LEACHING TECHNIQUE AND PARTIAL DIGESTION USING MICROWAVE OVEN: TREATMENT PROCEDURES FOR U-Pb DATING OF METAMICTIC AND OVERGROWN ZIRCONS

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THE ANALYTIC METHOD

The leaching technique was used to treat metamict zircons showing overgrowth rims and high uranium concentration. Petrographic studies using cathodeluminescence (CL) and transmitted light (TL) was carried out in order to evaluate the method efficiency. Crystals (around 10) of the same typology were photographed. These crystals, previously washed with HNO3, were then put in a savillex cup and attacked by an with 600µl of concentrated HF. The savillex cup was placed inside a microwave oven at temperatures not higher than 130 ^{o}C and 4 cycles of 90 minutes of heating and 10 minutes in stand-by (oven turned off) were applied in order not to overload or damage the microwave. After 360 minutes of heating, less than 5% of the acid solution evaporated from the savillex cup. With a micropipette the acid solution containing Pb and U leached from zircon rims was carefully separated from the remaining crystals and transported to another clean savillex cup, to which ^{205}Pb spike and H_3PO_4 were added. HF was completely evaporated on a hot plate, remaining only H_3PO_4 and leached material at the bottom of the cup.

To the first savillex containing partially leached zircons a reload of acid solution containing 600 µl of concentrated HF was again added and the whole procedure was repeated for 4 times (I, II, III and IV).

Before the final chemical attack, the grains were recounted and photographed. The dissolution using HF and HNO_3 took place in a digestion bomb with a special stainless steel protector placed inside a resistive oven kept at 180 ^{o}C during 3 days for a complete chemical digestion of the zircons. Pb and U were purified using the ion exchange column. The resulting H_3PO_4 solution containing Pb and U was loaded with silicagel on the Re filament and the isotopes ratio were measured by Finnigan MAT 262 Thermal Ionization Mass Spectrometer (TIMS).

DESCRIPTION OF THE SAMPLES

For the proposed experiment, 3 zircon fractions were selected from the two different zircon typologies observed in the leucosome veins present in the Atuba migmatites from Atuba quarry (north of Curitiba). These rocks are located in the Atuba Complex - SE Brazil.

- Group I: coarse-grained zircons, of average sizes between 200 and 400 µm x 130 µm, prismatic habit, relatively homogeneous, soft brown in reflective light and dark in transmitted light.

- Group II: Similar to group I in habit and in size but with intense metamictization. The grains are dark brown and inhomogeneous in reflective light and opaque in transmitted light

Figure 1 illustrates the above-mentioned characteristics using cathodeluminescence images (CL). The CL images show grains generally homogeneous, but locally with hints of oscillatory zoning parallel to the grain boundaries. There are some recrystallisation domains which appear bright and homogeneous in the images. All SHRIMP analyses show high U content (3000 – 7000 ppm, Sato et al., 2003), with variable and generally high common Pb content. The Fig. 2 show TeraWasserburg concordia diagram from SHRIMP analysis. One zircon yielded the oldest ^{207}Pb/^{206}Pb date of 795 ± 15 Ma. A group of four analysis yielded a weighted mean ^{207}Pb/^{206}Pb date of 573 ± 55 Ma with 96.5% confidence and MSWD of 0.4.

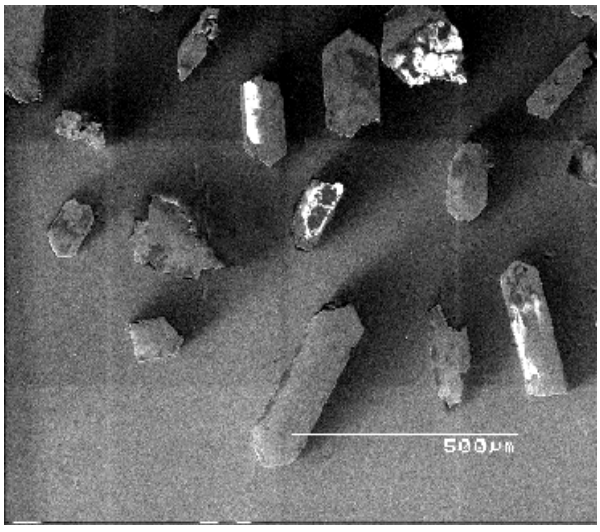

Figure 1. Cathode luminescence image.

DISCUSSION OF THE RESULTS

The grains were completely destroyed after 2 leaching phases of 6 hour cycles for each phase (Fig. 3).

It is possible to see in the Fig. 4a that U content goes down in the exponential form. These results may to indicate a preferential U leaching or of a possible higher U concentration in the zircon rim. The total amount of Pb and U extracted for the 4 leaching stages exceed 85% for Pb (Fig 4b). The values of ^{207}Pb/^{206} x ^{238}U /^{206}Pb ratios of the 3 initial phases plot on a straight line that intercepts

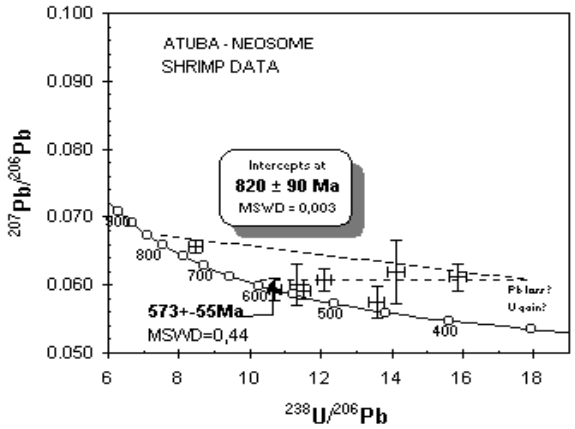

Figure 2. TeraWasserburg diagram. SHRIMP zircon analyses.

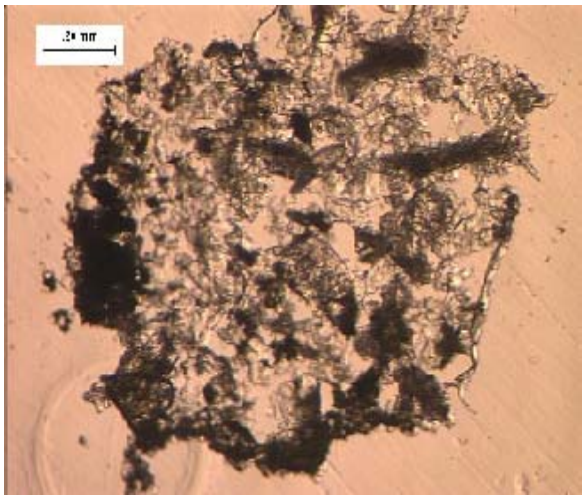

Figure 3. Leach residue after IV step. The zircons are completely destroyed in very small fragments.

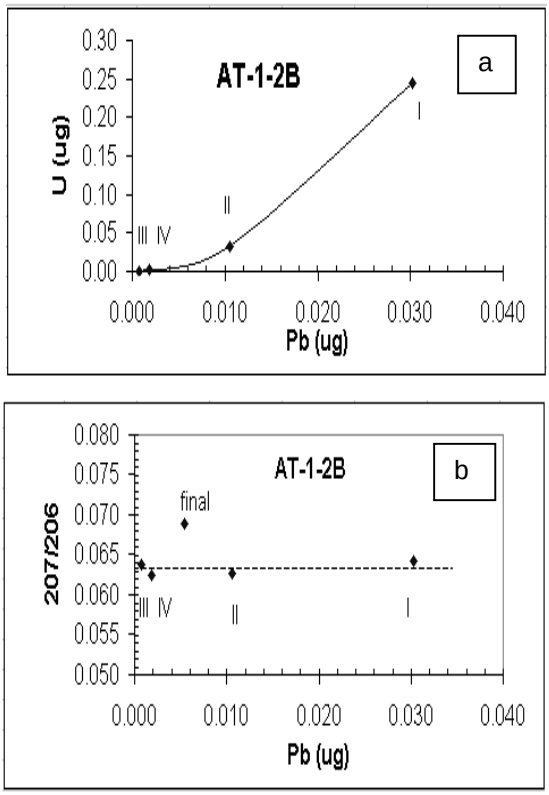

Figure 4. a) The U content decrease relative to Pb in the exponential mode from leach step I to IV; b) The fig. 3b shows the ratio $^{207}Pb/^{206}Pb$, with Neoproterozoic age, no change to 4 initial phases but the final residue data indicate Paleoproterozoic inherited component.

the concordia curve between 700 and 800 Ma (Figs 5 a, b, c, d, e, f). This value is very close to the zircon age obtained by SHRIMP (Fig. 2, $^{207}Pb/^{206}Pb$ age of 795 ± 15 Ma). The large age variation are due: very large amounts of common Pb, intense metamictization, continuous lead loss and also mixture age between inherited zircon (around 1700 Ma, Fig. 6) and the Neoproterozoic overgrowths around 570 Ma (SHRIMP ages; Sato et al., 2003). In addition for each leaching step it also is possible to see high U for step 1 (normal discordance) and decreasing to low U content for step IV (reverse discordance).

The final leaching fractions (attack in the bomb) yielded the U/Pb age (upper intercept) of 1635 ± 25 Ma (Fig. 6). These values registered in the residue material may reflect a mixed age with major contribution from inherited Paleoproteozoic material.

FINAL CONSIDERATIONS

The partial chemical attack using HF and HNO_3 was done in savillex cup and microwave oven. Normally the dissolution occur only in the zircon rim due low temperature (< 130 ^{o}C), but highly metamictic zircons the chemical digestion was very strong (>85%). The initial leaching fractions indicate Neoproterozoic ages (~ 750 Ma). These ages are probable a mixture between the Neoproterozoic recrystallized grain and the Paleoproterozoic inherited material that was evidenced in the final residues after the dissolution in the bomb.

REFERENCE

Sato, K; Siga Jr., O; Nutman, A. P.; Basei, M.A.S.; McReath, I.; Kaulfuss, G., 2003. The Atuba Complex. Southern South American Platform: Archean Components and Paleoproterozoic to Neoproterozoic Tectonothermal Events. Gondwana Research, 6 (2):.251-263.

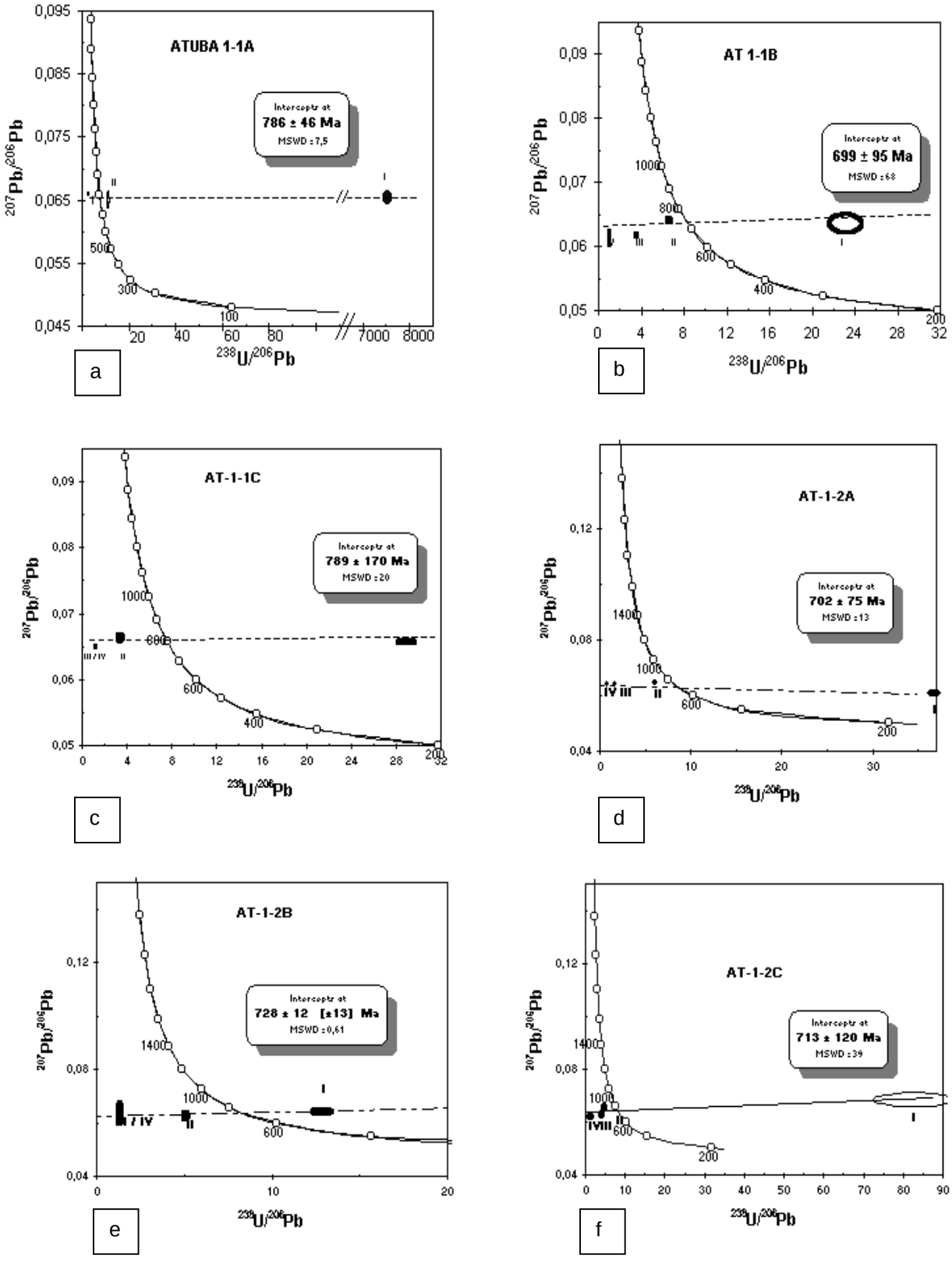

Figure 5. TeraWasserburg diagram. The group I correspond for diagrams a, b and c and group II correspond to diagrams d, e and f. The Pb-Pb ages for the first four leach steps, varied between 700 and 790 Ma for different fractions,but is relatively constant for the leaches into same fraction.

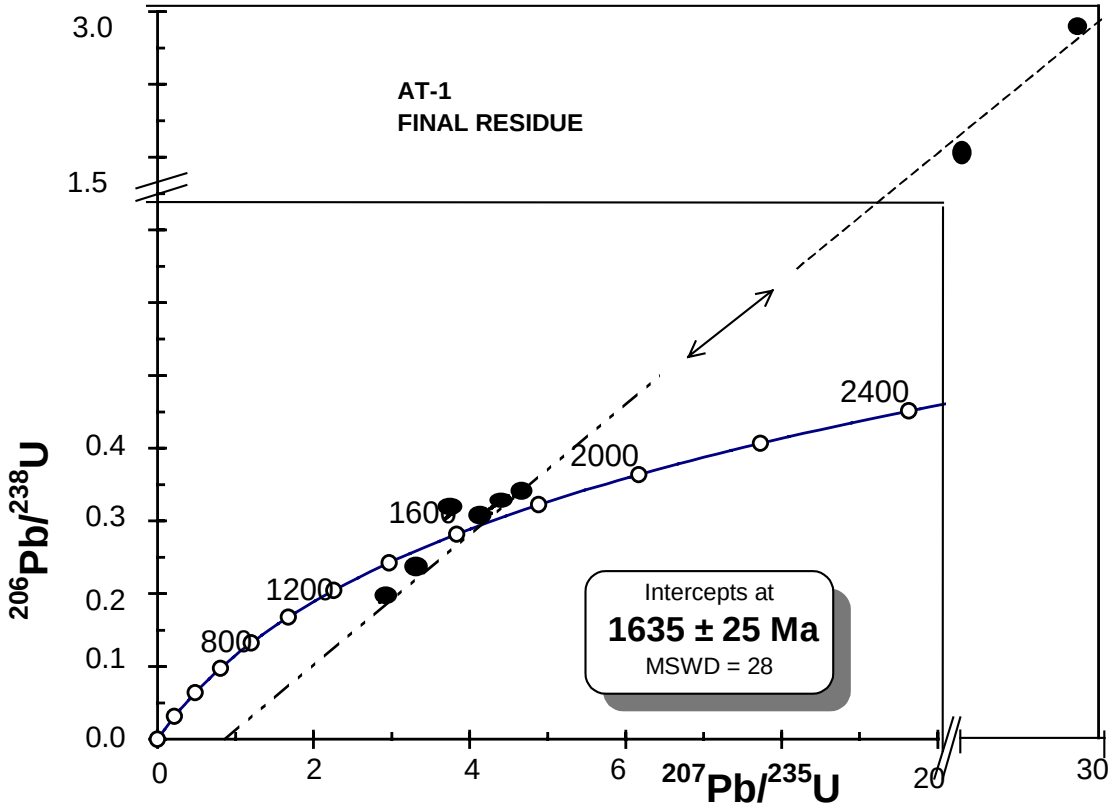

Figure 6. Concordia diagram for final residue. The upper intercept indicate 1635 ± 25 Ma. Some groups indicates reverse discordance that may be due preferential U removing during the first leach step or due a possibility of the migration these element from core to rim during zircon recrystallization during Neoproterozoic thermal event.