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# Earliest Evidence for the Ivory Trade in Southern Africa: Isotopic and ZooMS Analysis of Seventh–Tenth Century AD Ivory from KwaZulu-Natal

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**Abstract** KwaGandaganda, Ndongondwane and Wosi were major Early Farming Community settlements in what is today the KwaZulu-Natal province of South Africa. These sites have yielded, among other remains, abundant evidence of ivory and ivory working dating to the seventh–tenth centuries AD, pre-dating by approximately 200 years the better-known ivory artefacts from sites in the Limpopo River Valley and surrounding regions. We report the results of carbon, nitrogen and

strontium isotope analysis to explore the origins and procurement of this ivory, in combination with Zooarchaeology by Mass Spectrometry (ZooMS) to identify the species of animals from which it was derived. All of the ivory studied using ZooMS was elephant, despite the presence of hippopotamus remains on all three sites. Some ivory was probably obtained from elephant herds that lived close to the sites, in the densely wooded river valleys favoured by both elephants and early farmers. Other material came from savannah environments further afield. Ivory found at these three sites was drawn from different catchments, implying a degree of landscape/resource partitioning even at this early stage. These communities clearly invested substantial effort in obtaining ivory from across the region, which speaks to the importance of this commodity in the economy of the time. We suggest that some ivory items were for local use, but that some may have been intended for more distant markets via Indian Ocean trade.

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Time period: Early Iron Age  
Country and region discussed: South Africa, KwaZulu-Natal

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**Résumé** Les sites de KwaGandaganda, Ndongondwane et Wosi ont été des peuplements majeurs de communautés d'agriculteurs précoces dans ce qui est aujourd'hui la province de KwaZulu-Natal, en Afrique du Sud. Ces sites ont donné, parmi d'autres vestiges, des preuves abondantes d'ivoire et d'ivoire travaillé datant des siècles VIIe à Xe de notre ère, ce qui précède, par environ 200 ans, des artefacts d'ivoire mieux connus provenant de sites de la vallée du Limpopo et des régions environnantes. Ici nous rapportons des résultats d'analyses d'isotopes de carbone, d'azote et de strontium, en combinaison avec de la zooarchéologie par

spectrométrie de masse (ZooMS) pour identifier les espèces d'animaux à partir de lesquelles l'ivoire archéologique a dérivé, et aussi pour explorer ses origines et ses marchés. Tout l'ivoire étudié en utilisant de la ZooMS a dérivé d'éléphant, malgré la présence de vestiges d'hippopotame sur les trois sites. Une partie de cet ivoire a probablement été obtenu à partir des troupeaux d'éléphants qui vivaient près des sites dans les vallées densément boisées qui étaient les favoris des éléphants aussi bien que ceux des agriculteurs précoces. D'autre matériel est. provenu de milieux de savane plus éloignés. L'ivoire trouvé aux trois sites est., donc, provenu de différents bassins versants, ce qui implique un certain degré de partitionnement de la paysage et des ressources, même à ce stade précoce. Ces communautés ont clairement investi des efforts considérables dans l'obtention de l'ivoire de toute la région, ce qui témoigne l'importance de cette denrée dans l'économie de cette époque. Nous suggérons que certains articles manufacturés de l'ivoire étaient probablement à l'usage local, mais que d'autres peuvent avoir été destinés à des marchés plus éloignés via le commerce de l'océan Indien.

**Keywords** Elephant · Carbon · Nitrogen · Strontium · Early Farming Community · Early Iron Age

## Introduction

In this paper, we report on the earliest and most southerly evidence for ivory trade in precolonial Africa. The sites of KwaGandaganda, Ndongondwane and Wosi in the South African province of KwaZulu-Natal (Fig. 1) have yielded large quantities of waste chips from ivory working (Fig. 2) and a number of (mostly fragmentary) ivory artefacts (Figs. 3 and 4). This evidence dates from the Msuluzi and Ndongondwane phases of the Early Iron Age sequence, c. seventh–tenth centuries AD. Ivory seems surprisingly abundant, so an intriguing question is the destination of this material. Overlapping with the later part of this time period, we see similar evidence of ivory working at the site of Schroda (tenth–eleventh centuries AD) further to the north in the Limpopo River Valley, and thereafter at K2, close to Mapungubwe Hill (Hanisch 1980, 1981; Raath 2014; Voigt 1983). The Limpopo River Valley is well known to have been part of a trans-Indian Ocean trade network, exporting local products that likely included ivory in exchange for glass

beads, glazed ceramics and other luxury goods. How and where did trans-oceanic trade to and from southern Africa first begin? Was ivory from KwaZulu-Natal linked to such a network, several centuries earlier?

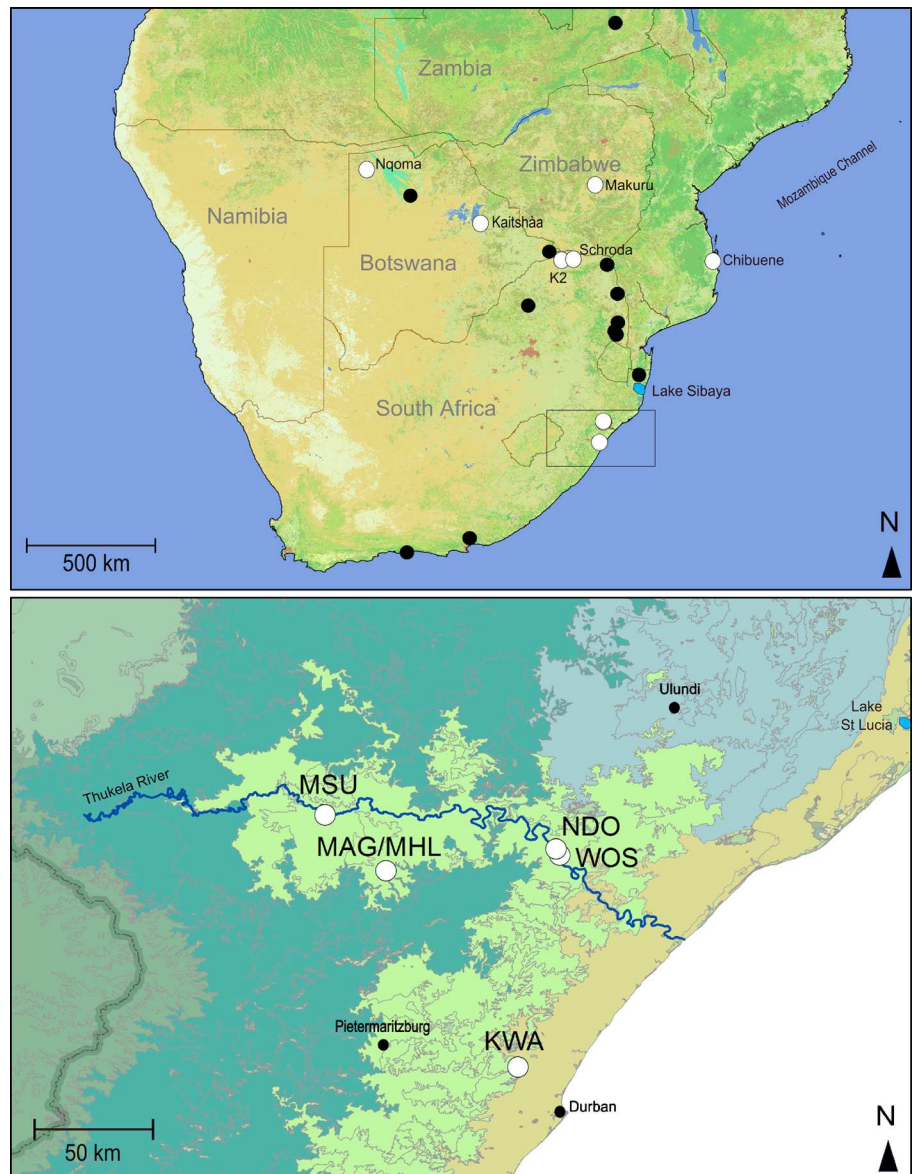
Here, we try to gain a better understanding of patterns of ivory procurement by analysing the stable carbon ( $^{13}\text{C}/^{12}\text{C}$ ) and nitrogen ( $^{15}\text{N}/^{14}\text{N}$ ) isotope composition of archaeological ivory from the sites of KwaGandaganda, Ndongondwane and Wosi, together with strontium isotopes ( $^{87}\text{Sr}/^{86}\text{Sr}$ ). Carbon and nitrogen isotopes reflect the diets the elephants consumed, informing us about the habitats in which they lived and thus the likely catchment of this material. Strontium isotopes reflect the geological substrates on which the animals lived. Did the ivory derive from local elephant populations, or was it sourced from further afield, presumably in response to greater demand? Did Early Iron Age communities use ivory from hippopotamus (*Hippopotamus amphibius*) and/or warthog (*Phacochoerus africanus*), as well as elephant (*Loxodonta africana*)? Zooarchaeology by Mass Spectrometry (ZooMS) enables us to identify even very small fragments of ivory to species. Here, we present the first such analyses in southern African archaeology.

## Early Farmers in KwaZulu-Natal

The first farmers arrived in KwaZulu-Natal c. AD 400 as part of an expansion southwards from East Africa through the eastern coastal regions of the subcontinent (Huffman 2007, pp. 331–359; Phillipson 1977; Russell et al. 2014). Links with East Africa are attested by remarkable similarities between the earliest South African ceramics and Kwale ware from Kenya and Tanzania (Huffman 2007, p. 122; Maggs 1980). These communities brought with them domesticated cattle and small stock, as well as crops such as sorghum and millet. These are tropical grains adapted to summer rainfall conditions, so farming communities settled in the northern and eastern parts of South Africa, where it is possible to grow these crops without irrigation. These groups also brought knowledge of iron smelting and iron working and were the first societies in South Africa to live in settled villages.

The chronological sequence of early farming communities in KwaZulu-Natal is based on the identification of sequential ceramic assemblages, tightly anchored in a series of radiocarbon dates. The earliest phase is called Mzonjani (AD 400–600), followed by Msuluzi

**Fig. 1** Upper map shows location of wildlife reserves (black dots) with modern elephant populations that yielded the published ivory isotope data referenced in Fig. 10. White dots indicate locations of archaeological sites in KwaZulu-Natal (KwaGandaganda, Wosi and Ndongondwane—the latter two incorporated into one white dot at this scale). Base map of vegetation cover from GlobCover © European Space Agency 2010 and UCLouvain (GlobCover 2008). Lower map is shaded by vegetation bioregions according to Mucina and Rutherford (2006) with white dots indicating locations of archaeological sites mentioned in the text (Kwa KwaGandaganda, *Wos* Wosi, *Ndo* Ndongondwane, *Msu* Msuluzi Confluence, *Mag* Magogo, *Mhl* Mhlopheni). Dark shading to the west of the sites represents sub-escarpment grassland (dark green in online version), lighter shading where the sites are located represents sub-escarpment savanna, shading northeast of the sites where Ulundi is located represents lowveld savanna (blue in online version) and shading where Durban is located represents the Indian Ocean coastal belt (brown in online version)



(650–780), Ndongondwane (780–910) and Ntshekane (910–1030), each named for the site at which it was first identified (Whitelaw 2013).

The few Mzonjani phase sites are found near the coast. From the Msuluzi phase onwards, settlements were located mainly within river valleys which provided deep, fertile soils for agriculture and ready access to water. These habitats would also have been prime elephant territory. Models of historic African elephant densities have shown that, prior to large-scale human encroachment, densities were positively correlated with high mean annual precipitation, and related ecological

variables such as biomass productivity (de Boer et al. 2013). In KwaZulu-Natal, high-resolution diatom records from Lake Sibaya (Fig. 1) indicate that conditions between AD 750 and 1000 were generally wetter than today, with a marked spike in rainfall between AD 790 and 830 (Stager et al. 2013). Vegetation in the deeply incised river valleys occupied by Early Farming Communities was denser than it is currently. Remains of nyala antelope (*Tragelaphus angasi*) at several sites in the Thukela Basin provide one proxy for environmental conditions during the second half of the first millennium (Maggs and Michael 1976; Voigt 1984). Nyala live in

**Fig. 2** Ivory working waste from KwaGandaganda, Square 22 midden, Level 2 (photo—A. Coutu)



dense bush and thicket. Today, the nearest suitable habitat occurs in northern KwaZulu-Natal. The archaeological evidence shows that during the Early Iron Age, the Thukela and other river valleys to the north were sufficiently densely vegetated to support nyala and other bush-loving species. A combination of climate change and bush clearance by early farming communities subsequently led to the extirpation of nyala at the southern end of its range. These river valleys would have supported populations of elephants that came into increasing contact with humans as settlements became larger and agricultural production grew from the Msuluzi phase (seventh c. AD) onwards. Contact and conflict between elephants and farmers in more recent times are well documented, as both seek out areas with productive soils and permanent water sources (Balfour et al. 2007; O'Connell-Rodwell et al. 2000; Sitati et al. 2005; Stone 1972).

In the next section, we summarise relevant aspects of the archaeology of the sites of Ndongondwane,

KwaGandaganda and Wosi, from which we obtained the ivory samples analysed in this study.

#### Ndongondwane

Excavations at Ndongondwane were initiated by Maggs (1984b) in 1978, followed by a second phase in 1982–1983 (Loubser 1993) and third phase beginning in 1995 (Greenfield and Van Schalkwyk 2003). Excavations into a low mound (Fig. 5) yielded a large quantity of cultural material and food waste, as well as abundant evidence of iron smelting and ivory working together with pottery, fragments of ceramic sculpture and other finds. At the base of the mound, the excavation revealed channels and post-holes, interpreted as the base of stockades supported by poles and filled in with reeds or branches (Loubser 1993; Maggs 1984b). This stockade may have served to screen off a special activity area where people worked iron and/or ivory, or it may simply have

**Fig. 3** Ivory armband found at KwaGandaganda in the red crust (burnt layer) of Grid 4, dating to the Ndongondwane phase. The armband was approximately 9.5 cm in diameter, 4.5 cm tall and a maximum of 1 cm thick (UCT lab no 14415; KZN no 22) (photo—A. Coutu)





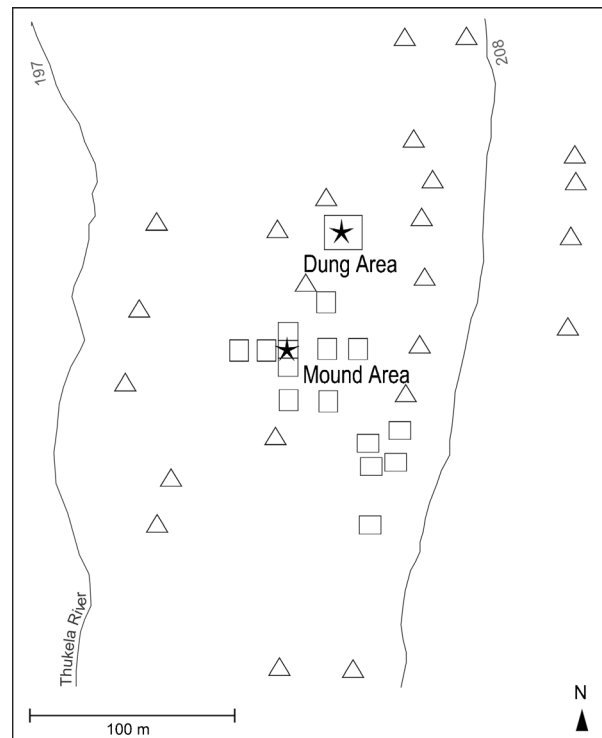
**Fig. 4** From left to right, elephant ivory copy of canine tooth from KwaGandaganda, Square 25, Level 3 (UCT 14406, KZN no 13), bone copy of a canine tooth pierced for suspension from KwaGandaganda, Square 25, canine tooth pierced for suspension

from KwaGandaganda, Trench 6, and four bone copies of canine teeth, three with perforations, from Ndongondwane, Mound Area (photos—A. Coutu)

demarcated an area where waste from these activities was deposited. The second phase of fieldwork extended the excavations into the Dung Area, interpreted as a cattle byre, and the Daga Area, an area of domestic activity. In addition, 12 auger samples were taken across the site to assess the presence of subsurface remains. The third phase of work expanded excavation of the Dung and Daga areas and also investigated additional middens to the east of earlier excavations. These yielded mainly domestic debris, although one locality revealed evidence

of charcoal and ore preparation. The occupation at Ndongondwane dates to the ninth century AD based on both radiocarbon dates and similarity in the ceramics recovered across the site; this is the name site of the Ndongondwane phase of the ceramic sequence for the region (Greenfield and van Schalkwyk 2003; Loubser 1993; Maggs 1984b). In this respect, it contrasts with many other Early Iron Age sites in the region at which occupation extended over more than one ceramic phase (see descriptions of KwaGandaganda and Wosi below).

**Fig. 5** Site map of Ndongondwane, re-drawn from Maggs (1984b, p. 73) and Loubser (1993, p. 111). Squares represent excavated areas, triangles represent auger sampling locations and stars indicate areas where ivory was found. Ivory samples taken for ZooMS and isotope analysis listed in Table 1 are from the excavation areas labelled on the map



Food waste includes both domesticated and wild species. The bulk of the remains are domesticated animals, including cattle, sheep, goats, chickens and dogs. Elephant (represented almost entirely by ivory) and hippopotamus are notable among the wide variety of wild species identified, not all of which are game animals (Fread 2007; Voigt and Von den Driesch 1984).

Ivory fragments come overwhelmingly from the Mound Area, which was likely a specialised craft activity zone (Maggs 1984b). It is interesting to note the spatial co-distribution of the remains of iron working—a male activity throughout sub-Saharan Africa—and ivory working, historically also a male activity amongst southern African Bantu speakers (Shaw 1974; Whitelaw 1994, 1994–1995). The initial excavation of the Mound Area, extending over 40 m<sup>2</sup>, produced 1,960 fragments of ivory waste weighing 1,150 g and also fragments of at least 22 armbands varying in thickness from 5 to 51 mm (Voigt and von den Driesch 1984). The amount of ivory present as well as the occurrence of both finished artefacts and working waste clearly indicate that the armbands were being manufactured on site. Near the Mound Area was what appeared to be a dump of hippopotamus bones, close to the river. These derived from at least four individuals, with 173 bones present, some still articulated and many showing butchering marks (Voigt and von den Driesch 1984). These authors suggested that this was a butchering site adjacent to the river, to avoid unnecessary transport of heavy carcasses. There was also a large unworked hippopotamus ivory fragment found in the Dung Area during the 1995 excavations. The abundance of hippopotamus remains raises the question of whether some of the worked ivory on the site might be hippopotamus, rather than elephant ivory.

Excavations at Ndondondwane also yielded a number of ostrich eggshell beads. Ostriches do not occur in bushy environments such as the Thukela Valley. Maggs (1984b) noted that none of the ostrich eggshell beads he recovered were broken in the course of manufacture, as often seen at sites in drier parts of South Africa. Beads made from the shell of the locally available land snail *Metachatina*, however, included several examples broken during manufacture. Maggs inferred that ostrich eggshell beads were not made on site, but imported from some distance away, probably from the grasslands to the west. A copper bead (Maggs 1984b) and three short pieces of copper wire (Loubser 1993) were probably also trade items. There is no evidence for local copper

production at this time and copper objects were probably traded from copper-rich regions north of the Vaal River (Miller and Whitelaw 1994).

### KwaGandaganda

KwaGandaganda was excavated from 1986 to 1989 by Whitelaw, as part of a cultural resource management project in the Mngeni Valley before the construction of the Inanda Dam (Whitelaw 1994). The site is now flooded by the dam. An area of almost 300 × 400 m was investigated by scraping away strips of topsoil with a bulldozer to assess the presence of ceramics and other archaeological remains (Fig. 6). Subsequent excavation targeted the richest areas, different types of remains and different ceramic phases. Occupation at KwaGandaganda extended through the Msuluzi, Ndondondwane and Ntshekane ceramic phases, from the seventh to early eleventh century AD. The middle phase of occupation at KwaGandaganda is therefore contemporaneous with that at Ndondondwane.

The highest density of ivory at KwaGandaganda was found in Square 25. Here, excavations extending across 18 m<sup>2</sup> yielded 3,659.7 g of ivory shavings and 10 fragments of ivory bangles (Whitelaw 1994) ranging in thickness from 18 to 27 mm. Square 25 also yielded Msuluzi phase pottery, metal and ostrich eggshell beads, worked bone, fragments of ceramic sculpture and canine teeth pierced for suspension. This concentration of material therefore dates to the Msuluzi phase and the ivory predates that at the ninth century AD site of Ndondondwane. Clearly, ivory procurement and the manufacture of bangles/armlets were already well established in the seventh and eighth centuries AD in KwaZulu-Natal.

A second concentration of ivory was found in Square 22, a Ndondondwane phase midden located close to a cattle byre. Here, finds included domestic ceramics and ceramic sculpture, faunal material, ivory shavings and an entire elephant tusk measuring 80 cm in length. Other Ndondondwane phase ivory includes a thick elephant ivory bangle recovered from the red crust in Grid 4 (Fig. 3), ivory shavings from Byre 1 (Grid 4) and Byre 2 (Grid 3) and armlet fragments from Byres 1 and 4 (Test Square 5). Skeletal elements identified as elephant were found in all phases: 11 in the Msuluzi, 5 in the Ndondondwane and 3 in the Ntshekane phase (Beukes 2000). In contrast to the large quantity of hippopotamus remains at Ndondondwane, only five identifiable

**Fig. 6** Site map of KwaGandaganda, re-drawn from Whitelaw (1994, pp. 5–6).

Squares represent excavated areas, triangles represent survey points and archaeological features, and stars represent areas of site where ivory was found. Ivory samples taken for ZooMS and isotope analysis listed in Table 1 are from the excavation areas labelled on the map



fragments of hippopotamus bone were found at KwaGandaganda, one in the Msuluzi phase, three in the Ndongondwane phase and one in the Ntshekane phase. Thirty-one fragments of warthog were also found, predominantly in the Ndongondwane and Ntshekane phases (Beukes 2000).

Many ostrich eggshell beads were recovered at KwaGandaganda, most from the Msuluzi phase with smaller numbers from later deposits. Some of these beads were unfinished, indicating that ostrich eggshell bead manufacture was occurring on site; apparently, eggshell was brought to KwaGandaganda in the form of eggs or large fragments rather than traded only as beads. There were also many marine shells, likely both food-waste and decorative items. This is not surprising given that the site is only about 30 km from the coast. It should be noted, however, that not all the marine shells were local species: one was a fragment of the abalone *Haliotis midae*. The closest recorded occurrence of this species today is at Port St Johns, approximately 250 km south of KwaGandaganda (Whitelaw 1994). Nine copper beads and one copper earring were probably also traded from some distance away.

KwaGandaganda has also yielded artefacts that originated outside Africa, including the earliest evidence of

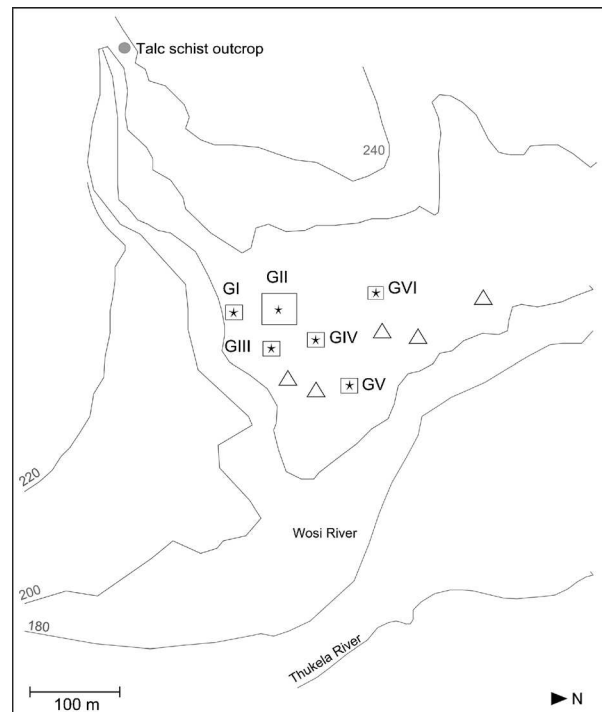
glazed ceramics thus far in southern Africa: a single sherd similar to ware recovered from ninth century AD sites in Iraq (Miller and Whitelaw 1994). One Zhizo series glass bead was found at the point of contact between Pits 2 and 4 in Grid 6. Based on ceramic style, the two pits date to the ninth and tenth centuries AD (see Whitelaw 1994, p. 17).

## Wosi

The site of Wosi lies at the confluence of the Thukela and Wosi rivers and extends over 10 ha. Six middens were excavated with extensive work carried out on the largest midden (Grid II) (Fig. 7). Occupation at Wosi occurred primarily in the Msuluzi and Ndongondwane phases. Ceramic sculptures of animal and human figures, similar to those excavated at Ndongondwane, were found in Grid II. Shell disc beads ( $n = 1,742$ ), made from the large land snail *Metachatina kraussi*, suggest a closed, wooded habitat, as this species requires shade (van Schalkwyk 1994). Faunal species such as blue duiker (*Philantomba monticola*) also signal a woodland-savanna or forest environment, further evidence for a relatively closed, bushy landscape around Wosi at the time of occupation (Voigt and Peters 1994).



**Fig. 7** Site map of Wosi, re-drawn from van Schalkwyk (1994, p. 66). *Squares* represent excavated areas, *triangles* represent surveyed middens and *stars* represent areas of site where ivory was found. Ivory samples taken for ZooMS and isotope analysis listed in Table 1 are from the excavation areas labelled on the map



Ivory was found in all of the middens investigated, in both raw and worked form. It occurred in both the Msuluzi and Ndongondwane phases of occupation (Voigt and Peters 1994). The largest number of ivory fragments derives from the lower levels of Grid II, dating to the seventh century AD (Voigt and Peters 1994). This reinforces the conclusion, drawn above on the basis of finds at KwaGandaganda, that ivory working was already well established in this area in the seventh century. Of the 427 worked ivory fragments recovered at Wosi, 379 were identified as “trimming flakes” or waste fragments and 48 were fragments of armbands. Of these, 31 were wider than 15 mm and 17 were between 0 and 15 mm, a smaller range than at Ndongondwane. Three larger fragments of ivory were identified as hippopotamus, two fragments from Grid II and one from Grid III (Voigt and Peters 1994). Hippopotamus and warthog bones were also found, though in smaller numbers (NISPs of 5 and 26, respectively), in different areas of the site and both phases of occupation. An abundance of talc-schist was found on the site, probably obtained from the nearby outcrop (Fig. 7). Objects made from talc-schist include pendants and beads for personal adornment and possibly also for trade. Over 200 ostrich eggshell beads were found, all in their complete form. Several ornamental items made

from marine shells provide evidence of coastal connections (Voigt and Peters 1994). The occurrence of stone artefacts, conforming to standardised Later Stone Age types, suggests interaction between hunter-gatherers and early farming communities. Trade in ivory may well have played a part in this interaction.

#### Other Early Iron Age Sites

Early Iron Age sites contemporary with KwaGandaganda, Ndongondwane and Wosi, but located further inland, have not yielded significant quantities of ivory. Msuluzi Confluence is the name site of the Msuluzi Phase and thus contemporary with Square 25 at KwaGandaganda and the lower levels at Wosi. This site lies on the Thukela River, upriver from Ndongondwane and Wosi (Fig. 1). It is a large site and since the excavations extended over only a limited area, there may be some as-yet-undiscovered ivory. Faunal preservation was poor, but the faunal sample recovered thus far lacks ivory (Maggs 1980). A single cowry shell and three ostrich eggshell beads provide evidence of trade or exchange with communities further afield. The sites of Magogo and Mhlopeni, in the Muden area slightly to the south-east (Fig. 1), also have not yielded significant amounts of ivory, although there is a single ivory bangle fragment in the sizeable faunal assemblage from Magogo

(Voigt 1984). The ivory working fragments that are so prominent at KwaGandaganda, Ndongondwane and Wosi were not found. Twenty-seven ostrich eggshell beads were recovered from Magogo, as well as one marine shell (*Monodonta australis*), drilled for suspension, and one estuarine shell (*Nassarius kraussianus*). Later deposits at Ntshokane and site 2931AD17 yielded one Zhizo glass bead each (Maggs and Michael 1976, p. 726; KwaZulu-Natal Museum records).

## Identifying Ivories

Ivory is a specialised tooth that grows outwardly, protruding from the mouth of the animal, and is composed primarily of dentine. Although the term ivory is usually associated with the upper incisors of an elephant, technically many species grow ivory; in this paper, we consider elephant, hippopotamus and warthog as possible sources of southern African ivory. Hippopotamus ivory may derive from the upper or lower canines, which have a distinctive curved shape, or the incisors, which are straight. Warthog ivory originates from the upper or lower canines, which have deeply grooved surfaces and strong curvature. They are much larger than the small tusks of the bushpig (*Potamochoerus larvatus*). All of these ivories can be identified by a trained analyst by eye or by microscope, on the basis of the growth structure of the dentine. Elephant ivory has a distinctive growth pattern in that it grows outwards throughout the life of the elephant in a cone-in-cone structure. This structure produces an effect called the Schreger pattern, visible even to the naked eye when examining elephant ivory in cross-section (Locke 2008; Raubenheimer et al. 1990). It is, however, necessary to have relatively large pieces of ivory in order to see these patterns, and many of the ivory fragments found at Iron Age sites in southern Africa are small, especially waste from ivory working (Fig. 2). These require biomolecular methods for identification.

Large caches of hippopotamus ivory have been found at Iron Age trading sites such as Gao in West Africa (Insoll 1995). Since all three of the KwaZulu-Natal sites investigated here are riverine sites, and several butchered hippopotami were found at Ndongondwane, it was unclear whether hippopotamus ivory was also being used to make artefacts such as bangles. We therefore wanted to investigate whether ivory fragments at these three sites were consistently derived from elephant or might have come from diverse species.

## Biomolecular Methods for Identifying Ivories

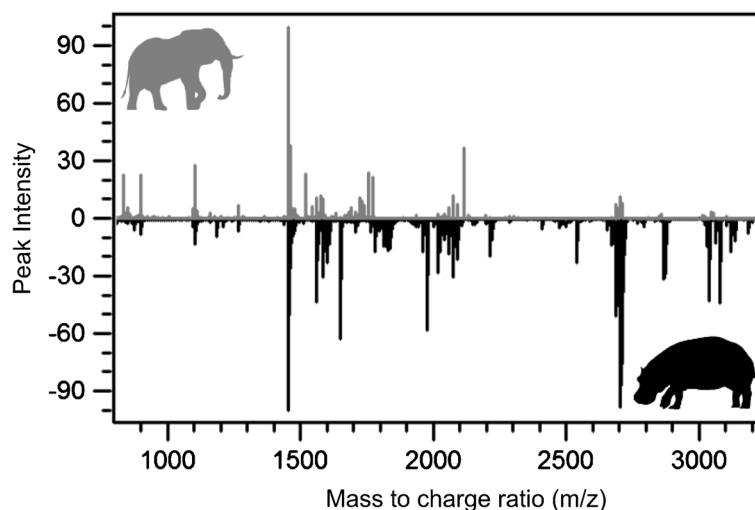
ZooMS is a method of taxonomic identification based upon peptide mass fingerprinting (the acronym ZooMS and method defined by van Doorn 2014); when applied to ivory and other skeletal hard tissues, it uses the structural protein collagen. Differences in the sequence of amino acids in the collagen of various species result in characteristic peptide profiles. In combination with a knowledge of the archaeozoology of the site, it is possible to use these to identify unknown fragments of bone, tooth or other materials (e.g., hair, Hollemeyer et al. 2012; or eggshell, Stewart et al. 2014). Very small amounts of collagen are extracted, converted to gelatin and the gelatin enzymatically fragmented into small peptides. The masses of these peptides are determined by time-of-flight mass spectrometry and compared with known reference sequences (Buckley et al. 2009, 2010; Buckley and Collins 2011; Kirby et al. 2013; von Holstein et al. 2014). Collagen is more robust than DNA, and ZooMS requires smaller samples than equivalent DNA sequencing analysis, so it was the preferred method here. Indeed, ZooMS analysis can be effectively nondestructive since a sample sufficient for analysis may be obtained by rubbing the surface of the object with a PVC eraser and analysing the peptide sequences in the protein remaining on the eraser “crumbs” (Fiddymet et al. 2015). ZooMS is an effective way to distinguish between hippopotamus and elephant because these two species have distinctive mass spectra (Fig. 8; Buckley et al. 2009). If one of the peptides characteristic of elephant or hippopotamus is preserved, it is often possible to distinguish between the two species even if the overall quality of the spectrum is poor due to bad protein preservation. ZooMS cannot distinguish between the two species of African elephant: forest (*Loxodonta cyclotis*) and savanna (*Loxodonta africana*). In Early Iron Age KwaZulu-Natal, only savanna elephants would have been present in the landscape.

## Isotopes as Tracers of Diet and Environment

### Carbon Isotopes

In tropical and subtropical Africa, where rain falls predominantly in the summer, most grasses utilise the C<sub>4</sub> photosynthetic pathway, while shrubs, trees and other

**Fig. 8** Mass spectra from elephant (*grey*) and hippopotamus (*black*) reference samples, showing the difference in peptide mass to charge ratio versus peak intensity for each species



dicotyledonous plants utilise the  $C_3$  pathway. These two pathways result in very different  $^{13}C/^{12}C$  ratios in plant tissue. Carbon isotope ratios are expressed as  $\delta^{13}C$  values, where

$$\delta^{13}C_{\text{sample}} = \left( \frac{^{13}C/^{12}C_{\text{sample}}}{^{13}C/^{12}C_{\text{standard}}} - 1 \right) \times 1000 \text{‰}$$

the standard is Pee Dee Belemnite (PDB) which by definition has  $\delta^{13}C = 0 \text{‰}$ . Samples with negative  $\delta^{13}C$  values therefore contain less of the heavy  $^{13}C$  isotope than the standard, while those with positive values contain more. The mean  $\delta^{13}C$  for modern  $C_3$  plants is  $-27.1 \pm 1.6 \text{‰}$ , with a range from  $-20$  to  $-37 \text{‰}$  (Kohn 2010). The mean for modern  $C_4$  grasses is  $-12.5 \pm 1.1 \text{‰}$  with a range from  $-9$  to  $-17 \text{‰}$  (Cerling et al. 1997). The greater range in  $C_3$  plants is due to wide diversity of habitats and also to environmentally induced shifts in  $\delta^{13}C$  as a result of variations in moisture, light, temperature and similar factors. Before the Industrial Revolution and the combustion of large quantities of fossil fuels,  $\delta^{13}C$  of atmospheric carbon dioxide and of food chains as a whole were approximately  $1.5 \text{‰}$  more positive than today (Francey et al. 1999). We have taken this into account in our interpretation of the analyses of archaeological ivory.

The carbon isotope composition of consumers reflects the foods consumed. Most tissues have more positive  $\delta^{13}C$  than the food due to further partitioning of carbon isotopes during digestion and tissue synthesis. Elephants are mixed feeders, relying on both  $C_3$  browse and  $C_4$  graze; hippopotami are primarily grazers; and warthogs are solely grazers.  $\delta^{13}C$  values of ivory can therefore be used to discriminate between species. African savanna elephant diets are seasonally variable, with animals tending to graze in the wet season, when fresh

new grass is available, and browse in the dry season (Skinner and Chimimba 2005). This pattern has been documented in both East and South Africa in  $\delta^{13}C$  measurements of sequential subsamples of elephant hair (Cerling et al. 2009) and multiple small samples of ivory from cross-sections of tusks (Codron et al. 2012). Such isotopic heterogeneity has implications for the sampling strategy in this study (see below).

Comparison of the  $\delta^{13}C$  values of elephants and hippopotami in East Africa shows that elephants have more negative values, as expected given that a considerable proportion of their diet is  $C_3$  browse (Ambrose and DeNiro 1986; Tieszen and Imbamba 1980). The difference between the two species is, however, geographically and temporally variable, depending on the local environment and vegetation (Cerling et al. 2007, 2008). This will need to be determined for the Early Iron Age of KwaZulu-Natal, especially since river valleys were more densely vegetated at that time. Warthog in both East and South Africa show  $\delta^{13}C$  values typical of  $C_4$  grazers (Ambrose and DeNiro 1986; Botha and Stock 2005; Cerling et al. 2008; Codron et al. 2007; Tieszen and Imbamba 1980).

Isotope values for collagen extracted from ivory (dentine) are very similar to those for bone collagen, although it should be noted that dentine is laid down incrementally and does not remodel as bone does. The large number of  $\delta^{13}C$  values for bone collagen from modern southern African mammals suggest that animals that eat only browse ( $C_3$  plants) have  $\delta^{13}C_{\text{collagen}}$  of about  $-21 \text{‰}$ , while those eating only  $C_4$  grasses have values

of about  $-7\text{‰}$  (Codron et al. 2012; Lee-Thorp et al. 1989; Sponheimer et al. 2003a; Vogel 1978). Animals consuming a mixture of the two have intermediate  $\delta^{13}\text{C}$  values, depending on the proportions of graze and browse. Variations in moisture and temperature that affect the  $\delta^{13}\text{C}$  values of plants (especially  $\text{C}_3$  plants) can lead to small variations in consumer  $\delta^{13}\text{C}$ .

### Nitrogen Isotopes

Variation in stable nitrogen isotopes ( $^{15}\text{N}/^{14}\text{N}$ ) is more complex and less well understood than variation in carbon isotopes. This is, in part, because of the complexity of the nitrogen cycle, with a large number of steps involved in nitrogen fixation and conversion of fixed nitrogen between different species.  $\delta^{15}\text{N}$  values (calculated in the same way as  $\delta^{13}\text{C}$ , the standard is atmospheric  $\text{N}_2$  or AIR) of soils, plants and animals tend to be higher in arid environments (Amundson et al. 2003; Aranibar et al. 2004; Murphy and Bowman 2006, 2009). They are typically also higher in animals that obtain most of their moisture requirements from their food, compared with those that drink water regularly (Ambrose and DeNiro 1986; Ambrose 1991; Sponheimer et al. 2003b). Since elephant, hippopotamus and warthog are all water-dependent (Skinner and Chimimba 2005), we do not expect inter-species differences in  $\delta^{15}\text{N}$  to be as large as those in  $\delta^{13}\text{C}$ . Wet/dry season variation in  $\delta^{15}\text{N}$  of growth increments of elephant hair and ivory is less pronounced than in  $\delta^{13}\text{C}$  (Cerling et al. 2009; Codron et al. 2012).

Previous studies of African fauna (including elephants) have documented a negative correlation between  $\delta^{15}\text{N}$  in animal tissues and mean annual precipitation (Coutu 2011; Heaton et al. 1986; Ishibashi et al. 1999; van der Merwe et al. 1990). Elephants living in moist areas may have  $\delta^{15}\text{N}$  as low as  $2\text{‰}$ , while in arid areas (Ethiopia, Namibia and Somalia) values up to  $17\text{‰}$  have been recorded, although there is considerable variation according to local nitrogen cycling (Coutu 2011; van der Merwe et al. 1990).

### Strontium Isotopes

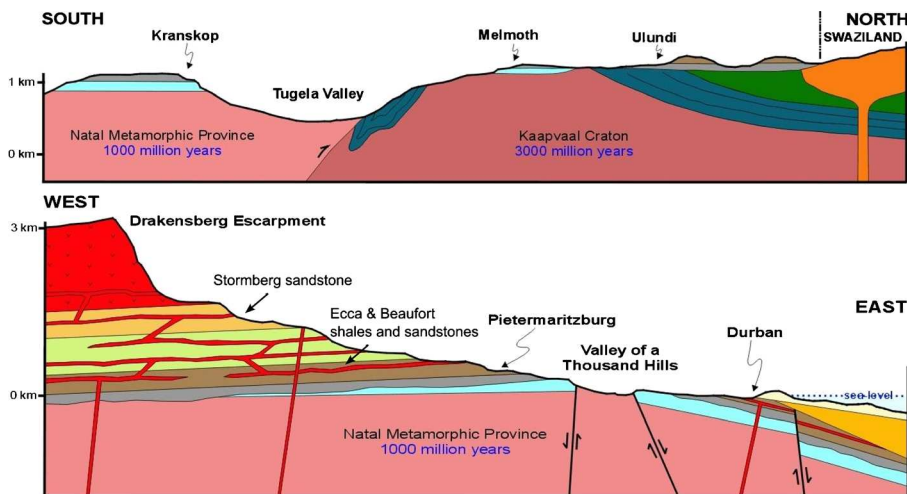
Strontium isotope ratios ( $^{87}\text{Sr}/^{86}\text{Sr}$ ) vary in different geological formations depending on the relative amounts of Rb (rubidium) and Sr that the rock contains, and the age of the formation.  $^{87}\text{Rb}$  is radioactive and decays to  $^{87}\text{Sr}$ , hence older rocks or those with high Rb/

Sr elemental abundance ratios evolve to high present-day  $^{87}\text{Sr}/^{86}\text{Sr}$  values. In KwaZulu-Natal, the geology in the environs of Wosi and Ndongondwane consists of very ancient, highly metamorphosed rocks, mainly gneisses of the Dondwana Gneiss Unit, part of the Mandleni tectonostratigraphic package of the Tugela terrane which forms the northernmost part of the Natal Belt (Johnston et al. 2002). There is, however, considerable complexity, with many intrusions. KwaGandaganda lies in the deeply incised Mngeni Valley (Valley of a Thousand Hills) which cuts through younger Karoo sedimentary rocks into older granite. The surrounding highlands consist of Natal Group sandstones/quartzites and Karoo sedimentaries (Fig. 9). Soils in river valleys may be mixed if the rivers carry down sediments from geological formations upstream. In addition, biologically available strontium can have isotope ratios that differ from whole-rock values if the various components of the rock are differentially soluble and have varied  $^{87}\text{Sr}/^{86}\text{Sr}$  (Hodell et al. 2004; Sillen et al. 1998).

For these reasons, Arnold et al. (2013) measured nine modern *Achatina* shells from the lower Thukela Valley to assess bioavailable  $^{87}\text{Sr}/^{86}\text{Sr}$ , obtaining values between 0.7081 and 0.7159. As these authors note, this is a large range. This is unsurprising given that the local geology can be extremely variable. It should be noted that individual specimens of *Achatina* have limited mobility, so  $^{87}\text{Sr}/^{86}\text{Sr}$  of single shells represent values integrated over small areas. More mobile species such as cattle and elephants will average  $^{87}\text{Sr}/^{86}\text{Sr}$  over larger scales. Analyses of archaeological cattle teeth from the sites of Ndongondwane, Wosi and Mamba (very close to Wosi) yielded a range of 0.7094 to 0.7216. Extreme values were thought to indicate animals brought in from outside the Thukela Valley (Arnold et al. 2013).

$^{87}\text{Sr}/^{86}\text{Sr}$  measurements on modern small mammals from different geological formations in KwaZulu-Natal analysed by the authors are as follows: 0.71706 to 0.71931 for three samples from the Natal Group, 0.71448 to 0.71686 for three samples from Karoo dolerite, 0.71229 to 0.71558 for 10 samples from Ecca/Beaufort and 0.71022 to 0.71304 for three samples from the Berea group (see [Supplementary Material Online](#) for data and Fig. 9 for a schematic diagram of the geology of KwaZulu-Natal).

Thus, while the geology in the environs of KwaGandaganda, Ndongondwane and Wosi is complicated,  $^{87}\text{Sr}/^{86}\text{Sr}$  measurements of archaeological ivory



**Fig. 9** Geological map of the KwaZulu-Natal region (Whitmore et al. 1999)

may be able to cast some light on the geological substrates on which the elephants lived, complementing information obtained from carbon and nitrogen isotopes.

## Laboratory Methods

### Carbon and Nitrogen Isotope Analysis

Elephant, hippopotamus and warthog ivory grows continuously and incrementally throughout the lifetime of the animal. In order to obtain meaningful isotopic measurements for each ivory fragment, it is important to sample across multiple growth layers. A guiding principle in our sampling strategy was to take sufficiently large, clean pieces, oriented in such a way that ivory from more than one season was incorporated into the material analysed. As far as possible, an effort was also made to select samples from different spatial and stratigraphic units within each archaeological site, in order to minimise the chances of repeat sampling pieces from an individual tusk.

For collagen extraction, fragments were removed or the object was drilled using a clean diamond-tip drill bit on a Dremel hand drill and the sample collected as powder. Collagen extraction followed the method of O'Connell and Hedges (1999) with some modifications. Briefly, cleaned chips or ivory powders were demineralised in 0.3 M HCl at room temperature for a period of up to 1–2 weeks, then rinsed with distilled water. They were then treated with 0.1 M NaOH to

remove humic acids derived from the burial environment, then rinsed with distilled water. The samples were then put into pH 3, 0.01 M HCl and heated to 70 °C for 48 h to denature the collagen (“gelatinization”). They were then filtered through 60–90 µm Ezeec® filters to remove large debris and subsequently lyophilised for analysis.

Approximately 0.5 mg of each sample was weighed into a tin capsule for combustion into N<sub>2</sub> and CO<sub>2</sub> gases on a Thermo Flash Elemental Analyser 2000. These were then introduced to a Delta V Plus mass spectrometer for measurement of <sup>13</sup>C/<sup>12</sup>C and <sup>15</sup>N/<sup>14</sup>N ratios as well as elemental compositions (%C, %N). The results are expressed in the delta (δ) notation in parts per thousand (‰), relative to the international standards Vienna Pee Dee Belemnite (VPDB) for carbon and Ambient Inhalable Reservoir (AIR) for nitrogen. All samples were run in duplicate and the results averaged to give the values listed in Table 1. The reproducibility of repeated measurements of homogeneous standard materials was 0.2 ‰ or better for both carbon and nitrogen.

### Strontium Isotope Analysis

Strontium isotope analysis was performed in the Department of Geological Sciences at the University of Cape Town. Four samples were subjected to a leaching experiment in order to assess whether the ivory might have absorbed Sr from the burial environment, leading to erroneous assessment of <sup>87</sup>Sr/<sup>86</sup>Sr. Post-depositional contamination (or diagenesis) may occur via Sr adsorption or incorporation of contaminating ions into

**Table 1** ZooMS and isotope data for all samples analysed in this study

UCT number	KZN number	ZooMS ID	$\delta^{15}\text{N}$ (‰)	%N (by weight)	$\delta^{13}\text{C}$ (‰)	%C (by weight)	C/N (atomic)	$^{87}\text{Sr}/^{86}\text{Sr}$	Location within site
KwaGandaganda									
17450	60		9.9	14.1	-22.4	40.7	3.4		Grid 3
17458	68		7.0	15.0	-19.4	41.9	3.3		Grid 3
14415	22		7.9	11.8	-21.2	31.8	3.2		Grid 4
14414	21							0.71612	Grid 6
14401	8		9.8	10.9	-20.2	33.7	3.6		Test Square 5
14402	9		8.6	11.9	-21.4	35.7	3.5		Test Square 21
14409	16							0.71712	Test Square 22
14397	4		11.6	11.8	-22.5	35.5	3.5		Square 22
17451	61		8.5	14.5	-19.8	40.6	3.3		Square 22
14405	12		11.4	12.7	-17.0	38.1	3.5		Square 25
14407	14		9.5	14.8	-19.4	41.4	3.3		Square 25
14411	18	Elephant	8.3	15.0	-20.6	41.6	3.2	0.71548	Square 25
14413	20		7.8	13.5	-20.2	37.8	3.3		Square 25
17452	62	Elephant	9.6	14.6	-18.7	41.2	3.3		Square 25
17464	74		9.3	14.9	-19.6	42.0	3.3		Square 25
17465	75		7.4	14.9	-20.0	41.4	3.2		Square 25
17488	98		7.6	14.5	-20.2	40.9	3.3		Square 25
17456	66		9.0	14.9	-19.5	42.0	3.3		Square 25a
17457	67	Elephant	9.4	14.7	-18.8	41.1	3.3		Square 25a
14403	10		10.0	11.3	-16.2	30.3	3.1		Square 25b
14404	11		7.4	12.1	-21.0	33.2	3.2		Square 25b
14410	17		8.1	13.6	-21.0	37.7	3.2	0.71626	Square 25b
17453	63	Elephant	13.0	16.2	-17.3	45.4	3.3		Square 25b
17459	69		10.2	14.9	-19.9	42.5	3.3		Square 25b
17460	70		8.6	15.5	-20.0	43.0	3.2		Square 25b
17461	71	Elephant	10.3	15.3	-16.9	42.4	3.2		Square 25c
14406	13	Elephant	14.3	13.5	-17.3	37.9	3.3		Square 25d
14412	19		7.1	14.1	-20.9	42.6	3.5	0.71498	Square 25d
17454	64		11.8	14.5	-19.1	42.5	3.4		Square 25d
17455	65	Elephant	11.0	14.6	-17.1	41.6	3.3		Square 25d
17491	101	Elephant	8.6	14.8	-18.5	41.7	3.3		Square 25d
17462	72		7.8	14.7	-20.0	40.8	3.2	0.71611*	Square 25e
17463	73		10.5	15.0	-20.2	41.9	3.3		Square 25e
17466	76		8.4	14.9	-19.6	41.7	3.3		Square 25e
17467	77		7.6	14.8	-20.5	43.1	3.4		Square 25e
17468	78		7.2	15.0	-20.1	42.4	3.3		Square 25e
17469	79		8.5	14.8	-19.8	41.2	3.3		Square 25e
17470	80		6.9	14.6	-20.1	41.2	3.3	0.71532*	Square 25e
17471	81		8.1	14.3	-22.2	40.0	3.3		Square 25e
17489	99		7.9	15.1	-20.0	41.9	3.2		Square 25e
17490	100		8.1	15.1	-20.3	42.0	3.3		Square 25e
Ndongondwane									
17482	92		9.4	15.5	-22.6	43.9	3.3		C10
14428	35		7.4	15.0	-20.4	42.2	3.3		E8

**Table 1** (continued)

UCT number	KZN number	ZooMS ID	$\delta^{15}\text{N}$ (‰)	%N (by weight)	$\delta^{13}\text{C}$ (‰)	%C (by weight)	C/N (atomic)	$^{87}\text{Sr}/^{86}\text{Sr}$	Location within site
14429	36		7.6	13.1	-21.0	39.2	3.5		E8
17474	84		7.7	14.8	-20.4	41.5	3.3		E10
14423	30		7.6	14.7	-21.2	40.8	3.2	0.71255*, 0.71289	G9
14424	31	Elephant	10.1	14.8	-19.3	42.8	3.4		G9
14427	34	Elephant	10.2	14.5	-21.0	40.6	3.3		G9
17479	89		7.4	15.6	-20.4	43.7	3.3		G9
17480	90		6.1	14.5	-20.8	40.5	3.2		G9
17481	91		8.3	14.5	-20.6	40.9	3.3		G9
14417	24		12.2	12.8	-20.5	35.3	3.2	0.71321	G10
14419	26		7.6	14.2	-20.6	41.0	3.4	0.71366	G10
17475	85		11.1	14.7	-20.7	41.4	3.3		G10
17476	86		8.2	15.0	-20.8	41.8	3.2		G10
17478	88	Elephant	7.6	14.9	-18.6	42.1	3.3		G11
14426	33	Elephant	9.9	12.7	-20.7	35.4	3.3		G12
14433	40							0.71242	G12
14446	52	Elephant	12.0	13.7	-19.8	38.7	3.3		H9?
14448	54	Elephant	10.9	14.4	-20.5	40.6	3.3		H9?
14420	27	Elephant	7.1	11.3	-20.3	31.6	3.3	0.71398	H10
14421	28		10.7	14.9	-21.0	41.9	3.3	0.71329	H10
14422	29							0.71363	H10
17487	97		8.8	14.4	-21.7	40.4	3.3		H10
14447	53	Elephant	12.0	14.5	-19.3	42.2	3.4		H11
14449	55	Elephant	7.9	14.3	-20.4	41.5	3.4		H11
14430	37		7.7	14.8	-19.3	41.9	3.3		I10
17472	82		11.3	15.1	-21.9	42.8	3.3	0.71429*	I10
17484	94		7.1	15.4	-20.6	42.6	3.2		I10
17485	95		7.2	14.6	-20.9	41.0	3.3		I10
14431	38	Elephant	7.3	14.6	-19.8	42.0	3.4	0.71268*	K10
Ndongondwane Hippopotamus									
14450	56		9.1	14.6	-9.4	40.7	3.3		Dung Area
14434	41		8.2	12.5	-9.7	34.9	3.3	0.71497	G10
14436	43		8.0	13.6	-12.5	41.5	3.6		G10
Wosi									
17499	109		10.5	14.4	-19.4	41.6	3.4		Grid 1
17494	104	Elephant	10.2	14.7	-18.2	41.6	3.3		Grid 2
17495	105	Elephant	12.7	14.8	-15.2	41.1	3.2		Grid 2
17497	107	Elephant	11.9	15.0	-16.8	41.3	3.2		Grid 2
17501	111		8.9	14.8	-20.0	41.5	3.3		Grid 2
17504	114	Elephant	13.1	11.2	-17.5	31.1	3.3		Grid 2
17506	116	Elephant	11.7	14.8	-18.3	41.2	3.2		Grid 2
17509	119		11.8	14.5	-17.3	40.2	3.2	0.71289*	Grid 2
17510	120		8.0	14.4	-19.6	40.6	3.3		Grid 2
17496-A	106-A	Elephant	12.9	13.8	-17.0	39.0	3.3		Grid 2
17508	118	Elephant	12.3	14.9	-17.0	41.8	3.3		Grid 3
17500	110		7.9	16.0	-20.6	45.0	3.3		Grid 4

**Table 1** (continued)

UCT number	KZN number	ZooMS ID	$\delta^{15}\text{N}$ (‰)	%N (by weight)	$\delta^{13}\text{C}$ (‰)	%C (by weight)	C/N (atomic)	$^{87}\text{Sr}/^{86}\text{Sr}$	Location within site
17502	112	Elephant	12.5	12.9	-17.6	36.7	3.3		Grid 4
17503	113		8.9	14.7	-19.2	41.9	3.3		Grid 4
17505	115		8.2	14.2	-21.2	42.5	3.5		Grid 6
Wosi hippopotamus									
17496	106		6.5	14.4	-10.5	43.6	3.5		Grid 5
KwaZulu-Natal Museum historic elephants									
17492	102		10.0	15.9	-20.3	45.7	3.3		Shot 1916
17492-A	102-A		10.2	14.9	-20.8	41.7	3.3		Pre-1880

$^{87}\text{Sr}/^{86}\text{Sr}$  values with asterisks indicate samples analysed by laser ablation MC-ICP-MS. Others were analysed by solution. For more detailed provenance information for each sample, please refer to Online Resource 3

recrystallised mineral. Either way, diagenetic material is more soluble than biogenic mineral, so that leaching in dilute acid preferentially removes the diagenetic component. Approximately 50 mg of each powdered sample was washed 30 times in a 100 mM acetic acid/sodium acetate buffer adjusted to pH 4.5 following the method outlined in Sealy et al. (1991). If there is significant diagenetic contamination by Sr with an isotopic composition different from that of biogenic Sr, then  $^{87}\text{Sr}/^{86}\text{Sr}$  of the early washes should differ from the later washes. Every five washes were pooled for analysis of  $^{87}\text{Sr}/^{86}\text{Sr}$ .  $^{87}\text{Sr}/^{86}\text{Sr}$  was also measured in the undissolved residue, using the solution method described below.

“Solution” analyses were carried out as follows. Up to 25 mg of each ivory sample was dissolved in 2 ml 65 % 2B  $\text{HNO}_3$  in a closed Teflon beaker and placed on a hotplate at 140 °C for an hour. It was then dried down and re-dissolved in 1.5 ml 2 M  $\text{HNO}_3$  for strontium separation chemistry, following the method of Pin et al. (1994). The separated strontium fraction for each sample was dried down, dissolved in 2 ml 0.2 %  $\text{HNO}_3$  and diluted to 200 ppb Sr concentrations for isotope analysis using a Nu Instruments NuPlasma high resolution multi-collector inductively coupled plasma mass spectrometer (HR MC-ICP-MS). Analyses were referenced to bracketing analyses of NIST SRM987, using  $^{87}\text{Sr}/^{86}\text{Sr}$  reference value of 0.710255. All strontium isotope data are corrected for isobaric rubidium interference at 87 amu using the measured signal for  $^{85}\text{Rb}$  and the natural  $^{85}\text{Rb}/^{87}\text{Rb}$  ratio. Instrumental mass fractionation was corrected using the measured  $^{86}\text{Sr}/^{88}\text{Sr}$  ratio and the exponential law, and a true  $^{86}\text{Sr}/^{88}\text{Sr}$  value of 0.1194. Analytical error associated with measurements by solution is  $\pm 0.00002$  ( $2\sigma$ ).

Laser ablation MC-ICP-MS Sr isotope analysis followed the procedure originally presented in Copeland et al. (2008) and recently evaluated further by le Roux et al. (2014). The same analytical instrumentation described in those publications, a NuPlasma MC-ICP-MS coupled to a NewWave UP-213 laser ablation unit, was used in this study. The error associated with  $^{87}\text{Sr}/^{86}\text{Sr}$  measurements by laser ablation on this system is  $\pm 0.0003$  ( $2\sigma$ ).

#### ZooMS Analysis

Selected samples were also analysed in the BioArCh laboratories for ZooMS at the University of York, UK. Where sufficient sample material was available, approximately 0.5 mg of collagen extracted for stable isotope analysis as described above was used for ZooMS by heating in 50 mM ammonium bicarbonate buffer at 65 °C for 1 h to solubilise a small fraction of the collagen. This soluble fraction was trypsinated overnight at 37 °C to break the protein into peptide fragments, which were then concentrated and eluted using C18 Zip Tips. Mass spectra (as shown in Fig. 8) were measured using a calibrated Ultraflex III (Bruker Daltonics) MALDI-TOF MS instrument in reflector mode and compared with reference spectra (Buckley et al. 2009).

## Results

#### Collagen Quality Indicators

Collagen extracts in this study had C/N ratios ranging from 3.1 to 3.6 (Table 1). Values between 2.9 and 3.6 are



generally considered characteristic of collagen that is sufficiently well preserved that measurement of the stable isotope ratios will yield reliable results (Ambrose 1990; DeNiro 1985; Van Klinken 1999). %C (by weight) varied from 30.2 to 45.4 and %N from 10.9 to 16.2, also within the ranges seen in well-preserved collagen.

### ZooMS

All of the 26 collagen samples analysed using ZooMS could be successfully identified to species, and all proved to be elephant ivory. Six of the samples were selected for ZooMS because “hippo?” was written on their museum labels, probably due to the presence of ridged surfaces resembling hippopotamus teeth. Another specimen, from Square 25 at KwaGandaganda (UCT 14406, KZN no 13), is an ivory copy of a perforated canine tooth like that found in the same context (Fig. 4) (Whitelaw 1994). The ZooMS results showed that this piece is also elephant ivory (spectra in Online Resource 1). All of the worked ivory fragments identified thus far using ZooMS are therefore elephant ivory, despite the presence of hippopotamus skeletal remains at all three sites.

### Carbon Isotopes

Three fragments of clearly identifiable non-artefactual hippopotamus teeth from Ndongondwane and one from Wosi yielded  $\delta^{13}\text{C}$  values between  $-12$  and  $-9$  ‰. These indicate consumption of substantial quantities of  $\text{C}_4$  grasses and are significantly more positive than the  $\delta^{13}\text{C}$  of the rest of the ivory analysed (Fig. 10). In the late first millennium AD Thukela Valley, hippopotamus clearly grazed to a much greater extent than elephants.  $\delta^{13}\text{C}$  values of 39 ivory samples from KwaGandaganda ranged from  $-22.5$  to  $-16.2$  ‰, with a mean of  $-19.7 \pm 1.5$  ‰. Most of our material from KwaGandaganda derives from Square 25, which dates to the Msuluzi phase of the Early Iron Age.  $\delta^{13}\text{C}$  values of the six specimens from areas other than Square 25 tend to cluster at the negative end of the range. This might be taken to hint at changes in ivory source areas over time, but we wish to be cautious in our interpretation of this small sample.  $\delta^{13}\text{C}$  values of 28 ivory samples from the site of Ndongondwane ranged from  $-22.6$  to  $-18.6$  ‰, with a mean of  $-20.5 \pm 0.8$  ‰, and 15 from Wosi ranged from  $-21.2$  to  $-15.2$  ‰, with a mean of  $-18.3 \pm 1.6$  ‰. The distributions of values from each pair of sites is significantly different (Mann-

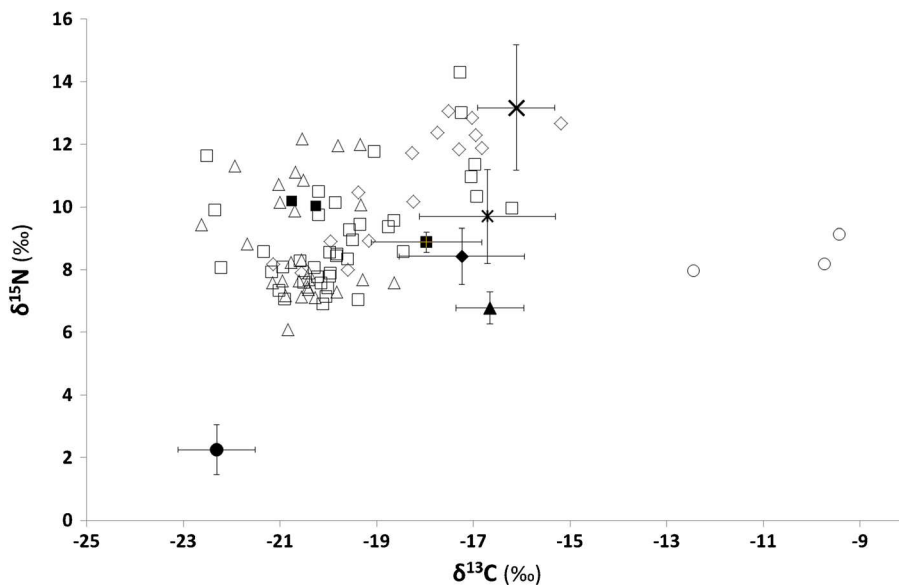
Whitney  $Z$ -value for KwaGandaganda vs. Ndongondwane is 2.79, for KwaGandaganda vs. Wosi 2.65, for Ndongondwane vs. Wosi 3.96,  $p < 0.01$  in each case). As can be seen from Fig. 10, values for Wosi tend to be  $^{13}\text{C}$  enriched compared with the other two sites, indicating that these animals consumed more grass. Samples from two historic elephants, on display in the KwaZulu-Natal Museum, fall in the middle of the range of archaeological samples, with  $\delta^{13}\text{C}$  values of  $-20.3$  and  $-20.8$  ‰, respectively. One, the skeleton of a bull of 55 years, lived on the Mfolozi flats in the Mtubatuba area, inland of the St. Lucia estuary (no known museum accession number). He was illegally shot and killed in 1916. The other, a single, heavily weathered tusk, was found buried in the bank of the Dorpspruit in Pietermaritzburg in about 1880 (museum accession number 542).

Although there is a wide range of  $\delta^{13}\text{C}$  values, the median of those from KwaGandaganda and Ndongondwane is  $-20.3$  ‰. This is close to the value expected for purely  $\text{C}_3$  diets, when adjusted for depletion of  $\delta^{13}\text{C}$  in atmospheric carbon dioxide due to the burning of fossil fuels (see caption to Fig. 10). Many of these elephants, therefore, ate largely or entirely  $\text{C}_3$  diets and did not consume  $\text{C}_4$  grasses. These animals must have lived in forested or dense bushy habitats.

The fact that the  $\delta^{13}\text{C}$  values of hippopotamus are so much more positive than those of elephants, and clearly distinguishable from them, is consistent with the results of the ZooMS analyses. All of the waste ivory fragments in this study from all three sites derive from elephants and not hippopotami. Warthog would also have very positive  $\delta^{13}\text{C}$ , so are clearly not represented here.

### Nitrogen Isotopes

$\delta^{15}\text{N}$  values of 39 ivory samples from KwaGandaganda ranged from 6.9 to 14.3 ‰, with a mean of  $9.1 \pm 1.7$  ‰. Those from Ndongondwane ranged from 6.1 to 12.2 ‰, with a mean of  $8.9 \pm 1.8$  ‰ ( $n = 28$ ), and 15 samples from Wosi ranged from 7.9 to 13.1 ‰, with a mean of  $10.8 \pm 1.9$  ‰. The distributions of  $\delta^{15}\text{N}$  values from KwaGandaganda and Ndongondwane are not significantly different (Mann-Whitney  $Z$ -value is 0.72,  $p = 0.47$ ), but both are significantly different from Wosi (Mann-Whitney  $Z$ -value for KwaGandaganda vs. Wosi 2.85, for Ndongondwane vs. Wosi 3.11,  $p < 0.01$  for both).  $\delta^{15}\text{N}$  values for Wosi tend to be slightly more positive compared with the other two sites. In this data set, higher  $\delta^{15}\text{N}$  values are much more characteristic of



**Fig. 10**  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  values for archaeological ivory from KwaZulu-Natal compared with reference samples from southern African wildlife parks. Samples from KwaGandaganda are shown as *open squares*, Ndongondwane as *open triangles*, Wosi as *open diamonds* and non-artefactual hippopotamus ivory from Ndongondwane and Wosi as *open circles*. Reference samples include two KwaZulu-Natal Museum historic elephants shown as *filled squares* and modern elephants plotted as means  $\pm$  one standard deviation. Ivory from Addo (South Africa) identified as larger bold *X* (van der Merwe et al. 1990; Vogel et al. 1990)

( $n = 14$ ), Botswana as smaller *X* (van der Merwe et al. 1990) ( $n = 7$ ), Tembe (South Africa) as *filled square with cross* (van der Merwe et al. 1990) ( $n = 6$ ), Kruger (South Africa) as *filled diamond* (Codron et al. 2012; van der Merwe et al. 1990; Vogel et al. 1990) ( $n = 44$ ), Zambia as *filled triangle* (van der Merwe et al. 1990) ( $n = 4$ ) and Knysna (South Africa) as *filled circle* (van der Merwe et al. 1990; Vogel et al. 1990) ( $n = 5$ ). 1.5 ‰ has been added to  $\delta^{13}\text{C}$  values of modern elephants to correct for depletion of  $^{13}\text{C}$  in atmospheric  $\text{CO}_2$  since the Industrial Revolution, due to burning of fossil fuels (Francey et al. 1999)

more positive  $\delta^{13}\text{C}$  values, as expected if these animals came from drier, savanna-type environments. The two historic KwaZulu-Natal elephants fall in the middle of the range of archaeological samples, with  $\delta^{15}\text{N}$  values of 10.0 ‰ for the 1916 bull and 10.2 ‰ for the 1880 weathered tusk, respectively.  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  in elephant ivory reflect the vegetation the elephant consumed, as well as the climate in which it grew, meaning these two isotope systems are indicative (although not definitive) of the habitat in which the elephant lived. The range of isotope values documented here is large enough to conclude that this ivory came from different environments and was not all from local elephant populations. Some ivory almost certainly did come from local elephants—samples with the most negative  $\delta^{13}\text{C}$  and low  $\delta^{15}\text{N}$  values derived from relatively moist environments with dense forest/bush vegetation, like that of the KwaZulu-Natal river valleys at the time. They may also have come from forests on higher ground, such as the Qudeni and Nkandla forests about 20–30 km north of Ndongondwane, and from dense coastal bush or forest.

Other ivory, particularly that with more positive  $\delta^{13}\text{C}$  and higher  $\delta^{15}\text{N}$  values, derived from more open grassy regions.

#### Strontium Isotopes

The four samples that were subjected to the leaching experiment included two from Ndongondwane (UCT 14431/KZN 38 and 14423/KZN 30), one from KwaGandaganda (UCT 17470/KZN 80) and one from Wosi (UCT 17509/KZN 119). None of the samples showed a consistent pattern of alteration in  $^{87}\text{Sr}/^{86}\text{Sr}$  through the leaching profile (see Online Resource 2). The first three samples showed only very slight variation in  $^{87}\text{Sr}/^{86}\text{Sr}$  through the profiles, with ranges from 0.00009 to 0.00042. KwaGandaganda (UCT 17470) yielded a more variable profile, with a range of 0.00174. Given the wide range of local variation in  $^{87}\text{Sr}/^{86}\text{Sr}$  mentioned above, differences in the fourth decimal place do not significantly affect the interpretation of the results presented here. It appears that the influence of diagenetic strontium is minimal. Sample

UCT 14423/KZN 30 from Ndongondwane was analysed by both laser and solution chemistry, yielding  $^{87}\text{Sr}/^{86}\text{Sr}$  of  $0.7126 \pm 0.0003$  and  $0.71289 \pm 0.00002$ , respectively. We therefore consider laser and solution measurements to be equivalent.

$^{87}\text{Sr}/^{86}\text{Sr}$  of five samples of ivory from KwaGandaganda range from 0.71498 to 0.71626, eight from Ndongondwane from 0.71255 to 0.71497 and the single value from Wosi is 0.71289. The range of values from Ndongondwane/Wosi falls within the ranges of both the modern *Achatina* shells and the archaeological cattle teeth from those sites analysed by Arnold et al. (2013). We note, however, that range of  $^{87}\text{Sr}/^{86}\text{Sr}$  from KwaGandaganda does not overlap with that from Ndongondwane and Wosi. This supports our statement above, based on light isotope analyses, that ivory brought to the three sites came from different catchments.

## Discussion

In Fig. 10, we compare  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  of archaeological ivory from KwaZulu-Natal with ivory from modern elephants from wildlife reserves in South Africa, Botswana and Zambia. Unfortunately, these include only a restricted range of habitats; in the past, elephants were much more common and widespread. Historical accounts describe an increase in elephant hunting for ivory exports when the first colonial settlement was established at Port Natal (now Durban) in 1824 (Carruthers et al. 2008). Records from the mid-nineteenth century describe elephants roaming near the Mfolozi River (Klingelhoefter 1987). We recognise that the restricted distribution of our modern reference ivory samples is a limitation, but we use them as best we can to characterise elephant habitats and help interpret the measurements of archaeological ivory.

The range of  $\delta^{13}\text{C}$  values in archaeological ivory analysed in this study is large ( $-22.6$  to  $-15.2$  ‰). It extends from values seen in densely forested  $\text{C}_3$  environments (such as Knysna) to much more open grassland savannas (such as Kruger National Park, Tembe Elephant Park). The range of  $\delta^{15}\text{N}$  values is also large. Our 39 ivory specimens from KwaGandaganda alone show a range of 7.4 ‰, compared with 6.5 ‰ for modern Kruger National Park elephants—the largest range from a single region in the modern data set. The Kruger Park is 19,485 km<sup>2</sup> in extent, and encompasses several different vegetation types and rainfall zones, from 400 to 700 mm per annum. Thus, while it is

currently not possible to reconstruct exactly where the ivory at Ndongondwane, KwaGandaganda and Wosi originated, we are confident that it was drawn from different elephant habitats. The most negative  $\delta^{13}\text{C}$  values identify elephants that lived in forested or bushy environments, with little grass. Some elephants from KwaGandaganda may have come from the densely vegetated coastal belt, which was probably moister than the environs of Wosi because of its proximity to the warm Indian Ocean (Whitelaw and Moon 1996, p. 70). Values towards the positive end of the range indicate that the animals consumed substantial quantities of  $\text{C}_4$  grasses. These likely included some drier areas, given that some of the  $\delta^{15}\text{N}$  values are similar to those seen in elephants from more arid environments such as Addo National Park, where average annual rainfall is less than 445 mm. It is important to note that the isotope values reflect diets averaged over several years, so the differences we see indicate substantial differences in elephant diet and environment. There may also be some chronological variation: Msuluzi phase ivory from KwaGandaganda and Wosi probably predates the wetter interval c. AD 750–1000 recorded at Lake Sibaya (Stager et al. 2013), while Ndongondwane phase material derives from this period.  $\delta^{13}\text{C}$  of the small number of Ndongondwane phase ivory fragments analysed from KwaGandaganda cluster towards the more negative end of the range recorded at that site.

We need to bear in mind that the number of samples analysed here is limited, but nevertheless, the fact that there are statistically significant differences in  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  at the three sites is intriguing. It may be that there was some partitioning of the landscape in terms of ivory sourcing even at this early stage. Wosi and Ndongondwane, for instance, are only 1.3 km apart, though they are separated by the Thukela River. The results suggest that the river possibly functioned as a political boundary and thus points to an organisation of ivory procurement based on political territories or chiefdoms. We cannot, unfortunately, draw any conclusions about shifts in the area of ivory catchment over time. Material from Square 25 at KwaGandaganda shows highly varied  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$ , clearly indicating ivory of heterogeneous origins even in the Msuluzi phase. Given the inter-site differences already mentioned, one would have to compare earlier and later assemblages from the same site to investigate possible shifts in catchment areas through time. We have insufficient data to do this.

Whether Early Iron Age people hunted elephants in more distant areas themselves or whether they obtained

ivory through trade, as part of a network that also involved other goods, is unknown. As mentioned above, all three sites preserve evidence of contact with other parts of southern Africa (copper, ostrich eggshell, marine shell), although Early Iron Age communities lived settled lives, sometimes (as at KwaGandaganda) continuing to maintain and rebuild villages in the same locality over several centuries (Whitelaw 1994). These sites may have been more involved than previously recognised in trade networks. The possible scope and extent of these networks are discussed further below. This raises interesting and important questions about the role that trade may have played in the development of larger-scale economic and political organisation.

If a well-developed system of ivory procurement and processing existed from the seventh century onwards, what was this ivory for? Was it for the local market, and/or was it being fed into more widespread distribution networks? We cannot answer this question definitively, but there are a number of lines of evidence that, in our opinion, suggest that both applied.

There is no doubt that in historical times in Africa, elephants—and by association, ivory—were imbued with powerful symbolic qualities. In west, east and central Africa, elephants were a potent origin symbol (Cosentino 1992). They were considered female in character, reflecting their matriarch-led herds. In this sense, elephants resemble origin pools, while tusks, seemingly analogous to reeds in origin pools (Whitelaw 2015, p. 14), can stand for a ruler's authority and virility. Elephants can take on human form, while humans can become elephants on death. In southern Africa, elephants can represent the authority and power of the Zulu king. His queen, like the Swazi queen mother, is called She Elephant. The Swazi king is Ngwenyama (Lion), but early on the main day of the big *incwala*, the annual Swazi ritual of kingship, he walks before his people wearing only an ivory penis cover. The people praise him: “You of the Elephant” and “Elephant of the Ngwane [i.e., of the Swazi]” (Kuper 1973, p. 622). Amongst other things, the performance presents the king as the fount of the life force of the nation. This connection between elephants and royalty seems an elaboration of a more general relationship because even among ordinary people, the spirit of a deceased homestead head can appear as an elephant (Raum 1973, p. 435). Indeed, Zulu ethnography indicates that elephants were believed to so resemble humans that young people avoided eating the flesh, for young women feared that they would give birth to one (Krige 1962, p. 388).

In historical times, elephant hunting for the ivory trade is well documented (e.g., Carruthers et al. 2008). Tusks moved upwards in hierarchical exchange systems that were genealogical in character (son > father > (chief) > ancestors) and in return came the means to produce and procreate (semen, rain, cattle) (Hunter 1936, p. 387; Bryant 1967, p. 686; Kuper 1982, p. 17). The association of elephants with power and procreation, and the widespread distribution of an essentially common elephant symbolism in Africa, hints that these beliefs are likely to be of considerable antiquity. If so, ivory may have been valued in Early Iron Age farming communities in KwaZulu-Natal in the same way as in later centuries. Perhaps there is a connection here with the apparent preference for elephant ivory, rather than that from hippopotami or other species, found in this study.

Some manufactured ivory items, such as the tooth-shaped object from KwaGandaganda shown in Fig. 4, were very likely intended for local use. This is an ivory version of an artefact type also found in other materials (pierced canine teeth, copies of such teeth in bone) at this site and at Ndongondwane. It therefore fits well with the overall material culture assemblages of the time.

Ivory bangles or armlets were the largest, most frequent and most standardised type of artefact manufactured from ivory, and they occur not only at the KwaZulu-Natal sites but also at later sites in the Limpopo Valley such as Schroda, Pont Drift and K2 (Hanisch 1980; Raath 2014; Voigt 1983), and sites in Botswana such as Mosu and Kaitshaa (Denbow et al. 2015; Reid and Segobye 2000). Much of the ivory working waste recovered (in very substantial quantities) from Ndongondwane, KwaGandaganda and Wosi likely relates to the shaping of these bangles. This point may be relevant to our identification of all the ivory in this study as elephant—the shapes and sizes of hippopotamus and warthog tusks are unsuitable for making bangles like these. Abundant working waste suggests craft specialisation at Ndongondwane, KwaGandaganda and Wosi, as other Early Iron Age sites in KwaZulu-Natal, by contrast, yield no evidence of ivory working and few, if any, ivory artefacts. At present, we do not know what role (if any) ivory armlets may have played in local society. None of the Early Iron Age burials from KwaZulu-Natal include bangles, although this picture may change with further finds. Further to the north and several centuries later, two children at K2 and a male skeleton from Happy Rest were interred wearing ivory bangles (Steyn and Nienaber 2000; Voigt and Plug 1984). Detailed consideration of the ivory from sites in the Limpopo River

Valley is beyond the scope of this paper. Nevertheless, we note that ivory from the tenth–twelfth century sites in the Limpopo region has long been assumed to be part of the range of goods exported in the Indian Ocean trade, either in raw or manufactured form (Forssman et al. 2014; Hall 1987; Huffman 2007; Maggs 1984a).

In the late first millennium AD, we see evidence for exotic goods being imported into KwaZulu-Natal. Some came from other parts of South Africa (ostrich eggshell beads, probably also copper items), but a few were traded across the Indian Ocean: glass beads found at KwaGandaganda (Whitelaw 1994) and Ntshekane (Maggs and Michael 1976) and the glazed ceramic sherd from KwaGandaganda (Miller and Whitelaw 1994). There are very few of these, but given that they must have been rarities when first obtained, that only a small proportion of objects find their way into the archaeological record, and that excavations sample only limited areas of archaeological sites, this is not surprising. We do not, as yet, have foreign imports from Msuluzi phase occupations, but that may well be a sampling issue. What did communities in KwaZulu-Natal offer in exchange? Ivory is one possibility.

We note that trading for ivory on the East African coast was well established by the early first millennium AD. *The Periplus of the Erythraean Sea*, probably written at Alexandria c. AD 100, mentioned large quantities of ivory obtained from the East African coast (Casson 1989). KwaZulu people, expanding southwards into Mozambique and South Africa in the first few centuries AD, may well have known about the ivory trade or even participated in it from their East African homeland.

In the ninth century, the Chinese writer Tuan Ch'eng-shih cites the East African coast as a source of ivory, ambergris and slaves. The early tenth-century traveller al-Mas'udi described ivory exported from East Africa to Oman and thence to India and China for making dagger handles, chess pieces and likely also bangles for Hindu brides (Freeman-Grenville 1962, pp. 14–16). Documentary evidence attests to this in the eighteenth century, as more than half of the ivory imported into India at that time was used for wedding bangles, as soft African savanna elephant ivory was valued over their own local ivory (Freeman-Grenville 1988, p. 11).

Trans-Indian Ocean voyaging may be far more ancient than any documentary records, if bananas had indeed reached Uganda by the fourth millennium BC (Lejju et al. 2006). A variety of other food crops were dispersed across the Indian Ocean in succeeding millennia, with African domesticates reaching India by at least 2000–1700 BC

(Boivin et al. 2013, 2014). This commerce relied on suitable winds and ocean currents, and it has long been thought that vessels relying on trade winds to cross the Indian Ocean probably did not make landfall on the African mainland further south than the northern end of the Mozambique Channel; winds and currents in this channel are challenging for sailors even today. Wood (2011) has shown that during the late first millennium AD, imports into south-eastern Africa included beads likely made east of the Euphrates River and traded through the Persian Gulf. Many of these imported beads have been found at the trading depot of Chibuene (near modern Vilanculos), close to the southern end of the Mozambique Channel (Fig. 1). This was a very active trading depot in the late first millennium (Sinclair et al. 2012; Wood et al. 2012); indeed, at that time, glass beads were imported in larger numbers into southern Africa than into eastern Africa (Wood 2011; Wood et al. 2016). The eighth- to mid-tenth century Zhizo series is the best known of these beads, but Wood recently identified a “Chibuene series” that possibly predates the Zhizo beads (although the low numbers of Chibuene beads might relate to colour and “feel” preferences, with people in the interior preferring Zhizo beads). Chibuene and Zhizo series beads were traded inland to Kaitshàa and as far as Nqoma in north-western Botswana (Fig. 1) (Denbow et al. 2015; Wood 2011; Wood et al. 2012), a distance of approximately 1,500 km as the crow flies (and almost 1.5 times the distance from KwaGandaganda to Chibuene). Zhizo series beads have been identified from a number of areas in southern Africa, including, in Zimbabwe, Makuru, Leopard's Kopje Main Kraal, Zhizo Hill, Matsheumhlope and Three-Mile-Water (Wood 2005, p. 41). Beads that may belong to the Zhizo series also occur in a number of other assemblages from Zimbabwe and Botswana. Since the earliest trade in beads overlaps with at least part of the Msuluzi phase, some KwaZulu-Natal ivory may have been destined for a trading post such as Chibuene, then shipped onwards to more distant markets.

## Conclusions

Stable carbon and nitrogen isotope analysis, in combination with species identification using ZooMS, shows that all of the ivory artefacts and fragments of worked ivory that we studied are elephant, rather than hippopotamus or warthog ivory. The elephants were drawn from

a wide catchment that included both heavily bushed/forested habitats and more open environments with abundant C<sub>4</sub> grasses. Inferences from strontium isotope analysis are imprecise, given high regional variation in <sup>87</sup>Sr/<sup>86</sup>Sr, but are consistent with ivory at KwaGandaganda coming from different areas from ivory at Ndongondwane and Wosi. The principal manufactured items were large bangles or armllets. Production of these appears to be confined to a limited number of settlements, where it continued over several hundred years. These sites may have been politically and economically more important than others.

The abundant evidence of ivory working at KwaGandaganda, Wosi and Ndongondwane dates from the seventh–tenth centuries AD, several hundred years earlier than the better-known evidence from the Limpopo River Valley. There, ivory is widely considered to be part of the goods exported through Indian Ocean trade. We suggest that at least some of the ivory from the KwaZulu-Natal sites may also have been destined for trans-oceanic trade based on (1) large quantities of ivory on some KwaZulu-Natal sites and (2) the new evidence reported here that ivory procurement was not merely local but was conducted over considerable distances, with a greater degree of organisation than previously suspected. Ivory export at this time would be consistent with the growing body of evidence for imported glass beads and other goods that increasingly indicate trade activity in the first millennium AD. KwaGandaganda, Wosi and Ndongondwane preserve the earliest evidence for large-scale ivory processing in southern Africa, the precursor of a larger, more expansive network of goods—including gold—that would flow through this region in the centuries to come.

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### Compliance with Ethical Standards

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**Conflict of Interest** The authors declare that they have no conflict of interest.

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