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ABSTRACT

We demonstrate fast analysis of ^{39}Ar/Ar at the 10^{-16} level using a mass spectrometer for isotope pre-enrichment and an atom trap for counting. An argon gas sample first passes through a dipole mass separator that reduces the dominant isotope ^{40}Ar by two orders of magnitude while preserving both the rare tracer isotope ^{39}Ar and a minor stable isotope ^{38}Ar for control purposes. Measurements of both natural and enriched samples with atom trap trace analysis demonstrate that the ^{39}Ar/^{38}Ar ratios change less than 10%, while the overall count rates of ^{39}Ar are increased by one order of magnitude. By overcoming the analysis-speed bottleneck, this advance will benefit large-scale applications of ^{39}Ar dating in the earth sciences, particularly for mapping ocean circulation.

I. INTRODUCTION

The cosmogenic ^{39}Ar (half-life = 268 ± 8 years[1]) is an ideal isotope for tracing groundwater flow, dating mountain glacier ice, and mapping ocean circulation.[2–5] Its unique age span of 50–1800 a bridges an existing gap between ^{14}C and, on the young side, the transient tracers, such as ^{3}H, CFCs, and ^{85}Kr.[6–8] Owing to its extremely low isotopic abundance in the range of 10^{-17}–10^{-15}, ^{39}Ar analysis had previously required large sample sizes (many tons of water) and had been used mostly in groundwater studies in the past.[9–12] Recently, the laser-based Atom Trap Trace Analysis (ATTA) technique has enabled ^{39}Ar dating on kilogram-sized ocean water and ice samples.[13,14] This development has greatly expanded the applications of ^{39}Ar dating in the earth and environmental sciences and has triggered a dramatic increase in demand.

The argon element has three stable isotopes, and their atmospheric isotopic abundances are ^{40}Ar ~ 99.6%, ^{38}Ar ~ 0.063%, and ^{36}Ar ~ 0.334%.[15] In order to determine the isotopic abundance of the rare and radioactive isotope ^{39}Ar, the stable ^{38}Ar is chosen as the control isotope. For dating applications, we measure the ^{39}Ar/^{38}Ar ratio of a sample and compare the result with the ^{39}Ar/^{38}Ar ratio of the atmosphere.[16] The age is then derived from the isotope abundance IA_{39} in the units of "pMAr," which stands for percent of modern argon,

$$IA_{39} = \frac{(^{39}\mathrm{Ar}/^{38}\mathrm{Ar})_{\mathrm{sample}}}{(^{39}\mathrm{Ar}/^{38}\mathrm{Ar})_{\mathrm{air}}} \times 100. \quad (1)$$

At present, the main limiting factor for the ^{39}Ar-ATTA method is the low atom count rate of ^{39}Ar,[10,16–18] which constrains both the measurement precision and the number of samples that can be processed in a given operation period. Even with the state-of-the-art ATTA setup, the ^{39}Ar count rate for a modern argon sample (^{39}Ar/Ar = 8 × 10^{-16}) is only ~10 atoms per h, and an analysis still needs more than 10 h to complete.[16] In order to overcome this analysis-speed bottleneck, we have developed an isotope pre-enrichment system for ^{39}Ar based on a 40 kV mass spectrometer and used it as the pre-enrichment stage to increase the ^{39}Ar concentration, thus boosting the ^{39}Ar atom count rate and reducing the sample measurement time. The pre-enrichment system removes

the dominant isotope ^{40}Ar from the sample while preserving the ^{39}Ar/^{38}Ar ratio of the sample. We define the isotope enrichment factor as the ratio of ^{39}Ar/Ar ratios between the sample after and before the enrichment process. The technical details and preliminary results of the system have been provided in the work of Jia *et al.*[19] Here, we report on quantitative ^{39}Ar analysis with ATTA using this pre-enrichment step. Similar approaches for the ^{81}Kr isotope have been attempted.[20,21] However, quantitative analysis has yet to be demonstrated.

II. METHODS

The measurement procedure is as follows: First, the argon sample is processed in the ^{39}Ar pre-enrichment system located at the Institute of Modern Physics in Lanzhou. Most of the ^{36,40}Ar are removed, while ^{38,39}Ar and the residual ^{36,40}Ar are collected in an aluminum foil. The foil is then sent to the ATTA laboratory at the University of Science and Technology of China (USTC) in Hefei and is melted to release the sample gas. The gas is purified and, finally, fed into the ATTA system for ^{39}Ar analysis.

A. Sample preparation

Two sets of samples are prepared. The first set (set I) consists of modern standard Ar samples with the following sizes: 0.5 ml STP, 2 ml STP, and 5 ml STP (several samples for each size). These are made from commercial argon acquired in 2018, originally collected from air, all with ^{39}Ar abundances at 100 pMAr. The second set (set II) is standard Ar samples with the following ^{39}Ar abundances: 20 pMAr, 50 pMAr, and 100 pMAr (several samples for each abundance). Samples with ^{39}Ar abundance less than 100 pMAr are prepared by mixing the modern standard Ar sample with an Ar sample depleted in ^{39}Ar. The depleted sample is obtained from air trapped in very old ice cores retrieved from a glacier in the Tibetan plateau. The ^{39}Ar abundance of the depleted Ar sample is measured with ATTA to be less than 2 pMAr. By mixing the depleted sample and the modern standard Ar with precisely controlled mixing ratios (<1% uncertainty), samples of 20.0 ± 1.6 pMAr and 50 ± 1.0 pMAr abundances are prepared.

B. Sample enrichment procedure

The pre-enrichment of Ar sample set I and set II is carried out at the Institute of Modern Physics using the ^{39}Ar enrichment Ion beam System (ARIS). The technical details of ARIS have been previously reported.[19] ARIS is an electromagnetic isotope separation system based on a dipole mass separator [see Fig. 1(a)]. It is equipped with a 2.45 GHz ECR (Electron Cyclotron Resonance) ion source capable of producing a beam of 40 keV Ar^+ ions with a current of 1–10 mA. The ECR ion source is chosen for its high ionization efficiency (50%–60%), which is essential in this application because of the limited Ar sample size.[22,23] At a typical beam current of 2 mA, the time to process a sample of 1 ml STP of Ar takes about 1 h. The Ar^+ ions extracted from the ECR ion source pass through a high resolution, high transmission efficiency spectrometer system consisting of two quadrupole focusers and a 95°dipole separator. The two quadrupole focusers can help constrain the size of the ion beam, suppress its envelope increase due to the space charge effect of the intense beam. The dipole separator bends and spatially separates the ion beams of Ar isotopes with different masses [Fig. 1(b)]. The mass separation system has a mass resolution of >100. The beam optics are optimized for M/Q = 39. The transmission efficiency for ^{39}Ar is typically better than 50%. On the focal plane is a target made of an aluminum foil intended to collect $^{39}Ar^+$ and $^{38}Ar^+$ ions and to avoid $^{40}Ar^+$ and $^{36}Ar^+$. The $^{40}Ar^+$ beam is directed to a beam dump. The $^{36}Ar^+$ ions are collected with a Faraday cup and used as a beam current monitor. The foil is mounted on a servo motor driving system to avoid saturation. More than 99% of the ^{40}Ar isotope is directed away from the foil and thus removed from the enriched sample. The sample imbedded in the foil is significantly enriched in ^{39}Ar, i.e., the ^{39}Ar/Ar ratio becomes two to three orders of magnitude higher. However, the ^{39}Ar/^{38}Ar ratio is not expected to change since, by design, both isotopes are collected into the foil. This arrangement allows us to use the ^{38}Ar isotope as the reference isotope in the later ATTA analysis.

C. Sample extraction

The foil containing the enriched sample is shipped to USTC, where ^{39}Ar analysis with ATTA is performed. To release the imbedded Ar sample, the foil is placed in an alumina boat inside a vacuum

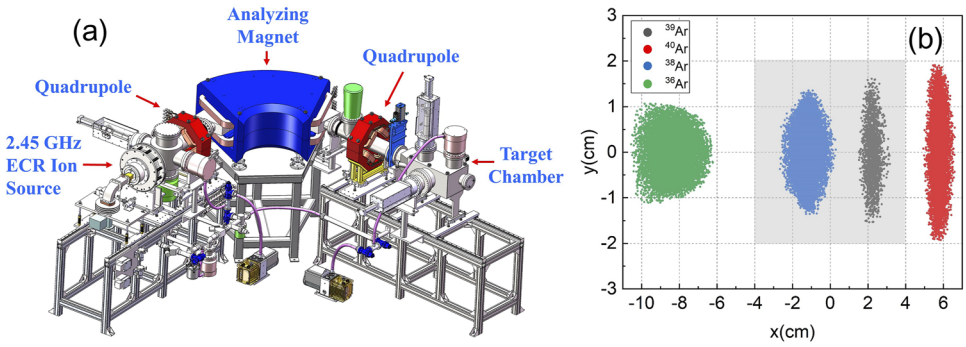

FIG. 1. The pre-enrichment system. (a) ^{39}Ar enrichment Ion beam System (ARIS). (b) Simulation of ion beam profiles on the target plane. The shaded area represents the aluminum foil target.

melter in a high temperature oven. The melter is made of a standard 2–3/4 inch diameter stainless steel pipe. The melter is first evacuated by a turbomolecular pump, pre-baked at 150 °C for 30 min to release water, and residual gas adsorbed on the foil and the inner wall of the melter. The melter is then sealed by closing the valve between it and the pump and is heated to 850 °C for 50 min. At this temperature, the aluminum foil is melted, and the imbedded Ar sample is released. The melter is then cooled down to 200 °C, and the Ar gas is transferred to a reservoir and purified with a heated getter pump to remove the impurities. The size of the pre-enriched Ar is typically below 10 μl STP.

D. ATTA analysis

The details about the ATTA analysis of ^{39}Ar have been reported in the work of Tong *et al.*[16] Single-atom counting is used to detect ^{39}Ar atoms. Every 10 min, the system switches over and takes a measurement on the reference isotope ^{38}Ar, which is measured with the "quench fluorescence" method because its abundance is too high to use the single-atom counting method. By switching between the two isotopes frequently and taking the ratio of ^{39}Ar/^{38}Ar, many effects that cause varying counting efficiencies, common to both isotopes, are largely canceled.

During the ATTA measurement, the Ar sample gas is circulated in the high vacuum system. For the ATTA system to operate, the pressure of the system needs to be maintained at a certain level (usually at 10^{-5} Torr level in the transverse cooling stage).[16] The reason is a discharge needs to be sustained so that it can excite the Ar atom into a particular metastable atomic state during the measurement.[16] The ATTA system has a flow conductance control function that allows us to operate the ATTA system at the optimal pressure with the maximal ^{39}Ar atom count rate (10 atoms/h for modern Ar sample) for samples of different sizes [150 μl to 2 ml STP (standard temperature and pressure is assumed for all following discussions)]. However, the size of the pre-enriched Ar sample is below the minimum value that can be accommodated by the flow conductance system. Therefore, Kr gas is added to make the total amount of gas (Ar + Kr) about 150 μl so that the pressure is high enough to make the discharge operate normally. For metastable ^{39}Ar production, Ar, Kr, or Xe works reasonably well as the carrier gas. The ^{39}Ar atom count rate is determined by the sample size reduction ratio and can be written as $V_{eff}/V_{opt} \times 10$ atoms/h. V_{eff} is the effective Ar sample size before pre-enrichment, i.e., the Ar sample that is actually enriched in the ARIS system. Currently, the V_{eff} is about 30% of the size of the original Ar put into the ARIS system, limited by the single-pass efficiency. Here, we want to point out that V_{opt} is 150 μl, the minimum sample size required to operate the ATTA system at the optimal pressure, not the Ar sample size after pre-enrichment. From this estimation, it is clear that the ^{39}Ar atom count rate enhancement is limited by V_{opt} of the ATTA system. If more enriched Ar sample is available, the atom count rate can be higher. Further reduction of V_{opt} leads to more enhancement in the ^{39}Ar atom count rate.

III. RESULT AND DISCUSSION

The sizes of the Ar samples are greatly reduced after the pre-enrichment, from 1 ml STP to less than 5 μl STP, as most ^{40}Ar atoms are removed. This is confirmed by the residual gas analyzer (RGA) installed on the ATTA system. Figure 2 is a typical scan over the mass range of 34–42 amu. It shows that both the ^{40}Ar and ^{36}Ar peaks have been reduced significantly relative to ^{38}Ar, compared to the atmospheric Ar composition. This confirms that the foil for sample collection is positioned correctly on the target plane in ARIS.

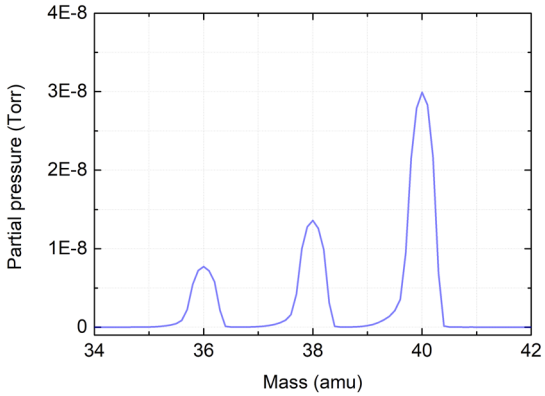

FIG. 2. Typical mass scan of a pre-enriched argon sample with a residual gas analyzer.

Based on the amount of ^{38}Ar collected, we estimate the single-pass efficiency of ARIS to be 30%. In other words, 30% of the ^{38,39}Ar atoms in the original sample are ionized, directed through the mass separator, and implanted into the foil, and the rest is pumped away. This is comparable to the value based on estimations of the ionization efficiency and the beam transport efficiency of ARIS. In the future, operating ARIS in a gas re-circulation mode would increase the efficiency.[19] From the sample sizes before and after the pre-enrichment, the ^{39}Ar/Ar enrichment factor is calculated to be between 200 and 600. The enrichment factor and the efficiency do not have a strong dependence on the sample size, although they do show a variation of about 50% from run to run, which could be related to the various operation conditions of ARIS, such as the field strength of the dipole magnet and position of the target.

With enriched samples, the ^{39}Ar count rates are increased by up to an order of magnitude (Fig. 3). The enhancement shows a

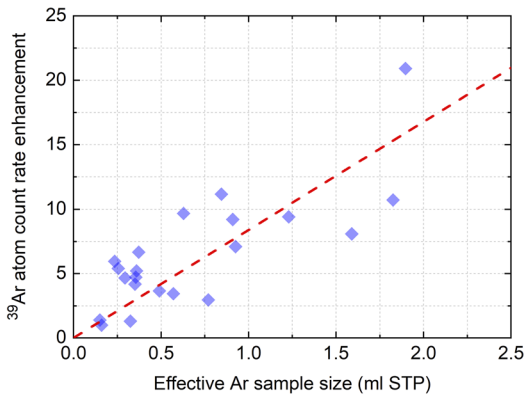

FIG. 3. ^{39}Ar atom count rate enhancement vs effective Ar sample size before pre-enrichment. The red dashed line is the fit.

linear dependence on the sample size. The larger the sample the higher the enhancement factor (see discussion in Sec. II D). For Ar samples below 0.3–0.5 ml STP, the advantage of pre-enrichment is almost completely canceled out by the small volume after pre-enrichment. As a result, the ^{39}Ar atom count rate is unchanged before and after the sample pre-enrichment. To take full advantage of the pre-enrichment enhancement, further optimization that allows the ATTA system to reach the optimal condition with smaller Ar samples is needed. Nevertheless, even under the current condition, it is clear that the pre-enrichment can increase the ^{39}Ar count rate by one order of magnitude for Ar samples larger than 1 ml STP in the ATTA analysis, thus reducing measurement time and boosting measurement precision.

For set I samples, the measured IA_{39} values are consistent with 100 pMAr (see Fig. 4). This confirms that ARIS operates with the same transmission and collection efficiency for ^{39}Ar and ^{38}Ar. As a result, ^{39}Ar/^{38}Ar ratios do not change after pre-enrichment [see Eq. (1)]. The results for set II samples are shown in Fig. 5. The data show a good proportional relationship between the isotopic abundances measured on the enriched samples and the expected values according to the mixing ratios. The slope of the fit line is 1.07 ± 0.02, which indicates a small but measurable difference between the collection efficiencies of ^{39}Ar and ^{38}Ar in ARIS. Set I and set II samples are pre-enriched in different runs. This suggests that the transmission efficiencies of $^{39}Ar^{+}$ and $^{38}Ar^{+}$ ions through the ion beam optics may change from run to run, possibly due to the space charge effect. In practice, this deviation can be measured and corrected with a standard modern Ar sample introduced into each batch of Ar samples during the pre-enrichment.

The other thing worth noting is that the set II samples are enriched at three different dipole magnet current settings. This is done deliberately to check the consistency of the pre-enrichment process and to explore the systematics caused by potential drifts due to the hysteresis of the dipole magnet in ARIS. The slight changes of the current result in small horizontal displacement of the Ar ion beams on the target plane [see Fig. 1(b)]. As the data show, small changes (1%) on the dipole magnet current have no measurable effects on the collection efficiencies of ^{39}Ar and ^{38}Ar, i.e., all data fit

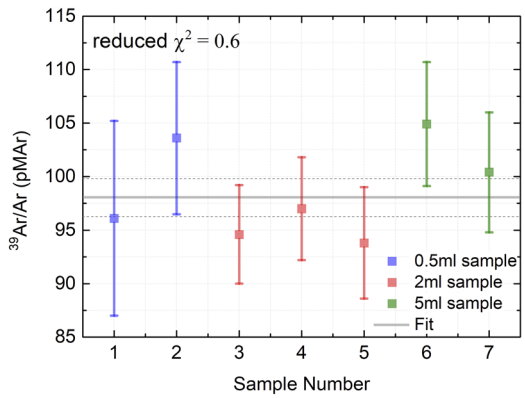

FIG. 4. Ar sample set I. The measured isotopic abundance of modern sample shows the collection efficiency of ^{38}Ar and ^{39}Ar is same during the enrichment process.

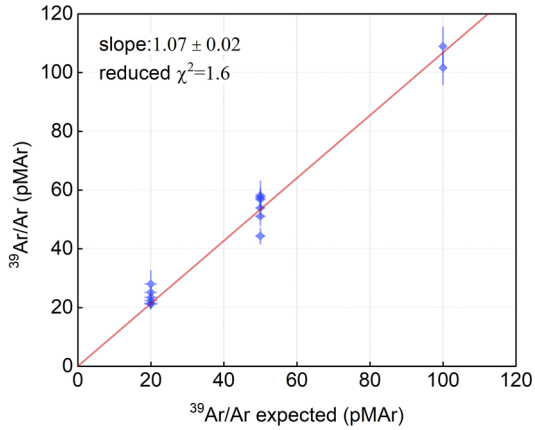

FIG. 5. Ar sample set II. The measured isotopic abundance IA_{39} of the pre-enriched samples shows a linear relationship with the expected isotopic abundance IA_{39} [Eq. (1)].

to a straight line. This is reasonable because their positions are in the center region of the foil target. However, the amount of residual ^{40}Ar and ^{36}Ar ions collected on the two edges of the foil are much more sensitive to the displacement. This can be seen from the ^{40}Ar/^{38}Ar and ^{36}Ar/^{38}Ar ratios of the enriched sample (see data in the supplement). Different current settings can change ^{36}Ar/^{38}Ar ratios by as much as a factor of 10 and ^{40}Ar/^{38}Ar ratios by a factor of 2.

IV. CONCLUSION

In summary, we have demonstrated fast quantitative ^{39}Ar analysis using an isotope pre-enrichment system (ARIS) and ATTA. The introduction of the pre-enrichment system can increase the ^{39}Ar atom count rate by one order of magnitude, which reduces the measurement time accordingly. The higher atom count rate also increases statistics of the measurement, especially for old samples with low ^{39}Ar abundances, leading to higher precisions and extended age ranges for ^{39}Ar dating. To increase the performance further, a few improvements can be implemented in the near future. One is to operate ARIS in a gas re-circulation mode. This will allow a more efficient usage of the Ar sample and make the pre-enrichment also useful for small samples below 0.5 ml of argon. Moreover, the ^{39}Ar atom count rate can be increased by optimization of the ATTA vacuum system for the small pre-enriched Ar samples, i.e., lowering V_{opt}, thus fully realizing the potential of ARIS and ATTA.

SUPPLEMENTARY MATERIAL

Measurement results for set I and set II samples are available in the supplementary material.

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AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

DATA AVAILABILITY

The data that support the findings of this study are available within the article and its supplementary material.

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