Experimental Replication of a Granulated Gold Bead from an Ancient Tomb at Bat, Oman

David Loepp and Alexander Maass

Keywords
Archaeometallurgy, autogenous welding, experimental archaeology, goldsmith, reaction solder

Abstract

During excavation of a tomb at Bat, Oman, an unusual gold bead entirely composed of 96 granules was discovered. The bead comes from an unstratified context in the bottom of the fill of the tomb, which means that it could date anywhere during the use-life of the tomb between the 3rd and 1st Millennium BC. The technical problems surrounding the manufacture of this bead are discussed and an experimental approach is used to explore a possible method of fabrication. The results of the experiments show that a combination of autogenous welding and brazing with copper salts to construct and join the individual units of the bead was successful to produce a visual replica of the bead. The method of bead construction based on the serial production of units and sub-units also has implications on the practice and organization of goldsmithing during this era.

Introduction

One of the richest periods in Oman's prehistory is the early Bronze Age with the so-called Umm-an-Nar culture, which started in the first half of the 3rd millennium BC and lasted until circa 2000 BC. Oman had an important position on the trading route between the advanced civilization of Harappa in the Indus Valley and Mesopotamia with the cities of Ur, Babylon and Akkad, and Oman was itself rich in raw mineral materials and may have supplied these civilizations with products such as copper and pigments (Begemann, et al., 2010; Hauptmann, et al., 2016; Weisgerber, 1981).Particularly the trade of Omani copper was essential for the ancient high cultures of Mesopotamia, who describe Oman as Magan and the land of copper in cuneiform clay tablets of the 3rd millennium BC.

The economic situation in Oman at the beginning of the 3rd millennium gave rise to a society which created remarkable architecture and artwork. Beside the great towers in the settlements with a diameter of 30 m, the "cairn graves" are some of the most impressive buildings of this period in Oman's prehistory. These "cairn grave" tomb structures are often clustered in small groups forming necropoli. They were collective tombs where possibly a single family or family lineage was interred. At the end of the 1970s many of these tombs were destroyed due to the robbing of stones for house construction.

In Bat, in the north of Oman, in the district of Ibri there is such a necropolis, which is a UNESCO listed World Heritage site. In 2006 Gerd Weisgerber, one of the first archaeologists to start investigations in Oman, created a project to reconstruct completely or partially some of the destroyed tombs to enrich and enhance the site. One of the tombs chosen for the reconstruction was tomb 154, where Klara Frifelt already performed small-scale archaeological excavations in the 1950s. The reconstruction project was financed by the Oman government and was led by Manfred Böhme. Before the work on the reconstruction of tomb 154 could begin, the 1.20 m thick fill of the collapsed tomb had to be excavated. The excavation of the tomb in the years 2007 and 2008 was financed by the Deutsch-Omanische Gesellschaft.

The excavation brought to light some interesting details about the tomb architecture and burial ceremonies connected with the tomb. The objects in the fill indicate that the tomb was used from the Early Bronze Age until the Iron Age, even after the collapsing of the roof of the tomb. The objects were irregularly mixed in the fill because of animal burrowing and the continual reuse of the tomb, and this made a precise dating of non-diagnostic objects difficult; however, during the excavation many significant and spectacular objects were found. Numerous pottery fragments, which partially showed

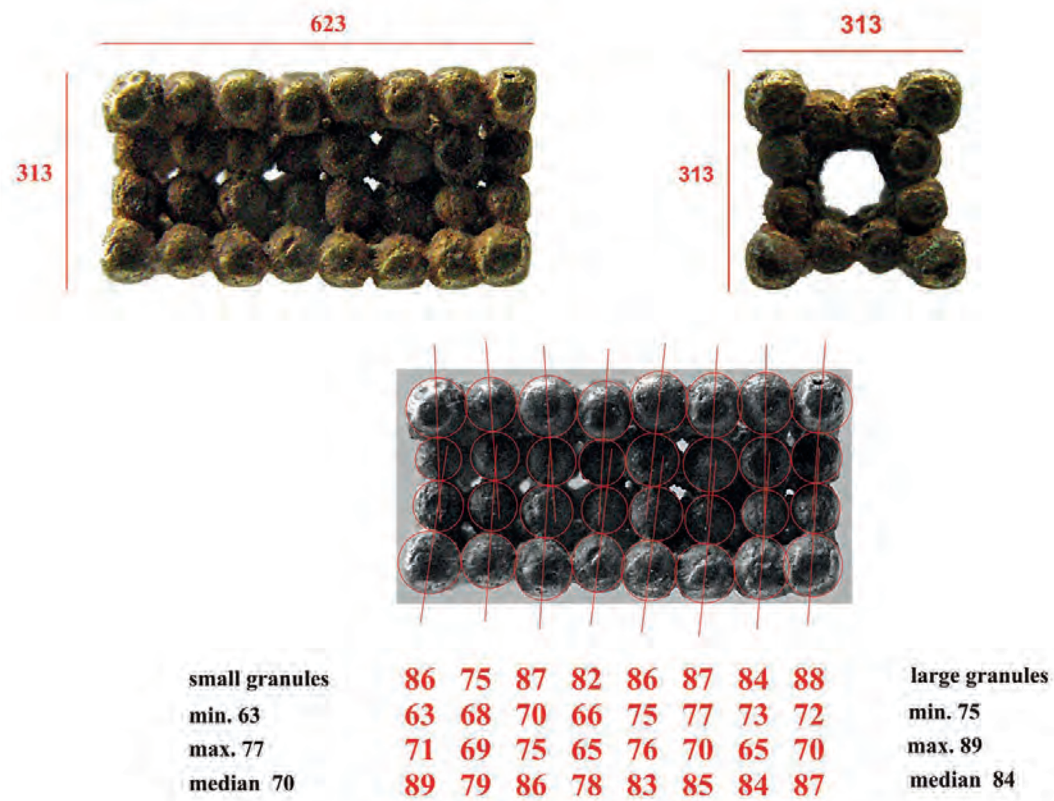

Figure 1: The bead from Bat composed completely of granules. To begin with the reconstruction, a practical model has to be made. The varying degrees of alignment among the granule planes suggest that the planes were constructed of four three-granule triangular units joined together. Measurements were taken from the optical photograph, though no substitute for precise measurements, must suffice in this case. The measurements are in hundredths of a millimeter, and since this is an optical image, there is an error margin of under ca. 5 hundredths of a millimeter. Photos / Illustrations: A. Maass and D. Loepp.

connections to India and Iran, fragments of steatite vessels, arrow heads from EBA until Iron Age made of copper or iron, a short sword made of copper from the Wadi Suq Culture, a razor made of copper and a cylindrical seal were found. Beside these, inside of the tomb around 2000 beads were discovered. Most of them were of carnelian or agate probably from India, but also many of them were made of bone, shell, local stones, steatite vessel fragments, a few of rock crystal or amethyst. But there were also some beads of copper, silver and 30 beads of gold. The number of beads increased with the depth. In the deepest layer of the tomb, one extraordinary and currently unknown type of gold bead was found. Even though it was found just above the floor surface, nothing can be said about its precise age, which could be anywhere between the middle of the 3rd Millennium and the 1st Millennium BC during the use-life of the tomb.

The Bead

This unique gold bead, approximately 6 mm long and 3 mm wide, consists of eight identical planes stacked to form a square-section, tubular system. Each plane is composed of eight spheres packed on a regular truncated square grid with four larger spheres placed on the corner vertices, creating a square-like lattice. The artifact therefore consists of 64 identical inner spheres and 32 larger external spheres. The small granules are circa 0.7 mm diameter, the larger circa 0.85 mm (Figure 1).

There are few published examples of technically related granulated structures. Allegedly, the oldest known granulated artifacts a small ring made of six granules thought to come from the tomb of Queen Pu-abi from Ur (Maxwell-Hyslop, 1977; Ogden, 1982, p.62); a reproduction of the bead by the first author using autogenous welding can be seen in Figure 2a. The archaeological documentation of this bead is however lacking in Wolley's excavation report but appears in Plenderleith without a catalogue number (1934, p.297). No comparable example has been found from the 3rd Millennium BC, and thus it is likely that the bead came from a younger period (Born, et al., 2009, p.20).

There are several beads of unknown provenance from the Henry Anavian collection that are similar, dated Iran, 1000 BC (Dublin, 1987, p.52, Figure 40) (Figure 2b). Maxwell-Hyslop (1971) discusses earring pendants from Hasanlu (p.189, plate 135) and Marlik (p.190, Plate 137

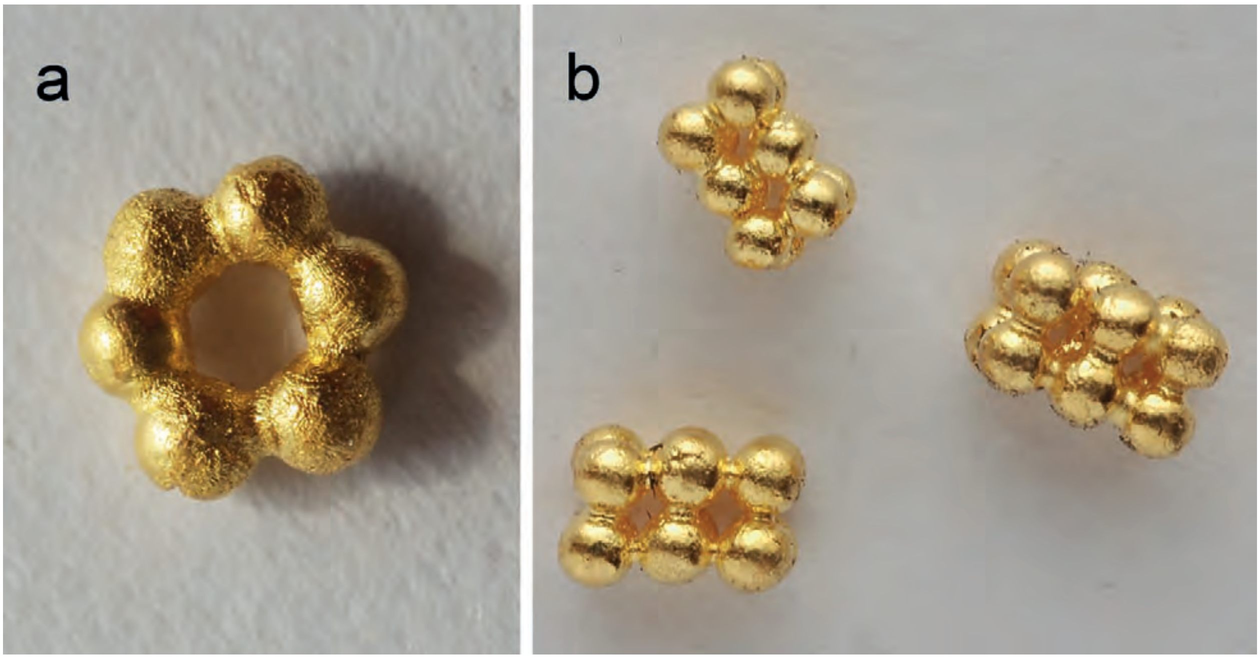

Figure 2: a) Modern replication of the hexagonal bead composed of six granules attributed to Queen Pu'abi's tomb from Ur (the context and date of this object are now controversial). The bead is 2 mm diameter like the original. Compare Maxwell-Hyslop 1977, Plate Ia. b) Modern replicas of Iranian granulated beads (c. 1000 BC) from the Henry Anavian collection. Compare Dublin, 1987, p.52, Figure 40. The beads were produced by the first author and joining was achieved through autogenous welding. Photo: D. Loepp.

and p.195, Plate 147), dated from the 12th to 8th century BC, constructed with triangular or pyramidal elements to form, respectively, larger triangles or pyramids. Wolters (1986) also illustrates a similar pyramidal structure (p.181, plate 200). It appears that the same technical strategies were used to construct these artifacts. One may note however that the spheres used on these later artifacts are somewhat larger, more qualified as shot rather than granule. A further difference to note on the earring pendants from Hasanlu is that, at the least, the upper part is made with hollow spheres fabricated from sheet rather than solid spheres.

Due to the rarity of this type of granulation there is no present agreement on terminology. There are a number of descriptive categories that have been proposed: granulation à jour, lattice granulation, openwork granulation (Shalem, 2002), Traubengranulation (Wolters, 1986, p.18-19). None of these terms clearly dissociates structures that are fabricated exclusively with granules from openwork granulated patterns that are soldered to a substrate. David Huycke has proposed "structural granulation" (structurele granulatie) and "constructive granulation" (constructieve granulatie) (Huycke, 2010, p.17): "In constructive granulation the granules are, however, essential to the structure of the work, they are the necessary building blocks of (part of) the object or piece of jewelry."

Technical description

Pending analyses, the following considerations can be adduced from the photographic evidence in light of the author's practical knowledge. The bead was likely made in a gold alloy containing silver with the component granules soldered together by a copper-salt based composition (Ferro, et al., 2008, Parrini, Formigli and Mello, 1982). Autogenous welding, that is the welding of elements together by heat alone, is tentatively excluded for the entire object due to the difficulty of repeated welds at temperatures within a few degrees of the liquidus phase. Binary Ag-Au alloys weld above the liquidus temperature of pure silver, 962°C. Copper-salt brazing, instead, initiates at circa 780°C and completes at a range of 100°- 60° C lower than the liquidus phase of the ternary alloy, thus allowing a safety zone for repetitive brazing.[1] Autogenous welding could, however, have been used advantageously to braze the initial units.

When the technological discovery of copper-salt brazing occurred remains an open question (Eluère, 1990, p. 200-207; Ogden, 1982, p.58-70). Analyses of the Ur bead and a granulated strip by Nigel Seeley (Maxwell-Hyslop, 1977) are not totally conclusive. While the bead has no traces of copper and is interpreted to have been made using autogenous welding (Ogden, 1982, p.62), the granulated strip does have values on the order of 5% Cu, likely a deliberate addition. The former has well-defined menisci while the latter is flooded, an indication of poor execution. The Ur tombs do contain numerous pigments that could potentially have been used in cross-over technologies, in which a product or technique may be valued for different uses. Copper acetate putties (see Hauptmann, et al., 2016; Woolley, 1934) may have been cosmetics pigments or may have served a medical function, but this material could have also been used as a copper-based brazing paste.

Replicating the bead

A valuable method for understanding an artifact and its context, and gather insights into the problem-solving process of ancient fabrication, is to replicate it with tools that likely existed at the time of its construction. In absence of any analytical element concerning the original artifact, one must rely on knowledge of ancient technique and choose those methods that best replicate the aesthetic and technical aspects of the object.

Initial attempts to reproduce granulation and related techniques in an academic context can be traced to the Castellani laboratories as reported by Alessandro Castellani to the Academie des Inscriptions e Belles-Lettres in Paris, 1860. Archaeological experiments on granulation and soldering were first conducted with modern analytical protocols by Parrini, Mello and Formigli. Edilberto Formigli continued both as a goldsmith and researcher restorer to replicate ancient techniques combined with micro-analytical methods, often in collaboration (Formigli, 1985; Formigli, 1995; Nestler and Formigli, 1993; Pacini, 2006). Robert Baines adopted a similar protocol in the study and replication of ancient artifacts (Baines, 1993a; Baines, 1993b; Baines, 2005). Experimental archaeology coupled with modern analytical methods has been conducted by Daniela Ferro at the Institute of Nanostructured Materials (ISMN-CNR) Italy (Ferro, et al., 2008).

Based on the experience of previous studies, it is possible to observe indicative details concerning the plausible techniques of fabrication: the granules are homogenous for dimension and roundness while the menisci partially cover the curve of most of the granules, masking their roundness. The formation of this aspect may be accomplished by accurate, prolonged heating by autogenous welding, or by brazing with a lower temperature alloy. The use of copper salts, instead, produces very fine menisci if adeptly managed as Alessandro Castellani said, "*saldato sine alia materia*." None of these solutions appear satisfying for a variety of reasons. It is very rare to find artifacts made of round, uniformly sized granules before the 7th century BC. It is possible that certain gold artifacts were joined by autogenous welding or brazing with an alloy in earlier periods. However, autogenous welding is a very high-risk technique for an object made entirely of granules.[2] Brazing with metallic solder in this specific case poses numerous problems, such as the placing of very minute quantities of metallic solder on each tangential point as the integrity of the joins suggests. In light of these considerations a first cycle of trials was undertaken as here reported.

In the case of granulation very few instruments are necessary. Preliminary production of a stock of granules requires a hammer and a flat hard surface to produce thin sheet, and a sharp instrument to cut snippets from the sheet or strip wire fabricated with the sheet. The actual construction of the artifact requires no more than small sticks or twigs and feather brushes to apply brazing mediums to the joins. Heating to brazing temperature may have been performed in a furnace or with an oil lamp with the aid of a blowpipe.

Preliminary operations - stock production

In order to produce a consistent number of beads, a large stock of granules is necessary. The most plausible method for the fusion of the granules is to charge a small coarse ware vessel with alternating layers of fine charcoal powder and gold snippets, and heat the vessel to circa 1100-1200°C in a furnace with a forced draft.[3] This method can reduce randomness by the targeting of a specific range of sizes correlated to the standard size of the bulk of the snippets. Due to random and unavoidable contact within the charge, granule sizes is distributed on a normal probability curve.[4]An alternative method for granule production by forming each granule singularly on a block of charcoal would have been time consuming and probably was not practiced save for small quantities.

It follows that, regardless the targeting of a specific size range by producing quasi-calibrated snippets, the majority of the granules within a batch will not be used for a specific project. It is noteworthy however that in this specific case the larger spheres are approximately twice the volume of the smaller spheres. This suggests that the larger spheres could also have been fabricated by fusing two smaller spheres together, if necessary. This presupposes, nevertheless, that the artisan had a considerable stock of identical spheres on the order of 0.7 mm diameter.

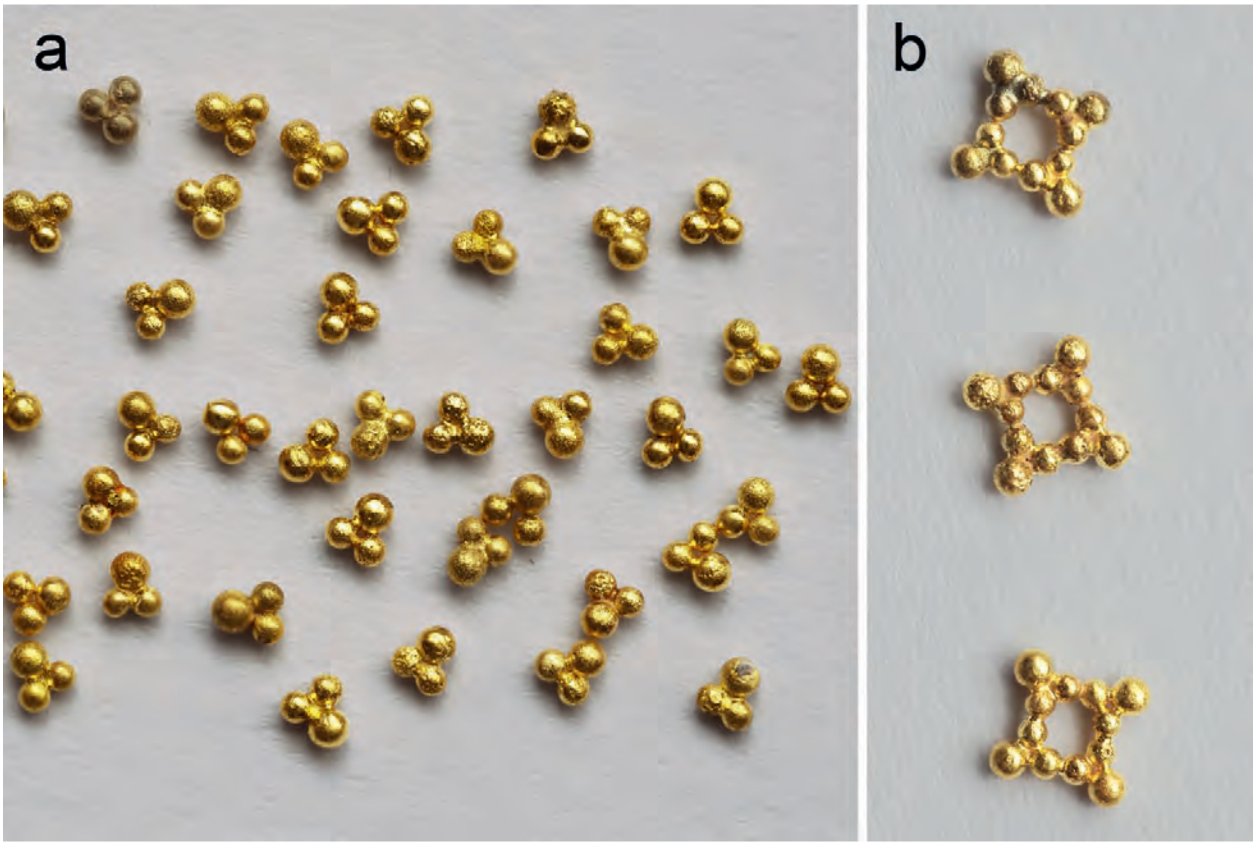

Figure 3: a) Triangular units consisting of one large granule and two small granules joined by autogenous welding. b) Plane units consisting of four triangular units joined by brazing with copper salts. Photo: D. Loepp.

Fabricating the planes

Two small granules and one large granule are welded to form an isosceles triangular configuration. It suffices to wet the surfaces of the granules with water to draw and hold them together in what is called a hexagonal close-packed configuration. A diluted organic binder may be used while copper salts are not necessary at this stage to braze the element. Direct heat is sufficient to weld each unit (Figure 3a).

The basic unit is the isosceles triangle composed of three spheres arranged in a triangle with a larger sphere at the vertex. Once a sufficient number of identical triangular units are prepared, groups of four, identical as possible, are then arranged in a regular octagonal configuration with the four large granules at the corner vertices (Figure 3b). This configuration is fairly unstable, as elements tend to maximize boundary contact. Granulated patterns require deliberate arrangement and fixing by the artisan and risk collapsing into hexagonal close packed configurations both during the application of the copper salts on spheres and subsequent brazing, all the more so when there is no substrate (Figure 4). In order to reduce unwanted displacements before and during firing, an organic binder, at

Figure 4: Application of copper salts between the triangular units to form the planes. Photo: A. Celauro.

this point containing copper salts, helps fix the design in place. However, a certain number of elements may shift slightly during the brazing cycles which leads to discarding if an element cannot be adjusted. Given that the triangular units have no axial symmetry, unwanted displacement is limited by sliding friction whereas single granules or pairs tend to roll.

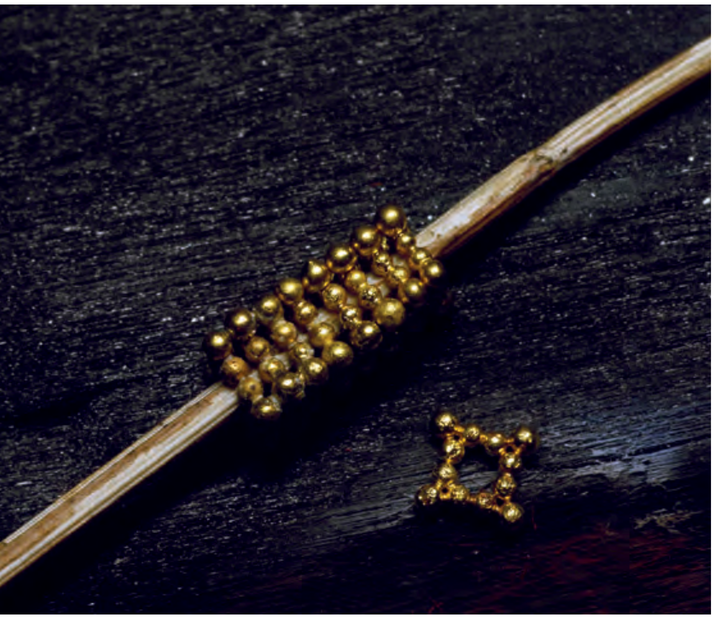

Figure 5: Plane units are aligned using a stem. Copper salts are applied to the four corners of each of the planes. Photo: D. Loepp.

Figure 6: The final brazing, which joins the planes together. Photo: D. Loepp.

The four contact points of each plane are then brazed with a copper-salt composition following the recipe of Dioscorides for *Ios Scolekos*, translated literally in English 'rose-worm rust'[5] (Beck 2005, Matthioli 1586). Attempts to weld the four triangles together by autogenous welding alone was unproductive.

After firing the plane, the lattice is not centered along the horizontal axis of the larger spheres. Gentle pressure with fingernails or a small stick is sufficient to align each plane so that the smaller spheres are centered along the horizontal. The fact that each plane alone can be easily manipulated calls attention to negative aspects and its structural weakness: a plane is subject to torsion, buckling and breakage.

Structural solidity is acquired by uniting the planes in a stack to form the bead. The larger spheres, once joined to form corner columns, act as torsion free nodes. In effect, the only joins to be brazed to complete the bead are between the external, larger spheres.

Stacking the planes to form the bead pattern

Due to the complexity of the plane with the outer spheres fairly distant from the plane's center, it was found that the four larger spheres were not always perfectly aligned. This made it difficult to stack the planes without slippage or shifting. Slightly flattening the contact points did not appreciably resolve the problem. The simplest solution was to thread the eight planes on a dried culm or stem of whatever indigenous grass or similar plant may have been common in the ancient environs (Figure 5). This would have allowed the ancient artisan to choose and rotate each plane to fit, and align all planes to form a square section tubular bead. The bead would then be painted with a copper-salt compost containing organic material on the outer sphere boundaries and be fired after drying. Although the culm quickly burns out, the carbonized organic binder holds the structure together throughout the brazing cycle (Figure 6).

Alternative procedures

In order to assess other possibilities for the construction of the plane, the first author attempted alternative methods of fabrication. The strategy adopted in this set of experiments was to produce four close-packed triangular elements arranged in a truncated square pattern with four joins to be brazed. Another possibility would be to join units of two small spheres to form four linear units. These would then be arranged in a regular octagonal pattern with four larger spheres situated at four vertices to form a square-like plane with 12 points of contact to be joined. An alternative would be to arrange all 12 spheres into the pattern and braze the 16 points of contact all at once on the off-chance that none of the 12 spheres might roll or slide during the brazing cycle. If we are to accept the premise that an artisan develops economic strategies that optimize productivity, these alternative solutions would not have been used.

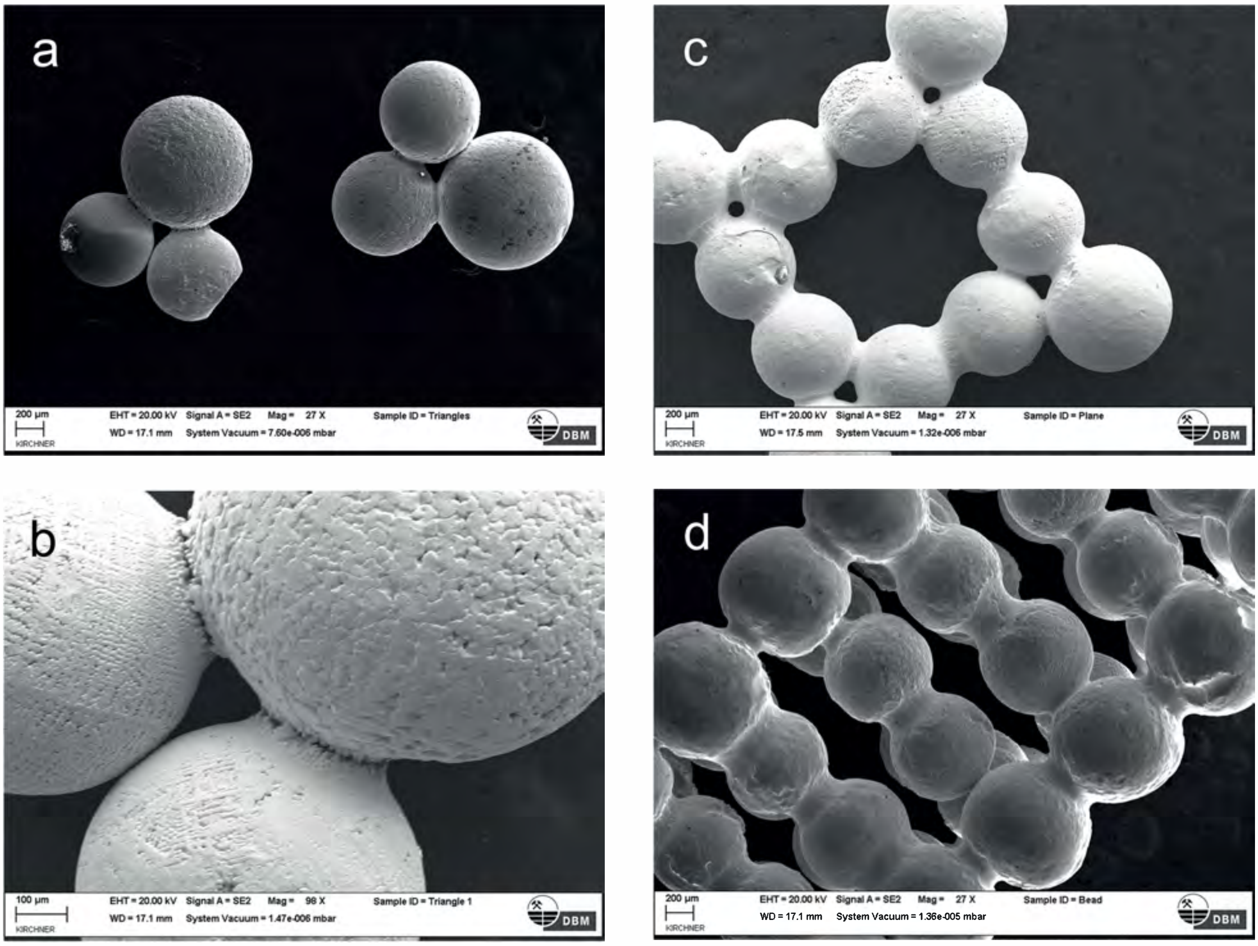

Figure 7: Scanning electron microscope images (Secondary Electron). a) Triangles consisting of three granules. The granules were joined by autogenous welding. b) Detail of autogenous weld joints. c) Plane formed by soldering four triangle units together. The menisci are significantly more pronounced. d) Scanning electron microscope image of the finished bead showing the brazed joints at the corners of the plains. Images: S. Merkel.

SEM-EDS Analyses

Scanning electron microscopy with energy dispersive spectroscopy (SEM-EDS) analyses were undertaken at the DBM to look at the triangle and plane elements and the completed bead. First the autogenously welded triangle elements were analyzed (Figure7b). A characteristic encountered on several of the triangle elements is that the granules have slightly different compositions and reflect the fact that different batches of granules were used. The silver contents range from 8 to 12 wt. % and the copper contents range between 0.6 to 3 wt. %. The analysis of several menisci between the welded granules shows that they tend to reflect the composition of the granule with the higher copper content. Weld joins between copper poor granules (c. 0.6 wt. % Cu) show no compositional difference from the granules. On the triangular units there is no significant variation in alloy composition at the joints. The use of copper salts to join the triangles to form planes has left a mark on the compositions of granules and joints. There is a rather consistent amount of copper over the entire plane (c. 2 wt. %) and the menisci between the granules are notably more robust and well developed (Figure7c).

The completed bead (Figure 7d) show that all joins have an increase of copper on the order of 4 wt. %, which is in agreement with previous research (Ferro, et al., 2008, Parrini, Formigli and Mello, 1982). This is no doubt due to surface spreading of copper from contingent areas, thus masking the fact that the basic triangular units were welded autogenously. In light of these results ulterior testing is warranted to characterize the phenomena of surface alloy masking.

Discussion

The complexity of the object here described implies that a number of sub-units were likely discarded during the various stages of fabrication. The formation of the isosceles triangles presupposes that the smaller spheres would not have differed by more than 0.05 millimeters diameter as this would have had repercussions on the subsequent geometry of the plane. Further, the brazing of each triangular unit yields a mean, as the forces and timing involved in brazing cannot produce constantly identical units even if the spheres are perfectly calibrated. Yet in order to make a plane, four quasi-identical units are necessary to respect the geometry of the plane, vis-à-vis its assemblage with the other planes. Were each plane to stand as a separate unit, however weak, the problem would not be posed, for any irregularity in their assemblage on a thread would simply add to the overall aesthetic charm of the composite artifact. On the contrary, as each plane will be soldered to other planes, respect for a near perfect alignment of the corners is indispensable. It is true that once brazed together the bead could be slightly pressed to even out irregularities, as the flattened surfaces in the original artifact suggest. But this final adjustment cannot belay the fact that a certain degree of discernment, and discarding, was necessary at all stages of the bead's fabrication.

The probability that any given unit floods, fuses or deforms during firing does not entirely depend on the artisan's ability. Given identical sphere values and consistent joining techniques, triangular units may result with varying degrees of compactness after brazing. This leads to a certain percentage of the units being discarded at each stage of the *chaine opératoire*. Any meaningful statistical analysis would involve the fabrication of a large number of units, which exceeds the scope of this work. Discard rate for the few exemplars produced was on the order of 10% for triangular units and 5% for planes. This would be reduced by acquired routine proficiency and large production volume. The entire productive cycle would also have to take into account the quantity of granules produced in excess of the targeted sizes, no doubt to be used on other projects. The circumstances around the production of the bead, such as the manufacture and selection of granule sizes and subunits, point towards a goldsmith who relied on stocks of prefabricates. Whether this prefabricate stock belonged to the goldsmith or to a workshop cannot be known, but either instance would suggest that the craftsperson was specialized and was involved in serial jewelry / ornament production.

Conclusion

The bead is a rare and unique example of structural granulation with a composition entirely composed of granules. It is surprisingly modern in its concept, recalling framed tube systems in which corner columns support an inner tubular array. The use of two sizes of granules precludes the formation of shear walls, thus adding lightness and a sense of length to the aesthetics of the bead. The dimensions of the bead, the underlying factors of production, and the probable signs of wear suggest that it was likely part of a larger assemblage counting dozens (if not hundreds) of identical elements.

The inner diameter of the bead, approximately 1.2 mm, indicates that the bead was designed to allow thick thread to pass through it. This suggests that it may have been a spacer bead to be used with stone beads which require thicker thread to guard against wear breakage. The issue may be resolved by studying wear traces on the two ends of the original bead. If instead a group of beads were strung together, they would tend to pack, creating a stepped quadruple helix design, each bead sitting ajar on the previous bead. The issue of whether a culm was used to string and arrange the planes may also be addressed by appropriate analytical techniques to verify the alignment of the holes.

The bead is remarkable engineering feat, all the more so for the ancient period in question. Due to the burial customs of the Umm-an-Nar and later Bronze Age and Iron Age cultures it is hard to determine an exact date or origin of the artifact. Analyses of the object and further excavations would certainly contribute to a better understanding of the questions posed by this artifact. It bears testimony to a mature technical mastery and style that suggests a cultural and technical context in which objects of this tenure, however small, were recognized and appreciated.

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Notes

1 The issue of temperature gradients is still very complicated. There are a number of articles and standards from which one can derive the statement on the temperatures of brazing, mostly from phase diagrams. It is true that granulation could be accomplished by soaking temperatures around 800°C, transient liquid bonding. However, it is apparent from studies on ancient copper-salt brazing that higher temperatures were used. Further, it depends entirely on bulk the ternary alloy composition. SEM_EDS analyses usually place the meniscus at 4 to 7 % more Cu than the bulk. This corresponds in general to a temperature that is 60-100°C less than the fusion point of most ternary Au-Ag-Cu alloys that were used. There are many factors involved such as the type of flux, melting point depressants, wettability, surface alloying for which there is a substantial body of research, although not on this specific case. The following works were consulted: Butrymowicz, et al., 1974; Kuntz, et al., 2013; Prince, Velikanova and Turchanin, 2006; Roberts, 1973, p.117; ASM Metals Handbook.

2 Attempts to fabricate the bead entirely by autogenous welding proved laborious and ultimately unsuccessful.

3 There are two ways to produce granules based on the first author's practical experience: either by melting small bit of gold individually on a charcoal block or by heating a crucible filled with gold bits in powdered charcoal. The main difference is the efficiency. For large quantities, the use of crucibles with powdered charcoal is the only rational way. At the moment there is no way to prove which method was used to produce the granules.

4 Of six charges that were measured on a scale of 0.1 mm differences, three were random and three were targeted. Distribution by weight of the random groups follow a normal symmetrical probability curve. Distribution in targeted charges are asymmetric with circa 55% within +/- 0.1 mm of the targeted diameter. Diameters less than the targeted range are negligible while larger granules are more frequent. Distribution by unit can also be calculated based on weight.

5 This is the author's translation. Both Beck and Matthioli are in error. The passage was correctly translated into French in the 18th century (Ferro, et al., 2011).

Authors

David Loepp
Via Ambrogio Fusinieri 50
00149 Rome, Italy
loepp@tiscali.it

Alexander Maass
Deutsches Bergbau-Museum Bochum
Forschungsbereich Archäometallurgie
Herner Str. 45
44791 Bochum, Germany
alexander.maass@bergbaumuseum.de