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Research paper

High-reliability zircon separation for hunting the oldest material on Earth: An automatic zircon separator with image-processing/microtweezers-manipulating system and double-step dating

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ABSTRACT

Despite the recent development in radiometric dating of numerous zircons by LA-ICPMS, mineral separation still remains a major obstacle, particularly in the search for the oldest material on Earth. To improve the efficiency in zircon separation by an order of magnitude, we have designed/developed a new machine – an automatic zircon separator (AZS). This is designed particularly for automatic pick-up of 100 μm -sized zircon grains out of a heavy mineral fraction after conventional separation procedures. The AZS operates in three modes: (1) image processing to choose targeted individual zircon grains out of all heavy minerals spread on a tray, (2) automatic capturing of the individual zircon grains with micro-tweezers, and (3) placing them one-by-one in a coordinated alignment on a receiving tray. The automatic capturing was designed/created for continuous mineral selecting without human presence for many hours. This software also enables the registration of each separated zircon grain for dating, by recording digital photo-image, optical (color) indices, and coordinates on a receiving tray. We developed two new approaches for the dating; i.e. (1) direct dating of zircons selected by LA-ICPMS without conventional resin-mounting/polishing, (2) high speed U-Pb dating, combined with conventional sample preparation procedures using the new equipment with multiple-ion counting detectors (LA-MIC-ICPMS). With the first approach, Pb-Pb ages obtained from the surface of a mineral were crosschecked with the interior of the same grain after resin-mounting/polishing. With the second approach, the amount of time required for dating one zircon grain is ca. 20 s, and a sample throughput of >150 grains per hour can be achieved with sufficient precision (ca. 0.5%).

We tested the practical efficiency of the AZS, by analyzing an Archean Jack Hills conglomerate in Western Australia with the known oldest (>4.3 Ga) zircon on Earth. Preliminary results are positive; we were able to obtain more than 194 zircons that are over 4.0 Ga out of ca. 3800 checked grains, and 9 grains were over 4300 Ma with the oldest at 4371 ± 7 Ma. This separation system by AZS, combined with the new approaches, guarantees much higher yield in the hunt for old zircons.

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1. Introduction

Zircon geochronology is a tremendous addition to the panoply of research techniques in the investigation of igneous, sedimentary and metamorphic rocks. In addition to the latest highly-tuned Nano secondary ion mass spectrometer (Nano-SIMS) and sensitive high-resolution ion-microprobe (SHRIMP) instruments, well-developed laser ablation inductively-coupled plasma mass spectrometry (LA-ICPMS) has dramatically enhanced the determination of mineral/rock ages, and the ability to handle numerous mineral grains (e.g., [lizuka and Hirata, 2004](#)); these new techniques have changed the style of research in geosciences in the last decade. Despite such improvements in dating techniques *per se*, mineral separation still remains a major obstacle because it requires enormous amounts of time and human effort. Crushing rocks, sieving, panning, and heavy liquid/magnetic treatments are the conventional procedures for mineral separation, and the hardest part is the final hand-picking of individual grains under the microscope. When we look for rare material, such as the oldest zircon on Earth, efficient and accurate separation of mineral grains is essential but may be difficult, even for well-trained researchers who may misidentify minerals and overlook small-sized targets.

In order to enhance the efficiency in mineral separation by an order of magnitude, and to emancipate many researchers from painstaking, time-consuming work, we have designed and developed a new separation system specifically customized for zircons, i.e. an automatic zircon separator (AZS). This AZS machine is designed for the automatic separation of 100 μm -sized individual zircon grains from a condensed mineral fraction after conventional separation procedures. We have modified a micromanipulator machine originally designed for picking up microscopic objects and relevant image-processing software, and thus have assembled a proto-type AZS ([Fig. 1A](#)) to test its practical efficiency.

The preliminary results are positive, as we have been able to reliably separate particular target minerals with this new machine. For a preliminary test, we analyzed the heavy mineral fraction of the well-known 3.0 Ga conglomerate from the Narryer gneiss complex in Western Australia, which contains the oldest zircons (>4.3 Ga) ever reported on Earth ([Wilde et al., 2001](#)). Still under progress, this machine likely provides a promising new tool for all

zircon seekers. This article briefly reports the results of our first attempt. More detailed descriptions and geological implications of our new Hadean zircons will be reported in a sister article elsewhere by S. Yamamoto and others.

2. Automatic zircon separator (AZS)

2.1. Basic concept

The new zircon separation system (AZS; [Fig. 1A](#)) operates in three functions: i.e. (1) optical recognition of targeted mineral grains on a tray with randomly scattered heavy minerals, (2) mechanical capturing of individual grains one by one, and (3) placing and aligning the captured grains on a receiving tray with registered coordinates. The first function is an application of pre-existing image-processing software, which we have further customized it for a particular mineral (e.g. zircon) by adding more specific constraints on optical characteristics (color, transparency, reflectance etc.) and the external morphology of mineral grains on the basis of our empirical knowledge of hand-picking procedures. The second and third functions also come from the modification of the micromanipulator, which is designed for capturing microscopic small-objects with a built-in digital microscope connected to a monitor screen of a desktop computer and with a mechanical micro-tweezer (vacuum-driven fine tube) ([Fig. 1B](#)). For the mechanical motion of the AZS machine and its procedure, readers may find a good analogy in the so-called “crane game machine” for capturing reward bunny-dolls in amusement parks, although obviously the size is larger.

2.2. Specification

The basic framework of the AZS machine is a micromanipulator system (Axis Pro™, MicroSupport Co.), which was primarily designed for picking up small objects grain-by-grain with a semi-manual control on a monitor screen. Users can manipulate the tip of the tweezers in enlarged images on the monitor screen. For capturing/releasing grains, a vacuum tweezer is used as an interlocking device ([Fig. 1B](#)). To complement these mechanical

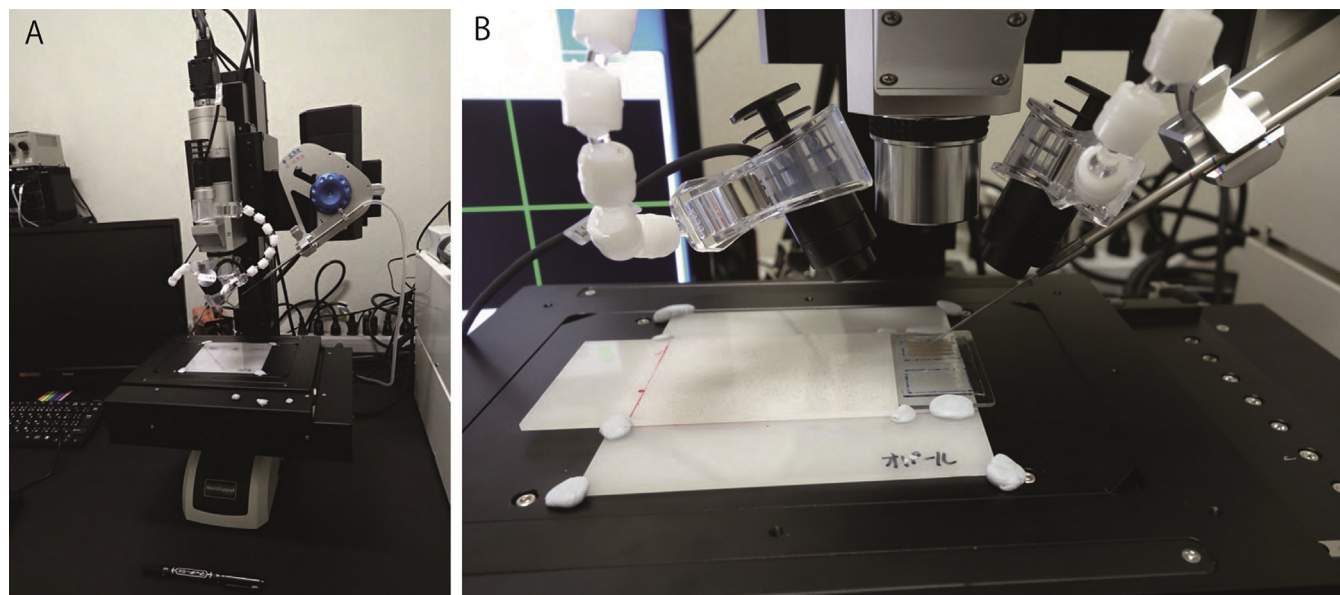


Figure 1. The automatic zircon separator (AZS). (A) The main body of the machine with an arm of microtweezers, (B) enlarged image of the microtweezers.

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