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Earth and Planetary Science Letters 203 (2002) 499–506

EPSL

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The interpretation of inverse isochron diagrams in $^{40}Ar/^{39}Ar$ geochronology

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Received 6 December 2001; received in revised form 7 June 2002; accepted 17 July 2002

Abstract

The concepts involved in the construction and interpretation of inverse isochron diagrams used in $^{40}Ar/^{39}Ar$ geochronology are reviewed. The diagrams can be useful as a means of recognising atmospheric argon and excess ^{40}Ar, incorporated in the mineral lattice, which cannot be recognised from $^{40}Ar/^{39}Ar$ spectra. The age established using an inverse isochron plot, unlike that yielded by a spectrum, is not affected by trapped argon $^{40}Ar/^{36}Ar$ ratios that are different from the atmospheric argon ratio (e.g. due to excess ^{40}Ar), and may contribute to a better age interpretation. However, a heterogeneous distribution of excess ^{40}Ar or heterogeneous argon loss can cause 'false' isochrons, with axial intercepts indicating an incorrect age and an incorrect trapped argon composition. Inconsistency between the ages from a spectrum and from the associated inverse isochron plot may indicate the degree of inaccuracy of isochrons. However, it is possible that both the spectrum and inverse isochron yield the same incorrect age. The importance of considering all possible interpretations before assigning an age to a specimen is stressed.

Keywords: Ar/Ar; absolute age; isochrons; interpretations

1. Definitions and introduction

1.1. Types of argon

In $^{40}Ar/^{39}Ar$ geochronology, there are two types of argon: radiogenic and non-radiogenic. Definitions in this section are based on those of Dalrymple and Lanphere [1] and McDougall and Harrison [2]. *Radiogenic argon* includes ^{40}Ar accumulated from the decay of ^{40}K within the mineral or rock of interest. *Relic ^{40}Ar* is radiogenic argon that remains following a partial resetting event (e.g. by reheating), and can only in favourable cases be distinguished from radiogenic argon accumulated after re-closure (e.g. [3]). *Non-radiogenic argon* is argon that did not accumulate within the mineral of interest by radioactive decay of K, and comprises blank, trapped, cosmogenic and neutron-induced argon. *Blank argon* is unavoidable surficial argon introduced into the mass spectrometer with the sample. The argon ratios used in this paper are all hypothetical, and it is assumed that they have been corrected for blank argon. *Trapped argon* is incorporated within the mineral, and can consist of atmospheric argon

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PII: S0012-821X(02)00833-6

with or without a component of excess ^{40}Ar. *Excess ^{40}Ar* is released from older K-bearing minerals, typically during a heating event, and is subsequently trapped in the mineral of interest as it cools below its argon closure temperature. *Atmospheric argon* is argon from the Earth's atmosphere, which has an ^{40}Ar/^{36}Ar ratio of 295.5. In extraterrestrial samples, the 'atmospheric' argon composition commonly differs considerably. Extraterrestrial samples also contain *cosmogenic argon*, which is produced from cosmic-ray interaction with elements such as Ca, Ti and Fe, involving spallation reactions or neutron capture [2]. *Neutron-induced argon* is produced during irradiation of a sample in a nuclear reactor. While ^{39}Ar is produced from ^{39}K (see Section 2.1), other argon isotopes are produced by neutron interactions with K, Ca and Cl (e.g. ^{40}Ar from ^{40}K, ^{36}Ar from ^{40}Ca, and ^{39}Ar from ^{42}Ca), and need to be corrected for [2,4]. All argon ratios used in this paper are assumed to have been corrected. *Inherited argon* includes both radiogenic and non-radiogenic argon introduced into the mineral or rock specimen by contamination of older material (e.g. as inclusions). It involves the types of argon discussed above, and will not be discussed separately in this paper.

1.2. Introduction

Isochron plots and inverse isochron plots are widely used in ^{40}Ar/^{39}Ar geochronology. The main advantage of these plots over ^{40}Ar/^{39}Ar spectra is that excess ^{40}Ar, in addition to atmospheric argon, can be detected. In a step-heating spectrum, all steps are corrected for non-radiogenic ^{40}Ar, assuming that any non-radiogenic argon in the specimen has atmospheric composition. Using the atmospheric ^{40}Ar/^{36}Ar ratio of 295.5 (for terrestrial samples), the amount of non-radiogenic ^{40}Ar can then be calculated from the ^{36}Ar measured. The disadvantage of this method is that, where present, the additional trapped non-radiogenic ^{40}Ar (excess ^{40}Ar) cannot be detected. Consequently, after correction, all the ^{40}Ar, including the excess ^{40}Ar, is assumed to have been derived from decay of ^{40}K in the mineral, resulting in apparent ages that are too old.

Isochron plots (^{40}Ar/^{36}Ar versus ^{39}Ar/^{36}Ar) and inverse isochron plots (^{36}Ar/^{40}Ar versus ^{39}Ar/^{40}Ar) do not assume a non-radiogenic ^{40}Ar/^{36}Ar ratio of 295.5, and therefore they can be useful for the recognition of excess ^{40}Ar (but not relic ^{40}Ar). ^{36}Ar is the isotope present in the lowest concentrations, thus any measurement inaccuracies can introduce large errors on both axes of the isochron plot, resulting in large correlated errors [5]. Ratios calculated excluding ^{36}Ar from the nominator, as used in the inverse isochron diagram, consequently have a higher precision. Errors in the ratios also have a much smaller correlation, owing to the small fractional errors in the typically high concentrations of ^{40}Ar relative to the other isotopes [5]. The differences between isochron diagrams and inverse isochron diagrams are discussed in more detail in Roddick et al. [5], Dalrymple et al. [6] and McDougall and Harrison [2]. The inverse isochron diagram is generally considered more advantageous than the isochron diagram and is more commonly used. The aims of this paper are to review the basic concepts of the inverse isochron plot and to illustrate circumstances which may lead to an erroneous interpretation of this diagram.

2. Concepts and laboratory artifacts

2.1. Concepts

In order to comprehend the inverse isochron plot, the basic concepts of ^{40}Ar/^{39}Ar geochronology must be understood. A detailed description of the theory and methods is given by McDougall and Harrison [2] and references therein. An abbreviated explanation is given here. ^{40}Ar/^{39}Ar geochronology is based on decay of ^{40}K to ^{40}Ar. An age can be derived from the ^{40}K/^{40}Ar ratio and therefore this ratio needs to be determined. In ^{40}Ar/^{39}Ar geochronology, ^{39}Ar is formed from ^{39}K by irradiation in a nuclear reactor. The quantity of ^{39}Ar produced is proportional to the amount of ^{39}K present in the sample. With a knowledge of the natural abundances of ^{39}K relative to ^{40}K, the amount of ^{40}K can be determined, and the ^{40}K/^{40}Ar ratio and age calculated.

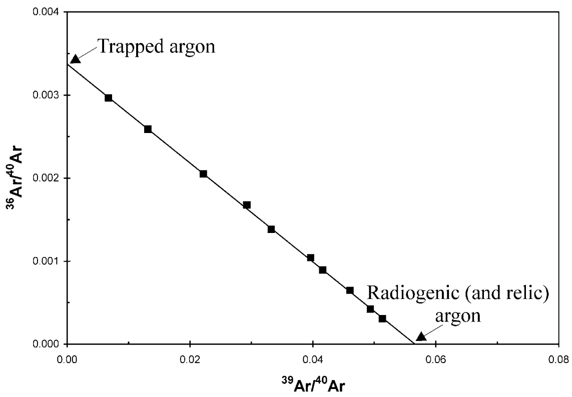

Fig. 1. An inverse isochron plot. The intercept with the $^{36}Ar/^{40}Ar$ axis gives the trapped argon composition. The intercept with the $^{39}Ar/^{40}Ar$ axis gives the radiogenic argon composition, from which the inverse isochron age can be calculated. All figures are corrected for blank argon.

The main advantage of $^{40}Ar/^{39}Ar$ geochronology, as opposed to $^{40}K/^{40}Ar$ geochronology, is that only the argon isotopes need to be measured (as opposed to measurement of potassium and argon separately) in order to establish the $^{40}K/^{40}Ar$ ratio and age.

The data presented in an inverse isochron plot represent different temperature steps from one mineral if the step-heating method is used, or different spots within one mineral if laser-ablation techniques are used (Fig. 1). The locations of the data presented on this isochron depend on their ratios of trapped argon relative to radiogenic argon; measurements with a high radiogenic component plot close to the $^{39}Ar/^{40}Ar$ axis, whereas measurements with a high trapped argon component plot close to the $^{36}Ar/^{40}Ar$ axis. If the gas released is a simple two-component mixture of trapped argon and radiogenic argon, then the points are likely to be distributed along a line, and the data can be regressed and an isochron can be plotted (Fig. 1; see [2]).

All ^{36}Ar is non-radiogenic. If the non-radiogenic argon component contains ^{36}Ar, then the age of the sample, unaffected by non-radiogenic argon, can be calculated from the intercept with the $^{39}Ar/^{40}Ar$ axis, where $^{36}Ar = 0$. However, the age can still be affected by relic ^{40}Ar. If the ^{36}Ar concentration is very small relative to the total ^{40}Ar concentration, then data will cluster along the $^{39}Ar/^{40}Ar$ axis, and non-radiogenic ^{40}Ar cannot be distinguished from radiogenic ^{40}Ar (see also Section 4.2).

The intercept of the isochron with the $^{36}Ar/^{40}Ar$ axis (where $^{39}Ar = 0$), represents the hypothetical $^{36}Ar/^{40}Ar$ ratio expected in the absence of any radiogenic argon. This can be visualised as follows. In a hypothetical sample containing no K, no ^{39}Ar will be produced during irradiation and $^{39}Ar/^{40}Ar = 0$. No radiogenic ^{40}Ar will accumulate and all argon present must be non-radiogenic. This non-radiogenic argon is trapped argon, which was included in the mineral when it cooled through its argon closure temperature. Therefore, the intercept with the $^{36}Ar/^{40}Ar$ axis indicates the $^{36}Ar/^{40}Ar$ ratio of the trapped argon.

In terrestrial samples, trapped ^{40}Ar can be atmospheric and/or excess ^{40}Ar. If the intercept of the isochron with the $^{36}Ar/^{40}Ar$ axis occurs at a ratio of 1/295.5 (the composition of atmospheric argon), then all trapped ^{40}Ar has an atmospheric origin. If the $^{36}Ar/^{40}Ar$ ratio of the intercept is lower, then excess ^{40}Ar is present in addition to atmospheric argon. The presence of excess ^{40}Ar will be addressed more extensively in Section 2.2. In extraterrestrial samples, the $^{36}Ar/^{40}Ar$ ratio of 'atmospheric' argon is different from that on Earth, and cosmogenic argon may be present. The intercept of the isochron with the $^{36}Ar/^{40}Ar$ axis (unaffected by excess ^{40}Ar) may be different from the terrestrial atmospheric $^{36}Ar/^{40}Ar$ ratio. In this paper, an atmospheric $^{40}Ar/^{36}Ar$ ratio of 295.5 is used, and absence of cosmogenic argon is assumed. For extraterrestrial samples, the intercept of the isochron with the $^{36}Ar/^{40}Ar$ axis may be different, but the principles discussed in this paper remain the same.

2.2. Laboratory artifacts

In this paper it is assumed that isotope ratios are correct. However, one must bear in mind that the measured isotopic ratios, and therefore the spectra and inverse isochrons, are affected by the accuracy of the applied corrections, e.g. for neutron-induced argon, recoil, and laboratory contamination (see also [7]). For example, if only a fraction of the neutron-induced ^{36}Ar is corrected for, then the $^{36}Ar/^{40}Ar$ ratios may be systematically too high and the inverse isochron may have erroneous intercepts (an erroneously elevated initial $^{36}Ar/^{40}Ar$ ratio and lowered apparent age). Koppers et al. [8] describe an example of recoil effects in altered seamount basalts from the Western Pacific and their influence on the inverse isochron diagram. Errors in the correction for laboratory contamination may be introduced during step-heating of the sample, because the system blank is only calibrated for a certain temperature range. For the temperature steps outside this range, argon introduced from the laboratory may differ in composition from the blank correction, thus inducing systematic errors. Other artifacts may be introduced, e.g. owing to the interlaboratory differences in error calculations and/or to uncertainties in the calculation of the value of the irradiation parameter, J [9]. It is important to understand the laboratory artifacts that affect the data. However, a detailed discussion is beyond the scope of this paper.

3. Homogeneous excess ^{40}Ar incorporation and argon loss

In this section the incorporation of excess ^{40}Ar and argon loss are assumed to be homogeneous within minerals, in order to simply explain the effects of these processes on data presented on inverse isochron plots. Realistic problems, including heterogeneous argon loss and excess ^{40}Ar distribution will be addressed in Section 4.

The effect of excess ^{40}Ar on the isochron plot is depicted in Fig. 2A. When part of the trapped argon is excess ^{40}Ar, analyses with a component of trapped argon will have a lower $^{36}Ar/^{40}Ar$ ratio than when all trapped argon is atmospheric. Assuming the trapped argon/radiogenic argon ratio remains the same for all data points, all points will plot at lower $^{36}Ar/^{40}Ar$ values, being lowered by an amount proportional to their component of trapped argon relative to radiogenic argon (e.g. point A in Fig. 2A moves down farther than point B). Analyses close to the $^{36}Ar/^{40}Ar$ axis have a high component of trapped argon, and would therefore also contain the largest amounts of ex-

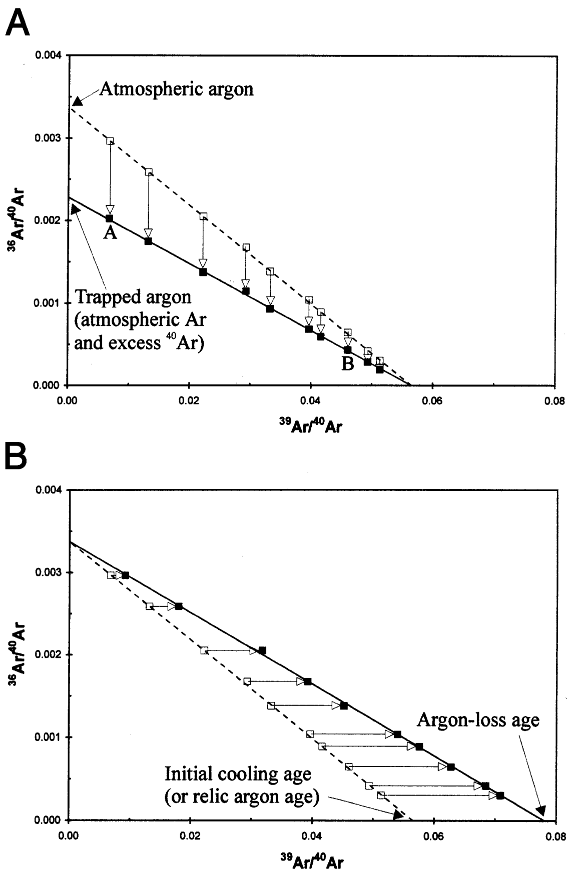

Fig. 2. The effects of an excess ^{40}Ar trapped argon component (A) and argon loss (B) on the inverse isochron plot. Undisturbed data are open squares and undisturbed isochrons are dashed. Solid squares and line show disturbed data and the isochron derived from those. Arrows show movement of individual data points as a result of excess ^{40}Ar (A) or argon loss (B). See text for further discussion.

cess ^{40}Ar, if present; consequently they would be lowered more than analyses close to the $^{39}Ar/^{40}Ar$ axis, which contain less trapped argon. In this way, the gradient of the isochron will be lowered though the intercept, while the $^{39}Ar/^{40}Ar$ axis remains fixed. The intercept with the $^{36}Ar/^{40}Ar$ axis will become lower, and the trapped $^{40}Ar/^{36}Ar$ ratio higher (which reveals the presence of excess ^{40}Ar). The inverse isochron age remains unchanged despite the incorporation of excess ^{40}Ar. This is an advantage of inverse isochron plots over spectra, because the apparent ages of spectra do become older in the presence of excess ^{40}Ar. Heizler and Harrison [10] (see also [2]) give examples in which the inverse isochron plot makes it possible to distinguish two compositionally distinct components of trapped argon within one mineral (i.e. by plotting two isochrons on one diagram, with the same ^{39}Ar/^{40}Ar intercept, but different ^{36}Ar/^{40}Ar intercepts). Also, they correct the spectrum steps for excess ^{40}Ar, using the trapped argon ^{36}Ar/^{40}Ar compositions of the isochrons on which the corresponding data points of the spectrum steps lie. In this way, uncorrected disturbed spectra can become plateaux after correction.

It is generally believed that, in nature, argon isotopes remain unfractionated during their escape from a mineral (by reheating or interaction with fluids). If this is true, then the ^{36}Ar/^{40}Ar ratio does not change (Fig. 2B) during loss. Because K is not released, the amount of ^{39}Ar that represents ^{39}K (produced in the nuclear reactor), remains unaffected by argon loss and the ^{39}Ar/^{40}Ar ratio increases as ^{40}Ar is lost. Consequently, the gradient of the isochron will decrease while the intercept with the ^{36}Ar/^{40}Ar axis remains fixed. The ^{39}Ar/^{40}Ar axis intercept will indicate a younger apparent age. If complete argon loss occurs, then the new age indicates the timing of the end of argon loss. If argon loss is not complete, then the inverse isochron age will represent a meaningless value between that of the initial cooling event and the end of the argon loss event (see Section 4.3 and Fig. 3).

4. Problems and pitfalls

4.1. Heterogeneous excess ^{40}Ar distribution and argon loss

In this paper, heterogeneous and homogeneous argon loss, or excess ^{40}Ar distribution, refer to changes in isotopic composition between the steps of an incremental heating spectrum. When different steps are lowered (or elevated) by various amounts, then argon loss (or excess ^{40}Ar incorporation) was heterogeneous and the spectrum is disturbed. Heterogeneity can be caused by the distribution of different argon compositions (e.g. excess ^{40}Ar, atmospheric argon and radiogenic argon) over different mineral lattice sites, which are therefore released under different conditions (during natural argon loss, or step-heating). Non-uniform relative amounts of the various types of argon in different physical locations within a mineral also cause heterogeneity. For example, a mineral rim may have more excess ^{40}Ar relative to radiogenic argon than the core, and a

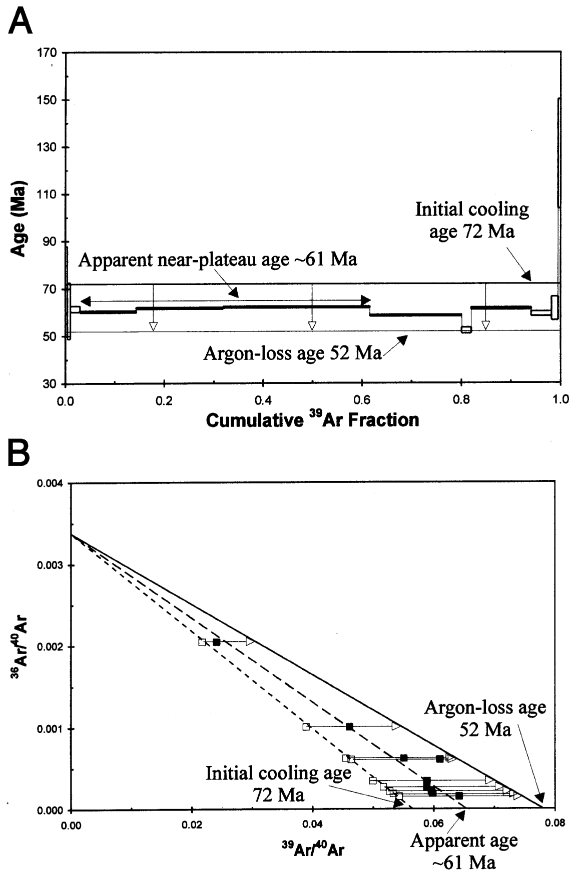

Fig. 3. The development of a false near-plateau (A) and isochron (B) with the same apparent age as a result of argon loss. The initial plateau and isochron as a result of cooling at 72 Ma, and the plateau and isochron, which would have resulted from complete argon loss at 52 Ma are dashed. Data from Spark [17]. The short dash indicates the initial isochron, the long dash indicates the isochron as a result of partial argon loss and the solid line indicates the isochron as it would be after complete argon loss. Other symbols as in Fig. 2.

rim or a crack may lose a large amount of radiogenic argon relative to the core. These mechanisms result in a heterogeneous isotopic composition of the remaining argon. Heterogeneous argon loss or excess ^{40}Ar distribution does not simply refer to the variable total amounts of argon present in (or lost from) different parts of the mineral.

Phase changes take place during step-heating, which (1) affect the conditions of argon release from lattice sites, (2) can homogenise the argon isotopes within the mineral, and (3) preferentially release argon from areas in the mineral in which the phase changes take place. Therefore, the composition of the argon may be unrelated to its corresponding distribution in the original mineral, and data from temperature steps cannot simply be related to specific locations within a mineral [11–14].

The incorporation of excess ^{40}Ar or argon loss is commonly heterogeneous. Data points on the resulting plots usually are too scattered to define precise inverse isochrons, intercept ages or trapped argon compositions, and the spectra are commonly disturbed.

4.2. Location of data points on the inverse isochron plot

If all analyses from one sample have a high radiogenic argon component relative to their trapped argon component, or if the ^{36}Ar component of the trapped argon is relatively small, then the data points will cluster close to the ^{39}Ar/^{40}Ar intercept. In this case, although the apparent inverse isochron age will be more precisely defined, it is difficult or impossible to accurately determine the composition of the trapped argon. Furthermore, the presence of trapped argon may remain unnoticed, and the inverse isochron age may be affected by undetected excess ^{40}Ar. Conversely, if all data represent large amounts of trapped argon relative to radiogenic argon, then the points will cluster close to the ^{36}Ar/^{40}Ar intercept and the inverse isochron age is difficult or impossible to determine. Additionally, if the trapped argon composition is not homogenous throughout the mineral, or argon loss occurred heterogeneously, then the data will plot with a large degree of scatter and cannot be regressed accurately. In this case, both the trapped argon composition and the intercept age become imprecise.

4.3. False inverse isochron plots

In some cases of argon loss or excess ^{40}Ar incorporation, data may yield 'false' plateaux and plot along a 'false' inverse isochron with meaningless intercepts. Spectra and inverse isochron plots can even give the same incorrect age, as demonstrated below.

Argon loss within a short period of time (e.g. <20 My) after the closure of the mineral for argon could lead to false (near-)plateaux and isochron ages. For example, in Fig. 3, hornblende yields an apparent age from both its spectrum and its inverse isochron plot of $\sim$61 Ma. However, the mineral may have cooled through its argon closure temperature at $\sim$72 Ma, and subsequently lost argon at $\sim$52 Ma. If argon loss occurs after a longer period of time after closure of the mineral for argon, false (near-)plateaux may still result if argon loss or gain is relatively homogeneous within the mineral. Similarly, excess ^{40}Ar can produce false (near-)plateaux that are older than the actual age, although, in general, excess ^{40}Ar does not result in older apparent inverse isochron ages, because the ^{39}Ar/^{40}Ar intercept remains the same (see also Section 3). False plateaux and inverse isochrons can also result from different mechanisms, e.g. mixing of two generations of minerals [15], or overprinting diffusion loss events [7].

More complicated false isochrons can be produced in unusual cases of heterogeneous argon loss or excess ^{40}Ar incorporation. Fig. 4A shows a hypothetical case in which excess ^{40}Ar does not result in the expected lowering of the isochron gradient while the intercept with the ^{39}Ar/^{40}Ar axis remains fixed. Instead, regression of the disturbed data results in an ^{36}Ar/^{40}Ar intercept at the atmospheric argon composition, and an apparent determined age from the ^{39}Ar/^{40}Ar intercept that is older than the real cooling age. In this case, the spectrum age would also be elevated. Sherlock and Arnaud [16] describe a geological example

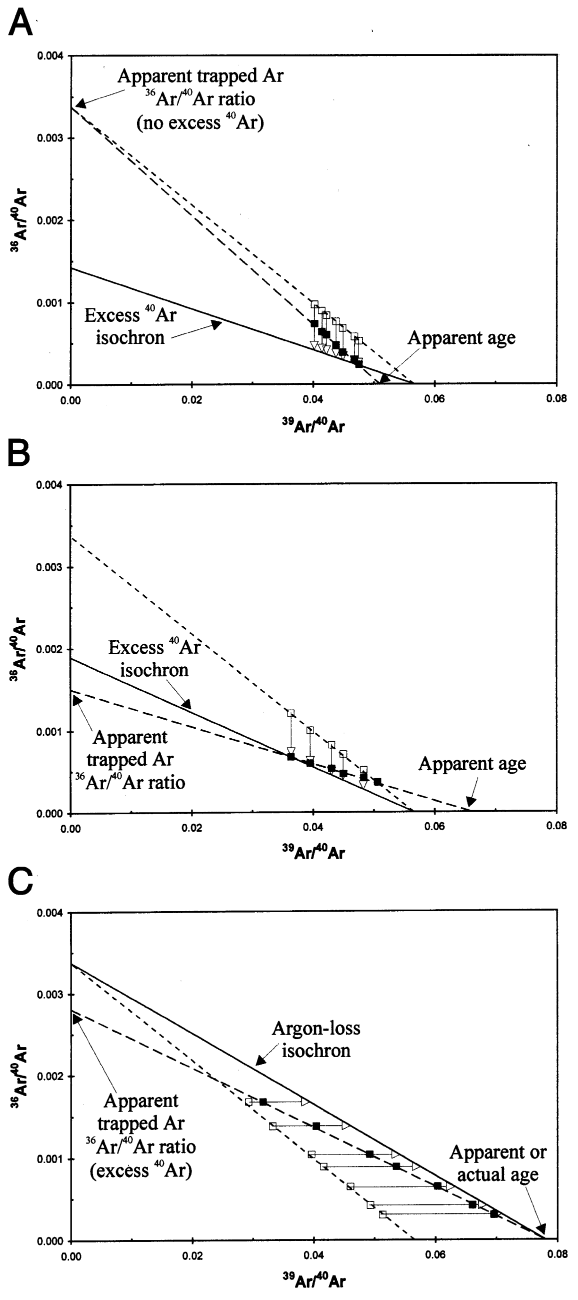

Fig. 4. The development of false isochrons as a result of heterogeneous excess ^{40}Ar distribution (A, B) or heterogeneous argon loss (C). Symbols as in Figs. 2 and 3.

of this from the Tavsanli Zone high-pressure metamorphic belt in Northwest Turkey.

Fig. 4B shows an inverse isochron plot with an apparent age younger than the real cooling age as a result of heterogeneously distributed excess ^{40}Ar. In this case, the spectrum age may still be older than the real age. However, if the excess ^{40}Ar has an excessively heterogeneous distribution within the mineral, it might not be possible to derive a (near-)plateau age and the inverse isochron age may be the only age obtainable.

Fig. 4C shows an inverse isochron plot displaying the effects of argon loss, but showing apparent excess ^{40}Ar. Here, the spectrum age could be the same as the isochron age, and the fact that argon loss occurred (resulting in a younger apparent age), rather than excess ^{40}Ar incorporation (which would result in an older spectrum age and an accurate isochron age), would remain unnoticed.

Examples of misleading inverse isochrons are numerous, and the interpretation of ^{40}Ar/^{39}Ar ages can become even more challenging when there is a complicated history with overprinting events, e.g. excess ^{40}Ar incorporation followed by argon loss. It is stressed here that the possibilities of false plateaux and inverse isochrons must always be considered carefully before a valid age interpretation can be made.

5. Conclusions

Inverse isochron diagrams can be useful when used in conjunction with spectra for the interpretation of ^{40}Ar/^{39}Ar ages. They allow the derivation of an ^{40}Ar/^{39}Ar age, without the assumption of a non-radiogenic ^{40}Ar/^{36}Ar ratio of 295.5. The ^{40}Ar/^{36}Ar ratio of trapped argon and the amount of excess ^{40}Ar can be determined, allowing subsequent correction of the spectra for excess ^{40}Ar. However, data can be too scattered to derive an isochron due to heterogeneous argon loss and/or a heterogeneous non-radiogenic argon distribution. Also, if isochrons can be derived, extreme care should be taken in the interpretation of these diagrams, because false isochrons exist with erroneous intercept ages and trapped argon compositions. Comparison with spectrum ages may not indicate that the inverse isochron is false. Therefore, false isochrons may lead to incorrect age interpretations, and consequently incorrect geological interpretations. Valuable age interpretations can only be made if possibilities for false isochrons and plateaux are examined thoroughly.

Acknowledgements

I thank Paul Williams, Peter Reynolds and especially James Whitehead for critically reading earlier drafts of this paper, and for valuable suggestions which greatly improved the manuscript. A constructive journal review by Jan Wijbrans enriched this paper significantly. This work was supported by an NSERC grant to Paul F. Williams.***[Boyle]***

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