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USING RAPID ATMOSPHERIC ¹⁴C CHANGES IN THE 7TH CENTURY BC TO PRECISELY DATE THE FLOATING CHRONOLOGY FOR PINE WOOD FROM JÓZEFOWO (NORTHERN POLAND)

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ABSTRACT. The floating dendrochronological sequence of pine wood from Józefowo, N. Poland was expected to cover the ∼660 BC radiocarbon (¹⁴C) excursion. The sequence was radiocarbon dated using the OxCal wiggle matching procedure and the IntCal20 calibration curve. 14C concentrations were measured in one-year α-cellulose samples from around 660 BC. The published data on the ∼660 BC 14C excursion from Grabie, Poland were used to absolute date the Józefowo chronology with 1-year accuracy. The results confirm the occurrence of a rapid increase in $\Delta^{14}C$ in 664/663 BC and its potential to be used as a fixing point for floating dendrochronological sequences.

KEYWORDS: dendrochronology, Miyake event, radiocarbon AMS dating, tree rings.

INTRODUCTION

The use of dendrochronology, one of the most precise methods of absolute dating allowing annual precision, is limited by the availability of standard chronologies for individual tree species. In Central Europe, the most common taxa are oak (Quercus robur L . and Quercus *petraea* L — anatomically indistinguishable wood) and pine (*Pinus sylvestris* L .). For oak, a chronology covering thousands of years has been compiled (Leuschner and Delorme [1988](#page-8-0); Becker [1993;](#page-8-0) Krąpiec [2001;](#page-8-0) Leuschner et al. [2002](#page-8-0); Friedrich et al. [2004\)](#page-8-0), while for pine the standard chronologies cover the last millennium (Heussner [1996](#page-8-0); Zielski [1997](#page-9-0); Szychowska-Krąpiec and Krąpiec [2005](#page-9-0); Szychowska-Krąpiec [2010\)](#page-9-0). Their extension is often limited by the poor availability of wood in both archaeological and geological sites. A chance to compile older chronologies is provided by subfossil pine wood found in peat deposits excavated during peat mining. Based on subfossil pine wood, a number of floating chronologies have been compiled in Poland and neighbouring areas, the time placement of which was determined by radiocarbon dating and the wiggle-matching technique (Pukienè [2001;](#page-8-0) Barniak et al. [2014](#page-8-0); Edvardsson et al. [2016a](#page-8-0); Krąpiec et al. [2016](#page-8-0); Achterberg et al. [2018\)](#page-8-0). In Central Europe, the lack of accumulation of pine trunks in peatlands in the last two millennia is characteristic, and it is probably climatically determined (Edvardsson et al. [2016](#page-8-0)b), making it impossible to dendrochronologically date older floating chronologies based on standards going up to the present day. A new opportunity to date such floating chronologies has been provided by the discovery of abrupt changes in radiocarbon concentrations ascending within a single year by Miyake et al. [\(2012](#page-9-0), [2013,](#page-9-0) [2014](#page-8-0), [2017\)](#page-8-0). Such events from 774–775 AD and 994–995 AD have been used to date with annual accuracy floating chronologies or to confirm dendrochronological dates from Switzerland (Wacker et al. [2014\)](#page-9-0), Japan (Hakozaki et al. [2018](#page-8-0)), and Poland (Krąpiec et al. [2021](#page-8-0)).

Another rapid increase in radiocarbon concentration has been noted around 660 BC and confirmed by Park et al. ([2017\)](#page-9-0), Rakowski et al. [\(2019](#page-9-0)), Koldobskiy et al. [\(2023](#page-8-0)), and Sakurai et al. ([2020](#page-9-0)). Similarly, to events from 774–775 AD and 994–995 AD, an increase in the production rate of 10 Be and 36 Cl has been noted, confirming the solar origin of this event

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Figure 1 Location of Józefowo village $(54.135763 \text{ N}, 19.818319 \text{ E})$ and Grabie village (50.0391 N, 19.992 E).

(O'Hare et al. [2019](#page-9-0)). The event occurring around 660 BC is characterized by a prolonged increase in the Δ^{14} C values between 665 and 661 BC, with differences in radiocarbon concentrations (Δ^{14} C) from 665 BC and 664 BC presented in the literature as 8.9 ± 0.4‰ (Park et al. [2017\)](#page-9-0), 8.3 \pm 2.9‰ (Rakowski et al. [2019](#page-9-0)), and 9.8 \pm 2.2‰ (Sakurai et al. [2020\)](#page-9-0).

The amplitude of the event observed between 665 BC and 661 BC was $9.3 \pm 2.6\%$ in the data of Park et al. ([2017\)](#page-9-0), 19.3 \pm 3.5‰ in the data of Rakowski et al. ([2019\)](#page-9-0) and 16.3 \pm 2.1‰ in the data of Sakurai et al. ([2020\)](#page-9-0). For all datasets, the minimum occurred around 665 BC and the maximum around 661 BC.

In this publication, we show for the first time the successful use of the Miyake event from 664–663 BC to date floating pine chronologies, which will serve as a reference to build a long absolute pine chronology from northern Poland for the first millennium BC, which is an important period for archaeological research in the region (Rose et al. [2022\)](#page-9-0).

MATERIALS AND METHODS

All the used wood samples are in the archives of the AGH Dendrochronology Laboratory in Kraków. The pine (Pinus sylvestris L.) wood samples were taken from a peat bog exploited by the peat mining company AGARIS Poland in Józefowo, located about 25 km east of Elbląg (N Poland, GPS: 54.135763 N, 19.818319 E) (Figure 1). In 2013–2021, more than 500 wood samples were taken from the trunks of subfossil pines in the form of round discs (wood slices) cut with a chainsaw. Annual tree rings were precisely measured (with an accuracy of 0.01 mm) using Dendrolab 1.0 equipment (Zielski and Krąpiec [2004\)](#page-9-0). The measured tree-ring sequences

Figure 2 Diagram of the temporal extent of growth sequences forming the 2JOZ_SA3 pine chronology from Józefowo.

were processed using the TREE-RINGS software (Krawczyk and Krąpiec [1995\)](#page-8-0) and the TSAP computer program (Rinn [2005](#page-9-0)).

Crossdating of the sample was statistically validated using "t-coefficient" (Baillie and Pilcher [1973\)](#page-8-0) and "Gl coefficient" (Gleichläufigkeit; Eckstein and Bauch [1969\)](#page-8-0). The accuracy of the measurements and the quality of the series were verified by COFECHA software (Holmes [1999](#page-8-0)).

From the 78 individual best-correlated sequences, a local 2JOZ_SA3 chronology covering 292 years was constructed (Figure 2). For absolute dating, 10 tree rings were selected from two samples, 2JOZ228 and 2JOZ504. The dendrograms (annual increment width graphs) of these samples have a clear resemblance to the 2JOZ_SA3 chronology: 2JOZ228 between 1 and 168 relative years with t=12, Gl=70%***, and 2JOZ504 between 131-278 relative years, with t=9.5, Gl=73%***, where the t-value represents the significance of the correlation of two series in relation to their overlap (Baillie and Pilcher [1973](#page-8-0)). The Gleichläufigkeit (Gl) was developed by Eckstein and Bauch [\(1969](#page-8-0)) as a special tool for crossdating of tree-ring series. The degree of similarity based on the positive or negative trend of each width is expressed as a percentage of the number of intervals.

The Green [\(1963](#page-8-0)) protocol was used to extract a-cellulose from wood. Additionally, this method was modified by using an ultrasonic bath (Pazdur et al. [2005\)](#page-9-0), with HCl instead of acetic acid to avoid any possible acetylation (Nemec et al. [2010](#page-9-0)).

After the pretreatment, around 4 mg of α -cellulose, extracted from each sample (one ring, one trunk) was combusted to $CO₂$ and subsequently reduced to graphite (Krapiec et al. [2018](#page-8-0); Wiktorowski et al. [2020\)](#page-9-0). The resulting mixture of graphite and Fe powder was pressed into a target holder for AMS ¹⁴C measurements. All the prepared targets contained approximately 1 mg of carbon and were measured at the Center for Applied Isotope Studies at the University of Georgia, USA (laboratory code UGAMS; Cherkinsky et al. [2010](#page-8-0)). The 14C concentrations

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Figure 3 ¹⁴C wiggle matching of the floating pine chronology 2JOZ_SA3. Calibrated positions of the first and last rings of the pine chronology 2JOZ_SA3 (lower part of figure).

are reported as Δ^{14} C in per mil (‰) deviations from the standard sample, 0.7459 activity of NBS oxalic acid (SRM- 4990C). Vitrinite was used as a background sample. The background sample was subjected to standard acid-alkali-acid pretreatment. Age correction and isotopic composition correction were calculated following formulas presented elsewhere (Stuiver and Polach [1977](#page-9-0)). The correction for isotopic composition was made based on $\delta^{13}C$, measured with an AMS system.

UGAMS#	Sample name and no. of annual ring	Relative year of the chronology	${}^{14}C$ age (BP)
58079	2JOZ 228/12	12	2683 ± 20
58080	2JOZ 228/46	46	2622 ± 20
58081	2JOZ 228/86	86	2475 ± 22
59460	2JOZ 504/35	165	2544 ± 24
59461	2JOZ 504/40	170	2441 ± 25
59462	2JOZ 504/45	175	2477 ± 25
59463	2JOZ 504/50	180	2458 ± 24
59464	2JOZ 504/55	185	2477 ± 24
59465	2JOZ 504/60	190	2449 ± 25
59466	2JOZ 504/65	195	2476 ± 24

Table 1 List of radiocarbon dates for pine wood samples used for wiggle matching from the Józefowo site.

The age of the floating chronology was obtained using the wiggle-matching technique and the results are presented in Figure [3.](#page-3-0) The start of the Józefowo 2JOZ_SA3 sequence was determined by the wiggle-matching method to be between 845–825 cal BC (2 σ). The end of the sequence falls at 554–534 cal BC (2 σ).

Preliminary matching of the floating chronology 2JOZ_SA3 to the absolute time scale was performed using the wiggle-matching method in the OxCal v4 program (Bronk Ramsey et al. [2010\)](#page-8-0).

Subfossil oaks (*Quercus robur L*.) were recovered from a site in a gravel pit by the Vistula River in the village of Grabie near Kraków and were control series, of known calendar ages and known changes of radiocarbon concentrations around 660 BC (Rakowski et al. [2019](#page-9-0)).

To precisely determine the calendar age of the studied trunk $2JOZ504$ from Józefowo, the measured ¹⁴C concentration values expressed in pMC (Table [2](#page-5-0)) were converted into Δ^{14} C while shifting the entire J *o*zefowo sequence year by year. The resulting $\Delta^{14}C^{Józefow}$ values were then compared to Δ^{14} C^{G58} values for the G58 trunk (Rakowski et al. [2019\)](#page-9-0). The G58 tree from Grabie was used for this purpose because it was taken from the absolutely dated dendrochronological sequence and is an excellent reference point for the period and region. For each shift (k), the sum of the squared difference of the $\Delta^{14}C$ for Józefowo and G58 trunks was calculated. The sum of the squared differences was normalized $(nSSD)$ by dividing it by the number of years (n) for which the difference could be calculated:

$$
nSSD(k) = \frac{\sum_{i=1}^{n} (\Delta^{14} C_i^{Józefowo} - \Delta^{14} C_i^{G58})^2}{n}
$$

The best fit of the J *o*zefowo sequence to the G58 sequence is described by the minimum of $nSSD(k)$.

RESULTS AND DISCUSSION

In the first stage, the measurements were made for ten single-ring, one-trunk (either JOZ228 or JOZ504 tree), α -cellulose samples, collected from the increments representing 12, 46, 86, 165,

UGAMS#	Sample name and no. of annual ring	pMC	$\Delta^{14}C$ (%o)	${}^{14}C$ age (BP)	Obtained age BC)	(calendar year Astronomical year
59466	JOZ 504/195	73.48 ± 0.23	4.4 ± 2.3	2476 ± 24	634	-633
59465	JOZ 504/190	73.72 ± 0.23	8.3 ± 2.3	2449 ± 25	639	-638
59464	JOZ 504/185	73.46 ± 0.23	5.5 ± 2.3	2477 ± 24	644	-643
60530	JOZ 504/184	73.30 ± 0.19	3.3 ± 1.9	2495 ± 20	645	-644
60529	JOZ 504/183	73.33 ± 0.19	3.9 ± 1.9	2491 ± 20	646	-645
60528	JOZ 504/182	73.14 ± 0.19	1.5 ± 1.9	2512 ± 20	647	-646
60527	JOZ 504/181	73.50 ± 0.19	6.5 ± 1.9	2473 ± 20	648	-647
59463	JOZ 504/180	73.64 ± 0.23	8.5 ± 2.3	2458 ± 24	649	-648
60526	JOZ 504/179	73.44 ± 0.19	5.9 ± 1.9	2479 ± 20	650	-649
60525	JOZ 504/178	73.48 ± 0.19	6.5 ± 1.9	2475 ± 20	651	-650
60524	JOZ 504/177	73.86 ± 0.19	11.9 ± 1.9	2433 ± 20	652	-651
60523	JOZ 504/176	73.01 ± 0.19	0.4 ± 1.9	2526 ± 20	653	-652
59462	JOZ 504/175	73.46 ± 0.23	6.7 ± 2.3	2477 ± 25	654	-653
60522	JOZ 504/174	74.03 ± 0.19	14.6 ± 1.9	2415 ± 20	655	-654
60521	JOZ 504/173	73.75 ± 0.19	10.8 ± 1.9	2446 ± 20	656	-655
60520	JOZ 504/172	73.35 ± 0.20	5.5 ± 2.0	2489 ± 21	657	-656
60519	JOZ 504/171	74.02 ± 0.19	14.8 ± 1.9	2416 ± 20	658	-657
59461	JOZ 504/170	73.79 ± 0.23	11.8 ± 2.3	2441 ± 25	659	-658
60518	JOZ 504/169	73.74 ± 0.20	11.2 ± 2.0	2447 ± 21	660	-659
60517	JOZ 504/168	74.23 ± 0.20	18.1 ± 2.0	2393 ± 21	661	-660
60516	JOZ 504/167	74.04 ± 0.19	15.6 ± 1.9	2414 ± 20	662	-661
60515	JOZ 504/166	73.92 ± 0.19	14.1 ± 1.9	2427 ± 20	663	-662
59460	JOZ 504/165	72.85 ± 0.23	-0.5 ± 2.3	2544 ± 24	664	-663
61918	JOZ 504/164	72.42 ± 0.26	-6.3 ± 2.6	2592 ± 29	665	-664
61917	JOZ 504/163	72.57 ± 0.25	-4.2 ± 2.5	2576 ± 28	666	-665
61916	JOZ 504/162	73.03 ± 0.26	2.4 ± 2.6	2524 ± 28	667	-666
61915	JOZ 504/161	73.35 ± 0.25	6.8 ± 2.5	2490 ± 28	668	-667
61914	JOZ 504/160	73.08 ± 0.26	3.3 ± 2.6	2519 ± 29	669	-668

Table 2 Δ^{14} C in the Józefowo samples calculated for the fitted period.

Figure 4 Fitting of Józefowo $\Delta^{14}C$ sequence to G58 sequence (Rakowski et al. [2019](#page-9-0)).

170, 175, 180, 185, 190, and 195 relative years in chronology $2JOZ$ _SA3. The results of the ${}^{14}C$ age determinations are presented in Table [1.](#page-4-0)

Dating results indicate that the increments between 170–180 relative years contained the diagnostic signal from 664/3 BC. In the two following sessions, 14 C concentrations were measured in α -cellulose from rings corresponding to relative years 160–185 (Table [2](#page-5-0)).

The precise fixing of the Józefowo floating chronology was possible based on the data presented in Figure 4. The minimum value of the $nSSD(k)$ is observed for the year 634 BC. However, the results for 635 and 634 BC do not differ significantly. Close inspection and visual comparison of the Józefowo dataset, the Sakurai et al. (2020) (2020) dataset and new, interannual results for the Grabie trunk (Rakowski et al. [2023\)](#page-9-0) presented in Figure [5](#page-7-0) suggests that 634 BC is the correct fixing point. The data from J ozefowo presented in Figure [5](#page-7-0) show a similar pattern as well as Δ^{14} C values to the G58 data sequence, the results of Sakurai et al. ([2020\)](#page-9-0), the data of Park et al. ([2017\)](#page-9-0) and new results for the Grabie trunk (Rakowski et al. [2023\)](#page-9-0). In the Józefowo sequence, the increase in average Δ^{14} C value between ranges 666–664 BC and 663–657 BC is 14.6 \pm 5.8‰, which is consistent with the data presented in Sakurai et al. ([2020\)](#page-9-0), Rakowski et al. ([2019\)](#page-9-0), Rakowski et al. [\(2023](#page-9-0)) and Park et al. [\(2017](#page-9-0)). The rapid increase in Δ^{14} C observed in Józefowo occurred in one or two years and is more abrupt than the one observed by Park et al. [\(2017](#page-9-0)) but similar to the peak observed in Japan (Sakurai et al. [2020](#page-9-0)) and Poland (Rakowski et al. [2019,](#page-9-0) [2023\)](#page-9-0).

This match shows that the 164th tree-ring of the 2JOZ_SA3 chronology represents 665 BC. This allows us to determine that the chronology covers the period 828–537 BC. The value of chronology 2JOZ_SA3 for absolute dating is difficult to overestimate; it serves not only as a tool for precise dating, but also as a potential record to be used in dendroclimatic studies both in traditional terms, using tree ring width variability, as well as carbon and oxygen composition of stable isotopes. Similar method has been already successfully applied by Pearson et al. ([2020\)](#page-9-0).

Figure 5 Józefowo $\Delta^{14}C$ sequence (red crosses) fitted to the G58 sequence (blue stars) and compared with data from Park et al. ([2017\)](#page-9-0) (blue-green circles), new Grabie sequence (green stars) (Rakowski et al. [2023](#page-9-0)), Sakurai et al. [\(2020\)](#page-9-0) sequence (black crosses) and IntCal20 data (grey shaded area) (Reimer et al. [2020](#page-9-0)).

It provides a repertoire that can enable the compilation of a long regional chronology for NE Poland for the first millennium BC, based on the available collections of subfossil pine samples from the surrounding peatlands covering more than 1900 samples.

CONCLUSIONS

The Δ^{14} C rapid increase in the Józefowo sequence confirmed the presence of the radiocarbon excursion ∼660 BC. The excursion is more visible in the Józefowo and Grabie data than in the data published by Park et al. ([2017\)](#page-9-0).

It has been demonstrated that the Δ^{14} C increase of ~660 BC can be used for dating a tree-ring sequence to an accuracy of 1 year. The floating tree-ring chronology from Józefowo was preliminarily fixed to the absolute time scale by radiocarbon dating using the wiggle-matching method. Later, the fixing was supported by Δ^{14} C matching to the known abrupt ¹⁴C increase in the atmosphere of 664/3 BC. The refined fixing allowed for 1-year accuracy. The absolute time period covered by the floating chronology of the Józefowo 2JOZ_SA3 sequence is between 828 BC and 537 BC. The method can be used to fix other floating dendrochronologies from this period.

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