SUPPLEMENTARY MATERIAL

Crust-mantle interaction and craton destruction: evidence from Late Mesozoic plutons in the North China Craton

Analytical methods

Sampling and petrography

Polished thin sections were prepared for petrographic and mineral chemical studies at the University of Tsukuba, Japan. Photomicrographs of representative samples are shown in Fig. 4. Mineral assemblages and abundance of minerals in granitoids and MMEs are summarized in Supplementary Table 1. Mineral chemical analyses were carried out using an electron microprobe analyzer (JEOL JXA8530F) at the Chemical Analysis Division of the Research Facility Center for Science and Technology, the University of Tsukuba. The analyses were performed under conditions of 15 kV accelerating voltage and 10 nA sample current, and the data were regressed using an oxide-ZAF correction program supplied by JEOL. Representative compositions of minerals in the analyzed samples are given in Supplementary Tables 2 to 6.

Whole-rock major and trace element analyses

Fresh MME and granitoid samples were selected for whole-rock geochemical analyses. The samples were reduced in a jaw crusher, and then manually fine-powdered in an agate mortar. Major oxides were analyzed by 'lithium metaborate/tetraborate fusion ICP whole rock (Code 4B)', and minor and trace element by 'trace element ICP/MS (Code 4B2)' techniques at Activation Laboratories of Ontario, Canada. Fused samples were diluted and analyzed by Perkin Elmer Sciex ELAN 6000, 6100 or 9000 ICP/MS. Three blanks and five controls (three before sample group and two after) are analyzed per group of samples. Duplicates are fused and analyzed every 15 samples. Instrument is recalibrated every 40 samples. Detection limits of major elements are 0.01%, where those of REE are 0.002-0.05 ppm. Detailed analytical conditions and detection limits are summarized in http://www.actlabs.com/. The geochemical data are summarized in Supplementary Material.

Zircon separation involved gravimetric and magnetic techniques from crushed rock samples, followed by hand picking under a binocular microscope at the Yu'neng Geological and Mineral Separation Survey Centre, Langfang city, Hebei Province, China. Zircons were imaged under both transmitted and reflected light, and were imaged using cathodoluminescence (CL) to study the morphology and internal structures, and to choose target sites for U–Pb analyses. The CL imaging at the Beijing Geoanalysis Centre used scanning electron microscope (JSM510) equipped with Gantan CL probe. Individual grains were mounted onto double-sided adhesive tape and enclosed in epoxy resin discs. The discs were polished to a certain depth and gold coated for CL imaging and U–Pb isotope analysis.

U-Pb dating and trace element analysis of zircon were simultaneously conducted by laser ablation–inductively coupled plasma–mass spectrometry (LA-ICP-MS) at the Wuhan Sample Solution Analytical Technology Co., Ltd., Wuhan, China. Detailed operating conditions for the laser ablation system and the ICP-MS instrument and data reduction are the same as description by Zong et al. (2017). Laser sampling was performed using a GeolasPro laser ablation system that consists of a COMPexPro 102 ArF excimer laser (wavelength of 193 nm and maximum energy of 200 mJ) and a MicroLas optical system. A 70 mJ laser energy was used in this study. An Agilent 7700e ICP-MS instrument was used

to acquire ion-signal intensities. Helium was used as carrier gas and argon as make-up gas and mixed with the carrier gas via a T-connector before entering the ICP. A "wire" signal smoothing device is included in this laser ablation system (Hu et al., 2015). The spot size and frequency of the laser were set to 32 μm and 8 Hz respectively in this study according to the size and the U content of the zircon. Each analysis incorporated a background acquisition of approximately 20-30 s followed by 50 s of data acquisition from the sample. Zircon 91500 and glass NIST610 were used as external standards for U-Pb dating and trace element calibration, respectively. Time-dependent drifts of U-Th-Pb isotopic ratios were corrected using a linear interpolation (with time) for every six analyses according to the variations of the 91500 zircon standard and preferred U-Th-Pb isotopic ratios used for 91500 are from Wiedenbeck et al. (1995). The weighted mean $^{206}Pb/^{238}U$ age for standard for 91500 is 1062.4 $\pm$ 3.9 Ma (n = 48, MSWD = 0.00041). Zircon standards GJ-1 (Jackson et al., 2004) and Plešovice (Sláma et al., 2008) were used as unknown samples to monitor the stability and accuracy of acquired U-Pb data. The obtained concordia U-Pb ages of GJ-1 (weighted mean $^{206}Pb/^{238}U$ age = 602.1 $\pm$4.5 Ma, n = 8, MSWD = 0.022) and Plešovice (weighted mean $^{206}Pb/^{238}U$ age = 338.1 $\pm$ 4.4 Ma, n = 8, MSWD = 1.9) are consistent within error with the recommended values. However, the uncertainties of standards could be overestimated according to the very low MSWD of weighted mean age from 91500 standards. An Excel-based software ICPMSDataCal (ver. 10.7) was used to perform off-line data reduction and integration of background and analyzed signals, time-drift correction and quantitative calibration for trace element analysis and U-Pb dating (Liu et al., 2008, 2010). Final assessment of uncertainty and data visualization were done in Excel™ using Isoplot/Ex_ver4.15 (Ludwig, 2012). All ages are reported as concordia ages (Ludwig, 1998). Errors are quoted at the 2σ level. Full isotopic data and details of the analytical method can be found in Supplememntary Table 8. Trace element composition analysis of zircon was calibrated with Si as internal standard. Except for those elements with low concentrations (e.g., La, Pr and/or Nd), the results of trace elements in GJ-1 are consistent within error with the recommended values.

Lu–Hf analyses of zircon were conducted at the same domains or immediately adjacent domains where the U–Pb data were analyzed with MC–ICP-MS) coupled with a GeoLas 2005 laser ablation system (Lambda Physik, Germany) at the State Key Laboratory of Geological Process and Mineral Resources, China University of Geosciences, Wuhan. A Neptune Plus MC–ICP-MS instrument was used to acquire ion-signal intensities, with a beam diameter of 44 μm and using a typical laser fluence of ~ 5 J/cm^2 and a repetition rate of 8 Hz. 91500 and Mud Tank was used as standard for U–Pb dating, and a pair of each were measured during every 4 analyses. The detailed analytical procedure and correction for interferences are similar to those described by Wu et al. (2008).The $^{176}Hf/^{177}Hf$ ratios of the standard zircon (Mud Tank) during analysis were 0.282506 $\pm$ 15 (2σ, n = 18). It is almost identical to the values based on LA-MC-ICP-MS analyses, which are 0.282504 $\pm$ 44 (2σ, n = 158; Woodhead and Hergt, 2005). All the Lu–Hf isotope analysis results are reported with an error of 2σ. The $^{176}Hf/^{177}Hf$ at time of crystallization is calculated from measurement of present-day $^{176}Hf/^{177}Hf$ using a decay constant of ^{176}Lu of $1.865 \times 10^{-11}year^{-1}$ was adopted (Scherer et al., 2001; Söderlund et al., 2004). Initial $^{176}Hf/^{177}Hf$ ratios and εHf(t) values were calculated with reference to the chondritic uniform reservoir (CHUR) of Blichert-Toft and Albarède (1997) at the time of zircon growth from the magma. Single-stage Hf model age (T$_{DM}$) was calculated with respect to the depleted mantle with present-day $^{176}Hf/^{177}Hf$ = 0.28325 and $^{176}Lu/^{177}Hf$ = 0.0384 (Griffin et al., 2000). Two-stage Hf model age (2s T$_{DM}$) was calculated with respect to the average continental crust with a $^{176}Lu/^{177}Hf$ ratio of

0.015 (Griffin et al., 2002). The Lu–Hf isotopic data are summarized in Supplementary Material.

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Supplementary Fig. 1. Wetherill U-Pb concordia plots and weighted average plots for LA-ICP-MS Zircon U–Pb reference materials.