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Dave + Nick.

Reassessment of the Evidence for Early Metallurgy in Niger, West Africa

David Killick[a], Nikolaas J. van der Merwe[b], Robert B. Gordon[c] and Danilo Grébénart[d]

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A large number of structures that appear to be forges or smelting furnaces have been excavated by D. Grébénart in the Agadez region of Niger. Many of the calibrated radiocarbon dates from these structures fall in the second and third millennia BC, more than a millennium older than the earliest previous dates for metallurgy in sub-Saharan Africa. Chemical and microstructural studies of the fused materials from these structures show that most of the samples dated prior to 1000 bc are partially vitrified soil and cannot be positively associated with a metallurgical process. The only positive evidence for metallurgy in this region in the second/third millennium BC is a single radiocarbon date of 1710 ± 110 bc (GIF-5176) for a copper-working furnace. This date may reflect the use of old charcoal and should be viewed with caution until thermoluminescence dates can be obtained for this furnace.

After 1000 BC, native copper and copper oxide minerals were processed in non-tapping shaft furnaces. Calcite, dolomite and aluminosilicate gangue minerals have combined to produce unusual red melilite slags. The scale of production appears to have been very small. Iron smelting came into general use in this region around 500 BC, but the origins of this technology are still unclear.

Keywords: TECHNOLOGY, METALLURGY, COPPER, NATIVE COPPER, IRON, SLAGS, AFRICA, SAHEL, NIGER, RADIOCARBON, MICROSCOPY.

Introduction

The arid plains southwest of the Air mountains in central Niger, west of the town of Agadez, are part of the Sahelian zone of dry savanna that abuts against the southern fringes of the Sahara desert (Figures 1 & 2). There was little archaeological exploration of this region before the late 1970s, when a concession was granted for the strip-mining of uranium. Between 1977 and 1981, a team of French scholars undertook a surface survey of the area covered by the concession, which is some 36,000 square kilometres in extent. While surveying Neolithic sites in this area, Danilo Grébénart recovered many artifacts of copper or copper alloys and located numerous structures of fired earth, which were

DMK S

[a]Department of Anthropology, Yale University, U.S.A.
[b]Department of Archaeology, University of Cape Town, South Africa.
[c]Department of Geology and Geophysics, Yale University, U.S.A.
[d]Laboratoire d'Anthropologie et de Préhistoire des Pays de la Méditerranée Occidentale, Aix-en-Provence, France.

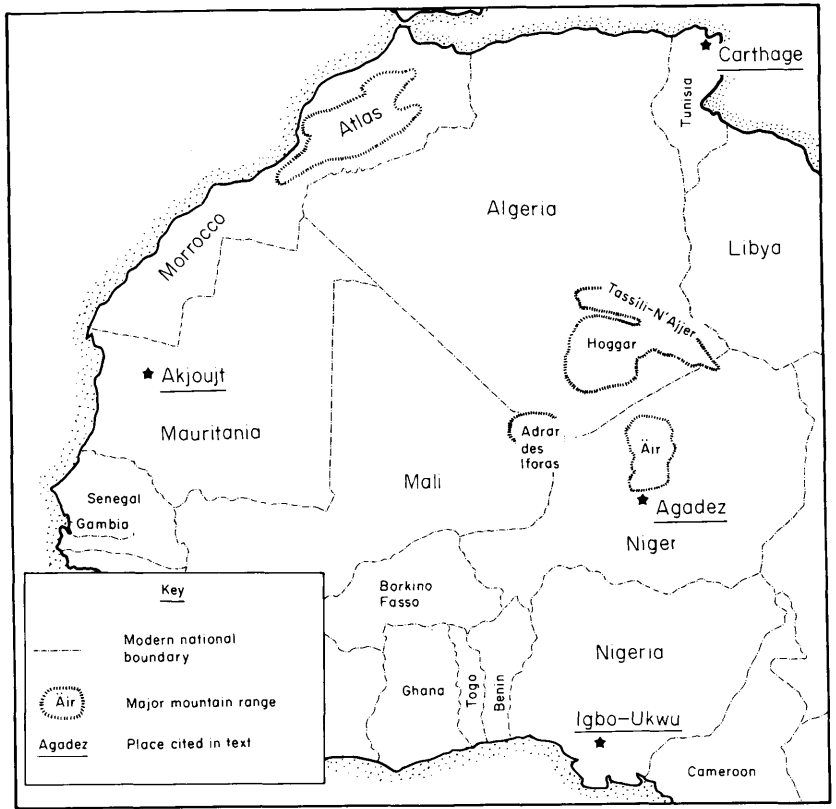

Figure 1. Map of West Africa, showing the location of Agadez and some other places referred to in the text.

tentatively identified as metallurgical furnaces (Grébénart, 1983, 1985, 1987). Several dozen of these were excavated, and more than 30 radiocarbon dates were obtained on wood charcoal from within the structures (Table 1). When calibrated, the oldest dates lie in the late second and third millennia BC. These are more than a millennium older than any previous radiocarbon date for metallurgy in sub-Saharan Africa.

Grébénart (1983) assigned the evidence to four overlapping periods, based on the radiocarbon dates, the shape of the structures and the visual appearance and chemical composition of the slags that they contained. The periods so defined (in radiocarbon years) were "Cuivre 1" (*c.* 2000 bc to *c.* 1000 bc), "Cuivre 2" (*c.* 1000 bc to *c.* 100 bc), "Fer 1" (*c.* 500 bc to *c.* 1 ad) and "Fer 2" (after 1 ad). The distinction between "Cuivre 1" and "Cuivre 2" followed the interpretation of J.-R. Bourhis, who has made several dozen chemical or spectrographic analyses of the metal artifacts and slags. Bourhis concluded that the materials classified as "Cuivre 1" derived from the melting of native copper, but that those from "Cuivre 2" were produced by smelting copper oxide minerals with a calcareous flux (Bourhis, 1983; Bourhis in Grébénart, 1985: 157–160, 247–255, 331–335). This distinction was not recognized by Tylecote (1982), who studied a subsample of these materials and concluded that only the melting of native copper was practised in either period.

The remarkably early radiocarbon dates for "Cuivre 1" have attracted much attention among African archaeologists and historians. The earlier work of Nicole Lambert at Akjoujt (Mauretania) had shown that copper was smelted in the Sahel as early as the mid-first millennium bc (Lambert, 1971, 1983). Some of the associated metal work had clear stylistic parallels in Phoenician North Africa, and it therefore seemed likely that the copper-smelting technology had been introduced from that region. By contrast, the earliest radiocarbon dates on apparent furnace structures in Niger are in the late second and early third millennia bc. There is at present no evidence of extractive metallurgy in North Africa that is of comparable age. The few bronze and copper objects of this period reported from North Africa appear to have been imported from Iberia (Harrison & Gilman, 1977). The dates from Agadez have therefore been widely, if tentatively, accepted as evidence for the independent development of metallurgy in West Africa (e.g. Phillipson, 1985: 170; Craddock & Picton, 1986: 5–6).

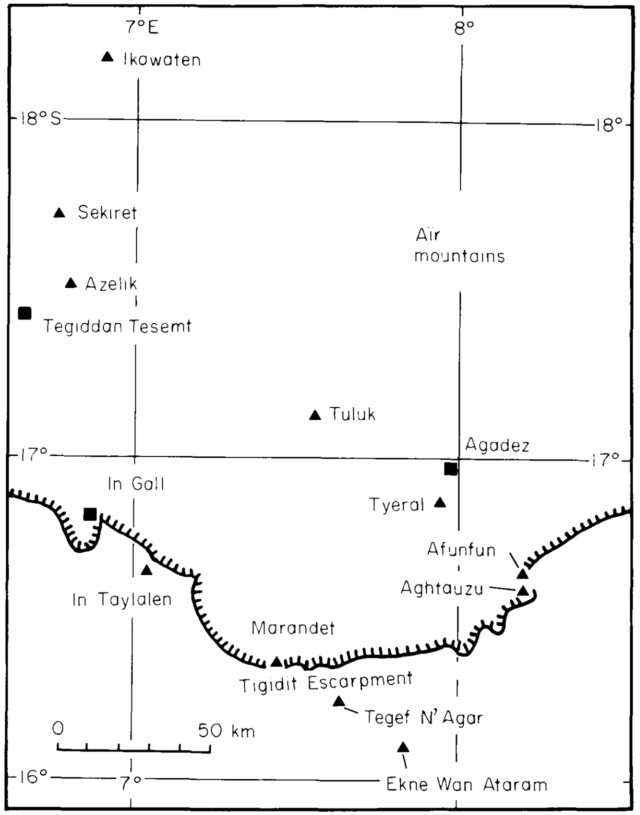

Figure 2. Map of the vicinity of Agadez, showing the location of modern settlements (■) and the archaeological sites (▲) listed in Table 1.

The evidence from Niger is also of relevance to the long-running debate on the origins of iron working in Africa. There are radiocarbon dates in the mid-first millennium bc for iron-smelting sites in several areas of West and East Africa (Quéchon & Roset, 1974; Tylecote, 1975; Schmidt, 1978: 190–191; Shaw, 1981; van Grunderbeek *et al.*, 1982; Schmidt & Childs, 1985). Some scholars have argued that these are evidence of the independent invention of iron smelting in Africa (e.g. Schmidt, 1978, 1983; Andah, 1979). One of the major points against this argument has always been the absence of evidence for a preceding tradition of high-temperature pyrotechnology in any of these regions. The

Table 1. Radiocarbon dates for "Cuivre 1", "Cuivre 2" and "Fer 1"

Site, structure	Radiocarbon date	Calibrated range
"Cuivre 1"		
Afunfun 175, furnace 1	1970±90 bc (MC-2398)	BC 2855–2824, 2657–2640, BC 2620–2180, 2166–2142
	2190±90 bc (MC-2379)	BC 2920–2470
	1730±60 bc (Pta-3964)	BC 2280–2230, 2210–1900
	440±100 ad (OxA-1115)	AD 268–275, 340–680
Afunfun 175, furnace 2	1650±90 bc (MC-2400)	BC 2200–1740
	1850±90 bc (MC-2401)	BC 2554–2547, 2490–2020, BC 2002–1979
Afunfun 175, furnace 3	1730±50 bc (GIF-5172)	BC 2200–1930
Afunfun 175, furnace 4	1150±70 bc (GIF-5173)	BC 1520–1250, 1246–1215
Afunfun 175, furnace 5	1630±100 bc (GIF-5174)	BC 2200–1680
	1520±50 bc (Pta-3969)	BC 1930–1680
Afunfun 175, furnace 7	1730±100 bc (GIF-5175)	BC 2453–2423, 2400–1870, BC 1842–1813, 1807–1777
Afunfun 175, furnace 8	1710±110 bc (GIF-5176)	BC 2453–2423, 2400–1750
Afunfun 175, furnace 12	1560±100 bc (GIF-5177)	BC 2135–2051, 2050–1610
Aghtauzu 178, furnace 1	950±110 bc (GIF-5179)	BC 1420–830
Sekiret, furnace 1	1360±100 bc (GIF-3862)	BC 1880–1410
Sekiret, furnace 2	950±100 bc (GIF-4177)	BC 1410–840
"Cuivre 2"		
Afunfun 162, furnace 9	570±90 bc (MC-2402)	BC 840–400
Afunfun 162, furnace 10	570±90 bc (MC-2403)	BC 840–400
Afunfun 162, furnace 25	850±90 bc (MC-2404)	BC 1258–1235, 1230–810
Afunfun 162, furnace 27	640±90 bc (GIF-5541)	BC 920–410
Afunfun 162, furnace 28	590±90 bc (MC-2405)	BC 892–882, 850–400
Afunfun 162, furnace 44	790±90 bc (MC-2406)	BC 1124–1113, 1110–790
Ikawaten 193, furnace 1	670±70 ad (GIF-5184)	AD 640–890
Ikawaten 193, furnace 2	140±90 bc (GIF-5185)	BC 380–AD 80
Ikawaten 193, furnace 3	210±90 bc (GIF-5186)	BC 400–AD 20
Azelik 210, furnace 1	560±70 bc (GIF-5546)	BC 810–400
Azelik 210, furnace a	540±90 bc (GIF-4175)	BC 820–390
Azelik 210, furnace b	530±90 bc (GIF-4330)	BC 820–390
Azelik 210, furnace c	450±90 bc (GIF-?)	BC 800–360, BC 283–257
Azelik 210, furnace d	90±90 bc (GIF-3863)	BC 362–282, BC 260–AD 130
Tyeral 207, furnace 1	460±90 bc (GIF-5542)	BC 800–370, 276–264
Tuluk 211, furnace 1	410±70 bc (GIF-5545)	BC 765–672, 668–612, BC 610–360, 283–258
"Fer 1"		
Tegef n'Agar 74, pit 1	140±90 bc (GIF-4172)	BC 380–AD 80
Ekne wan Ataram 119	490±90 bc (MC-2397)	BC 810–380
In Tayalen II/15, pit 1	260±90 bc (GIF-4170)	BC 410–40
In Tayalen II/15, pit 2	60±90 bc (GIF-4171)	BC 349–315, BC 210–AD 150, AD 162–194

All dates are on wood charcoal and are based on the 5568-year half-life. Pta-3964, Pta-3969 and OxA-1115 have not been previously published. All other dates are from Grébénart (1985). The calibrations used are those of Stuiver & Pearson (1986) and Pearson & Stuiver (1986). The calibrated ranges were calculated by the computer program CALIB 1.3, written by P. Reimer and M. Stuiver (University of Washington), and are at two standard deviations. No error multiplier has been applied.

early radiocarbon dates from Agadez appear to dispose of this objection, as Schmidt has noted (1983: 432–433). Furthermore, the sequence of metallurgical technique at Agadez, as reconstructed by Bourhis (1983), reproduces the sequence of technological stages known from the Near East (working of native copper/copper smelting/iron smelting).

There are, however, a number of problems with the primary evidence from Niger that must be resolved before any wider implications can be drawn. Many of the fired clay structures assigned to "Cuivre 1" have very irregular shapes, and it is by no means certain that these are metallurgical furnaces or forges. Only one of the "Cuivre 1" structures contained more than a few tens of grams of slag, and no slag heaps or tuyeres were recovered in association with these structures. The two previous studies of the slags from the "Cuivre 2" furnaces produced quite different reconstructions of the technology employed. Tylecote (1982) concluded that these materials derived from the melting of native copper in crucibles, while Bourhis (1983) argued for the smelting of copper oxide minerals in a furnace with a calcareous flux. The present laboratory study was undertaken to resolve these uncertainties. It has not yet been possible to re-examine these structures in the field.

Scope of this Study

We have made chemical and microstructural studies of new samples of the materials recovered from the structures of "Cuivre 1" and "Cuivre 2", and have compared our results to those previously reported by Bourhis and Tylecote. Bulk chemical analyses (by X-ray fluorescence) were obtained by van der Merwe. The optical and electron microscopy was done by Killick and Gordon. We also report two new conventional radiocarbon dates, provided by Dr J. C. Vogel (C.S.I.R., Pretoria), and an AMS radiocarbon date from the Oxford accelerator. Two additional chemical analyses were provided by Dr P. de Paepe (Laboratorium voor Aardkunde, Rijksuniversiteit Gent).

"Cuivre 1": Sample descriptions

Most of the material assigned to "Cuivre 1" was recovered from one site, Afunfun 175. The site consists of a roughly linear zone of several dozen pit structures, each of which is delineated by a hard fired lining (Grébénart, 1985: figure 78). Some are regular circular bowl furnaces or small cylindrical shaft furnaces, but in others the space enclosed by the fired lining is very irregular indeed. Eighteen of these structures were excavated. Full descriptions and plans of these have been published by Grébénart (1985: 113–160). Eight contained datable charcoal, from which a total of 13 radiocarbon dates have been obtained so far (Table 1). With one exception, to be discussed below, all fall in the second and third millennia bc.

Most of the excavated structures also contained small amounts of fused materials. Chemical analyses of samples of these materials have been made by Tylecote (1982) and Bourhis (1983), and are reproduced here in Tables 2 and 3. The chemical analyses cannot, however, be interpreted without knowledge of their mineralogy, and microstructure. We have therefore obtained chemical, microstructural and mineralogical data for the following additional samples.

Afunfun 175, furnace 9

This structure was a shallow, elongated basin 2·2 m long with two lobes of unequal area. The sides and floor of the structure were baked hard. The interior was scored with parallel grooves (Grébénart, 1985: figure 89), and a scatter of pieces of baked lining was excavated

Table 2. Analyses of fused products from "Cuivre 1" structures

	1	2	3	4	5	6	7	8	9	10	11
Site	Afunfun 175	Afunfun 175	Afunfun 175	Afunfun 175	Afunfun 175	Afunfun 175	Afunfun 175	Afunfun 175	Afunfun 175	Afunfun 175	Afunfun 175
Furnace	9	8	8	6	6	6	1	1	1	1	1
SiO_2	60·21	62·31	63·5	60·98	65·5	62·7	62·18	59·0	69·0	20·53	20·74
TiO_2	1·21	1·12	—	1·20	—	—	1·19	1·4	—	0·65	0·61
Al_2O_3	20·91	19·98	—	21·23	—	—	17·47	22·5	—	6·30	6·42
Fe_2O_3	8·76	7·69	—	9·12	—	—	6·66	—	—	72·42	69·80
FeO	—	—	31·2	—	28·0	33·1	—	6·7	27·8	—	—
MnO	0·10	0·13	0·15	0·12	0·15	0·05	0·11	0·2	0·1	0·07	0·07
MgO	1·75	1·74	—	1·80	—	—	1·75	1·0	—	1·05	0·97
CaO	1·37	1·98	4·2	1·68	2·8	3·7	2·20	1·4	2·5	0·32	0·45
Na_2O	1·33	1·46	—	1·35	—	—	1·97	1·3	—	0·07	0·06
K_2O	2·77	3·04	—	2·87	—	—	2·85	3·1	—	0·28	0·40
P_2O_5	0·19	0·22	—	0·22	—	—	—	—	—	0·77	0·94
Cu metal	·tr	·tr	1·0	·tr	3·5	0·5	·tr	n.d.	0·3	·tr	·tr
H_2O-	0·26	0·22	—	0·23	—	—	—	—	—	0·41	0·56
LOI	1·23	0·32	—	−0·13	—	—	—	—	—	−3·79	−2·34
Total	100·09	100·21	100·05	100·70	99·95	100·05	96·38	96·6	99·70	99·08	98·66
Cu (trace)	24 ppm	26 ppm		26 ppm			33 ppm	—		32 ppm	40 ppm
Analysis	van der Merwe	van der Merwe	Bourhis	van der Merwe	Bourhis	Bourhis	de Paepe	Tylecote	Bourhis	van der Merwe	van der Merwe
Method	XRF	XRF	?	XRF	?	?	?	EPMA	?	XRF	XRF

Key: —, Not analysed; ·tr, trace element; n.d., below limit of detection; XRF, major elements by X-ray fluorescence, H_2O- and loss on ignition (LOI) by gravimetry; EPMA, electron microprobe. Analyses by de Paepe and van der Merwe not previously published; others from Tylecote (1982) and Grébénart (1985).

Table 3. Semiquantitative analyses of fused products from "Cuivre 1" structures

	1	2	3	4	5	6	7	8
Site	Afunfun 175	Afunfun 175	Afunfun 175	Afunfun 175	Afunfun 175	Afunfun 175	Afunfun 175	Afunfun 175
Furnace	1	1	3	6	8	9	10	10
SiO_{23}	60%	60%	60%	60%	60%	60%	60%	60%
Al_2O_3	25%	25%	20%	25%	25%	25%	25%	25%
FeO	10%	10%	15%	10%	9%	10%	10%	10%
MgO	1%	2%	2%	2%	2%	2%	2%	2%
CaO	2%	1%	1%	1%	2%	1%	1%	1%
Na_2O	2%	2%	2%	1%	2%	2%	2%	2%
Cu metal	0·2%	100 ppm	10 ppm	50 ppm	10 ppm	n.d.	n.d.	n.d.

Analyses by Jean-Roger Bourhis (in Grébénart 1985: 157–160). The analyses are by arc spectrography. The copper values are quantitative; other values are semi-quantitative and were obtained by calibrating the spectra against those of chemically analysed reference slags. n.d., below limit of detection.

from the former ground surface at one end. These suggest that the structure was raked out before abandonment. Several pieces of fused material were excavated from the furnace base. There was no charcoal in the fill, and the structure is not dated. It was assigned to "Cuivre 1" because both the form and contents of this structure are very similar to those of adjacent structures, which produced radiocarbon dates in the second and third millennia bc.

Sample description: A sample of the fused material from this furnace is dark green and is of relatively low density. The exterior surfaces are smooth and bulbous and show evidence of rather viscous flow. Broken surfaces are rough and packed with tiny bubbles; the texture is similar to that of pumice. There are no charcoal impressions. In thin-section, small angular fragments of quartz, microcline, plagioclase feldspar and opaque minerals can be seen in a dark glass. The glass is evidently inhomogeneous, as there is great variation in its colour and transparency within the area sectioned. No metallic iron or copper was noted. The colour of the specimen was matched against the colour chips of the Munsell Color Chart (Munsell Color Company n.d.). It varied from deep blue-green (Munsell 7·5 BG 5/2) in polished section to deep brown. The streaks of different colours are evidence of poor mixing, which also suggests that the sample was very viscous at the temperature of its formation. The large amount of entrapped gas is further evidence of high viscosity. Some regions of the slide consist only of gas bubbles bounded by thin walls of glass. Some flow or slumping of the hot mass can be inferred from the arcuate shape of the largest voids.

A chemical analysis of this sample is given in Table 2 (analysis 1). The dominant constituents are silica (60%), alumina (21%) and iron oxide (9%). It contains only 24 ppm copper. This is very similar to the analysis of another sample from this structure, made by J.-R. Bourhis (Table 3, analysis 6).

Interpretation: This sample is not a metallurgical slag. The presence of the angular quartz, feldspar and opaque minerals indicate that this sample is a partially vitrified soil. The glass has probably formed by reaction between the clay minerals and metal oxides (CaO, FeO, Na_2O, K_2O) in the finest size fractions of the soil. Samples of this kind are often found in and around ancient furnaces, and were formed by partial fusion of the earth walls or the tuyeres. But they do not in themselves prove that a metallurgical operation was carried out in this structure. Similar materials may be produced in pottery-firing pits, for example.

Afunfun 175, furnace 8

This structure is the base of a circular shaft 30 cm in internal diameter with a single opening at base level (Grébénart, 1985: 121–123). The circular shaft was inclined away from the opening at an angle of 15° to the vertical. The furnace fill contained several pieces of fused material and wood charcoal. A sample of the latter gave a radiocarbon date of 1710 ± 110 bc (GIF-5176).

Three samples of the fused material have been chemically analysed. One sample analysed by J.-R. Bourhis (Table 2, analysis 3) contained 63·5% SiO_2, 31% FeO, 4% CaO and 1% metallic copper (Grébénart, 1985: 159). The sample analysed in this study (Table 2, analysis 2) contains 62% SiO_2, 20% Al_2O_3 and 8% Fe_2O_3, with only 26 ppm copper. This is very similar to a third sample analysed by Bourhis (Table 3, analysis 5).

The thin-section corresponding to analysis 2 consists largely of isotropic brown glass enclosing small spherical and larger irregular voids (Figure 3). Angular fragments of strained quartz, magnetite, microcline and plagioclase feldspars are scattered throughout the glass. At 400 ×, tiny crystallites can be seen in the glass, but it was not possible to identify them. No metallic iron or copper was noted. The high volume of entrapped gas suggests that the glass was very viscous at furnace temperature.

Interpretation: This sample is very similar in all respects to that described above for furnace 9 and is also a partially vitrified soil. As the chemical composition of one of the samples analysed by Bourhis (Table 3, analysis 5) is almost identical, we infer that this too is a fired soil. The other sample analysed by Bourhis (Table 2, analysis 3) is, however, very different as it contains 1% copper. We consider this to be strong evidence for some form of copper metallurgy. Unfortunately, no microsection was made from this specimen before it was analysed, so we can say little about the technology employed. The liquidus temperature of the non-metallic portion of this sample (65% SiO_2, 31% FeO, 4% CaO) is well above 1700°C (Osborn & Muan, 1960; figure 7). It is therefore highly unlikely that this sample was entirely molten. It may, however, be a normal fayalitic smelting slag (*c.* 70% FeO, 30% SiO_2, liquidus temperature *c.* 1200°C) enclosing fragments of undissolved silica.

Afunfun 175, furnace 6

This structure was a simple bowl 100 cm in diameter, with a maximum depth of 40 cm. No charcoal was recovered from the fill, and the structure is undated. It was provisionally placed in "Cuivre I" because it is surrounded by other structures (furnaces 1, 5 and 7) with radiocarbon dates older than 1500 bc (Grébénart, 1985: 115). The major feature of interest in this case is that one of the slag samples from the fill contained a small prill, which was analysed by J.-R. Bourhis. The composition was 82% copper, 15% tin, 1% lead, 1% SiO_2, 0·5% arsenic and 0·15% iron. The slag sample from which the prill was extracted (Table 2, analysis 5) is 65·5% SiO_2, 28% FeO, 2·8% CaO and 3·5% copper metal. A second piece of slag analysis by Bourhis (Table 2, analysis 6) was very similar.

Other samples from this same structure have quite different chemical compositions. A third sample analysed by Bourhis (Table 3, analysis 4) is *c.* 60% SiO_2, 25% Al_2O_3 and 10% FeO, with only 50 ppm copper. This is very similar to the sample analysed by van der Merwe (Table 2, analysis 4) which contains only 26 ppm copper.

The hand specimen of this last sample is of very low density, comparable to that of pumice. The colour of a polished surface is dark green, Munsell 5 BG 3/1. In thin-section, the sample consists largely of films of glass enclosing abundant spherical voids. The glass varies in colour from clear through greenish brown to almost opaque. Above 400 ×, tiny crystallites can be seen in the glass. The glass contains about 20% (by volume) angular fragments of quartz, magnetite and a little plagioclase feldspar.

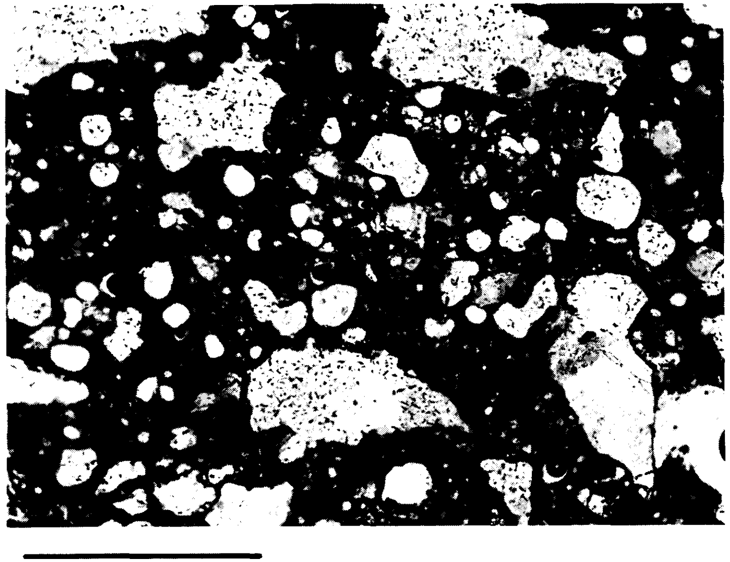

Figure 3. Plane-polarised, transmitted-light micrograph of a sample from Afunfun 175, furnace 8. The white areas containing small dark speckles are voids; the speckles are a filler in the mounting epoxy. Smaller irregular white areas are angular grains of quartz and plagioclase feldspar. The matrix is a brown glass. The scale bar is 0·5 mm.

This sample was examined with an energy-dispersive X-ray analyser (EDAX) coupled to a scanning electron microscope. This showed that the residual magnetite grains are titaniferous and that the tiny crystallites in the glass are of two types. The first type contains iron and some titanium, while the only significant peaks for the second type are those of silicon. The composition of the glass is very variable, which suggests poor mixing because of high viscosity. The dominant oxides in the glass are silica and alumina, with variable proportions of iron, calcium and potassium oxides.

Interpretation: The specimen studied in thin-section is a partially vitrified soil, similar in all respects to the samples described above from furnaces 9 and 8. The third sample analysed by Bourhis (Table 3, analysis 4) is chemically almost identical. It, too, is probably a fired soil. The other two samples analysed by Bourhis are quite different (Table 2, analyses 5 and 6). They are, however, very similar to the sample with 1% copper from furnace 8, discussed above. We consider both samples to be firm evidence for copper metallurgy. No microsections of this material are available, and we cannot therefore say much about the technology employed.

Afunfun 175, furnace 1

This extraordinary structure is of a particular interest and has been the subject of much controversy. Several cycles of analysis and radiometric dating have been made on the contents of furnace 1. These are recalled here in chronological order.

Excavation of the structure revealed a very complex shape (Figure 4), with an asymmetric chamber below ground level surmounted by two circular apertures about 50 cm in diameter. Irregular funnel-shaped tunnels projected from the east side of the chamber (Grébénart, 1985: 114–121). Some of these were entirely filled with wood charcoal. Two radiocarbon dates were obtained on samples of charcoal from these tunnels. The uncalibrated dates were 1970 ± 90 bc (MC-2398) and 2190 ± 90 bc (MC-2379). These are the earliest radio carbon dates obtained for "Cuivre 1".

The walls of the chamber were baked hard. Some 10 kg of fused material was present in the fill or fused to the walls of the chamber. Samples of these were studied by Tylecote (1982) and Bourhis (1983). Tylecote analysed one sample by electron microprobe. His analysis is reprinted here as Table 2, analysis 8, and shows the dominant oxides to be SiO_2 (59%), Al_2O_3 (22·5%) and FeO (6·6%). He also examined several samples of materials from "Cuivre 2". His conclusion was that the "Cuivre 1" and "Cuivre 2" materials were both products of the same technology, which was the melting of native copper in crucibles. On this interpretation, the sample from furnace 1 is presumably a piece of partially melted crucible or furnace lining, although Tylecote does not provide an explicit identification.

J.-R. Bourhis (1983) analysed a further three samples from this structure. Two of these (reprinted here as Table 3, analyses 1 and 2) are similar to that reported by Tylecote. A third sample (Table 2, analysis 9) has a composition of 69% SiO_2, 28% FeO, 2·5% CaO and 0·3% copper metal. This is very similar to the composition of samples from furnaces 8 and 6, discussed above. Bourhis concluded that these samples—and all others from "Cuivre 1"—were products of the refining of native copper in a furnace (Bourhis, 1983; Bourhis, in Grébénart, 1985: 158). As this interpretation was incompatible with that suggested by Tylecote, Grébénart sought a third opinion from van der Merwe, who examined all the remaining materials and selected samples for further analysis.

Sample descriptions: Hand samples of the fused materials from this structure are of two very different types. Samples of the first type are essentially identical in hand specimen to those described above for other "Cuivre 1" structures. The density and texture resemble that of pumice. The colour of freshly broken surfaces is generally dark green (Munsell 5 BG 3/1) or greyish purple (Munsell 5 P 4/4). Streaks of different colour in the same piece attest to incomplete mixing of the material. The external surfaces are angular to subrounded. Some pieces contain patches of a red-brown material (Munsell 5 YR 5/6) that appears to be baked earth. Flow structures are poorly developed, and there are no clear charcoal impressions. The material was evidently very viscous at the temperatures attained within the furnace. Analyses of these materials by Tylecote and Bourhis are cited above. An additional analysis of this type of material was made by P. de Paepe (letter of 4 July 1984 to D. Grébénart), and is reprinted here as Table 2, analysis 7. It is essentially the same as that obtained by Tylecote.

The second type of fused material from furnace 1 is quite different. The samples are much denser and are matt black with orange to brown weathering stains. Cut surfaces show only a few large circular gas cavities. Small pieces of charcoal have been enveloped by the slag. Where these have eroded out, the impressions left in the slag preserve fine detail of the surface of the charcoal pieces. This type of slag was therefore much more fluid at furnace temperatures than the other samples from "Cuivre 1" structures, described above. Chemical analyses of samples of this material were obtained by van der Merwe (Table 2, analyses 10 and 11). Both samples contain about 70% FeO, 20% SiO_2 and 6% Al_2O_3. They differ markedly from the silica- and alumina-rich, iron-poor analyses of other "Cuivre 1" samples.

Six sections of this material were examined in reflected or transmitted light. All were very similar and contained the following minerals.

Fayalite: This is the dominant mineral in all six sections. It is present in the form of skeletal laths, often crystallized around grains or dendrites of opaque minerals (Figure 5). The optical properties and EDAX spectrum identify this mineral as fayalite (Fe_2SiO_4), which is the most commonly reported mineral from ancient iron- and copper-smelting slags (Bachmann, 1980; Fells, 1983).

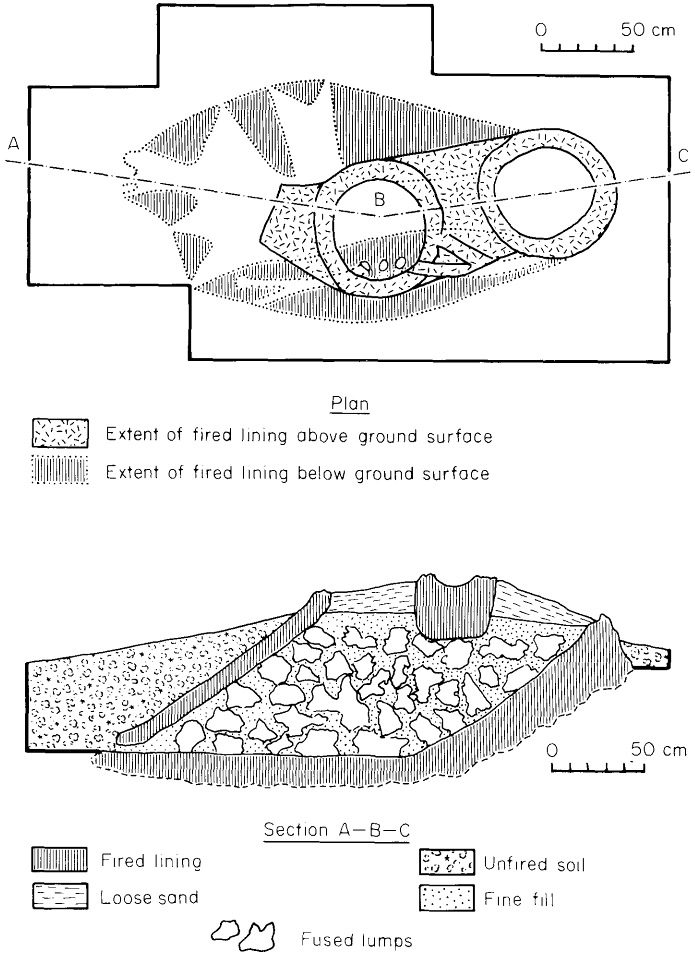

Figure 4. Plan and section of Afunfun 175 furnace 1 (after Grébénart, 1985: figure 79).

Wustite: An opaque constituent of moderate reflectivity forms rounded dendrites, the larger examples of which clearly crystallized before the fayalite crystals. The only EDAX peaks for this constituent are those of iron, and its Vickers microhardness is 540 (25 g load, $n = 10$, range 503–572). These characteristics identify the constituent as wustite (Fe[1-x]O), which is one of the most commonly observed mineral phases in the microstructure of prehistoric iron- and copper-smelting slags. Wingrove (1970) reported a Vickers microhardness of 506 (25 g load, $n = 10$, range 483–525) for pure synthetic wustite.

Hercynite: In thin-section, this constituent is opaque and is euhedral with rhombic or polyhedral crystal habit. In polished section, it has very low reflectivity, slightly less than

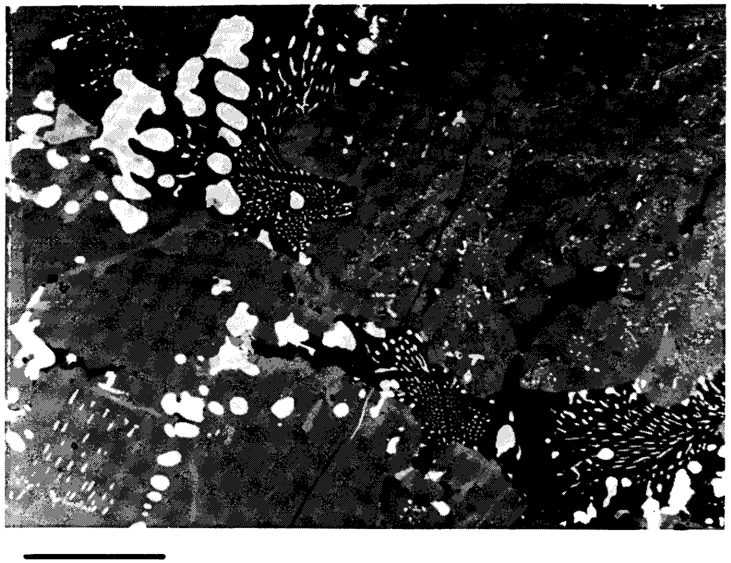

Figure 5. Reflected-light micrograph of a slag sample from Afunfun 175 furnace 1. The large white globular crystals are wustite. The medium grey areas that fill most of the picture are fayalite crystals. Some of these contain fine secondary wustite. The large dark patches and the smaller dark patches between fayalite crystals are leucite, which contains oriented rods of secondary wustite. The grey areas between fayalite crystals are patches of glass. The scale bar is 0·1 mm.

that of fayalite, but is easily distinguished by its high hardness, which causes the rhombs to develop positive relief against the other constituents during the final polishing of the sample (Figure 6). The EDAX spectrum and characteristic habit identify this as hercynite ($FeAl_2O_4$), which is often reported as a constituent of prehistoric slags produced from ores or fluxes containing alumina (e.g. Fells, 1983).

Leucite: Between the fayalite laths are rounded or angular patches of a constituent that has very low reflectivity, and often contains oriented inclusions of wustite (Figures 5 & 6). In thin-section, these patches have very low birefringence, best detected at high magnification with the full-wave accessory plate. The optical properties and chemical composition identify this phase as leucite ($KAlSi_2O_6$). Leucite is commonly seen in smelting slags, and the striking leucite/wustite intergrowths illustrated in Figure 5 have been reported in pre-Roman and Roman iron-smelting slags from Britain (Fells, 1983: 113–114).

The outer portions of all specimens have been altered by weathering and now consist of maghemite and other iron oxides, often concentrically banded (Figure 6). The inner portions are unaltered except where cracks lead from the exterior to the interior.

Interpretation: With one exception, the samples of the first group are fired soil. They strongly resemble those described above from furnaces 9, 8 and 6 in hand specimen and in chemical composition. The exception (Table 2 analysis 9) has a composition of 69% SiO_2, 28% FeO, 2·5% CaO and 0·3% copper. This is very similar to specimens described from furnaces 6 and 8. We are unable to study this specimen in thin-section but think that it is probably a fayalitic slag contaminated with undissolved silica.

The second type of material is a fayalitic iron-smelting slag. Similar slags were often produced in copper smelting, where the usual fluxes were iron oxides. We noted in the last paragraph that one of the samples of the first type of slag contains 0·3% copper. However, no metallic copper was seen in the six microsections, and the copper content of the two samples analysed is only 30–40 ppm. In our experience, slags from copper smelting always contain some entrapped copper prills, even though they may be very small. We think it highly unlikely that separation of the copper from the fayalite slag by gravity in a small furnace could lower the copper content to 40 ppm. We therefore feel confident that these slags are the residues of iron smelting.

A third sample of charcoal from the basal tunnels was submitted for radiocarbon dating in a different laboratory. The date obtained was 1730 ± 50 bc (Pta-3964). While this probably does not date the same event as that dated by MC-2379 and MC-2398, it would still make this one of the earliest known iron-smelting furnaces in the world. We were reluctant to accept this conclusion for two reasons. The first was that there is as yet no other evidence of the use of iron in this region until *c.* 500 bc. The second was that the extraordinary plan and section of the furnace raises doubts that the radiocarbon dates are associated with the smelting operation.

There is good evidence that the smelting operation took place in this furnace, as some of the slag was fused to the wall of the chamber. But the complex plan of the structure, and especially the charcoal-filled basal tunnels, suggest that this may have been the lower trunk and root structure of a tree. One of the authors (D.K.) has seen similar structures in the savannas of central Africa, where dead tree stumps ignited by grass fires may smoulder slowly underground for days or weeks. Shaw (1980) has noted the same phenomenon in Nigeria. If furnace 1 is in fact a carbonized tree stump, then radiocarbon samples taken from the basal tunnels would date the life time of the tree, not the smelting operation.

We therefore decided to try to date the iron slag directly. The small pieces of charcoal that had been enveloped by the slag during the smelting operation were insufficient for conventional radiocarbon dating but were more than adequate for accelerator mass spectrometry. A small piece of charcoal prised out of one of the pieces of iron slag was converted to carbon dioxide and then to iron carbide targets at the Oxford accelerator. The sample was counted in duplicate, and the corrected radiocarbon age obtained was 440 ± 100 ad (OxA-1115). When calibrated at two standard deviations by the method of Stuiver and Pearson (1986), this yields calendar age ranges of 268–275 AD or 340–680 AD. A span of more than two millennia therefore separates the formation of this structure from its use as an iron-smelting furnace.

"Cuivre 1": Discussion

We need to ask three major questions when attempting to interpret the evidence from each of the structures at Afunfun 175. In the first place, is the structure of artificial or natural construction? Secondly, is there any definite evidence that the structure was used in a metallurgical process? Thirdly, the radiocarbon dates from furnace 1 clearly show that fossil charcoal was available on the site. Given that humans could have made use of old charcoal, can we trust the radiocarbon dates?

Four of the 18 structures excavated at Afunfun 175 are definitely furnaces of some kind. Furnaces 3, 8 and 18 are shaft furnaces with an internal diameter of *c.* 30 cm and a rake hole in front. Furnace 6 is a bowl of regular hemispherical cross-section. The remaining 14 structures have a wide variety of irregular shapes, each of which is unique. They comprise single or multiple basins or pits, delineated by a hard fired shell. In some of these, the shell forms pockets or tube-like extensions around masses of charcoal. In most cases, it was from these pockets that the radiocarbon samples were obtained.

Some of these structures may be of human construction, but there is no positive evidence that any of these were used in the practice of metallurgy. We have suggested above that furnace 1 may be a burnt-out tree stump. Some of the other structures on this site may also be fired casts of stumps or fallen trunks of trees, as suggested by Bernus (1983: 168–169). We think that fallen trunks may have been partially buried by wind-blown soil and subsequently ignited by grass fires. The heat evolved by the burning wood would bake a thin jacket of the enclosing soil, producing a fired cast of the trunk. Slow smouldering combustion might also carbonize buried limbs or roots, producing the charcoal masses. This process has been well described by Shaw (1980), and the interested reader should examine the photographs provided in his account.

Thirteen of the 19 analysed samples of fused materials excavated from the structures at Afunfun 175 form a distinct cluster. In all of these samples, the sum of the SiO_2 and the Al_2O_3 is 80–90% of the total by weight (SiO_2 59–69%, Al_2O_3 17–25%). Iron oxides are 5–15%, and no other single element is as much as 4% of the total in any of the samples. Twelve of the 13 samples have copper contents of 100 ppm or less; the single exception (Table 3, analysis 1) has only 0·2%. Three of these samples have been studied in thin-section and consist of small angular fragments of quartz, feldspar and opaque minerals in glass. Their chemical compositions are so similar to those of other members of the cluster that we can infer that the mineral content of the latter is similar.

Members of this cluster are partially vitrified soil. Although similar materials were often produced in ancient metallurgical processes by partial melting of furnace wall or crucibles, they can be produced in many other ways. They may even be formed naturally if sufficient heat can be supplied to initiate the reaction between the glass-forming constituents. It follows that the presence of this type of fused product is not in itself evidence for the practice of metallurgy. The copper content of 12 of the 13 samples is less than 100 ppm. These levels cannot be taken as evidence for copper working and almost certainly reflect the copper content of the soil at this locality. The single remaining sample contains 0·2% copper metal (2000 ppm) and is from furnace 1, where there is definite evidence of a metallurgical process.

The remaining six samples provide more positive evidence for some form of metallurgical process. Three of these came from furnace 1. Two are fayalite/wustite/hercynite/leucite iron-smelting slags with only 30–40 ppm copper. The third sample from furnace 1 is similar to two samples from furnace 6 and one from furnace 8 (Table 2, analyses 3, 5, 6 and 9). Each of these four samples contains 60–70% SiO_2 and 27–33% FeO. They contain very little Al_2O_3 (0–3%). The copper content of these samples (0·3%–3·5%) is high enough to constitute plausible evidence for copper working. Unfortunately, no thin-sections were made from these samples before they were used for chemical analyses, so we cannot be sure what they represent. It seems likely that they are fayalitic copper-smelting slags, similar to those recorded in most regions of the Old World (Bachmann, 1980), physically mixed with undissolved silica from the charge. Samples of these compositions could not have been fully molten in a small shaft furnace, as the liquidus temperatures of the non-metallic portion of these four samples is well above 1700°C (Osborn & Muan, 1960: figure 7).

This brings us inevitably to the problem of dating these sites. The only structures at Afunfun 175 that can be considered as evidence for early metallurgy are those that (1) contain residues definitely associated with a metallurgical process, and (2) are dated by radiocarbon or some other technique. Only two of the 18 excavated structures at Afunfun 175 (furnaces 1 and 8) meet both these requirements.

Among those excluded is Afunfun 175 furnace 6, which produced a prill of leaded bronze but is undated. Although this is situated within a cluster of structures with radiocarbon dates in the second millennium bc, it cannot be assumed to be contemporary with them. The composition of the alloy is similar to those recovered from the site of Marandet, which is dated by radiocarbon to the first millennium ad (Grébénart, 1985: 354–379). Furnace 3 is a small shaft furnace with a radiocarbon date of 1730 ± 50 bc (Grébénart, 1985: 121–122). The single sample analysed (Table 3, analysis 3) is partially vitrified earth and contains only 10 ppm copper. There is therefore no conclusive proof that this furnace was employed in a metallurgical process. The same applies to furnace 9.

Furnace 1 has been eliminated from this group by a direct radiocarbon date of 440 ± 100 ad on the iron-smelting slag it contained. This leaves only furnace 8, which has a single radiocarbon date of 1710 ± 110 bc obtained on charcoal from the furnace fill. Three samples of fused materials from the fill have been analysed. Two of these (Table 2, analysis 2 and Table 3, analysis 5) are vitrified earth of furnace lining, containing 26 ppm and 10 ppm copper, respectively. The third sample (Table 2, analysis 3) contains 63·5% SiO_2, 31·2% FeO, 4·2% CaO and 1·0% copper metal. This constitutes definite evidence of copper working, but without an examination of the microstructure we cannot infer anything about the technology employed.

A number of the irregular structures at Afunfun 175, including furnace 1, contain masses of charcoal that are fired into the lining. There is therefore no reason to doubt that radiocarbon samples from these masses date the time of formation of the structure. This is not, however, true of furnaces 3 and 8; the radiocarbon samples for these were excavated from the furnace fill. There is therefore no proof that the age of these charcoal samples is the age of the structure. The artisans who built these structures may have made use of the fossil charcoal that was available in the surrounding "Cuivre 1" structures. The radiocarbon date of 1710 ± 110 for furnace 8 cannot therefore be taken at face value. It must be checked by some other technique, such as thermoluminescence dating of the furnace lining.

"Cuivre 2": Sample Descriptions

The materials grouped together as "Cuivre 2" are much less variable than those from "Cuivre 1". In all cases, the sites were the remains of small non-tapping shaft furnaces in the shape of a truncated cone. The maximum internal diameter varied from 0·5–1·0 m. The shafts were apparently broken down to extract the contents and probably rose no more than 1 m above ground level. These smelting sites were scattered throughout the region and were often surrounded by light scatters of slag and tuyere fragments. Grébénart has excavated 20 of these furnaces, 16 of which produced charcoal for radiocarbon dating. The uncalibrated dates (Table 1) range from 850 ± 90 bc to 670 ± 70 ad, although 15 of 16 lie between 850 ± 90 bc and 90 ± 90 ad (Grébénart, 1985: 175–259).

Eight chemical analyses of slags from these furnaces were made by Bourhis, Tylecote and de Paepe, and are reprinted in Table 4. With one exception (Table 4, analysis 6), they are strikingly similar. Their composition is also most unusual for prehistoric copper-smelting slags, which are generally rich in iron oxide (Bachmann, 1980). With the one exception noted above, none of the "Cuivre 2" slags contain more than 2% FeO. Apart from their copper content, they are strikingly similar to historic iron-smelting blast-furnace slags, which were fluxed with limestone or dolomite. As no microstructural or mineralogical data were available for these eight samples, we studied thin-sections and obtained chemical analyses for samples from three additional furnaces.

Ikawaten 193, furnace 3

This was the largest of four similar furnace bases from this site. It was a non-tapping shift furnace, since broken down to the former ground level. The sides of the base have a pronounced taper, the internal diameter being 0·55 m at floor level and 0·42 m at a height of 20 cm (Grébénart, 1985: 215–6). Wood charcoal from the fill was dated to 210 ± 90 bc (GIF-5186).

Table 4. Analyses of fused products from "Cuivre 2" structures

	1	2	3	4	5	6	7	8	9	10	11
Site	Tuluk 211	Ikawaten 193	Ikawaten 193	Ikawaten 193	Ikawaten 193	Ikawaten 193	Afunfun 162	Afunfun 162	Afunfun 162	Afunfun 162	Azelik 210
Furnace	1	2	3	SP1	SP4	GRB	28	4	10	24	Surface
SiO_2	47·44	42·91	43·87	42·5	39·5	49·5	44·94	45·40	44·40	49·0	41·6
TiO_2	0·54	0·63	0·32	n.d.	n.d.	—	0·57	0·70	0·53	0·3	—
Al_2O_3	9·82	9·02	7·09	5·5	6.4	—	8·75	7·52	7·10	7·7	15·5
Fe_2O_3	1·39	1·47	1·23	—	—	—	2·40	—	—	—	1·0
FeO	—	—	—	0·2	0·4	18·3	—	1·70	1·50	1·2	—
MnO	0·39	0·20	0·35	n.d.	n.d.	—	0·43	n.d.	0·38	0·3	—
MgO	10·51	12·49	11·34	10·6	9·8	10.5	9·19	10·20	10·80	8·9	10·0
CaO	22·91	24·59	27·96	37·3	37·8	21.0	24·86	27·90	29·60	22·7	20·0
Na_2O	3·10	3·60	2·29	2·4	2·4	—	2·37	2·26	2·99	1·1	—
K_2O	0·85	0·70	0·77	n.d.	0·1	—	0·98	0·87	1·10	1·2	5·0
P_2O_5	0·97	—	0·66	—	—	—	1·25	—	—	—	—
Cu metal	0·61	0·64	0·49	n.d.	n.d.	0.7	0·94	0·95	0·34	5·7	5·2
SrO	—	0·28	—	—	—	—	—	—	—	—	—
H_2O-	0·14	—	0·15	—	—	—	0·32	—	—	—	—
LOI	0·75	—	0·77	—	—	—	1·57	—	—	—	—
Total	99·42	96·53	97·29	98·5	96·4	100·0	98·57	97·80	98·70	98·4	97·7
Analysis	van der Merwe	de Paepe	van der Merwe	Tylecote	Tylecote	Bourhis	van der Merwe	Tylecote	Tylecote	Tylecote	Bourhis
Method	XRF	?	XRF	EPMA	EPMA	?	XRF	EPMA	EPMA	EPMA	?

Key: —, Not analysed; n.d., below limit of detection; XRF, major elements by X-ray fluorescence, H_2O- and loss on ignition (LOI) by gravimetry; EPMA, electron microprobe. Analyses by de Paepe and van der Merwe not previously published; others from Tylecote (1982) and Grébénart (1985).

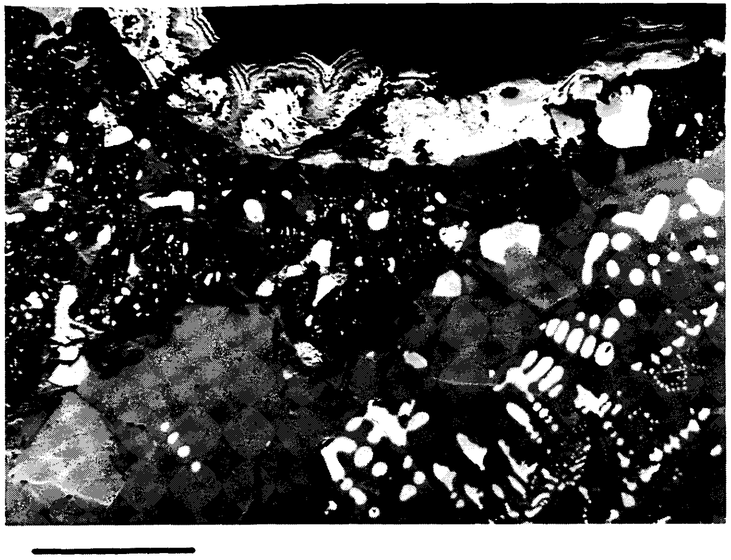

Figure 6. Reflected-light micrograph of a slag sample from Afunfun 175, furnace 1. This picture shows an area bounding a cavity (black area) originally formed by entrapped gas. The margins of the cavity have been altered by weathering and contain maghemite (white) and banded microcrystalline iron oxides. In the unweathered portions, the continuous light grey area is fayalite. The light grey polygonal crystals with positive relief against the fayalite are hercynite. The rounded white dendrites are wustite. The cavity is surrounded by glass (medium grey) with numerous rounded cystals of leucite (dark), most of which contain fine secondary wustite. The scale bar is 0·1 mm.

The hand specimen is a well-rounded piece with smooth surfaces. The surface colour is chalk white, but on a cut face this can be seen to be a weathered rind. The colour of the unweathered interior on a polished surface is dull red (Munsell 7·5 R 4/2). The bulk chemical composition is given in Table 4, analysis 3. This analysis is quite unlike any analysis from "Cuivre 1". The most striking features are the very high levels of CaO (28%) and MgO (11%). The balance is made up of SiO_2 (44%), Al_2O_3 (7%), sundry oxides and 0·5% copper metal. Note also that the FeO content is only 1%.

A thin-section of this sample contains only one silicate mineral, accompanied by a little interstitial glass, in which are dispersed small spheres of copper (diameter 1·5–12·5 μ; Figure 7). The silicate mineral is mostly in the form of rectangular phenocrysts. They are colourless, have high positive relief against the mounting epoxy, show parallel extinction, have low birefringence, are length-fast and give a uniaxial positive interference figure. The optical characteristics and composition identify the mineral as a melilite towards the end member äkermanite ($Ca_2MgSi_2O_7$). Melilites show extensive solid solution over a wide range of composition (Nurse & Midgley, 1953). The bulk composition of this sample is too complex to be fitted satisfactorily to any of the binary phase diagrams within the system, but is closest to the binary system äkermanite–sodium melilite ($Ca_2MgSi_2O_7$–$NaCaAlSi_2O_7$; Table 5). The phenocrysts show no visible evidence of compositional zoning, although the comparison of EDAX spot analyses from the core and margins of several crystals shows slight enrichment in iron and alkalis at the margins. Most of the alkali is in the interstitial glass. Above 400 × most phenocrysts show the characteristic "peg structure" of melilites, evident as small rods of an exsolved second phase parallel to the *a* or *b* crystallographic axis (Figure 7). The glass is clear to light green in colour and

Table 5. Composition of some melilite end-members (weight %)

		CaO	MgO	Na_2O	FeO	Al_2O_3	SiO_2
Äkermanite	$Ca_2MgSi_2O_7$	37·8	13·6				40·5
Sodium melilite	$NaCaAlSi_2O_7$	21·7		12·0		19·7	46·5
Ferroäkermanite	$Ca_2FeSi_2O_7$	36·9			23·6		39·5
Gehlenite	$Ca_2Al_2SiO_7$	40·9				37·2	21·9

completely isotropic. Alteration of the sample is apparent only in the thin white rind around the exterior. Several copper droplets were examined by EDAX. No elements other than copper were detected.

Tuluk 211, furnace 1

The sample examined was excavated from within the collapsed remnants of a non-tapping low shaft furnace with a basal diameter between 0·6 and 0·8 m (Grébénart, 1985: 234–5). A sample of wood charcoal in good association with the slags was dated to 410 ± 70 bc (GIF-5545).

The hand specimen has a maximum length of 10 cm and is smooth and bulbous with a glossy red surface (Munsell 7·5 R 3/6; a polished interior surface is Munsell 7·5 R 4/6 with lighter patches, Munsell 2·5 YR 4/4). A recognizable charcoal impression is preserved in one part of the surface, although the edges are rounded and the mould preserves none of the crisp surface detail of charcoal impressions in iron-smelting slags. The bulk composition is given in Table 4, column 1, and is similar to that for Ikawaten 193, except that the cation/anion ratio is lower.

The only silicate mineral present is melilite near the composition of äkermanite. There are a few rectangular phenocrysts, but most of the melilite forms delicate fan-shaped dendrites without visible zoning. The glass is light green and isotropic, and is packed with very small spheres of copper (diameter 1·0–19·5 μ). In a few instances, there is a second constituent associated with the copper that is a deep grey colour and is probably lead. Alteration of the sample is evident around the rim of the specimen and as a few brown patches in the interior, within which the dendritic crystal habit has been destroyed. The alteration product of the melilite has inclined extinction and interference colours in the high second order, but was not positively identified.

Afunfun 162, furnace 28

This furnace base is of the same tapered shape as Ikawaten 193, as indeed are the majority of furnaces in "Cuivre 2". The internal diameter at floor level is 0·85 m, and the diameter at 30 cm height (the former soil surface) is 0·80 m (Grébénart, 1985: 195–200). Charcoal from the furnace fill was dated to 590 ± 90 bc (MC-2045).

The hand specimen has a smooth chalk-white surface with several rectilinear pits, which are clearly the impressions of charcoal pieces. The charcoal pieces were 1·0–1·5 cm in maximum length. The intersections of the planes are, however, slightly rounded, and none of the surface detail of charcoal impressions in fayalite/wustite iron slags can be seen here. This implies that this slag had higher viscosity than typical iron slags. On a sawn surface, the white surface is only a rind a few mm thick. The interior is dark red (Munsell 10 R 6/4 on a wet sawn surface, Munsell 10 R 2/2 on a polished surface). The bulk chemical composition of this sample is given in Table 4, analysis 7, and is very similar to those of other analysed "Cuivre 2" samples.

Figure 7. Plane-polarized transmitted-light micrograph of a slag sample from Ikawaten 193, furnace 1, at high magnification. An area of glass (in the centre of the frame) is bounded by four rectangular crystals of melilite, near the composition of äkermanite. The glass contains abundant tiny spheres of metallic copper. Copper spheres are also trapped within the melilite crystals, although at a lower density. The characteristic "peg structure" of melilites, caused by the exsolution of a second phase, is apparent as small tadpole-shaped bodies oriented at right angles to the long axis of the crystals. The scale bar is 0·1 mm.

The thin-section reveals the same three constituents as the two previous samples, namely melilite near the composition of äkermanite, glass and small copper spherules (diameter 1–10 μ). The melilite is entirely in dendritic form (Figure 8). Beneath the thin weathered rind, the specimen is quite unaltered. The glass is pale green in thin-section and is completely isotropic. Near one edge, the specimen contains a few small fragments of relict quartz surrounded by reaction rims.

"Cuivre 2": Interpretation and Discussion

The three samples described are very similar. Apart from a little relict quartz in the sample from furnace 28, all of the phases in each sample have formed the cooling of a fully molten mass. It is clear that these were copper-working slags and that they were in direct contact with the charcoal fuel. The chemical analyses of these three samples are very similar to those of seven of the eight samples analysed previously, and we presume that they were also composed of melilite, glass and copper. The exception (Table 4, analysis 6) contains little Al_2O_3 and has 18% FeO. We do not know the mineralogy or microstructure of this sample and cannot be sure what it represents.

The chemical composition of the remaining 10 "Cuivre 2" slags have not, to our knowledge, been previously recorded from prehistoric copper-smelting sites. The high calcium and magnesium content (and the low content of iron) are strikingly similar to the composition of slags from historic iron-smelting blast furnaces. Blast-furnace slags are known to have substantially higher viscosity at given temperature than the usual fayalitic slags of ancient copper and iron smelting. We must therefore ask whether drops of molten copper could have separated from slags of these compositions within reasonable limits of time and temperature for a small shaft furnace. Tylecote (1982) suggested that the slags

Table 6. Experimentally determined viscosities of slag melts

	1	2	3	4	5	6
Composition (weight %)						
SiO_2	50·0	45·0	45·0	48·0	32·2	29·4
Al_2O_3	10·0	5·0	10·0	10·0	0·0	0·0
CaO	30·0	40·0	30·0	25·0	0·0	0·0
MgO	10·0	10·0	10·0	12·0	0·0	0·0
alkalis	0·0	0·0	0·0	5·0	0·0	0·0
Fe_2O_3	0·0	0·0	0·0	0·0	0·0	0·0
FeO	0·0	0·0	0·0	0·0	64·3	70·6
			Viscosity (poise)			
Temperature (°C)						
1500	7·09	2·99	4·07	—	—	—
1450	9·92	4·19	5·92	—	—	—
1400	15·00	6·56	8·79	—	—	—
1350	23·80	10·30	13·40	—	0·82	0·50
1325	—	—	—	31·20	—	—
1300	39·10	—	23·00	—	0·95	0·55
1250	75·00	—	45·70	—	1·10	0·75
1240	—	—	—	96·20	—	—
1200	—	—	—	—	3·20	4·80
Liquidus temperature	—	—	—	1240	1212	—
Source	Machin *et al.* (1952)	Machin *et al.* (1952)	Machin *et al.* (1952)	Lakatos *et al.* (1981)	Williams *et al.* (1983)	Williams *et al.* (1983)

The slag compositions of columns 3 and 4 correspond most closely to those of the analysed "Cuivre 2" slags (Table 4). Columns 1 and 2 show the effect upon viscosity of variation in the alumina and silica content. Columns 5 and 6 have the compositions of typical prehistoric fayalite slags and demonstrate the marked effect of FeO on viscosity.

would originally have had much higher alkali content (since leached away by weathering) and would therefore have been less viscous than their present composition would imply. This is a problem that must be assessed on a sample-by-sample basis. Tylecote does not report the degree of weathering in the samples he examined, but the samples we have studied are not substantially altered. Weathering is largely confined to the thin, chalk-white rind on the outer surface. The glass in the interior (where most of the alkalis reside) is generally clear and isotropic. We therefore think that the present composition of these samples can be used to infer approximate liquidus temperatures and viscosities. However, in future we would remove the outer rind before obtaining bulk chemical analyses.

Viscosity–temperature relationships in the system $CaO–MgO–Al_2O_3–SiO_2$ have been studied by Machin *et al.* (1952) and by Turkdogan & Bills (1960). The liquidus temperatures and viscosities of some compositions in the system $CaO–MgO–Al_2O_3–SiO_2$–alkalis–Fe_2O_3–FeO were determined by Lakatos *et al.* (1981), and those for a few compositions in the system $FeO–Fe_2O_3–SiO_2$ by Williams *et al.* (1983). Table 6 shows viscosity–temperature relationships for those compositions that are closest to the bulk analyses of "Cuivre 2" slags from Niger.

For comparison, the data for two FeO/SiO_2 melts close to the composition of a fayalite slag are also given. Note that molten fayalite slags have viscosities one to two orders of magnitude lower than molten melilite slags at the same temperature. It is also apparent that limited substitution of alkalis (sodium and potassium) for the alkali earths (calcium and magnesium) does not change slag viscosity appreciably in this particular region of the system $CaO–MgO–Al_2O_3–SiO_2$–alkalis–Fe_2O_3–FeO. The substitution of FeO for alkalis or alkali earths produces a marked decrease in viscosity (Lakatos *et al.*, 1981: figure 1). With one exception, the FeO content of analysed "Cuivre 2" slags is less than 2%, and these slags must therefore be more viscous than the usual fayalitic slags from prehistoric smelting sites.

Table 7. Time of fall of copper spheres through a 10 cm slag bed

	Viscosity (poise)					
Particle diameter (mm)	1·0	3·0	6·0	12·0	30·0	100·0
5·00	0·1 s	0·4 s	0·7 s	1·5 s	3·6 s	11·5 s
1·00	3·0 s	9·1 s	18·5 s	37·0 s	1·5 min	5·1 min
0·10	5·1 min	15·3 min	30·6 min	1·03 h	2·55 h	8·50 h
0·01	8·50 h	25·48 h	51·00 h	101·94 h	254·84 h	850·00 h

The rate of descent of a spherical particle (a copper drop, for example) under gravity in a viscous medium is given by Stokes Law,

$$\mu = \frac{1}{18}\,\frac{l_s - l_L g D^2}{\eta},$$

where μ is fall speed ($m \times s^{-1}$), l_s is the density of the falling sphere ($kg \times m^{-3}$), l_L is the density of the viscous medium ($kg \times m^{-3}$), g is the gravitational acceleration ($9{\cdot}8\ m \times s^2$), D is the diameter of the falling sphere (m) and η is the viscosity of the medium ($kg \times m^{-1} \times s^{-1}$).

We have calculated the time taken for copper particles of various diameters to fall through a slag bed of arbitrary thickness at various values of slag viscosity. The values given in Table 7 are for a slag depth of 10 cm. They suggest that copper particles with a diameter of 1 mm or more will separate successfully from even the most viscous of the slags in Table 6 in a few minutes. The fall speed varies with the square of the diameter, however, and small particles descend much more slowly. The maximum diameter of the copper droplets in the three "Cuivre 2" slags described above is 0·01–0·02 mm (10–20 μ). This is in good agreement with the calculated values in Table 7. The times taken for drops of 0·1 mm diameter to pass through a 10 cm slag bed are all reasonable values for the duration of a smelting operation (except perhaps for slags of 30–100 poise), whereas the times for drops of 0·01 mm are clearly excessive (Table 7). From the experimental results quoted in Table 6, it appears likely that the operators of the "Cuivre 2" furnaces were working with slag viscosities of between 6 and 15 poise.

We conclude from this that most of the copper charged to the furnace would have been recovered from these slags by the operators. We do not know what the initial size of the copper drops would have been, nor can we be certain that collisions between descending drops do not result in the formation of larger drops with faster fall rates. Consequently, we cannot infer the operating temperatures of these furnaces from the size of the copper droplets suspended in the slags. If the operators were working with slags of less than 15 poise, as we suggest, we can infer a furnace temperature above 1350°C (Table 6). Slags with viscosity of 6 poise are free-flowing (J. E. Rehder, pers. comm.). The surface morphology of the "Cuivre 2" slags we have examined does not suggest free flow, and the viscosity must therefore have been greater than 6 poise. From the data in Table 6, this suggests an approximate upper limit of 1450°C for the temperature of the furnace.

Tylecote (1982: 61) assumed that copper would not have separated from these slags in a furnace because of their relatively high viscosity. He therefore concluded that the native copper must have been processed in crucibles, and that the slag formed by reaction between the crucible fabric and residual dolomite gangue in the charge. Our calculations show that copper would in fact have separated from these slags in the shaft furnaces of "Cuivre 2". It should also be noted that no crucible fragments have yet been recovered from any site of "Cuivre 1" or "Cuivre 2", although they are abundant on sites of the first and second millennia ad, which fall outside the scope of this discussion (Bernus, 1983; Grebenart, 1985: 349–378). We conclude that the copper was in fact processed in the short shaft furnaces, and that successful separation of metal and slag in the furnace was achieved.

Bourhis (in Grébénart, 1985: 24) argued that "Cuivre 2" represents the beginning of true smelting in the Agadez region. He concluded that copper oxide minerals rather than native copper were smelted. Tylecote, on the other hand, concluded that these slags were a byproduct of the refining of native copper. To evaluate these arguments, we need to examine the geological context of the native copper and copper minerals in the region of Agadez.

Copper Resources of the Agadez Region

The geological strata exposed in the region south and west of Agadez are fluviatile and deltaic sediments of Permian and Jurassic age. They comprise sandstones, arkoses, conglomerates, shales, marls and lenses of limestone and dolomite. Uranium, vanadium and copper minerals have been deposited from solution in some of these strata, forming low-grade deposits of the Colorado Plateau type (Imreh & Nicolini, 1962). The ore mineral assemblage and host rock are variable. In most studied localities, the dominant copper mineral is chrysocolla ($CuSiO_32H_2O$), which is associated with cuprite, native copper, malachite, tenorite, copper vanadates and uranium minerals (Imreh & Nicolini, 1962: 70–77).

We examined a small specimen containing native copper. The hand specimen is well rounded and varies in colour from cream to dark pink. The granules of copper are small (2 mm or less) and form dark protrusions on the surface. In polished section, most of the copper particles have a central core of metallic copper and cuprite (Cu_2O), surrounded by a rim of cuprite (Figure 9). Some of the individual copper grains within the core contain growth twins (cf. Vernon, 1986: 159–160). Other nodules consist only of cuprite. No malachite or chrysocolla was seen in this section. The only impurity detected in the metallic copper by EDAX was arsenic, which was barely above the detection limit of the instrument (*c.* 0·2%). Some of the native copper from the Agadez region is evidently more arsenical; two hand-picked concentrates prepared by Bernus (1983: 167) contained 0·80% As (63·7% Cu) and 0·40% As (50·6% Cu). The copper ingots obtained by melting the concentrates in a crucible contained 1·5% As and 0·5% As, respectively.

In thin-section, most of the specimen is composed of very small carbonate crystals. Area scans with the electron microprobe suggest that this is mostly calcite with some dolomite. The finely divided carbonates have been replaced along numerous small veins by the pyroxene enstatite ($MgSiO_3$), calcite ($CaCO_3$) and octagonal crystals of analcime ($NaAlSi_2O_6H_2O$), which display complex twinning and have very low birefringence. Some portions of the vein filling show very strong EDAX peaks for barium. As no other minerals were noted on re-examination of the filling, the barium is probably present as the orthorhombic carbonate witherite ($BaCO_3$), which is difficult to distinguish from calcite in thin-section.

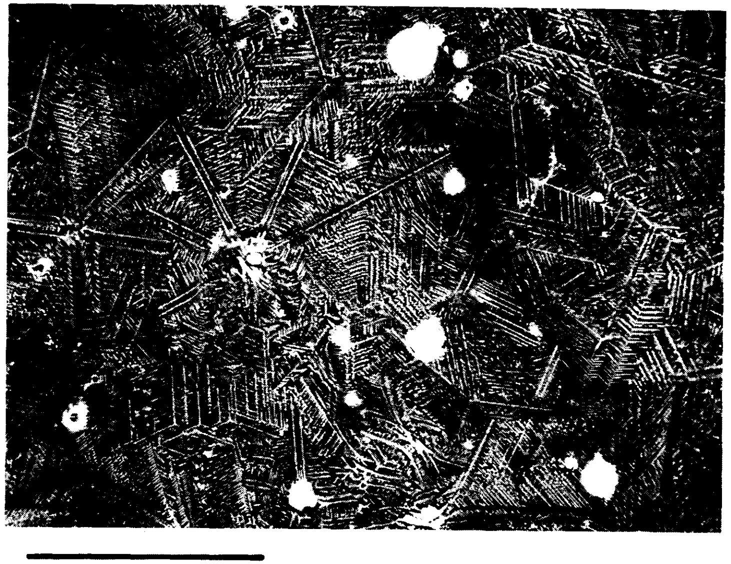

Figure 8. Plane-polarized, transmitted-light micrograph of a slag sample from Afunfun 162, furnace 28. The sample is almost entirely composed of interlocking dendrites of melilite near the composition of äkermanite. The interstices are filled with glass containing very small spheres of copper. Those regions that appear darkest in this frame contain higher concentrations of copper spheres. The white circular regions are gas cavities. The scale bar is 0·5 mm.

Interpretation: Tylecote (1982) concluded that only native copper was processed in "Cuivre 2", while Bourhis (in Grébénart 1985) argued that sites of this period represent the smelting of oxide minerals. The difference between these positions is more apparent than real. Copper oxide minerals occur in intimate association with native copper in the host rocks (Figure 9; Imreh & Nicolini, 1962). Both types of material would probably have been present in a prepared charge for the furnace. Copper oxide is very readily reduced by carbon monoxide, so the metal recovered from the furnace would be a mixture of melted native copper and smelted copper.

The high magnesium and calcium content of the slags must derive from the presence of dolomite in the furnace charge. The ratio of calcium to magnesium varies from about 2:1 to about 4:1 in these samples. This presumably reflects varying proportions of calcite and dolomite in the host rocks, although some calcium (together with sodium, potassium and phosphorus) may have been introduced in the ash of the charcoal fuel. The phosphorus level is relatively high (0·66, 0·97 and 1·25%) in the three slag samples in which it was determined. It is not clear whether this derives from the rock, the charcoal or both. The silica and alumina in the slags probably derive in part from the ore. If the slags had formed only by the reaction of a dolomitic charge with the furnace wall, we might expect to see the same silica/alumina ratio in these samples as in those "Cuivre 1" samples that are interpreted as vitrified earth. This lies between 2·4 and 3·7 ($n = 13$). The silica/alumina ratios of the iron-poor "Cuivre 2" slags lie between 2·7 and 7·7 ($n = 10$; nine of 10 samples between 4·8 and 7·7). This suggests that these oxides derive at least in part from the furnace charge.

Native copper and copper minerals in this region occur in arkose, shale, marl and carbonate lenses (Imreh & Nicolini, 1962). The ratio of calcium and magnesium to silica and alumina in the host rock is therefore variable, and this presumably explains the variation in the relative proportions of these elements in the analysed slags of Table 4. The grade of copper mineralization available in the Agadez region today is only about 2%, and the minerals are widely dispersed in the host rocks (Imreh & Nicolini, 1962; Bernus & Gouletquer, 1976: 16). Although the grades available to the earliest metal workers were probably higher than this, it is likely that laborious hand beneficiation of the native copper and copper oxides would have been essential in preparing a furnace charge. We think that the slags of "Cuivre 2" derive from the mineral gangue remaining in the charge after hand concentration, and that relatively little slag would have been produced in each run of the furnace. There is also a good technical reason to remove as much carbonate-bearing gangue as possible from the furnace charge. The decomposition of magnesium and calcium carbonates to their oxides are strongly endothermic reactions. The heat required to decompose the carbonates in the charge would initially drive the furnace temperature down and would therefore increase the fuel consumption of the process very considerably (J. E. Rehder, pers. comm.).

Conclusions

The great majority of the radiocarbon dates for "Cuivre 1" derive from samples taken from within a series of baked clay structures at the site of Afunfun 175. Many of the structures have very irregular shapes. There is no positive evidence that these structures were used for any type of metallurgical operation. The small amounts of slag-like material that they contained are partially vitrified soil. Some of these may be of human construction, although we have not been able to determine what functions they may have served. Others may be baked linings formed when partially buried dead tree trunks or stumps were ignited by grass fires.

The most remarkable structure at Afunfun 175 is furnace 1. We think that this hollow structure may have formed by the burning of a tree stump below ground level. At the base of the structure are several tapering tunnels filled with charcoal, which we interpret as roots carbonized *in situ*. Three radiocarbon samples taken from these tunnels date to the late second and early third millennia bc. The central cavity was later employed as an iron-smelting furnace; charcoal enclosed within the iron slag gave an AMS radiocarbon date of 440 ± 100 ad. This example shows very clearly that fossil charcoal was available at this site and could therefore have been used at any later time.

Four of the excavated structures at Afunfun 175 (furnaces 3, 6, 8 and 18) are undoubtedly of human construction (Grébénart, 1983: figures 83–87). Charcoal from furnace 3 gave a radiocarbon date of 1730 ± 50 bc (GIF-5172), but there is no definite evidence that it was used for metallurgy. Furnace 18 contained no slags or other residues, and is undated. Furnace 6 produced a prill of leaded bronze, but no charcoal for radiocarbon dating. This leaves only Afunfun 175 furnace 8 as evidence for metallurgy in Niger before 1000 bc.

A sample of the residue from furnace 8 has been analysed and contains 63·5% SiO_2, 31·2% FeO, 4·2% CaO and 1% copper metal. We consider this to be plausible evidence for copper smelting. A radiocarbon date of 1710 ± 110 bc (GIF-5176) was obtained on charcoal in the fill. When calibrated at two standard deviations, this date falls in the calendar range 2450–1750 BC (Pearson & Stuiver, 1986).

We think that a pervasive "old wood" problem may exist at Afunfun 175 and suggest that this radiocarbon date be viewed with great caution until it can be corroborated by another method, such as thermoluminescence dating of the fired lining of the furnace. More field work will be required to collect appropriate samples. Until these are available, the evidence for metallurgy in Niger prior to 1000 bc must remain in doubt.

The "old wood" problem appears to be common in arid areas. For example, Schiffer (1986) has reported radiocarbon dates as old as 1500 bp on wood samples from the present ground surface in the Sonora region of southern Arizona. Archaeologists working in arid regions clearly should be aware of this problem when submitting samples of wood charcoal for dating, and they should seek where possible to check radiocarbon dates by an independent method. In the case of iron-smelting furnaces, thermoluminescence dating of ceramic tuyere tips or of well-fired furnace wall would be appropriate. We do not see thermoluminescence as a substitute for radiocarbon, as the precision of thermoluminescence dates is relatively poor. The precision should however be adequate to detect major errors of association caused by the use of old wood or charcoal.

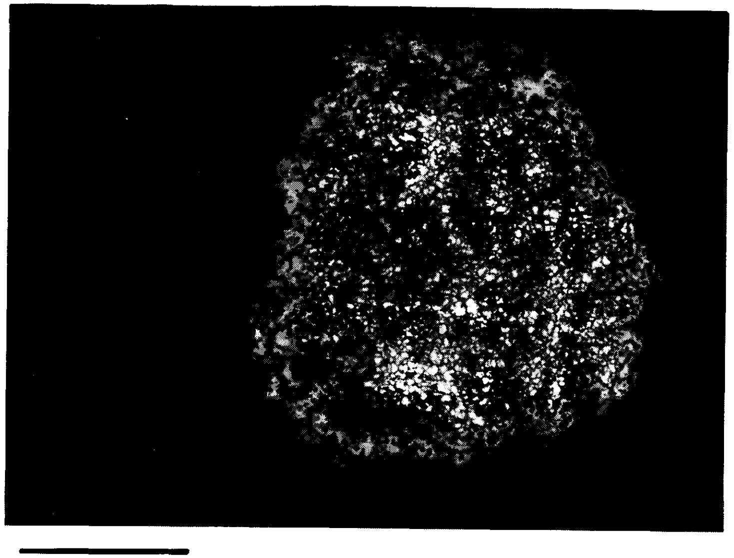

Figure 9. Reflected-light micrograph of a sample of copper minerals in a carbonate matrix from the Agadez region, etched with ammonia and hydrogen peroxide. The core of this small nodule consists of both fine-grained native copper and cuprite (Cu_2O). The dark areas are voids. The nodule is enclosed by a continuous rim of cuprite. The matrix around the nodule is composed of fine-grained calcite and dolomite. The margin of a vein is visible in a corner of the picture; the medium grey mineral on the edge of the vein is recrystallized calcite. The scale bar is 0·5 mm.

The sites assigned to "Cuivre 2", all of which have radiocarbon dates more recent than 1000 bc, were used for the processing of native copper, copper oxides and carbonates, which are found in a variety of sedimentary host rocks in this region. Calcite, dolomite and aluminosilicate gangue minerals have reacted to form distinctive red melilite slags. Although these are more viscous than the usual fayalitic copper-smelting slags, most of the copper appears to have been recovered from them by gravitational separation. From the limited experimental data available, we infer furnace-operating temperatures of 1350–1450°C. As the native copper and copper minerals are dispersed at low grade in the host rocks, laborious beneficiation would have been needed to produce a concentrate for the furnace. The scale of copper production in the Agadez region during the first millennium bc must have been very small, as the total volume of slag on all known "Cuivre 2" sites is only 200 cubic metres.

Four radiocarbon dates for "Fer 1" are listed in Table 1. All are from pits containing iron artifacts or iron slag (Grébénart, 1985: 263–348). A graph of the calibrated ranges of the radiocarbon dates for "Cuivre 2" and "Fer 1" is given in Figure 10. This also includes a radiocarbon date of 678 ± 120 (Dak-145) obtained on charcoal from an iron-smelting furnace in the Termit massif, southeast of Agades (Quéchon & Roset, 1974). If all of these

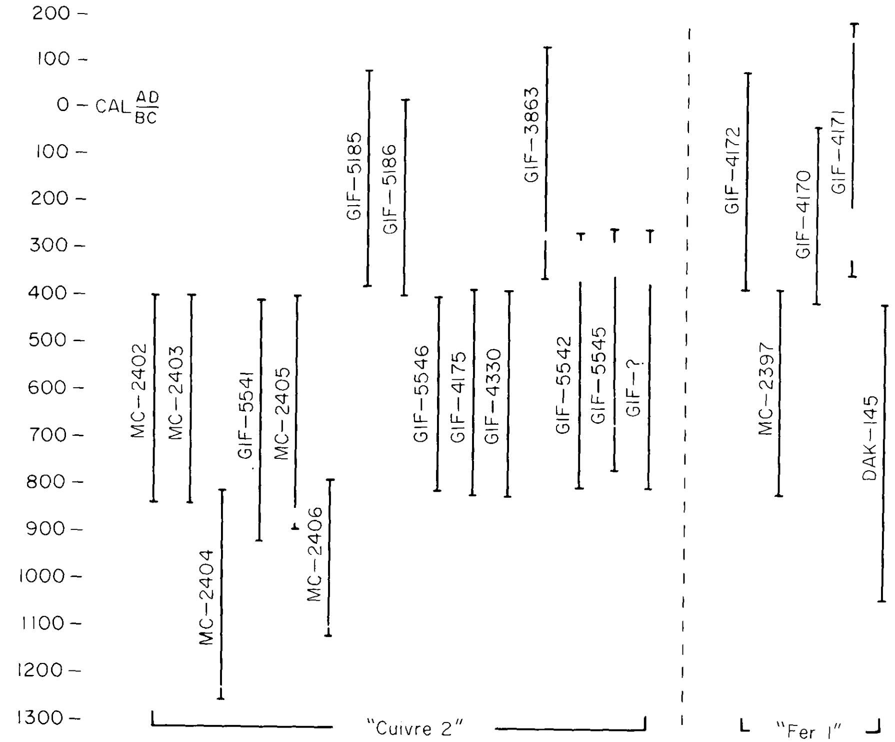

Figure 10. Calibrated ranges (two standard deviations) of radiocarbon dates for "Cuivre 1" and "Fer 1". The calibrations used are those of Pearson & Stuiver (1986) and Stuiver & Pearson (1986). Dak-145 is from Quéchon & Roset (1974). Details for the other dates are in Table 1 of this paper.

dates are taken at face value, these two technologies may be of similar antiquity in this region. Some of these dates may, however, reflect the use of old wood or charcoal.

The problems encountered in the course of this work suggest that some modificiations should be made in field methods. The collection of thermoluminescence samples should be standard practice in excavations of smelting furnaces in arid regions. Paired radiocarbon and thermoluminescence dates will provide more reliable estimates of age for smelting furnaces than will either technique used in isolation. The contents of an excavated furnace are usually very variable and may include partially reduced ore, vitrified ceramics, slags and metal. Archaeologists should take particular care to bring back samples that cover the full range of variation, as expressed in density, colour, surface texture and magnetic properties. As far as the laboratory analysis is concerned, both the microstructure and the chemical composition of metallurgical residues should be studied. It is often impossible to reconstruct the technology from chemical data alone.

This article has been confined to the evidence for metallurgy in the Agadez region before *c.* 100 ad. There is also abundant evidence of metallurgical activity in later periods, and many interesting problems await investigation. One question of major interest is whether the tin sources in the Air mountains near Agadez (Greigert & Pougnet, 1967: 26–28) were exploited for the lcoal production of bronze. The stratified village site of Marandet has a series of radiocarbon dates between 250 ± 100 ad (GIF 5547) and 1420 ± 80 ad (GIF-5549). It contains huge numbers of metallurgical crucibles (Grébénart, 1985: 354–379), and evidence of the processing of lead, copper/lead alloys and brass. The sequence of use of these various alloys, the problem of distinguishing locally smelted from imported metals and the scale of production at this site are priorities for future work, as is the assessment of possible links between this metallurgical tradition and those of Nigeria. Copper production evidently continued in this area through mediaeval times. Bernus & Gouletquer (1976) have identified the archaeological site of Azelik wan Birni as the historic town of Takkeda, where Ibn Battuta reported copper mining during his visit in 1354 AD. It is clear that the Agadez region is one of major importance in the development of metallurgy in Africa, but much more field and laboratory study will be required before this sequence and its external relationships are fully understood.

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