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Potentiality of Charcoal as a Dendrochronological and Paleoclimatic Archive: Case Study of Archaeological Charcoal from Southeastern Altai, Russia

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Abstract: Archaeological charcoal from ancient nomad iron-smelting furnaces collected in the highland southeastern Russian Altai has great potential as a material for tree ring analysis. Dendrochronological dating was applied to 355 viable samples (>80% of the 448 collected ones), prepared using a new protocol. Individual tree ring series of 155 (~43%) samples were used to construct nine floating chronologies from 76 to 290 rings long. The archaeological and radiocarbon data on charcoal that fueled the hearths of the Kosh-Agach type bracket the floating tree ring chronologies between the second and tenth centuries AD. The results demonstrate that long tree ring "steppe" chronologies can be obtained for intermontane basins in the arid zone of Southern Siberia, using the analysis of charcoal samples. A strong climate signal imprinted in the annual growth of trees allowed for crossdating samples with relatively few rings. The revealed common climate signal for larches from different locations indicates similar paleoclimate conditions of their growth despite the strong modern southeastward aridization trend in the region, which was not so pronounced ca. 1.5 ka ago. The further matching of these chronologies to the calendar timeline will provide reference for the precise comparison of climatic conditions in the floors of intermontane basins and in the flanking mountains.

Keywords: archaeological charcoal; dendrochronology; iron-smelting furnace; long tree ring chronology; Russian Altai

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1. Introduction

Time is the key parameter in studying geological, geographic, or climate history, as well as the history of human societies. The events recorded in archaeological artefacts, rock samples, landforms, or tree rings remain vague unless they are arranged chronologically. Dendrochronology is a dating method with advantages over radiocarbon, thermal luminescence, or other radiometric techniques, as the ages it can provide are accurate to years or even seasons.

Dendrochronological dating implies the analysis of information recorded in tree rings using various criteria of annual growth: ring width, mechanical and chemical properties, and the anatomical structure of wood, etc. Most of the natural phenomena and archaeological artefacts are dated in greenwood cores or in wood cross sections of dead trees. Wood can be preserved for thousands of years in arid climates [1]; in submarine anoxic conditions; when buried rapidly under moving glaciers, landslides or rockslides, mudflows, etc.; during geohazard events [2]; or at archaeological funerary sites in permafrost areas [3–5]. The preserved anatomic and structural features of annual rings can also be found in charcoal from areas of growing or extinct forests. Carbonization, which results from wildfires or combustion in industries and households, prevents trees from rotting and, thus, ensures the prolonged preservation of the material.

The first anthracological reports from the Mas d'Azil cave in Ariège (France) date back to the 1900s [6], while the paleoclimatic implications of charred vegetation were recognized in the 1940s [7,8]. At the same time, charcoal has long remained out of use in dendrochronology because the fragile and crumbling samples were hard to prepare for analysis. Obtaining thin 3D specimens was long painstaking work up until the 1970s. The related methodological issues have been discussed since the middle of the 20th century [7,9]. The amount of available samples increased and their quality improved with the advent of microscopy, which saved much time and effort for charcoal processing. However, despite the experience gained in analyzing both charcoal [10–14], etc., and combined charcoal + wood [15–17] samples, the preparation protocols for dendrochronological dating are still imperfect and poorly formulated, and the potentiality of charcoal as a unique chronological and paleoclimatic archive remains underestimated.

The problem in using charcoal for tree ring analysis is that the samples represent shrunk wood and are small and crumbly. Wood shrinking in carbonization leads to a loss of 12–20% and 7–13% in the radial and axial dimensions, respectively [18], while the small size and fragility of the samples pose the greatest problems. Thus, much caution is required in the choice of charcoal material preserved during burial, as well as special protocols for sampling, sample preparation, and measurements. Charcoal samples contain many fewer tree rings than the saw-cut or cored wood due to wood fragmentation during burning and destructive environment effects. Combusted wood retains mainly the heartwood but lacks sapwood and pith. In this respect, the presence of prominent age trends in such samples has to be taken into account in dendrochronological applications. Furthermore, obtaining smooth transversal planes with perfectly visible rings is challenging. The methods of charcoal preparation for tree ring analysis have been a subject of discussion for a long time, but the main recommendations have been limited to fixing the samples with a thermo-retractable sheath [19] or an adhesive tape. Other approaches imply solidifying the samples in resin [18,20,21], impregnating with various compounds, or freezing and subsequent trimming [11]. However, the freezing procedure can reduce the tree ring width.

Investigating the potential of charcoal in the southeastern Russian Altai area (SE Altai), Southern Siberia, Russia, an arid mountainous terrain in Central Asia, for tree ring dating was motivated by its abundance at the archaeological sites and in Holocene sediments. We suggest a sample preparation protocol that allows the use of numerous fragments of charcoal from iron-smelting hearths of ancient Altai nomads. The collection prepared with the new method was used for constructing long floating chronologies (in contrast to absolute chronologies, these are tree-ring histories whose beginning- and end-dates are not known), and the reported results can provide reference for chronological and climatic reconstructions.

2. Study Area

The mountainous southeastern part of Russian Altai (SE Altai, Figure 1) includes the Chuya and Kurai intermontane basins and the bordering mountains. The rugged surface topography of the area results from Cenozoic orogenic movements [22,23]. The floor of the Chuya Basin, the largest in the Altai Mountains, lies at 1750–2000 m a.s.l., while the elevation of the Kurai basin floor is 1500–1600 m a.s.l. The ranges rise up to 3500–4200 m a.s.l. and are high enough for the formation of glaciers. The number and extent of Pleistocene glaciations remains a subject of discussion [24], and the middle–late Holocene glacier advance was inferred to have occurred in three events [2,25].

Southeastern Altai is an area of ultra-continental arid climate with permafrost. The mean annual air temperature is -5.2 °C, according to records from a weather station located in the Chuya Basin. The mainly western and partly northern moisture transport leads to a southeastward aridity increase; the mean annual precipitation is 150–200 mm in the Kurai

Basin and only 80–120 mm in the Chuya Basin [26]. The rocky steppe on the floor of the Kurai Basin grades into taiga vegetation on the slopes. The mountains flanking the Chuya Basin are grown with sporadic forest patches in the west and are almost bare in the eastern part. The flat-topped highlands are covered by arid mountain steppes. Alpine landscapes predominate in the belt of the higher mountains, with mountain tundra and tundra–steppe vegetation grading upwards into a glacial zone.

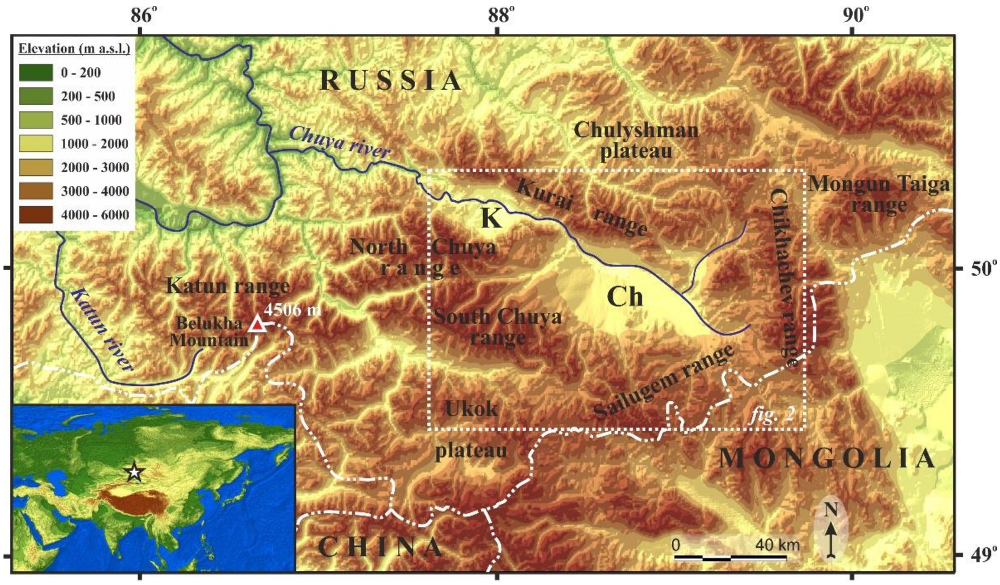

Figure 1. Location map of study area in mountainous southeastern Russian Altai and adjacent territories. Abbreviations stand for names of intermontane basins: K = Kurai, Ch = Chuya. Dotted box frames the Chuya–Kurai metallurgic province (Figure 2).

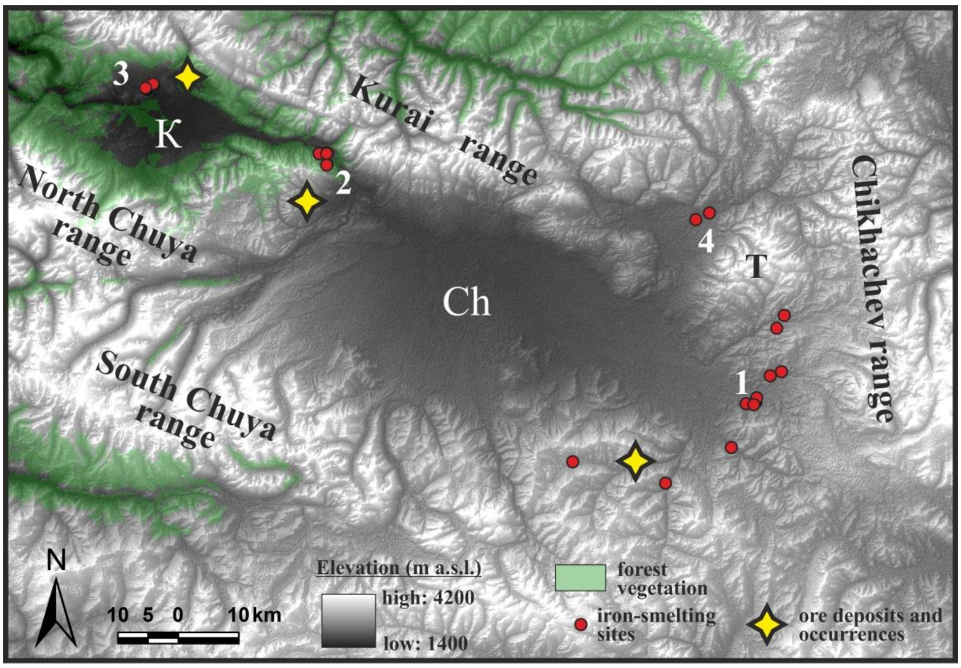

Figure 2. Location of some known ancient iron-smelting sites, iron deposits and occurrences, and modern forest fuel resources within the Chuya–Kurai ferrous metallurgy province. Arabic numerals stand for sampling sites in river valleys: 1 = Yustyd valley; 2 = Chuya valley, near Kuektanar mouth; 3 = Turgun valley; 4 = Buguzun valley; and Derek site. Abbreviations stand for basin and range names: Ch = Chuya Basin, K = Kurai Basin, T = Talduairy Massif.

The forest growth in the mountains is generally controlled by air temperatures during the vegetation season. For instance, the mean July temperature in the North Chuya Range is 9 °C, which is the forest thermal limit [27]. Mature forests spread within 2220–2330 m a.s.l., and single trees or young trees are found up to 2400–2470 m a.s.l. under favorable conditions. The present upper timberline is slightly above or equal to the altitudes of glacier toes. The forest growth is additionally limited by moisture shortage and depends on the level of groundwaters (often associated with permafrost) and slope exposure. As a result, Siberian pine (*Pinus sibirica* Du Tour) is no longer a forest-forming species near the upper timberline, though it grows on the slopes of the Katun and North Chuya Ranges, together with larch (*Larix sibirica* Ledeb). Larch patches grow sporadically on the northern slope of the South Chuya Range (mainly in its western part); on the northwestern slope of the Talduairy Massif, at the northern border of the Ukok Plateau; and in the Mongun–Taiga Range.

3. Nomad Iron-Smelting Industry in SE Altai and Natural Prerequisites for the Use of Charcoal in Tree Ring Analysis

Many Late Paleolithic to medieval archaeological sites in southeastern Russian Altai have been well preserved due to the arid climate, the presence of permafrost, and scarce population [4,28], etc. Among them, the numerous sites of ancient iron-smelting industry (Figure 2), including 29 sites in the Chuya–Kurai province of ferrous metallurgy distinguished by Ziniakov [29]. The developed iron smelting in the area left a record in the geographic names: *Kuektanar*, the name of a sampling site in this study, is from the Altai language for *putting on chain armor*. Currently, the former hearths located mostly on river terraces are barely recognizable on the surface being exposed to erosion and degradation [30].

Most of the ancient iron-smelting sites in the area are of the Kosh-Agach type, referred to as 'box-shaped', 'linear', or 'rectangular' bloomery hearths [29,31] of multiple use. Furnaces of this type were most often placed at the edge of river terraces, in a slightly inclined position for discharging liquid slag into a pit connected with the hearth via a special channel (Figure 3), which allowed the use of the natural air flow along the valley. The hearths had a rectangular shape, 100–180 cm long and 40–80 cm wide, with a volume of 1 m^3, and commonly existed in groups of two to six. Their top was made of clay while the box-shaped underground part was lined with stone plates. The hearth length and volume were extended due to numerous tuyere holes (up to 26) on the long side approximately 1 m above the bottom, 45° to the horizon.

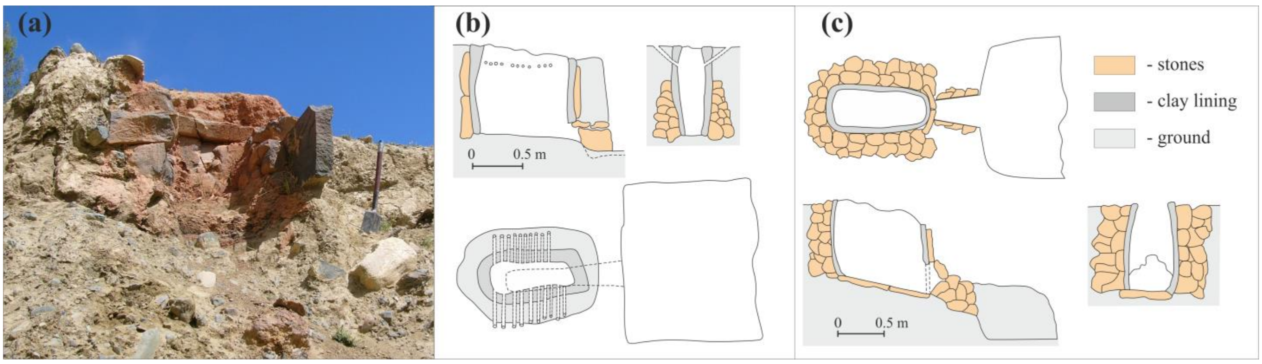

Figure 3. (**a**) Survived fragment (back and side walls) of a hearth at the Kuektanar-1 site. The shovel is 0.6 m long for scale, photograph by Agatova; (**b**,**c**) sketches of hearths of the Kosh-Agach type at Turgun (No. 3 in Figure 2) and Kuektanar-1 (No. 2 in Figure 2) sites, respectively, after [29].

The development of ferrous metallurgy in the area was maintained by the abundance of iron deposits and forests, which, at that time, grew also in the currently deforested arid eastern periphery of the Chuya Basin [30,32]. Ancient nomads consumed much wood for iron production [29]. Charcoal was used for ore dressing by annealing and for smelting proper, while the timber/ore ratio in the hearths during smelting was more than 3:1, and the charcoal output from combustion was approximately 30–65%. The bloomery hearths of the Chuya–Kurai province were fueled with larch trees (*Larix sibirica* Ledeb). The age of the trees exceeded 100 years, while the extant larch trees in the Chuya and Kurai basins are up to 400–450 years old or more [33]. Therefore, the dendrochronological dating of charcoal samples from the Altai hearths can be expected to yield long chronologies.

Voluminous wood combustion for the large-scale iron-smelting industry of ancient nomads produced a great number of localized charcoal occurrences as carbonized fragments of coeval trees cut within one or two of the same years. The area, with its severe climate and southeastward increasing aridity, mostly grows *Larix sibirica* Ledeb, which is the only forest-forming tree species in the mountains around the Chuya Basin and one of several main tree species in the Kurai Basin surroundings. The harsh climate produces prominent signals in the radial tree growth records but reduces the width of annual rings, down to a single cell in some years or even to zero (missing rings). The small width of annual rings poses problems for crossdating but narrow rings are much more numerous, which makes even small samples suitable for tree ring dating.

Thus, the iron-smelting industry of ancient nomads and the severe climate created favorable prerequisites for the use of charcoal from different parts of the Chuya–Kurai province for tree ring analysis and for building long tree ring chronologies (TRC), due to

- the abundance and concentrated location of charcoal deposits;
- the similar ages of carbonized wood fragments in collections from one site;
- *Larix sibirica* Ledeb being a single or one of the main forest-forming species;
- the long lifespan of larch trees in the area (400–450 years);
- the prominent climate signals in the radial tree growth record;
- the large number of annual rings even in small charcoal fragments.

4. Operation of Iron-Smelting Hearths in SE Altai: Time Constraints from Archaeological Evidence and Radiocarbon Ages of Charcoal

The time constraints on the use of hearths in the Chuya–Kurai iron-smelting province of SE Russian Altai and the respective time span of the charcoal-built floating tree ring chronologies were obtained from archaeological evidence and radiocarbon dating.

The onset of the metallurgic industry in the region dates back to the time when people mastered the bloomery process for making iron. Although unique artefacts made of meteorite iron were found at sites of the ca. 3000–2000 BC Afanasiev culture [34,35], long before the Iron Age, the iron industry proper did not exist at that time yet. Frequent findings of iron artefacts are from the Pazyryk culture of the Scythian period, such as tools, horse harness elements, and arms at burial sites from the 5th to 3rd centuries BC [36,37]. Meanwhile, there was no evidence either for the existence of smelting industry then [38].

Excavations by Ziniakov [29] revealed two types of bloomery furnaces in the Chuya–Kurai province: widespread box-shaped hearths of the Kosh-Agach type (see above) and a few hearths with an oval-shaped back wall were found in the Yustyd valley, which were possibly made in the Hunnu (Xiongnu) time, 2nd century BC to 5th century AD [39].

In general, only a few artefacts may provide age reference as it is hard to apply the traditional archaeological methods to the timing of hearths within the Chuya–Kurai province because the smelting sites were located outside the residential communities. Nevertheless, some findings in box-shaped furnaces allow for an idea of when they were used [29]. A fragment of a ceramic vessel rim decorated with thumbnail depressions and pits [29] was found inside a hearth at the Kuektanar-1 site (Figure 2). Pottery with such a decoration was found among artefacts of the 6–7th centuries AD at the Kudyrgue burial site [40], as well as at some other Altai sites of the 7–9th centuries AD [41].

Another example is a harness buckle with a tongue in a joint, such as those known from other Altai sites of the 7–8th centuries AD [41], which was discovered in the pit filling of a box-shaped furnace from the Buguzun valley (Figure 2). Such buckles are also known from sites of the 6th through 8th centuries AD in the neighboring Tyva area [42,43]. The Buguzun valley artefacts also included a three-lobed arrowhead similar to those from the

Kyrgyz sites of the 6–8th centuries AD [44]. Two more findings were reported from a furnace at the Derek smelting site: an iron fire striker and a stirrup fragment. The similarity of these findings to artefacts from other Altai areas [40] implies the age limits of the 7–8th and 6–10th centuries AD, respectively, i.e., the ancient Turk period (the late 5th through 11th centuries AD).

Additional age constraints on the box-shaped hearths from the Chuya–Kurai province were provided by their features [29]. They lack immediate analogs in adjacent territories, but some elements are comparable with their counterparts of the 6–9th centuries AD from the Khakassia–Minusa Basin [45] and with the Saltov-type furnaces of the 8–9th centuries AD [46].

Thus, the available archaeological evidence shows that the box-shaped bloomery hearths of the Kosh-Agach type in the Altai region were used in the ancient Turk period between the 6th and 10th centuries AD [29].

The available radiocarbon age constraints are due to obtaining 30 dates for charcoal fragments from hearths in the Chuya–Kurai province (Supplementary Table S1): 18 dates for samples from the Chuya valley (Kuektanar mouth); 11 dates for charcoal from the Yustyd Valley, including a sample from a Hunnu pottery kiln; and 1 more date for samples from the Turgun valley (Figure 2). The radiocarbon dates confirm the timing by different authors, who place the use of bloomery hearths in the Chuya–Kurai province within the ancient Turk (late 5th century BC through 11th century AD [30,32,47] and, possibly, Hunnu (2nd century BC through 450 AD) [31,39] periods.

5. Results

5.1. Preparation of Charcoal Samples for Tree Ring Analysis

5.1.1. Sampling

Our collection comprises fragments of carbonized wood (charcoal) and slag with high charcoal percentages sampled during the field campaigns from 2011 to 2022 from eroded hearths, slag dumps, and river terrace surfaces at archaeological iron-smelting sites in the Kurai and Chuya Basins, as well as in the Chuya River valley between them (Figure 4).

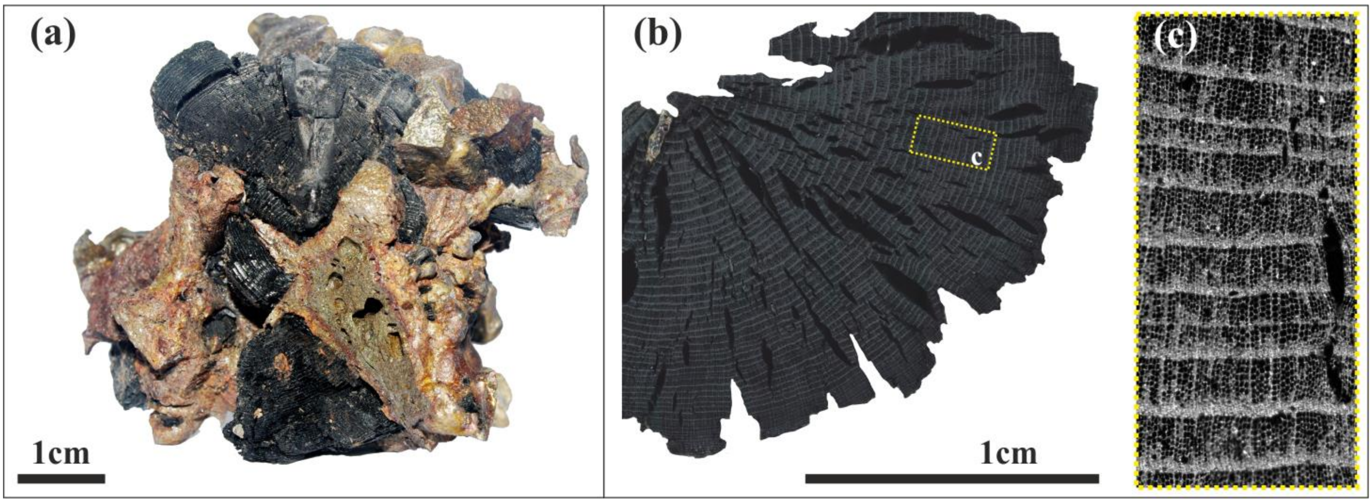

Figure 4. Photographs of slag with charcoal inclusions (**a**) and prepared charcoal sample (**b**,**c**).

The samples from the Yustyd valley were 40–50 cm slag pieces with numerous charcoal inclusions, found on the ground surface along the edge and at the toe of floodplain terrace I, at the site excavated by Ziniakov in 1978 [29]. Although the slag had stayed on the surface for 35–40 years, it was preserved well enough for the transportation and preparation procedures.

Sampling in the Chuya valley was performed in the mouth of the Kuektanar River, at the Kuektanar-2 and Kuektanar-1 sites, likewise, studied before by the team of Ziniakov [29]:

slag pieces with charcoal inclusions scattered on the terrace surface at the Kuektanar-2 site and charcoal fragments from rapidly degrading hearths of the Kuektanar-1 site (Figure 3a). Later, the collection was extended with samples from a dump that was preserved after the excavation campaign of 2019 by Vodyasov et al. [31] at the Kuektanar-2 site.

Note that the size of the charcoal fragments is not the only guide in field sampling. A close visual inspection is required additionally to estimate the approximate number of tree rings and the fragility of charcoal samples. In general, the three properties jointly determine the feasibility of sample preparation and further measurements along one or several radial directions for subsequent crossdating of the individual tree ring series.

5.1.2. Sample Preparation

The most difficult things in the preparation of charcoal samples for tree ring analysis are to separate coal from slag and to obtain smooth transverse planes. Breaking along natural fractures causes significant losses in charcoal and produces very rough surfaces. Manual surfacing under a microscope with the subsequent use of contrasting agents is labor-consuming and hardly applicable to large collections.

The suggested protocol was tested at the Siberian dendrochronological laboratory of the Siberian Federal University (Krasnoyarsk, Russia). The procedures ensure fast and high-quality processing of large collections with samples of almost any size and shape, using ordinary sanders and optical microscopes. Namely, the procedures include:

- cutting (breaking) slag samples with a pick hammer, a diamond sanding disc, or a band saw, as in [19]. Altogether, 448 charcoal specimens were extracted in this way;
- grinding and polishing to obtain transverse planes of the largest possible surface area, with clearly visible growth rings, using a belt and disc sander (belt P600 or P1000 and disc P1000) for hard samples;
- cleaning the surfaces of pre-dried samples by vacuuming (up to 5 bar depending on fragility) in order to remove coal dust, which fills tracheids and masks the cell structure. The dried samples should have residual moisture contents of at most 7%, otherwise the dust particles agglutinate, harden upon drying, and impede the cleaning;
- checking the surface quality under a Leica M80 stereo zoom microscope and repeating the grinding, polishing, or cleaning procedures, if necessary.

This protocol was applied to prepare 355 viable samples for tree ring analysis out of 448 samples in the initial collection from the SE Altai archaeological smelting sites.

5.1.3. Digitizing Prepared Sample Surfaces and Measuring Tree Ring Parameters

The prepared surfaces were digitized by photographing in reflected light at $\times 30$ magnification on a Carl Zeiss AXIO zoom V16 microscope, with the subsequent stitching of image tiles using the Carl Zeiss ZEN software. The work with digital images has advantages over direct measurements on natural samples for several reasons.

- Handling digitized data on a screen is safer for the user's eyes, increases the output, and improves the quality of results. Note that a stereo microscope is required for checking the surfacing quality (see above) and cross checks for some data.
- Digital images provide a full picture of the sample surface and can be scaled, which facilitates tracing the tree ring series.
- Digital images can be processed in different ways (highlighting and tracing features, improving resolution, etc.) using special software, which is impossible on natural sample surfaces.
- Measuring tree ring parameters in digital images is easier and more accurate than on natural surfaces (except for samples with fresh cracks or cleavage that have to be moved and may require re-digitizing or additional surfacing).
- Digital archives are safe, unlike the natural samples exposed to the effects of rotting, beetles, etc.
- Digitized surfaces can be measured again at any time in the future as new opportunities become available due to the rapidly developing dendrochronological techniques.

The images were processed and converted to the TIFF format for further measurements. The tree ring width, as well as the earlywood and latewood linear parameters, were measured manually using the CooRecorder 9.3 (CR) software [48]. The measured tree rings were dated in a standard way by graphical and visual crossdating techniques [49] combined with cross-correlation analysis using the DPL program package especially designed for dendrochronology [50] and the TSAP V3.5 software [51]. The age trend in each series was removed by spline standardization, ⅔ of the series length [52]. This standardization technique was chosen to correct for short events of rapid growth, which are typical of trees in forest–steppe landscapes.

5.2. Dendrochronological and Radiocarbon Dating of Charcoal from Ancient Iron-Smelting Sites in SE Altai

The dating was performed using eleven referenced individual tree ring series with maximal length, which were selected after checking all measured samples for growth stability (absence of short periods of rapid tree ring width increase). These eleven series included six series for the Kuektanar site and five for the Yustyd site. The crossdating of all measured samples yielded eleven mean tree ring chronologies for the Kuektanar and Yustyd sites. Moreover, two pairs of chronologies from different sites (Nos. 1k-1u and 2k-2u) were crossdated and resulted in a combined TRC (1ku and 2ku) (Figure 5). Then, standardized chronologies were built where the climatic signal (the main factor determining the parameters of the tree rings) was more clearly expressed. Thus, it became possible to crossdate some individual tree ring series that were not dated previously. At this stage, one sample from the Yustyd site was dated with a 3k Kuektanar chronology, resulting in another mixed Kuektanar–Yustyd chronology (No. 3ku).

The measurements for 155 samples (~43% of the collection body) yielded 9 floating tree ring chronologies (Figure 5, Table 1), from 76 to 290 rings.

Table 1. The parameters of archaeological charcoals from iron-smelting hearths at the Kuektanar and Yustyd sites and floating tree ring chronologies (Figure 5).

TRC	Site	TRC Length, Years	Number of Samples	Average Number of Rings in the Sample	Multiple Correlation Coefficient	Visible Tree Ring Width, mm		
						Minimal	Maximal	Average
1ku	Kuektanar/Yustyd	290	106	67	0.64	0.03	1.43	0.31
2ku	Kuektanar/Yustyd	176	9	81	0.68	0.02	2.13	0.32
3ku	Kuektanar/Yustyd	115	13	60	0.51	0.02	0.94	0.24
4u	Yustyd	76	9	37	0.55	0.04	1.08	0.33
5u	Yustyd	83	2	58	0.78	0.05	0.57	0.27
6u	Yustyd	108	2	88	0.53	0.03	0.58	0.16
4k	Kuektanar	99	3	82	0.41	0.04	0.46	0.17
5k	Kuektanar	117	7	88	0.72	0.02	1.02	0.17
6k	Kuektanar	175	4	95	0.45	0.05 (0) *	0.69	0.20
total Yustyd and Kuektanar		1239	155	68	0.58	0.02 (0) *	2.13	0.27

* (0) means that some tree rings were missing.

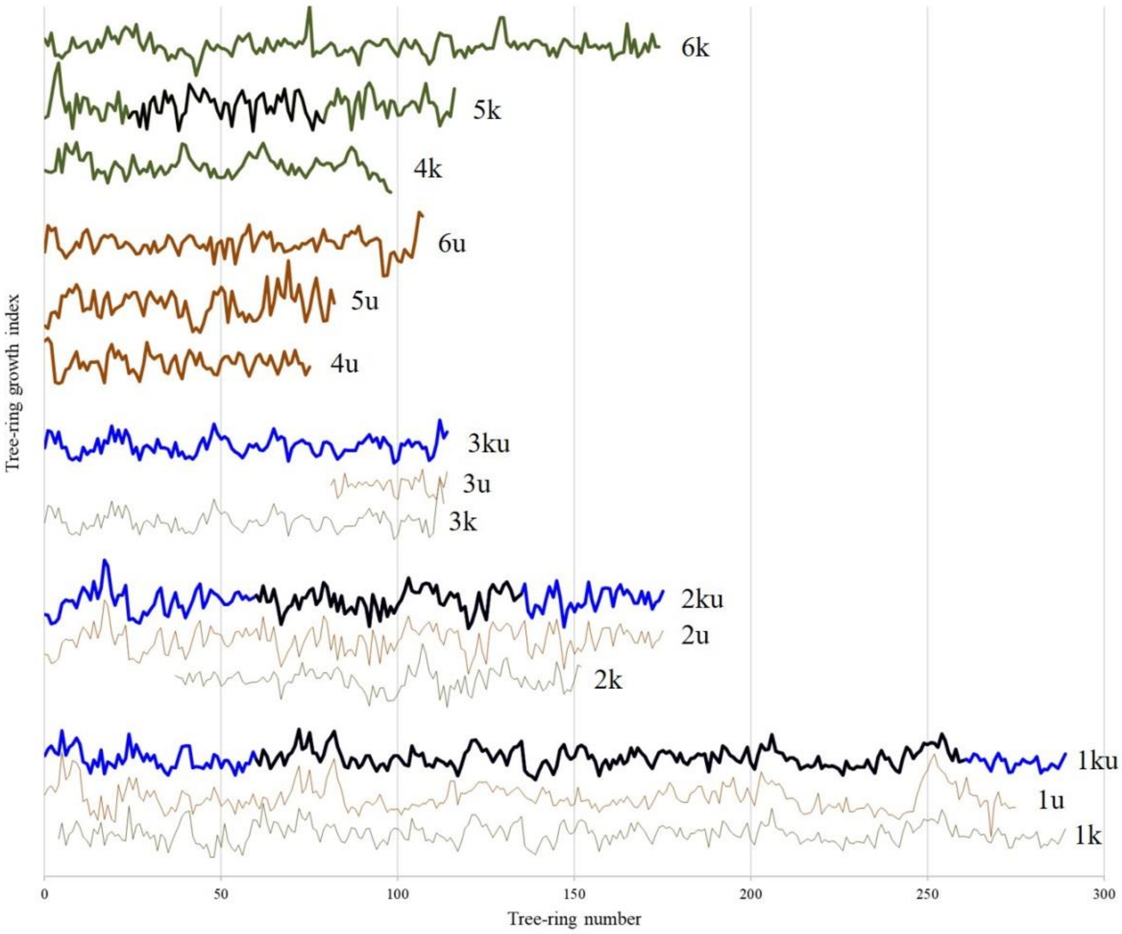

Figure 5. The resulting floating tree ring chronologies (bold line) built on charcoal from Kuektanar (green) and Yustyd (brown) sites. The combined chronologies are in blue (intervals with EPS > 0.85 are shown in black). The thin line indicates the intermediate chronologies that were cross-dated to construct the combined ones.

The obtained tree ring chronologies were verified in the COFECHA program applying the standard procedure. The good quality of the crossdating was confirmed by the absence of discrepancies in the variability of the individual growth series. The multiple correlation coefficient varied from 0.41 (TRC 4k) to 0.78 (TRC 5u), 0.58 on average, and did not depend directly on the completeness of the chronologies. In three chronologies (TRC 1ku, 2ku and 5k), the expressed population signal (EPS) parameter has significant value (>0.85) (Figure 5), i.e., the annual growth in chronology reflects the signal of the general population [53,54].

Among the dated samples, ~7% are charcoal fragments containing 15 to 30 rings, and another ~33% are those containing 32 to 50 rings. The undated samples are more often (~54%) individual series with less than fifty rings, while the others are long series with large deviations in growth parameters. These samples may possibly be crossdated in the future as the collection is extended.

The crossdating of individual tree ring series and some mean chronologies from different sites constitutes an important result of the reported study. It provides evidence that trees at the Kuektanar and Yustyd sites, which are spaced at more than 80 km, grew in relatively similar ecological conditions and that some hearths of the two sites were used at approximately the same historic time.

We have not been able yet to correlate the constructed floating “steppe” chronologies to the regional 3200 tree ring Mongun chronology, the longest for the SE Altai and Tuva territory, which was constructed for the upper timberline in the Siberian Dendrochronological Laboratory, Siberian Federal University. The reason is that the trees that the nomads charred in the hearths grew on the bottom of basins and valleys in temperature and moisture conditions different from those at the upper timberline. Further matching of our chronologies to the calendar timeline may be possible with the wiggle-matching technique using AMS ^{14}C dating or with reference to the chronologies for the high-altitude SE Altai intermontane basins that are currently under development.

Two new calibrated (2σ) radiocarbon dates for in-slag charcoal fragments (Table 2) at the excavation site of Ziniakov [29] on the left bank of the Yustyd River fall between the 3rd and 6th centuries AD. This time interval spans the transition between the Hunnu and ancient Turk periods in the Altai region. The archaeological and radiocarbon data on the charcoal that fueled the hearths of the Kosh-Agach type (Supplementary Table S1) bracket the floating tree ring chronologies between the second and tenth centuries AD.

Table 2. New radiocarbon dates for charcoal fragments from the iron-smelting site previously excavated by Ziniakov [29] in the Yustyd valley. Dates are calibrated using OxCal 4.4.4 [55] and the IntCal20 calibration curve [56].

Lab. Code	Material	Dating Technique	^{14}C Age	Calibrated Date (2σ)
$IGAN_{AMS}$ 7165	Charcoal	AMS	1565 ± 20	432AD (95.4%) 560AD
$IGAN_{AMS}$ 7166	Charcoal	AMS	1720 ± 20	254AD (26.9%) 287AD 324AD (68.6%) 406AD

6. Discussion

The first taxonomic and ecological studies of wood charcoals from archaeological contexts date back to the late 19th century and the first half of the 20th century in Europe and North Africa [57], but early anthracology was limited by the lack of sample preparation techniques and ways of proper material selection. The amount and quality of samples increased considerably with the progress in optical microscopy [58], and special methods were developed for the quantitative analysis of charcoal fragments [57]. By the 1990s, important data quality criteria were formulated, including the requirements of studying large statistically significant collections, taking into account the spatial representativeness of field sampling, charcoal fragmentation patterns, and palaeoecological representativeness of the relative frequencies of charcoal taxa, as well as selecting appropriate archaeological contexts and deposits to sample in the field [59] and references therein.

Further progress in dendrochronology became possible due to the recent rapid advance of digital technologies, e.g., [60,61]. Among other advantages, it allowed for the simultaneous measurement of several variables in digital images instead of the labor-consuming and less efficient measurements of rings in natural wood samples. Thus, tree ring analysis can use material that was previously impossible to process. In this respect, charcoal is among the underestimated materials for tree ring dating. The charcoal from the mountainous SE Altai has shown good prospects due to the landscape and climate features of the area, together with the abundance of smelting archaeological sites.

The study has yielded floating chronologies with a length of 290 rings, one of the world longest charcoal chronologies at the time being.

6.1. Minimum Number of Tree Rings for Dendrochronological Dating

The dendrochronological analysis of charcoal is often limited to a few tree rings because the samples are crumbly and small, and only a small percentage of individual tree ring series can be dated. That was, for instance, the case of a collection with less than twenty five rings in most of the samples and <25% of dated samples [17]. This number of rings is twice as small than the previously suggested minimum required for successful analysis and even less than the thirty rings that may work for large collections [62]. Earlier, the minimum number of rings for the crossdating of individual tree ring series was also estimated as fifty [63], before Backmeroff and Di Pasquale [64] reported that 190 out of 268 charcoal fragments from the upper timberline charcoal-production kilns in the Central

Italian Alps were dated successfully from measured ring widths. The charcoal samples of that collection contained 31 to 149 annual rings, and the dating covered two thirds of all measured samples with 31 to 50 rings and 90% of samples with >60 rings. The success was mainly due to a strong climate signal in the data, because the combusted larches grew at the upper timberline where trees are especially sensitive to climate change. The annual growth record of larch trees from the SE Altai area likewise stores a strong climate signal, which allows for crossdating samples with relatively few rings.

The mean chronology obtained for the multi-period archaeological site of Uşaklı Höyük in Turkey had a length of 49 rings [13]. However, it turned out to be unsuitable for secure dendrochronological dating of any sample because it could not be matched to the regional reference (note that matching tree ring series to the regional master chronologies often poses a separate problem). The reasons were: insufficient chronology length (49 rings), shortness of individual tree ring series (max. 34 rings), and scarcity of reference chronologies available for cross-dating [13]. In this case, the AMS ^{14}C technique with subsequent wiggle-matching analysis becomes the main dating approach. The preliminary ages of material inferred from single AMS ^{14}C dates provide good reference in the beginning of geochronological studies of charcoal collections.

The smallest charcoal sample we dated was 2.57 mm in size and contained 15 rings. Every sixths dated sample from our collection was less than 1 cm, the average ring width was within 0.22 mm in one third of the samples, and 40% of the samples had 50 rings or less. Nevertheless, the prominent climate signal in the tree growth record allowed for the successful dating of charcoal from the ancient nomad smelting sites of SE Altai.

6.2. Other Factors of Dating Success with Charcoal Used for Tree Ring Chronologies

The dating success also depends on other factors in addition to the minimum number of rings: the reference to archaeological and natural contexts, the use of additional sample material, the availability of master chronologies, etc. The understanding of the general archaeological and natural contexts of the analyzed material is a key point. The knowledge that single charcoal samples belong to the same tree may simplify the analysis and ensure dating success even when few samples or few rings are available. For instance, it was useful in the analysis of 95 charcoal fragments corresponding to eight taxa, which were sampled in different archaeological contexts and belonged to the same trees or same cultural layers [19]. As a result, 8 out of 14 floating chronologies, with lengths from 8 to 59 rings, demonstrated very good concordance and allowed for testing of the suggested approach on 2 to 8 samples from 4 to 100 rings (29 on average) from the same context [19].

The analysis of wood samples that represent a similar archaeological context can extend the charcoal data and improve the quality of tree ring chronologies, as crosschecking multiple lines of dendrochronological evidence enhances the accuracy of interpretations. That was the case of the tree ring dating of the historical charcoal-production sites in the Great Basin, USA [16]. The collection included large fragments of carbonized and non-carbonized wood, which were sampled on the surface or were unearthed in the vicinity of charcoal-production kilns, as well as samples of tree stumps and old-growth live trees. The analysis of all available timber material from the site, with at least 30 rings in the samples, yielded floating chronologies as long as 100 rings for charcoal and 202 rings for all timber types. The calendar dates were assigned to each sample based on crossdating with the master chronologies [16].

The death time of trees used as geoarchaeological samples, i.e., the age of the last ring below the bark, is of special interest as it records certain natural or anthropogenic events. However, the carbonized wood samples found in hearths very rarely retain bark that would allow for the exact dating of the cut time [30]. Most of the charcoal samples in our collection lack the pith and generally have few annual growth rings: 68 rings on average over all dated samples (Table 1). Meanwhile, the trees growing currently in the Kurai and Chuya Basins and its border ranges are at least 300 years old, with a maximum of 450 years for some trees in the Kurai steppe [33] and at the foot of NW slope of the Talduairy Massif in the Chuya basin. Thus, the sub-bark rings in individual charcoal samples are not always detectable from growth stabilization, whereas the SE Altai larches reach approximately an age of 150 years. The most preferable approach in this respect is to handle whole tree ring chronologies rather than making chronological reconstructions by bringing together single dated fragments.

The visualization of the annual rings and the measurements of their parameters in charcoal depend on processes in green trees, as well as on the preservation of wood during combustion and subsequent burial [19]. The problems with using charcoal samples from the SE Altai ancient iron-smelting sites are mainly due to biological factors. Larch trees growing in harsh climate conditions of mountains develop very thin annual rings, sometimes reduced to a single cell, or some rings may be missing at all. As in the case of classical dendrochronological samples, this impedes the earlywood and latewood identification and ring measurements [65]. Furthermore, the boundaries of annual rings can become less visible under destructive pre- and post-carbonization effects caused by various agents: wood beetles in green trees [11]; vitrification of carbonizing wood [66]; sediments penetrating into carbonized wood during and after burial [19]; etc. Furthermore, shrinking cracks make charcoal samples prone to breakage.

6.3. Paleoenvironmental and Paleoclimatic Contexts

Dendroanthracological studies of in-slag archaeological charcoal preserved in ancient hearths of the Altai nomads were performed at two localities: (i) the Kuektanar River mouth in the Chuya valley, between the Chuya and Kurai basins, and (ii) the Yustyd River valley on the eastern periphery of the Chuya Basin (Figure 2). Being 80 km apart, the two sites are more than 350 m different in elevation (1730 and 2095 m a.s.l.) and differ markedly in landscapes. The bottom and steep sides of the narrow Chuya valley at the Kuektanar mouth are grown with larch, spruce, willow, poplar, and sporadic birch, whereas the eastern periphery of the Chuya Basin drained by the braided Yustyd River is bare.

However, the Yustyd area did grow forests in ancient times (*yustyd* means *one hundred larches* in the Altai language) but became deforested under anthropogenic loads from the dense nomad population since the Bronze Age, because the cut wood failed to recover in the extremely dry climate of the area [32]. The crossdating of individual tree ring series and some mean chronologies from the two localities have paleoenvironmental and paleoclimatic implications. The results indicate that the trees burnt for iron smelting were growing synchronously throughout the vast Chuya–Kurai metallurgical province. Therefore, the now forestless eastern Chuya Basin still preserved tree vegetation in the time of iron smelting with the box-shaped furnaces, while the metallurgical use of timber was one of the main causes of deforestation in the Yustyd valley. Another important inference concerns the common climate signal in the annual growth of *Larix sibirica* Ledeb and, hence, similar paleoecological conditions in the Kuektanar and Yustyd areas despite the strong modern southeastward aridization trend in the region. Furthermore, the aridization intensity might have varied over the Holocene, i.e., during the time of larches growth; therefore, this spatial trend was not so pronounced.

The identity of the climate signal in the Kuektanar and Yustyd records is essential for obtaining long tree ring chronologies based on larch trees from the Kurai and Chuya highland steppes. The tree growth is controlled by moisture patterns depending on summer precipitation and air temperatures in basins within elevations 1730–2000 m a.s.l., but it is the soil surface temperature in the summer season that is the principal control at the upper timberline (2200 m and higher) in the flanking mountains [1,33].

The difference in the limiting factors makes it impossible to match the charcoal-based tree ring chronologies to the calendar timeline with reference to the available master regional chronologies. The reported charcoal data represent highland steppe landscapes with elevations 1730 to 2000 m a.s.l., where the ecological conditions differed from those for *Larix sibirica* Ledeb from the upper timberline (2220–2450 m a.s.l). They are, namely, the larch trees used in the master 3200-rings Mongun and 1900-rings Jelo chronologies [1,33], as well as in another regional 424-rings Pazyryk chronology [67].

6.4. Dating Iron Production Sites by Numerical Methods

Archaeological sites of iron-smelting industry are most often dated using the tools of archaeomagnetism, thermoluminscence (TL), and LSC or AMS radiocarbon dating [68]. Archaeomagnetism is a perfect method for determining the last episode of hearth use at a site [69,70], but it is applied to in situ furnace structures, as in the case of TL dating [71]. Furthermore, the techniques are time consuming and require specialized field procedures and calibration. Currently, radiocarbon dating is the most broadly used method due to abundance of charcoal found at iron production sites.

At the time being, 29 radiocarbon dates of charcoal samples from the Yustyd and Kuektanar hearths are available for tree ring analysis: 11 dates for the Yustyd samples (Figure 6), and 18 dates for samples from three box-shaped hearths at the Kuektanar sites (#2 at Kuektanar-1 and #3, #4 at Kuektanar-2, Figure 7). The former group of samples includes a charcoal fragment from a Hunnu pottery kiln and charcoal from box-shaped iron-smelting hearths or from those with an oval back wall. Unlike the Kuektanar samples, the in-slag charcoal fragments scattered at the terrace toe and near the excavation sites of Ziniakov [29] in the Yustyd valley can be only provisionally attributed to specific furnaces.

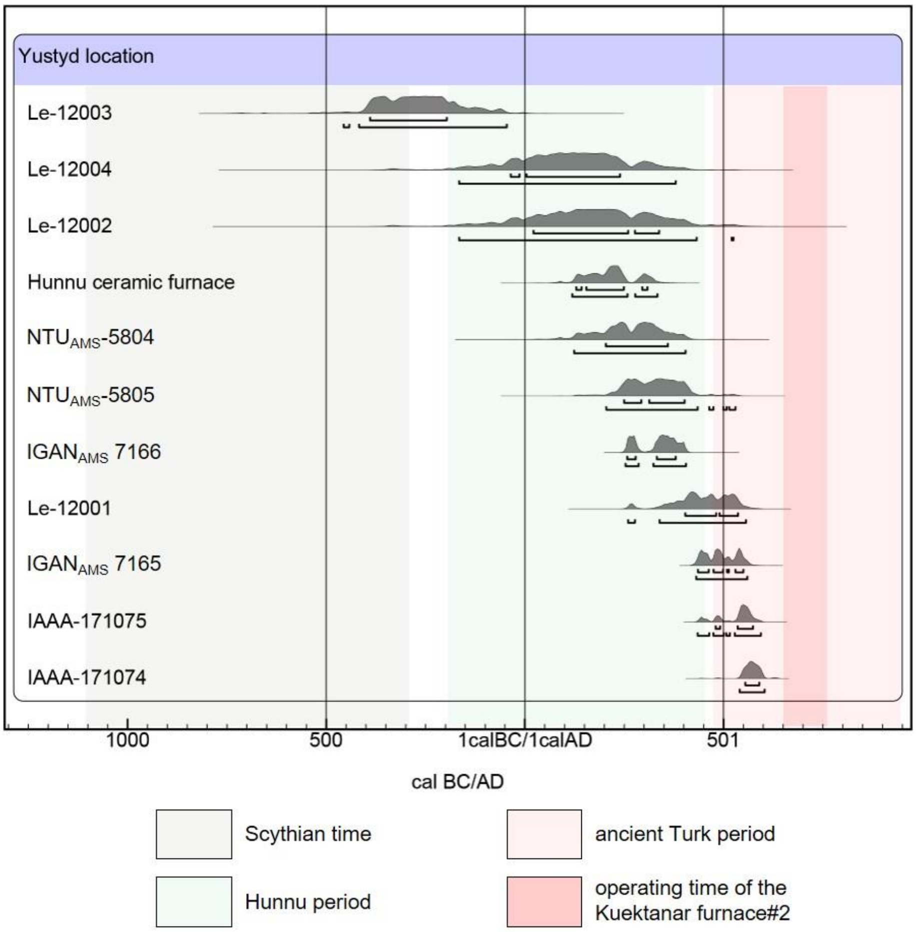

Figure 6. Radiocarbon dates of charcoal samples (Lab. codes are shown on the vertical axis) from the hearths of different types in the Yustyd valley, calibrated (2σ) in OxCal 4.4.4 [53] using the IntCal20 curve [54]. For each sample, the chart presents the probability distribution function (gray fill); bars for 1σ (upper line) and 2σ (low line) standard deviation indicate the calibration results. The dates were published previously [39,47,72] and are cited in Supplementary Table S1. The dates of IGAN$_{AMS}$ 7165 and IGAN$_{AMS}$ 7166 are reported for the first time.

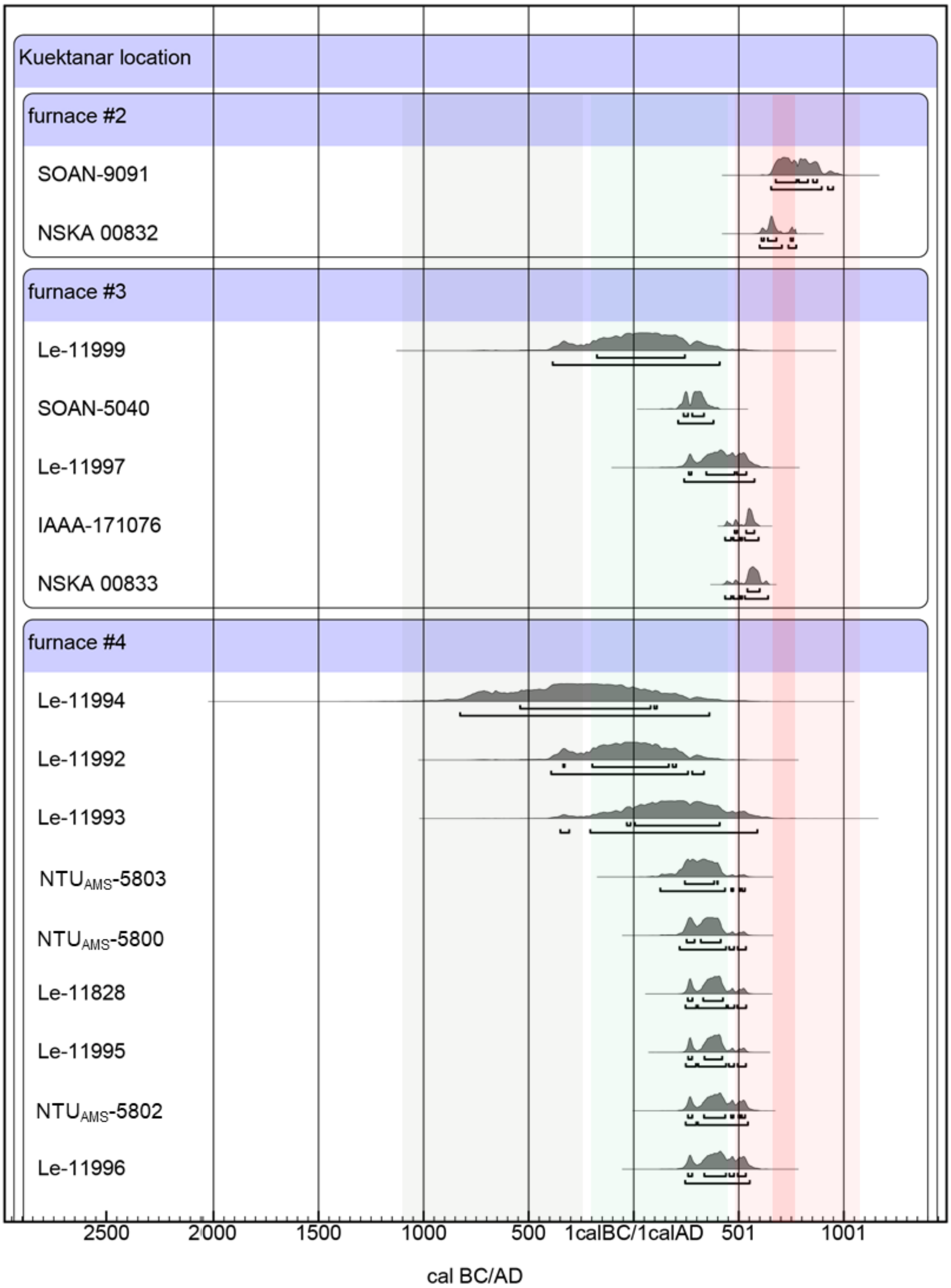

Figure 7. Radiocarbon dates of charcoal samples (Lab. codes are shown on the vertical axis and grouped for each furnace) from box-shaped iron-smelting hearths in the Kuektanar mouth, calibrated (2σ) in OxCal 4.4.4 [53] using the IntCal20 curve [54]. For each sample, the chart presents the probability distribution function (gray fill); bars for 1σ (upper line) and 2σ (low line) standard deviation indicate the calibration results. The dates were published previously [30–32,47,73] and are cited in Supplementary Table S1. For the legend, see Figure 6.

The numerous cultural artefacts bracket the lifespan of the Yustyd pottery kiln between the 2nd century BC and the 1st century AD, or the Hunnu period, which is supported by the radiocarbon date found for charcoal fragments from the kiln [72]. Some in-slag charcoal samples scattered on the ground near the hearths of different types show similar ages older than the box-shaped furnaces. The synchronicity of the samples indicates that the Hunnu kiln and some iron-smelting hearths from the Yustyd valley were operated during the same time span [39].

At the same time, the youngest radiocarbon ages of the charcoal samples obtained from the slags of iron-smelting furnaces of the Kosh-Agach type, as well as a fragment of unburned bark from the filling of one of these furnaces (Figures 6 and 7, Supplementary Table S1, [30,32,47]) indicate that trees used in box-shaped hearths grew, including in the ancient Turkic period. These youngest dates obtained for all hearths of the same type allow for the minimization of the influence of the old wood effect [30,39,74–77] and the loss of outer rings during the combustion of wood. Therefore, it results in more precise chronological reconstructions of iron-smelting events. Obviously, during charcoal burning and iron-smelting production, fragments of the most ancient parts of wood are concentrated in slags, and, accordingly, ^{14}C dates of the younger outer rings are generally less presented. The seven youngest radiocarbon dates were obtained by two scientific groups in five different laboratories applying both AMS and LSC techniques (Figures 6 and 7, Supplementary Table S1). These dates characterize three locations within the Chuya–Kurai metallurgic province, from the westernmost Turgun and then Kuektanar sites to the easternmost Yustyd (Figure 2) and directly indicate the functioning of Kosh-Agach type furnaces in the ancient Turkic period.

In general, the accuracy of the ^{14}C dating method, with regard to the old wood effect, allows for quite a wide time interval when the combusted trees could have lived: from the 5th century BC to the 10th century AD. The similarity of ^{14}C dates for charcoal from box-shaped hearths from different sites within the Chuya–Kurai metallurgic province ([30–32,39,47], Supplementary Table S1) means that the floating tree ring chronologies can be crossdated. Indeed, we have performed such crossdating and expectedly arrived at the conclusion supported by the archaeological evidence [29], that the box-shaped hearths of the Kosh-Agach type were used during approximately the same time span.

Note that radiocarbon analysis, even applying the AMS technique, is not accurate enough for chronological reconstructions of prehistoric events. For the case of charcoal from the Altai iron-smelting hearths, the uncertainty of calibrated (2σ) radiocarbon ages varied from 63 years (sample IAAA-171074, Figure 6, Supplementary Table S1, which is an exceptionally successful dating result) or 128 years (sample $IGAN_{AMS}$ 7165, Table 2, Figure 6, Supplementary Table S1, the next most precise result) to as many as >1000 years (sample Le-11994, Figure 7, Supplementary Table S1), which is much longer than the duration of each archaeological period considered in this study. The required dating accuracy is hardly achievable even for charcoal from large iron slag pieces [68], where the organic material was sheltered safely from the destructive external effects and contamination with younger carbon.

Furthermore, the radiocarbon ages of the charcoal used to date archaeological events correspond to the lifespan of trees or, at best, to the time when they were cut, which may differ from the time of the hearth operation. Therefore, these radiocarbon ages represent an interval around the smelting episode and, at least, provide a terminus post quem of metallurgical activity at a given locality. Earlier, we [30] found a unique fragment of only slightly charred larch bark (sample NSKA-00832, Supplementary Table S1) in the pit filling of hearth #2 at the Kuktanar site, with its age corresponding to the time of tree cutting and, to a large probability, to the time of the last smelting episode. The age of a nearby charcoal sample SOAN-9091 (Figure 7, Supplementary Table S1) from the same hearth calibrated using that information provided time constraints on the last fire-use episode between 655 and 765 AD, within the ancient Turk period [30]. This is the most accurate yet available radiocarbon-based chronological reconstruction, but it exceeds 100 years anyway. Obviously, tree ring analysis, with its annual or even seasonal resolution, is advantageous in this respect.

The chronological reconstructions based on wood and charcoal dating can be biased additionally by the old wood effect [30,39,74–77]. Both radiocarbon and dendrochronologically derived charcoal ages used to date archaeological artefacts may be overestimated as the number of lost peripheral rings remains unknown. A possible solution is to sample the outer rings where their width corresponds to that of the sub-bark rings. However, as noted above, the sub-bark rings of *Larix sibirica* Ledeb may be difficult to identify from the growth stabilization age, which is far less than the survival time in the present conditions of highland Altai and Tyva: ~150 years against 450 years or more [33,78]. Rather, trees were found to live longer in more severe conditions [79], and the age of the oldest living tree in the Tyva area adjacent to Altai is 778 years [80]. Thus, the signs of growth stabilization in charcoal samples cannot be reliable evidence of the sub-bark rings. Another solution is to date small charcoal fragments that preserved pith, possibly representing young branches or trunks, with their ages close to the time of cutting. However, neither approach can allow for the missing peripheral rings.

Thus, the use of tree ring analysis can improve the accuracy of charcoal dates, which is within at least 100 years or mainly within 1000 years in the radiocarbon method. As for the old wood problem, tree ring analysis facilitates the choice between two strategies of selecting samples.

The impossibility to crossdate some constructed standardized chronologies at the time may indicate that the combusted trees were of different ages. This may indicate that iron-smelting production at the Kuektanar and Yustyd sites existed for a long time, and the hearths were from several generations. The same hearths were often used repeatedly (up to seven times), judging by the stratification of their pit filling [29]. On the other hand, the long lifespan of *Larix sibirica* in the harsh conditions of the Altai intermontane basins, where trees could live at least 450 years, may cause problems for the crossdating of TRC from one site for the poor applicability of charcoal samples from different parts of the same tree.

The old wood effect is apparently responsible for the old 14C ages of some Kuektanar charcoal samples falling before the ancient Turk period, as well as for the impossibility to crossdate some charcoals from that site with the tree ring analysis. This possibility is more so likely that all Kuektanar hearths we studied are of the same type, whereas those from the Yustyd valley are different: the iron-smelting traditions can hardly have remained the same when the archaeological cultures had changed.

The expansion of the sample collection will solve the problem of constructing long tree ring chronologies and will ensure the further precise dating of charcoals from iron-smelting hearths (though not the problem of dating the hearths themselves). Therefore, all obtained floating TRC can, to a large probability, be combined into one continuous chronology in the future, as far as more samples become available.

7. Conclusions

Dendroanthracological studies can be successful in the arid mountainous area of southeastern Russian Altai where abundant charcoal from iron-smelting archaeological sites is well preserved in dry and cold climatic conditions. New time- and cost-saving preparation protocols tested on charcoal samples from the Altai smelting sites can yield numerous high-quality samples for digitizing with advanced instruments and software. The digital archive of photographed sample surfaces can be used to perform all necessary measurements of ring parameters and to process the data.

Despite the problems in processing charcoal samples, the suggested techniques have allowed us to prepare 355 viable charcoal fragments out of 448 samples in the whole body of the collection (more than 80%) from iron-smelting furnaces left by ancient Altai nomads. The tree ring parameters were measured in the prepared surfaces of all 355 samples, and 155 samples (~43%) were used to construct floating TRC as long as 76 to 290 rings. The one with 290 rings is among the world's longest charcoal-based chronologies.

The work with charcoal from SE Altai can be continued to obtain a floating "steppe" TRC up to 1000 rings (for larch trees from the intermontane basins) and for its subsequent matching to the calendar timeline. The relative simplicity of the sample preparation protocol opens new avenues in tree-ring dating for geoarchaeology and paleoecology in this part of Central Asia, as well as for dendroanthracological research in general.

The crossdating of the tree ring chronologies built on charcoal from the box-shaped hearths (Kosh-Agach type) found at two locations in the SE Altai region indicates that trees grew in the now forestless eastern periphery of the Chuya Basin from the 2nd to 10th centuries AD, which is consistent with the ^{14}C dates. The archaeological evidence and radiocarbon dates indicate that the Altai nomads apparently used box-shaped hearths between the 5th and 10th centuries AD. On the other hand, older ^{14}C ages of samples from the Kuektanar mouth, where all hearths are of the Kosh-Agach type, may be due to the old wood effect.

Some floating tree ring chronologies were not crossdated for several reasons: they may be poorly provided by samples; the charcoal samples may represent different wood parts of old trees (up to 450 and even more years); and trees used for iron smelting may have lived in different periods. The latter inference agrees with the presence of two types of hearths in the Yustyd valley, as well as with older ^{14}C ages of the respective charcoal samples. Correspondingly, constructing a long charcoal-based tree ring chronology for the highland steppes of SE Altai has good perspectives. The long chronologies can be further used for reference in the precise comparison of climatic conditions in the floors of intermontane basins and at the upper timberline in the flanking mountains.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/cli11070150/s1, Supplementary Table S1: Available radiocarbon ages for charcoals from iron-smelting furnaces in the Kosh-Agach ferrous metallurgy province.

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