LETTER

K-Ar biotite and fission-track zircon ages of the Nisatai Dacite, Iwate Prefecture, Japan: A candidate for Tertiary age standard

TAKAHIRO TAGAMI¹, KOZO UTO², TAKAAKI MATSUDA³,
NORIKO HASEBE¹ and AKIKAZU MATSUMOTO²

¹Department of Geology and Mineralogy, Faculty of Science, Kyoto University, Kyoto 606, Japan

²Geological Survey of Japan, Higashi 1-1-3, Tsukuba 305, Japan

³Department of Geology, Faculty of Science, Himeji Institute of Technology, Himeji 671-22, Japan

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K-Ar biotite and fission-track (FT) zircon ages were determined on the Nisatai Dacite (ND), Iwate Prefecture, Japan, to assess its suitability for a geologic age standard. The concentration of potassium and argon isotope ratios were determined on a biotite sample twice and three times, respectively, and yielded reproducible results within analytical errors. A K-Ar age was given as 21.0 ± 0.3 (2 sigma) Ma. FT zircon ages were determined on two samples from different localities as 21.8 ± 1.4 (2 sigma) and 23.9 ± 1.4 Ma. The K-Ar age was obtained from an outcrop adjacent to that for the former FT age, with a good agreement between the two methods. The concordance in age, in conjunction with previous zircon FT length data, suggests a potential applicability of the ND to both the K-Ar and FT system calibration in Cenozoic time.

INTRODUCTION

Analysis of age standards provides an important baseline for reliable interlaboratory comparison of radiometric age data (e.g., Alexander and Davis, 1974; Hurford and Green, 1983). In particular, age standards are routinely employed for the $^{40}\text{Ar}/^{39}\text{Ar}$ and fission-track (FT) methods, both of which involve neutron irradiation processes in a nuclear reactor and accordingly require precise assessment of neutron dose. Repeated measurements of a set of standards varying in age and other characteristics also provide various information about the reproducibility and accuracy of age determination at individual laboratories. Such a procedure was formally commended to standardize the FT system calibration by the I.U.G.S. Subcommittee on Geochronology (Hurford, 1990).

Several age standards have been successfully used for the system calibration as well as for the

interlaboratory comparison of radiometric ages: e.g., P207 muscovite (Lanphere and Dalrymple, 1967); Bern 4B biotite and 4M muscovite (Flish, 1982); Fish Canyon Tuff biotite, sanidine, apatite and zircon (Naeser *et al.*, 1981; Cebula *et al.*, 1986); and MMhb-1 hornblende (Samson and Alexander, 1987). As a working standard for Ar analysis in Japan, the Sori granodiorite biotite of ~91 Ma was collected from the geochemical standard JG1 locality (Ando *et al.*, 1975) and has been widely used (e.g., Uchiumi and Shibata, 1980; Matsumoto *et al.*, 1989). As was discussed in detail elsewhere (Uto *et al.*, 1994), the precise age determination of Neogene to Quaternary rocks is highly important for reconstructing various geologic processes at active continental margins, particularly volcanic and tectonic histories. In this regard, it is strongly needed to establish a set of K-Ar and FT age standards from such young rocks by detailed intercalibration between the two techniques. The aim of this study is to characterize a

candidate for such standards, i.e., the Nisatai Dacite (ND), which contains a sufficient amount of fresh, euhedral biotite and zircon crystals suitable for K-Ar and FT analyses, respectively. Note that the suitability of the ND to a FT zircon standard has also been suggested through track length analyses (Matsuda, 1992; Hasebe *et al.*, 1994; Yamada *et al.*, 1995).

SAMPLE PREPARATION AND ANALYTICAL PROCEDURES

The Nisatai Dacite (ND) is a dacitic volcanic formation that occurs in and around Ninohe city, Iwate prefecture, Japan. The ND consists primarily of dacitic welded tuffs and occupies the lowest horizon of the Miocene Ninohe Group, overlying the pre-Cenozoic basements (Samata, 1976; see also Otuka, 1934; Chinzei, 1966). A K-Ar age of 21.8 Ma was previously reported on the ND (Kimura, 1986), although no detailed information was given about the sampling localities and analytical procedures therein. The rock samples analysed in this study were collected from dacitic welded tuff outcrops, correlative to the upper part of the ND (Samata, 1976). The sampling sites (NST, 40°15.7' N, 141°20.9' E; NS-630, 40°17.3' N, 141°20.0' E; NS-704B, 40°15.7' N, 141°20.8' E; see Tables 1 and 2) are located to the west of the Mt. Oritsume, where the thickness of the formation reaches the maximum at around 200 m. The rock sample NST for K-Ar dating was collected from a fresh, large outcrop adjacent to NS-704B, one of the rock samples for which FT ages were measured prior to the sampling for K-Ar dating.

K-Ar dating

The NST sample contains a sufficient amount of fresh biotites suitable for K-Ar dating, which were separated by the following procedures. After crushing with a rock trimmer, a Jaw-crusher and a disk mill grinder, the sample was washed with water to remove fine rock powders. Subsequently, biotites were concentrated with bromoform heavy liquid solution, Frantz ferrofilter and Sumitomo

Table 1. K-Ar age determination on NST biotite from the Nisatai Dacite

Analysis No.	Sample wt. (g)	K ₂ O (%)	³⁸ Ar/ ³⁶ Ar	⁴⁰ Ar/ ³⁶ Ar	Atm. ⁴⁰ Ar (%)	Rad. ⁴⁰ Ar (10 ⁻⁶ ml STP/g)	Age (Ma)
#1	0.08896		0.1875 ± 0.0014	814.0 ± 2.2	36.3	5.732	21.07 ± 0.48
#2	0.04480	8.386, 8.366	0.1890 ± 0.0030	795.9 ± 3.6	37.1	5.715	21.01 ± 0.48
#3	0.04219		0.1902 ± 0.0026	867.8 ± 3.4	34.1	5.682	20.89 ± 0.48
	average	8.38 ± 0.08				weighted mean	20.99 ± 0.28
Air*			0.1869	295.5			

Samples were analysed by T. Tagami, N. Hasebe and K. Uto. Errors are ±2 sigma throughout.

*Isotopic ratios for air are from Krummenacher (1970).

Table 2. Analytical results of fission-track dating on zircon samples from the Nisatai Dacite

Sample name	<i>n</i>	$\rho_s (N_s)$ ($\times 10^6 \text{ cm}^{-2}$)	$\rho_i (N_i)$ ($\times 10^6 \text{ cm}^{-2}$)	$\rho_d (N_d)$ ($\times 10^5 \text{ cm}^{-2}$)	<i>T</i> (Ma)	$P(\chi^2)$ (%)	<i>r</i>
NS-630	17	3.21 (2469)	4.13 (3175)	1.67 (7054)	23.9 $\pm$ 1.4	5	0.961
NS-704B	11	2.94 (2133)	3.93 (2850)	1.58 (6690)	21.8 $\pm$ 1.4	25	0.977
weighted mean					22.8 $\pm$ 1.0		

n = number of crystals counted, ρ_s = spontaneous track density of a sample, N_s = number of tracks counted to determine ρ_s , ρ_i = induced track density of a sample measured in a muscovite external detector, N_i = number of tracks counted to determine ρ_i , ρ_d = induced track density of glass dosimeter NBS-SRM612 measured in a muscovite external detector, N_d = number of tracks counted to determine ρ_d , *T* = fission-track age with ± 2 sigma Poisson error, $P(\chi^2)$ = probability of obtaining the observed value of Galbraith's (1981) χ^2 parameter, for *N* degrees of freedom, where *N* = (number of crystals) - 1, quoted to the nearest 5% or 10%, *r* = correlation coefficient between ρ_s and ρ_i measured for individual crystals.

Samples were analysed by T. Matsuda.

TA-Neomax magnet, and then completely separated by a Frantz isodynamic magnetic separator and tapping technique. Finally, the biotite separates were sieved to serve the 42–60 mesh size grains for potassium and argon measurements.

The concentration of K in biotite was determined by the flame emission spectrometry using the Kotaki FIP-3D system, in which lithium internal standard and peak integration were adopted. The laboratory procedure for K analysis was described in detail elsewhere (Matsumoto, 1989). The isotope analyses of Ar were performed with a VG Isotopes 1200C mass spectrometer. We employed the peak height comparison method (Takaoka *et al.*, 1989), wherein the concentration of ^{40}Ar in sample is obtained by comparing peak intensities with the known amount of air standard. The laboratory procedures of Ar analysis adopted in this study were documented by Matsumoto *et al.* (1989).

Ages were calculated using the following constants and atmospheric initial $^{40}\text{Ar}/^{36}\text{Ar}$ ratio (i.e., 295.5): $\lambda_\beta = 4.962 \times 10^{-10} \text{ y}^{-1}$, $\lambda_e = 0.581 \times 10^{-10} \text{ y}^{-1}$, $^{40}\text{K}/\text{K} = 0.01167\%$ (Steiger and Jager, 1977).

Fission-track dating

A sufficient amount of euhedral zircons suitable for FT analysis were separated from the two rock samples of the ND, using the conventional heavy liquid and magnetic separation techniques. FT ages were determined by the external detector

method (e.g., Naeser, 1976), as will be briefly described below. After mounting onto a PFA teflon sheet, zircon crystals were ground then polished to expose internal surfaces. The mount was then etched with KOH:NaOH eutectic etchant (Gleadow *et al.*, 1976) at 225°C for ~20 hr, until spontaneous tracks were optimally revealed. An Indian Bihar muscovite sheet was firmly attached on the mount, and the samples were packed for irradiation between two NBS-SRM612 glass dosimeters. The neutron irradiation was performed for 30 min at the Irradiation Pit facility of the Musashi Institute of Technology reactor. In this facility, a nominal thermal neutron flux and a Cd ratio for Au monitor are $\sim 8 \times 10^{11} \text{ cm}^{-2}\text{s}^{-1}$ and 13–15, respectively (T. Honda, pers. commun., 1986). The external detector muscovite was etched with 46%HF at 20°C for 20 min to reveal induced tracks optimally. Tracks were counted throughout under a Nikon Biophot optical microscope with 100 $\times$ dry objective and 10 $\times$ eyepieces. FT ages were calibrated by the zeta calibration method (Hurford and Green, 1983) using a zeta factor of 365.8 ± 7.0 (2 sigma).

RESULTS AND DISCUSSION

Table 1 presents the analytical results of K-Ar dating on the NST biotite sample. The potassium content and argon isotope ratios were determined twice and three times, respectively, on individual

aliquots of the sample. The repeated determinations of potassium show a good agreement, giving an average of $8.38 \pm 0.08\%$ (errors for all data are consistently given at ± 2 sigma hereafter) as K_2O . Meanwhile, the argon isotope ratios are also reproducible at $\sim 5.7 \times 10^{-6}$ ml STP/g. A weighted mean age for the NST biotite sample is thus given as 21.0 ± 0.3 Ma.

FT zircon ages were determined as 23.9 ± 1.4 and 21.8 ± 1.4 Ma on samples NS-630 and NS-704B, respectively, showing a reasonable concordance with each other (Table 2). The latter FT age also shows a good agreement with the K-Ar age, both of which were obtained from outcrops adjacent to each other (see their sampling sites and explanations). The concordant results between the two methods suggest a potential applicability of the ND to both the K-Ar and FT system calibration. In addition to these age data, mean horizontal-confined-track (HCT) lengths are concordant between spontaneous and induced tracks in the NST zircon, and this confirms both the rapid cooling of the rock subsequent to its formation and the absence of effective thermal overprints (Yamada *et al.*, 1995). It is also remarked that the mean induced HCT length of NST zircon is similar to those of other natural zircons of different geneses (Hasebe *et al.*, 1994). These lines of evidence further support that the ND is a potential candidate for a geologic age standard.

With respect to stratigraphic implication, the present results accord with the regional existence of unconformity at about 19 Ma, covering nearly the whole of the Tohoku district (Kimura, 1986). In the Ninohe city area, the Yotsuyaku formation that includes the Kadonosawa fauna unconformably overlies the Nisatai formation (e.g., Otuka, 1934; Chinzei, 1966; Samata, 1976). The Kadonosawa fauna is an important age marker indicating the occurrence of ~ 15.5 Ma (Kimura, 1986; see also Samata, 1976). The Keiseitoge andesite, which has an interfingering relationship with the middle part of the Yotsuyaku formation, yields a K-Ar age of 15.9 Ma (Kimura, 1986). Hence the stratigraphic discontinuity at about 16–20 Ma is confirmed between the Nisatai dacite

and the Keiseitoge andesite, which probably represents the overall Yotsuyaku formation in age.

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