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Terrestrial age measurements using natural thermoluminescence of a drained zone under the fusion crust of Antarctic ordinary chondrites

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Abstract–Miono *et al.* (1990) and Miono and Nakanishi (1994) have proposed that the build-up of natural thermoluminescence (TL) in a drained layer directly below the meteorite fusion crust can be used to determine terrestrial ages of meteorites in the 40 to 200 ka range. We have measured the natural TL of the drained layer of 15 meteorites. The data indicate that this technique could be used to determine terrestrial ages of meteorites with ages <200 ka, after which TL equilibrium is reached. Comparison of TL build-up with terrestrial ages for a suite of Antarctic meteorites suggests that the meteorites have been exposed to temperatures of 0 to 5 °C. The close correspondence between natural TL levels and surface exposure TL growth curves suggest that Allan Hills meteorites with ages <200 ka have spent a significant portion of their terrestrial history exposed on the ice surface, rather than being buried in the ice sheet. The technique is, however, sensitive to thermal history; and, for Antarctic meteorites with terrestrial ages <200 ka, natural TL of the drained zone largely reflects exposure on the ice surface.

INTRODUCTION

Most meteorites found in hot deserts are susceptible to high degrees of weathering that limits their lifetime on Earth to less than ~40 ka (Bland *et al.*, 1998). However, the lower Antarctic weathering rates allow meteorites to survive up to 2 Ma (Nishiizumi *et al.*, 1989; Welten *et al.,* 1997, 1999). Thus, Antarctic meteorites are suitable for studies of possible variations in the meteoroid flux on Earth on the 2 Ma timescale (Schultz *et al.*, 1991; Michlovich *et al.*, 1995; Benoit and Sears, 1996). Other types of studies include weathering rate variations over time, weathering rate as a function of climate, and concentration mechanisms (Jull *et al.*, 1990, 1998; Benoit, 1995; Bland *et al.*, 1998). However, all these studies require a knowledge of terrestrial age.

Terrestrial ages have been determined using the abundance of cosmogenic radionuclides and natural thermoluminescence (TL) of the interior of meteorites (*e.g.*, Nishiizumi *et al.*, 1989; Jull *et al.*, 1990; Benoit *et al.*, 1993; Welten *et al.*, 1999). While a meteorite is in space, it is exposed to a large flux of galactic cosmic rays that result in the production of radionuclides (*e.g.*, ^{26}Al, ^{36}Cl, ^{14}C). The absorbed dose also causes a proportional increase in natural TL levels. When the meteorite falls to Earth, it is shielded from most of the galactic cosmic-ray flux and ambient temperatures are higher so that, like cosmogenic radionuclide activity, the level of stored natural TL decays. If the original levels and decay rates are known, terrestrial ages can be calculated. Typically, original levels are estimated from recent falls. The activity of ^{14}C and ^{36}Cl in recent falls exhibits significant variation due mainly to depth variations in the original samples that cause significant uncertainties in terrestrial ages (Jull *et al.*, 1994, 1989; Freundel *et al.*, 1986). A further constraint on applicability is that ^{36}Cl has a half-life of ~300 ka and thus terrestrial ages <200 ka bear high uncertainties. On the other hand, the 5.73 ka half-life of ^{14}C limits its range to <40 ka when it will have decayed below background levels. Other cosmogenic nuclides have also been used to estimate terrestrial ages, notably ^{26}Al, ^{81}Kr, and ^{41}Ca. The 705 000 year half-life of ^{26}Al is too long to allow accurate terrestrial ages to be calculated for most Antarctic meteorites (Nishiizumi *et al.*, 1989). Krypton-81 has a half-life of only 210 ka, but meteorites contain only one-tenth the required concentrations of Sr, the target element for the production of ^{81}Kr (Freundel *et al.*, 1986). Calcium-41 has a convenient half-life of ~100 ka (Paul *et al.*, 1991; Dickin, 1995; Nishiizumi and Caffee, 1998) but has not been widely applied. There is thus a large gap between 40 and 200 ka in which terrestrial ages are poorly constrained (Fig. 1).

The level of the natural TL in meteorites has also been shown to depend on sample depth, temperature, and orbit (Benoit *et al.*, 1991;

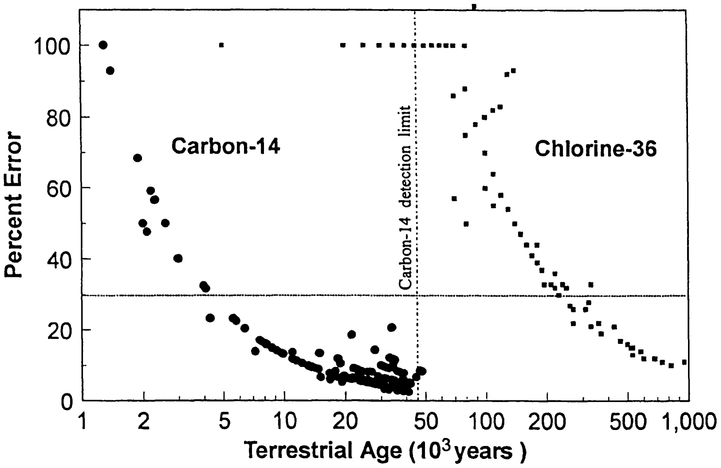

FIG. 1. Percent error in the terrestrial age measurement of Antarctic ordinary chondrites, calculated from ^{14}C and ^{36}Cl abundance (Nishiizumi *et al.*, 1989; Jull *et al.*, 1998) *vs.* terrestrial age. For ages up to ~40 ka, the ^{14}C method has uncertainties of <30%, whereas the ^{36}Cl method has <30% uncertainties for ages <200 ka. The upper boundary for the ^{14}C at ~40 ka is imposed by background levels, whereas the large uncertainties for ^{14}C-dated meteorites with terrestrial ages <5 ka and ^{36}Cl-dated samples with terrestrial ages <200 ka are the result of uncertainties in saturation levels. No meteorites in the current collection have terrestrial ages large enough to approach the upper limit of the ^{36}Cl method. There is a gap between ~40 and ~200 ka in which terrestrial ages cannot currently be determined with accuracy. Samples for which only upper or lower limits are available are omitted.

Benoit and Chen, 1996). The rate of natural TL decay is dependent on storage temperature (Benoit, 1995). Although terrestrial age estimates for bulk natural TL are consistent with ^{14}C estimates for the Prairie States and North Africa, they are consistently lower than terrestrial age estimates based on ^{36}Cl for Antarctic finds (Benoit *et al.*, 1993). This is because meteorites buried in the ice are subject to lower temperatures and thus decay at lower rates than those on the surface (Benoit *et al.*, 1993). Therefore, bulk natural TL measurements only provide an indication of the duration of exposure on the surface, and not the complete terrestrial age.

Another natural TL technique that has been proposed for terrestrial age estimation involves the fusion crust or material just beneath the crust (Miono *et al.*, 1990; Miono and Nakanishi, 1994). Natural TL levels in or near the crust will have been reset to zero during atmospheric passage but will grow during exposure to internal and cosmic radiation while on Earth. A similar technique is in widespread use for pottery dating (where firing in a kiln drains the natural TL; Fleming, 1979) and, in the case of meteorites, might be applicable to the 40 to 200 ka range. Miono *et al.* (1990) and Miono and Nakanishi (1994) demonstrated that the natural TL levels of samples taken just under the fusion crust tended to increase as a function of terrestrial age. The purpose of this study is to define the strengths and limitations of the method for terrestrial age determination. We describe a detailed experimental procedure and apply the method to 15 meteorites with particular emphasis on the uncertainties related to the measurements and calculations.

THEORY

During atmospheric passage, temperatures are high enough to completely drain the natural TL in the first millimeter or so of material under the fusion crust of a chondritic meteorite (Sears, 1975). This surface layer may be referred to as the "drained zone." In order to use the drained zone as a terrestrial age indicator, it is necessary to have a theoretical framework for predicting natural TL build-up. Several attempts have been made to determine the build-up of natural TL in mineral systems under ionizing radiation, but it is difficult because each mineral appears to be affected by ionizing radiation differently (McKeever *et al.*, 1985). Cosmic-ray dose rates in Antarctica are estimated to be between 0.04 and 0.06 rad/year, whereas for ordinary chondrites internal radionuclides contribute ~0.01 rad/year (Kerridge and Matthews, 1988; Prescott *et al.*, 1994). However, the dose rate experienced by the meteorite will be significantly less because of TL production inefficiencies. For the purposes of the present study, an estimate of the dose rate can be determined by comparing TL levels of meteorites with theoretical build-up curves for meteorites with terrestrial ages between 30 and 45 ka.

Thermoluminescence is produced by the release of electrons from "traps," defects in the crystal lattice of the phosphor responsible for the TL. The process follows second-order kinetics:

$$\frac{dn}{dt} = -sn^2 \exp\left(\frac{-E}{kT}\right) \qquad (1)$$

where n is the number of excited electrons, T is the temperature during irradiation, s is the frequency (Arrhenius) factor, E is the trap depth, and k is Boltzmann's constant (McKeever, 1980). If the apparent dose rate (or rate of excitation of electrons) is r, then

$$\frac{dn}{dt} = r - sn^2 \exp\left(\frac{-E}{kT}\right) \qquad (2)$$

The values of s and E can be determined using the methods of McKeever (1980) and Randall and Wilkins (1945). Figure 2 shows the calculated build-up and decay of natural TL at various temperatures and dose rates appropriate for Antarctic environments. A meteorite on the surface will be exposed to cosmic rays as well as internal radiation giving it an effective dose rate of 5 mrad/year. While the meteorite is buried in the ice, the major source of radiation dose will be internal radiation, or an effective dose rate of ~1 mrad/year (Prescott *et al.*, 1994). Although some variation in dose rate is expected for different classes of meteorite and different terrestrial environments, it has less influence on TL than temperature (Eq. 2). Most of the curves level out when the TL of the drained zone begins to reach a state of equilibrium, the equilibrium level depending on temperature and dose rate.

In some cases, feldspar (the mineral that produces TL in meteorites) has been found to exhibit "anomalous fading" (Wintle, 1973), notably in howardite–eucrite–diogenite (HED) meteorites and lunar samples (Hasan *et al.*, 1986; Sears *et al.*, 1991; Benoit *et al.*, 1996). Equilibrated ordinary chondrites do not appear to display anomalous fading (Sears *et al.*, 1991), but because fusion crust samples have experienced major alteration, it is necessary to ensure that their TL properties have not been altered. Fortunately, anomalous fading can easily be detected by laboratory experiments (Wintle, 1973).

METHODS

We acquired samples of 19 Antarctic equilibrated ordinary chondrites from the Johnson Space Center and the Smithsonian Institution, each sample containing surface with fusion crust (Table 1). The drained zones of Allan Hills (ALH) A76008, ALHA77004, ALHA77297, and ALHA81015 were removed in ~0.5 mm cuts using a diamond blade saw. Portions of the drained zone within <1.2 mm of the meteorite fusion crust surface of the remaining samples were removed with a chisel. The chipped pieces were then ground to ~100 mesh and the magnetic portions removed

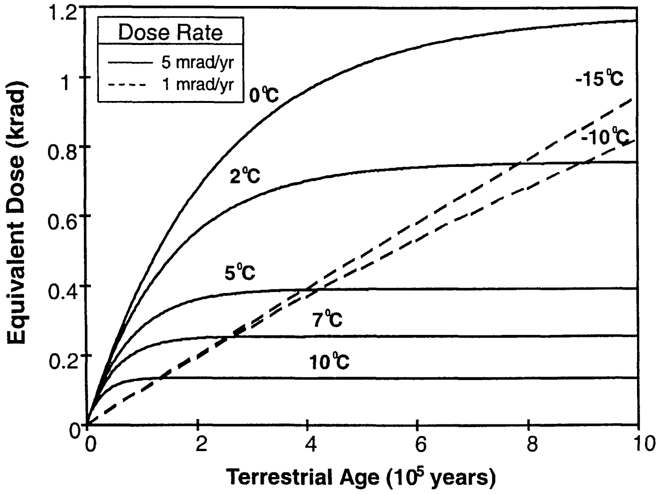

FIG. 2. Calculated TL build-up curves for the drained zone of equilibrated ordinary chondrites. Solid lines are for various temperatures and a dose rate of 5 mrad/year, approximating exposure on the ice surface, the radiation sources including both cosmic rays and internal radionuclide decay. The dashed lines are for a dose rate of 1 mrad/year, approximating burial in the ice, where most of the radiation dose is from internal radionuclide decay. The temperatures chosen for the solid and dashed line series approximate summer meteorite surface exposure temperatures (Schultz, 1990) and subsurface ice temperatures (Mayewski *et al.*, 1990), respectively.

TABLE 1. Equivalent doses measured and their corresponding terrestrial ages.

Meteorite name	Equivalent dose (krad)	AMS terrestrial age (ka) *	Drained zone terrestrial age (ka)†
ALHA76008,38	0.16 ± 0.04	100 ± 70	38 ± 13
ALHA77002,29	0.50 ± 0.11	820 ± 80	>200
ALHA77004,9	0.30 ± 0.02	170 ± 70	99 ± 30
ALHA77231,51	0.52 ± 0.02	600 ± 70	>200
ALHA77272,10	0.57 ± 0.02	580 ± 80	>200
ALHA77294,75	0.05 ± 0.01	10 ± 2	10 ± 2
ALHA77297,4	0.14 ± 0.03	80 ± 40	33 ± 10
ALHA78045,12	0.43 ± 0.08	740 ± 80	>200
ALHA78076,5	0.23 ± 0.01	130 ± 70	62 ± 11
ALHA78114,13	0.33 ± 0.04	460 ± 80	>200
ALHA80132,31	0.30 ± 0.04	110 ± 60	109 ± 46
ALHA81015,72	0.33 ± 0.00	180 ± 70	114 ± 31
ALHA83070,21	0.04 ± 0.01	2 ± 1.3	8 ± 3
ALHA84005,23	0.16 ± 0.03	34 ± 2	39 ± 11
ALHA85033,27	0.10 ± 0.01	29 ± 2	23 ± 3

*Terrestrial ages estimated using accelerated mass spectrometry (AMS) measurements of the abundances of cosmogenic radionuclides, ^{14}C and ^{36}Cl (Nishiizumi *et al.*, 1989; Michlovich *et al.*, 1995; Jull *et al.*, 1998).
†Terrestrial ages estimated using Eq. (3) and the natural TL of the drained zone.

with a magnet. Three 4 mg portions of each sample were heated in the TL apparatus at a rate of 7.5 °C/s in a N_2 atmosphere. Corning filters 4-69 and 7-59 were placed between the tube and the sample to restrict the measurement of wavelengths between 350 and 500 nm. The natural TL, the light emitted as a function of temperature, was measured using an EMI 9635B photomultiplier tube. The samples were then given a known dose of radiation from a ^{90}Sr/^{90}Y β-source and the induced TL measured in the same manner as the natural TL. The TL data for the three aliquots were averaged and equivalent doses calculated by dividing the natural TL at 250 °C by the induced TL at 250 °C and then multiplying by the artificial dose given. This method is similar to that used in pottery dating (Fleming, 1979). Reported uncertainties for the data reflect only the precision of the triplicate measurements. The four meteorites cut with the saw had reproducibly low natural TL as a result of the heat generated during cutting. Their equivalent dose values were corrected using an empirical value obtained from meteorites that were subjected to both methods of sample preparation (see Akridge, 1998, for details). Other possible sources of error are discussed below.

Four samples of material from the drained zones of ALHA76008 and ALHA81015 were irradiated and stored in a dark room. Figure 3 shows the fraction of TL remaining after storage times of up to 1000 h. There is no detectable loss of TL, indicating that anomalous fading is not significant.

Four of the samples we received were not suitable for fusion crust dating. The natural TL exhibited by ALHA81111 and ALHA77261 was below the detection limits, perhaps due to preterrestrial shock. These meteorites may have very young terrestrial ages but this is unlikely in view of their ^{36}Cl ages. Samples ALHA78131 and ALHA78102 were extensively weathered and the fusion crust and drained zones were completely destroyed.

RESULTS

The equivalent doses we determined are listed in Table 1, with terrestrial ages estimated from ^{14}C and ^{36}Cl (Nishiizumi *et al.*, 1989; Michlovich *et al.*, 1995; Jull *et al.*, 1998). We calculated terrestrial ages from equivalent doses assuming storage temperatures of 0 and 5 °C using a simplified version of Eq. (2):

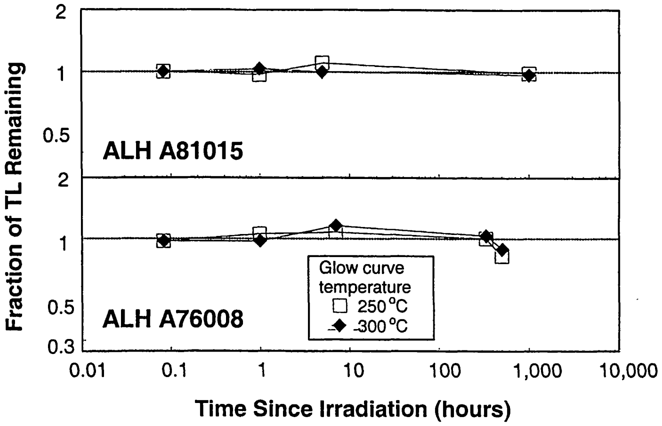

FIG. 3. Fraction of induced TL remaining as a function of time for samples of the drained zone of ALHA76008 and ALHA81015 after storage in the dark at room temperature. The TL at 250 and 300 °C in the glow curve are shown. No measurable decay of TL was observed. In contrast, some feldspar samples exhibit "anomalous" fading by up to 50% on the order of a few days (Wintle, 1973).

$$\frac{n_2 - n_1}{\Delta t} = r - s n_1^2 \exp\left(\frac{-E}{kT}\right) \tag{3}$$

The approximation is valid if the time increment is very small. We use the difference between the data at these two temperatures as an estimate of uncertainties on ages. The results are given in Table 1.

Figure 4 shows natural TL profiles for ALHA76008 and ALHA81015. As expected, natural TL levels are significantly lower for the first 1.2 mm from the surface than in the interior. Similar results were obtained for ALHA77004 and ALHA77297 and agree with the earlier observations of Sears (1975) and Miono *et al.* (1990).

The TL of four of our samples (ALHA76008, ALHA77294, ALHA77297, and ALHA78076) were also measured by Miono and

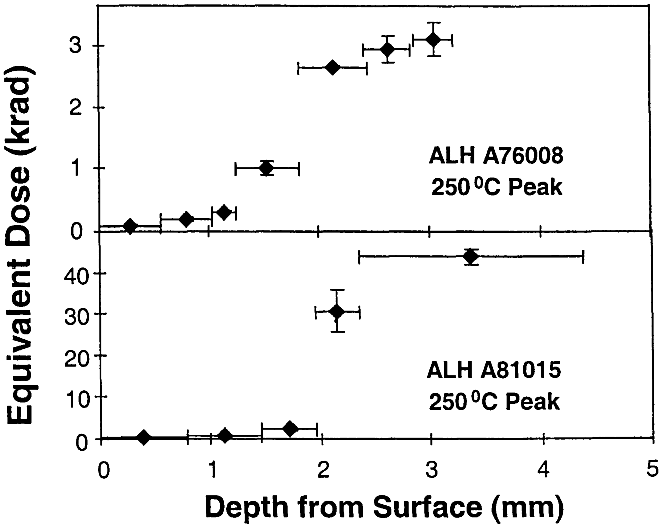

FIG. 4. Equivalent dose at 250 °C in the glow curve as a function of depth from the fusion crust surface for ALHA76008 and ALHA81015. There is a significant difference in TL levels above and below 1.2 mm depth. The TL at >1.2 mm predominately reflects exposure to radiation in space, whereas TL at <1.2 mm reflects terrestrial radiation exposure.

Nakanishi (1994). However, it is not possible to directly compare data between studies because Miono *et al.* (1990) and Miono and Nakanishi (1994) did not report equivalent dose for their samples.

DISCUSSION

Depth Profiles

It is apparent from the depth profiles in Fig. 4 that even modest amounts of "contamination" of samples of the drained zone with material from the interior would greatly affect our results. Figure 5 shows glow curves for two of our samples. "Contamination" is apparent in the curve for ALHA78131 in the large signal in the high-temperature portion (>350 °C) of the glow curve. On the other hand, this is not observed for the sample of ALHA76008. The most likely instances of "contaminated" samples are when the drained zone was lost through weathering or when the drained zone was extremely thin (*e.g.*, the surfaces that were frontal during atmospheric passage; Sears, 1975).

Terrestrial Age Estimation

Comparison of our natural TL data and terrestrial age estimates suggest storage temperatures ranging between 0 and 5 °C (Fig. 6). Schultz (1990) found similar temperatures produced by solar heating in the interior of a meteorite analog on the ice surface in Antarctica. The temperature estimate is an effective storage temperature averaged over the terrestrial history of the meteorites. Thus, for example, modeling of TL variation suggests that a meteorite exposed to 10 °C temperatures for only 25% of its history and 0 °C for the remaining 75% will have an apparent storage temperature of 5 °C, reflecting greater rate of TL decay at the higher temperature, balanced by the lower decay rate at the lower temperature (Akridge, 1998). However, the choice of temperature is obviously an important constraint on the age estimates and uncertainties (Fig. 2).

In Fig. 7, we compare inferred TL terrestrial ages against terrestrial ages determined from cosmogenic radionuclide abundances for meteorites with terrestrial ages <200 ka. As an estimate of uncertainty on the TL ages, we use the full range calculated for storage temperatures of 0 and 5 °C, this source of uncertainty giving a wider age range than analytical uncertainties in all cases except for very low terrestrial ages (<40 ka). The uncertainties on the TL ages are generally smaller than those associated with ^{36}Cl age estimates and become larger with increasing terrestrial age due to the onset of TL equilibrium (Fig. 6). This is shown graphically in Fig. 8, where we compare uncertainties associated with the TL of the drained zone and the abundance of ^{14}C and ^{36}Cl in meteorite finds. For samples with ages >200 ka, however, uncertainties associated with TL of the drained zone become very large because of the TL reaching equilibrium (Fig. 6). Our detection limit prevents TL terrestrial age estimation for samples with terrestrial ages <30 ka.

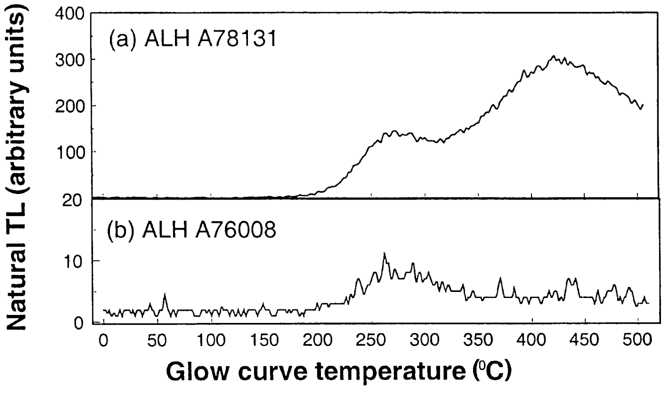

FIG. 5. Glow curves of samples of the drained zone from two meteorites. (a) The glow curve of ALHA78131 exhibits two peaks, including a peak with significant intensity in the high temperature (>350 °C) region of the glow curve. Under typical terrestrial conditions, >10^8 years would be required to build high-temperature TL to these levels, greater than the terrestrial ages of most Antarctic meteorites. The high-intensity TL in this region of the glow curve probably reflects the inclusion of material not drained of TL during atmospheric passage, and thus this sample is unsuitable for TL dating. (b) Sample ALHA79008 does not exhibit significant TL in the high-temperature portion of the glow curve. It is likely that the sample was completely drained of TL during atmospheric passage and thus is suitable for TL dating.

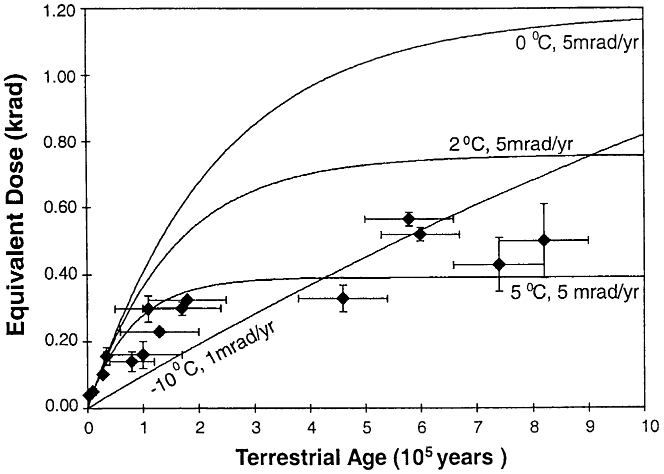

FIG. 6. Natural TL of our samples compared with terrestrial ages estimated from ^{14}C and ^{36}Cl activity. Some of the TL build-up curves from Fig. 2 have been superimposed on the data. The data most closely correspond to the 5 °C surface exposure curve. Cosmogenic radionuclide activity data are from Nishiizumi *et al.* (1989), Michlovich *et al.* (1995), and Jull *et al.* (1998).

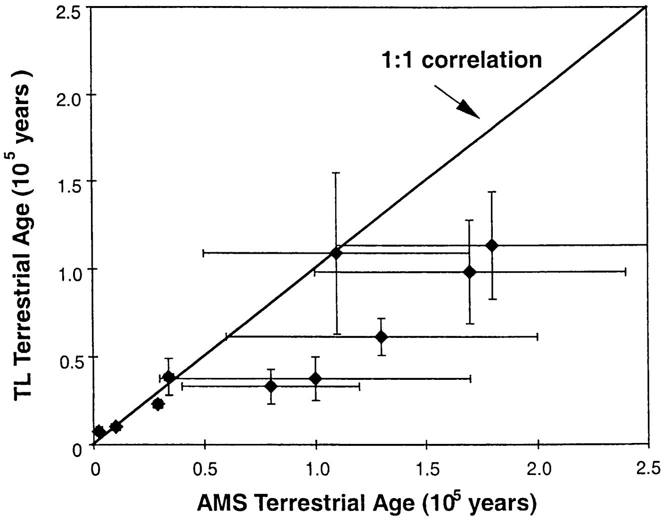

FIG. 7. Terrestrial ages calculated from natural TL of the drained zone for Antarctic ordinary chondrites compared to their terrestrial ages estimated from ^{14}C and ^{36}Cl (Nishiizumi *et al.*, 1989; Michlovich *et al.*, 1995; Jull *et al.*, 1998). Meteorites with terrestrial ages >200 ka are not shown (see Fig. 6). The TL terrestrial age uncertainties include the effect of analytical and storage temperature uncertainties. The uncertainties on natural TL increase with increasing age because of the onset of TL equilibrium (Fig. 6).

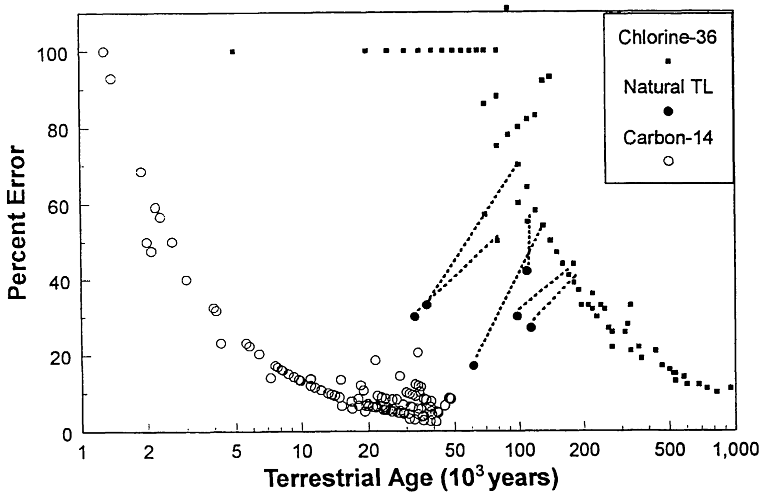

FIG. 8. Figure 1 revised to show ages calculated from the natural TL of the drained zone (filled circles). Tie lines connect natural TL ages to their corresponding ^{36}Cl ages. Only samples with terrestrial ages between 30 and 200 ka are shown.

The data for our samples more closely follow the 5 °C, 5 mrad/year build-up curve than the –10 °C, 1 mrad/year build-up curve (Fig. 6). These conditions were chosen to model the two environments available to Antarctic meteorites, exposure on the ice surface and burial within the ice, respectively. Whereas 5 °C seems warm for Antarctic conditions, this temperature only applies to the outermost black surface exposed to the sun. The temperature estimate of –10 °C was chosen on the basis of measurements on ice cores (Mayewski *et al*., 1990), but TL build-up curves for lower temperatures are essentially the same. On the basis of these data, we suggest our samples spent a significant portion of their terrestrial histories on the ice surface. All of our samples came from a single region, namely the icefields near the Allan Hills (Table 1). It is possible that long surface exposure is typical of this region and that these icefields are fairly stable entities over the time span represented by the Antarctic meteorite collection (Huss, 1990). Benoit (1995) suggested on the basis of a TL profile that a large Allan Hills meteorite had lain exposed on the ice surface in a single orientation for at least 150 000 years.

Another potential complication is the possible effect of multiple episodes of burial and surface exposure. For meteorites with large terrestrial ages, TL cannot distinguish between simple surface exposure and multiple episodes of exposure and burial in the ice. In addition, sample heterogeneity could influence the results, especially for large meteorites with significant thermal profiles due to solar heating and contact with the ice (*e.g*., Benoit, 1995). However, during the period of rapid TL build-up (<200 ka), deviations from the build-up curve for surface exposure should be readily apparent. Most of our data plot close to the curve calculated for surface exposure (Fig. 6). We therefore infer that these meteorites have spent a significant portion of their terrestrial history on the ice surface. Notably, some meteorites do exhibit shorter TL ages than implied by their terrestrial ages (Fig. 7), although uncertainties in the terrestrial ages prevent us from definitely attributing this to burial in the ice.

CONCLUSIONS

A layer ~1.2 mm thick under the fusion crust of meteorites is completely drained of its natural TL during atmospheric passage. The natural TL exhibited by samples from this layer thus reflects build-up on Earth. In the case of Antarctic meteorites, meteorites exposed on the ice surface will experience significantly different thermal and radiation conditions than meteorites buried in the ice, and these differences affect natural TL build-up rates. Natural TL levels of the drained zone can thus be used to estimate surface exposure, if independent terrestrial age estimates are available. Because of detection limits and TL equilibrium, the TL drained zone method is limited in application to meteorites with surface exposure ages between 30 to 200 ka. For meteorites with long ice burial histories, terrestrial ages of up to 1 Ma could be estimated.

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