K-Ar biotite ages of the coarse-grained granites from the Inada area, Ibaraki Prefecture: Evaluation of suitability for a new K-Ar dating standard

Takahiro Muto^{†,‡}, Naoyoshi Iwata[†] and Kazuo Saito[†] (Received May 19, 2005)

Abstract

K-Ar ages have been determined on biotite fractions of three coarsegrained granites from the Inada area, Ibaraki Prefecture. Ages are 65.0 + / - 2.3 Ma for Inada-I, 63.7 + / - 2.2 Ma for Inada-II and 62.1 + / - 2.8 Ma for Inada-III. The Inada-II sample is a better candidate than the other two samples as a K-Ar dating standard on the basis of thin section observation and K-Ar dating results. However, a chlorite removal step is necessary to make the new dating standard.

1 Introduction

For K-Ar dating, various kinds of inter- and intra-laboratory dating standards have been used in order to check the reliability of dating procedures. It is important that the properties (age, mineral) of the standard samples are close to those of the unknown samples. However, no good K-Ar dating standards are available for late Cretaceous to early Tertiary samples.

The Inada area in Ibaraki Prefecture is a famous center for the production of granitic building stone in Japan, and so we can easily obtain fresh rock samples from the quarry. The age of this granitic rock was estimated to be late Cretaceous. Based on these advantages, the granitic rock is a good

[†]Department of Earth and Environmental Sciences, Faculty of Science, Yamagata University, Yamagata, 990–8560, Japan

[‡]Present address: Ibaraki Hitachi Information Service Co., Ltd.

candidate for a new dating standard. In this study, we collected the coarsegrained granite from quarries in the Inada area and measured the K-Ar ages to evaluate the suitability of the biotite fraction of the Inada granite as a new dating standard.

2 Geological background and sample descriptions

In the Tsukuba district, granitic rocks are intruded into surrounding sedimentary and metamorphic rocks. Detailed geological and petrographical investigations were performed by Takahashi (1982). A simplified geological map around the Inada area was modified from Takahashi (1982) and is shown in Fig. 1. He divided the granitic rocks into the following seven



Fig. 1. Simplified geological map of the northern Tsukuba area (modified after Takahashi, 1982) and the sampling sites for the K-Ar dating. 1. Coarse-grained granite, 2. Medium-grained granodiorite, 3. Fine-grained granodiorite, 4. Kabasan fine-grained granite, 5. Gabbroic rock, 6. Sedimentary rocks. The Yamanoo fine-grained granite, porphyritic granodiorite and two-mica granite are distributed outside of this map area.

bodies, namely coarse-grained granite, fine-grained granodiorite, mediumgrained granodiorite, Kabasan fine-grained granite, Yamanoo fine-grained granite, porphyritic granodiorite and two-mica granite. These seven bodies were continuously emplaced, and solidified during a short time interval of less than 3 Ma (Arakawa and Takahashi, 1989). The rocks from the Inada area belong to the coarse-grained granite group.

We collected three samples from the quarries in Inada Town at sample locations indicated in Fig. 1. Samples of Inada-I and Inada-III were obtained from working granite quarries, whereas Inada-II was collected from an old quarry where work has finished. About 20 kg of granitic rocks were collected from each location.

Sampling positions, modal compositions and relative abundances of the chlorite in the samples are tabulated in Table 1. The samples seem fresh, but biotite is partially chloritized as seen in thin section.

Sample No.	Location		Mode					chlorite
	Latitude	Longitude	qtz	pl	K-fel	bi	other	
Inada-I	N 36°22.5'	E 140°11.7'	++	+	+++	-		rich
Inada-II	N 36°20.5'	E 140°14.3'	++	-	+++	+		poor
Inada-III	N 36°22.8'	E 140°13.1'	++	-	+++	-		moderate

Table 1. List of samples for K-Ar dating

Abbreviations

qtz: Quartz, pl: plagioclase, K-fel: K-feldspar, bi: biotite +++: > 50%, ++: ~30%, +: ~10%, -: ~5%, --: < 3%

Samples for K-Ar dating were prepared by the following procedure. Roughly broken rock samples were crushed in a stainless-steel mortar and sieved to $0.15\sim0.25$ mm (60–100 mesh size) in diameter. The sieved fraction were washed in distilled water in an ultrasonic bath, and then dried overnight at 110°C in an oven. Subsequently, the biotite fraction was separated magnetically using a permanent magnet and a Franz type isodynamic magnetic separator. To separate biotite from other minerals, tapping was also carried out. Finally, samples were washed in acetone with ultrasonic treatment and sieved to $0.15\sim0.25$ mm again. The purity of the biotite fraction was about 97% after the above procedure.

3 K-Ar dating method

Potassium contents of samples were measured in Yamagata University by a flame photometer, FP-33D (Hekisa Kagaku), using a lithium internal Takahiro Muto, Naoyoshi Iwata, and Kazuo Saito

standard and the peak integration method. To verify the accuracy of potassium analyses, we performed measurements of standard samples (Geological Survey of Japan Geochemical Reference samples, Igneous rock series, JG-2 and HD-B1) and unknown samples at the same time. Measured potassium contents of the standard samples were 3.89 + / -0.45 wt. % for JG-2 and 7.93 + / -0.33 wt. % for HD-B1, which are consistent with the reference values (JG-2: 3.91 wt. %, Terashima et al., 1994; HD-B1: 7.956 wt. %, Hess and Lippolt, 1994) of the samples within the error range. This guaranteed the accuracy of our potassium measurements. The average potassium contents of the unknown samples of replicated analyses (n = 5) and their standard deviations were indicated as uncertainties in the measurement procedures.

Abundances of radiogenic 40 Ar were measured in Yamagata University by the isotope dilution method using a 38 Ar spike. Samples were degassed at 1500°C in a Mo crucible using a resistance furnace. Extracted gases were purified by two Ti-Zr getters and a Zr-Al alloy getter, and were then introduced into the mass spectrometer. Argon isotopes were analyzed by a 15 cm radius and 60° deflection sector type of mass spectrometer. To calculate the amount of radiogenic 40 Ar, corrections of mass discrimination and hot blank were carried out during the argon isotope analyses. The amount of 38 Ar in a spike is often calibrated with an international age standard, HD-B1 (Hess and Lippolt, 1994).

For K-Ar age calculation, the following constants, $\lambda_{\rm e} = 0.581 \times 10^{-10}$, $\lambda_{\beta} = 4.962 \times 10^{-10}$, ${}^{40}\text{K/K} = 0.01167\%$ (Steiger and Jaeger, 1977) were used. Uncertainty in a K-Ar age was calculated from the propagation of analytical errors in potassium and radiogenic ${}^{40}\text{Ar}$ contents (1 sigma level).

4 Results and Discussion

The results of K-Ar dating of Inada samples are shown in Table 2. The K-Ar ages of Inada-I, Inada-II and Inada-III are 65.0 + / - 2.3, 63.7 + / - 2.2, 62.1 + / - 2.8 Ma, respectively, and are within the error range.

K-Ar and Rb-Sr ages of plutonic and metamorphic rocks from the Tsukuba district have been reported in the literature (Kawano and Ueda, 1966, Shibata, 1968, Shiba et al., 1979, Arakawa and Takahashi, 1988, Shibata and Uchiumi, 1992), and a summary of these radiometric ages is given in Table 3. For plutonic rocks, radiometric ages range from 60 Ma to 65 Ma, while the K-Ar biotite ages of the granitic rocks around the Inada area were 64.5 Ma (Kawano and Ueda, 1966) and 61.4 Ma (Shibata, 1968). Our results are in good agreement with these previous works. A ⁴⁰Ar-³⁹Ar dating result (Total age = 59.9 Ma and Isochron age = 59.9 +/- 0.6 Ma,

Sample No.	K content	40 Ar/ 36 Ar	rad. ⁴⁰ Ar	A.C.	Age
-	(wt. %)		$(\times 10^{-5} \text{ cm}^{3}/\text{g})$	(%)	(Ma)
Inada-I	6.63 +/- 0.33	7068 +/- 644	1.68 +/- 0.01	4.2	64.2 +/- 3.2
		8767 +/- 529	1.72 +/- 0.01	3.4	65.7 +/- 3.2
Average					65.0 +/- 2.3
Inada-II	7.10 +/- 0.35	7478 +/- 327	1.80 +/- 0.01	4.0	64.1 +/- 3.1
		6206 +/- 478	1.77 +/- 0.01	4.8	63.2 +/- 3.1
Average					63.7 +/- 2.2
Inada-III	6.85 +/- 0.43	9872 +/- 876	1.68 +/- 0.01	3.0	61.9 +/- 3.9
		10935 +/- 444	1.68 +/- 0.01	2.7	62.2 +/- 3.9
Average					62.1 +/- 2.8

Table 2. K-Ar dating results of the Inada samples

N.B.

1) rad.⁴⁰Ar: radiogenic ⁴⁰Ar.

2) A.C.: air contamination ratio in the 40 Ar.

Table 3.	Radiometric	ages	of	plutonic	rocks	in	the	Tsukuba	district
rabie o.	reactioniconic	agos	O1	protonic	100100	***	0110	roundou	anounce

Rock type	Method	Material	Age	Reference
Coarse-grained granites	K-Ar	biotite	61.4(*)	2
Coarse-grained granites	K-Ar	biotite	64.5(*)	1
Coarse-grained granites	K-Ar	hornblende	64.0 +/- 2.1	6
Coarse-grained granites	K-Ar	K feldspar	61.9 +/- 1.9	6
Coarse-grained granites	Rb-Sr	minerals	60.2 +/- 0.4	5
Coarse-grained granites	Rb-Sr	whole rocks	61.6 +/- 4.3	5
Fine-grained granodiorite	K-Ar	biotite	60.4	1
Porphyritic granodiorite	K-Ar	biotite	52.9	3
Porphyritic granodiorite	K-Ar	biotite	62.9	3
Two-mica granite	K-Ar	biotite	60.4	1
Two-mica granite	K-Ar	biotite	60.4	1
Mafic inclusion of	K-Ar	hornblende	76.8 +/- 3.2	6
fine-grained granodiorite	10 20			
unknown	⁴⁰ Ar- ³⁹ Ar	whole rocks	59.9	4

References

1 Kawano and Ueda (1966)

2 Shibata (1968)

3 Shiba et al. (1979)

4 Ozima et al. (1979)

5 Arakawa and Takahashi (1988)

6 Shibata and Uchiumi (1992)

(*): K-Ar ages were recalculated using the constants of Steiger and Jaeger (1977).

whole rock) was obtained by Ozima et al. (1979). They showed a flat pattern in the age spectrum of an Inada sample. It means that no significant thermal disturbance occurred in the Inada area. Unfortunately, no sample and no detailed Ar isotope information were indicated in their paper. Thus, the relationship between our dating results and the ${}^{40}\text{Ar}$ - ${}^{39}\text{Ar}$ dating result is ambiguous.

Thin section observations indicated that the Inada samples contain different amounts of chlorite (Table 1). Chlorite was formed by the alteration of biotite with potassium being lost during the chloritization process (e.g. Eggleton and Banfield, 1985, Kogure and Banfield, 2000). Thus, decreasing potassium contents in the Inada samples equals to increasing amounts of chlorite. Since the Inada-II sample has the least amount of chlorite and the highest potassium content among the three samples, it is the most suitable candidate as a dating standard. However, even small amounts of chlorite bearing sample heterogeneity problems, and so K-Ar dating results of chloritebearing samples may be complicated. To make the Inada samples into a dating standard sample, another sample preparation process, which removes chlorite from biotite, is required.

5 Acknowledgement

This document is a summary of an undergraduate research project undertaken by the first author. We acknowledge Prof. Kazuo Nakashima, Dr. Masao Ban, Takeo Arai, Jun Takiguchi, Maki Syoji and Hitoshi Takahashi of the Department of Earth and Environmental Sciences, Yamagata University and Dr. Naoto Hirano of the Tokyo Institute of Technology who helped to carry out this study.

References

- [Arakawa, Y. and Takahashi, Y., 1988] Arakawa, Y. and Takahashi, Y., Rb-Sr ages of granitic rocks from the Tsukuba district, Japan, J. Mineral. Petrol. Econ. Geol., 83, 232-240, 1988.
- [Arakawa, Y. and Takahashi, Y., 1989] Arakawa, Y. and Takahashi, Y., Strontium isotopic and chemical variations of the granitic rocks in the Tsukuba district, Japan, Contrib. Mineral. Petrol., 101, 46-56, 1989.
- [Eggleton, R.A. and Banfield, J.F., 1985] Eggleton, R.A. and Banfield, J.F., The alteration of granitic biotite to chlorite, Am. Mineral., 70, 902-910, 1985.

- [Hess, J.C. and Lippolt, H.J., 1994] Hess, J.C. and Lippolt, H.J., Compilation of K-Ar measurements on HD-B1 standard biotite 1994 status report, in Phanerozoic Time Scale, G.S. Odin, Ed., Paris, 19-23, 1994.
- [Kawano, Y. and Ueda, Y., 1966] Kawano, Y. and Ueda, Y., K-A dating on the igneous rocks in Japan (IV)-Granitic rocks in northeastern Japan-, J. Mineral. Petrol. Econ. Geol., 56, 41-55, 1966 (in Japanese with English abstract).
- [Kogure, T. and Banfield, J.F., 2000] Kogure, T. and Banfield, J.F., New insights into the mechanism for chloritization of biotite using polytype analysis, Am. Mineral., 85, 1202-1208, 2000.
- [Ozima, M., Kaneoka, I., and Yanagisawa, M., 1979] Ozima, M., Kaneoka, I., and Yanagisawa, M., Temperature and pressure effects on ⁴⁰Ar-³⁹Ar systematics, Earth Planet. Sci. Lett., 42, 463-472, 1979.
- [Shiba, M., Ueda, Y., and Onuki, H., 1979] Shiba, M., Ueda, Y., and Onuki, H., K-Ar ages of metamorphic rocks from the Tsukuba district, Ibaraki Prefecture, J. Mineral. Petrol. Econ. Geol., 74, 122-125, 1979 (in Japanese with English abstract).
- [Shibata, K., 1968] Shibata, K., K-Ar age determinations on granitic and metamorphic rocks in Japan, Geol. Surv. Japan, Report, 227, 1-71, 1968.
- [Shibata, K. and Uchiumi, S., 1992] Shibata, K. and Uchiumi, S., K-Ar age results -4- New data from the Geological Survey of Japan, Bull. Geol. Surv. Japan, 43, 359-367, 1992 (in Japanese with English abstract).
- [Steiger, R.H. and Jaeger, E., 1977] Steiger, R.H. and Jaeger, E., Subcommission on geochronology: convention on the use of decay constants in geo- and cosmochronology, Earth Planet. Sci. Lett., 36, 359-362, 1977.
- [Takahashi, Y., 1982] Takahashi, Y., Geology of the granitic rocks in the Tsukuba area, J. Geol. Soc. Japan, 88, 177-184, 1982 (in Japanese with English abstract).
- [Terashima, S., Imai, N., Itoh, S., Ando, A., and Mita, N., 1994] Terashima, S., Imai, N., Itoh, S., Ando, A., and Mita, N., 1993 compilation of analytical data for major elements in seventeen GSJ geochemical reference samples, "Igneous rock series", Bull. Geol. Surv. Japan, 45, 305-381, 1994.