

An Archaeometallurgical Study of Mixtec Silver Gold Alloy Foils from Tomb no. 7, Monte Alban, Oaxaca, México

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The main pre-Columbian collection of gold artifacts in México, belongs to Mixtec culture, this is exhibited in the Gold Room of the Museo de las Culturas de Oaxaca, México. It is well known that the funerary chamber called Tomb No. 7 from Monte Alban Oaxaca was discovered by Mr. Alfonso Caso in 1932; between an extensive quantity of artifacts manufactured with precious materials, 121 gold pieces were recovered. Few studies on metalwork was determined in particular parts of Mesoamerica, developing various methods to yield metal artifacts as reported by Motolinia, Sahagún, Las Casas, Bernal, Cortés, and others. Most archaeologists believe that the Mesoamerican metallurgy (México and Guatemala) is related to cultures from other regions of Central and South America because of certain similarities. The Mixtec goldsmiths employed several techniques such as ordinary casting, hammering, cold-working, lost-wax casting, false filigree, welding and gilding to obtain artifacts and ingots of metals that had previously been molten. Ethnohistoric sources mention that "laminated gold" was worked exclusively via hammering to achieve hardness employing stone or copper tools.

There are few studies on the gold alloy artifacts from Tomb No. 7 at Monte Alban, usually they present results of nondestructive techniques which give information about elemental composition mainly; in this work we analyze Au/Ag/Cu alloy foil samples from Tomb No. 7, which were called silver gold alloy foils because of their main elemental composition and they were classified as received according to coloration as LG1, LG2, LG3, and LG4 groups. An important characteristic of Au/Ag/Cu alloys is their coloration, which can vary between reddish, yellowish, and silvery depending on the metals' proportions, superficial changes for gilding processes, corrosive effects and exposure to heat to mention some causes. Gold (Au), silver (Ag), and copper (Cu), show FCC crystalline structure and a compact atomic order, ideal for hammering because of their malleability, toughness, and resistance to brittle fractures. For small fragments of silver gold alloys foils characterization, we start with a superficial examination without cleaning, chemical etching or any metallographic preparation by mean of LV-SEM/EDS, observing superficial damage such as pitting caused by corrosion processes during burial and/or gilding processes; topographic heterogeneities, micrometric pores and longitudinal marks due to manufacturing processes and ancient foils management. According to the first EDS approach, the sample richest in gold showed minor porosity because this metal dominates the properties of the Au/Ag/Cu alloy, which is less prone to corrosion than copper; on the other hand, EDS of many materials sucked in the surface and incrustated in the pores suggest the presence of minerals from the burial site as feldspars, clays and quartz.

As second step we selected one piece of each group of foils and cut a small section of 4x2 mm trying to avoid deformation, each section

was mounted in transversal section using resin to be polished and subsequently etched for thickness measurement, to reveal their microstructure applying ASTM standards and made a second EDS approach by elemental mapping. Foils thickness was in the range from 27 to 61 microns and the Au, Ag and Cu elemental mapping was very homogeneous in the samples with a light gold enrichment on the surface's foils. Unfortunately, after chemical etching, we did not observe defined grain boundaries at 600x magnification, but green, purple and red colorations corresponding to copper, silver and gold were appreciated, suggesting that the grain size could be extremely small. Then we decide preparing two FIB cross sections in each specimen mounted in resin: one in transverse cross section and the other in longitudinal cross section with the propose to reveal the microstructure and identify the elemental composition without traces of abrasives and burial impurities. All samples analyzed contain Au, Ag and Cu in different proportions with compositions close to modern 6-karat, 7-karat, and 14 karat gold. Comparing our results for silver gold alloy foils from Tomb No. 7 with other authors, the compositions are different than those reported for artifacts belonging to different museum collections (Maya, Caxonos and Fisherman's treasure items), including other gold collections from Oaxaca (Zaachila) and pieces from Tomb No. 7; three silver gold alloy samples analyzed in this work are in the gap of the Au/Ag/Cu phase diagram reported by Peñuelas et. al. between 20%wt Au and 50%wt Au.

FIB erosions revealed microstructural details at the micrometric and nanometric levels, which are not visible using traditional metallographic observations; three groups of samples showed well-developed equiaxial grains in the order of 1-14 microns in transverse and longitudinal cross sections, annealing twins due to operation at proper temperatures and times, and degasification macropores (from 54-68 nm) and mesopores smaller than 50nm in the core of the foils. The fourth group of samples was probably cooled very rapidly because non-recrystallized grains in both cross sections domain but there are no signals of grain enlargement in the deformation direction, neither annealing twins, iron inclusions and intergranular micro fractures as in the rest of the samples. Finally, nanoindentation tests were recorded to extract the elastic modulus (EIT) and indentation hardness (HIT) of the samples from load-displacement measurements with a Berkovich indenter, finding that the thinnest foil (27 μm) has the highest hardness value (HIT=1781.7 MPa), Au content (56.9 wt%) and the lowest Cu content (1.5 wt%) unlike the non-recrystallized sample that showed the lowest hardness value (HIT=325.1 MPa) because of a deficient work metal. All result together, revealed that the original casting Au/Ag/Cu alloys were cold worked and annealed using a mixed technique of hammering with intermediate annealing for grain size refinement, modifying the mechanical properties of these solid solutions.