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TWO NEW MILLENNIUM-LONG TREE-RING OXYGEN ISOTOPE CHRONOLOGIES (2349–1009 BCE AND 1412–466 BCE) FROM JAPAN

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ABSTRACT. We present two new millennium-long tree-ring oxygen isotope chronologies for central and northern Japan, based on 9693 annually resolved measurements of tree-ring oxygen isotopes from 39 unearthed samples consisting mainly of Japanese cedar (*Cryptomeria japonica*). These chronologies were developed through cross-dating of tree-ring widths and $\delta^{18}O$ data from multiple samples covering the periods 2349–1009 BCE (1341 yr) and 1412–466 BCE (947 yr) for central and northern Japan, respectively. In combination with our published chronology for central Japan, the tree-ring $\delta^{18}O$ dataset currently available covers the past 4354 yr (2349 BCE to 2005 CE), which represents the longest annually resolved tree-ring $\delta^{18}O$ dataset for Asia. Furthermore, the high-resolution temporal record of ^{14}C contents independently developed by Sakurai et al. (2020) was reproduced by our ^{14}C measurements of earlywood and latewood in annual rings for the period 667–660 BCE.

KEYWORDS: Dendrochronology, oxygen isotopes, tree-ring width, ^{14}C spike matching.

INTRODUCTION

Tree rings are widely used to date wood samples using the principle of cross-dating between different trees (Stokes and Smiley 1968). The longest tree-ring chronology so far produced in Japan dates back to 1313 BCE, based on tree-ring width measurements of *Cryptomeria japonica* (Mitsutani 2000). Another long tree-ring width chronology that extends back to 912 BCE was developed using *Chamaecyparis obtusa* in Japan (Mitsutani 2000). These two chronologies have been widely utilized to date woods recovered from buried forests, archaeological sites, and old temples (Nara National Research Institute for Cultural Properties 1990). However, due to the limited climatic sensitivity of tree growth in Japan, samples with a short tree-ring sequence (<100 rings) are often challenging to date using tree-ring width data. In addition, samples of different species cannot typically be cross-dated between each other, because of species-dependent variations in tree-ring width data. Therefore, many wood samples in Japan are yet to be dated.

Recent progress in oxygen isotope dendrochronology has shown considerable promise in overcoming the limitations described above (Loader et al. 2019, 2020, 2021; McCarroll et al. 2019; Nakatsuka et al. 2020). This is because oxygen isotope ratios ($\delta^{18}O$) of tree-ring cellulose are mainly controlled by hydroclimatic parameters during the growing season, irrespective of the tree species. Due to its strong climatic sensitivity, annual variations in tree-ring $\delta^{18}O$ values are well correlated in samples from the same and different species. In addition, tree-ring $\delta^{18}O$ time-series are also significantly correlated between samples that are spatially distant from each other (Seo et al. 2019; Sano et al. 2022). Recently a 2600-yr-long tree-ring record was developed for Japan using both oxygen and hydrogen isotopes, in order to reconstruct decadal–centennial hydroclimatic variability (Nakatsuka et al. 2020). The original $\delta^{18}O$ time-series was then modified by extracting high-frequency components

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to produce a master chronology that is suitable for tree-ring dating (Sano et al. 2022). While the updated $\delta^{18}O$ chronology works well for dating archaeological samples with short tree-ring sequences (30–60 rings), the tree-ring $\delta^{18}O$ chronology needs to be extended both spatially and temporally in Japan.

Annually resolved ^{14}C data in which rapid changes in ^{14}C contents occur, such as at 774/5 CE (Miyake et al. 2012) and 993/4 CE (Miyake et al. 2013), are a promising approach to single year dating. For example, Wacker et al. (2014) dated a wooden beam in a historically important and well-preserved chapel in Switzerland using the 774/5 CE event. Similarly, Oppenheimer et al. (2017) and Hakozaki et al. (2018) successfully dated the so-called “Millennium Eruption” of Baitoushan Volcano to late 946 CE using the 774/5 CE event, whereas the wood samples cannot be dendrochronologically dated using just tree-ring width data. The ^{14}C spike matching method is therefore suitable for independent validation of dendrochronological dating (Büntgen et al. 2018).

In this study, we developed two new millennium-long tree-ring oxygen isotope chronologies for Japan. These chronologies are for central and northern Japan, and cover the periods 2349–1009 BCE (1341 yr) and 1412–466 BCE (947 yr), respectively. Dendrochronologically dated samples for the period 667–660 BCE were further used for radiocarbon measurements at sub-annual resolution, to verify our dates by identifying a rapid ^{14}C spike observed in Germany (Park et al. 2017) and Japan (Sakurai et al. 2020). In combination with a published tree-ring $\delta^{18}O$ time-series from central Japan (Nakatsuka et al. 2020; Sano et al. 2022), our tree-ring $\delta^{18}O$ dataset currently available for Japan covers the past 4354 yr (2349 BCE to 2005 CE), which represents the longest annually resolved tree-ring $\delta^{18}O$ record for Asia. Tree-ring data for the master chronologies are archived as Supplementary Material to this article.

MATERIALS AND METHODS

The tree-ring data used in this study are for the Chōkai and Kurota sites, which are located in northern and central Japan, respectively (Figure 1). We collected disk or block samples of *Cryptomeria japonica* unearthed from Chōkai volcano. These woods are locally recognized as “Chōkai–Jindai cedar”, which were buried in debris avalanche deposits produced by a sector collapse of Chōkai volcano (Inokuchi 1988). Because of the limited sample size, which is not sufficient for developing a tree-ring chronology, we also collected samples of *Zelkova serrata* and *Quercus* sp. Although tree-ring width patterns of these samples cannot be matched with those of the cedar samples, tree-ring $\delta^{18}O$ values can be matched between conifer and broad-leaf trees (Sano et al. 2022). A total of 34 samples, consisting of 28, 5, and 1 samples of *Cryptomeria*, *Zelkova*, and *Quercus,* respectively, were obtained from a lumber dealer and local authority. We also collected a total of 63 samples of *C. japonica* unearthed from the Kurota site, where a swamp forest dominated by *Alnus*, *Fraxinus*, and *Cryptomeria* existed (Tsuji et al. 1995).

In addition to tree-ring oxygen isotope data, tree-ring widths from the cedar samples were utilized to independently conduct pattern matching amongst the samples. The samples were polished with progressively finer sandpaper until the annual ring boundaries could be clearly recognized. Tree-ring widths were then measured using images of the samples that were scanned into a personal computer at resolutions of 600–3700 dots per inch depending on the tree-ring width. Following the standard methodology of tree-ring dating based on tree-ring width data (Baillie and Pilcher 1973), standardization was conducted to extract

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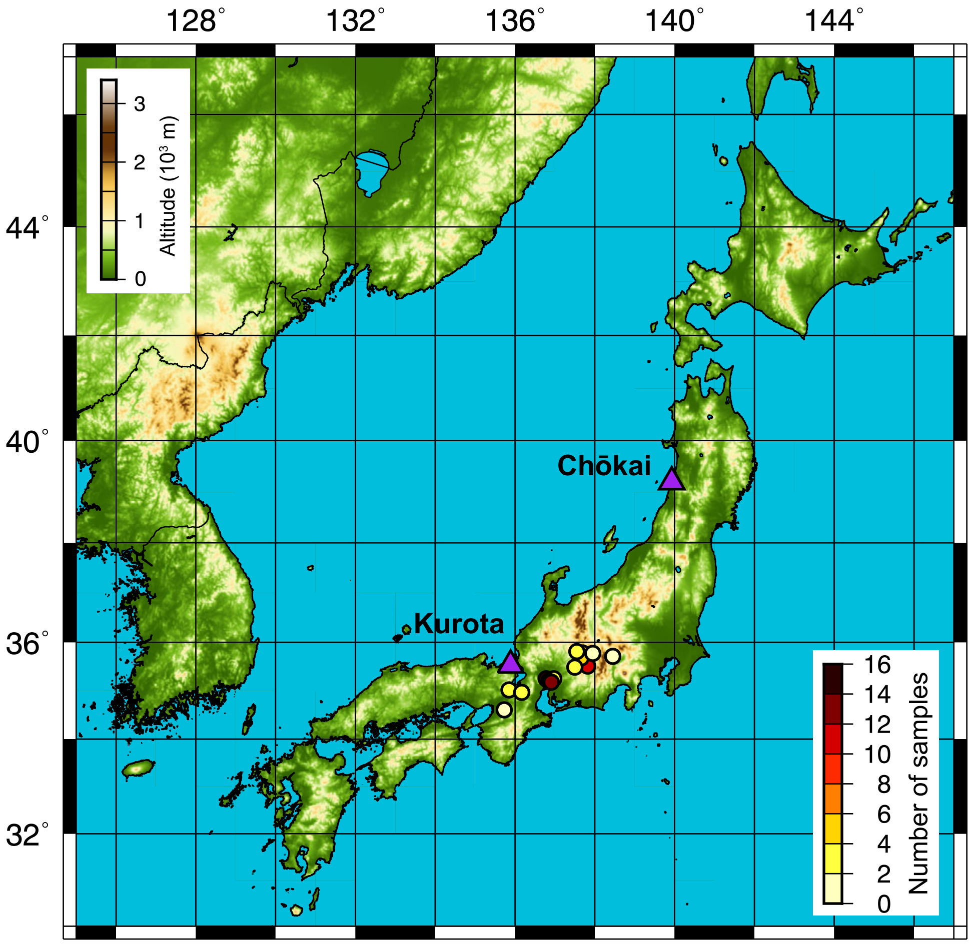

Figure 1 Map of Japan showing the locations of the Chōkai and Kurota sites (triangles), and the sites (circles) where a total of 67 samples were collected for the 2617-yr-long master chronology extending to the present-day (Nakatsuka et al. 2020; Sano et al. 2022).

the high-frequency variability component from individual tree-ring width series. Specifically, the tree-ring widths were expressed as the percentage departures from the 5-yr running mean fitted to the raw tree-ring width series, and then transformed using the natural logarithm. Pattern matching of tree-ring width variations was conducted using the standardized series to assign relative years for each ring. For the buried wood from the Chōkai site, the outermost tree ring of samples with exterior bark was previously dated to 466 BCE based on tree-ring width records, which were independently measured by Mitsutani (2001). The cedar samples used by Mitsutani (2001) are different from our samples. As such, we provisionally considered that the last tree rings of our four samples had an exterior bark date of 466 BCE, which was further verified by the tree-ring $\delta^{18}O$ and ^{14}C data. In this study, we report calendar years using the "Gregorian calendar" (i.e., with no "year zero", whereby 1 BCE is followed by 1 CE).

Based on the pattern matching results using the tree-ring width data, a total of 17 and 22 samples from the Chōkai and Kurota sites, respectively, were selected for oxygen isotope

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analysis. For the Chōkai samples, 11, 5, and 1 of the 17 samples were from *Cryptomeria, Zelkova*, and *Quercus,* respectively. The plate method was used to directly extract cellulose from 1-mm-thick wood plates whilst preserving the cell wall structures (Kagawa et al. 2015). Each individual ring (100–300 μg) was sub-sampled from a cellulose plate using a razor blade under a microscope. The oxygen isotope ratios ($^{18}O/^{16}O$) of the tree-ring cellulose were determined using a continuous flow mass spectrometer (Delta V Advantage; Thermo Fisher Scientific) coupled to a pyrolysis-type elemental analyzer (TC/EA High Temperature Conversion Elemental Analyzer; Themo Fisher Scientific) interfaced with a Conflo III (Thermo Fisher Scientific). The oxygen isotope ratios are reported as $\delta^{18}O$ values (in ‰) relative to the Vienna Standard Mean Ocean Water (VSMOW) standard. Working standard material (Merck cellulose) was measured every eight samples, and the analytical uncertainty was less than ±0.20‰ (1σ). Similar to the tree-ring width data, a high-pass filter was used to standardize the raw tree-ring $\delta^{18}O$ time-series. As described by Loader et al. (2019), the statistical properties of tree-ring $\delta^{18}O$ time-series differ from those of tree-ring width time-series, and thus another standardization method is suitable for the oxygen isotope data. Based on Loader et al. (2019), an 11-yr rectangular filter, which was previously applied to data from Japan (Sano et al. 2022), was utilized to standardize each of the tree-ring $\delta^{18}O$ time-series. Specifically, each of the raw $\delta^{18}O$ time-series was filtered and subtracted to produce anomalies with a mean of zero. Our analyses were performed using dplR (Bunn 2008) and some packages in the R environment (R Core Team 2020). The statistical analyses of the tree-ring $\delta^{18}O$ data are given in Supplementary Tables 1–2.

Pattern matching of the detrended tree-ring $\delta^{18}O$ time-series was conducted between different samples from the Chōkai or Kurota sites. The result was then confirmed to be consistent with the dating result obtained from the tree-ring width data. Our pattern-matched time-series were then averaged to produce a master chronology for each site. While the outermost year of the Chōkai chronology is known to be 466 BCE, our $\delta^{18}O$ chronology from the Chōkai site was independently cross-dated against a 2617-yr-long $\delta^{18}O$ chronology from central Japan (Nakatsuka et al. 2020; Sano et al. 2022). Subsequently, the Kurota $\delta^{18}O$ chronology was cross-dated against the dated Chōkai $\delta^{18}O$ chronology.

To further confirm the robustness of our dendrochronological dating, we precisely measured the ^{14}C contents of earlywood and latewood in annual rings of a Chōkai sample (Ck-W13) for the period 667–660 BCE, during which a rapid ^{14}C increase was observed in German oak (Park et al. 2017) and Chōkai–Jindai cedar (Sakurai et al. 2020). The Chōkai–Jindai cedar used by Sakurai et al. (2020) is a different sample to our Chōkai sample. Based on our dating results of the Chōkai samples, eight consecutive rings corresponding to this period were isolated from the sample for this analysis. Our ^{14}C time-series was then compared with an independently dated ^{14}C time-series developed by Sakurai et al. (2020). For the ^{14}C analysis, cellulose was extracted from small pieces of earlywood and latewood by acid–alkali–acid and sodium chlorite treatments (Miyake et al. 2017). The graphite extraction and ^{14}C analysis was conducted at the Yamagata University Accelerator Mass Spectrometry (YU-AMS) facility (Tokanai et al. 2013).

RESULTS AND DISCUSSION

Pattern-matched time-series and their correlation matrix are presented in Figures 2–3, respectively, for the tree-ring widths and $\delta^{18}O$ data from the Chōkai site. Figures 4–5 are the same as Figures 2–3, but for the Kurota site. Enlarged plots are also presented in

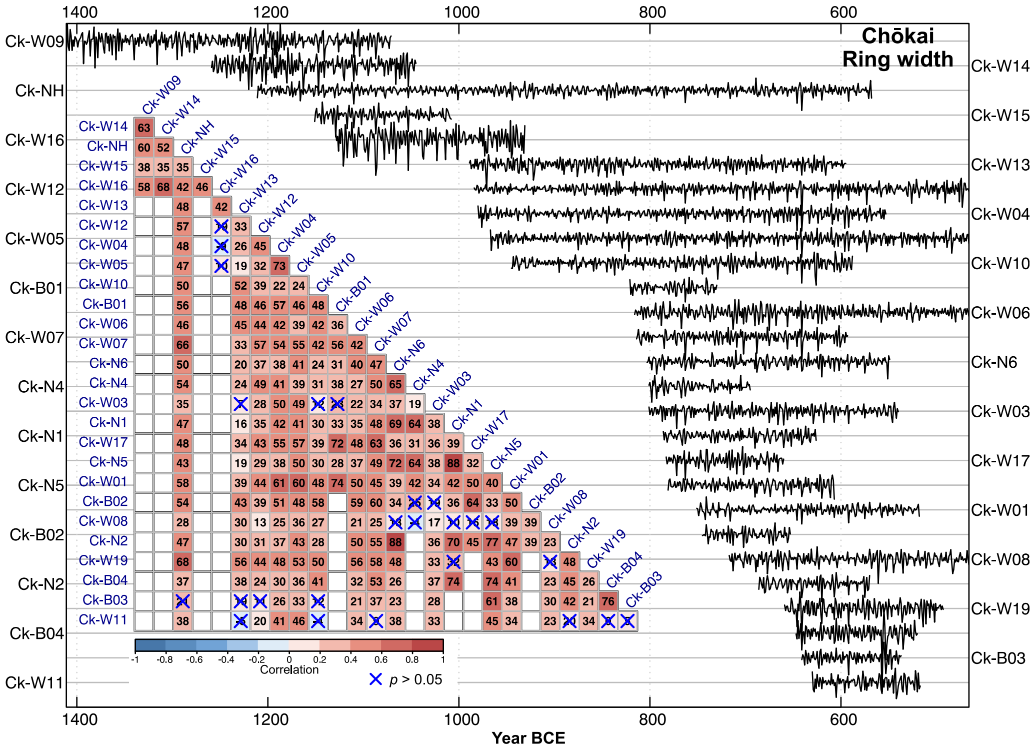

Figure 2 Plots of cross-dated tree-ring width series from the Chōkai site, along with their correlation matrix, in which pairs of overlapping periods of <20 yr were masked out. Note that the tree-ring width series were standardized using a 5-yr running mean fit and natural logarithmic function to extract the high-frequency variability component (see the text for more details). Enlarged plots are presented in Supplementary Figure 1.

Supplementary Figures 1–4. In addition, correlations of all pairs of tree-ring width and $\delta^{18}O$ time-series from the two sites are plotted in Figure 6.

Mean inter-series correlations for tree-ring width are 0.40 and 0.20 for the Chōkai and Kurota sites, respectively. The tree-ring width patterns of the Chōkai samples are well matched between different trees, with 89.8% of pairs showing significant correlations (Figure 2). The cedar samples from this site are thus considered to have been climatically stressed. In contrast, the tree-ring width time-series from the Kurota site exhibit relatively weak correlations between different trees, with 62.4% of pairs showing significant correlations (Figure 4). The cedars from the Kurota site grew in a swamp forest, which might explain the weaker correlations (i.e., less climatically stressed).

As expected, the mean inter-series correlations for the tree-ring $\delta^{18}O$ data are higher than those for the tree-ring widths, being 0.54 (95.7% of pairs at $p < 0.05$) and 0.61 (98.8%) for the Chōkai and Kurota sites, respectively (Figures 3 and 5). Importantly, variations in tree-ring $\delta^{18}O$ values are not affected by the growing environment, which significantly modulated the tree-ring widths of the Kurota samples. Furthermore, $\delta^{18}O$ patterns of the cedars are well matched with those of the broad-leaf trees from the Chōkai site (Figure 3). In general, the tree-ring $\delta^{18}O$ time-series are well correlated between different samples from both study sites, indicating that our samples can be robustly cross-dated. It should be noted that all

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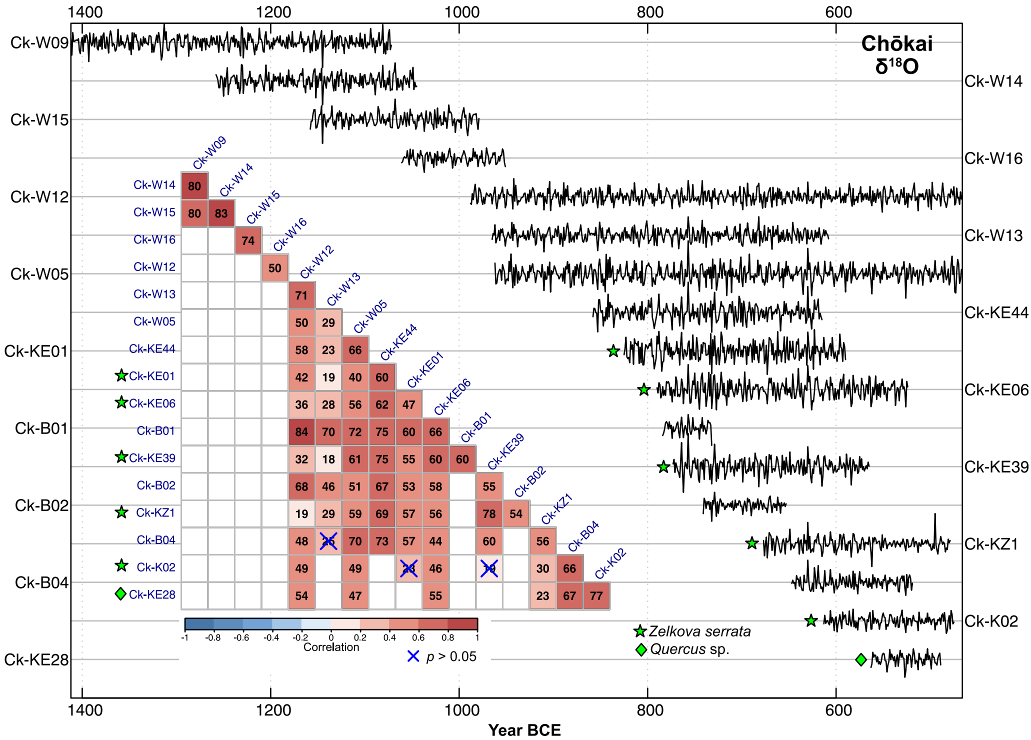

Figure 3 Plots of cross-dated tree-ring $\delta^{18}O$ series from the Chōkai site, along with their correlation matrix, in which pairs of overlapping periods of <20 yr were masked out. Note that the tree-ring $\delta^{18}O$ series were standardized using an 11-yr rectangular filter to extract the high-frequency variability component (see the text for more details). Enlarged plots are presented in Supplementary Figure 2.

the dating results from the tree-ring width data are consistent with those from the tree-ring $\delta^{18}O$ data. Based on the relative years determined by pattern matching, all of the standardized tree-ring $\delta^{18}O$ time-series were individually averaged for the Chōkai and Kurota sites to construct the final $\delta^{18}O$ chronologies.

Our $\delta^{18}O$ chronologies are significantly correlated with each other (Figure 7). Specifically, the Chōkai chronology was successfully cross-dated against the master chronology from central Japan (Nakatsuka et al. 2020; Sano et al. 2022). The overlapping period was 147 yr, which has a significant correlation of 0.26 ($t = 3.22$; $p < 0.01$) between the two chronologies. As expected, the end rings of samples with a bark exterior from Chōkai were dated to 466 BCE, which is consistent with an earlier tree-ring width study (Mitsutani 2001). The Kurota chronology was subsequently cross-dated against the dated Chōkai chronology. There is also a significant correlation of 0.33 ($t = 6.94$; $p < 0.01$) between these chronologies over the overlapping period of 404 yr.

Seo et al. (2019) reported that a tree-ring $\delta^{18}O$ chronology from southern Korea was well correlated with that from central Japan over the past 142 yr ($r = 0.47$; $t = 6.31$; $p < 0.01$), even though these records are located ~1000 km apart. However, the Chōkai site is only 400 km from the central Japan site, yet the correlation of 0.26 is lower than that with the tree-ring data from southern Korea. This difference is due to the hydroclimatic variability

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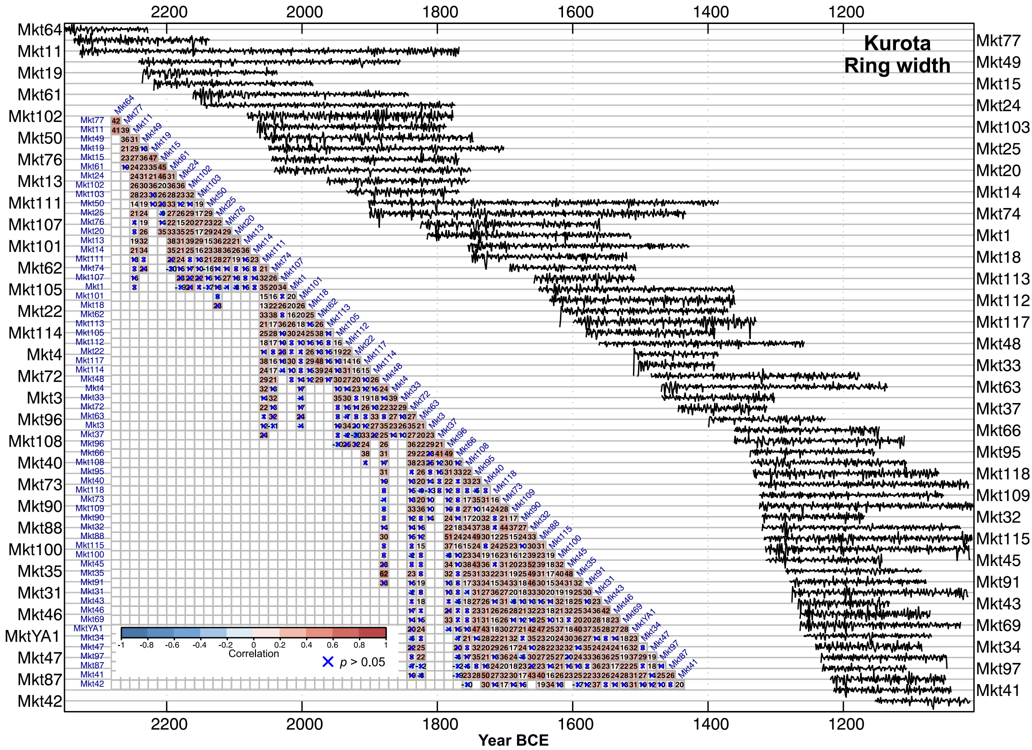

Figure 4 Same as Figure 2, but for the Kurota site. Enlarged plots are presented in Supplementary Figure 3.

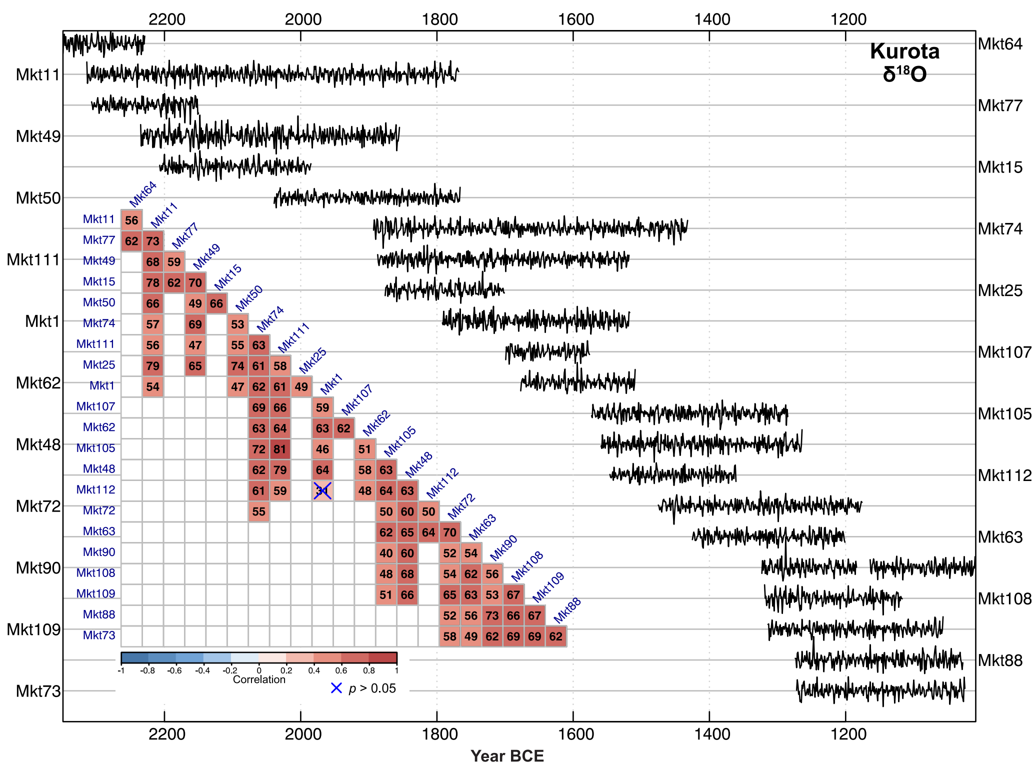

Figure 5 Same as Figure 3, but for the Kurota site. Enlarged plots are presented in Supplementary Figure 4.

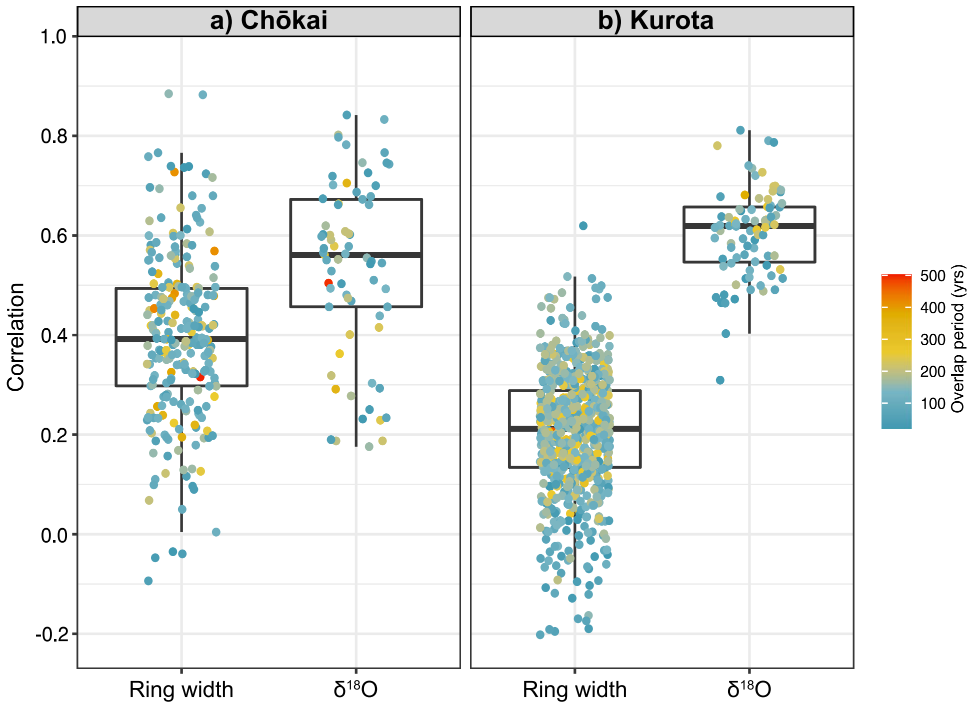

Figure 6 Correlations of dated tree-ring widths or $\delta^{18}O$ data for all possible pairs for the a) Chōkai and b) Kurota sites. Pairs of overlapping periods of <20 yr were masked out.

of East Asia, including South Korea and Japan, whereby the June–July season is controlled by the Meiyu–Baiu front that occurs as a zonally oriented rain band. Variations in precipitation or relative humidity, both of which are recorded by tree-ring $\delta^{18}O$ values, are thus better correlated zonally than meridionally. In fact, our tree-ring record from central Japan shows significant correlations with June–July precipitation over the same latitude (Nakatsuka et al. 2020). This climatological mechanism apparently affects the spatial correlations of tree-ring $\delta^{18}O$ time-series. It should also be noted that the earliest part (i.e., 612–544 BCE) of the master chronology from central Japan comprises only one sample, which is partly responsible for the lower correlation of 0.24 with the corresponding part of the Chōkai chronology. In fact, these two chronologies have a higher correlation of 0.30 for 537–466 BCE, during which the master chronology from central Japan comprises at least three samples. Nevertheless, there are still significant common signals in these different chronologies during the overlapping periods.

The high-resolution temporal record of ^{14}C contents independently developed by Sakurai et al. (2020) was well reproduced by our ^{14}C measurements of earlywood and latewood in annual rings for the period 667–660 BCE (Figure 8). The agreement between the two ^{14}C time-series is reasonable (reduced chi-squared $\chi_{red} = 1.1$), and it becomes significantly worse ($p < 0.01$) if either age is changed by more than 1 yr. Therefore, our dendrochronologically dated ages are further supported by the ^{14}C spike matching.

In summary, we developed two millennium-long tree-ring $\delta^{18}O$ chronologies (947 and 1341 yr for the Chōkai and Kurota sites, respectively) for Japan, back to 2349 BCE. In combination

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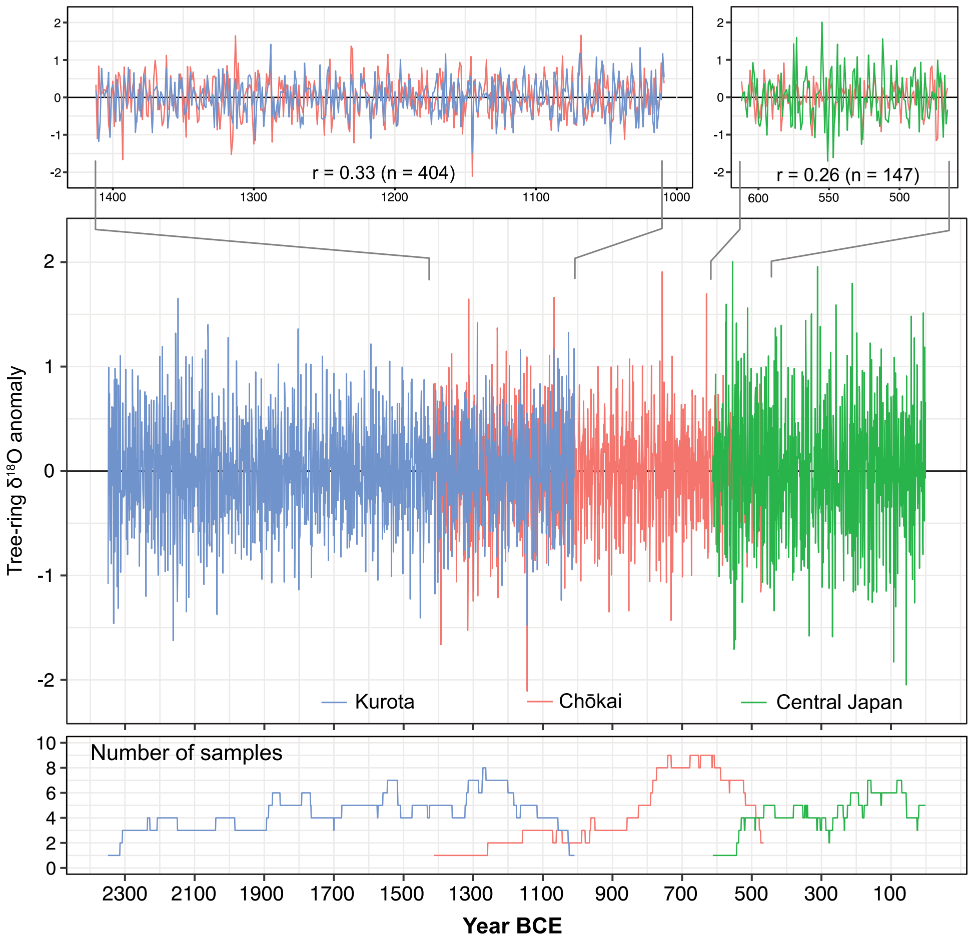

Figure 7 Cross-dated master chronologies for the Chōkai site, Kurota site, and central Japan (Nakatsuka et al. 2020; Sano et al. 2022), with the number of samples used for each chronology.

with the 2600-yr-long $\delta^{18}O$ chronology from central Japan (Nakatsuka et al. 2020; Sano et al. 2022), our tree-ring $\delta^{18}O$ dataset currently available in Japan covers the past 4354 yr (2349 BCE to 2005 CE). To our knowledge, this is the longest annually resolved tree-ring $\delta^{18}O$ record produced for Asia. The datasets developed in this study will contribute significantly to tree-ring dating of unearthed wood samples in Japan, and possibly in regions of Korea and China, as indicated by Sano et al. (2022). Furthermore, tree-ring $\delta^{18}O$ records from monsoonal Asia are known to be highly sensitive to hydroclimatic variability in summer (e.g., Nakatsuka et al. 2020; Yang et al. 2021). Although the present study focused on the high-frequency variability component required for precise dating of tree rings, low-frequency signals related to hydroclimate can also be extracted using other methods (Nakatsuka et al. 2020). High-resolution paleoclimatic records over the past several millennia are important to understand the timing and nature of climate variability, including abrupt changes such as the 4.2 ka event observed in many regions of the world (Cullen et al. 2000; Hong et al. 2003; Drysdale et al. 2006; Berkelhammer et al. 2013). Further paleoclimatic reconstructions will shed new insights into the decadal–centennial-scale climate variability over the past several millennia in Japan.

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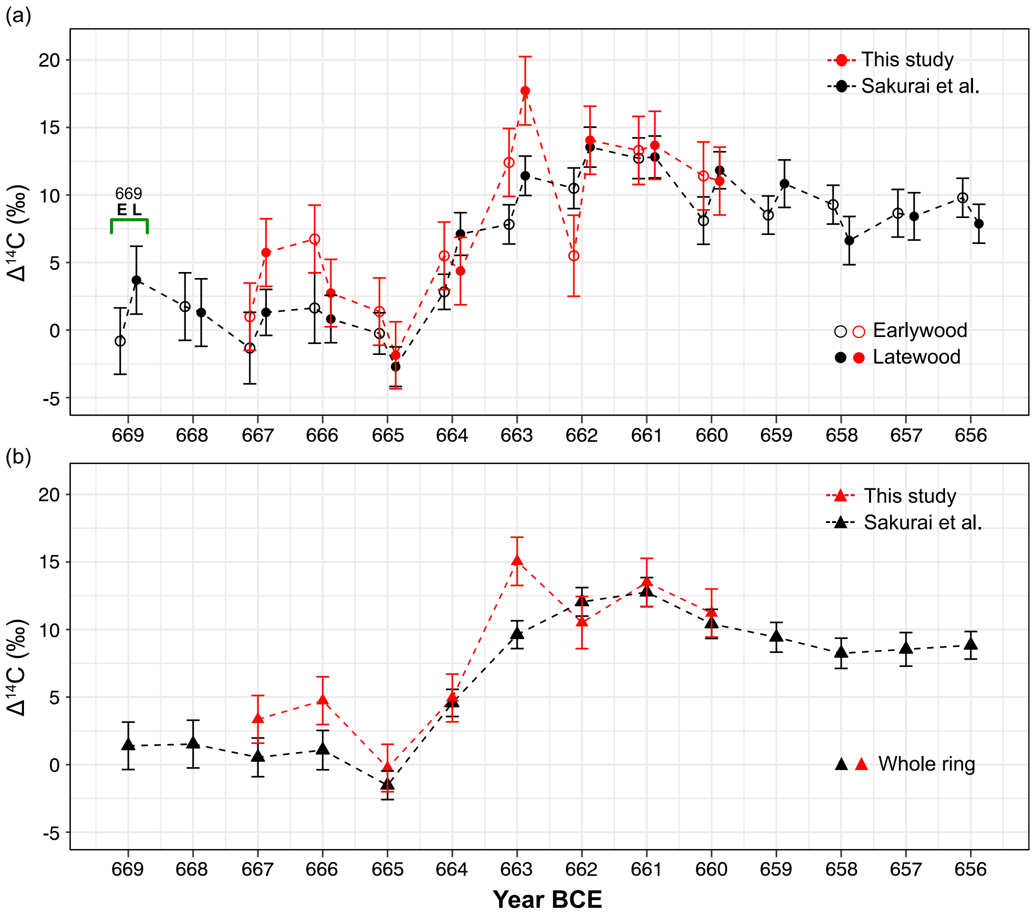

Figure 8 Comparison of the $\Delta^{14}C$ time-series for (a) earlywood and latewood, and (b) whole tree rings for our samples with those from Sakurai et al. (2020).

SUPPLEMENTARY MATERIAL

To view supplementary material for this article, please visit https://doi.org/10.1017/RDC.2023.29

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DECLARATION OF COMPETING INTERESTS

The authors declare no conflicts of interest.

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