research has focussed on finding corrections for these effects so that P_{inc} measurements can be used within the isotropic model. For example, different inclusion shapes lead to different amounts of mutual elastic relaxation, but shape correction factors can be calculated (Mazzucchelli et al., 2018). Anisotropic stress in the inclusion moves the Raman peaks from their positions under hydrostatic stress, but these effects can be calculated (Murri et al., 2018, 2022) allowing measured peak shifts to be more correctly interpreted (Angel et al., 2019; Gonzalez et al., 2021) and a mean P_{inc} to be determined for use in isotropic isomeke calculations. Full anisotropic analyses are also available (Zhong et al., 2021). But measurement of inclusion strains also opens up the possibility of using them to define separate linear isomekes of each cell parameter of the inclusion with the host (Adams et al., 1975a; Gonzalez et al., 2021) whose crossing point in $P - T$ space will uniquely identify the entrapment (or resetting) conditions of the inclusion (e.g. Alvaro et al., 2020). Multiphase inclusions can also be treated within the isotropic model by calculating the elastic properties of the composite inclusion from the properties of the phases (Angel et al., 2023), and the effects of phase transitions in the inclusion can be addressed through more complex visco-elastic models (e.g. Zhukov and Korsakov, 2015).

With these extensions of the simple spherical inclusion model in place, there is now more focus on the geological interpretation of inclusion pressures. Many inclusions clearly retain pressures that reflect entrapment conditions (e.g. Endo et al., 2012; Gianola et al., 2023). The P_{inc} of other inclusions, however, do not, indicating that the inclusion stress state has been modified by non-elastic processes during exhumation. This is a hazard that has long been recognised (e.g. Rosenfeld and Chase, 1961; Carstens, 1971). But now such processes can be modelled so that the inclusion pressures become "tiny timekeepers" of the exhumation of their host rocks (Zhong et al., 2018, 2020). At the same time, some inclusion pressures can be reset even on laboratory timescales (Pummell and Thomas, 2024) that can be as short as minutes under some conditions (Campomenosi et al., 2023). The P_{inc} of such inclusions then provides an indication of the conditions prevailing in the rock at the time that resetting ends. In combination with dating inclusions (e.g. Kinny and Meyer, 1994), these few examples show that there is much more geology still to be learned from solid inclusion piezobarometry beyond a simple pressure and temperature of entrapment.

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