



PII S0016-7037(02)00839-6

An investigation of artificial biasing in detrital zircon U-Pb geochronology due to magnetic separation in sample preparation

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(Received December 14, 2001; accepted in revised form January 23, 2002)

Abstract—The application of detrital zircon geochronology for provenance analysis is complicated by the presence of biases induced by natural processes and sample preparation. The biasing of age distributions as a result of magnetic susceptibility is illustrated using sensitive high resolution ion microprobe dating of detrital zircon from a metaquartzite sample partitioned using a Frantz magnetic barrier separator. The relationship of paramagnetism with U content, α -dose, and discordance is demonstrated, but no relationship between grain size and discordance or age is found. The data also demonstrate that previous limits of zircon survival in sedimentary processes based on U content alone are too simplistic. Two age modes at ~ 3150 and ~ 2960 Ma are present in all the paramagnetic fractions; there is a bias toward the ~ 3150 Ma mode being more prominent in the least-paramagnetic fractions. While the ~ 2960 Ma is present in the least-paramagnetic fraction, it is argued that such fortuitous representation cannot be assumed before analysis. Such “there or not” provenance interpretations are considered simplistic, and at the very least there is no harm in broadening the range of paramagnetic fractions sampled for analysis. The results indicate a compromise between broad representation and analytical efficiency (avoiding discordant and thus unreliable results) can be made with a Frantz setting of ~ 1.8 A and 10° side-slope. Copyright © 2002 Elsevier Science Ltd

1. INTRODUCTION

Detrital zircon geochronology for provenance analysis has become an increasingly used method for tectonic and crustal evolution issues (e.g., Dodson et al., 1988; Davis et al., 1994; Rainbird et al., 1998). Like all analytical methodologies, detrital zircon geochronology is subject to a variety of assumptions and limitations. In particular, detrital zircon grains in any sedimentary unit sampled for geochronology have been subject to various natural processes before and during deposition that bias interpretations. These include underrepresentation of source rocks with low zircon content, preferential destruction of metamict grains during transportation, and grain-size sorting during deposition. Artificial biases are induced during multiple levels of sample selection (from collection at the outcrop to crushing and hand picking and even ion probe spot location). These biases preclude the ideal situation of being able to exactly match the quantity of analysed grains with the volume of contributing source rock in a pure quantitative interpretation.

One of the fundamental procedures in zircon U-Pb geochronology is the use of magnetic separation to obtain sample fractions that yield more analyses that are concordant. Concordant detrital zircon ages are clearly more powerful than discordant ages. The positive correlation between Pb-loss/discordance, U content, and magnetic susceptibility was noted by Silver (1963a). Refinements to the magnetic separation techniques and the introduction of air abrasion to minimize discordance were developed by Krogh (1982). Heaman and Parrish (1991) later speculated that the routine selection of grains from

the least-magnetic fraction introduced the possibility of an artificial bias in the resulting U-Pb age distribution. Such a bias may be negligible for zircons of generally unimodal origin such as those in most igneous and some metamorphic units. However, it is a major concern in sedimentary units where zircons of diverse origin are typical, and reasonable representation is sought.

The aims of this paper are to (1) review the known causes of zircon magnetic susceptibility and the functioning of the Frantz magnetic barrier separator, (2) examine the potential for biasing in a typical provenance analysis project based on detrital zircon geochronology, and (3) provide some guidelines for researchers conducting similar provenance projects to monitor and/or avoid potential artificial paramagnetic biasing.

2. MAGNETIC SUSCEPTIBILITY OF ZIRCON

The use of the Frantz magnetic barrier separator to fractionate zircon based on paramagnetism or magnetic susceptibility has become so common in geochronology that it barely receives a mention in the modern literature. This section will discuss the features of magnetic susceptibility in zircon relevant to this investigation. A detailed review of the functioning of the Frantz magnetic barrier separator based on these principles is given in the Appendix.

Magnetic susceptibility is the measure of the ease with which a material is magnetized by the application of a magnetic field. This value can be considered in terms of volume magnetic susceptibility (χ_v) or mass magnetic susceptibility (χ_g), which is derived by dividing volume magnetic susceptibility by the density of the material being examined ($4.6\text{--}4.7\text{ g/cm}^3$ for zircon; Deer et al., 1992). The density of zircon changes with metamictization up to a saturation level at $\sim 3.9\text{ g/cm}^3$ or a density decrease of $\sim 17\%$ (Murakami et al., 1991). However, the density change itself is not important in the discussion here,

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because the Frantz separator fractionates by relative magnetic susceptibility only. Although magnetic susceptibility is in itself dimensionless, in many references, mass magnetic susceptibility is reported in values based on CGS units such as gauss, centimetre, and gram, and this is often indicated by the nomenclature, e.m.u./g. CGS mass magnetic susceptibility can be converted to a value based on SI units by multiplying by $4\pi/1000$ (Payne, 1981). SI-based values are distinguished by m^3/kg . Although by historical necessity the discussion here will use CGS-based values, both values will be included.

There are three magnetic states of matter relevant for this discussion. Diamagnetic materials have negative magnetic susceptibilities and are weakly repelled by a magnetic field. Paramagnetic materials have positive magnetic susceptibilities and are moderately attracted by a magnetic field. Ferromagnetic materials, such as iron and many iron compounds, have high positive magnetic susceptibilities and are strongly attracted by a magnetic field.

The typical value of mass magnetic susceptibility for zircon ranges from diamagnetic to paramagnetic. Lewis and Senftle (1966) calculated from a theoretical basis that pure zirconium orthosilicate ($ZrSiO_4$) is diamagnetic at -0.39×10^{-6} e.m.u./g (-4.9×10^{-9} m^3/kg). They also measured diamagnetic and paramagnetic values for their samples ranging from -0.25×10^{-6} to $+6.6 \times 10^{-6}$ e.m.u./g (-3.1×10^{-9} to 8.3×10^{-8} m^3/kg) with most specimens having susceptibilities $<1 \times 10^{-6}$ e.m.u./g (1.3×10^{-8} m^3/kg). Other reported measurements of mass magnetic susceptibility in zircon range from 0.163×10^{-6} to 0.258×10^{-6} e.m.u./g (2.0×10^{-9} to 3.2×10^{-9} m^3/kg) (Powell and Ballard, 1968). S. G. Frantz in the Appendix of Gaudin and Spedden (1943) reported "negative mass susceptibility in the order of -0.3×10^{-6} " (-3.8×10^{-9} m^3/kg). The possibility of anisotropic magnetic susceptibility in zircon was raised by measurements of Voigt and Kinoshita (1907): -0.170×10^{-6} e.m.u./g (-2.14×10^{-9} m^3/kg) || a-axis, and 0.732×10^{-6} e.m.u./g (9.20×10^{-9} m^3/kg) || c-axis. These anisotropic values have since been repeated in Landolt-Börnstein tables, although the original analysts appear to have used an impure zircon sample and stated that they considered the values to have low reliability.

The actual cause of magnetic susceptibility in zircon has only been briefly examined. Using a series of leaching and heating/oxidizing experiments Lewis and Senftle (1966) demonstrated the cause to be the combination of a readily soluble but unidentified mineral on the zircon surface and iron impurities in the crystal lattice. Lewis and Senftle (1966) also discussed the metamictization of the zircon-enhancing diffusion of iron from external sources, such as groundwater, into the crystal lattice.

3. SAMPLE DESCRIPTION

The sample used in this study is from the Slave craton, Northwest Territories, Canada, and forms part of a Mesoproterozoic metasedimentary succession that has been extensively studied in the region (Bleeker et al., 1999; Isachsen and Bowring, 1997). The sample had been previously analysed as part of a regional provenance project (Sircombe et al., 2001) that tested and established a regional correlation across lithostratigraphically similar, but widely dispersed, metasedimentary

units. The sample was selected because of our familiarity with the location and the provenance issues involved. The previously analysed age distribution contains a broad range of ages, and in particular, two distinct modes that were considered a suitable target to test for provenance biasing. Previous investigations in the area have also provided a geochronologic constraint from overlying volcanics. Finally, the sample had also previously yielded a couple of grains older than 3800 Ma. Because the search for such ancient grains is of particular interest in many studies of Precambrian sedimentary rocks, there was also a desire to understand how the presence of these ancient grains related to magnetic susceptibility.

The sample is a white-to-green fuchsitic metaquartzite sampled on the south shore of Dwyer Lake, ~30 km north of Yellowknife. The quartzite succession overlies ~2900 Ma or older basement granitoids (Isachsen and Bowring, 1997). Remnant decimetre-scale cross-beds are preserved at the sample locality, commonly outlined by fuchsitic (\pm relict detrital chromite) laminae. The quartzite is overlain by a thin felsic volcanic-to-volcaniclastic unit dated at $2853 \pm 2/-1$ Ma (Ketchum and Bleeker, 2000), which in turn is overlain by BIF and the main volcanic succession of the Yellowknife greenstone belt.

The age distribution in the sample spans one billion years and includes two principal age modes. The first is a ~2960 Ma component and is correlated with a widespread plutonic event in the Central Slave Basement Complex involving the intrusion of tonalites, i.e., Event VII in the nomenclature of Bleeker and Davis (1999). The second component is ~3150 Ma and is correlated with plutonic-metamorphic event in the same basement complex involving the intrusion of tonalites to granites and their deformation (Event V: Bleeker and Davis, 1999). A range of older ages with no distinct mode occur up to 3900 Ma.

4. ANALYTICAL METHODOLOGY

Detrital zircon grains were extracted using standard crushing, milling, Wilfley table, and heavy liquid techniques. Five fractions were separated using the Frantz magnetic barrier separator at the settings shown in Table 1 after removing ferromagnetic minerals with a hand-magnet. The range of mass magnetic susceptibilities represented by each fraction, from A, the least paramagnetic, to E, the most paramagnetic, is illustrated in Figure 1. Ideally, the absolute mass magnetic susceptibility should be used when describing paramagnetic fractions. The theoretical relationship between these values and the settings on a Frantz is discussed in the Appendix with an example of direct calibration. However, it must be acknowledged that this relationship probably varies to some extent between separators, and the significance of this variation remains to be investigated. For the purposes of this study, it is assumed that the performance of the Frantz used matches the theoretical calculations.

The handpicking process in detrital zircon geochronology attempts to ensure the most-representative sampling possible within the limitations imposed by handling small-sized samples. No preferential selection of zircon was made based on size, color, shape, roundness, and metamictization. This aim is best achieved by minimising individual grain handling and hand picking only to ensure purity of zircon content as a group rather than the quality of individual grains. Extracted zircon grains were poured into a mound under alcohol in a Petri dish. This mound was directly mixed and then a swathe swept from the middle. Non-zircon grains were removed from this swathe, and the remaining zircon grains, typically numbering 100 to 150 grains, were taken from the dish using a pipette. Zircon grains were mounted on double-sided adhesive tape before being set in an epoxy disk and polished to reveal half-sections. Zircon internal structures were analysed and interpreted from backscattered electron imaging. During analysis, an array of mounted zircon grains was systematically analysed with the only

Table 1. Frantz settings and calculated mass magnetic susceptibility values used in this study.

Setting	θ° side-slope	i A amps	$H \cdot dH/dx$ "Gauss ² /cm" [†]	χ_g cgs "emu/g"	χ_g SI "m ³ /kg"	Fraction [§]
1	5	0.5	3.74×10^7	2.28×10^{-6}	2.87×10^{-8}	E
2	10	1.0	2.21×10^8	7.69×10^{-7}	9.66×10^{-9}	D
3	10	1.8	3.60×10^8	4.72×10^{-7}	9.66×10^{-9}	C
4	5	1.8	3.60×10^8	2.37×10^{-7}	2.98×10^{-9}	B
5	1	1.8	3.60×10^8	4.75×10^{-8}	5.96×10^{-10}	A

[†] Magnetic energy gradient strength values obtained from documentation supplied by S.G. Frantz Company Inc. and discussed in Appendix.

[§] Fraction identification next to upper bound of magnetic susceptibility range, i.e. fraction E comprises grains ranging from 2.87×10^{-8} to 9.66×10^{-9} (m³/kg). Grains in fraction A potentially range from 5.96×10^{-10} to a theoretically pure zircon diamagnetic value -4.9×10^{-9} (m³/kg) Lewis and Senftle, 1966.

operator choice being the avoidance of any obvious imperfections in the grain surfaces.

Because grain size is a frequent consideration in conventional zircon analysis (Silver, 1963b; Silver and Deutsch, 1963) and a potential provenance feature (Morton et al., 1996), the sizes of analysed grains were also measured using digital image analysis. This approach assumes that the 2-D section of a grain is an accurate measurement of the overall size of that grain. Because zircon grains are typically elongate (with mean breadth/length ratio typically 0.6–0.7), three measurements are reported here: breadth, length, and equivalent diameter. The latter is the diameter of a circle of equivalent area as the digitized grain image (Russ, 1994). This measurement better reflects the potential hydraulic behavior of the grain than breadth alone. Breadth reflects the equivalent sieve mesh size and is thus compatible with conventional sedimentological grain-size measurements. The sorting and skewness parameters described here have been calculated using standard grain-size statistical procedures (Folk and Ward, 1957; Folk, 1980; Boggs, 1995).

Using standard analytical techniques on the Geological Survey of Canada's SHRIMP II ion microprobe (Stern, 1997), Pb/Pb ratios were measured directly and Pb/U ratios by comparison with known standards (BR266, ²⁰⁶Pb/²³⁸U: 0.09059; Kipawa, ²⁰⁶Pb/²³⁸U: 0.16654) analysed sequentially with the unknown samples. As described above, at least 60 individual grain analyses are required to satisfy statistical adequacy for detrital investigations (Dodson et al., 1988; Sircombe, 2000a). To reach such a target for all five fractions within a reasonable schedule, the number of scans per analysis was reduced to three. While this has resulted in poorer precision in the age measurements (median ± 1 s.e. in the five fractions range from 47–117 Ma) the results remain adequate to test for any age component bias between paramagnetic fractions. Analyses were corrected for common Pb content using ²⁰⁴Pb measurements and examined for concordance/discordance. Because one purpose of this study was to examine compositional correlations with magnetic susceptibility, the otherwise routine canceling of analyses with high ²⁰⁴Pb was avoided. Nevertheless, only measurements with between 5% and –5% discordance have been utilized for subsequent provenance interpretation. Because analyses with extremely high

common-Pb and U content have been included, average values may be skewed high. To avoid this, median compositional values are reported.

Grain ages are calculated using the ²⁰⁷Pb/²⁰⁶Pb ratios and reported with errors at ± 2 s.e. In the cases where multiple analyses have been made on a single grain, statistically similar ages were pooled, whereas significantly different ages, for instance those associated with rims and cores in electron imaging, were treated as separate provenance ages. Mixture modeling procedures (Sambridge and Compston, 1994) were also applied to deconvolve complex age distributions.

α -dosage values based on U and Th content and age were calculated using the procedures of Murakami et al. (1991) and are used as the basis for quantifying metamictization.

Isotopic, grain-size, and α -dosage data are presented in supplementary tables. Univariate age data are displayed using a combination of histograms and probability/density distributions created by accumulating individual gaussian distributions (Fig. 2) (Sircombe, 2000b).

5. RESULTS

5.1. Previous Analysis: 1.8 A, 5° Non-Magnetic

In the previous examination of this sample (Sircombe et al., 2001), detrital zircon was extracted at the "typical" level as the non-magnetic fraction at an electromagnet current of 1.8 A and 5° side-slope. Grains were analysed with five mass scans. A total of 88 analyses were made on 80 individual grains, yielding 68 concordant ages. The majority of grains have a generally clear appearance and few inclusions. U and common-Pb content have median values of 148 ppm and 3 ppb, respectively, with an average discordance of 2.2%. The concordant ²⁰⁷Pb/²⁰⁶Pb ages can be subdivided broadly into three groups: (1) ~2960 Ma (29% of total concordant grains), (2) ~3150 Ma (34% of total concordant grains), and (3) a broad range between

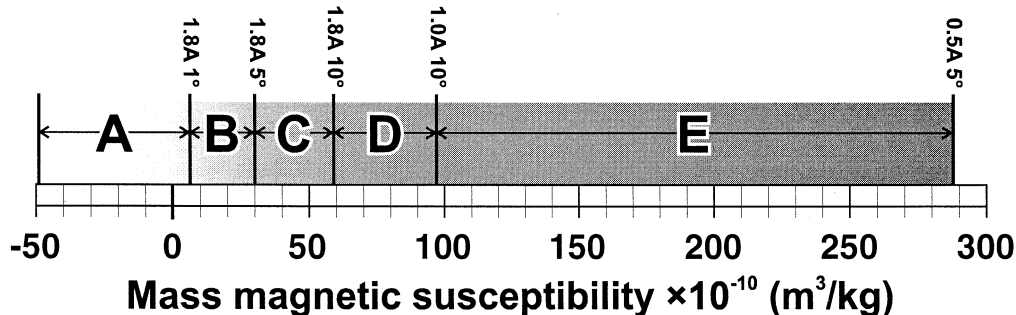


Fig. 1. Illustration of the potential range of mass magnetic susceptibilities represented by the five fractions used in this study ranging from A, the least paramagnetic and potentially also diamagnetic, to E the most paramagnetic. Magnetic susceptibility values as calculated in the Appendix and given in Table 1.

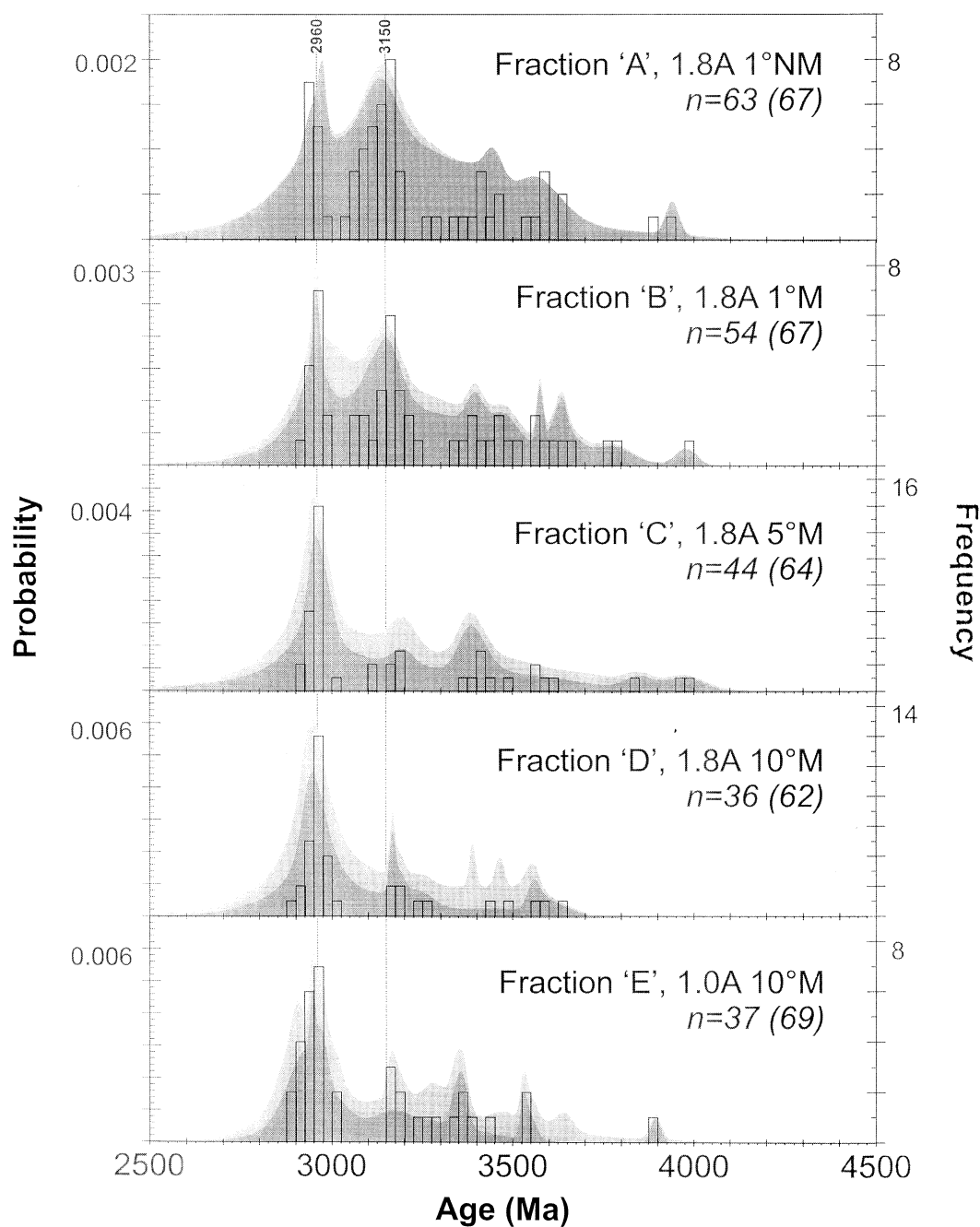


Fig. 2. Age probability density distributions and histograms for the five paramagnetic fractions. Dark-grey-filled curves represent the accumulation of concordant $^{207}\text{Pb}/^{206}\text{Pb}$ ages scaled to total 1. Light-grey-filled curves represent the combination of concordant and discordant analyses. n = indicates number of concordant ages (between -5 and $+5\%$ discordance); (n) indicates total number of analyses.

3300 and 3600 Ma (26% of total concordant grains). The oldest analysed grain is at 3918 ± 10 Ma. Mixture modeling subdivides these groups into several smaller subgroups (Table 2).

5.2. Fraction E: 1.0 A, 10° Paramagnetic

A total of 69 analyses on 68 grains were made, yielding 37 concordant ages. Almost all grains are cloudy and mottled in

appearance with common inclusions and corroded surface texture. U and common-Pb content have median values of 714 ppm and 54 ppb, respectively, with an average discordance of 11.7%. The concordant $^{207}\text{Pb}/^{206}\text{Pb}$ ages are dominated (57%) by an age mode, as defined by mixture modeling, at 2950 ± 10 Ma (Table 2). Two smaller modes occur at 3183 ± 76 Ma (14%) and 3351 ± 17 Ma (19%). Ages ranging between 3300 and 4000 Ma comprise 22% of the total with one age older than 3800 Ma.

Table 2. Summary of mixture modeling age components, grain-size and compositional data.

<i>Previous Dwyer Lake[†]</i>	<i>Fraction</i>					<i>Event[‡]</i>
	A	B	C	D	E	
2945 ± 13 (3)				2949 ± 17 (24)	2950 ± 10 (22)	VII
2962 ± 4 (15)	2960 ± 17 (17)	2958 ± 13 (14)	2959 ± 16 (25)			
3042 ± 21 (3)						VI
3111 ± 11 (8)	3131 ± 34 (26)					
3146 ± 8 (10)		3148 ± 11 (20)				V
				3170 ± 13 (6)		
3180 ± 11 (5)			3193 ± 56 (4)		3183 ± 76 (5)	IV
various individual grains	3305 ± 80 (4)	3398 ± 36 (6)	3392 ± 30 (12)		3351 ± 16 (7)	III
	3439 ± 42 (8)	3475 ± 55 (4)				
	3570 ± 62 (6)	3586 ± 5 (4)		3564 ± 26 (5)		II
		3637 ± 26 (3)				
0.29	0.25	0.27	0.26	0.24	0.21	Sorting ϕ
0.09	-0.12	-0.12	-0.10	0.05	-0.03	Skewness
66 ± 29	56 ± 29	66 ± 29	72 ± 29	69 ± 29	70 ± 29	Breadth μm
148	96	181	317	457	714	U ppm
3	17	12	16	39	54	²⁰⁴ Pb ppb
2.2	0.1	4.4	5.6	6.0	11.7	Discordance

All age values given with ± 2 standard errors. The number of individual analyses (n) assigned to the modeled age component is given in brackets. Note that each sample will have several analyses that are independent of these modeled groups and thus will not be accounted for in the table. U ppm and ²⁰⁴Pb ppb values are median values for each fraction. Discordance is the average percentage value of each fraction. [†] Data from original analysis of Dwyer Lake quartzite sample Sircombe et al., 2001. [‡] Data are placed in the context of Slave province crustal formation/reworking events defined by Bleeker and Davis, 1999.

5.3. Fraction D: 1.8 A, 10° Paramagnetic

A total of 62 grains were analysed, yielding 36 concordant ages. The appearance of the grains ranges from cloudy to clear with infrequent inclusions and rare corroded surface textures. U and common-Pb content have median values of 457 ppm and 39 ppb, respectively, with an average discordance of 6.0%. The concordant ²⁰⁷Pb/²⁰⁶Pb ages are dominated (67%) by a mode defined by mixture modeling at 2949 ± 17 Ma (Table 2). Two smaller modes occur at 3170 ± 13 Ma (17%) and 3564 ± 26 Ma (14%). Ages ranging between 3300 and 4000 Ma comprise 14% of the total with no ages older than 3800 Ma.

5.4. Fraction C: 1.8 A, 5° Paramagnetic

A total of 64 analyses on 63 individual grains were made, yielding 44 concordant ages. Grains are generally clear in appearance with infrequent inclusions. U and common-Pb content have median values of 317 ppm and 16 ppb, respectively, with an average discordance of 5.6%. The concordant ²⁰⁷Pb/²⁰⁶Pb ages are dominated (57%) by a mode defined by mixture modeling at 2959 ± 16 Ma (Table 2). Two smaller modes occur at 3193 ± 56 Ma (9%) and 3391 ± 30 Ma (27%). Ages ranging between 3300 and 4000 Ma comprise 31% of the total, including three ages older than 3800 Ma.

5.5. Fraction B: 1.8 A, 1° Paramagnetic

A total of 67 analyses on 66 individual grains yielded 54 concordant ages. Grains generally have a clear appearance, infrequent inclusions, and rare overgrowths. U and common-Pb

content have median values of 181 ppm and 12 ppb, respectively, with an average discordance of 4.4%. The concordant ²⁰⁷Pb/²⁰⁶Pb ages are dominated by two principal modes at 3148 ± 21 Ma (37%) and 2958 ± 13 Ma (26%) (Table 2). Other minor modes occur in the range from 3300 to 4000 Ma and comprise 37% of the total. The oldest age within all five fractions occurs within this latter fraction at 3979 ± 58 Ma (#B25.1).

5.6. Fraction A: 1.8 A, 1° Non-Magnetic

A total of 67 analyses on 65 individual grains yielded 63 concordant ages. The appearance of the grains is generally very clear with rare inclusions and rare preservation of euhedral crystal faces. U and common-Pb content have median values of 96 ppm and 17 ppb, respectively, with an average discordance of 0.1%. The concordant ²⁰⁷Pb/²⁰⁶Pb ages are dominated by two principal modes at 3131 ± 34 Ma (41%) and 2960 ± 17 Ma (27%) (Table 2). Other minor modes occur in the range from 3300 to 4000 Ma and comprise 29% of the total, including two individual grain ages greater than 3800 Ma.

6. DISCUSSION

6.1. Age Modes, Magnetic Susceptibility, and Metamictization

The most striking feature of this set of data is the presence or absence of grain ages associated with the ~3150 Ma component previously identified (Sircombe et al., 2001). While ages associated with the ~2960 Ma component are ubiquitous in all

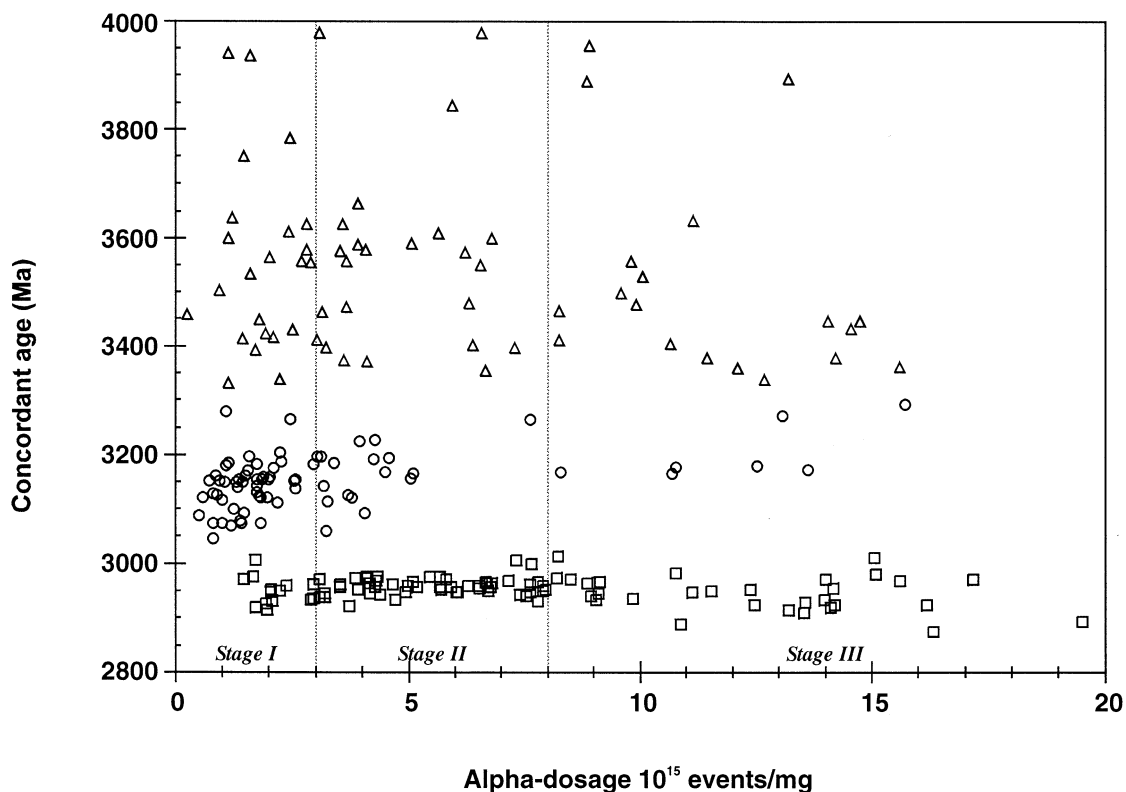


Fig. 3. Scatterplot illustrating the relationship between α -dosage/metamictization and age in the concordant results in the entire set of data. Vertical lines indicate the three divisions of metamictization defined by Murakami et al. (1991) from purely crystalline to highly metamict. Squares, circles, and triangles highlight the ages in the ~ 2960 Ma, ~ 3150 Ma, and >3300 Ma age components, respectively.

five fractions, ~ 3150 Ma ages are only seen prominently in the two least-paramagnetic fractions (Fig. 2). The numerical ratio of ~ 2960 Ma ages to ~ 3150 Ma ages varies markedly from 0.41 to 0.70 in fractions A and B, to 3.29, 4.33, and 2.63 in fractions C, D, and E, respectively. The magnetic susceptibility fractionation of the sample clearly influences the presence of zircon grains associated with the ~ 3150 Ma component. Another feature of the age distributions is the reduction in grains in the >3300 Ma range from 29, 35, and 32% in fractions A, B, and C, respectively, to 14 and 21% in fractions D and E, respectively.

The explanation of these age distribution features lies in the correlations between metamictization, magnetic susceptibility, and age. When concordant ages are plotted against α -dose (Fig. 3), it can be seen that, overall, the ~ 3150 Ma ages have distinctly lower α -doses than the ~ 2960 Ma ages, with median values of 2.0 and 6.5×10^{15} α -decay events/mg, respectively.

The correlations between magnetic susceptibility, U content, and discordance, as discussed by Silver (1963a) and Krogh (1982), are further illustrated in this study by Figure 4. The median U content increases with increasing magnetic susceptibility from fraction A to E. Coincident with increased U content is the greater potential for crystal lattice damage as quantified by α -dose (Fig. 4b). The positive correlation between paramagnetism and discordance seen in Figure 4c is consistent with the previous results. These results reinforce the ideas of Lewis and Senftle (1966) that metamictization and the

subsequent introduction of Fe-compounds into the zircon lattice contributed to paramagnetism.

6.2. Management of Discordant Results and Paramagnetism

Managing discordant results has long been a major concern of zircon geochronology. Indeed, avoiding discordance is the purpose of the development and continuing wide use of magnetic separation techniques. The relationship between magnetic susceptibility and discordance is further illustrated in this study in which many results, particularly those in the two most-magnetic fractions, are classified as discordant with unreliable age interpretations. Such discordant analyses are routinely excluded from further interpretation—but does this in itself introduce a bias?

Undoubtedly, some biasing does occur. The discordant analyses are more likely to be from the more paramagnetic grains, as shown in Figure 4c. Given sufficient time between formation and transportation, these grains have already been subject to natural biasing because of preferential destruction during transportation. Assuming a representative selection of available grains during hand picking, artificial biasing against discordant grains occurs in two stages. First, although the SHRIMP ion probe increases the range of material that can be analysed, spot location is not truly random because of limitations imposed by internal structures and the avoidance of obvious surface imper-

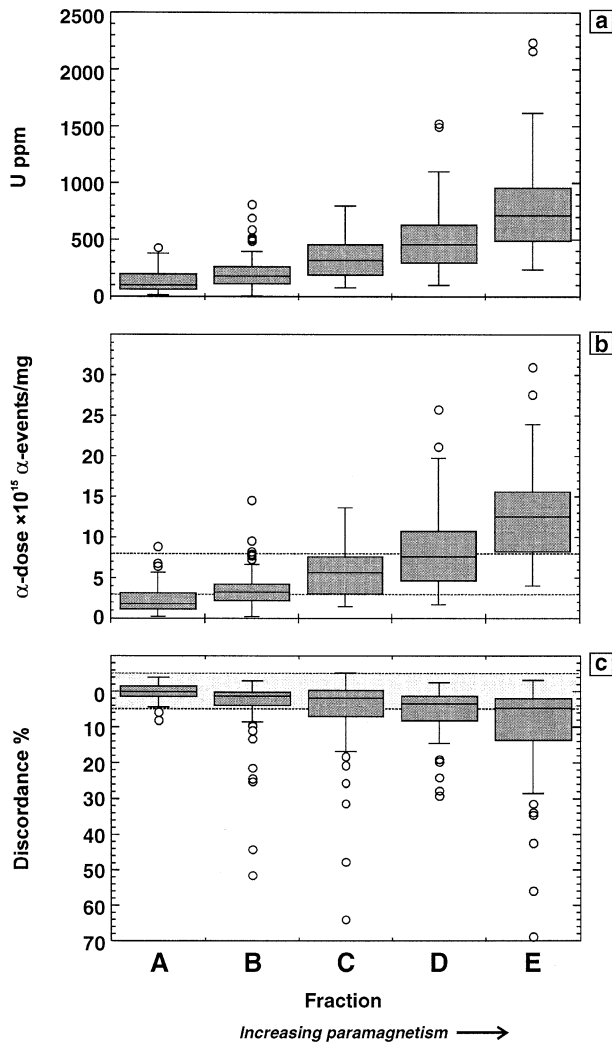


Fig. 4. Box diagrams illustrating the relationships between (a) U content and magnetic susceptibility, (b) α -dosage and magnetic susceptibility, with horizontal lines indicating the metamict stages defined by Murakami et al. (1991), and (c) discordance and magnetic susceptibility represented by the five paramagnetic fractions. All data are included. Boxes represent 50% of the data between the lower and upper quartiles. Circles represent outliers defined as being greater or less than the upper quartile plus 1.5 times the interquartile distance.

fections and/or areas of metamictization. In these cases, it is more important to use the spatial ability of the SHRIMP technique to gain some insight into the age and history of the grain rather than arbitrarily analysing a random spot. The impact of spot selection biasing is considered minimal, because at least part of the grain is still being analysed and is available for interpretation.

The second stage of biasing is the exclusion of results with a particular level of discordance. Quantifying this discard bias is difficult because by definition, the results discarded are unreliable, and, therefore, comparisons between complete and edited data are equally unreliable. However, the results presented here do provide some insight into this complex issue and suggest some limits for future work. Even if the $^{207}\text{Pb}/^{206}\text{Pb}$ ages of the discordant grains were not to be discarded, it can be

seen in fractions A to C (light-grey curves on Fig. 2) that the overall age distribution does not change significantly. It is in the two most-paramagnetic fractions, D and E, that significant problems emerge. Both fractions require the discarding of over 40% of the analyses—clearly inefficient use of analytical time. The age distributions also alter between the complete age data and concordant-only age data, with various modes altering markedly. Finally and more seriously, some of the supposedly concordant results show signs of unreliability. The age of the youngest mode in fractions D and E are ~ 10 Myr younger than the ~ 2960 Ma components seen in the other fractions. In addition, some “concordant” measurements in fraction E are below the minimum limiting age given by overlying volcanics. This suggests that these apparently concordant grains have sufficient U content, metamictization, and related Pb-loss to cause slight discordance that is not detectable with this analytical method on an individual basis.

These results indicate that the most-paramagnetic fractions (D and E) are generally unreliable and their analysis is inefficient. A good compromise between representation and analytical efficiency/reliability occurs with fraction C at a Frantz separator setting of 1.8 A and 10° side-slope.

6.3. Provenance Interpretations

On a first pass, it could be stated that the least-paramagnetic fraction alone is sufficient to illustrate the age distribution because the major modes are present and their presence does not change in the other fractions. This view is challenged in two ways.

The results here indicate that this “sufficient” representation in the least-magnetic fraction cannot be assumed. The bulk of the ~ 2960 Ma ages occurs in Stage II metamictization between 3 and 8×10^{15} α -events/mg (Fig. 3). In this case, some of the ~ 2960 Ma mode have relatively lower U content and levels of metamictization and are seen in the least-paramagnetic fraction at the Frantz settings used for this study. Can this fortunate coincidence be automatically assumed to occur in all samples examined for detrital zircon geochronology? An even more rigorous setting, especially a diamagnetic setting (reverse slope on the Frantz chute), could easily eliminate the presence of this mode altogether. There are only five ~ 2960 Ma mode grains with α -doses below 2×10^{15} α -events/mg and none below 1×10^{15} α -events/mg.

The second argument against the apparently sufficient representation in the least-paramagnetic fraction involves the nature of the interpretation itself. While it has been acknowledged that quantitative provenance is not possible, this does not mean that the relative size of particular modes has no significance. For example, the prevalence of a particular mode occurring in multiple samples over a wide area would point to the importance of such rocks in the area being eroded. High throughput methods such as available with spot dating methods allow such statistical geochronology to be accomplished, and thus steps should be taken to avoid potential analytical biases, such as taking the least-magnetic fraction.

The study here illustrates what is possible with comparisons between the two principal age modes. By including data from fractions B and C, it can be seen that the ~ 2960 Ma age mode is more prevalent than would be considered using fraction A

alone. This can be added to the provenance interpretation, because it indicates that the ~ 2960 Ma source was a major supplier of the sediment, possibly even more than the ~ 3150 Ma source. The concentration of the ~ 3150 Ma mode in the least-paramagnetic fraction suggests a compositional control and/or the selective destruction of ~ 3150 Ma grains with higher degrees of metamictization during transportation.

The latter interpretation is tempting, because it potentially offers further provenance information about distal and multi-cycle sources. However, as discussed below, further investigation of the results suggests the assumption of selective destruction during transportation needs careful consideration.

6.4. Metamictization, Sedimentary Transportation, and Paramagnetic Biasing

Grain survivability has been correlated in some cases with U content (Heaman and Parrish, 1991), but survivability depends on metamictization, which in turn depends on uranium content and, possibly most importantly, age. A high U grain will survive transportation through to deposition and subsequent analysis if transportation occurs before α -decay damage becomes too great. The combination of high-energy environments and relatively recent dominant sources (typically 650–150 Ma) can be seen in data from zircon in modern beach sand in eastern Australia (selected for SHRIMP analysis with Frantz settings at 2 A and 5° side-slope and non-biasing criteria; Sircombe, 1999). With over 800 analyses, the median α -dose is 0.44×10^{15} α -events/mg and the 95 percentile is 1.91×10^{15} α -events/mg. (The comparable U content values are 209 ppm and 629 ppm, respectively, again reinforcing the unsuitability of U content alone as a limit.) In comparison, for the Dwyer Lake metaquartzite, the 95 percentile of α -dose in fraction A alone is 5.7×10^{15} α -events/mg. By incorporating concordant results in fractions B and C, this rises to a 95 percentile at 8.5×10^{15} α -events/mg.

The comparison between the ancient Dwyer Lake sample and modern beach sand is relevant to the discussion of biasing here. As illustrated in Figure 4, metamictization is correlated to paramagnetism. Hence, this paramagnetic biasing may only occur when metamictization has reached a sufficient level. For instance, biasing is not a problem in the recent beach sands of eastern Australia, because metamictization has not occurred to the degree where the intrusion of Fe-compounds into the damaged lattice will induce paramagnetic biases during sample preparation.

Provenance interpretations explaining modes with restricted U content (such as the ~ 3150 Ma mode discussed above) by “loss of higher-U grains during transportation” are also incorrect if the time between formation and deposition is not accounted for. The 95 percentile of α -dose on modern beach sands at 1.91×10^{15} α -events/mg is taken as an indicator of the degree to which zircon metamictization is destroyed in a high-energy environment. Only those grains from the ~ 3150 Ma source with extreme U content of >1700 ppm would have reached such a level of α -dose in the ~ 300 Myr between formation and deposition. The time between formation and deposition in the ~ 2960 Ma mode is even shorter. The low U content and low α -dose in the ~ 3150 Ma mode (and conversely the relatively higher values in the ~ 2960 Ma mode) are

taken as reflecting original source composition control rather than sedimentary processes.

6.5. Size, Magnetic Susceptibility, and Age

This set of data also provides an opportunity to assess the relation between zircon grain size in a sedimentary setting and such parameters as discordance and age. Partitioning of zircon samples based on size is often performed in conventional geochronology (Silver, 1963b; Silver and Deutsch, 1963). A correlation between size and age could also yield important provenance information.

However, in this sample, size is not correlated to either discordance (Fig. 5a) or concordant age (Fig. 5b). This suggests, at least for heterogeneous sedimentary zircon suites, that size is not related to discordance and is thus not a useful way to fractionate and select the “best” zircon samples. This example also indicates that zircon grain size may not be a diagnostic provenance feature in mature, well-sorted sediments.

7. CONCLUSIONS

Detrital zircon geochronology is subject to limitations imposed by natural and artificial biases. Natural biases are induced during sedimentary processes and are an acknowledged fact of any provenance analysis project. In contrast, artificial biases are induced during sample selection and preparation and can be monitored and mitigated. This study has focused on one of these artificial biases caused by paramagnetic fractionation during sample preparation using the Frantz magnetic barrier separator.

The results presented here further illustrate the known relationship between paramagnetism and U content/metamictization (Fig. 4). The data clearly illustrate that there is a bias in the distribution of ages depending on paramagnetism. The assumption that despite this biasing, the least-paramagnetic fraction remains sufficient for “there or not” interpretations is considered unreliable and, potentially, an unnecessary source of bias. The nature of the “there or not” provenance interpretation is also considered simplistic, and while a pure quantitative interpretation is not possible, further provenance information is available in a sample selected from a broader paramagnetic range.

The data presented also provide insight into other issues regarding detrital zircon geochronology and provenance interpretations. Careful consideration must be given to metamictization. Previous assumptions about U content and grain survival in sedimentary processes are too simplistic. In addition, paramagnetic biasing may only be an issue in Archean zircons in which sufficient metamictization has occurred to induce a range of paramagnetism. It is possible to differentiate in provenance interpretations between compositional control and possible losses during sedimentary transport by accounting for α -damage and the time between formation and deposition. A secondary issue is grain size, and at least for the heterogeneous sedimentary zircon suite from a mature sediment presented here, grain size has no control over grain discordance.

The management of discordant grains is always of concern in detrital zircon geochronology. While at the very least, including zircon grains from a broader paramagnetic range can only enhance

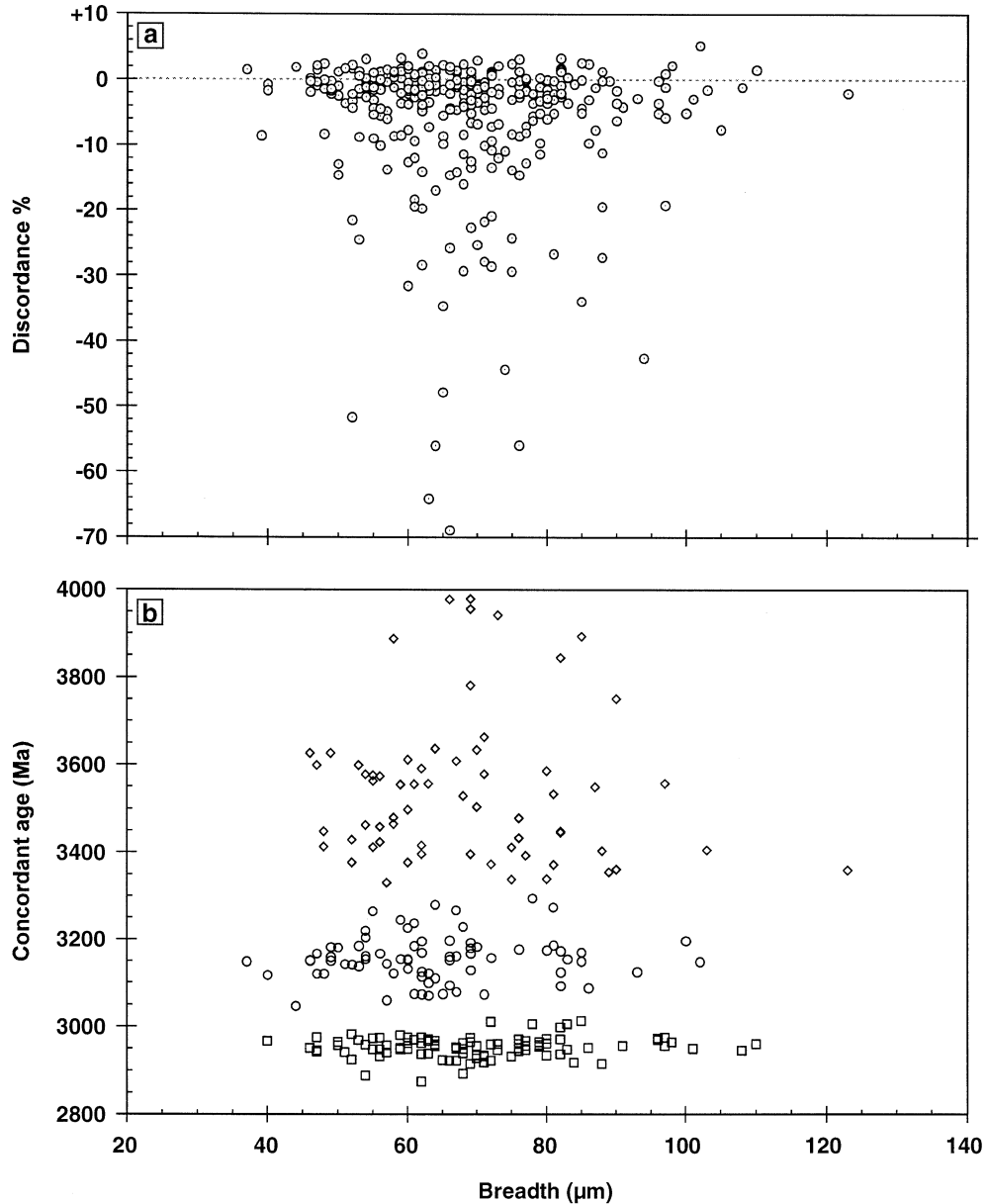


Fig. 5. Scatterplot illustrating the lack of relationship between (a) breadth and the discordance of all the analysed grains and (b) breadth and concordant $^{207}\text{Pb}/^{206}\text{Pb}$ ages only. Squares, circles, and triangles highlight the ages in the ~ 2960 Ma, ~ 3150 Ma, and >3300 Ma age components, respectively.

provenance interpretations, results from higher paramagnetic fractions become increasingly unreliable and thus analytically inefficient. The results of this study indicate that taking the non-magnetic fraction of a detrital zircon sample at a Frantz setting of $1.8 \text{ A } 10^\circ$ side-slope, or a mass magnetic susceptibility of $4.72 \times 10^{-7} \text{ e.m.u./g}$ ($5.93 \times 10^{-9} \text{ m}^3/\text{kg}$), is a reasonable compromise between representation and analytical efficiency.

Acknowledgments—This paper greatly benefited from reviews by K. Ansdell, W. Bleeker, D. Davis, R. Ernst, V. McNicoll, and A. Nemchin. J. Sun (S.G. Frantz Co. Inc.) is thanked for his prompt and informative responses to inquiries. KNS acknowledges support of a NSERC Canadian Laboratories Visiting Fellowship. GSC Contribution number 2000196.

Associate editor: Y. Amelin

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APPENDIX

OPERATION AND CALIBRATION OF THE FRANTZ MAGNETIC BARRIER SEPARATOR

Operation

There have been a number of descriptions of the operation of the Frantz magnetic separators (Gaudin and Spedden, 1943; McAndrew, 1957; Rojas et al., 1965; Gabenisch et al., 1972; Oberteuffer, 1974). Some of these descriptions were based on the earlier L-1 “Isodynamic” model and are often focused on a largely empirical approach to mineral separation (e.g., Rosenblum, 1958; Flinter, 1959). However, the theoretical basis for operation is largely the same for the LB-1 “Barrier” model. (The LB-1 model introduced pole pieces that produced a planar isodynamic magnetic field with an energy gradient approximately four times greater than the broad isodynamic field of the L-1—hence, the “magnetic barrier” nomenclature. This makes the LB-1 more sensitive in separating materials with low ranges of paramagnetic or diamagnetic susceptibilities. The sense of tilt on the side-slope is also reversed with paramagnetic materials being attracted toward the inner edge of the chute in the LB-1 rather than the outer edge of the L-1 chute as described in the earlier literature.)

The two principal forces that act on a grain moving down the chute are illustrated in Figure A1. The gravitational force, F_g , acts down the direction of maximum slope, while the magnetic force, F_m , induced in the grain, acts perpendicularly to the chute. Following the nomenclature of McAndrew (1957) in CGS units:

$$F_m = \chi_g m H \frac{dH}{dx} \quad (1)$$

where a paramagnetic or diamagnetic particle has mass m grams and mass magnetic susceptibility χ_g and is acted on by a magnetic field with strength H oersteds (or gauss, assuming a free-space permeability of 1) and a gradient of dH/dx measured in distance of cm. The gravitational component acting across the chute is given by:

$$F'_g = mg \sin \theta \quad (2)$$

where g is the gravitational constant (980 cm/s²) and θ is the side-slope of the chute (Fig. A1). The forward slope of the chute is not considered critical to the separation process, provided the rate of flow is slow

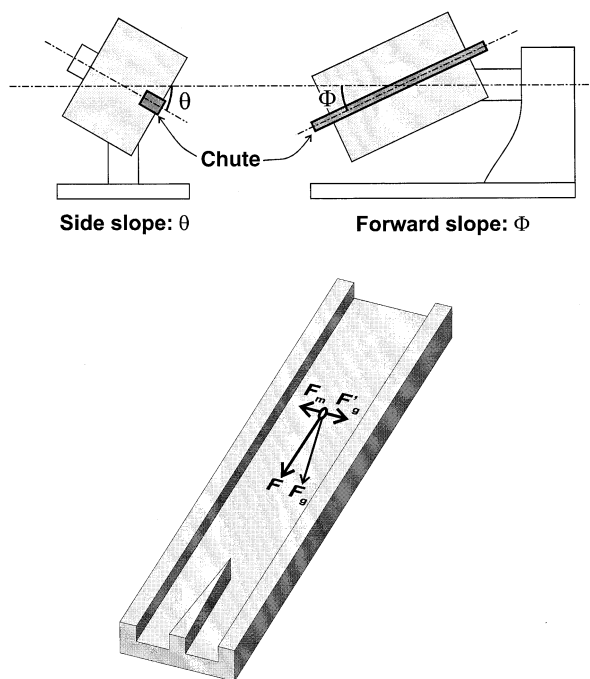


Fig. A1. Schematic illustration of the Frantz mechanical parameters and forces acting on a grain moving in the chute.

enough to allow grains moving in the chute to interact effectively with the magnetic barrier (McAndrew, 1957; Flinter, 1959). A forward slope of 10° was maintained throughout the separations described here.

Eqn. 1 and 2 can be combined to provide a calculation of mass magnetic susceptibility:

$$\chi_g = g \sin \theta \frac{1}{H} \frac{dx}{dH} \text{ (emu/g)} \quad (3)$$

This can be converted to SI values by multiplying by $4\pi/1000$. The value of magnetic energy gradient for a given setting of the electromagnet current can be determined using two methods described below.

Magnetic Energy Gradient: Theoretical Calculation

The strength of the magnetic energy gradient (HdH/dx) at the magnetic barrier of the LB-1 is illustrated in Figure A2 using data supplied by S. G. Frantz Co. Ltd. Using a modified sigmoid function, this relationship can be modeled as:

$$H \frac{dH}{dx} = \left(\frac{0.0038544}{0.00032445 + e^{-10.296i}} + \frac{1.0511}{0.037619 + e^{-3.051i}} - 1.0168 \right) \times 10^7 \quad (4)$$

where i is the electromagnetic current in amperes. This calculation of magnetic energy gradient can then be used in Eqn. 3 to calculate magnetic susceptibility at various side-slope angle settings as illustrated in Figure A3. However, it must be acknowledged that these values are theoretical only. The actual strength of the magnetic field produced by any individual Frantz separator depends on the state of the electromagnet and the accuracy of the current meter. These features will undoubtedly vary from machine to machine, but the significance and range of the resulting inconsistency is unknown at this stage. Ideally, paramagnetic fractions should be reported in absolute terms of mass magnetic susceptibility that is then independent of any individual Frantz machine. As a step toward this, a method for direct calibration of an individual Frantz machine is described below.

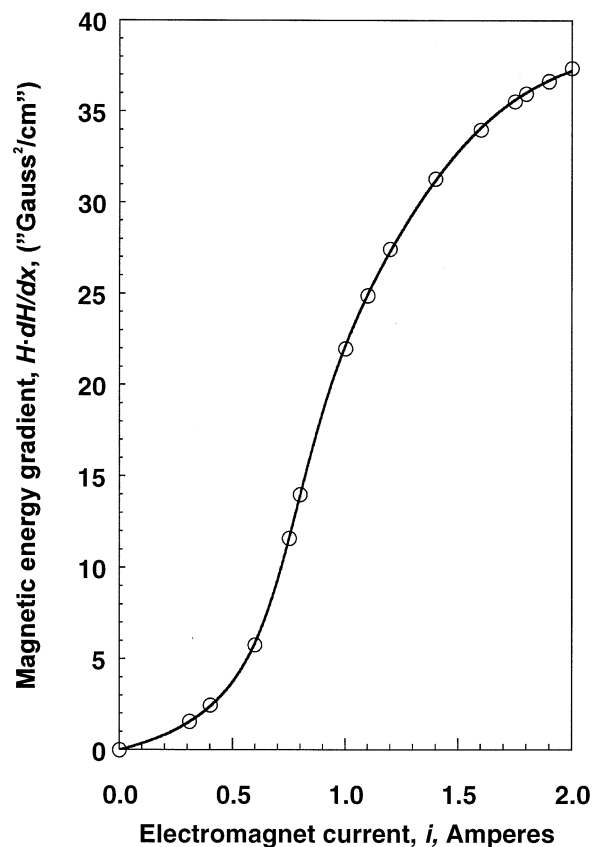


Fig. A2. Graph illustrating the magnetic energy gradient vs. electromagnet current for the LB-1 magnetic barrier separator. Based on graph supplied by S.G. Frantz Co. Inc.

Magnetic Energy Gradient: Direct Calibration

McAndrew (1957) presented a method for calibrating the L-1 separator, and although the calculations do not directly apply to the LB-1 separator, the methodology can be applied. By equating Eqn. 1 and 2:

$$\frac{\sin \theta}{\chi_g} = \frac{1}{g} H \frac{dH}{dx} \quad (5)$$

and assuming that the magnetic field strength will increase at the same rate as the electromagnet current, particularly at low currents, Eqn. 4 can be written as a power-law relationship:

$$\frac{\sin \theta}{\chi_g} = ki^n \quad (6)$$

where i is the electromagnet current, n is a constant close to 2, and k has a constant value. Applying this relationship, McAndrew (1957) used substances of known magnetic susceptibility to find the calibration for the L-1 separator ($n = 2$, $k = 52,000$). Using data from S.G. Frantz that directly defines the relationship between magnetic field strength and electromagnet current for the L-1 separator, the derived calibration is close to that based on direct measurement of the magnetic field for electromagnet current values from ~ 0.5 to 1.0 A. A similar approach was used to check the calibration of the LB-1 separator used in this study.

Two inorganic compounds used by McAndrew (1957), ammonium iron(II) sulphate, $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 0.6\text{H}_2\text{O}$, and copper (II) sulphate, $\text{CuSO}_4 \cdot 0.5\text{H}_2\text{O}$, were selected as having mass magnetic susceptibilities suitable for calibrating the Frantz separator at 32.3×10^{-6} e.m.u./g (40.6×10^{-8} m³/kg) and 6.13×10^{-6} e.m.u./g (7.7×10^{-8} m³/kg), respectively, at 20°C (Kaye and Laby, 1973). One limitation is that these substances tend to be only practically useful in the lower range of

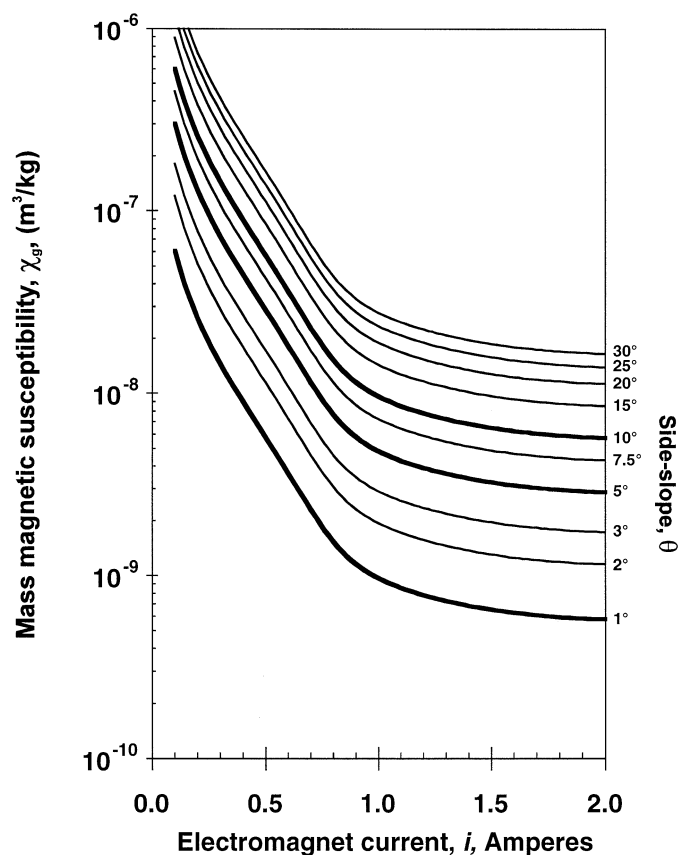


Fig. A3. Graph illustrating the relation between Frantz settings (side-slope and electromagnet current) and magnetic susceptibility derived using calculations for the magnetic energy gradient as discussed in the Appendix.

electromagnet current/magnetic field strength, thus requiring extrapolation of the results to higher field strengths more typical of zircon separation.

The separation process used during calibration was identical to that typically used in mineral separations. Materials used were sieved at 48 mesh (~340 microns) to remove large crystals. Material moved from the inside chute toward the magnetic barrier under the influence of gravity, depending on the side-slope. The electromagnet current was increased until a balance between the amount of material flowing along and passing through the barrier was achieved. These measurements are shown in Table A1 and plotted in Figure A4. The resulting best fit ($R = 0.99$) power-law equation results in $k = 87686$ and $n = 1.0821$.

Figure A5 illustrates a comparison between the two methods for calculating magnetic susceptibility as discussed above. The two calculations are within an order of magnitude of each other, indicating that the Frantz separator used in this study is not a radical departure from the expected magnetic field strength. However, it does highlight that the assumption of a power-law relationship between current and magnetic energy gradient is too simplistic for the LB-1 separator. Further work is required, particularly with substances with lower magnetic

susceptibility that is closer to that commonly seen in zircon, to fully develop the Frantz calibration method into an independent test of magnetic field behavior. In the interim, the magnetic susceptibility values discussed in this study have been calculated using Eqn. 3 and 4.

Supplementary data tables

GSC Sample ID: BNB97-008
 GSC Geochronology ID: Z6238
 GSC Ion-probe mount ID: IP125
 Topographic map location: 85-J 9637275E 6953175N
 Latitude/Longitude: 62°41'6.8"N 114°19'5.6"W

Uncertainties reported at one sigma and are calculated by numerical propagation of all known sources of error (Stern, 1997). Data have been corrected for common-Pb according to procedures outlined in Stern (1997).

Spot name: Unique identification of spot analysis, in the form

Table A1 Frantz separator electromagnet amperage settings required to paramagnetically balance streams of ammonium iron(II) sulphate and copper (II) sulphate in the chute at various side-slopes.

Side-slope	2.5	5.5	8	10.5	13	15.5	18	20.5	25.5	30.5
Substance					<i>Electromagnet amps</i>					
Fe(NH ₄) ₂ (SO ₄) ₂ ·6H ₂ O			0.05	0.075	0.10	0.12	0.14	0.15	0.175	0.225
CuSO ₄ ·5H ₂ O	0.10	0.25		0.35		0.55		0.60	0.70	

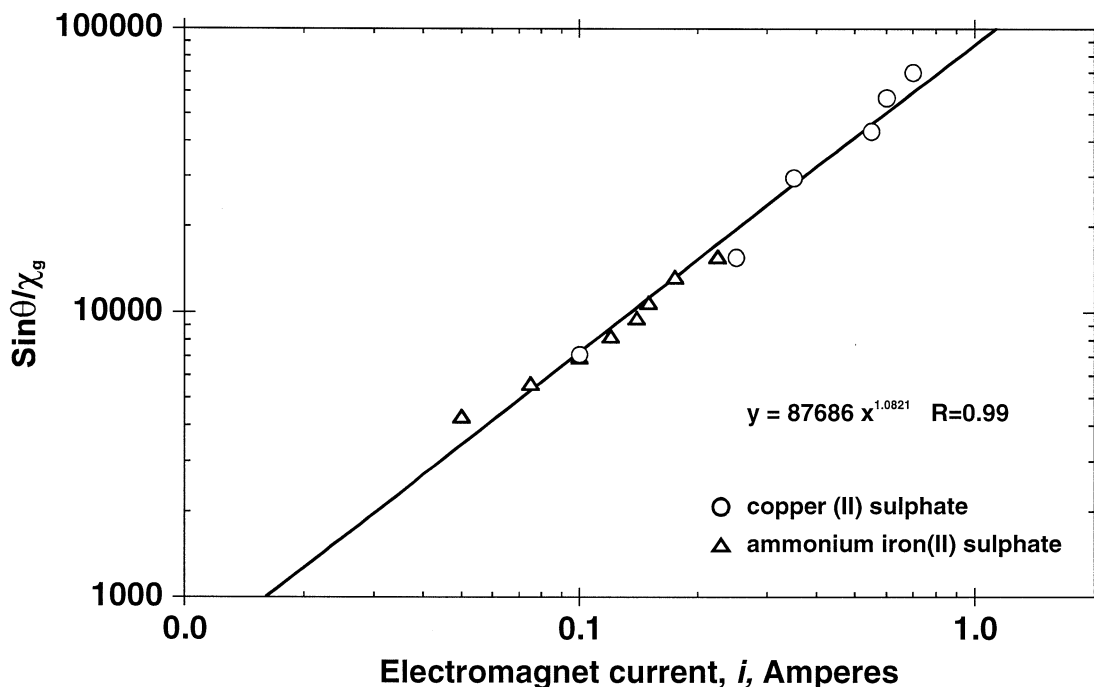


Fig. A4. Graph illustrating the direct calibration measurements using substances of known magnetic susceptibility on the Frantz magnetic barrier separator.

grain-number · spot-number, e.g. 12.2 is the 2nd spot on grain 12. 'C' indicates an analysis on a suspected core area of a grain, 'R' indicates an analysis on a suspected rim. † indicates spots < 95% concordant, ‡ indicates spots >105% concordant, § indicates spots that have been

discarded from further interpretation because of extreme error related to analytical problems, high common-Pb content or are erroneously younger than geochronologic constraints in the case of Fraction E as discussed in text.

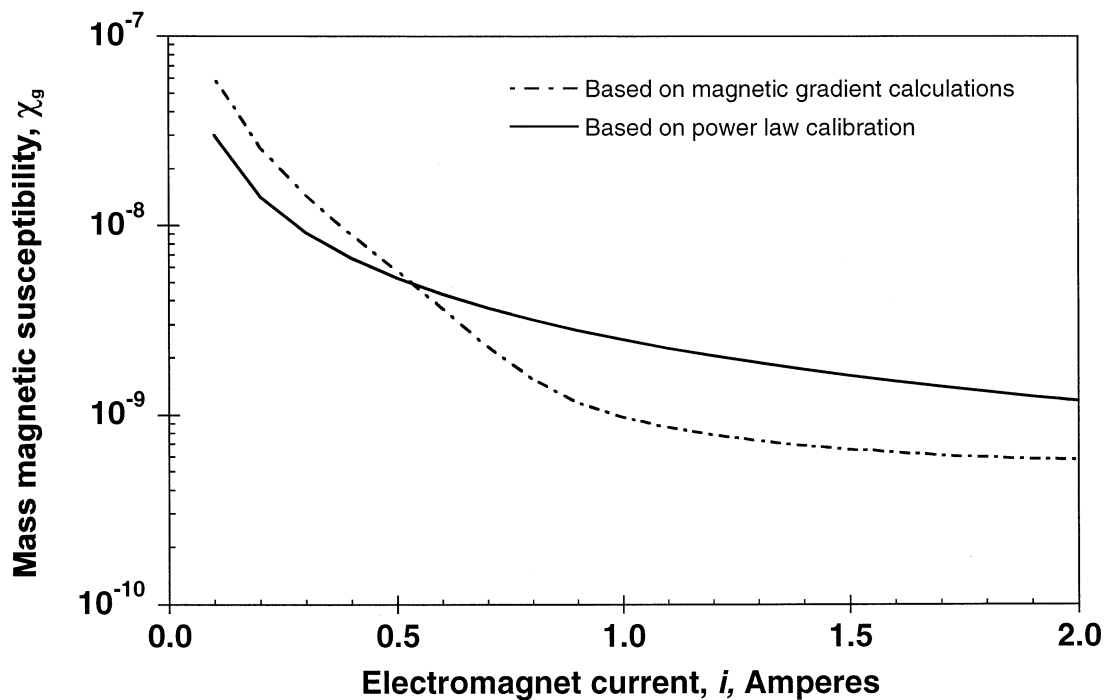


Fig. A5. Graph illustrating the comparison between magnetic susceptibility values derived by using a calculation of magnetic energy gradient and values derived from a power-law relation based on direct measurement of substances of known magnetic susceptibility.

U (ppm): Parts per million uranium abundance. Calculated by calibration against Zr20 in standard zircon.

Th (ppm): Parts per million thorium abundance. Calculated by calibration with U content.

Th/U: Thorium to uranium abundance ratio.

Pb (ppm): Parts per million radiogenic Pb content.

204Pb (ppb): Parts per billion 204(common) Pb content.

204Pb/206Pb: As directly measured.

f206: Refers to mole fraction of total 206Pb that is due to common Pb. Calculated with 204Pb method. A negative value indicates where common Pb content is calculated as less than background.

208Pb/206Pb: As directly measured.

206Pb/238U: As calculated by calibration to standard zircon.

207Pb/206Pb: As directly measured.

Apparent Ages (Ma): Apparent age calculated from 204Pb-corrected 206Pb*/238U and 207Pb/206Pb ratios.

Cone. %: $100 \times (206\text{Pb}/238\text{U age}) / (207\text{Pb}/206\text{Pb age})$.

Disc. %: 100 – concordance%

Alpha-dose: alpha-dose as calculated using formula in Murakami et al. (1991)

Breadth: Minimum axis of digitized image in μm .

Length: Maximum axis of digitized image in μm .

EqDiam: Equivalent diameter of a circle encompassing the same area as the grain.

Form factor: An estimate of roundness varying from 0 to 1 for a perfect circle, defined by $4\pi \cdot \text{area}/\text{perimeter}^2$; (Russ, 1994, p. 525).

Dwyer Lake Quartzite, Slave Province, Magnetic Fraction 'C' 1.8A 5 M

Table with columns: Spot name, U (ppm), Th (ppm), Pb (ppm), 206Pb/238U, 207Pb/235U, Apparent Ages (Ma) for 206Pb/238U, 206Pb/207Pb, 206Pb/238U, 206Pb/207Pb, 206Pb/238U, 206Pb/207Pb, Conc. %, Disc. %, a-dose events/g x 10^15, Length EquDiam, Factor. The table contains 360 rows of data points.

