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Preliminary results on the use of pyroxene for ^{10}Be surface exposure dating

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Abstract

We report ^{10}Be data from two sequential dissolution series of pyroxenes separated from Antarctic dolerites with exposure ages around 2 Ma. The goal of these experiments was to test whether pyroxene is suitable for exposure dating with ^{10}Be or whether meteoric ^{10}Be cannot be quantitatively removed from this mineral. Because pyroxene retains ^3He and ^{21}Ne quantitatively, it is an attractive mineral for exposure dating. The measurement of ^{10}Be in the same mineral would complement the noble gas results by revealing possible episodes of prior exposure (when there has been an intervening period of burial). In the first of two dissolution series, the final ^{10}Be concentration was 26% greater than the concentration expected based on the ^3He and ^{21}Ne exposure age of 2.4 Ma. Given production rate uncertainties of perhaps up to 20%, it is possible that meteoric ^{10}Be was completely removed from this sample after two dissolution steps. However, in the second series the final ^{10}Be concentration was almost four times greater than that expected from the noble gas exposure age of 1.8 Ma. This indicates that, using the standard hydrofluoric leaching technique, meteoric ^{10}Be is in some cases not removed completely from pyroxene grains. This is in contrast to quartz, where after only one hydrofluoric leaching step meteoric ^{10}Be is completely removed. An explanation is that meteoric ^{10}Be resides within secondary minerals inside weathered pyroxene grains, and thus cannot be removed even with a rigorous cleaning.

Keywords: ^{10}Be , pyroxene, cosmogenic isotopes, surface exposure dating, Antarctic Sirius Group.

Introduction

Cosmogenic isotopes are produced in measurable quantities in situ in the upper decimeters of rocks exposed to cosmic rays. Measurement of the concentration of these isotopes allows the calculation of the amount of time elapsed since formation or uncovering of the rock surface (for appropriate equations see LAL, 1991). ^{10}Be is produced in the Earth's atmosphere at a rate around three orders of magnitude higher than in rock surfaces (LAL, 1991) and can therefore easily contaminate surface rock samples. To solve this problem HF leaches of the minerals are employed (KOHL and NISHIZUMI, 1992).

Results from CERLING (1990), POREDA and CERLING (1992) (summarized in CERLING and CRAIG, 1994), BRUNO et al. (1997) indicate that ^3He and ^{21}Ne are quantitatively retained in pyroxene. As ^{10}Be ($t_{1/2} = 1.5$ Ma) decays, while ^3He and ^{21}Ne do not, periods of earlier exposure followed by periods of burial would show up as an older apparent ^{21}Ne , respectively ^3He , exposure age in relation to the ^{10}Be age. The use of stable and radionuclides in concert, is often the only way to reveal the "true" exposure history of the rock surface.

We present ^{10}Be measurements from sequential leaching tests of two different pyroxene mineral separates. For comparison, we show leaching results from three quartz samples, two from sand-

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stones and one from granite. The objective of these tests was to determine if pyroxene could be used for ^{10}Be exposure dating studies. Although quartz is a ubiquitous mineral, enlarging the cast of minerals that can be used for ^{10}Be exposure dating would increase the number of geological questions that can be addressed.

No ^{10}Be exposure dating studies have been attempted as yet with pyroxene. NISHIZUMI *et al.* (1990) reported ^{10}Be and ^{26}Al exposure ages and calculated erosion rates from olivine separated from a 500,000 year old lava flow from Hawaii. In their study they removed 11% of the outer surface of the grains by HF etching. Olivine was also used to determine the ^{10}Be exposure age and therefore the eruptive age of a 45,000 year old lava flow (SHEPHERD *et al.*, 1995). SEIDL *et al.* (1997) measured ^{10}Be in olivine as part of a study to determine knickpoint propagation rates (Hawaii). In all of these examples, the HF leaching technique developed for quartz (and used here) was employed. A similar HF leaching technique was also used by BERNER *et al.* (1980) to simulate natural weathering.

The samples investigated are part of a project to determine the age of the Sirius Group sediments and related landscape elements in the Dry Valleys region of Antarctica, using in situ produced cosmogenic ^3He , ^{10}Be , ^{21}Ne and ^{26}Al (IVY-OCHS *et al.*, 1995, 1997; BRUNO *et al.*, 1997). Quartz-bearing clasts unambiguously associated with the Sirius Group are rare; the possibility to analyze pyroxene for ^{10}Be , in addition to ^3He and ^{21}Ne is therefore crucial. Interpretation of the exposure age data and the implications for both the age of the Sirius Group sediments and the stability of the East Antarctic Ice Sheet are discussed in detail in IVY-OCHS *et al.* (1995, 1997) and BRUNO *et al.* (1997). Additional ^{10}Be and ^{26}Al exposure dates from Sirius Group samples are found in BROOK *et al.* (1995).

Samples and analytical procedures

All of the samples discussed are from Table Mountain, Dry Valleys, Antarctica. Rock surface 218 is from a bedrock outcrop of Beacon Sandstone at the top of Table Mountain, while 213a was a loose cobble that had weathered out of the Beacon Sandstone bedrock. Granite boulder 217b is located at the lower margins of the Sirius Group outcrop (locations are shown in IVY-OCHS *et al.*, 1995). Samples 4 and 5a were chipped from the top surfaces of two dolerite boulders found embedded in the Sirius Group sediments (locations shown in BRUNO *et al.*, 1997).

EXTRACTION OF Be FROM QUARTZ AND PYROXENE

From sandstone (samples 213a and 218) and granite (217b), selective chemical dissolution with a weak HF solution was used to obtain a pure quartz fraction (KOHL and NISHIZUMI, 1992). Thereafter, the procedure we used for ^{10}Be sample preparation generally follows that of KOHL and NISHIZUMI (1992). In the case of the pyroxenes, minor modifications were necessary to isolate the Be from the significantly more abundant major element cations (OCHS and IVY-OCHS, 1996).

In order to obtain monomineralic grains, the 0.2–0.5 mm fraction was chosen for the dolerites (based on thin sections). For sample 4, the pyroxene grains were hand-picked under a binocular microscope. For 5a, we used the heavy liquid methylene iodide and a Frantz magnetic separator to separate the pyroxene from the plagioclase. The pyroxenes are augitic; major element compositions (Tab. 1) were measured by ICP-AES (BRUNO, 1995).

The mineral separates were placed in 60 ml closed teflon jars, weighed, then put on a shaker table with approximately 40 ml 18 M Ω water, 5 ml HNO_3 and 5 ml 48% HF. After several hours, the HF solution was decanted into a teflon beaker and the remaining mineral grains were rinsed several times with 18 M Ω water, the rinse water being added to the teflon beaker containing the decanted sample solution. The material left in the teflon jar was put on a shaker table overnight in 18 M Ω water. In the morning, this water was also added to the beaker containing the sample solution, and the sample was rinsed again as before. The teflon jar containing the undissolved portion was dried and weighed to record the amount of pyroxene dissolved in that step. ^9Be carrier was immediately added to the decanted solution. After fuming

Tab. 1 Major element concentrations in weight percent oxide.

Element	4	5a
SiO_2	51.84	50.38
Al_2O_3	4.54	5.99
Fe_2O_3	16.4	17.87
MnO	0.29	0.3
MgO	14.14	13.53
CaO	10.99	10.27
Na_2O	0.48	0.59
K_2O	0.18	0.27
TiO_2	0.63	0.78
P_2O_5	0.13	0.12

Tab. 2 Sample weight that was dissolved in each step and the respective AMS-measured ^{10}Be concentrations.

Sample No.	Mineral	Rock type	Altitude (m)	Step	Weight (grams)	Dissolved (%)	Cumulative (%)	^{10}Be atoms/gram	Error (%)
4	pyroxene	dolerite	2030	HCl	0.0478	2.0	2.0	1.54×10^9	6.0
				HF1	1.9248	82.2	84.2	1.67×10^8	7.8
				HF2	0.3697	15.8	100	5.30×10^7	10.9
5a	pyroxene	dolerite	2030	HF2	0.6905	14.2	25.7	1.48×10^8	5.5
				HF3	0.2691	5.5	31.2	1.29×10^8	7.1
				HF4	0.4376	9.0	40.2	1.50×10^8	7.6
				HF5	0.4301	8.8	49.0	0.85×10^8	7.8
				HF6	2.4826	51.0	100	1.18×10^8	6.1
217b	quartz	granite	1820	HF5	1.3971	8.6	31.3	3.52×10^7	9.2
				HF6	2.9749	18.3	49.6	3.77×10^7	5.6
				HF7	2.9935	18.4	68.0	4.03×10^7	5.6
				HF8	5.1831	32.0	100	3.61×10^7	6.1
213a	quartz	sandstone	2080	HCl	see text	1.0	1.0	8.00×10^8	5.2
				HF2	4.7063	14.2	57.6	5.57×10^7 *	6.5
				HF4	11.1623	33.7	100	5.70×10^7 *	5.9
218	quartz	sandstone	2170	HF2	1.5461	5.7	15.6	3.37×10^7 *	10.3
				HF3	3.1343	11.6	27.2	3.39×10^7 *	7.8

All data are presented. Errors are at the 1σ level, including the statistical (counting) error, the error due to the normalization to the standards and blanks, and the reproducibility of several measurements of the same sample (IVY-OCHS, 1996). HF1 etc. indicates the leaching step from which the sample was made.

Starting weights are as follows: 4, 2.3423 g; 5 a, 4.8627 g; 217 b, 16.2454 g; 213 a, 33.1449 g; 218, 26.9366 g.

* Data from IVY-OCHS et al., 1995.

off SiF_4 , Be and Al were separated on a cation exchange column. Excess Fe was separated from the Be fraction by using a methylisobutylketone extraction (KNAUER, 1994) or an anion exchange column. When Al was apparently still present in the Be fraction, the complete cation column procedure was repeated. Finally, the BeOH was precipitated and then converted to BeO at 850°C . $^{10}\text{Be}/^9\text{Be}$ ratios were measured at the PSI/ETH tandem AMS facility (Tab. 2).

^3He AND ^{21}Ne MEASUREMENT IN PYROXENE

The ^3He and ^{21}Ne measurement procedure and data analysis are discussed in detail in BRUNO (1995) and BRUNO et al. (1997). The noble gas concentrations in table 3 are corrected for atmospheric Ne and nucleogenic He and Ne. Atmospheric ^{21}Ne was determined by assuming that all measured ^{20}Ne is atmospheric. The respective correction is only $\sim 5.0\%$ for sample 4 and $\sim 3.3\%$ for the least air-contaminated split of sample 5a. Nucleogenic ^3He was determined via the measured ^4He concentration, which was assumed to be completely radiogenic (and quantitatively retained). In all cases, nucleogenic contributions were less than 1.1% . The samples should not contain any inherited mantle ^3He since the trapped Ne is atmo-

spheric, i.e. no traces of a MORB (mid-ocean ridge basalt) Ne component are visible. The concentrations of the cosmogenic noble gases are accurate to within 5% . This number includes the reproducibility of the measurements, the uncertainty of the standard gas amounts and the corrections for non-cosmogenic contributions.

PRODUCTION RATES

The production rate of ^{10}Be in pyroxene irradiated at Earth's surface by galactic-cosmic-ray particles is calculated using LCS, the Los Alamos LAHET Code System (PRAEL and LICHTENSTEIN, 1989), which is a general-purpose, Monte Carlo computer code that treats the relevant physical processes of particle production and transport. LCS, its tests, the basics of its physical model, and its adaptation to the production of cosmic-ray-produced (cosmogenic) nuclides in meteorites as well as the Earth's surface and atmosphere are described in detail by MASARIK and REEDY (1994; 1995).

Using calculated fluxes of particles in the Earth's surface and measured and evaluated cross sections, we calculated production rates of cosmogenic nuclides. In pyroxene, ^{10}Be is produced from O, Mg, Al, Si, and Fe. For these elements, absolute

Tab. 3 Measured ^{10}Be , ^3He and ^{21}Ne concentrations and calculated exposure ages.

Sample	4	5a
^{10}Be (10^6 atoms/g)	53 ± 5.8	118 ± 8.3
^{10}Be P.R. (atoms/g yr) (at sea level)	5.2	5.1
^{10}Be P.R. (atoms/g yr) (at sample altitude)	28.8	28.5
Age (Ma) (^{10}Be)	$4.1^{+2.1}_{-1.0}$	*
^3He (10^6 atoms/g)	1522 ± 75 #	1185 ± 45 #
^3He P.R. (atoms/g yr) (at sea level)	115	115
^3He P.R. (atoms/g yr) (at sample altitude)	657	657
^3He Age (Ma)	2.4 ± 0.01	1.9 ± 0.06
^{21}Ne (10^6 atoms/g)	357 ± 18 #	273 ± 11 #
^{21}Ne P.R. (atoms/g yr) (at sea level)	25.4	25.3
^{21}Ne P.R. (atoms/g yr) (at sample altitude)	151	150
^{21}Ne Age (Ma)	2.4 ± 0.03	1.8 ± 0.04

* The measured concentration was higher than the secular equilibrium concentration thus an exposure age could not be calculated.

Data from BRUNO et al. (1997).

For details on calculation of production rates (P.R.) see text.

elemental production rates for ^{10}Be were calculated:

$$P(^{10}\text{Be}) = 10.87[\text{O}] + 0.39[\text{Si}] + 0.45[\text{Al}] + 0.52[\text{Mg}] + 0.16[\text{Fe}] \quad (1)$$

where the weight percent of the target element is contained in brackets (MASARIK and REEDY 1996). These ^{10}Be production rates have uncertainties estimated at $\sim 20\%$. The sea level production rates (Fig. 1) for the pyroxenes we used are both about 5×10^6 ^{10}Be atoms per gram pyroxene per year (Tab. 3). In comparison, empirically determined production rates for quartz are on the order of 6 atoms ^{10}Be per gram SiO_2 per year (NISHIZUMI et al., 1989; 1996; KUBIK et al., 1998).

In order to calculate the elemental contributions from Mg and Al to the production of ^{21}Ne , we used the production ratios of MASARIK and REEDY (1996) combined with the measured production rate for ^{21}Ne from SiO_2 (NIEDERMANN et al., 1993), i.e. $P(^{21}\text{Ne}_{\text{Mg}}):P(^{21}\text{Ne}_{\text{Si}}) = 3.32$ and $P(^{21}\text{Ne}_{\text{Al}}):P(^{21}\text{Ne}_{\text{Si}}) = 1.64$. The resulting contributions are 149.4 for Mg, 73.8 for Al and 45 for Si. The ^3He production rate we used is 115 atoms ^3He per gram pyroxene per year (CERLING and CRAIG, 1994). The production rates were then scaled to the altitudes of our samples (LAL, 1991).

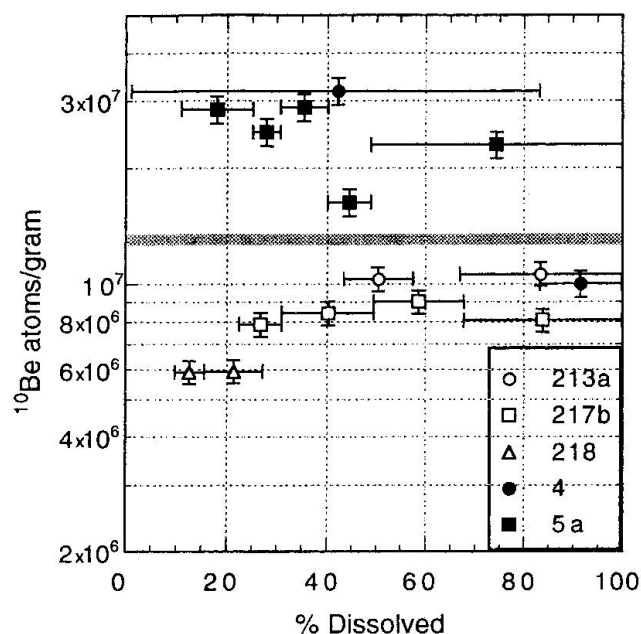


Fig. 1 ^{10}Be concentrations normalized to sea level. Filled symbols are for pyroxene, open symbols are for quartz. The shaded band indicates the sea level secular equilibrium concentration of ^{10}Be for pyroxene. Note that the very high concentrations measured in the initial leach step (samples 213a and 4, data listed in Tab. 2) have not been plotted.

The agreement of ^3He and ^{21}Ne ages from many pyroxene samples (BRUNO et al., 1997) and ^{10}Be , ^{26}Al and ^{21}Ne ages from several quartz samples (IVY-OCHS et al., 1995; BRUNO et al., 1997) reflects the internal consistency of the production rates used here.

Results and discussion

QUARTZ

A ^{10}Be concentration of 8.0×10^8 atoms/gram was obtained for the HCl leach of quartz sample 213a (Tab. 2). This was calculated using the total initial sample weight. This is at least an order of magnitude greater than the ^{10}Be measured in steps HF2 and HF4 of the same sample. This indicates firstly, that meteoric ^{10}Be is indeed adsorbed at the surface of quartz grains and that secondly, the concentration of ^{10}Be had leveled off after HF2. In sandstone 218, we measured ^{26}Al as well, and the age of 1.3 Ma (IVY-OCHS et al., 1995) agrees well with the ^{10}Be age (1.4 Ma). This verifies that the ^{10}Be concentration reached is the in situ component with no remaining meteoric contamination. The ^{10}Be concentrations for steps HF5, HF6, HF7 and HF8 for quartz from granite sample 217b are

the same within the stated errors, and an exposure age of 2.6 Ma was calculated. The agreement with the ^{26}Al results (2.7 Ma; IVY-OCHS *et al.*, 1995) shows that the HF etchings have indeed cleaned away the meteoric ^{10}Be .

Such dissolution series experiments support the idea that the majority of the meteoric ^{10}Be is weakly adsorbed on the surfaces of the quartz grains (BROWN *et al.*, 1991; 1995; KOHL and NISHIZUMI, 1992). Individual quartz grains easily survive many HF washings. The resistance of quartz to weathering, i.e. no secondary mineral formation within the grains, prevents the uptake of meteoric ^{10}Be . Figure 2 shows scanning electron micrographs of unetched (2a) and etched quartz (2b;

following two HF steps). It is difficult to distinguish the leached from the unleached quartz grains.

PYROXENE

We performed two separate dissolution series on pyroxenes (samples 4 and 5a), from two dolerite boulders (Tab. 2). For sample 4, the ^{10}Be concentration in the HCl leach was, similar to quartz sample 213a, two orders of magnitude greater than the concentration measured in the last dissolved fraction, indicating the presence of meteoric ^{10}Be at the onset. From the ^{10}Be concentration in the final step, an exposure age of $4.1^{+2.1}_{-1.0}$ Ma was

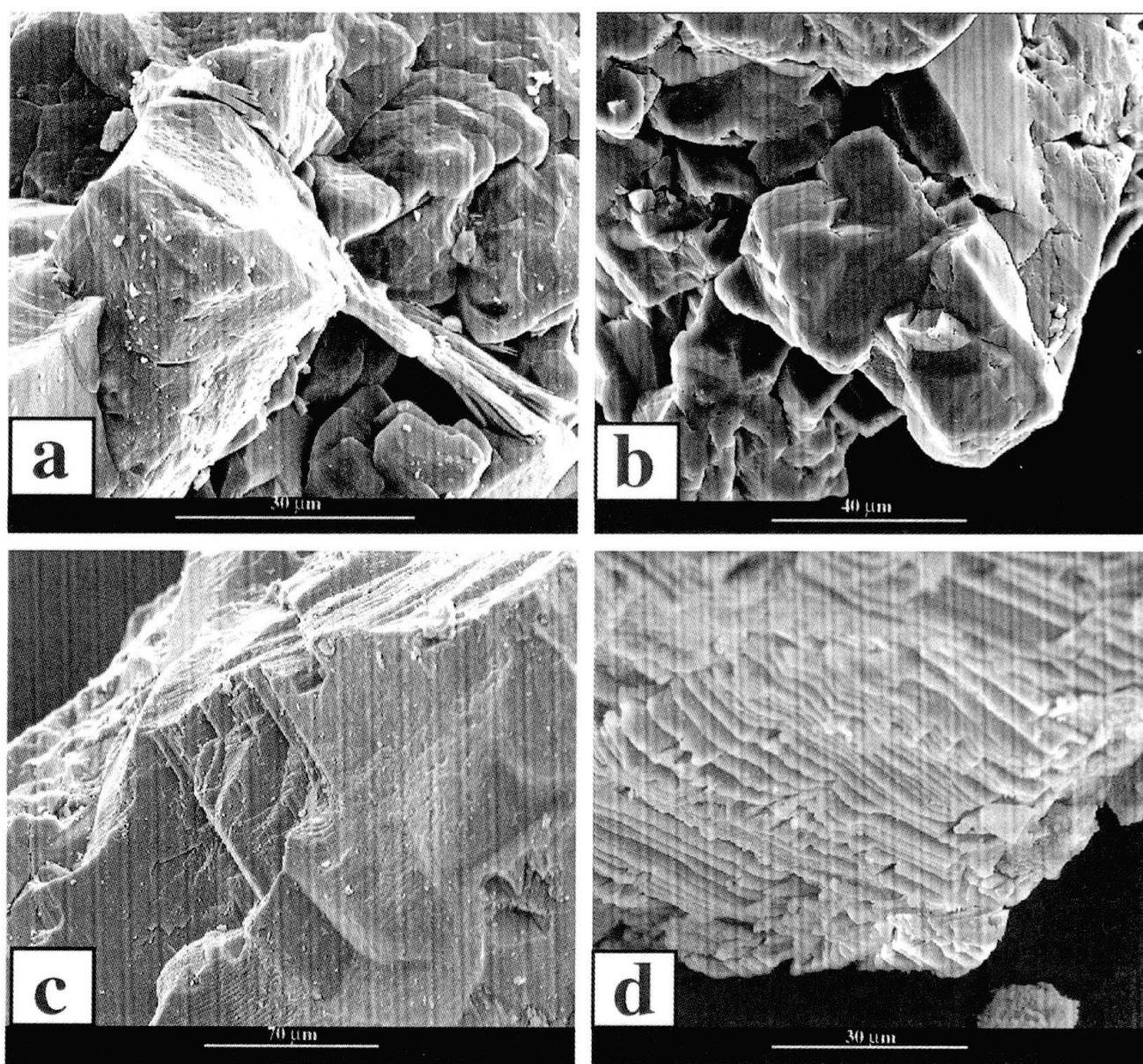


Fig. 2 Scanning electron micrographs of (a) unleached quartz; (b) HF leached quartz; (c) unleached pyroxene; and (d) HF leached pyroxene. Note the well-developed fin or blade structure of the leached pyroxene grain, which can also be seen to a lesser extent in the unleached grain (bottom center in c).

calculated. The stated errors are the 1σ measurement errors with a sample reproducibility error of 5% included quadratically (IVY-OCHS, 1996). The ^{10}Be age is apparently too old in comparison to the ^3He and ^{21}Ne age of 2.4 ± 1.0 Ma, although it may not be unreasonable in light of possible production rate uncertainties of 20%. The measured ^{10}Be concentration differs from the expected (using an exposure age of 2.4 Ma) concentration (4.2×10^7 atoms ^{10}Be /gram pyroxene) by 26%.

For pyroxene sample 5a, we measured the ^{10}Be concentrations in HF2, HF3, HF4, HF5 and HF6 (Tab. 2). Prior to the final step almost half of the original sample weight had already been dissolved away (49%). At this point, the ^{10}Be concentration appears to have leveled off. We did not perform more dissolution steps after HF6 because the remaining pyroxene grains were completely bleached and could easily be mechanically destroyed. The final concentration of ^{10}Be in pyroxene 5a is roughly twice the saturation concentration for this elevation (which is 6.7×10^7 atom ^{10}Be /gram pyroxene) or almost four times the expected concentration based on the 1.8 Ma ^3He and ^{21}Ne exposure age. There appears to be an excess of ^{10}Be in the pyroxenes of sample 5a and probably in the pyroxenes of sample 4 as well.

For radiogenic production of ^{10}Be (SHARMA and MIDDLETON, 1989), the U, Th or Li concentrations (BRUNO, 1995) in our pyroxenes are far too low. Therefore, we suggest that we were unable to remove all of the meteoric ^{10}Be from the pyroxene grains. This may also be an explanation for the scatter in the data, indicating that more or less of the meteoric ^{10}Be was removed inconsistently in the different cleaning steps.

Meteoric ^{10}Be derived from atmospheric production has probably been introduced into the pyroxene grains during mineral weathering. Weathering and dissolution studies of pyroxenes in both the laboratory and in naturally weathered pyroxenes (BERNER et al., 1980; SCHOTT and BERNER, 1985; EGGLETON, 1986; COLIN et al., 1993) indicate that pyroxene does not weather uniformly just on the mineral surface. Conversely, etch pits concentrate along crystallographically weak zones. As the pyroxene dissolves, clay minerals and Fe-(hydr) oxides form along these zones, taking up structural elements from the dissolved pyroxene and from the surrounding aqueous medium (EGGLETON, 1986; CASEY et al., 1993; BANFIELD et al., 1995). Be is preferentially concentrated in secondary clay minerals, substituting for Al and for some divalent cations in the clay structure (WEDEPOHL, 1974; KABATA-PENDIAS and PENDIAS, 1992).

Figure 2 shows scanning electron micrographs

of unetched (2c) and etched (2d; following two HF steps) pyroxene grains. A fin- or blade-like structure is visible in the leached grain. BERNER and SCHOTT (1982) were able to reproduce this structure (as observed as well by them in naturally weathered pyroxenes) using an HF + HCl leaching technique (i.e. similar to the leaching used here). Etch pits were clearly crystallographically controlled, in their study, being concentrated along the basal exsolution lamellae. In thin section, pyroxenes of sample 5a and 4 exhibit patchy alteration and replacement by clays and iron oxides. This is especially noted in the grain cores and as irregular blebs related to the (001) plane (concentrated in the cores as well). Macroscopically, both dolerite boulders 4 and 5a exhibit a thin rust-colored rind clearly indicating oxidation of the primary minerals. We can see no difference in the degree or nature of weathering either macro- or microscopically between our samples 4 and 5a.

Although Antarctica is a polar desert, thin films of water are present and are likely enough to weather the primary minerals over the course of millions of years (UGOLINI and ANDERSON, 1973; UGOLINI, 1986; CAMPBELL and CLARIDGE, 1987). For example, CAMPBELL and CLARIDGE (1987) noted Mg-rich smectites as weathering products of dolerites in Antarctica. Weathering or hydro-cryogenic diagenesis (without liquid water except as films) including the formation of clay minerals after plagioclase, pyroxene and glass in stony meteorites in Antarctica has also been reported (GOODING, 1986).

Concentrations of ^{10}Be too high to have been formed in situ by spallation have previously been noted in studies of secondary mineral deposits: e.g. bauxite ores (MIDDLETON et al., 1994) and hydrothermal vent deposits (VALETTE-SILVER et al., 1987) and hydrothermally altered sediments (BOURLÈS et al., 1992). The bauxite ores were formed by the sub-aerial weathering of silicates whereby meteoric ^{10}Be was incorporated.

Difficulty in removing meteoric ^{10}Be using HF leaching from olivine was encountered by SEIDL et al. (1997) in her multi-isotope study of a 5.2 Ma lava flow. ZREDA (1994) noted contamination by meteoric ^{36}Cl in olivine grains altered to iddingsite. Recently, KLEIN et al. (1997) reported a comparison of the ^{10}Be concentrations in weathered granite (whole rock analyzed) and in quartz separated from the same rock. Concentrations in the former were several times higher than in the latter and were highest in the most weathered rock.

In summary, the problem is twofold. Firstly, weathering of the pyroxenes may result in precipitation of meteoric ^{10}Be -containing clay and/or

(hydr)oxide minerals deep inside the pyroxene grains. Secondly, the pyroxene grains are not robust enough to make it through the numerous HF steps during which the contamination might be removed. In each grain, the weathered (possibly contaminated) zones are difficult to separate from primary unaltered pyroxene.

Conclusions

The two dissolution series we performed indicate that meteoric ^{10}Be may be difficult to separate in even weakly altered mineral grains. If meteoric ^{10}Be were only adsorbed on the surface of the pyroxene grains, it should have been removed by our sequence of seven etching steps. One possible explanation is that crystallographically controlled dissolution during weathering of the pyroxene accompanied by clay formation allows meteoric ^{10}Be to penetrate and to be locked in the interior of the grains. This is in contrast to quartz, where all of the meteoric ^{10}Be is only adsorbed on the surface. It may be that, similar to olivine, ^{10}Be exposure ages can be measured in pyroxenes from young rocks where individual grains are unaltered. Leveling off in a dissolution series does not necessarily prove the absence of meteoric ^{10}Be . The multi-nuclide approach is important for checking for gain or loss of one of the isotopes. Finally, we would like to point out that the open structure of pyroxene could just as easily lead to premature leaching of in situ produced ^{10}Be during the chemical cleaning which would also lead to spurious results.

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