U-Pb AGES OF ZIRCONS FROM THE GRÃO-PARÁ GROUP AND SERRA DOS CARAJÁS GRANITE, PARÁ, BRAZIL*

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ABSTRACT Zircons collected from weathered rhyolites in the lower metavolcanic sequence of the Grão-Pará Group yield a U-Pb upper intercept age of 2,758 ± Ma. The zircons show no sign of a premagmatic history, and the age is interpreted as the age of volcanism. This Archean age is within the calculated uncertainty of a Rb-Sr whole-rock isochron age for interstratified basalts, reported separately. The U-Pb age is the first indication that the Grão-Pará Group, including the Carajás Formation iron deposits, is Archean, much older than previously thought. Zircons were also collected from weathered Serra dos Carajás Granite adjacent to the quarry near Serra Norte. These yield a U-Pb age of 1,820 ± 49 Ma, which agrees well with previously published age determinations for the granite, and for the Uatumã Supergroup volcanic and shallow plutonic rocks that occur throughout the Amazonian Craton. Zircons from both the Grão-Pará rhyolite and Carajás Granite show evidence for a single stage loss of radiogenic lead, possibly due to dilatancy during Mesozoic uplift of the region. All of the zircons were panned from saprolites, and most exhibit well-defined crystal forms. Tropical weathering is an asset to the collection and field concentration of zircons, and our results confirm that standard U-Pb dating techniques can be successfully applied in deeply-weathered terranes.

RESUMO Os zircões coletados em riólitos decompostos na seqüência metavulcânia inferior do Grupo Grão-Pará produzem uma idade U-Pb de 2.758 ± 39 Ma. Os zircões não apresentam sinais de história pré-magmática e o resultado é interpretado como sendo a idade do vulcanismo. Esta idade arqueana é estatisticamente similar à idade do isócrona Rb-Sr em rocha total de basaltos da mesma seqüência, publicado separadamente. A idade U-Pb é a primeira indicação que o Grupo Grão-Pará, incluindo os depósitos de ferro da Formação Carajás, é arqueano, sendo, portanto, bem mais antigo do que previamente suposto. Zircões foram também coletados em Granito Serra dos Carajás decomposto, nas adjacências da pedreira, junto a Serra Norte. Suas análises produzem uma idade U-Pb de 1.820 ± 49 Ma, que coincide com as determinações de idade publicadas anteriormente relativas ao granito e às rochas vulcânicas e plutônicas do Supergrupo Uatumã que ocorrem no Cráton Amazônico. Ambos os grupos de zircões demonstram uma única perda de chumbo radiogênico. A perda do chumbo foi provavelmente devida à *dilatancy*, durante o soerguimento da região durante o Mesozóico. Todos os zircões foram obtidos em saprólitos e exibem formas cristalinas perfeitas e bem definidas. O intemperismo tropical facilita a coleta e concentração de zircão e o resultado de nossos estudos confirma que a técnica de datação U-Pb pode ser aplicada com sucesso em terrenos profundamente decompostos.

INTRODUCTION Previous studies have shown that the metamorphosed volcanic and sedimentary rocks in the Serra dos Carajás region are at least as old as the Trans-Amazonian Cycle (2,100-2,000 Ma). Model ages ranging from 2,700-2,900 Ma have been obtained by K-Ar and Rb-Sr methods for rocks from Serra do Tapirapé (Tassinari & Basei 1980), Serra do Inajá, Salobo, and the Maroni-Itacaiúnas region (Tassinari *et al.* 1982, Cordani *et al.* 1984). The Grão-Pará Group has generally been considered Early Proterozoic. Gomes *et al.* (1975) obtained ages of 1,900-740 Ma from metavolcanics in the Grão Pará Group using the K-Ar method. The K-Ar data indicated that substantial argon loss had occurred in some samples, and Gomes *et al.* suggested a minimum age of 1,900 Ma for the group.

Previous geochronological studies in the Serra dos Carajás region have primarily made use of the Rb-Sr and K-Ar methods. These isotopic systems are susceptible to resetting at low to medium metamorphic grades, and tend to record younger tectonic and thermal events. Metamorphism associated with the Trans-Amazonian cycle was sufficiently intense over most of the Amazonian Craton to reset these isotopic systems. The uranium and lead isotopic system is much less susceptible to resetting by metamorphism (e.g. Peucat *et al.* 1985) and can provide more accurate crystallization ages.

U-Pb isotopes in zircons derived from weathered saprolites of rhyolites in the Grão-Pará Group conclusively show that the lower metavolcanic sequence, the lowest unit in the group is Archean in age. This age probably applies to the Carajás Formation iron deposits immediately overlying the metavolcanic unit. Considering the evidence that the Grão-Pará volcanics were erupted onto older continental crust (as discussed in Gibbs *et al.* 1986), these isotope data confirm the presence of even older Archean continental crust in the Central Brazilian Shield. The Grão-Pará Group is underlain by rocks that have features in common with high-grade gneiss terranes, greenstone belts, and rifted continental crust.

SAMPLING Zircons were sampled from outcrops of saprolite along roads, streams, and adjacent to the quarry. Approximately 30-50 kg of saprolite were collected at each location. Caution was taken during sampling to insure that the saprolites were clean and that they were entirely composed of the product of in-situ weathering of local bedrock. All exposed surfaces at the outcrop were carefully cleaned, and root holes, fractures, and geologic contacts

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were avoided during sampling.

The samples were disaggregated with a disc grinder set at 1-2 mm. Clays were removed by washing in a bucket prior to the pre-concentration of zircons by panning. Zircons were separated from the other heavy minerals in the panned concentrates by settling in heavy liquids, magnetic separation, and hand picking. The nearly pure zircon fractions were further subdivided according to magnetic susceptibility and size.

ANALYTICAL PROCEDURES Low contamination methods for the digestion of zircon, extraction of U and Pb, and determination of U and Pb concentrations by isotope dilution (Krogh 1973) were used in this study. Isotopic analyses were made using a 9 inch radius, Nier-type mass spectrometer at the University of New Hampshire. Each isotopic ratio presented below is the average of greater than 20, repeated, 16 seconds counting cycles. The data have been statistically cleaned to eliminate cycles deviating more than two standard deviations from the mean. In most cases, enough counting cycles were measured to reduce the mean standard deviation to less than 0.5%. A lead blank for the digestion, dilution, separation, and loading procedures contained 5 ng of Pb contamination. This is comparable to previous lead blanks measured in the same laboratory, and is probably due to contamination during loading (Gibbs & Olszewski 1982). Decay constants used in determining ages are those of Steiger & Jäger (1977). The original thorium concentrations were estimated from the ^{208}Pb concentrations, apparent lead losses, and U-Pb upper intercept ages. A more detailed description of the analytical procedures used in this study are presented in Wirth (1986).

ZIRCONS Zircons were collected from two separate exposures of weathered rhyolite in the lower metavolcanic sequence of the Grão-Pará Group along the road between Serra Norte airport and the Parauapebas River (Fig. 1). One sample (GB-123) is from a massive flow unit at least 10 m in thickness, and contains conspicuous phenocrysts of quartz and strongly altered plagioclase. The other sample (GB-50) is from a fragmental unit that consists of angular blocks (2-50 cm diameter) of rhyolite in a medium-grained matrix of tuffaceous material.

The zircons range from clear and light pink to yellowish brown in color, and average 0.06-0.25 mm in length. Typical length to width ratios range from 1:1 to 4:1. Although many grains appear equant and sub-rounded at low magnification, they are actually bounded by well-defined crystallographic faces (Fig. 2). No relict zircon cores or overgrowths are present, but growth zoning is evident. The zircons are interpreted to have crystallized directly from the felsic magma, rather than having been inherited from older crustal materials.

Zircons were also collected from granite saprolite exposed adjacent to the quarry south of savanna N2. These zircons are different from those in the weathered rhyolites, and are characterized by elongate, well-faceted and terminated crystal forms (Fig. 3). Some of the zircons are translucent and light pink, while others are opaque and white.

RESULTS Analytical results for zircons from rhyolites in the Grão-Pará Group are given in tables 1 and 2. All of the samples have isotopic compositions that plot below the concordia line (Fig. 4), indicating the loss of some radiogenic

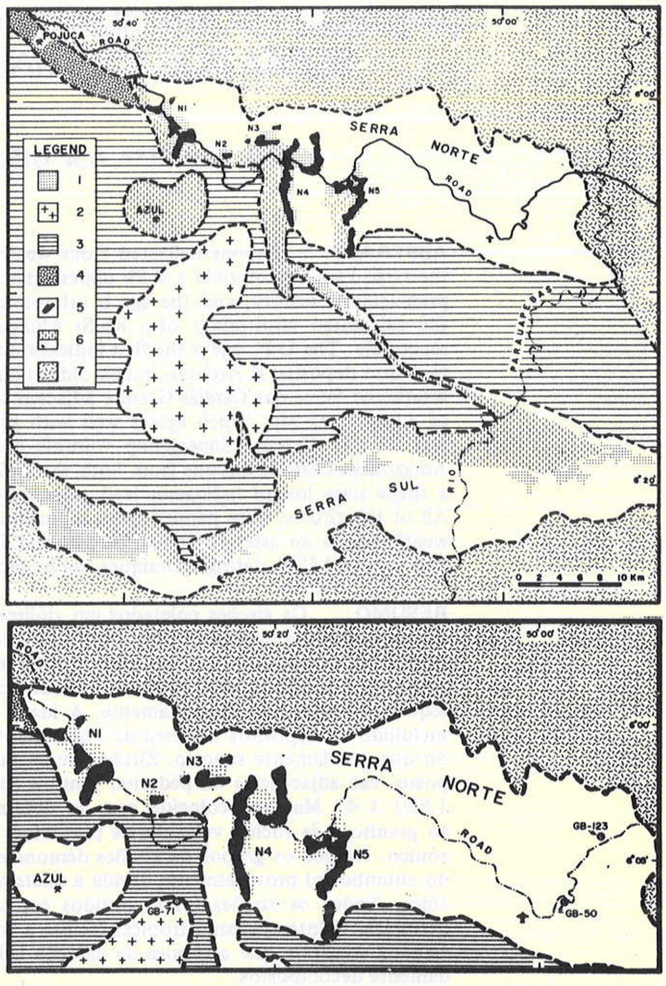

Figure 1 – a) Generalized lithologic map of Serra dos Carajás. Key: 1 – savannas; 2 – Serra dos Carajás Granite; 3 – sandstone and siltstone; 4 – Pojuca Sequence; 5 – Carajás Formation, Grão-Pará Group; 6 – Grão-Pará Group: Lower Metavolcanic Sequence (unpatterned), Upper Sequence (vertical dashes); 7 – Xingu Complex. b) Map showing the location of zircon samples in Serra Norte, Serra dos Carajás. Sample locations are shown with open circles; all other symbols same as figure 1a

lead. Sample 1e was not included in the calculation of the regression line because of its low $^{206}Pb/^{204}Pb$ ratio and large statistical error. The six remaining rhyolite samples define a chord (mean weighted standard deviation, MWSD = 4.65) with an upper intercept age of 2,758 ± 39 Ma (1σ), and a lower intercept age of 50 ± 121 Ma. Although the zircon samples are from two different rhyolite units, their U and Pb concentrations and calculated Th/U ratios are very similar. When the zircon data for each rhyolite are plotted on separate concordia diagrams, they have similar upper and lower intercept ages. The addition of sample 1e to the regression calculation changes the upper intercept age to 2,759 ± 33 Ma (1σ) and lowers the MWSD to 3.74. Together, the zircons define a chord that could be interpreted in terms of a Mesozoic to Recent single-stage lead-loss event, which might have occurred due to weathering or dilatancy during uplift (Stern *et al.* 1966, Goldich & Mudrey 1975).

Zircons from the Serra dos Carajás Granite have much higher U concentrations, and these vary directly with

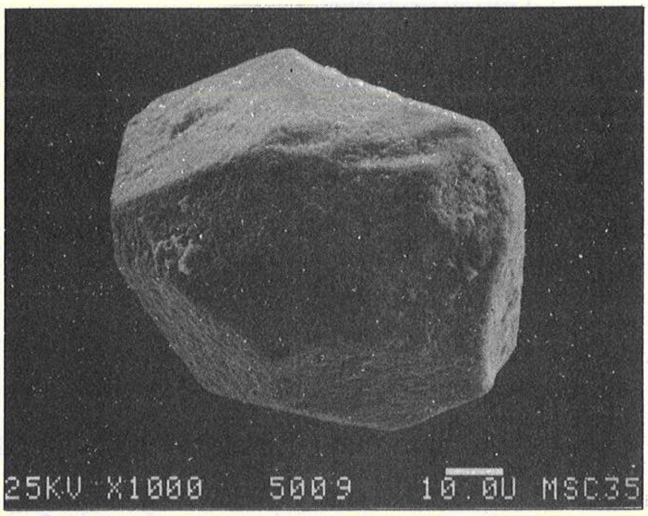

Figure 2 – Scanning electron photomicrograph of zircon from weathered rhyolite of the Grão-Pará Group. Scale bar in lower right corner of figure is 10 microns. Small pits (approximately 1 micron in diameter) on the surface of the grain are similar to pits observed in other saprolite zircons and have been interpreted as solution pits formed during weathering (Gibbs & Olszewski 1982)

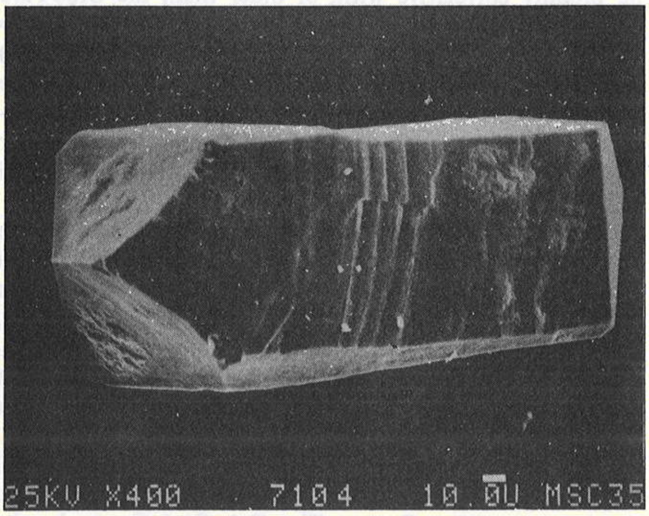

Figure 3 – Scanning electron photomicrograph of zircon from weathered Serra dos Carajás Granite. Scale bar in lower right corner of figure is 10 microns

magnetic susceptibility (Table 3). X-ray diffraction studies indicate a large 2θ reflection angle for the (112) planes in the opaque and white zircons, indicating that they are very metamict (Hurley & Fairbairn 1953). Metamict zircons, having undergone atomic disordering by radioactive decay, are more susceptible to lead loss during weathering and low-grade metamorphism. One zircon split contains a high percentage of white and opaque metamict zircons (split 1a, sample GB-71; Tables 3 and 4; Fig. 5). The analysis of this split lies above the lead-loss chord defined by the other samples. Presumably, these metamict zircons lost some radiogenic lead during an earlier event that did not affect the less metamict zircons. The metamict split was not included in the calculation of the regression line for this reason.

The three remaining samples define a chord (MWSD = 0.222) with an upper intercept age of 1,820 ± 56 Ma and a lower intercept age of 151 ± 58 Ma. All of the samples have discordant apparent ages, possibly indicating a single-stage lead-loss event similar to the one described above for zircons from the rhyolites.

Table 1 – U-Pb isotope analyses of zircons from rhyolites in the Grão-Pará Group

Sample	Split*	Weight (mg)	Pb (ppm)	U (ppm)	Pb loss (%)	Original U (ppm)	Original Th (ppm)	Original Th/U
50 1a	0° M > 200	2.6	57	140	39	228	149	0.67
1b	1° M > 200	4.9	57	139	40	226	155	0.70
1c	2° M > 200	6.0	59	155	45	252	180	0.73
1d	1° M 200–270	8.5	50	150	51	244	167	0.70
1e	2° M > 200 (E)	1.5	59	121	27	197	133	0.69
123 2a	1° M (W)	3.1	57	146	42	238	156	0.67
2b	0° M > 200	3.3	63	203	53	331	197	0.61

* Top number: degrees side tilt on Franz Magnetic Separator; bottom number: U.S. Standard Sieve sizes; M = magnetic fraction; NM = non-magnetic fraction; E = euhedral grains only; W = whole fraction. Pb loss estimated from discordance of points on concordia diagram. Original Th: estimated Th molar concentrations calculated from loss-corrected ^{208}Pb; age 2.760 Ga. Measured lead contamination 204/206/207/208 = 1/19.49/16.30/40.33.

Table 2 – U-Pb isotope ratios measured in zircons from rhyolites in the Grão-Pará Group

Sample	$^{204}Pb/^{206}Pb$	Corrected ratios* $^{207}Pb/^{206}Pb$	$^{208}Pb/^{206}Pb$	$^{206}Pb/^{238}U$	$^{207}Pb/^{235}U$
50 1a	0.003284 ±0.000020	0.192387 ±0.001192	0.265925 ±0.002725	0.326833 ±0.001904	8.6693 ±0.0742
1b	0.001630 ±0.000099	0.187236 ±0.001923	0.271973 ±0.004445	0.329077 ±0.002691	8.49513 ±0.11183
1c	0.001723 ±0.000023	0.188987 ±0.000802	0.283651 ±0.016023	0.300282 ±0.002319	7.82426 ±0.06936
1d	0.000649 ±0.000044	0.189722 ±0.001024	0.274594 ±0.002227	0.26687 ±0.00153	6.9807 ±0.0553
1e	0.005449 ±0.000178	0.191151 ±0.003834	0.274551 ±0.009003	0.389776 ±0.010255	10.294 ±0.341
123 2a	0.001963 ±0.000029	0.191149 ±0.000863	0.266145 ±0.001738	0.312165 ±0.006166	8.22695 ±0.16689
2b	0.002155 ±0.000178	0.194156 ±0.003336	0.242430 ±0.007643	0.250563 ±0.001208	6.70732 ±0.11987

* Isotopic ratios: lower number refers to standard error of mean. Isotopic composition of common lead same as in table 1.

DISCUSSION The physical and isotopic properties described above, for zircons from rhyolites of the Grão-Pará Group, support the interpretation that the upper intercept age (2,758 ± 39 Ma) represents the age of rhyolite volcanism. This age is older than was previously suspected for the Grão-Pará Group and it verifies the presence of Archean crust in the region. Although there have been some previous Archean age determinations in the region, they are based on relatively less reliable K-Ar and Rb-Sr whole-rock and model ages. This U-Pb age for rhyolites in the Grão-Pará

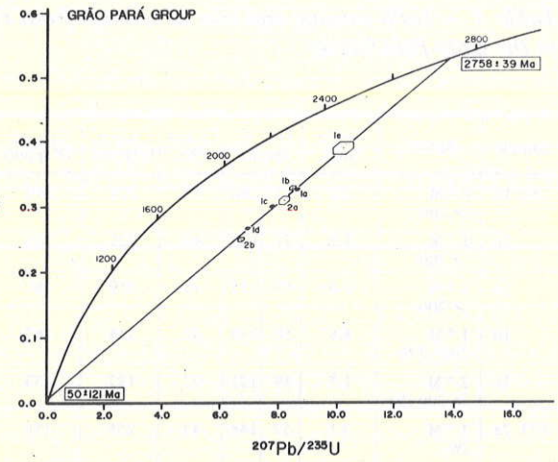

Figure 4 – Concordia diagram for zircons from rhyolites of the Grão-Pará Group at Serra dos Carajás. Sample 1e, with a large statistical error, was not included in the calculation of the regression line. The intercept ages and standard errors (1 σ) were calculated using the method of York (1969)

Table 3 – U-Pb isotope analyses of zircons from the Serra dos Carajás Granite

Sample	Split*	Weight (mg)	Pb (ppm)	U (ppm)	Pb loss (%)	Original U (ppm)	Original Th (ppm)	Original Th/U
71 1a	2 °M > 80 (W)	13.8	191	1496	76	2431	1116	0.47
1b	2 °M 80–140	10.0	118	816	71	1329	613	0.47
1c	2 °M > 80 (P)	12.4	93	724	75	1185	533	0.46
1d	1 °M	3.85	128	670	57	1091	427	0.40

* Top number: degrees side tilt on Franz Magnetic Separator; bottom number: U.S. Standard Sieve sizes; M = magnetic fraction; NM = non-magnetic fraction; P = pink zircons; W = white and opaque zircons. Pb-loss estimated from discordance of points on concordia diagram. Original Th: estimated Th molar concentrations calculated from loss-corrected ^{208}Pb; age 1.82 Ga. Isotopic composition of common lead same as in table 1.

Table 4 – U-Pb isotope ratios measured in zircons from the Serra dos Carajás Granite

Sample	Corrected ratios* $^{204}Pb/^{206}Pb$	$^{207}Pb/^{206}Pb$	$^{208}Pb/^{206}Pb$	$^{206}Pb/^{238}U$	$^{207}Pb/^{235}U$
71 1a	0.000329 ±0.000009	0.098206 ±0.000321	0.131796 ±0.000532	0.121661 ±0.000455	1.64730 ±0.00833
1b	0.000714 ±0.000093	0.104247 ±0.001590	0.144534 ±0.003861	0.135101 ±0.000577	1.94180 ±0.03082
1c	0.000448 ±0.000128	0.103060 ±0.002150	0.136665 ±0.005255	0.11984 ±0.000492	1.70285 ±0.03626
1d	0.001368 ±0.000092	0.107290 ±0.001637	0.137017 ±0.003962	0.179002 ±0.000953	2.64789 ±0.04285

* Isotopic ratios: lower number refers to standard error of the mean. Isotopic composition of common lead same as in table 1.

Group also agrees within the calculated uncertainty with a whole-rock Rb-Sr isochron age (2,687 ± 54 Ma) for basalts in the Grão-Pará Group (Gibbs *et al.* 1986). The analytical uncertainties are too large to detect any systematic differences in the isotopes or ages of the two rhyolite units analyzed.

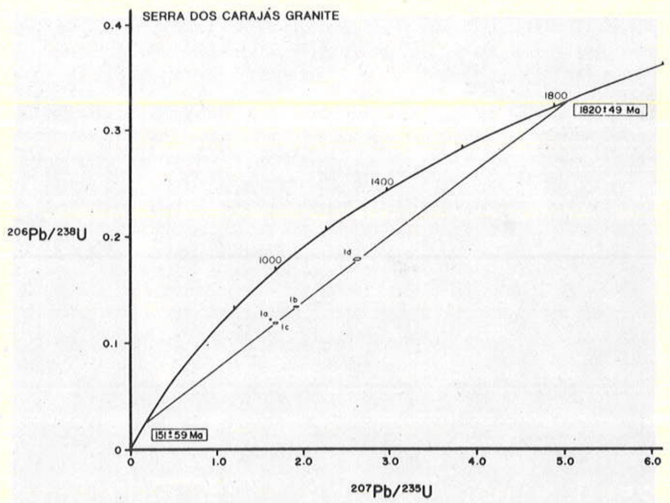

Figure 5 – Concordia diagram for zircons from the Serra dos Carajás Granite. The intercepts and standard errors (1 σ) were calculated using the method of York (1969). Sample 1a contains a high proportion of metamict zircons and was not included in the calculation of the regression line

A whole-rock Rb-Sr reference isochron for the more intensely metamorphosed Salobo formation, which may be broadly correlative with, or older than, the Grão-Pará Group, yielded a model age of 2,700 ± 150 Ma (IR = 0.717) (Tassinari *et al.* 1982). Cordani *et al.* (1984) suggested a model age of 2,900 Ma (IR = 0.710) for the same rocks at Salobo. Gneisses at Serra do Inajá, approximately 300 km south of Serra dos Carajás, give a Rb-Sr whole-rock model age of 2,750 Ma (IR = 0.701) (Tassinari *et al.* 1982).

There are few other Archean radiometric ages in the Amazonian Craton. The Imataca Complex, in eastern Venezuela, yielded an age of > 3.6 Ga by whole-rock U-Pb and Pb-Pb methods (Montgomery & Hurley 1978), and shows evidence of a metamorphic event at approximately 2.7 Ga. Montgomery (1977) interpreted an Archean age for the Imataca banded iron formations as well as for the gneisses.

The Archean age of the Grão-Pará Group has several important implications for Precambrian crustal evolution in the Amazonian Craton. The Grão-Pará Group basalts are similar to modern continental basalts (Gibbs *et al.* 1986), suggesting that they were erupted on an even older continental basement. The surrounding Xingu Complex and low-grade greenstone belts may be part of this basement, and thus may be considerably older than the Grão-Pará Group.

Recent isotopic investigations have shown that the apparently ensimatic greenstone belts in the northern Guiana Shield were formed at about 2.3-2.1 Ga, without evident contribution of older continental crust (Klar 1979, Gibbs & Olszewski 1982, Gruau *et al.* 1985). The geology of the northern Guiana Shield thus contrasts sharply with the Late Archean continental-type volcanism observed in the Serra dos Carajás region. A fundamental age boundary must separate the two terranes, and some workers (Tassinari *et al.* 1982, Cordani *et al.* 1984) have suggested that it lies just north of Serra dos Carajás. Gibbs (1986) proposed that this boundary might be located in northern Amapá based on the differences between the belts in Amapá and those occuring in the northern Guiana Shield. Additional dating of rocks in northern Pará and Amapá is needed to determine the location of this boundary.

The Archean age of the Grão-Pará Group might also have some important implications for models of deposition of banded iron formations. Many large iron formations, including the Imataca Complex in Venezuela (Montgomery 1977), the Nova Lima Group (Dorr 1973), Hamersley Basin of Australia (Richards & Blockley 1984), and the Liberian of West Africa (Cahen *et al.* 1984) have also been shown to be of comparable Archean age. Together, these iron formations account for much of the total of global Precambrian iron resources.

The Early Proterozoic U-Pb zircon age (1,820 ± 56 Ma) for the Serra dos Carajás Granite agrees well with some published K-Ar and Rb-Sr age determinations (Silva *et al.* 1974, Gomes *et al.* 1975, Tassinari *et al.* 1982, Cordani *et al.* 1984), and is considered to be the age of crystallization of the granite. This age is common among the felsic to intermediate composition volcanics and shallow intrusives of the Uatumã Supergroup exposed across much of the Amazonian Craton. The age of the granite provides a minimum age for the intruded sandstones adjacent to the quarry at Serra dos Carajás.

The lower intercept ages for both the rhyolites and the granite are similar within the large calculated uncertainties, and can be explained by single-stage lead-loss during the Mesozoic or Cenozoic. These results are similar to those observed by Klar (1979) and Gibbs & Olszewski (1982) in the Guiana Shield. Regional uplift in the Mesozoic could account for lead-losses due to decompression and dilatancy of the zircons, as suggested by Goldich & Mudrey (1975). It is also possible that the lead-loss occurred due to weathering, which may have begun as early as the Mesozoic. Two of the zircon samples were collected within a few tens of meters of the southern Pará peneplain, which forms the summits of the Carajás range. This surface is considered to date from the pre-Cretaceous (Boaventura 1974).

This study once again demonstrates that zircons panned from saprolites are well-suited for isotopic dating, as shown previously by Gibbs & Olszewski (1982). The ease of sample collection and zircon pre-concentration in the field make this method a useful tool for geological investigations in deeply weathered and remote field areas.

CONCLUSIONS U-Pb analyses of zircons from weathered metarhyolites of the Grão-Pará Group indicate a crystallization age of 2,758 ± 39 Ma and verify the presence of Archean crust in the Serra dos Carajás region. U-Pb analyses of zircons from saprolite in the Serra dos Carajás Granite near Serra Norte indicate a crystallization age of 1,820 ± 49 Ma. This age agrees well with previously determined ages for the granite and for the Uatumã Supergroup. The age of the Serra dos Carajás Granite provides a minimum age for the sandstones it intrudes near Serra Norte. This study confirms that zircons collected from weathered bedrock can provide reliable and accurate U-Pb dates.

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