Direct dating of mid-crustal shear zones with synkinematic allanite: new in situ U-Th-Pb geochronological approaches applied to the Mont Blanc massif

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ABSTRACT

Dating the timing of motion on crustal shear zones is of tremendous importance for understanding the assembly of orogenic terranes. This objective is achieved in this paper by combining petrological and structural observations with novel developments in in situ U-Th-Pb geochronology of allanite. A greenschist facies shear zone within the Mont Blanc Massif is documented. Allanite is synkinematic and belongs to the mylonitic assemblage. LA-ICP-MS U-Th-Pb isotope analyses of allanite reveal high contents and highly radiogenic isotopic compositions of the common-Pb component. The use of measured Pb-isotope compositions of associated minerals (feldspars and chlorite) is critical for accurate common-Pb correction, and provides a powerful mechanism for linking allanite growth to the metamorphic assemblage. A mean 206Pb/238U age of 29.44 ± 0.95 Ma is accordingly taken for synkinematic allanite crystallisation under greenschist facies conditions. This age reflects the timing of the Mont Blanc underthrusting below the Penninic Front and highlights the potential of directly dating deformation with allanite.

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Introduction

Resolving the timing and rates of crustal deformation is fundamental to our understanding of tectonic and orogenic processes. Direct dating of deformation features by isotopic techniques has great potential to provide new insights into the complexities of orogenesis and tectonics in general. Of particular interest are shear zones, which can accommodate vast lateral and vertical crustal motions, control the development of tectonic features, and provide important pathways for orogenic and ore-forming fluids.

In contrast to constraining deformation ages through cross-cutting structural relationships, the direct dating approach focuses upon isotopic analysis of synkinematic minerals that belong to the metamorphic assemblage associated with deformation. Previous efforts at directly dating deformation focused upon Rb-Sr dating of high Rb/Sr phases (e.g. Freeman et al., 1997) and 40Ar/39Ar dating of white mica, amphibole and feldspar (e.g. Simon-Labric et al., 2009). In situ 40Ar/39Ar dating of re-crystallized micas by laser-heating techniques has successfully been applied to mylonites in low temperature conditions, but potential cm-scale mobility of Ar and excess-Ar may compromise successful thermochronology (Kelley, 2002; Mulch et al., 2005). Texturally controlled Rb-Sr dating also has great potential, but is prone to resetting by post-deformation fluid circulation (e.g. Wickman et al., 1983) and isotopic disequilibrium on the thin-section scale (Frey et al., 1976).

Uranium and thorium rich accessory phases, such as zircon and monazite, provide robust ages in many geological settings. Although mechanical modification of zircons in shear zones is relatively common, chemical modifications, and hence age resetting, are not (e.g. Wayne and Sinha, 1992; Moser et al., 2009). To a lesser extent, U-Pb techniques have been successfully applied to dynamically recrystallised titanite and monazite (Resor et al., 1996; Storey et al., 2004). Another accessory phase that is a prime target for geochronology in these environments is allanite, which is a rare earth element (REE) rich end-member of the epidote solid solution series [(Ca, REE, Th)[Fe, Al]3Si2O10(OH)]. The mineral is a key to the storage and mobility of REE, Th and U (Hermann, 2002; Gieré and Sorensen, 2004), and offers geochronological information that can be linked with physico-chemical conditions (e.g. pressure, temperature conditions), based upon petrological observations. Allanite is also commonly found associated with monazite (Janots et al., 2008). Epidote and clinozoisite generally surround allanite. As thermodynamic stabilities of these phases can be calculated, it is possible to make quantitative links between the timing of monazite/allanite/clinozoisite/epidote growth and
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Geological setting of the Mont Blanc shear zones

The MBM belongs to a suite of Variscan external crystalline massifs of the western Alps (Fig. 1). It is composed of gneisses and a granitic batholith that crystallised at 300 ± 3 Ma (Bussy and von Raumer, 1994). The general deformation pattern of the MBM consists of sub-vertical narrow (1–50 m) shear zones, arranged in a fan-like geometry, separated by low strain domains (100–500 m). Deformation, strain localisation and associated upper greenschist facies metamorphism in the MBM have been considered Alpine in age (Rolland et al., 2008).

The majority of shear zones are transpressive and form a complex network of anastomosing NNE-SSW (N40-60°E) and N-S (N160-20°E) components with sub-vertical stretching lineations (Fig. 1). These two groups of shear zones have a dextral and sinistral component, respectively, resulting in a NW-SE compression regime (Rossi et al., 2005). In addition, domains of distinct mineral assemblages within shear zones can be recognised with an NW to SE zoning (Fig. 1). In the NW part, the dominant assemblage is epidote, quartz and muscovite, in the central part, phlogopite, chlorite and quartz are present, and in the SE part, phengite dominates.

Microstructure and petrology of the sample

This study focuses on a greenschist facies shear zone in the chlorite- and phlogopite-bearing domain (central domain of Fig. 1). This shear zone may be observed along-strike inside the Mont Blanc tunnel, emphasising its regional two-dimensional exposure. Lineations are sub-vertical and shear sense indicators reflect exclusively pure shear, i.e. horizontal shortening. The main mylonitic foliation is marked by chlorite, elongated K-feldspars and recrystallised quartz. Locally, phlogopite is present as larger crystals, and albite crystals occur in low strain parts of the sections of the shear zone. Thermodynamic phase equilibria indicate that shear zone recrystallisation occurred at 0.51 ± 0.05 GPa and 400 ± 25 °C (Rolland et al., 2003).

The contact between high-strain domains and low-strain granite pods is anastomosing and finger-like. In addition, a low Fe-content of fabric minerals is associated with high bulk-rock Mg/(Mg + Fe) values compared to the unaltered, undeformed granite.

Fig. 1 Simplified geological map and cross-section (without vertical exaggeration) of the Mont Blanc Massif with main structural features and mineral assemblages (modified from Rolland et al., 2008). UTM Coordinates of studied sample: (WGS84): N5084.005; E343.502.
These evidence suggest a localised fluid alteration front associated with greenschist facies metamorphism. These are Mg-rich fluids percolating upwards in the core of the MBM, with high fluid/rock ratios (Rossi et al., 2005).

Small (<100 μm), newly crystallised REE-rich phases are parallel to, or occasionally overgrow, the main greenschist facies mylonitic foliation (Fig. 2). New aeschynite and elongate allanite crystals are idioblastic and contain chlorite, albite and uraninite inclusions. In addition, there are clear differences in zoning, texture, composition and REE patterns between allanite in the host granite and in the shear zone (Rolland et al., 2003), with the shear zone grains being homogeneous in major and REE elements composition (Fig. 3). These observations indicate that, texturally, allanite is not inherited, is in equilibrium with the mylonitic assemblage and therefore synkinematic.

**Allanite dating methods**

Laser ablation (LA)-ICP-MS U-Th-Pb isotope analyses were undertaken at the University of Portsmouth, using a New Wave 213 nm Nd:YAG laser coupled with an Agilent 7500cs ICP-MS. Analytical protocols and instrument conditions are described in detail by Darling et al. (2012a). Key points of the methodology are: (i) line-raster ablation, in order to minimise time-dependent elemental fractionation; and (ii) external normalisation to the zircon standard Plešovice (Slama et al., 2008); (iii) the use of measured 204Pb to correct for inherited common-Pb. Accuracy was monitored via analyses of the allanite reference materials Tara, Mucrone, BONA and SISS (von Blanckenburg, 1992; Gregory et al., 2007; Cenki-Tok et al., 2011), for each of which mean common Pb-corrected 208Pb/232Th ages are within uncertainty of reference values, with uncertainties of 0.5–1.5% (2σ; Table 1).

Pb-isotope measurements of albite and chlorite were undertaken in a polished block of the studied sample at the University of Bristol, using a New Wave Research 193 nm ArF Excimer laser coupled with a ThermoFinnigan Neptune multi-collector (MC)-ICP-MS. Analytical procedures followed those of Foster and Vance (2006) and Darling et al. (2012b). Sample measurements were normalised to the NIST 610 glass standard, and NIST 612 was used to monitor accuracy and precision, yielding mean 206Pb/204Pb, 207Pb/204Pb and 208Pb/204Pb values of 17.105 ± 0.008, 15.521 ± 0.006, and 37.035 ± 0.013 respectively (120 μm nominal beam diameter; n = 12; all uncertainties 2σ).

**Allanite U-Th-Pb systematics**

The results of allanite analyses are detailed in Table 2, and key points are summarised in Fig. 4. The measured grains typically have high common-Pb contents, with measured 206Pb/204Pb and 208Pb/204Pb varying from 32–184 to 60–474 respectively. A Tera-Wasserburg type concordia plot (Fig. 4A) highlights the dominance of common-Pb. The regression has a poorly defined lower intercept of 21 ± 18 Ma (all uncertainties 2σ, unless otherwise stated), but interestingly has a very low y-intercept (0.41 ± 0.02), which reflects the 207Pb/206Pb composition of the common-Pb component (Tera and Wasserburg, 1972). This value is far removed from model terrestrial Pb-isotope evolution curves (total 207Pb/206Pb range 0.84–1.11; Stacey and Kramers, 1975).

The isotopic composition of common-Pb in the metamorphic assemblage associated with allanite was...
further investigated via LA-MC-ICP-MS Pb-isotope measurements of albite and chlorite from the same sample (Table 3). The measured albite crystals have highly radiogenic age-corrected Pb-isotope values, with weighted mean $^{206}\text{Pb}/^{204}\text{Pb}$, $^{207}\text{Pb}/^{204}\text{Pb}$ and $^{208}\text{Pb}/^{204}\text{Pb}$ values of 29.75 ± 0.39, 16.08 ± 0.12 and 46.05 ± 0.34 respectively (Fig. 4B). These ratios were age corrected to 28.2 ± 2.6 Ma (the allanite Th-Pb isochron age; Fig. 4C) using measured $^{238}\text{U}/^{204}\text{Pb}$ and $^{232}\text{Th}/^{204}\text{Pb}$, although the magnitude of this correction is less than analytical uncertainty in albites due to low $^{238}\text{U}/^{204}\text{Pb}$ (<5.1) and $^{232}\text{Th}/^{204}\text{Pb}$ (<0.11). The single measurement of chlorite has $^{206}\text{Pb}/^{204}\text{Pb}$, $^{207}\text{Pb}/^{204}\text{Pb}$ and $^{208}\text{Pb}/^{204}\text{Pb}$ values within uncertainty of the albite mean. Two further analyses of chlorite were rejected due to ablation through Pb-rich inclusions and very low Pb concentration in one case. Importantly, these measured albite and chlorite values are within uncertainty of the initial $^{206}\text{Pb}/^{204}\text{Pb}$ and $^{208}\text{Pb}/^{204}\text{Pb}$ values provided by the Th-Pb and U-Pb isochrons (Fig. 4C–D). As suggested by textural and geochemical evidence, this indicates that allanite is in equilibrium with the mylonitic assemblage and offers new opportunities for accurate common-Pb correction.

Previous studies have advocated the use of assumed common-Pb compositions taken from model terrestrial Pb-isotope evolution curves (Stacey and Kramers, 1975), in order to correct Pb-isotope signals for common-Pb in magmatic allanite (e.g. Gregory et al., 2007). However, as shown in Figure 4E, Th/Pb ages corrected using a Stacey and Kramers (1975) composition are scattered and do not define a single age population. In contrast, when the measured Pb-isotope composition is used for correction, the data define a single age population, with a weighted mean of 29.44 ± 0.95 Ma (MSWD = 0.85; n = 30; Fig. 4F). This value is within uncertainty of the Th-Pb isochron age for these data (28.2 ± 2.6 Ma; Fig. 4C), which is independent of common-Pb correction. A necessary pre-requisite for this approach is that metamorphic allanite, chlorite and albite have

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Table 1 Summary of allanite U-Th-Pb isotope data for reference materials. The reference ages were measured by thermal ionisation mass spectrometry (TIMS; von Blanckenburg, 1992) or Sensitive High Resolution Ion Microprobe (SHRIMP; Gregory et al., 2007). All analyses at the School of Earth and Environmental Science, University of Portsmouth, UK.

| Material | No. | analyses | raster (μm) | 206Pb/204Pb | 207Pb/204Pb | 208Pb/204Pb | 206com % | 207com % | 208com % | Mean common-Pb age (Ma) | Notes | Ref. | age (Ma) | Notes | Ref. |
|----------|-----|----------|----------|------------|------------|------------|----------|----------|----------|------------------------|--------|------|--------|--------|------|
| SSS      | 4   | 25 × 40  | 2        | 57         | 4          | 59         | 3        | 93       | 4        | 48        | 33 353 2.5 2 2.2 2.2 31.5 ± 0.35 | Large excess | von Blanckenburg (1992) |
| BONA     | 4   | 25 × 40  | 4        | 59         | 9          | 79         | 14       | 56       | 5        | 82        | 25 45 35 310 0.7 30.1 ± 0.4 | Large excess | von Blanckenburg (1992) |
| Tara      | 6   | 25 × 40  | 4        | 59         | 9          | 79         | 14       | 56       | 5        | 82        | 14 42.4 11 416.4 3.4 414.9 ± 3.3 | Gregory et al. (2007) |
| Mucrone   | 4   | 25 × 40  | 90       | 40         | 588        | 159        | 23       | 936      | 7        | 285       | 34 316 55 55 126 ± 5.5 | Gregory et al. (2011) |

All analyses at the School of Earth and Environmental Science, University of Portsmouth, UK.

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| Identifier  | Pb 40% | Pb 5% | Pb 1% | Pb 0.1% | Pb 0.01% | Pb 0.001% | Pb 0.0001% | Pb 0.00001% | Pb 0.000001% |
|-------------|--------|-------|-------|----------|-----------|------------|-------------|---------------|----------------|
| Au16C07     | 25     | 40    | 15    | 7        | 1         | 1          | 37          | 52             | 64             |
| Au16C08     | 25     | 40    | 15    | 7        | 1         | 1          | 37          | 52             | 64             |
| Au16C09     | 25     | 40    | 15    | 7        | 1         | 1          | 37          | 52             | 64             |
| Au16C11     | 25     | 40    | 15    | 7        | 1         | 1          | 37          | 52             | 64             |
| Au16C12     | 25     | 40    | 15    | 7        | 1         | 1          | 37          | 52             | 64             |
| Au16C13     | 25     | 40    | 15    | 7        | 1         | 1          | 37          | 52             | 64             |
| Au16C15     | 25     | 40    | 15    | 7        | 1         | 1          | 37          | 52             | 64             |
| Au16C16     | 25     | 40    | 15    | 7        | 1         | 1          | 37          | 52             | 64             |

common Pb-corrected using measured Pb-isotope ratios from feldspars and chlorites.
equilibrated in the same common-Pb reservoir. In this case, this condition is fulfilled as: (i) allanites contain chlorite and albite inclusions; and (ii) initial $^{208}\text{Pb}/^{204}\text{Pb}$ from the allanite Th-Pb isochron ($47.1 \pm 2.3$) is within error of the measured albite and chlorite values. There is minor variability in the initial Pb-isotope values of matrix phases that is greater than analytical precision. For example, $^{206}\text{Pb}/^{204}\text{Pb}$ in albites range from $29.53 \pm 0.28$ to $30.40 \pm 0.31$. Further analyses are required to test the scale of Pb-isotope heterogeneity in such metamorphic systems, although this range of variability has a minor effect on calculated ages compared to uncertainty in measurement of $^{204}\text{Pb}$ in this study. In combination, this analysis of Pb-isotope systematics in allanite and other phases of the metamorphic assemblage provides confidence that the weighted mean $^{206}\text{Pb}/^{232}\text{Th}$ age represents the best estimate of crystallisation age of allanites in the studied sample.

Fig. 4 Allanite U-Th-Pb ICP-MS isotope data for sample C33. (A) Tera-Wasserburg plot of allanite analyses uncorrected for common-Pb. (B) Age-corrected Pb-isotope compositions of albites and chlorite. (C) Th-Pb and (D) U-Pb isochron plots for the allanite analyses of this study. A calculated U-Pb isochron corresponding to an age of 30 Ma, and using the albite and chlorite measured Pb-isotope composition, is also shown in B for comparison. Data are not corrected for common-Pb. All uncertainties are at the 95% confidence level. (E)–(F) Common Pb-corrected Pb/Th ages of allanites, showing the importance of using measured Pb-isotope ratios for common-Pb correction.
Common Pb-corrected $^{206}\text{Pb}/^{238}\text{U}$ ages are highly variable, and significantly older than Th-Pb ages. The slope of the U-Pb isochron (Fig. 4D) also provides a significantly older age ($122\pm 32\text{ Ma}$). Two lines of evidence suggest that the $^{238}\text{U}-^{206}\text{Pb}$ system is compromised by excess $^{206}\text{Pb}$, either from $^{230}\text{Th}$ disequilibrium (e.g. Schärer, 1984; von Blanckenburg, 1992), inherited radiogenic Pb from a U rich precursor (Romer and Siegesmund, 2003), labile $^{206}\text{Pb}$ from another source, or a combination of these factors: (i) the initial $^{206}\text{Pb}/^{204}\text{Pb}$ provided by the U-Pb regression is within uncertainty of the composition of albite and chlorite, suggesting variable levels of $^{206}\text{Pb}$ incorporation into different grains of allanite; and (ii) the low $^{207}\text{Pb}/^{206}\text{Pb}$ intercept of the Tera-Wasserburg regression ($0.41\pm 0.02$) compared to the albite-chlorite initial value ($0.540\pm 0.014$). Furthermore, there is a positive correlation between the Th/U ratios and $^{206}\text{Pb}/^{238}\text{U}$ ages of the allanite population, which suggests that U-Th fractionation during crystallisation of allanite, causing initial $^{230}\text{Th}$ disequilibrium ($t_{1/2} = 75\text{ kyr}$; e.g. Schärer, 1984; von Blanckenburg, 1992), may be the dominant control on excess $^{206}\text{Pb}$.

**Discussion and conclusion**

Significant improvement in understanding the petrology and geochemistry of allanite was made in the past decade (e.g. Janots et al., 2008), but the effects of deformation and recrystallisation on allanite U-Th-Pb systematics have as yet been little studied. A first attempt to test whether allanite may be used to infer the age of mylonitisation revealed that the mineral can be remarkably resistant to deformation in relatively dry conditions at eclogite facies due to mechanical shielding that prevents chemical equilibration (Cenki-Tok et al., 2011). This study shows that new crystallisation of allanite may occur in shear zones associated with fluid flux, which can efficiently reset U-Th-Pb isotopic ratios. In the studied shear zone, allanite texturally belongs to the greenschist facies assemblage.
allanite is expected to be stable at these PT conditions (ca. 0.5 GPa and 400 °C; Rolland et al., 2003).

The U-Th-Pb closure temperature of allanite is estimated to be above 700 °C (Heaman and Parrish, 1991), because: (i) allanite has been shown to remain close to Pb loss and retain trace element and Sr-Nd isotope zonation during prolonged magmatic conditions (Oberli et al., 2004; Gregory et al., 2009); and (ii) in general zoning patterns developed in allanites during prograde metamorphic growth may be retained through peak conditions (Janots et al., 2008). Due to the high closure temperature of allanite, the age of this study is interpreted as a crystallisation age that records shear zone activation under greenschist facies conditions.

The allanite U-Th-Pb in situ isotope data from this study also highlight the importance of using measured Pb-isotope compositions for common-Pb, particularly in metamorphic rocks in which several processes may fractionate Th/Pb, U/Pb or Th/U. Measured albite and chlorite indicate that fluids associated with the metasomatic event (Rossi et al., 2005) had highly radiogenic Pb-isotope compositions. Similar fluid compositions have already been recognised in this zone of the MBM (Marshall et al., 1998). These were interpreted as being related to the emplacement of the Penninic Front that tapped fluid from deep crustal and mantle sources (Rossi et al., 2005). Indeed, metasomatized rocks with similar 87 Sr/86 Sr calcite ratios may also be found at the Penninic Front itself (Rossi et al., 2005).

In summary, the mean Th-Pb age of 29.4 ± 1.0 Ma is taken as the crystallisation age of allanites in the studied shear zone, and hence the age of deformation and fluid percolation. In the SE domain of the MBM (Fig. 1), shear zones yielded younger 40 Ar-39 Ar crystallisation ages (16 Ma; Rolland et al., 2008). The diachronity revealed by these two studies highlights a succession of previously unrecognised events in the MBM including: (i) ductile deformation and fluid percolation at c. 29 Ma ascribed to activation of the Penninic Front, which is also recognised further to the South in the Pelvoux Massif (Simon-Labrie et al., 2009); and (ii) reactivation of the shear zones at 16–14 Ma is ascribed to the onset of exhumation in relation with the rotation of Apulia (Rolland et al., 2012).

More generally, allanite may be found in a wide variety of lithologies (felsic, pelitic and mafic). It is a petrologically important mineral in greenschist to amphibolite grade rocks typical of the upper-mid crust, particularly when found together with monazite. As its closure temperature is well above these moderate mid-crustal temperatures (ca. 700 °C; Oberli et al., 2004), allanite ages in these environments are likely to record crystallisation rather than cooling. Allanite therefore helps us understand the timing and rates of low to medium temperature processes, which are known to be difficult to date.

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