The radiocarbon dating of iron artefacts using accelerator mass spectrometry

Richard G Cresswell

Abstract

Rather than dating associated materials when the age of an iron artefact is required, a date may be obtained from the carbon that the iron has incorporated from the smelting process. Large sample sizes have forestalled this method in the past; radiocarbon dating using accelerator mass spectrometry has reduced the sample size requirements to less than 5 mg carbon equivalent. Thus, only 10 g of wrought iron is needed, while for high carbon (2%) steel or cast iron, only 250 mg may be required. Two historical irons, dated at the IsoTrace Laboratory, Toronto, illustrate the capabilities of this technique.

Introduction

The usual method used to date an iron object is to date some carbonaceous material associated with it. This material should, ideally, be intimately associated with the iron (e.g. handles of axes, or other tools), but this dates only the last use of the tool, and not the tool's manufacture. Dates from charcoal associated with iron slags, iron furnaces and hearths, iron ores and associated horizons can all give clues as to the iron's origin, but the artefact may be of a completely different age to the site and inferences may have to be made. A further problem is that of collected samples with no known origin, or those whose origin is suspect. In such cases, the dating of the artefact itself is the only recourse.

For iron artefacts, any carbon associated intimately with the iron is adequate for radiocarbon dating. Indeed, fragments of charcoal held within the iron itself must relate to the piece's manufacture and would therefore date that event. The sources of carbon other than from the fuel are negligible, except where pre-smelted iron is added to the charge (e.g. in the co-fusion process of the Chinese),[1] or if the ore contains a significant proportion of terrestrial, or native, iron or meteoritic iron. Both terrestrial and meteoritic iron can have quite a high carbon content. The iron found on Disko Island, off the central west coast of Greenland, for example, can assay over 4% carbon.[2] Meteoritic iron may contain up to 2.5% in both combined and graphitic forms.[3] A meteoritic input can be determined by the presence of appreciable nickel ($>4\%$)[4,5] while terrestrial sources are sufficiently rare and well-documented[6] not to be a problem in archaeological studies.

With these points in mind, in the 1960's, van der Merwe set out to establish a routine analytical line for the extraction, purification and dating of carbon in iron. The minimum size of sample required for the gas proportional counters at the Yale Radiocarbon Laboratory at that time was 500 mg carbon (1 litre CO_2 gas), which immediately placed constraints on the size of van der Merwe's apparatus which was being developed at the Yale Geochronometric Laboratory. 20 g of a very high carbon cast iron would be a minimum; losses during cleaning and combustion (incomplete extraction) meant that 40–50 g was more practical. For wrought iron, samples would have to be over a kilogram.[7]

The technique required cleaning and crushing the sample followed by heating in an electric (resistance) furnace to 1150°C. Carbon diffused to the surface where it was oxidized to carbon-dioxide in a stream of oxygen. The CO_2 was then trapped, purified and loaded into proportional gas counters for beta-decay counting.

The results[8] of 11 artefacts analyzed in this way (Table 1) clearly indicate the validity of this technique, the only problem was the great amount of material required for each run.

Other than van der Merwe, I am aware of only one other laboratory dating archaeological iron. This is the Brookhaven National Laboratory, New York, where dates for a Frobisher bloom were commissioned by the Smithsonian Institution in 1975. Using small proportional counters, 30 g of bloomery iron was required for each radiocarbon analysis, the iron being combusted as three 10 g powdered fractions in much the same way as van der Merwe. 2% precision (±160 years) was attainable, though results were ambiguous.[9]

With the advent of accelerator mass spectrometry (AMS)[10] in the late 1970's, whereby ^{14}C atoms in a sample are measured rather than the number of decays of this radionuclide, the problems of sample size and poor precision were considerably improved. To date, however, little has been done to apply the technique to the analysis of metallic materials, despite the fact that a number of accelerator radiocarbon laboratories initially used carbon-iron targets for their regular radiocarbon runs. A few laboratories have tried to measure the activity of some ultra-high carbon ($>6\%$) cast irons, but have abandoned this due to poor reproducibility. This iron is basically used as a catalyst

Table 1
ARTEFACTS ANALYZED PRIOR TO THIS STUDY

Artefact	%C	Weight (g)	Age (years BP)	Ref.
Saugus cast iron	2.9*	380	350 ± 60	8
	3.73	≈10	469 ± 144	9
Redding cast iron	2.3*	231.9	180 ± 60	8
	3.98	≈10	285 ± 145	9
Hopewell cast iron	2.0*	289.2	200 ± 60	8
Scottish iron nails	0.22*	1232.1	1850 ± 80	8
Sian cast iron	3.2*	187.4	2060 ± 80	8
Hunan cast iron	1.9*	200.4	400 ± 60	8
Honan cast iron	3.1*	46.4	2380 ± 80	8
Szechwan cast iron	2.6*	32.7	2130 ± 100	8
Yugoslavian steel sword	0.66*	216.0	2130 ± 60	8
South Dakota cast iron	3.0*	198.0	⩾25000 1)	9
North Dakota cast iron	2.7*	245.6	⩾6700 1)	8
Frobisher Bloom #1	0.051–0.127	≈30	679 ± 133	9
	0.048–0.061	≈30	792 ± 107	9

* Estimated values based on combustion yields.

Notes. The ages obtained by van der Merwe were used to corroborate the suspected dates of manufacture of the samples. The nature of the radiocarbon calibration curve, whereby a single radiocarbon date may give more than one calendric date for certain time periods, does not give specific dates for most samples, and precise calendric dates are not possible for the precisions cited. It is noted, however, that the Hunan sample gives a date 4 to 10 centuries too young based on stylistic grounds. The sample may be a Ming copy, or may be from a repaired section of the statue.[8]

1) These ages indicate that some measure of coal or coke was used in the manufacture of these ingots. Assuming a mixed fuel to have been used, the ratios of coke to charcoal would have been about 95 : 5 and 55 : 45 for the South and North Dakota samples, respectively.[8]

on which the carbon is deposited, and iron carbides and iron-carbon solutions have lost favour due to their relatively low efficiencies and precisions.[11]

This was the state of the art up to a few years ago. Despite suggestions of the use of AMS dating of such artefacts,[9] none had been attempted. Various carbon-metal mixtures, carbides and solutions have been tested for routine radiocarbon analysis, but with no mind towards the dating of untreated iron artefacts.[11] An attempt to date man-made iron at this laboratory, with a very much lower carbon content (<0.4% C), did not prove successful due to the extremely low carbon currents and matrix effects due to the inhomogeneity of the sample.[12]

To remedy this situation, a line for the extraction of carbon from iron and steels has been developed at the IsoTrace Laboratory in Toronto[12] and can now analyse samples containing as little as 1 mg of carbon. This is equivalent to roughly 2 g of wrought iron (0.05% carbon) or less than 30 mg for a high carbon cast-iron.

Experimental Procedures

The carbon extraction line (fig.1) is based on the design of van der Merwe.[7] The sample to be analysed is first checked for rust, which is removed by milling, and then undergoes ultrasonic cleaning in dilute nitric acid to expose a fresh surface. If the sample weighs less than 2g, 2–3 g of iron chip accelerator is added as a flux, and the sample is loaded into a zirconia boat and placed in the quartz combustion tube. The line is evacuated to better than 10^{-2} torr pressure, and the sample is heated to ≈800°C for 15 minutes to bake off surface adsorbants. Oxygen is circulated through the line, maintained at a positive pressure, and the sample's temperature is raised until melting occurs, at which point the carbon diffuses to the surface and exsolves from the carbides. The exsolved carbon is oxidized to CO_2 and trapped in liquid-nitrogen-cooled traps.[12] The trapped carbon dioxide is added to heated lithium metal to create lithium carbide, and water is added to convert this to acetylene. The acetylene is dried and bled into a "cracking chamber" where a

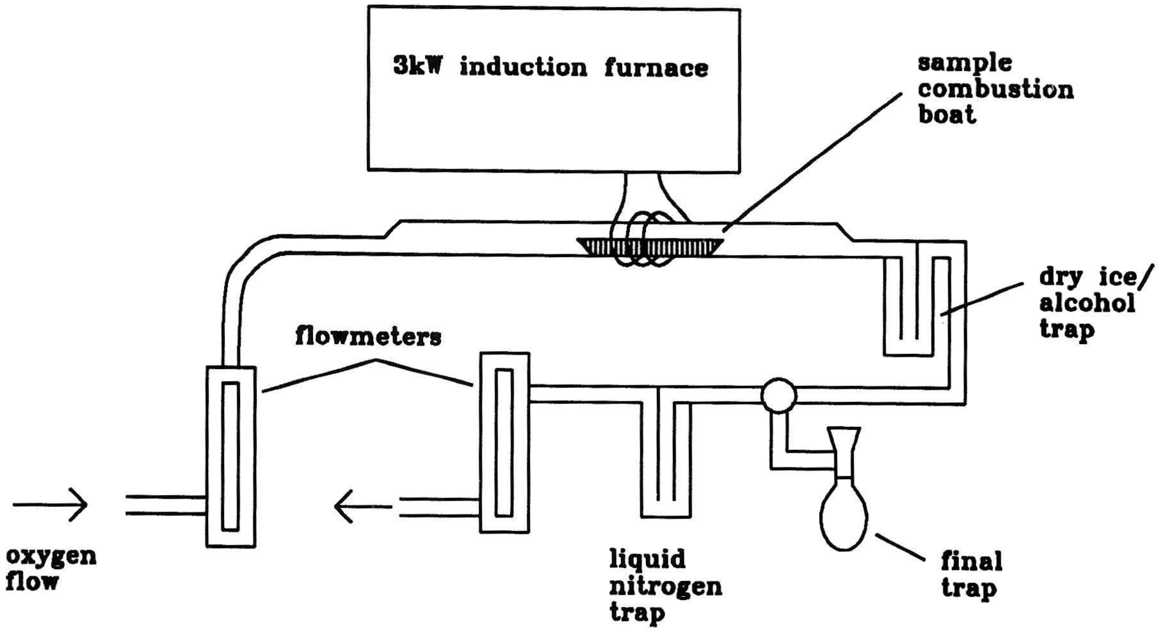

Fig. 1 Simplified schematic of the iron combustion line. Carbon dioxide, trapped in the final trap, is removed to be cracked as described in the text and reference 12.

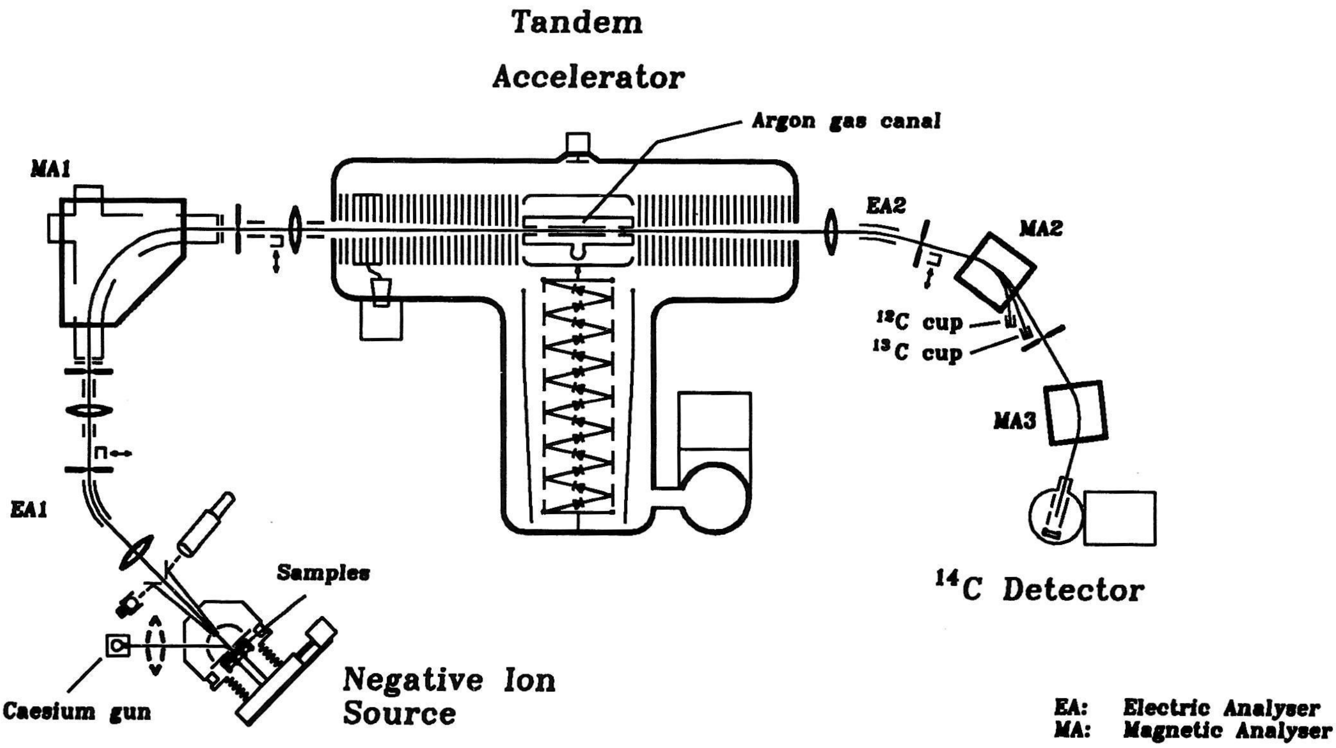

Fig. 2 Schematic of the IsoTrace Laboratory's accelerator mass spectrometer: set-up for radiocarbon analysis (see text).

1 kHz a.c. discharge between two $\frac{1}{4}''$ diameter aluminium slugs causes the acetylene to dissociate, depositing carbon on the surface of the aluminium.[13] This target is analyzed for its radiocarbon content using accelerator mass spectrometry on the IsoTrace spectrometer (fig. 2).[14] A beam of finely-focussed, energetic, positive cæsium ions are aimed at the carbon target, sputtering the surface, creating a plasma of energetic ions. Negative carbon ions are extracted from the surface and steered through electric and magnetic lenses into the tandem accelerator, where their energy is raised to 2 MeV. Passing through an argon stripper canal at these energies causes electrons to be stripped from their outer shells; molecules become unstable, and a beam of positive atoms are further accelerated back to ground. After further magnetic analysis the ^{12}C and ^{13}C beams are measured, while the ^{14}C undergoes further magnetic discrimination before being measured in an energy detector. The ability to analyse all three carbon isotopes allows us to correct for all fractionation effects, whether from the sample, sample preparation or from analysis, with ages quoted to a base $\delta^{13}C = -25‰$.

A sample of 5cc of CO_2 is currently preferred for cracking (equivalent too just over 2.5 mg of carbon). The size of the sample initially combusted, therefore, is strongly dependent on the carbon content of the material. Ideally an iron sample of 3 g or larger is preferred, thereby removing the need to add iron chip, which has been shown to contribute up to 0.02 cc of CO_2 per gram of chip used.[12]

The contamination level of the system was tested using the fact that modern coke-smelted iron should be essentially radiocarbon-free. Samples from the Stelco Steel Works, Hamilton, Ontario, were analyzed, together with a sample of terrestrial, or native, iron from Disko Island, off the west-central coast of Greenland (Table 2a). This native iron is believed to be derived from the interaction of intruding tholeiitic magmas with the host, Mesozoic, carbonaceous country-rocks.[15] The carbon should thus also be radiogenically "dead". The carbon content of these intrusions varies from less than 0.1% in the basalts to an estimated 6.7% in white cast-iron zones of the Kidlît Lens, the locality from which the analyzed sample was obtained.[15]

The results (Table 2a) suggest a small contribution to the sample carbon from the line itself, most likely from the oxygen used to flush the system. For dates younger than 5,000 years, however, this effect is within the statistics, except for high resolution dates ($<1\%$), for which the date may be too young by up to 100 years, depending on the age and amount of carbon analyzed. Improvements to the vacuum line are continuing to reduce this contribution.

Contemporaneous Carbon in Iron?

The usefulness of dating carbon in iron relies on the contemporaneity of that carbon with the time of manufacture. Thus, the carbon source should have a radiocarbon content indicative of the date of incorporation of that carbon into the metal. This is valid as long as the fuel used to smelt the ore consisted of contemporaneous charcoal or organic matter, while any coal or coke used would dilute that amount and result in an older age. Thus, just as recent carbon may contaminate the sample, and give it an apparently younger age, so ancient carbon may dilute the sample giving it an apparently older age. Recent sources may generally be removed by correct cleaning practices; the problem of inclusion of older material is harder to elucidate, as often, metallographically, no distinction may be made between different sources of carbon. A high sulphur content may be indicative of a contribution by coal; peat, however, can also contain appreciable sulphur, while contributing carbon that may be adequate for radiocarbon analysis. The antiquity of many peat bogs however can conspire to give erroneously old dates, thus the archaeological context of the discovery must give some clues to whether or not different fuels were used, and historical records can indicate the likelihood of obtaining an accurate date.

Fortunately, up until the Industrial Revolution, most smelting was carried out using charcoal-fired furnaces. Historical records indicate that much of the ancient iron was smelted using only freshly cut wood. As Hammurabi (ca. 1750 BC) once informed a servant:

> "Among the firewood that will be cut, there shall be no wood that died in the forest. They shall cut green wood only."[22]

It is probable that charcoal was discovered during the early development of copper smelting where temperatures of over 1000°C were required, significantly above that attainable from a simple open fire (700–800°C).[16] Dung can also produce such temperatures, as can peat, the latter being used in Scotland in the 17th century.[17] These fuels would normally yield ^{14}C in equilibrium with the prevalent atmospheric concentrations and would enable the dating of the artefact, though there is the possible problem with peat noted above.

For smelting, charcoal was the dominant fuel[17] though some important exceptions exist. As Sung Ying-Hsing said in 1637 AD:

> "(As for the fuel) sometimes hard wood is used, sometimes coal, and sometimes charcoal — whatever is convenient in the locality."[1]

The Chinese are known to have used coal from the 4th century AD though in 1690 AD Chhii Ta-Chin notes that when "the iron ore is put in the (blast) furnace quantities of hard charcoal are mixed with it", indicating that charcoal was also used up to recent times.[1]

Aristotle reports in the 4th century that the Chalybians

Table 2
SELECTED SAMPLES ANALYZED AT ISOTRACE

a) Samples analyzed as a check on contamination

IsoTrace date #	Sample	%C	Weight (g)	^{14}C activity (x modern) (% error)	"Background" age (years BP)
TO-128-2	FUM 175958-23				
	Disko Island Iron	≈0.65	3.193	0.0267 (4.23)	29,090 ± 340
	STELCO STEELS				
TO-134-3	C1090-71	0.960	2.0	0.0213 (4.48)	30,910 ± 360
TO-314-5	C1090-76	0.960	0.700	0.0220 (2.99)	30,660 ± 240
	(+iron chip)	<0.001	5.88		
TO-285	5665-1 cast iron	4.87	2.35	0.0064 (9.46)	40,630 ± 760

b) Iron artefacts analyzed at IsoTrace

				Radiocarbon age (years BP)	Date (cal. AD)
TO-712	FROB-2-75				
	Frobisher Bloom #2	≈0.3	1.338	1,340 ± 70	640–760[1]
	(+iron chip)	<0.001	6.48		
TO-1085	MOD-19-8-77				
	Sri Lankan wootz	1.79	0.274	980 ± 40	1012–1038
	(+iron chip)	<0.001	5.1		

[1] An extracted fragment of charcoal from this bloom gave a calibrated age of 1006–1150 cal.AD, significantly younger than the iron. For discussion, see text.

smelted ores with the stone "pyrimachus", which Goodale and Speer[18] interpret as coal, though this is far from certain. Theophrastus talks of "pyromachos lithos", possibly the same as Aristotle's "pyrimachus", which is translated by Lidell Scott as "fire-fighting, fire-proof stone" and is identified as limestone by Persson.[19] The Romans are also known to have used coal, but whether for smelting or just for forging is unclear.[16] Goodale and Speer report the use of coal by Anglo-Saxon monks in the late 9th century AD in Britain.[18]

Apart from the Chinese, all these reports of coal are curiosities. Through to the 18th century charcoal was the prime fuel. De Morveau, in 1786, expounds: "only charcoal was used as a cement, just as it alone is still used today",[20] indeed, so much was used that local shortages of wood for it occurred (e.g. on Elba and in Gaul during Roman times).[17] Agricola, in his 'De natura fossilium' written in 1546, notes that "they (the blacksmiths) use only iron obtained with charcoal except when it is not available".

In fact, such large quantities of charcoal were required to smelt iron (e.g. 16 lb (7.25 kg) of charcoal for 1 lb (0.45 kg) iron)[7] that vast deforestation took place in the 17th and 18th centuries (e.g. in Pennsylvania).[21] This was coupled with a great increase in demand for wood for fuel and a demand for the land for agriculture,[22] and was not aided, in Britain at least, by an act passed by Queen Elizabeth I in 1558 restricting the use of timber for fuelling iron smelters.[18]

The first patents for coal-fired furnaces in Britain were granted to Sturtevan (1611) and Rovenson (1613), though it appears that these attempts failed, as the patent was re-issued in 1619 to D. Dudley in Worcester[18] who may have been successful, due to the low sulphur content of the nearby iron ores.[23] Coking of coal was started by Abraham Darby I in the late 1600's, and by 1750 this had become established in Britain. Coke-fired furnaces had pervaded mainland Europe by the end of the 19th century,[16] and today, coke is the standard fuel for smelting of iron in the developed world, although up to World War I, Sweden stood out amongst the developed countries as still operating charcoal-fired furnaces.[24] For most of Europe though, the end of the 18th century also marks the end of the use of charcoal as a fuel, and thus the youngest age for which a radiocarbon date may be obtained, except in a few, fortunately well-documented, cases. By the 19th century, the America's and

Commonwealth had converted to coke, and by the 20th century most of Asia and Africa had also switched, though some developing countries (e.g. parts of Africa) still seem to be barely out of the European Iron Age stage of metallurgical development.[25]

All this is generally good news for radiocarbon dating, as it ensures that the carbon incorporated into the iron is essentially contemporaneous with the manufacture of the iron itself, at least up to the 18th century. The only worry is the fact that it is known that smelters would often re-smelt previously produced tools, which can add older carbon into the system.[7, 26]

That different irons possessed different properties was well accepted from the beginning of the use of iron. Attempts to combine irons with different attributes to produce the 'ideal' tool, or weapon, resulted in many elaborate techniques being developed, such as pattern welding (known from the 3rd century AD through to the Viking period) in the west;[23] Damascening (from about the 7th century AD) in the Middle East and India;[23] co-fusion (from the 4th century AD) in China[1] and Japanese sword forging (from the 12th century AD).[27] All methods attempt to combine the shock resistant properties of low-carbon wrought iron with the strength and hardness of high-carbon steel, either by welding the two end members as a series of sheets or sections, as in pattern welding and Japanese samurai sword forging, or by combining the two during the smelting phase: co-fusion.

The art of steel-making, as opposed to the modern science, appears to have reached its pinnacle with the development of wootz steel in the Middle East and the Indian sub-continent, with the process possibly dating back to before the time of Alexander the Great (ca. 300 BC).[28] The complicated procedure of repeatedly heating and cooling the iron in a highly carbonaceous environment produced a high-carbon (1.3–2% C) steel which was then forged at low temperatures (thus preventing de-carburization) to yield a steel of great strength and toughness, famed the world over. It is generally accepted that this is the raw material for the legendary Damascus swords and armour used by the Saracens during the Crusades. Similar steels were produced in Russia (the bulat) and Persia (poulad janherder).[28] The charge used to create the high carbon environment varied: Richardson[29] noted a charge of magnetite, bamboo, charcoal and green leaves of certain plants, placed in a crucible of clay. Buchanan[30] stated that neither charcoal nor wood was added to the steel in the crucibles, only rice husks, with the sealed clay crucibles then placed in circular charcoal furnaces. Either sources of carbon would provide reliable radiocarbon ages.

Thus, with a little historical and archaeological help, the feasibility of dating an iron artefact may be surmised, and the date obtained from analysis correctly interpreted.

Applications of the technique

The Frobisher Bloom #2

In addition to the aforementioned bloom dated by the Brookhaven group, three other blooms associated with the Canadian Arctic voyages of Sir Martin Frobisher in the 1570's have been recovered. A bloom discovered at the site of an encampment on Kodlunarn Island, Northwest Territories, was excavated by a Smithsonian Institution field team in the early 1980's. The bloom, recovered in 1981, has been described[31] and discussion of the results of this, and previous dates is presented elsewhere.[32] The dated sample consisted of 1.338 g of low-carbon ($\approx 0.3\%$) iron from the outer edge of the bloom. The calibrated date of 640–760 AD is considerably older than expected, superficially supporting the possibility of the discovery, or re-working, of Norse remains. During the slicing of the bloom for examination, a fragment of charcoal was found embedded in the iron. This has also been dated, giving an age of 1006–1150 AD, markedly younger than the iron date, but still within the range of known Norse occupation of the area. A third, unrelated piece of carbon from the same site has also been processed, and gives an age of 25,640 ± 220 years BP. This old material is indicative of the use of coal at the site, and the ship's records also note coal as cargo for one of the voyages. Whether the coal was used for smelting, or merely for re-heating for forging is unclear. If we assume that the bloom is in fact of Elizabethan age, this requires 6–14% of the fuel to be radiocarbon-free (i.e. coal). For such a bloom of 5 kg, this would relate to approximately 5–12 kg of coal added to the charcoal during roasting and smelting. The analysed iron sample was from the surface of the bloom, therefore may be the result of surface contamination from later firing in a coal-fired hearth. Further analyses from the interior of the bloom are required to shed light on this problem.

Sri Lankan Wootz

The other iron sample so far analysed consisted of 274 mg of high-carbon (1.79%) wootz steel, collected from central-eastern Sri Lanka.[33] This is the only date thus far performed on a sample of wootz steel and the calibrated date of 1012–1038 AD helps constrain the historical development of both the region and the use of wootz metal. The piece is from the site of a current dam development on the Maduru Oyo River, a project that will drown the remains of previous dams and settlements that date back to the 5th century AD. Little information is known on the archaeology of this region, except that settlement ceased in the 13th century, and the dams from previous occupations have all but been washed away by the river.[33] The analytical significance of this date lies in the fact that it represents the smallest sample of iron to be dated using the radiocarbon technique; three orders of magnitude less material was required than would have been necessary for the first such analysis.

Conclusions

Since the first radiocarbon analysis of iron over 20 years ago, only 12 iron artefacts have been dated using this technique. This has largely been due to the large sample sizes (up to 1 kg) required for accurate results using radiocarbon decay-counting of the carbon. Accelerator mass spectrometry, capable of determining ^{14}C activities on very small samples of carbon (<1 mg is possible) has opened the door to revitalize the interest in obtaining dates on metallic artefacts, as much smaller samples are required. The analytical method at IsoTrace has been tested, and has been used to give radiocarbon dates for two irons of historical interest. One of these weighed only 274 mg, emphasizing the potential of this method for determining the age of artefacts from very small fragments.

The laboratory is now routinely processing iron samples for dating. The minimum sample size is dictated by the carbon content of the material. Currently, 5 mg carbon is preferred (equivalent to 10 g for a wrought iron sample, but only 250 mg for a 2% cast-, or high-carbon, iron), assuming a clean and uncorroded artefact. Naturally, the larger the sample, the more precise the analysis, as replicate runs may be performed. For smaller samples (<1 mg carbon) doping is required to provide sufficient carbon dioxide for cracking, this leads to a poorer precision on the date.

In 1968, van der Merwe and Stuiver[8] expressed the hope that the dating of iron would become a routine procedure at radiocarbon laboratories, thereby facilitating the ability to constrain and refine our chronology of metallurgical developments by the dating of the metals themselves. This potential has been established at the IsoTrace Laboratory.

Acknowledgements

The author greatly appreciates the support and help from his colleagues at the IsoTrace Laboratory, in particular: Roelf Beukens, Ted Litherland and John Rucklidge; also to Carl Bowen in sample preparation. Thanks also to an anonymous referee for helpful comments.

About the author

Richard Cresswell received a BSc in geology from the University of Sheffield, then took up graduate work at the University of Toronto, where he is currently completing his PhD on radiocarbon in meteorites. The dating of metallic artefacts was studied as part of his MSc at Toronto, and this work is continuing, and expanding to look at other metals and slags.

References

1. J Needham, **The Development of Iron and Steel Technology in China, London, 1958.**
2. H Löfquist and C Benedicks, **Det stora Nordenskiöldska jårnblocket från Uivak-Mikrostruktur och bildungssått,** *K. svenska VestenlkAkad. Handl.,* 1941, 19.
3. C B Moore, C F Lewis and D Nava, **Superior analyses of iron meteorites,** in *Meteorite Research,* New York, 1969.
4. G F Zimmer, **The use of meteoric iron by primitive man,** *J. Iron and Steel Inst.,* 1916, ii, 306–356.
5. J K Bjorkman, **Meteors and meteorites in the ancient Near East,** *Meteoritics,* 1973, **8,** 91–130.
6. R W Van der Veen, **Mineralogy and Ore Deposition, The Hague,** 1925.
7. N J van der Merwe, **The Carbon-14 Dating of Iron,** Chicago, 1969.
8. N J van der Merwe and M Stuiver, **Dating iron by the carbon-14 method,** *Curr. Anthropol.,* 1968, **9,** 48–53.
9. E V Sayre, G Harbottle, R W Stoenner, W Washburn, J S Olin and W Fitzhugh, **The carbon-14 dating of an iron bloom associated with the voyages of Sir Martin Frobisher,** *American Chemical Society Symposium Series,* no. 176, **Nuclear and Chemical Dating Techniques,** L A Currie (ed), Washington, D.C., 1982, 441–451.
10. for a review of accelerator mass spectrometry see A E Litherland, **Fundamentals of Accelerator Mass Spectrometry,** *Phil. Trans. R. Soc. Lond. A,* 1987, **323,** 5–21.
11. D J Donahue, A J T Jull and T H Zabel, **Results of radioisotope measurements at the NSF-University of Arizona tandem accelerator mass spectrometer facility,** *Nucl. Instrum. Methods,* 1984, **B5,** 162–166.
12. R G Cresswell, **Radiocarbon Dating of Iron using Accelerator Mass Spectrometry,** *Thesis,* University of Toronto, 1987.
13. D Gurfinkel, **An assessment of laboratory contamination at the IsoTrace Radiocarbon Facility,** *Radiocarbon,* 1987, **29,** 335–346.
14. W E Kieser, R P Beukens, L R Kilius, H W Lee and A E Litherland, **IsoTrace radiocarbon analysis — equipment and procedures,** *Nucl. Instrum. Methods,* 1986, **B15,** 718–721.
15. F Ulff-Møller, **Solidification history of the Kidlît Lens: Immiscible metal and sulphide liquids from a basaltic dyke on Disko, West Greenland,** *J. Petrol.,* 1985, **26,** 64–91.
16. L Aitchison, **A History of Metals,** London, 1960.
17. G E Stahl, 1703, his work is summarized in T A Wertime, **The Coming of the Age of Steel,** Chicago, 1962.
18. *passim* S. L Goodale and J R Speer, **Chronology of Iron and Steel,** Pittsburgh, 1920.
19. R J Forbes, **Studies in Ancient Technology: vol. 9,** Leiden, 1964.
20. S Rinman, **Rön om etsning pa järn och stal,** *K. Vetenskaps Akad. Handl.,* **35,** 1–14, English

transl. by A Chisholm, printed in C S Smith, **Sources for the History of the Science of Steel,** Cambridge, Mass., 1968.
21. E F Heite, **The Delmarva bog iron industry,** *Northeast Hist. Archaeol.,* 1974, **3** (2), 18–34.
22. R J Forbes, **Studies in Ancient Technology: vol. 8,** Leiden, 1964.
23. R F Tylecote, **A History of Metallurgy,** London, 1976.
24. H H Coghlan, **Notes on Prehistoric and Early Iron in the Old World,** Oxford, 1977.
25. N J van der Merwe and R T K Scully, **The Phaloborwa story: archaeological and ethnographic investigation of a South African Iron Age group,** *World Archaeol.,* 1971, **3**, 178–196.
26. R F Tylecote, **The Prehistory of Metallurgy in the British Isles,** London, 1986.
27. M Chikashige, **Alchemy and other Chemical Achievements of the Orient,** Tokyo, 1936.
28. J Wadsworth and O D Sherby, **On the Bulat — Damascus steels revisited,** *Prog. Mat. Science,* 1980, **25**, 35–68.
29. H C Richardson, **Iron prehistoric and ancient,** *Amer. J. Archaeol.,* 1934, **38**, 555–583.
30. F Buchanan, **A Journey from Madras through the Countries of Mysore, Canara, and Malabar,** London, 1807, cited in 23.
31. H Unglik, **Metallurgical study of a recently recovered iron bloom and associated finds from Kodlunarn Island, Frobisher Bay, Canada,** manuscript on file, National Parks and Sites, Environment Canada — Parks, Ottawa, 1987.
32. G Harbottle, R G Cresswell, and R W Stoenner, **Carbon-14 dating of iron blooms from Kodlunarn Island.** In W W Fitzhugh and J Olin (eds) **The Archaeology of the Frobisher Voyages: a contribution to the archaeology of Kodlunarn Island, Frobisher Bay, Northwest Territories, Canada.** Washington, in press.
33. S Brown, pers. comm.

Obituary

Professor William Oliver Alexander died aged 81 in the South Warwickshire Hospital in February 1992. Few historical metallurgists have been so well known outside the profession as Bill Alexander who, in 1944 with his co-author Arthur Street, published in the Penguin format *Metals in the Service of Man.* It immediately became a best seller which had to be reprinted in 1945 and 46 and has since been revised and reprinted ten times, last appearing on the bookstalls in 1989. By then it had sold well over half a million copies, truly the most popular book ever written about metallurgy.

Bill was born in Bramford near Lowestoft on 8 July 1911 and so technically was an East Anglian. His father, working in Suffolk for a Birmingham sportsware manufacturer, returned to the Midlands shortly after and it is as a 'Brummie' both in attitude and accent that we came to know him.

King Edward's Grammar School, Camp Hill, followed by a degree at Birmingham University and post-graduate research led to this PhD and to the Metal Group of Imperial Chemical Industries, now known as IMI, where he stayed from 1937 to 1955. These were momentous years for non-ferrous metals and processes. During the war years Bill worked on extruded uranium and titanium developments which have since assumed great importance for IMI.

In 1955 Bill went to the USA, representing ICI in New York for three years, and after leaving ICI in 1961 became Technical Director of Forseco International Limited until 1967. On creation of the new University of Aston in Birmingham he became Professor and Head of the Department of Metallurgy, a position which he held from 1967 until he retired in 1976. From then he was emeritus professor and research fellow.

Metals in the Service of Man had incorporated the early industrial history of many metals and metal processes, establishing Bill's interest in the field of post-industrial revolution metallurgy, but it was in the sphere of twentieth-century metals history that he was pre-eminent. As a co-ordinator of metallurgical innovations he was unparalleled and after his 1965 publication by Pergamon Press, *Metallurgical Achievements*, celebrating sixty years of the Birmingham Metallurgical Society, in which he collected papers from fifteen eminent practitioners, he moved on to promote the 1984 HMS Alloys Conference in Birmingham.

In this, he enlisted the assitance of twenty-one contributors, many of them war-time colleagues, to present a resume of metallurgical development from 1900 to 1950 which has been published as *JHMS Volume 19, numbers 1 and 2*, a magnificent record of recent history.

However, Bill was more than mere metallurgist. From his early days he was an active sportsman, playing and then refereeing rugby, taking part in swimming and organising athletics. He became a Governor and Bailiff of King Edward VI Schools in Birmingham and President of the Birchfield Harriers. The main entrepreneur of this last organisation over 100 years ago was W W Alexander, Bill's grandfather, in whose honour the City of Birmingham athletics stadium is named.

In the recent past Bill's labour of love was to compile and see through to publication a history of the Birchfield club. When switching on to watch a television evening of athletics from the Alexander Stadium, I think of Bill. He just kept on running.

Roy Day