ArcheoSciences
Revue d'archéométrie

33 | 2009
Authentication and analysis of goldwork

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OpenEdition
Journals

Electronic version
URL: http://journals.openedition.org/archeosciences/2323
DOI: 10.4000/archeosciences.2323
ISBN: 978-2-7535-1598-7
ISSN: 2104-3728

Publisher
Presses universitaires de Rennes

Printed version
Date of publication: 31 December 2009
Number of pages: 281-287
ISBN: 978-2-7535-1181-1
ISSN: 1960-1360

Electronic reference
Roberto Cesareo, Angel Bustamante, Julio Fabian, Cristina Calza, Marcelino dos Anjos, Ricardo T. Lopes, Walter Alva, Luis Chero, Fidel Gutierrez, Maria del Carmen Espinoza, Rosendo Rodriguez, Marco Seclen, Victor Curay, Carlos Elera and Izumi Shimada, « Pre-Columbian alloys from the royal tombs of Sipán and from the Museum of Sicán », *ArcheoSciences* [Online], 33 | 2009, Online since 10 December 2012, connection on 19 April 2019. URL : http://journals.openedition.org/archeosciences/2323 ; DOI : 10.4000/archeosciences.2323

Pre-Columbian alloys from the royal tombs of Sipán and from the Museum of Sicán: non-destructive XRF analysis with a portable equipment

Alliages précolombiens des tombes royales de Sipán et du musée de Sicán : analyse non-destructive avec un système FX portable

Roberto CESAREO*, Angel BUSTAMANTE**, Julio FABIAN**, Cristina CALZA***, Marcelino DOS ANJOS***, Ricardo T. LOPES***, Walter ALVA****, Luis CHERO****, Fidel GUTIERREZ****, Maria del Carmen ESPINOZA****, Rosendo RODRIGUEZ****, Marco SECLEN****, Victor CURAY*****, Carlos ELERA***** and Izumi SHIMADA*****

Abstract: On the north coast of present-day Peru, approximately between 50 and 700 AD, flourished the Moche civilization. It was an advanced culture, and the Moche were sophisticated metalsmiths. The Moche metal working ability was impressively shown by the excavations of the "Tumbas Reales de Sipán", discovered by W. Alva and co-workers in 1987.

The Sicán culture is a successive civilization (750-1375 AD) which extended as far as present day Piura in the north and Trujillo in the south. The Sicán culture was strongly influenced by the Moche culture, particularly in its metallurgical development (Shimada and Griffin, 1994).

The metal objects from the Museums of Sipán and Sicán were analyzed with a portable equipment which uses energy-dispersive X-ray fluorescence (XRF). This portable equipment is composed of a small size X-ray tube and a Si-PIN thermoelectrically cooled X-ray detector. It was determined that the analyzed artefacts are composed of gold, silver and copper alloys, of gilded copper, of silvered gold, and of *tumbaga*, the last being a poor gold alloy enriched at the surface by depletion gilding, i.e. removing copper from the surface.

In the case of gold, silver and copper alloys, their composition was determined by EDXRF analysis employing standard alloys. In the case of gilded copper, silvered copper and of *tumbaga*, the ratio Cu(Kα/Kβ) was accurately determined from the X-ray spectra, first in order to clearly distinguish them, and subsequently in order to determine the thickness of the gilding. Mean values of 0.4 μm and 2.7 μm were determined for gilded copper and *tumbaga*, respectively. For gilded silver, the ratio Ag(Kα/Kβ) was measured. The ratios Au-Kα/Cu-Kα, Ag-Kα/Cu-Kα and Au-Kα/Ag-Kα (gilded copper, silvered copper, and gilded silver, respectively) also depend on the thickness of gilding or silvering, and were employed to this aim.

Résumé : *Sur la côte nord de l'actuel Pérou, entre environ 50 et 700 AD, prospérait la civilisation Moche. Cette culture était très avancée et les Moches étaient des métallurgistes avertis. Leur talent pour travailler le métal est illustré par les fouilles du site des "Tumbas Reales de Sipán", découvert par W. Alva et collaborateurs en 1987.*

La culture Sicán est une civilisation qui se succède de 750 à 1375 AD et qui s'étend au nord jusqu'à Piura et au sud jusqu'à Trujillo. La culture Sicán a été fortement influencée par la culture Moche, et tout particulièrement leur métallurgie (Shimada et Griffin, 1994).

Les objets métalliques des musées de Sipán et Sicán ont été analysés avec un spectromètre portable à fluorescence X à dispersion d'énergie. Cet équipement est constitué d'un tube à rayons-X miniaturisé et d'un détecteur de rayons-X de type Si-PIN à refroidissement thermoélectrique. Nous avons pu montrer

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rec. Sept. 2009 ; acc. Nov. 2009

que les objets sont fabriqués avec des alliages d'or, argent et cuivre, avec du cuivre et de l'argent doré ainsi qu'avec du tumbaga, alliage d'or enrichi par mise en couleur, c'est-à-dire par appauvrissement en surface du cuivre.
Les compositions des alliages en or, argent et cuivre ont été déterminées par ED-FX au moyen de standards d'or. Pour différencier les cuivres et les argents dorés ainsi que les tumbaga et ensuite déterminer l'épaisseur de la dorure, le rapport Cu(Kα/Kβ) a été déterminé avec précision à partir des spectres à rayons X. Des valeurs moyennes de, respectivement, 0,4µm et 2,7µm ont été déterminées pour les cuivres dorés et les tumbaga. Pour les argents dorés, c'est le rapport Ag(Kα/Kβ) qui a été mesuré. Les rapports Au-Kα/Cu-Kα, Ag-Kα/Cu-Kα et Au-Kα/Ag-Kα (cuivres dorés, cuivres argentés et argents dorés, respectivement) dépendent aussi de l'épaisseur de la dorure et de l'argenture.

Keywords: X-ray fluorescence, gold leaf thickness, Moche culture.

Mots-clés : *Fluorescence à rayons X, épaisseur de feuille d'or, culture Moche.*

1. Introduction

Objects belonging to the Moche civilization (Vetter Parodi, 2006; Alva, 2006) were analyzed in the past using various destructive techniques (Andrade *et al.*, 2005; Burger, 1992; Lechtman, 1998; Ruvalcada Sil, 2005; Saettone *et al.*, 2003; Scott, 2000); fragments of gold and silver artefacts (and also of silvered gold) from Loma Negra, Peru, were accurately studied and analyzed by Schorsch (1998) by employing EDXRF attached to a scanning electron microscope, and wave length dispersive X-ray Spectrometry. The gold objects showed the following composition: Au ~ 80%, Ag = 10-20%, Cu = 5-15%. The silver objects showed a high Ag content, of about 97-99%. Copper represents the rest of the composition, to 100%. An interesting and unusual feature is the case of silvered gold, with a silver sheet measured to have a thickness of about 5 µm.

Fragments from 17 Moche objects from the "Museo Tumbas Reales de Sipán" have been analyzed by Hörz and Kallfass (2000), using various techniques. These authors were able to identify: a) gilded copper objects: they are characterized by a thin gold film (2-6 µm). The coatings consist of a gold-copper alloy containing some silver; b) copper-silver alloys: fragments from several human head shaped beads have been analyzed, showing a mean composition of 79% Cu, 20% Ag, and 1% Au; c) copper-gold-silver alloys (*tumbaga*): fragments from a headdress, a chin ornament, an ornamental disc, and ornamental beads were analyzed; the average compositions were calculated to be varying between 20-60% Cu, 35-65% Au and 6-15% Ag. The alloy composition is strongly dependent on the distance from the surface.

For the systematic analysis of Sipán and Sicán metal objects, the use of non-destructive techniques may be proposed, and, among them, energy-dispersive X-ray fluorescence analysis (EDXRF) appears to be the most suited, because it is non-destructive, multi elemental, reliable, rapid, and may be carried out in situ (Cesareo *et al.*, 2004). This method is able to quantify the composition of a gold or silver alloy when standard samples are used. EDXRF analysis provides reliable results regarding the concentration of high carat gold alloys with a reduced quantity of copper, and also in the case of high concentration copper or silver alloys. In the case of *tumbaga*, of copper-rich gold alloys, of gilded copper, and of copper-rich silver alloys, the results provided by EDXRF are incomplete or erroneous.

EDXRF is a surface analysis, and it is useful in this case because the respective thickness of the alloy is of the order of a few µm to a maximum of tens of µm; because of this peculiarity, EDXRF is able to distinguish a gold alloy from gilded copper or *tumbaga*, and a silver alloy from gilded silver, by using the internal ratio of Cu and Ag-lines. The method is also able to determine the gold thickness value in the case of gilded copper or *tumbaga* gold.

2. Experimental set-up

The portable equipment employed for the analysis of Sipán and Sicán alloys is composed of an X-ray tube and a Si-PIN detector, both manufactured by AMPTEK. The X-ray tube has a weight of 300 g, length of 17 cm – including the collimator – and diameter of 3.7 cm. It has an Ag-anode, and works at 30 kV and 100 µA maximum voltage and current. X-rays from the tube irradiate areas of about 5 mm diameter, when the object is at a distance of ~2 cm. The X-ray beam intensity is too high when alloys are analyzed, because the Si-PIN detector is able to process only a few thousands of photons/sec without losing energy resolution. The X-ray beam is therefore collimated with a brass cylinder 2 cm long and with an internal hole of 2 mm

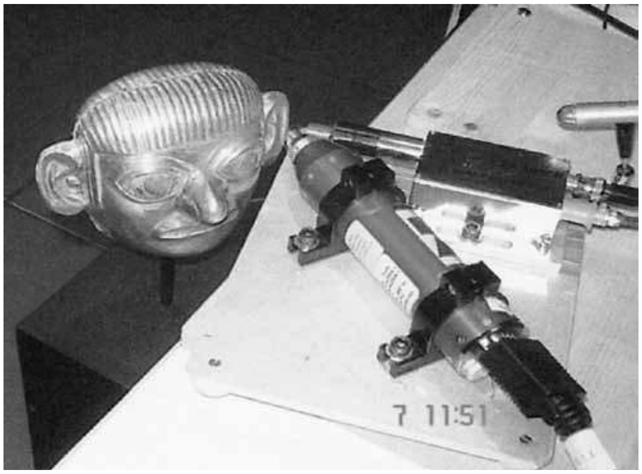

Figure 1: Experimental setup, showing, on the left, the Eclipse II X-ray tube and the Si-PIN detector (both collimated with a brass cylinder), measuring a Moche golden mask in the Museum "Tumbas Reales de Sipán".
Figure 1 : Configuration expérimentale avec, sur la gauche, le tube à rayons-X Eclipse II et le détecteur Si-PIN (les deux collimatés avec un cylindre en laiton) pendant l'analyse du masque d'or Moche au musée « Tumbas Reales de Sipán ».

in diameter. Furthermore, in order to excite silver in a more efficient manner, the X-ray tube output is also filtered with about 0.1 mm Ti.

The X-ray detector is a thermoelectrically cooled Si-PIN, with 300 µm thickness, 7 mm^2 area of the Si-crystal, and a thin Be-window. This detector has an energy resolution of about 180 eV at 5.9 keV, and an efficiency of 90%, 25% and 8% at 10, 20, and 30 keV, respectively. It has a weight of 150 g and a length of 14 cm. It is also collimated. The measuring time was of approximately 100 sec, according to sample composition and geometry.

Standard gold and silver alloys, with certified Au, Ag and Cu concentrations, were employed for calibration and for the quantitative determination of the alloy composition.

In order to measure the gilding thickness of gilded gold or silver, the Cu(Kα/Kβ) or Ag(Kα/Kβ) ratios, and the (Au-Lα/Cu-Kα) or (Au-Lα/Ag-Kα)-ratios were employed. Au leaves and Ag foils were employed for calibration (the foils were each 0.125 µm and 0.28 µm thick for Au and Ag, respectively). Thick sheets of pure Cu and Ag were also employed. Thickness values were tested by transmission measurements with mono-energetic X-rays.

3. Theoretical background

Quantitative analysis of gold, silver and copper alloys

Artefacts of very different sizes, compositions and surfaces were analyzed. It is therefore very difficult to reproduce a fixed geometry. For this reason, determination of the alloy components on the basis of the fundamental parameters method is difficult, and an alternative approach was preferred, using the intensity ratio of two components (for example Cu/Au, assuming that Au(%)+Cu(%)+Ag(%) = 100), which is not dependent on the geometry. Other elements present are determined by using fundamental parameters.

Gilding thickness in gilded Cu (or Ag) and in tumbaga through determination of (Kα/Kβ), (Lα/Lβ)- or (Au-Lα/Cu-Kα)-ratio, and (Au-Lα/Ag-Kα)-ratios

(Kα/Kβ), (Lα/Lβ), (Lα/Lγ)-ratios altered by self-attenuation

The Kα/Kβ, Lα/Lβ and Lα/Lγ-ratios are tabulated (Cesareo, 2000; Markowicz, 1992). For example, for copper, Kα/Kβ = 7.4, and for gold, Lα/Lβ = 1. These values are valid for an infinitely thin sample, corresponding to a thickness < 1µm, < 0.5µm and < 0.2µm for Cu, Ag and Au, respectively. For larger thicknesses, self attenuation effects must be considered (Cesareo and Brunetti, 2008).

(Kα/Kβ), (Lα/Lβ), (Lα/Lγ)-ratios of an element altered by attenuation by a second element

When a sheet of metal (a), for example copper, of infinite thickness, is covered by a sheet of another metal (b), for example gold, then the ratio $(K_\alpha/K_\beta)_a$ or $(L_\alpha/L_\beta)_a$ is altered because of the attenuation of the covering sheet:

$$(K\alpha/K\beta) = (K\alpha/K\beta)_{s.a.} \exp\text{-}[\mu_1 - \mu_2]\ d \qquad (1)$$

$(K\alpha/K\beta)_{s.a.}$ is the ratio of metal (a) according to possible self-attenuation; μ_1 and μ_2 (cm^{-1}) are the attenuation coefficients of element (b) at the energy of Kα and Kβ rays of element (a); d is the thickness (in cm) of the sheet of element (b) (Cesareo and Brunetti, 2008; Cesareo *et al.*, 2009). Similar equations may be calculated for Lα/Lβ and Lα/Lγ-ratios.

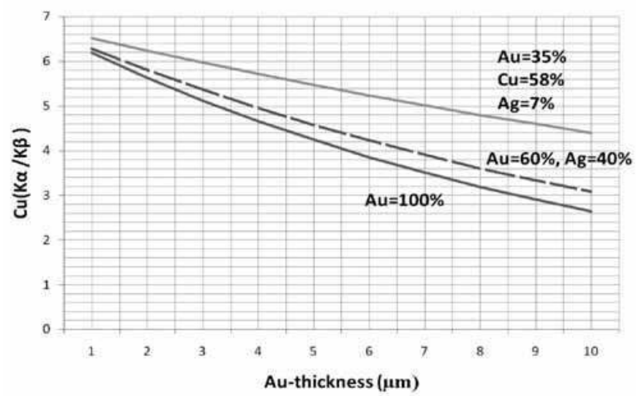

Figure 2: Gilded copper: attenuation of Cu(Kα/Kβ) ratio by Au (or Au alloys) vs Au thickness.
Figure 2 : Cuivre doré: atténuation du rapport Cu(Kα/Kβ) par l'Au (ou les alliages d'Au) versus l'épaisseur d'Au.

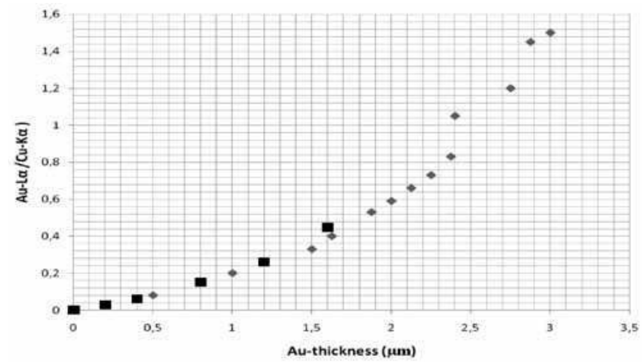

Figure 3: Theoretical (squares) and experimental values for (Au-Lα/Cu-Kα) ratio vs Au thickness, assuming Cu with infinite thickness.
Figure 3 : Valeurs théoriques (carrés) et valeurs expérimentales du rapport (Au-Lα/Cu-Kα) versus l'épaisseur d'Au, en assumant une épaisseur infinie pur le Cu.

(Au-Lα/Cu-Kα)-ratio versus Au thickness

Another way to experimentally determine, from the X-ray spectrum, the thickness of the second element (b) assuming that the first element (a) has an infinite thickness is the use of the X-ray ratio of the two elements, for example, the ratio (Au-Lα/Cu-Kα). This ratio, for two generic elements at fixed incident energy and geometrical arrangement, is provided by the following formula:

$$N_b/N_a \propto (\mu_{a0}+\mu_{aa})/(\mu_{b0}+\mu_{bb})[1-\exp-\{\mu_{0b}+\mu_{bb}\}\rho_b d_b] \exp\{\mu_{0b}+\mu_{ba}\}\rho_b d_b \quad (2)$$

$-\mu_{a0}$ or μ_{b0} (in cm^2/g) are the total mass attenuation coefficients of elements (a) and (b) at incident energy; μ_{ab} (in cm^2/g) indicates the mass attenuation coefficient of element (a) at energy of the involved line of element (b); ρ_b (in g/cm^3) is the density of element b.

In Equation (3), the proportionality should be quantitatively determined according to experimental data. Figure 3 represents theoretical values (from Eq. 3) and experimental measurements values for the (Au-Lα/Cu-Kα)-ratio versus Au thickness, assuming Cu with infinite thickness.

4. RESULTS

Alloys from "Tumbas Reales de Sipán"

About 50 objects from the "Tumbas Reales de Sipán" were analyzed, made of gold, gilded copper, *tumbaga*, silver and copper alloys, the majority of them originating from the tomb "Señor de Sipán".

Gold objects composition

Several objects are made of gold (an example is shown in Fig. 4) and are characterized by Au, Cu and Ag as main components. In some samples, traces of Fe, Zn and Br are visible. The mean results of the EDXRF analysis carried out on gold objects are the following:

Au = 69.5 ± 7%; Ag = 21 ± 4%; Cu = 9.5 ± 5%.

Gilded copper: analysis and Au-thickness measurement

Only a few objects are surely of gilded copper. They were identified by the exclusive presence of Cu in some analyzed areas, and by a deteriorated surface. In many cases, it was possible to clearly determine the Au leaf thickness from Cu (Kα/Kβ) and (N_{Au-L}/N_{Cu-K}) ratios.

Several sheets of gilded copper were analyzed. They are characterized by a Cu(Kα/Kβ) ratio of 6.1 ± 0.1, corresponding to a gilding thickness of 1.2 ± 0.5 μm. From the (Au-Lα/Cu-Kα) ratio of 0.1 results a value of ~ 0.5 μm, however. A beautiful mask of gilded copper was analyzed in detail, showing the following composition: Au ~97.5%, Ag ~2.5%. The gilding thickness was measured to be ~ 0.5 μm. Cu could not be determined, because it was present below the gilding.

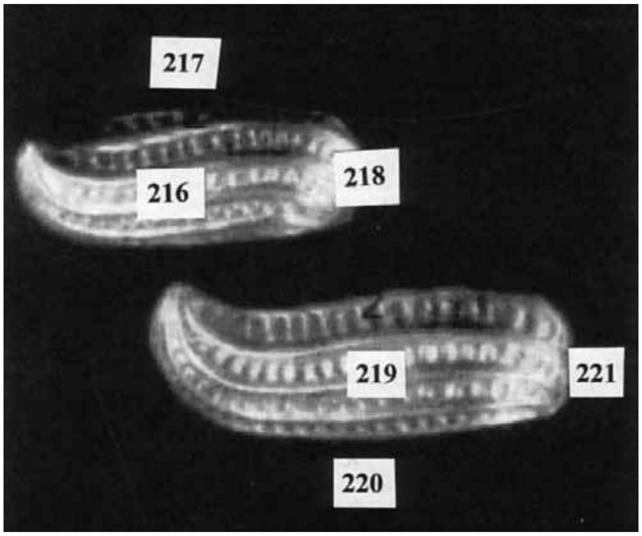

Figure 4: Peanut heads made of gold, belonging to a necklace. The average composition is: Au = 58%, Ag = 26%, Cu = 16%.
Figure 4 : Perles en forme de cacahuète exécutées en or et appartenant à un collier. D'après la Ref. (2). La composition moyenne est: Au = 58%, Ag = 26%, Cu = 16%.

Figure 5: Leg protector made of *tumbaga*. The ratio Cu(Kα/Kβ) is 4.8, corresponding to an Au thickness of 3.1 µm
Figure 5 : *Protecteur de jambe en* tumbaga. *Le rapport Cu(Kα/Kβ) est 4,8, correspondant à une épaisseur d'Au de 3,1 µm*

Tumbaga *(or gilded copper) mean composition and gold thickness measurement*

The majority of the gold alloys were identified as *tumbaga* (an example is shown in Fig. 5), which behaves in a similar manner as gilded Cu for EDXRF analysis. The 'gold-equivalent' surface thickness can be determined from Cu-Kα/Kβ and from (N_{Au-L}/N_{Cu-K}) ratios (see Section 3). The mean Au-Cu-Ag concentration and Au thickness values are:

Au = 60 ± 10%; Cu = 30 ± 9%; Ag = 10 ± 4%.

The (N_{Au-L}/N_{Cu-K}) ratio = 1.44 ± 0.7, corresponding to an Au thickness of 3.10 ± 0.7 µm;

The (Cu-Kα/Kβ) ratio = 5.35 ± 0.5, corresponding to an Au thickness of 2.5 ± 1.4.

Silver objects

The EDXRF analysis of silver objects shows that the silver content is relatively high, and that it systematically contains gold. The following mean concentration values were determined:

Ag = 92 ± 4%; Cu = 5 ± 2.5%; Au = 3 ± 1.5%.

Copper objects

Parts of a few objects are made of copper, composed of about 99% Cu, and traces of Fe and Ni.

Turquoises

Many of the gold objects include turquoise, which is a hydrated phosphate of aluminium and copper. The turquoise shows an occasional presence of Zn, Fe and Cr as impurities, resulting in deviations from the blue colour. Results pertaining to Sipán turquoise show a systematic presence of Fe and Zn, at an average concentration of 10% and 8.5%, respectively.

Alloys from the museum of Sicán

About 20 objects from the Museum of Sicán (Shimada and Griffin, 1994) were analyzed, the majority of which were of gilded copper; others were made of gold, *tumbaga*, silver and copper alloys.

Objects made of gold

The following mean values could be determined:

Au = 62 ± 4% ; Ag = 32 ± 3% ; Cu = 6 ± 2%.

Objects made of gilded copper

The following mean values could be determined for the gilding: Au = 67%; Ag = 33%.

Objects made of tumbaga

A few objects are of uncertain composition; they could either be made of gilded copper or of *tumbaga*. In fact, the gilded copper objects are identified because of the altered Cu(Kα/Kβ) ratio, and because of the presence of highly corroded areas on almost pure copper. In other cases, the ratio Cu(Kα/Kβ) is altered, but no corroded areas were detected.

One artefact, a beautiful mask, is certainly made of *tumbaga*, whose concentration and characteristic thickness parameters are the following:

Au = 34 ± 6%; Cu = 57 ± 5%; Ag = 7 ± 1.5%.

The Cu(Kα/Kβ) ratio = 4.9 ± 0.3, corresponding to an Au thickness of 3.8 ± 0.9 µm.

The (Au-L/Cu-K) ratio = 1.05 ± 0.02, corresponding to an Au thickness of 4.5 ± 0.8 µm.

Objects made of silver

Only one object is made of silver, i.e. a brooch in the form of a monkey, with the following composition: Ag = 94%, Cu = 3.7%, Au = 0.5%, Pb = 0.8%, Br = 1%.

Objects made of copper

The following mean values could be determined:

Cu = 98 ± 1%; Fe = 1.2 ± 0.5%; As = 0.8 ± 0.5%.

5. Conclusions

EDXRF analyses of precious objects from Sipán and Sicán demonstrate their complexity and variety; they are made of the following alloys: gold, gilded copper, *tumbaga*, silver, silvered copper, silvered gold, copper, and so on, and their nature is not always easy to identify.

From the metallurgical point of view, a comparison between the Moche (50-700 AD) and Sicán (750-1375) civilizations shows that:

– Moche precious objects seem to be more sophisticated in terms of beauty and technology;

– The average compositions of gold and silver are similar;

– The Moche civilization largely used *tumbaga*-gold (representing more than 50% of the 'gold' artefacts), while Sicán used more gilded copper;

– The Moche employed almost pure copper; the Sicán civilization had a knowledge of arsenical copper.

Acknowledgements

This work was partially carried out within the framework of the project IAEA-CRP (G4.20.02/1371) "Unification of nuclear spectrometry: integrated techniques as a new tool for material research". J. Fabian expresses his gratitude to the International Centre for Theoretical Physics Abdus Salam for a 5 months grant at the University of Sassari.

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