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An evidence-based approach to accurate interpretation of $^{40}Ar/^{39}Ar$ ages from basaltic rocks

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A B S T R A C T

Since its inception in the mid-1960s, the $^{40}Ar/^{39}Ar$ dating technique has been the premier method for determining the eruption ages of basaltic rocks, providing valuable insights into a plethora of terrestrial and planetary processes. Advances in multi-collector mass spectrometry and improved sample preparation procedures are enabling ever-improving analytical precision and clearer evaluation of the isotopic disturbances that affect many basaltic samples and cause discordant $^{40}Ar/^{39}Ar$ age spectra.

Here, we present $^{40}Ar/^{39}Ar$ step-heating data for multiple samples from two Quaternary basalt flows (0.8038 ± 0.0017 and 2.309 ± 0.009 Ma) of the intraplate Newer Volcanic Province, southeast Australia. A small proportion of these samples give concordant $^{40}Ar/^{39}Ar$ results, but most are variably discordant. The factors controlling these disturbances and implications for accurate age determination are examined and modelled in both step-heating spectra and inverse isochron space. We demonstrate that the proportion of radiogenic ^{40}Ar ($^{40}Ar^*$) present in these samples strongly influences the nature of the discordance reflected in $^{40}Ar/^{39}Ar$ data. Mass-dependent fractionation appears to have a major influence on low-$^{40}Ar^*$ samples, whereas ^{39}Ar recoil loss/redistribution effects are evident in samples with higher $^{40}Ar^*$ proportions. The impact of mass fractionation is quantified via step-heating analyses of unirradiated basalt, whereby a $\sim$4% difference in $^{38}Ar/^{36}Ar$ ratios is observed between low- and high-temperature heating steps.

On an inverse isochron plot ($^{39}Ar/^{40}Ar$ vs $^{36}Ar/^{40}Ar$), isotopic disturbance for groundmass samples primarily manifests as isochron rotation, leading to a negative correlation between initial $^{40}Ar/^{36}Ar$ ($[^{40}Ar/^{36}Ar]_i$) values and associated $^{40}Ar/^{39}Ar$ ages. We propose a new framework for the interpretation of $^{40}Ar/^{39}Ar$ step-heating data for basaltic samples, through judicious evaluation of inverse isochron data, $(^{40}Ar/^{36}Ar)_i$ ratios and inverse isochron ages. Results from this study suggest that only samples exhibiting both flat $^{40}Ar/^{39}Ar$ age spectra and atmospheric $(^{40}Ar/^{36}Ar)_i$ ratios yield accurate eruption ages; in the case of more discordant age spectra, intermediate temperature steps with atmospheric $(^{40}Ar/^{36}Ar)_i$ ratios may provide the closest approximation of the eruption age.

1. Introduction

Precise and accurate ages for basaltic rocks are critical for constraining research questions ranging from global tectonics to magma petrogenesis, paleoclimatology and volcanic hazard assessment (e.g., Rampino and Stothers, 1988; Storey et al., 1995). $^{40}Ar/^{39}Ar$ geochronology remains the primary method for obtaining eruption ages of basaltic rocks (e.g., Hirano et al., 2008; Jourdan et al., 2014; Oostingh et al., 2017; Singer et al., 2014). However, accurate $^{40}Ar/^{39}Ar$ dating of basaltic rocks is often challenging, due to relatively low potassium contents and difficulties separating target potassic mineral phases. *Whole-rock* dating, as was commonly used in earlier K–Ar studies (e.g., McDougall et al., 1966), provides one approach to this problem, but assumes that all mineral phases crystallised contemporaneously (i.e., no xenocrysts, early phenocrysts or alteration). $^{40}Ar/^{39}Ar$ analysis of *groundmass* basaltic material is a refinement of the whole-rock technique, whereby relatively high-K aggregate grains are separated from inherited and altered phases (Jicha et al., 2012; Koppers et al., 2000; Singer et al., 2004). This approach has been employed in several recent high-precision $^{40}Ar/^{39}Ar$ studies on basaltic rocks (e.g., Matchan and Phillips, 2014; Oostingh et al., 2017). In some cases, $^{40}Ar/^{39}Ar$ studies have targeted plagioclase feldspar (e.g., Pringle et al., 1991; Renne et al., 1990). However, basaltic plagioclase typically contains <0.5 wt.% K_2O and is often fine-grained, making mineral separation time-consuming, particularly for Quaternary

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samples. The current study focuses exclusively on $^{40}Ar/^{39}Ar$ analyses of groundmass basaltic material; see Koppers et al. (2000) for a discussion of typical mineral phases outgassed during $^{40}Ar/^{39}Ar$ dating of basaltic groundmass.

Fresh, holocrystalline groundmass basaltic samples that have not experienced radiogenic ^{40}Ar ($^{40}Ar^*$) loss, and are devoid of extraneous argon, sometimes produce relatively concordant step-heating age spectra (e.g., Singer et al., 2014). However, many basalts are non-ideal, possibly due to complex thermal histories, significant glass contents and/or alteration (e.g., Baksi, 2007; Fleck et al., 1977; Foland et al., 1993; Koppers et al., 2000, 2003), and the interpretation of $^{40}Ar/^{39}Ar$ ages from discordant age spectra is often subjective and open to debate (e.g., Baksi, 2014). The order of magnitude improvement in analytical precision afforded by new multi-collector mass spectrometers (Matchan and Phillips, 2014), reveals more subtle disturbances of argon isotopic ratios, such that perfectly concordant step-heating spectra are rare.

When confronted with discordant $^{40}Ar/^{39}Ar$ age spectra, additional insight can be provided from *inverse isochron diagrams*, plotting $^{36}Ar/^{40}Ar$ versus $^{39}Ar/^{40}Ar$ ratios (Turner, 1971). In inverse isochron space, the $^{39}Ar/^{40}Ar$ intercept is inversely equivalent to the $^{40}Ar^*/^{39}Ar$ ratio of the sample, and can be used to derive an apparent age. The $^{36}Ar/^{40}Ar$ intercept yields the 'initial' $^{40}Ar/^{36}Ar$ ratio [$(^{40}Ar/^{36}Ar)_i$], and undisturbed basaltic groundmass samples are expected to give values within error of the atmospheric ratio of 298.56 ± 0.62 (2σ; Lee et al., 2006). The primary use of isochron analysis in $^{40}Ar/^{39}Ar$ dating is to evaluate the assumption of an atmospheric composition for the *trapped* argon component (McDougall and Harrison, 1999). Previous work has attributed supra-atmospheric $(^{40}Ar/^{36}Ar)_i$ ratios to excess ^{40}Ar, and sub-atmospheric ratios to $^{40}Ar^*$ loss (e.g., Baksi, 2014; Phillips and Onstott, 1986). In some cases, non-atmospheric $(^{40}Ar/^{36}Ar)_i$ ratios have also been used to re-calculate $^{40}Ar/^{39}Ar$ ages to improve the concordance of age spectra (e.g., Heizler and Harrison, 1988; Hodges et al., 2015; Oostingh et al., 2017). However, the validity of these interpretations and data treatment approaches have yet to be rigorously tested.

In this study, we compare apparent ages and $(^{40}Ar/^{36}Ar)_i$ ratios measured on multiple samples of groundmass material collected from two basaltic lava flows from the Newer Volcanic Province, southeast Australia. These samples produced variably disturbed age spectra and characteristic signatures in inverse isochron space that can be linked to mass fractionation and ^{39}Ar recoil loss/redistribution processes. We model the various types of isotopic disturbance expected for basaltic samples, and provide an evidence-based approach for the interpretation of $^{40}Ar/^{39}Ar$ age data from basaltic samples.

2. Sample locations

Samples were selected from two well preserved basaltic lava flows in the Melbourne area of southeast Australia (Fig. 1a). These flows form part of the intraplate Neogene–Quaternary Newer Volcanic Province (NVP), a predominantly basaltic lava field that covers approximately 23,000 km^2 of Victoria and South Australia (e.g., van den Hove et al., 2017). Previous K–Ar (e.g., Aziz-ur-Rahman and McDougall, 1972; Gray and McDougall, 2009; McDougall et al., 1966; McDougall, 1975) and $^{40}Ar/^{39}Ar$ dating (e.g., Hare et al., 2005; Matchan and Phillips, 2011; Matchan and Phillips, 2014; Matchan et al., 2018; Oostingh et al., 2017), combined with cosmogenic dating studies (Gillen et al., 2010; Stone et al., 1997), thermoluminescence (Smith and Prescott, 1987) and ^{14}C (Gouramanis et al., 2010) work on the youngest eruption points (<100 ka), have constrained an age range of ~4.6 Ma to ~5 ka for the NVP.

The Melbourne area is located on the eastern margin of the NVP (Fig. 1b). A complex history of volcanism in this relatively confined area has been inferred from previous K–Ar work (Gray and McDougall, 2009; McDougall et al., 1966), spanning 4.6 to 0.8 Ma. Two large-volume lava flows in the Melbourne area with existing K–Ar age constraints were selected for this study, and are termed Flow A and Flow B. The lavas of Flow A travelled over 40 km, filling several tributaries and river channels to the north of Melbourne and extending southwards towards the current central business district (CBD) and Port Phillip Bay areas, forming a flow front up to 8 km wide (Fig. 1b). A total of 82 outcrop and borehole samples (0–57.6 m depth) were collected along Flow A, and the 11 freshest samples were selected for $^{40}Ar/^{39}Ar$ dating. The age of Flow A is constrained at 0.72 ± 0.08 Ma (2σ) to 0.99 ± 0.12 Ma (2σ) by K–Ar dating studies (Gray and McDougall, 2009; McDougall et al., 1966).

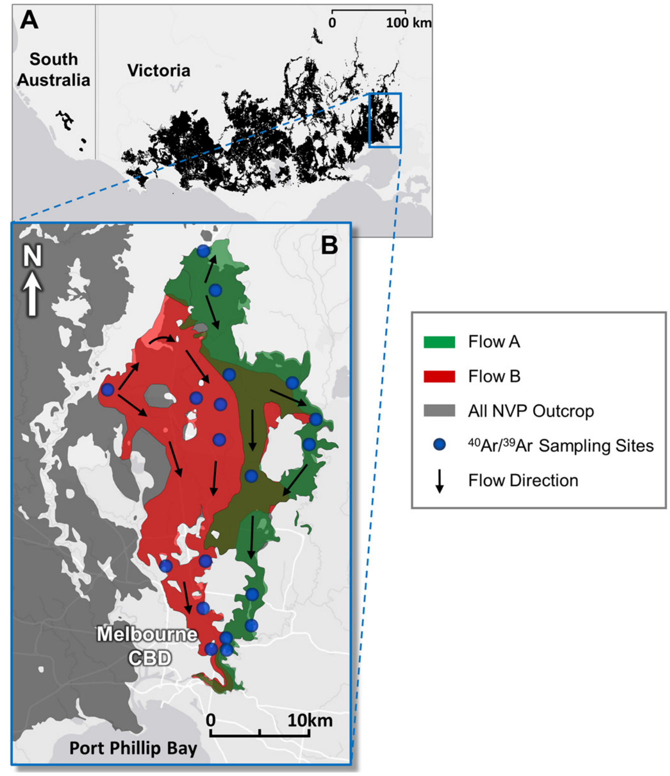

Fig. 1. (a) Location of the Newer Volcanic Province in southeast Australia (shown in black). **(b)** Lava flows A and B in the Melbourne area. Flow directions are inferred from surface topography and borehole stratigraphy. Lighter colours are where flows A and B do not outcrop at surface. (For interpretation of the colours in the figures, the reader is referred to the web version of this article.)

Flow B travelled a distance of nearly 40 km, beginning in an elevated area in Melbourne's north-west and filling several river valleys, following their courses towards the current Melbourne CBD and Port Phillip Bay (Fig. 1b). A total of 132 outcrop and borehole samples (0–57.9 m depth) were collected along Flow B, with eight samples selected for $^{40}Ar/^{39}Ar$ dating. Previous K–Ar studies of Flow B samples suggest an age between 2.26 ± 0.14 Ma (2σ) and 2.36 ± 0.10 Ma (2σ; Gray and McDougall, 2009; McDougall et al., 1966).

3. Methods

3.1. Sample preparation

Following screening of samples in thin-section to evaluate their suitability for $^{40}Ar/^{39}Ar$ dating, groundmass separates were prepared from rock fragments devoid of visible alteration, which were crushed, sieved to 150–250 μm, and washed to remove fine par-