

An Overview of Radiometric Techniques
used to Determine Kimberlite Emplacement
Ages

Andrea K. Noyes
Larry M. Heaman

University of Alberta, Edmonton Alberta

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A.K. Noyes and L.M. Heaman
University of Alberta, Edmonton, Alberta, Canada

Introduction

Emplacement ages of kimberlite provide us with knowledge on the timing of local and regional geotectonic controls associated with kimberlite formation and emplacement. Within a particular craton, varying emplacement ages may occur, providing information on the frequency and duration of magmatism for that region (Davis et al., 1996). Also, knowledge of the different kimberlite emplacement ages may provide clues to whether the kimberlites are diamond-bearing bodies. Conventional isotopic techniques used to determine kimberlite emplacement ages include Rb-Sr analyses on phlogopite and whole rocks; K-Ar and $^{40}\text{Ar}/^{39}\text{Ar}$ analyses of phlogopite and U-Pb analyses of perovskite and mantle zircon. However, an increasing number of kimberlites discovered in North America do not contain the 'traditional' minerals (phlogopite, perovskite and mantle zircon) often used to determine pipe emplacement ages. Typically, kimberlite magmatism is Jurassic to Cretaceous in age, however, kimberlite emplacement ages in Southern Africa are known to occur over a long period of geologic time (50 to 1700 Ma) (Allsopp et al., 1986). Kimberlites in North America have emplacement ages ranging from 50 Ma (Davis and Kjarsgaard, 1997) to 1100 Ma (Watson, 1967) (Eocene to Mesoproterozoic). A summary of the currently used radiometric techniques with examples from North American and South African kimberlites and an introduction to some preliminary results from new isotopic techniques for determining kimberlite emplacement ages are presented in this overview.

Isotopic Techniques

Kimberlites are mineralogically heterogeneous containing a proportion of xenocrysts making identification of primary minerals that have crystallized directly from the kimberlite magma, difficult. Therefore, it is preferable to apply more than one isotopic technique whenever possible to a single kimberlite pipe. One dating technique alone may yield geologically meaningless ages depending on such factors as alteration (surface weathering) and/or the presence of contaminants (calcite within fractures, xenocrystic mica, etc.).

Rb-Sr Techniques

Phlogopite occurs both as phenocrysts/megacrysts and in the groundmass of kimberlite. The coarser grained phlogopites are easier to date due to their large size, lower Sr content and thus higher Rb/Sr ratios (Allsopp et al., 1986). The main advantage of the Rb-Sr technique is that phlogopite is common in most kimberlites, phlogopite megacrysts are easy to extract and precise emplacement ages are attainable. However, problems do arise if the phlogopite grains are altered to chlorite and/or calcite or if xenocrystic mica is present in the sample. The presence of xenocrystic mica will result in excessive scatter on an isochron diagram (e.g. Rietfontein; Smith et al., 1985) and therefore, anomalously old ages for the kimberlite

(Allsopp et al., 1986). These problems can be avoided by selectively hand-picking the phlogopite grains under a binocular microscope to avoid altered grains and by applying an acid leaching technique to remove the presence of calcite along cleavage plains and grain fractures. This leaching technique does not disturb the Rb-Sr isotope systematics within the micas but reduces the amount of common Sr which improves the accuracy and precision of the calculated ages. Even though acid leaching techniques have been developed, the most reliable results are obtained from unaltered phlogopite grains (Brown et al., 1989). The Makganyene kimberlite in South Africa was used to test the acid leaching method (Brown et al., 1989). Three altered phlogopite fractions were picked of which one fraction was not leached, the second was leached for 10 minutes and the third fraction was leached for 12 hours. Sr contents within the samples decreased while Rb contents remained unchanged with prolonged leaching periods. Therefore, a contaminant with a high Sr content (presumably calcite or apatite) was removed from the phlogopite during the leaching process. As a result, leaching of the phlogopite samples increased the precision of the ages obtained for the Makganyene kimberlite. The unleached phlogopite samples yielded an isochron age of 120.9 ± 4.6 Ma, the samples that were leached for 10 minutes yielded an isochron age of 121.7 ± 3.6 Ma and the samples that were leached for 12 hours yield an isochron age of 121.2 ± 1.2 Ma (Brown et al., 1989).

Some examples of North American kimberlites dated with this technique include the Leslie and Panda kimberlite pipes in Lac de Gras (53.9 ± 2.0 Ma, 53.2 ± 3.8 Ma, respectively; Berg and Carlson, 1998), Kennedy Lake (539.0 ± 2.5 Ma; Carlson et al., 1998), Jericho pipe (171.9 ± 2.6 Ma; Heaman et al., 1997), Cross kimberlite, British Columbia (240-250 Ma; Smith et al., 1988), Somerset Island (100 Ma; Smith et al., 1989) kimberlites in the Northwest Territories, and the Attawapiskat kimberlites (155 to 170 Ma; Kong et al., 1998). An example of an anomalously old age is reported for the Cretaceous Ison Creek kimberlite, Elliott County, Kentucky (Rb-Sr biotite age of 257 ± 22 Ma; Zartman et al., 1967). This kimberlite has a 87 ± 3 Ma U-Pb perovskite age (Heaman, 1989), 80 ± 6 Ma K-Ar whole rock age (Basu et al., 1984) and 89 ± 2 Ma Rb-Sr phlogopite age (Alibert and Albarède, 1988).

K-Ar and $^{40}\text{Ar}/^{39}\text{Ar}$ Techniques

This isotopic system has been successfully used to date kimberlite whole rocks and micas. Micas selected for age determinations must be fresh and contain no inclusions unless they are cogenetic. Problems arise if samples contain excess argon or if samples have been subjected to argon loss due to metamorphic or heating events. The former situation will yield anomalously old and inaccurate ages for kimberlite emplacement (Allsopp et al., 1986). The Upper Canada mine kimberlites in Kirkland Lake Ontario, has been dated using the K-Ar whole rock technique yielding an age of 151 ± 8 Ma (Lee and Lawrence, 1968), which is in good agreement with a U-Pb perovskite age of 158 ± 2 Ma (Heaman, 1989) for this kimberlite.

The laser probe $^{40}\text{Ar}/^{39}\text{Ar}$ technique allows analysis of small groundmass phlogopite grains with precision. The selected grain is heated with the laser several times to release sufficient

argon gas for isotopic analysis (Faure, 1986). The advantages of this technique include analysis of individual grains avoiding alteration products or inclusions, rapid analyses (often analysed within thin sections for fine grained samples), several mineral grains can be analysed within the same rock sample, only small quantities of sample are necessary and good precision of age analysis (Faure, 1986; Phillips et al., 1998). The avoidance of altered and xenocrystic material enhances the precision of this technique. Some disadvantages include the rarity of groundmass phlogopite in some kimberlites and the susceptibility to alteration in small grain sizes. Also, the date obtained may represent the age of total release of argon, thereby, underestimating the age of the sample (Faure, 1986).

$^{40}\text{Ar}/^{39}\text{Ar}$ step heating technique involves the analysis of argon released during a series of heating experiments at varying temperatures. In an ideal situation, if the system has remained closed to argon and potassium since its cooling temperature, the dates at each step will be constant (Faure, 1986). However, if the grain exhibits varying amounts of argon loss, then a range of dates will result. The main advantage of the $^{40}\text{Ar}/^{39}\text{Ar}$ method is that it allows determination of a date that approaches the original cooling age minerals. However, the presence of excess argon can result in anomalously old ages and is always easy to detect, producing a plateau age that exceeds the crystallization age of the mineral (Faure, 1986).

U-Pb Technique

Groundmass perovskite is common in kimberlites and U-Pb perovskite geochronology is a highly successful tool for determining emplacement ages as perovskite contains high concentrations of uranium (11-348 ppm; Heaman, 1989). However, the small grain size hinders widespread usage of this mineral. Subhedral and euhedral perovskite grains are the most suitable for obtaining reliable ages. Chemical, magnetic and hand-picking techniques can further enhance the accuracy of U-Pb ages obtained from perovskite.

Examples of North American kimberlites dated by the U-Pb perovskite technique include Buffalo Hills, Alberta (86 ± 3 to 88 ± 5 Ma; Carlson et al., 1998), Somerset Island, Northwest Territories (105 Ma; Smith et al., 1989), Fort à la Corne, Saskatchewan (101 ± 2 Ma; Leckie et al., 1997) and Kirkland Lake kimberlites in Ontario and (158 ± 2 Ma; Heaman, 1989).

Mantle zircon is also used to date kimberlite emplacement ages by the U-Pb technique, due to its resistant nature. Zircon is a xenocrystic, rare accessory mineral in kimberlites but is commonly recovered in heavy concentrates with diamonds. Mantle zircons typically have lower U (4.0-9.9 ppm) and radiogenic Pb contents than crustal zircon (Davis et al., 1977; LeCheminant et al., 1998). Mantle zircons form fairly large (2-20 mm), well rounded crystals, are typically not zoned, have well developed cleavage, and are often coated with a layer of baddeleyite (ZrO_2) (Davis et al., 1977; Davis, 1978). When comparing mantle zircons to other minerals transported by kimberlites, mantle zircons are more advantageous because they are less susceptible to chemical and isotopic changes (LeCheminant et al., 1998). Information pertaining to timing and nature of mantle processes prior to kimberlite emplacement, can be preserved in mantle zircons. Great care must be taken while selecting

zircon grains by avoiding such problems as inclusions, fractures and/or turbidity within zircon crystals.

In all other isotopic techniques, the avoidance of xenolithic material is crucial when determining kimberlite emplacement ages. The difference with mantle zircons is that while zircons reside in high temperature regimes (1100°C), Pb is lost through diffusion (Davis et al., 1977). It is not until mantle zircons are entrained within kimberlitic magma and brought to lower temperatures, that lead begins to accumulate through radioactive decay processes. However, mantle zircons that record pre-eruption ages and have not been reset during emplacement, have been documented (Kinny and Meyer, 1994). This occurs when mantle zircons have been brought to cooler temperature regimes, recording a history prior to kimberlite emplacement. An example is the Cretaceous Mbuji Mayi kimberlites, Zaire where a U-Pb age of 628 Ma was obtained through analysis of a mantle zircon inclusion in diamond (Kinny and Meyer, 1994).

New Isotopic Techniques

U-Pb Ilmenite

Many kimberlites do not contain minerals such as phlogopite or perovskite that are traditionally used to determine kimberlite emplacement ages, so it is desirable to find alternative age dating techniques. Preliminary results are presented here for evaluating the feasibility of U-Pb geochronology. Ilmenite is chosen for its chemical and physical resistance and its widespread occurrence in kimberlites world wide. As well, extensive research regarding mineral chemistry and petrology have concluded that ilmenite is genetically linked to crystallization in kimberlitic or proto-kimberlitic magmas.

The Monastery kimberlite is an ideal test case for analysing the potential of ilmenite as a geochronometer, as the emplacement history has been investigated by several isotopic techniques (Figure 1). The best estimate for the emplacement ages of the Monastery kimberlite is 90 ± 1 Ma. Two ilmenite and five perovskite fractions have been analysed from the Monastery kimberlite in South Africa. Ilmenite has the following concentrations: U: 0.09-0.17 ppm; Pb: 0.15-0.2 ppm; and Th: 0.03-0.14 ppm. These concentrations are significantly lower than other U-bearing minerals (e.g. perovskite and mantle zircon). The isotopic variation between ilmenite and perovskite is illustrated in Figure 2A. When the five perovskite analyses are plotted alone (Figure 2B), they produce an isochron age of 88.1 ± 3.0 Ma. An age of 122 ± 48 Ma for ilmenite is obtained as illustrated on the isochron diagram, Figure 2C. The perovskite isochron is plotted along with the ilmenite to demonstrate the varying initial $^{206}\text{Pb}/^{204}\text{Pb}$ ratios. The age of ilmenite is slightly higher but overlaps within error of the reported age for the Monastery kimberlite (90 Ma). An effort in future studies of ilmenite will be to focus on evaluating blank uncertainties to decrease the errors in the $^{206}\text{Pb}/^{204}\text{Pb}$ and $^{238}\text{U}/^{204}\text{Pb}$ ratios. It will also be necessary to evaluate U/Pb variations within and between Monastery ilmenite megacrysts and core/rim relationships to determine whether the ilmenite geochemistry is homogeneous.

Re-Os Ilmenite

The purpose of this study is to apply a relatively new radiogenic isotope technique to gain information about the origin of ilmenite as well as the origin and age of kimberlitic magmas. This study is the first attempt to determine the Os isotopic composition of ilmenite from kimberlites.

Until recently, inaccurate Re-Os age dates with large errors were a direct result of difficulties in measuring the half-life of ^{187}Re and low concentrations of Os in silicate minerals (Faure, 1986; Stein, 1998). Recent advances in the Re-Os system have led to increased measurement sensitivity and analytical accuracy possibly representing a new technique that has the potential to provide alternative method for determining kimberlite emplacement ages. Equilibration of spike and sample has been improved due to methods involving acid dissolution in Carius Tubes (Shirey and Walker, 1995). These advances include establishing a more accurate half-life (Smoliar et al., 1996) and precise measurements of Re and Os attainable by use of negative thermal ion mass spectrometry (NTIMS) (Creasar et al., 1991; Faure, 1986). Re-Os has been widely used to determine the origin of iron meteorites and to date sulfide minerals.

Extensive research has been done on peridotitic xenoliths (Pearson et al., 1995a; Olive et al., 1997) and eclogitic xenoliths (Pearson et al., 1994; Pearson et al., 1995b) from kimberlites. These authors report that peridotite xenoliths have less radiogenic $^{187}\text{Os}/^{188}\text{Os}$ than average chondrites and eclogite xenoliths have highly radiogenic $^{187}\text{Os}/^{188}\text{Os}$ in comparison. Re and Os concentrations range from 0.10 to 0.45 ppb and 0.12 to 2.9, respectively (Pearson et al., 1995a), for peridotitic xenoliths and 0.087 to 1.6 ppb and 0.028 to 0.346 ppb, respectively (Pearson et al., 1995b) for eclogitic xenoliths. A diagram representing the different ranges in Re/Os ratios for sulfides, oxides and whole rock samples is illustrated in Figure 3.

Preliminary results of Os analysis of ilmenite yield concentrations of 0.9 ppt which is low compared to analyses obtained from kimberlitic xenoliths. Further work will be done to determine the potential of ilmenite as a geochronometer or as a tracer of kimberlite processes. As well, investigating Re-Os whole rock compositions along with analyses of sulfide minerals within kimberlites, will be a focus of study.

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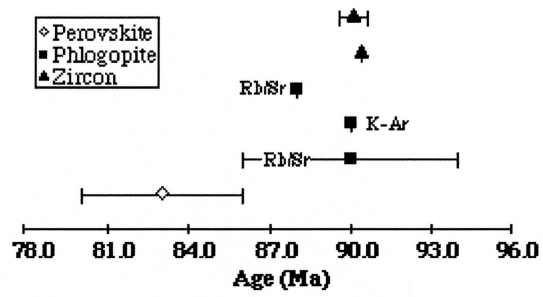


Fig. 1. Compilation of perovskite (U-Pb = 83 ± 3 Ma, Kramers and Smith, 1983), phlogopite (Rb/Sr = 90 ± 4 Ma; 88 Ma, Allsopp and Barrett, 1975; Smith and Barton, 1996; K/Ar = 90 Ma, McIntyre and Dawson, 1976), and zircon (U-Pb = 90.4 Ma; 90.1 ± 0.5 Ma, Davis et al., 1975; Zartman et al., 1998) ages for the Monastery kimberlite, South Africa.

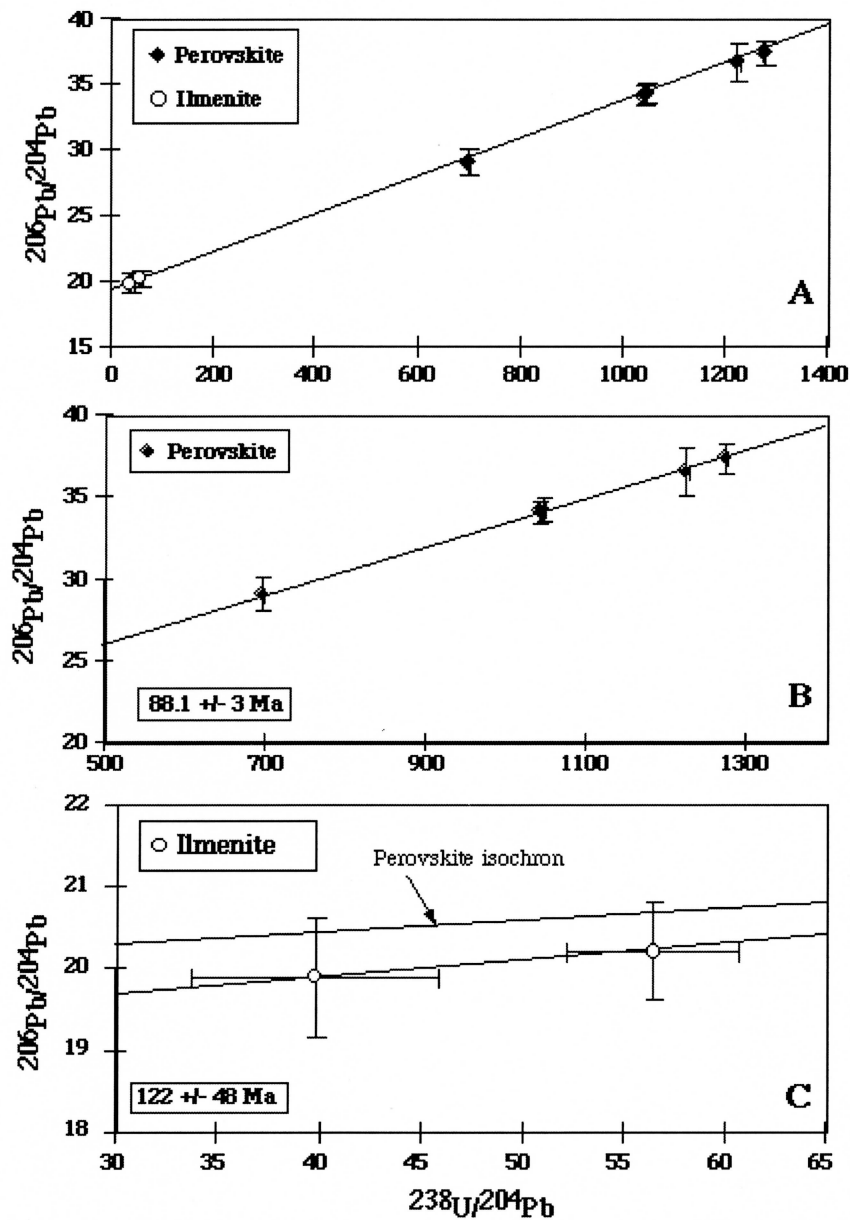


Fig. 2. Isochron plots for perovskite and ilmenite from the Monastery kimberlite, South Africa. A: illustrates the isotopic variation between ilmenite and perovskite, with a reference line between the two minerals, B: illustrates isochron age of $88.1 \pm 3.0 \text{ Ma}$ for perovskite, C: isochron age of $122 \pm 48 \text{ Ma}$ for ilmenite. Perovskite isochron included to demonstrate the difference in initial $^{206}\text{Pb}/^{204}\text{Pb}$ ratios.

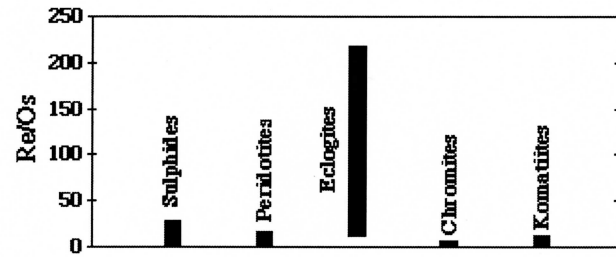


Fig. 3. Compilation of Re/Os isotopic ratios of whole rock,(Pearson et al., 1995a; Pearson et al., 1995b; Shirey and Walker, 1995) and sulphide (Freydie et al., 1997), and oxide minerals (Nagler et al., 1997).