# HISTORICAL TECHNOLOGY, MATERIALS AND CONSERVATION

## SEM AND MICROANALYSIS



# Historical technology, materials and conservation : SEM and microanalysis

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# A study of pre-Columbian gold beads from Panama

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**ABSTRACT** Research into the fabrication of pre-Columbian gold beads from Panama was carried out as part of an ongoing study on pre-Columbian gold at the Smithsonian Institution. Roughly 2100 beads were included in the study, originating from the collections of the National Museum of the American Indian (NMAI), the National Museum of Natural History (NMNH), and from recent excavations in Panama. Technical examination and X-ray fluorescence (XRF) analysis were carried out in the first phase, which permitted the beads to be divided into compositional, formal and technological categories. The most common type, the rolled and joined sheet bead, was chosen for more in-depth study. Variable-pressure scanning electron microscopy with energy dispersive X-ray analysis (VP-SEM-EDX) was carried out on selected beads in order to identify the joining method used. No elemental variation was detected along the exterior surfaces or over cross-sections of the join regions, suggesting that solder was not used as a bonding method for this type of bead.

KEYWORDS SEM-EDX, metallography, gold, beads, Panama

#### Introduction

While the techniques of gold jewellery fabrication in Europe have been extensively studied, to date there has been relatively little research on the production of pre-Columbian gold ornaments, and more specifically, the manufacturing techniques of gold beads. Ongoing research at the Smithsonian Institution on the history of pre-Columbian gold working in Panama provided an excellent opportunity to undertake an in-depth study of bead fabrication. This study is the latest phase of a larger research project that began in 2007, when researchers from the Smithsonian's Museum Conservation Institute (MCI), in collaboration with archaeologists at the Smithsonian Tropical Research Institute (STRI), conducted a technological study of two large collections of pre-Columbian gold in Panama. The second phase, which provided the context for the current study, focused on examination and analysis of the Panamanian gold in two Smithsonian collections in Washington, DC, those of the National Museum of the American Indian (NMAI) and the National Museum of Natural History (NMNH), in addition to well-contextualised finds from recent excavations in Panama curated at STRI. Access to the recently excavated material for analysis was approved by the Patrimonio Histórico of Panama through partnership with archaeologists at STRI.



Figure 1 Map of Panama indicating the location of Sitio Conte in the Gran-Coclé archaeological region.

## Background

Metallurgy arrived in Panama in the first several centuries AD through contact with the cultures of Colombia and Ecuador,<sup>1</sup> [1, 2]. While methods for smelting and casting had originated in South America, spreading from Peru to neighbouring regions and then into Central America, there remained a great deal of diversity in technology and style among cultures occupying this vast area. Panamanian goldwork shares many traits with the goldwork from Colombia, and the close relationship with Costa Rican material has led some researchers to consider these Isthmian countries as part of one larger metalworking region [3, 4].

Gold and copper were the metals of primary importance in Panama and were often combined in an alloy known as *tumbaga*. Many pre-Columbian gold objects, however, are made from a natural alloy of gold, silver and copper, which results from the use of gold grains and nuggets from secondary alluvial deposits that commonly contain small quantities of copper and silver. Gold fabrication techniques known to the pre-Columbian inhabitants of Panama include sheet forming, annealing, repoussé, chasing, lost-wax casting (closed core and open-back casting), soldering and surface enrichment, also known as *mise en couleur* or depletion gilding [4, 5].

Sitio Conte, a funerary site excavated by Harvard and University of Pennsylvania archaeologists in the 1930s, is the most well-known site for gold finds in Panama. It is located in Gran-Coclé, the region considered by archaeologists to encompass the culturally related pre-Columbian societies of central Panama (Figure 1). The gold objects excavated at Sitio Conte, which reportedly date to between AD 750 and AD 950, have provided the clearest evidence of complex, ranked societies from that time period [1]. The recently excavated beads discussed in this paper are roughly contemporary to

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Fabrication	Туре	Number	%	Total%
Cast beads	Small circular beads	4	0.2	1.32
	Large tubular beads	21	1.03	
	Animal/anthropomorphic effigy beads	2	0.1	
Sheet beads	Coiled with no join	46	2.26	98.68
	Biconical	2	0.1	
	Sheathing (complex shapes)	151	7.41	
	Rolled and joined	1812	88.91	

Table 1 Categorisation of beads included in study by fabrication method and type.

those from Sitio Conte and are believed to have been produced by a culturally related group in Gran-Coclé.

Gold beads provide an excellent starting point for looking at specific fabrication technologies used by Panamanian goldsmiths as they are a common and widespread class of artefact in Panama. Beads also represent a range of forming technologies from hammered sheet to lost-wax casting, allowing the study of a variety of production methods within one category of artefact. The large quantity of beads that have been found in Panama to date also provides a considerable sample for analysis.

Despite their prevalence, the references to Panamanian gold beads in the literature are rare and often contradictory. According to Emmerich, the necklaces excavated at Sitio Conte were composed of hundreds of identical cast gold beads, many of which were 'cast over inner cores' [6]. Conversely, the archaeologist at Sitio Conte, Samuel Lothrop, described the majority of beads as 'hammered or pressed' to shape over a perishable substrate [5]. While Stone and Balser also mention hammered beads among those that they examined, they suggest that the majority was made up of different types of cast gold beads, including cast tubes cut at regular intervals to produce identical smaller beads [4]. None of these assertions, however, is supported by analytical investigation.

#### Methodology

Examinations and analyses were carried out on the pre-Columbian gold beads from Panama in the two Smithsonian museum collections and from recent excavations in order to identify the fabrication techniques used in their manufacture. The study was divided into two phases: (1) preliminary investigation to determine the final sample set, and (2) analysis of the selected objects. Phase 1 included technical examination and X-ray fluorescence (XRF) analysis, which allowed the identification of different categories of beads by fabrication type, shape and features. Using this information, representative beads were selected from the most common categories for further analysis in Phase 2. In this second phase the selected beads underwent imaging and microanalysis using variable-pressure scanning electron microscopy with energy dispersive X-ray analysis (VP-SEM-EDX).

#### Preliminary investigation to select sample set

Roughly 2100 beads were included in the first phase of the study, comprising over 1700 beads at the National Museum of the American Indian, 60 beads at the National Museum of Natural History, and 231 beads from recent excavations in

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Panama. Of the recently excavated beads, 223 originated from the site of El Caño in the Gran-Coclé region. In cases where multiple beads were catalogued as a single item, such as the strung necklaces and bracelets, several representative beads were selected for examination and XRF analysis.

A total of 113 beads was analysed by calibrated XRF, using a portable bench-top ElvaX X-ray fluorescence analyser. Analyses were conducted at 45 kV for 100 seconds of live time in heavy element mode, which allows detection of elements from chlorine (Cl, atomic number 17) to uranium (U, atomic number 92). A set of approximately 35 matrix-matched gold reference standards (Royal Canadian Mint BCR 8079) were analysed by Jeff Speakman at MCI to create a material-specific calibration in the ElvaX software, reporting results in wt%. A selection of these standards was also analysed periodically during testing to adjust the calibration and monitor the instrumental precision and accuracy. The limits of detection for each element vary somewhat according to the matrix in which they are present. For copper and silver in a gold matrix, they are approximately 0.1-0.2% and c.0.25% respectively and for gold in a copper matrix, c.0.25-0.5%.

Using information gathered during technical examination, the beads were divided by fabrication technique into two main categories: cast and sheet fabrication (Table 1). Based on the presence of several visible casting features, such as dendrites, casting flaws and porous surface texture, only 27 beads were identified as cast, which represented 1.3% of the total beads examined. The cast beads ranged from large tubular beads to animal effigy beads and small plain circular beads. The beads fabricated from sheet were found to be far more numerous at just over 2000. Common features identified on these beads included flanges and burrs at edges, chisel lines around bead openings, rough texture or unidirectional striations on the interior surface, and crimped and creased edges. These beads were either coiled with no joined edge or alternatively joined with a bonded overlap (Figure 2). The beads with a bonded join were by far the most common type at 88.9% of all beads examined. While many of the beads examined were identified as rolled and joined sheet, a join line was visible on only a fraction of these beads. Finally, 151 of the sheet beads were considered to fall under the sub-category of sheathing, i.e. thin hammered sheet that had been formed by burnishing and crimping over organic cores in complex shapes such as shells and teeth.

Based on the information provided by the preliminary investigation, the sample set for further analysis was narrowed down to the most common category of bead – those formed from rolled and joined sheet. By focusing on the most frequently found bead type, it might be possible to characterise

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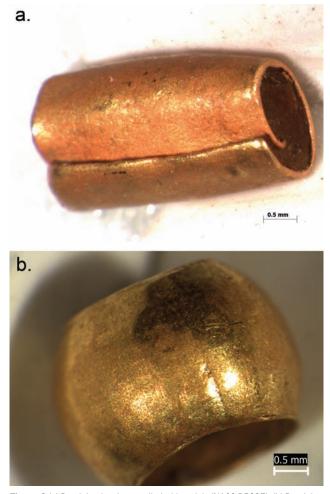


Figure 2 (a) Bead that has been coiled with no join (NA20 R5387). (b) Bead that has been rolled and joined (PAPG H7).

a large category of artefact from Panama through analysis of a small representative sample. Similarly, as the most numerous type of bead from recent excavations, the rolled and joined sheet beads had greater potential for the approval of sampling requests. This particular category of bead also raised several interesting research questions including the types of joining techniques used to form the bonded overlap.

#### Joining methods

The identification of methods used to bond the overlap of rolled sheet gold alloy beads is fundamental to our understanding of fabrication. The procedures for joining metals considered in this study include hot-working processes such as fusing, soldering, the formation of eutectic systems and sintering, and mechanical, cold-working methods such as crimping and burnishing.

Fusing is a metal bonding process using heat to unite two homogeneous metals without the addition of other materials. Adjacent surfaces are heated until they become fluid and diffuse into each other. This process takes place at the liquidus or melting temperature of the metal. Heat is rapidly transmitted through gold, silver and copper, owing to their superior conducting properties, so the possibility of melting the entire metal piece during the fusing process is greatly increased [7]. Although lack of variation in the metallic bond may make detection difficult, telltale signs of a roughened surface or wavy edges can be reasonable visual indicators of a fused seam.

Soldering is a method of joining metals by using heat to flow an alloy of a lower melting temperature metal between the two surfaces of a metal with a higher melting temperature. For our purposes, the solder would likely be formulated of gold, copper and silver in a ratio that reduces the melting temperature below that of the original alloy sufficient to flow the solder without melting the object. The solder required to achieve this disparity in melting temperatures may not be visible under magnification. However the compositional variation between the solder and original alloy may be measured by a variety of analytical techniques including SEM-EDX [8–10].

Another method of joining gold elements is the use of a eutectic system to form a bond. Eutectics are also referred to as proto-brazing [11], reaction-soldering [12] or – when attaching small granules of metal – granulation [13]. Gold alloys with a low percentage of copper will form a eutectic when copper salts in an organic adhesive are applied between the surfaces to be joined, followed by heating. Carbonisation of the adhesive reduces the salt to metallic copper, which forms an alloy with the original metal. The localised reduction in melting temperature results in bonding at the points of contact. The slight change in composition of the eutectic system has been identified in several previous studies using both surface analysis with SEM-EDX and analysis of cross-sections [14, 15].

A bond can be formed using powdered metal of the same composition as the bulk metal, which is packed into the seam with an organic adhesive. This process is called sintering. When the metal powder is heated to 65–80% of its solidus temperature the grains coalesce, closing the seam. Sintering may be distinguished by the granular appearance at the surface of the join; however, metallography highlighting the dendritic structure of the metal in the seam is a more reliable indicator [12].

Finally, cold-working approaches for joining gold beads include crimping and burnishing. For our purposes, crimping is the process of pushing one metal surface into another, thereby deforming one or both surfaces to form a join. Depending on the force applied, the metal surfaces may come into close contact along most of the seam. Crimped seams are more likely to be visible to the naked eye and will be obvious under magnification. Regions of work hardening may be visible in cross-sectional analysis.

Burnishing extends this process, bringing the two metal surfaces into intimate contact by applying sufficient force to the front of an object while the back is supported against a rigid surface. The metal surface is burnished or rubbed with a hard, rigid material, such as a polished stone, and the combination of pressure and friction compacts the two surfaces together. Burnished seams may not be visible to the naked eye although they may be visible under magnification, and regions of work hardening may be visible in cross-sectional analysis.

#### Beads selected for further analysis

To better understand the joining technology, more nuanced compositional information was required. SEM-EDX analysis of whole and sectioned beads was determined to be the best

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method available at MCI for this second phase of the study. As none of the Smithsonian museum material was permitted to undergo destructive sampling, 29 beads from the NMAI and NMNH collections (15 and 14, respectively) were selected to undergo SEM-EDX of the whole beads only. These 29 beads represent eight catalogued objects such as necklaces and bracelets, each with between 1 and 550 beads. Each of the selected beads exhibited obvious overlaps or visible seams and were clearly fabricated from sheet.

A representative sample of eight rolled and joined beads was selected from the 223 beads excavated at El Caño, for which export and analysis permission was granted. Four of these underwent further analysis at MCI, the results of which are featured in this paper. The four beads, R4853, R5101.2, R5341 and R5352, were all manufactured from hammered sheet with an overlap or seam visible on either the interior, exterior, or on both sides. The bead diameters measured between 2.15 mm and 4.05 mm and the lengths measured between 2.15 mm and 2.8 mm. While three of the beads are round in cross-section and in profile, one of the selected beads, R5352, is round in cross-section but tubular in profile. Bevels, burrs, chisel marks or a combination of these features was noted on the bead openings, or apertures. Three of the four beads also had evidence of abrasion in the form of a rough texture on the interior surfaces. XRF measurements indicated that all were high in gold, ranging from c.81 to c.93 wt%. Copper content was c.1-2 wt% while silver content ranged from c.5 to c.19 wt% (Table 2).

# Analysis of selected beads: SEM-EDX and metallography

Prior to analysis, the selected El Caño beads were cleaned in an ultrasonic bath; cleaning was not permitted for the NMAI and NMNH beads. SEM-EDX was carried out first on the whole beads, and then subsequently on the mounted and sectioned beads from El Caño, using a Hitachi S-3700N VP-SEM with Bruker EDX. All beads were analysed at 15 kV

Table 2 XRF data of El Caño beads selected for further analysis.

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Object	Au (%)	Ag (%)	Cu (%)
R5352.2	92.9	5.4	1.7
R4853	90.9	8.2	0.8
R5101.2	87.5	6.7	1.2
R5341	80.5	18.7	0.8

at approximately 10–15 mm working distance. Analysis of the whole beads was carried out under full vacuum while the sectioned beads were analysed in VP mode. Elemental mapping was conducted over the surface of the joins in order to determine whether any compositional variation from joining techniques could be detected. Elemental maps were acquired in the Hypermap mode of the Quantax software for approximately 15 minutes.

#### Surface analysis of whole beads

Elemental mapping of gold, copper and silver concentrations showed homogeneous composition over the exterior surface of visible joins on all beads that underwent SEM-EDX. The elemental maps of bead A396672 (Figure 3) in the NMNH collection are typical in that they reveal no compositional variation across the join region. Comparison of the elemental count rates in areas across and adjacent to the join quantitatively confirmed the uniformity of composition. While no solder or eutectic bonding was detected in this initial step, no conclusions could be reached regarding the presence of these materials inside the seam as only the surface composition of these beads was measured. Therefore, variation in composition at the interior of the join required further investigation. Although the Smithsonian beads could not undergo destructive sampling, confirmation of the initial results was possible through cross-sectioning of the representative beads from El Caño.

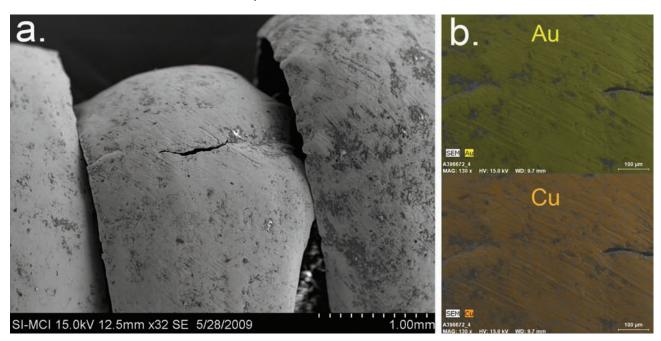


Figure 3 (a) SEM image of bead A396672 (NMNH). (b) Elemental maps of Au and Cu in the joint.

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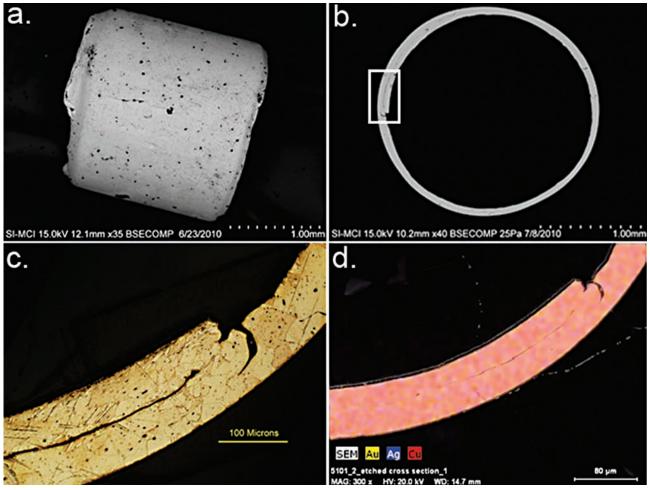


Figure 4 (a) SEM-BSE image of bead R5101.2. (b) SEM-BSE image of bead R5101.2 in cross-section. (c) Cross-section of R5101.2 etched in aqua regia. (d) Elemental map of Cu, Ag, Au concentrations in the join region of R5101.2.

#### Cross-sectional analysis

The four recently excavated beads (R4853, R5101.2, R5341 and R5352) were mounted in epoxy and polished down to reveal the cross-section along one edge. Bead number R5341 was also sectioned longitudinally with a low speed diamond saw to reveal the profile of the bead in addition to the remaining half of the cross-section. All of the samples were polished with a series of increasingly finer grit grinding papers up to 600 grit. The final polish was achieved on a lap wheel with 0.05 Alpha Alumina and water on a polishing cloth. The polished cross-sections were examined under the metallographic microscope, photographed and analysed by VP-SEM-EDX. After documentation and analysis of the un-etched samples, the cross-sections were etched in aqua regia and re-examined under the metallographic microscope, followed by carbon coating and analysis once again by VP-SEM-EDX.

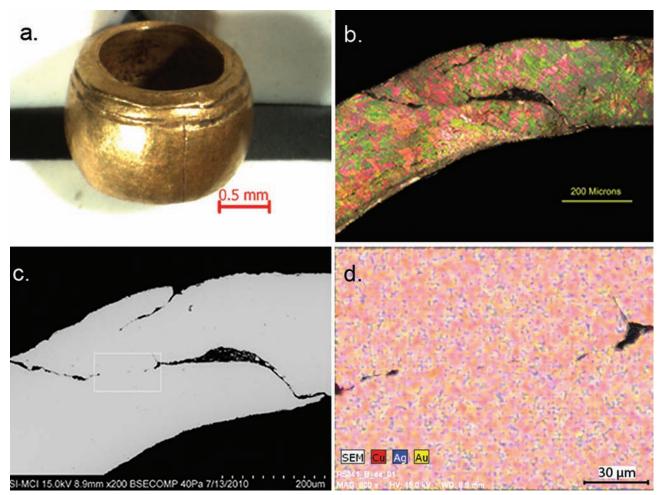
### **Results and discussion**

#### R5101.2

SEM-EDX analysis of bead number R5101.2 was first undertaken on the whole bead to measure the composition across the surface of the join. This small tubular bead, which measures just 2 mm long and 2 mm in diameter, has a join visible on both the exterior and interior surfaces, the overlap of which measures approximately 1.5 mm (Figures 4a and 4b). The inner surface of the bead is rough in texture while the exterior is polished with chisel marks present around the circumference of the bead aperture. An irregular flange is also visible on the interior of the bead openings. Elemental mapping at the surface of the join at  $\times 160$  revealed no variation in gold, copper and silver composition.

Examination of the cross-section with the metallographic microscope after etching in aqua regia revealed an irregularly shaped grain pattern from cold working (Figure 4c). The straight twinned grains throughout indicate that the sheet was annealed after working. Some of the twin lines at the join are slightly curved, however, suggesting additional localised deformation after the final annealing. The zone directly between the two layers at the very end of the overlap is tightly aligned across the grain boundary and a crack is present in the metal sheet adjacent to the end of the overlap, possibly having widened due to working at the join.

No variations in microstructure or colour were noted across the join region under the metallographic microscope, suggesting no addition of solder or other material. These initial findings were confirmed using EDX analysis. Elemental mapping of the overlapping region showed no compositional variation across the join other than that related to the variation in the microstructure of the metal itself (Figure 4d). The combined evidence from metallography and SEM-EDX suggests that the bead was joined without any solder or use of any other additive material. The chisel marks around the bead apertures indicate that the bead was cut from a longer tube.



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Figure 5 (a) Bead R5341. (b) Cross-section of R5341 etched in aqua regia. (c) SEM-BSE image of cross-section of the join region. (d) Elemental map of Cu, Ag, Au concentrations in the join region of R5341

#### R5341

Bead R5341 is fairly similar in shape to many of the other beads from El Caño; however, analysis by XRF revealed an unusually high percentage of silver, at 19 wt% with 80 wt% gold and approximately 1 wt% copper. The bead is round in cross-section and profile with an overlap visible on the interior and exterior of the bead (Figure 5a). The edges of the apertures are bevelled and a double chisel line is present around the circumference of one side.

As in the case of the NMAI and NMNH beads, SEM-EDX analysis across the exterior join revealed no anomalous variation in composition. Metallography exposed a distinct seam with no solder present but a small area where the grain boundaries appear tightly aligned across the join (Figure 5b). Elemental mapping using SEM-EDX provided further evidence of the homogeneity across the join region (Figure 5c). Count rates in areas at and adjacent to the join were also checked in the Quantax software to quantitatively confirm the visual interpretation of the elemental maps (Figure 5d).

#### R5352.1 and R4853

Analysis of the remaining two beads from El Caño produced very similar results to those already described. The sectioned beads revealed an overlapping join with no solder or compositional variation along the seam. The join regions were not found to be fused; however, the surfaces on either side were very closely aligned.

#### Discussion

Based on the results of our investigation, all of the beads from El Caño appear to have been fabricated using the same manufacturing technique: they were made from sheet that was formed into a tube with overlapping edges, joined, and then cut at regular intervals. The lack of compositional variation along the seams indicates that solder was not used as the joining method. Sintering with powdered gold alloy would leave no appreciable compositional variation but was ruled out as the characteristic microstructural features of this method were not present in our samples.

Eutectic bonding is also considered unlikely as we would anticipate a distinguishable difference in elemental distribution at the join. The compositional variation resulting from eutectic bonding has been identified through analysis in other studies; however, it should be noted that there are additional steps in fabrication that could prevent detection of this joining method. The diffusion mechanism that initially allowed the formation of a eutectic may continue with prolonged or repeated heating, causing continued diffusion of copper into the object [16]. The increased homogeneity in composition resulting from this mechanism could thus complicate detection of the eutectic zone by SEM-EDX analysis.

While burnishing of the seam over a substrate is considered a plausible joining method, there is some question as to whether this method alone can achieve a secure bond. Replication experiments in this area have had varied results suggesting that the specifics of the replication procedure probably play a crucial role. During experimental reproduction of burnished joins on sheet gold, Armbruster [16] was not able to produce a lasting join while Harling's experiments proved successful [17].Further research involving experimental reproduction with all of these joining techniques for comparison with the archaeological beads will be necessary in order to determine the exact method of manufacture.

#### Conclusions

Initial results from examination and analysis of the Panamanian gold beads in the Smithsonian collections and from recent excavations in Panama suggest that the majority of these beads were manufactured from hammered sheet, formed into a tube with overlapping ends, joined along the seam and cut at regular intervals. While XRF and microscopic examination were effective methods for the categorisation of beads by type and composition, cross-sectional analysis coupled with SEM-EDX was essential in the investigation of joining methods. Although the beads in the NMAI and NMNH collections did not undergo destructive sampling, crosssectional analysis of four representative beads recently excavated in Panama provided sufficient evidence to support our initial hypotheses regarding the fabrication of this category of bead. While results to date suggest that neither solder nor sintering methods were used to effect joins, experimental reproduction of beads using variations on the hypothesised method will be the next step in this research in order to fully work out the sequence, techniques and tools used in the manufacture of these beads.

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#### Note

 The earliest known metal artefacts excavated in Panama, including both copper and gold alloys, were discovered at Cerro Juan Díaz in a grave with a calibrated radiocarbon date of AD 130–370. R. Cooke, Smithsonian Tropical Research Institute, personal communication (May 2009).

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