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TECHNICAL NOTE

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$^{40}Ar/^{39}Ar$ age determination using ARGUS VI multiple-collector noble gas mass spectrometer: performance and its application to geosciences

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Abstract

Background: $Ar/^{39}Ar$ dating technique has been used to determine the age for low-temperature geological event. The introduction of the multiple collectors and the improvement in sensitivity in the noble gas mass spectrometry enable the single-grain $^{40}Ar/^{39}Ar$ age determination.

Findings: The protocol for $^{40}Ar/^{39}Ar$ age determination and performance of the new high-sensitivity noble gas mass spectrometer (ARGUS VI) combined with the infra-red laser heating device are shown.

Conclusions: ARGUS VI can produce the precise single-grain $^{40}Ar/^{39}Ar$ age of 28.4 ± 0.5 Ma (1σ, MSWD = 138, *n* = 24) within the recommended values of Fisher Canyon sanidine (FCS) (28.294 ± 0.036 Ma). For the young samples, we can also get the age of 1.185 ± 0.004 Ma (1σ, MSWD = 6.05, *n* = 26) of Alder Creek sanidine (ACS), equivalent to the recommended age (1.193 ± 0.001 Ma).

Keywords: $^{40}Ar/^{39}Ar$ age; Noble gas mass spectrometer; Quaternary; Geochronology; CO_2 laser

Findings

Introduction

Recent development in mass spectrometry, such as the increase of sensitivity and the adoption of multiple collector system, enables to analyze the isotopic ratio for the single grain and spot analysis. Since the introduction of in-situ age dating for the small geological samples such as SHRIMP (sensitive high-resolution ion micro probe), the complex geological events can be revealed more precisely. The U-Pb spot age dating for zircon grains suggests the new concept of early Earth (e.g., Wilde et al. 2001) and provides the important keys to solve the juxtaposed geological events in a certain region (e.g., Cheong et al. 2014). However, to reveal the full history of a certain orogeny, it is indispensable to get the age information for the low- to mid-temperature geological activity. The Ar isotope system, whose closure temperatures range from ca. 130 to 690°C (e.g., Faure and Mensing 2005), can provide good clue to solve it. In addition, for the basaltic rocks with rare minerals adequate for U-Pb dating, the Ar ages have become the good proxy for the eruption age. For these purpose, K-Ar age dating method was introduced in early 50s and had provided age information related to various studies, such as the geomagnetic polarity timescale, one of the essential foundations of plate tectonic concepts (McDougall, 2014). However, the possible inhomogeneity due to the separate analyses of K and Ar and no information about the Ar-loss and/or Ar-gain after the closure in Ar isotope system should be considered for the proper interpretation of K-Ar ages. The $^{40}Ar/^{39}Ar$ technique, a variant of K-Ar dating system, has now commonly replaced the conventional K-Ar method to overcome these disadvantages. In addition, the introduction of laser heating/ablation technique enables to measure Ar age of single grains and monitor the variation in Ar isotope within minerals. In this technical note, we describe the $^{40}Ar/^{39}Ar$ age dating protocol using the newly installed multi-collector noble gas mass spectrometer in Korea Basic Science Institute (KBSI) and also report its performance for some geological samples.

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Principle of $^{40}Ar/^{39}Ar$ age determination

$^{40}Ar/^{39}Ar$ age dating is based on the same principle of K-Ar method, which uses the decay of ^{40}K to ^{40}Ar through the electron capture and positron emission. The concentration of parent ^{40}K is measured indirectly the neutron irradiation process which converts a part of ^{39}K to ^{39}Ar through the $^{39}K(n, p)^{39}Ar$ reaction. The constant $^{40}K/^{39}K$ ratio in samples enables to estimate the content of ^{40}K in samples through the measurement of ^{39}Ar, if we exactly know how many portions of ^{39}K in samples are converted to ^{39}Ar during the neutron irradiation. The $^{40}Ar/^{39}Ar$ age is calculated using the following formula (McDougall and Harrison 1999):

$$t = \frac{1}{\lambda} \cdot \ln\left(1 + J \cdot \frac{^{40}Ar^*}{^{39}Ar_K}\right)$$

where

$$J = \frac{^{39}K}{^{40}K} \cdot \frac{\lambda}{\lambda_e + \lambda_e'} \cdot \Delta T \cdot \int \phi(E)\sigma(E)dE$$

t age
λ total decay constant of ^{40}K (5.534×10^{-10}/year)

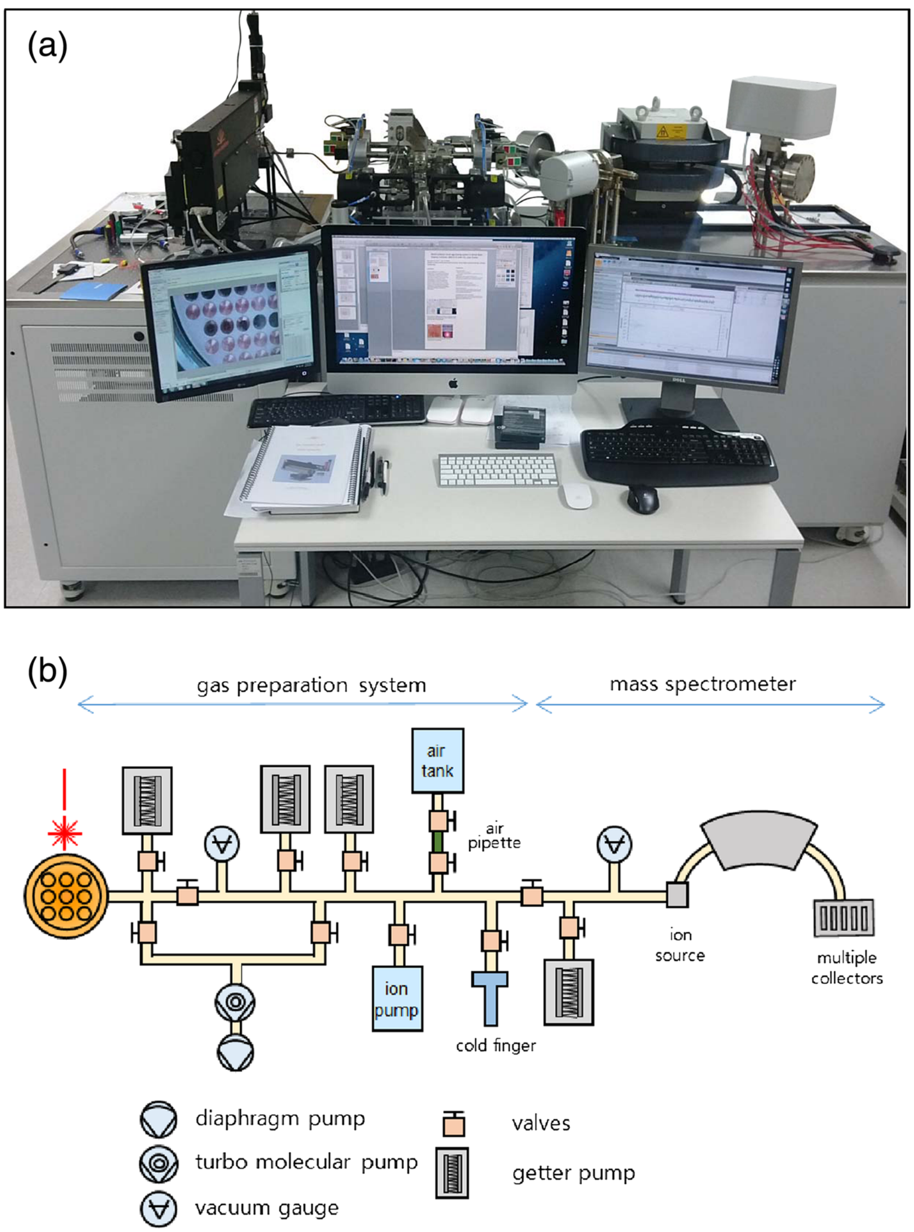

Figure 1 ARGUS VI system in KBSI. (a) Photo of whole $^{40}Ar/^{39}Ar$ age dating system and **(b)** schematic illustrations for the gas preparation system.

λ_e decay constant of ^{40}K to ^{40}Ar by electron capture (0.572×10^{-10}/year)
$\lambda_e{}'$ decay constant of ^{40}K to ^{40}Ar by emission of a positron (0.0088×10^{-10}/year)
$^{40}Ar^*$ radiogenic ^{40}Ar
$^{39}Ar_K$ ^{39}Ar derived from ^{39}K by the reaction with fast neutron
ΔT duration of irradiation
$\phi(E)$ neutron flux density at energy E
$\sigma(E)$ neutron capture cross section at energy E

As the precise values of $\phi(E)$ and $\sigma(E)$ are difficult to obtain, standard minerals of age-known are used to calculate J values using the following equation:

$$J = \frac{\exp(\lambda t) - 1}{^{40}Ar*/^{39}Ar_K}$$

During neutron irradiations, the isotopes of Ca, K, and Cl in the samples commonly produce interfering Ar isotopes through interactions of neutron. Reactor-derived ^{40}Ar from ^{40}K, and ^{39}Ar and ^{36}Ar from ^{42}Ca and ^{40}Ca, respectively, are corrected by analyzing K- and Ca-salts irradiated together with samples. For the detailed calculation for these interfering isotopes, refer to McDougall and Harrison (1999).

Availability and requirements

Outline of the Ar age dating system

$^{40}Ar/^{39}Ar$ age dating system in KBSI can be divided into the following three parts: (1) laser heating system, (2) gas preparation bench, and (3) high-sensitivity noble gas mass spectrometer (Figure 1).

Laser heating system

Laser heating system (Fusions 10.6, Photon Machines) consists of an integrated CO_2 laser source and beam-delivery system with CCD camera and sample chamber. The laser source generates the continuous CO_2 laser with the wavelength of 10.5 μm. Gantry type beam delivery system consists of three mirrors, one iris and one focusing lens. Whole laser delivery system also navigates 3-dimensionally over sample chamber. The intensity and beam size of laser including the movement of beam delivery system are controlled by the Chromium II software. The sample chamber is made up with a 114 mm diameter Conflat flange and differentially pumped ZnS viewport by turbomolecular pump. A copper sample holder of 43 mm diameter and 5 mm in thickness with 133 holes is loaded into sample chamber for single grain analysis. KBr glass covers the sample holder to prevent the spillover and scattering of sample from the hole during heating. After sample loading, sample chambers are heated by infrared lamp during several hours to achieve good vacuum level. Released gases from sample by laser beam diffuse from sample chamber into gas preparation system.

Gas preparation system

The outline of the gas preparation system is shown in Figure 1b. All pipe line is made from internally polished stainless steel. In order to purify argon from the extracted gases, three SORB-AC getter pumps (NP10) are used. They are constructed from a cartridge of getter material (ST101 alloy of zirconium with 16% aluminum) placed around an axial heater. At room temperature, these getters pump out hydrogen and carbon monoxide which are major background gases in the mass spectrometer.

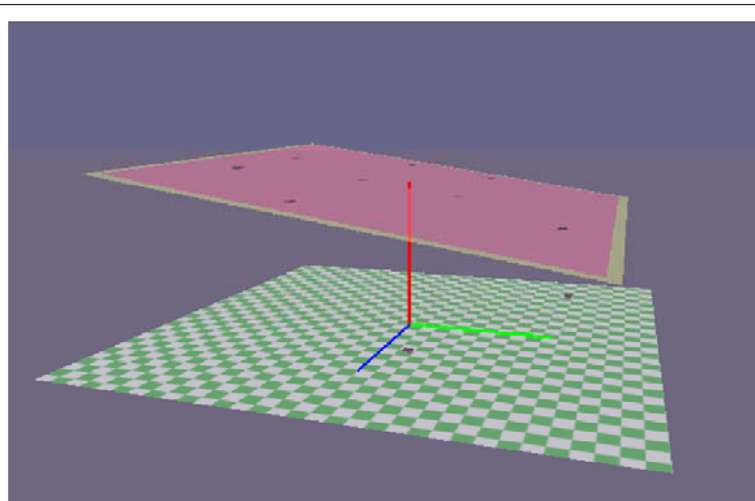

Figure 2 3-Dimensional variation in *J* values across one irradiation disk.

Table 1 Representative analysis of $^{40}Ar/^{39}Ar$ age for Fisher Canyon sanidine (FCS) and Alder Creek sanidine (ACS)

Relative isotopic abundances													Derived results				
Lab ID no.	*J* ($\times10^{-3}$)	±1σ	^{40}Ar	±1σ	^{39}Ar	±1σ	^{38}Ar	±1σ	^{37}Ar	±1σ	^{36}Ar	±1σ	^{39}Ar Mol $\times10^{-14}$	Ca/K	$\%^{40}Ar^*$	Age (Ma)	±1σ
FCS																	
84-02	0.2055	0.0003	56,989	8	748	3	57	3	22	2	1.27	0.09	0.025	0.161	99.4	27.83	0.10
84-03	0.2055	0.0003	96,025	11	1,239	3	87	3	26	2	1.77	0.09	0.041	0.116	99.5	28.32	0.06
84-04	0.2055	0.0003	79,364	11	1,033	7	65	9	9	3	0.56	0.14	0.034	0.050	99.8	28.17	0.20
84-05	0.2055	0.0003	36,927	10	478	7	29	9	7	3	0.14	0.14	0.016	0.087	99.9	28.32	0.42
84-06	0.2055	0.0003	70,660	11	913	7	49	9	10	3	0.87	0.14	0.030	0.065	99.6	28.34	0.22
87-01	0.1959	0.0003	60,413	8	759	3	44	3	14	3	1.99	0.08	0.025	0.100	99.0	27.61	0.10
87-02	0.1959	0.0003	63,977	16	583	3	30	3	0	3	2.47	0.09	0.019	0.000	98.9	33.90	0.18
87-03	0.1959	0.0003	54,528	7	686	3	49	4	11	3	0.83	0.08	0.023	0.091	99.6	27.72	0.14
87-05	0.1959	0.0003	103,304	9	1,264	3	77	2	0	2	3.63	0.09	0.042	0.000	99.0	28.32	0.08
87-06	0.1959	0.0003	46,783	10	581	7	36	8	4	3	0.54	0.13	0.019	0.043	99.7	28.10	0.32
87-07	0.1959	0.0003	35,618	9	445	7	33	8	1	4	0.58	0.13	0.015	0.011	99.5	27.91	0.42
87-09	0.1959	0.0003	78,316	11	969	7	55	8	8	3	1.29	0.14	0.032	0.045	99.5	28.17	0.19
93-01	0.1816	0.0001	63,628	8	724	2	37	3	5	2	1.79	0.09	0.024	0.038	99.2	28.28	0.10
93-02	0.1816	0.0001	127,664	12	1,453	3	70	4	9	3	1.53	0.10	0.048	0.034	99.7	28.41	0.07
93-03	0.1816	0.0001	108,347	10	1,228	3	65	4	7	2	0.59	0.09	0.041	0.034	99.8	28.59	0.08
93-04	0.1816	0.0001	124,168	11	1,424	3	78	4	14	3	2.15	0.09	0.047	0.056	99.5	28.16	0.07
93-05	0.1816	0.0001	51,898	7	595	3	37	3	1	2	1.06	0.08	0.020	0.007	99.4	28.16	0.15
93-06	0.1816	0.0001	100,604	10	1,167	3	77	3	13	3	0.95	0.09	0.039	0.064	99.7	27.90	0.08
93-07	0.1816	0.0001	81,501	9	935	5	52	3	3	3	0.99	0.08	0.031	0.019	99.6	28.18	0.14
93-08	0.1816	0.0001	108,398	9	1,225	5	72	3	51	2	1.04	0.09	0.041	0.233	99.7	28.65	0.11
93-09	0.1816	0.0001	100,317	10	1,154	3	78	3	8	3	3.84	0.09	0.038	0.037	98.9	27.91	0.09
93-10	0.1816	0.0001	120,283	10	1,386	3	96	3	29	2	0.72	0.08	0.046	0.119	99.8	28.13	0.06
93-11	0.1816	0.0001	89,456	9	1,022	3	77	3	25	3	2.80	0.09	0.034	0.139	99.1	28.16	0.08
93-12	0.1816	0.0001	141,018	12	1,631	4	92	3	16	2	0.31	0.08	0.054	0.056	99.9	28.05	0.06
														Average		28.38	0.47
ACS																	
75-01	0.2020	0.0003	11,629	11	3,238	6	184	6	18	5	3.14	0.14	0.108	0.023	92.0	1.176	0.006
75-03	0.2020	0.0003	4,741	5	1,419	4	76	3	14	2	0.27	0.09	0.047	0.047	98.5	1.169	0.008
75-04	0.2020	0.0003	11,887	4	3,330	5	166	3	45	3	2.44	0.10	0.111	0.066	94.0	1.195	0.004
75-05	0.2020	0.0003	11,502	6	3,112	4	172	4	5	3	3.72	0.11	0.103	0.007	90.3	1.189	0.004
75-06	0.2020	0.0003	12,491	5	3,668	3	198	3	15	2	0.90	0.10	0.122	0.020	97.9	1.186	0.003
75-08	0.2020	0.0003	16,880	19	3,793	7	215	6	17	6	14.91	0.30	0.126	0.022	73.5	1.170	0.010
79-01	0.1910	0.0002	13,275	12	3,623	6	245	6	22	5	1.48	0.14	0.120	0.026	96.8	1.194	0.005
79-02	0.1910	0.0002	13,908	12	3,866	6	209	6	25	5	0.39	0.14	0.128	0.027	99.3	1.202	0.004
79-03	0.1910	0.0002	25,059	10	6,147	5	382	3	41	4	11.51	0.15	0.204	0.033	86.3	1.187	0.003
79-04	0.1910	0.0002	17,716	6	4,857	5	275	3	37	4	1.96	0.10	0.161	0.037	96.8	1.189	0.003
79-05	0.1910	0.0002	14,645	5	3,944	4	241	3	17	3	2.89	0.11	0.131	0.021	94.2	1.177	0.003
79-06	0.1910	0.0002	7,592	5	2,118	5	130	5	14	4	0.63	0.10	0.070	0.032	97.6	1.178	0.006
79-07	0.1910	0.0002	11,883	6	3,176	5	175	3	19	3	2.72	0.09	0.105	0.029	93.2	1.175	0.004
79-08	0.1910	0.0002	10,674	5	2,921	3	207	3	18	2	1.68	0.10	0.097	0.031	95.4	1.173	0.004
79-09	0.1910	0.0002	7,120	6	1,973	3	117	4	18	4	0.33	0.10	0.066	0.043	98.8	1.200	0.006

Table 1 Representative analysis of $^{40}Ar/^{39}Ar$ age for Fisher Canyon sanidine (FCS) and Alder Creek sanidine (ACS) *(Continued)*

83-01	0.1788	0.0002	21,443	11	5,512	7	415	6	35	4	3.32	0.14	0.183	0.027	95.5	1.172	0.003
83-02	0.1788	0.0002	13,561	12	3,278	6	286	7	23	4	4.76	0.15	0.109	0.030	89.6	1.171	0.005
83-03	0.1788	0.0002	18,842	6	4,812	5	305	4	26	3	1.93	0.11	0.160	0.026	97.0	1.199	0.003
83-04	0.1788	0.0002	25,856	5	6,779	6	406	4	40	3	1.77	0.11	0.225	0.029	98.1	1.180	0.002
83-05	0.1788	0.0002	27,183	9	5,713	7	489	6	24	5	19.39	0.31	0.190	0.021	78.7	1.186	0.005
83-06	0.1788	0.0002	12,743	9	3,243	7	187	5	12	5	2.09	0.27	0.108	0.018	95.1	1.180	0.008
83-07	0.1788	0.0002	12,061	14	3,075	6	174	6	13	5	2.18	0.27	0.102	0.021	94.6	1.172	0.009
83-08	0.1788	0.0002	18,775	18	4,755	7	272	6	21	6	2.12	0.27	0.158	0.022	96.7	1.205	0.006
83-09	0.1788	0.0002	17,799	18	4,521	7	269	6	18	6	1.97	0.27	0.150	0.020	96.8	1.202	0.006
83-10	0.1788	0.0002	6,962	17	1,784	6	111	6	14	6	0.44	0.26	0.059	0.040	98.2	1.210	0.015
														Average		1.185	0.004

The getter can be run at 400°C to enhance the pumping of less reactive gases such as hydrocarbons. The vacuum level of gas preparation system reaches approximately 2×10^{-9} mbar by the turbomolecular pump and ion pump. The air of 0.1 cm^3 from the automatic pipette system consisting of standard volume and two pneumatic valves is routinely measured to derive the discrimination factor.

Mass spectrometer

The purified argon gas through gas preparation system is directly introduced into the mass spectrometer (ARGUS VI). ARGUS VI is an all-metal single focusing, 13 cm radius, 90° extended geometry magnetic sector mass spectrometer designed for operation in static mode (Mark et al. 2009). A Nier-type electron bombardment source is equipped with y- and z-focusing. All source parameters, including the 5 kV acceleration potential, are computer-controlled and monitored by electronic readbacks. This system is equipped with five Faraday detectors and one compact discrete dynode (CDD) detector for the simultaneous measurement of five isotopes of argon (^{36}Ar, ^{37}Ar, ^{38}Ar, ^{39}Ar, and ^{40}Ar). The ^{40}Ar beam is measured in the high-mass (H1) detector fitted with a 10^{11} ohm resistor. The ^{39}Ar to ^{37}Ar beams are collected in other Faraday detectors fitted with 10^{12} ohm resistor. The ^{36}Ar beam is measure by CDD detector. The amplifiers for Faraday detectors are kept in housing evacuated to 1–2 mbar in order to reduce electronic noise. Sensitivity at 200 μA trap current for Ar reaches to 1.15 Amps/Torr and the mass resolution (M/ΔM) is ca. 250. The background value during sample analysis is $<1.0 \times 10^{-16}$ mols ^{40}Ar and $<1.8 \times 10^{-18}$ mols ^{36}Ar in static mode.

System integration and data reduction

Laser heating system, gas preparation system, and mass spectrometer are integrated and controlled by the MassSpec software (Alan Deino Software). It communicates with Chromium II software in laser heating system and Qtegra software in ARGUS VI and controls each parameter in source electronics, magnet control, and steering plate in front of multiple collectors as well as the manipulation of gas preparation system. In addition, it enables the scheduled automatic analysis for tens of analysis. Fitting of raw data, age calculation, and data presentation are also performed using MassSpec software.

Experimental procedures for $^{40}Ar/^{39}Ar$ age

Neutron irradiation

As suggested in the above section, neutron irradiation process is indispensable for the $^{40}Ar/^{39}Ar$ dating. For irradiation, several grains of sample were dropped in each hole on aluminum sample disk with 10 holes of 5-mm diameter along with K_2SO_4 and CaF_2 monitors. Age-known standard minerals were placed in the center or margin of sample disk as the neutron flux monitor. Fisher Canyon sanidine (FCS, 28.294 ± 0.036 Ma: Renne et al. 2010) and Alder Creek sanidine (ACS, 1.193 ± 0.001 Ma: Nomade et al. 2005) were used as flux monitor minerals. Two or three sample disks were stacked and wrapped by 0.25-mm-thick cadmium foil in order to reduce the effect of thermal neutrons. Samples were irradiated for 100 h at IP-4(c) position of hydraulic rabbit irradiation facility in HANARO research reactor with the total neutron flux of ca. $3.7 \times 10^{13} n/cm^2 s$ in Korea Atomic Energy Research Institute (KAERI).

Argon isotope measurement

One or several grains of irradiated samples are placed in a hole on the 133-hole sample holder and then evacuated to ultra high vacuum of ~10^{-9} mbar. Multiple run sequences for sample analysis including air and blank measurement are executed on the MassSpec software. The heating of samples and subsequent cleaning of extracted gas and the measurement of isotopes are sequentially performed by previously defined procedure file. After heating samples for 6 seconds and purifying gases for another 4 minutes with 3 SAES getters, the argon gas is introduced to the mass spectrometer. Each signal of argon isotopes is measured by the multiple data collection using five detectors using H1 to CDD detector or by the peak jumping method using CDD for ^{36}Ar to ^{39}Ar and H1 detector for ^{40}Ar, respectively. For the baseline, the signals at mass 35.7 and 40.3 are measured at the start and end of the analysis. Blank analyses are made every three unknown sample analysis with the identical condition to the actual samples except for laser heating. For the mass discrimination, standard air is measured after every 10 analysis. Variation of blank and mass discrimination factor during analysis are corrected by parabolic regression. Net intensity of each Ar peaks is extrapolated to the zero time when the argon gas was introduced into the mass spectrometer. After correcting the blank and the mass discrimination, *J* values and ages are calculated for the standard and unknown samples.

Determination of J value

As neutron flux changes considerably across the sample disk, monitor minerals such as FCS and ACS sanidine are placed in three positions to correct the horizontal gradient in neutron flux. For real samples, the corrected

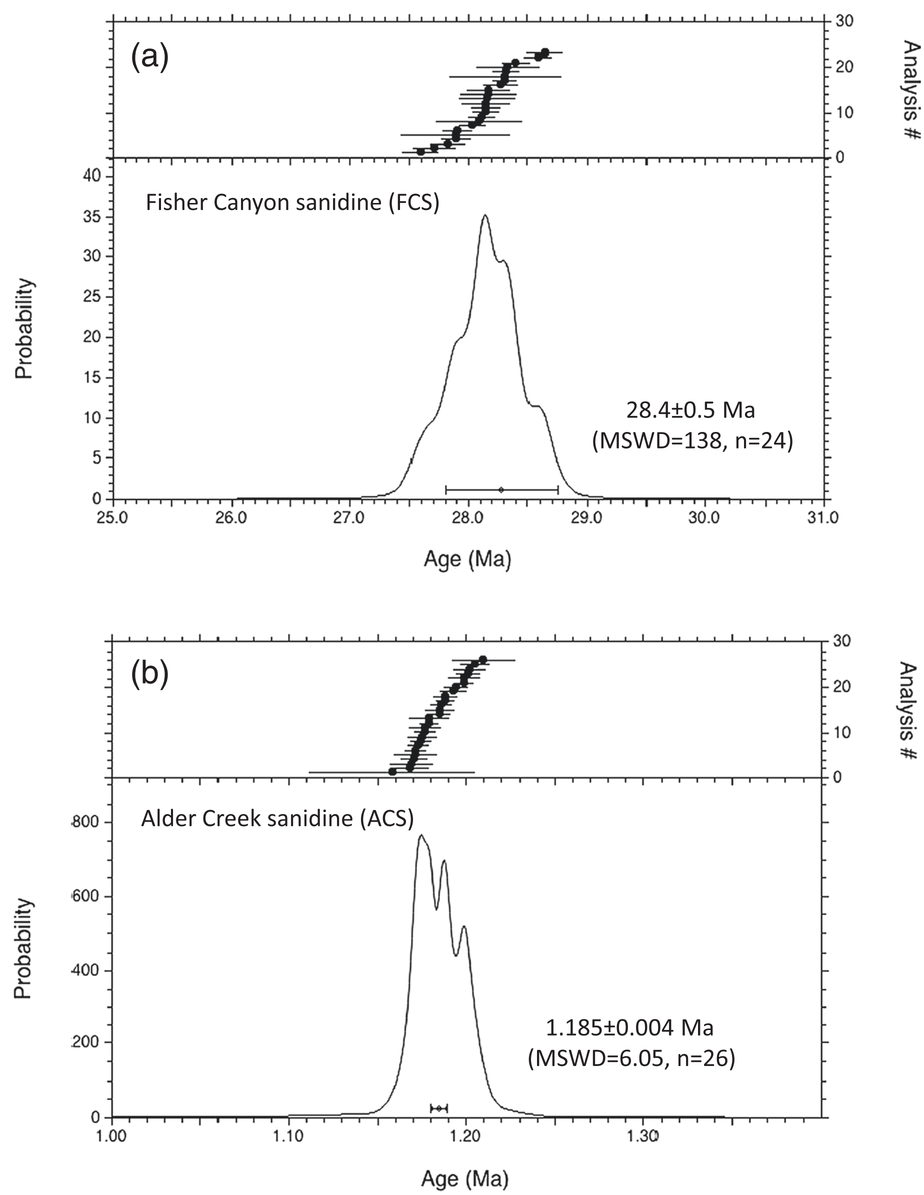

Figure 3 Age probability diagrams for the $^{40}Ar/^{39}Ar$ age of standard materials. (a) Fisher Canyon Sanidine. **(b)** Alder Creek Sanidine.

J value along the different position of samples in the sample disk is applied to the age calculation according to the 3-dimensional modeling for the horizontal flux gradient (Figure 2).

Results and discussion

Single-grain total fusion $^{40}Ar/^{39}Ar$ analyses were repeatedly performed on two standard materials in order to evaluate the performance of ARGUS VI system (Table 1). Figure 3 shows the age probability diagrams for the total fusion ages from single grains of FCS and ACS. The measured value of FCS (Figure 3a) is 28.4 ± 0.5 Ma (1σ, MSWD = 138, n = 24) which is consistent with recommended value (28.294 ± 0.036 Ma: Renne et al. 2010) within uncertainty. Figure 3b shows the single-grain total fusion ages of ACS sanidine (1.185 ± 0.004 Ma, 1σ, MSWD = 6.05, n = 26), which also coincide with recommended age value (1.193 ± 0.001 Ma, Nomade et al. 2005). Such performance shows that the abovementioned $^{40}Ar/^{39}Ar$ age protocol can be successfully applied to the Quaternary rocks such as ACS.

For age dating of young rocks (<1 Ma), the exact derivation of radiogenic ^{40}Ar ($^{40}Ar^*$) is indispensable to get meaningful $^{40}Ar/^{39}Ar$ ages, so that it needs to minimize the reactor-derived ^{40}Ar from ^{40}K. In this study, the $(^{40}Ar/^{39}Ar)_K$ with the 0.25-mm-thick cadmium shielding reaches approximately 0.08, which is higher than the majority of reactor facilities used for $^{40}Ar/^{39}Ar$ age dating (McDougall and Harrison 1999) owing to high slow/fast neutron flux of >780 in Hanaro (Nam S, personal communication). To reduce the incidence of slow neutrons and, thus, the production rate of ^{40}Ar, thicker cadmium shielding should be highly considered.

Conclusions

$^{40}Ar/^{39}Ar$ dating system with multi-collector mass spectrometer and CO_2 laser heating device has been established at KBSI in order to date young and very small amounts of minerals and volcanic rocks. Preliminary experiments on sanidine standards show the reliable age results of total-fusion dating for single grains, i.e., 28.4 ± 0.5 Ma (1σ, MSWD = 138, n = 24) for FCS sanidine, and 1.185 ± 0.004 Ma (1σ, MSWD = 6.05, n = 26) for ACS sanidine as Quaternary age standard, within recommended values (28.294 ± 0.036 Ma and 1.193 ± 0.001 Ma, respectively).

Competing interests
The authors declare that they have no competing interests.

Authors' contribution
JK conceived of the study and carried out the experiment using ARGUS VI as well as writing manuscript. SJ participated in the preparation of experiment and helped to draw the figures. Both authors read and approved the final manuscript.

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