Initial study of metallurgical technology from western Tinogasta, Catamarca, NW Argentina (1st-15th centuries CE)

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ABSTRACT: We undertook an initial study of the metallurgical technology developed in the Fiambalá and Chaschuil regions between the 1st and 15th centuries CE. In so doing we compiled data on macroscopic observations of metal objects deposited in museums, evidence of mineral resources within the regions, and archaeological studies of metal pieces recovered from archaeological sites. This included visual analysis of geometric and surface characteristics and scanning electron microscopy with X-ray energy dispersive spectrometry (SEM-EDS) to determine the chemical composition of the alloys. Collating this evidence allowed us to characterise the metal objects' production system and the different socio-political contexts in which they appeared. Furthermore, it highlighted the role played by western Tinogasta in the supply of tin, of vital importance in copper-tin metallurgy.

Introduction

Technology cannot be dissociated from the identities and roles of the agents who employ it within a given socio-historical context. Technology, therefore, constitutes a continuous process of material and social reproduction (Dobres and Hoffman 1999). The choices that a society makes when it adopts, rejects, or modifies a technical component involves other elements which have no apparent material purpose. These non-technical aspects affect the way one thinks, as well as in how the object is manufactured and used, including its effectiveness. It also impacts on those who use it where, even if mundane, it can evoke thoughts, behaviours and attitudes relating to distinct areas of social life. In effect, technology produces more than just things, but also social representations, relations, and symbols (Dobres 2000; Lemonnier 2012, 13-20). With this in mind, we believe that mining and metallurgy technological processes are social phenomena imbued with values, modes, attitudes and behaviours, which are articulated in the multiple decisions that the processes entail, from the search for, through to the extraction of the raw materials, culminating in its transformation into a finished product.

Lemonnier (1986; 1992) states that the stages of the *chaîne opératoire* – in our case of mining and metallurgy – have their own dynamics and involve a series of phases. Therefore, an archaeological study of mining and metallurgy is not limited only to describing the sequence of activities, but also to understanding its interaction with the physical and social environment of those who developed these mining processes in the past (González 1992; Angiorama 2001; Salazar 2003-4; Salinas and Salazar 2008 among others). Likewise, the *chaîne opératoire* allows us to understand the technical process as a coherent scheme of articulated sequential actions, which are composed of both physical, theoretical and practical elements as well as other abstract ones; it is the interweaving of these spheres that define the process (Salinas and Salazar 2008). The technical aspects mainly cover the choice of alloys and the processes used to manufacture each part with their respective tools and associated devices (moulds, crucibles, hammers, anvils, furnaces). Additionally, there is also the matter of the place chosen to undertake these activities.

Conversely, in a more conceptual manner, there is the whole facet of understanding the relationship of these technical systems to the local social organization, ritual life, as well as the maker's thought processes (Lemonnier 2012, 13-20). For example, the colour, brightness, and sound qualities of the objects are a product of the alloy types used but also constitute an interpretative bridge towards uncovering the symbolism behind the object's context of use (Pernot 2010; Rovira 2017). Thus, Prehispanic Andean metal mining and metallurgy entailed the appropriation, handling and transformation of raw materials to produce and use objects that were related to different spheres of society, ranging from the strictly functional to the religious-symbolic (Lechtman 1976; Salazar and Vilches 2014).

In this article, we present an initial study into the metallurgical technology developed in the Fiambalá and Chaschuil regions of Catamarca, both by village-level societies (1st to 11th centuries CE) and the Inca conquista (15th century CE) (Ratto 2013). We collated data at different scales of analysis: on the one hand, macroscopic observations made on metal objects from archaeological collections; on the other, the archaeometric study of six pieces of metal. These objects come from archaeological sites.

Metalliferous resources from the region of Chaschuil and Fiambalá

Knowledge of how minerals appear naturally is key to reconstructing Prehispanic metallurgical technology, given that extraction methods varied depending on whether it was an oxide or carbonate mineral rather than a sulphide. This is because most metals in their natural state are found as ores of various compositions. For example, copper can occur as a native element, as copper oxide (cuprite, tenorite), copper carbonate (malachite, azurite), or as copper sulphide (covellite, chalcocite, chalcopyrite) (González 1992; Angiorama 2001). Estimating the quality, sustainability, accessibility, and content of the mineral deposits present in a region are important aspects when studying Prehispanic mining as part of a *chaîne opératoire* (Stöllner 2014). In general, metallic minerals and associated gangue minerals (of no economic value) such as quartz, pyrite, carbonates, and tourmaline, are found in joints and faults in igneous or metamorphic rocks, or bedding planes in sedimentary rocks. Minerals precipitate from brines and sulfide solutions forced through these channels. These channels gradually fill to form veins and narrow sheets that can be very rich in metal. In prehistory, they were the most important sources of copper, lead, zinc, silver, and tin before the development of open pit mining.

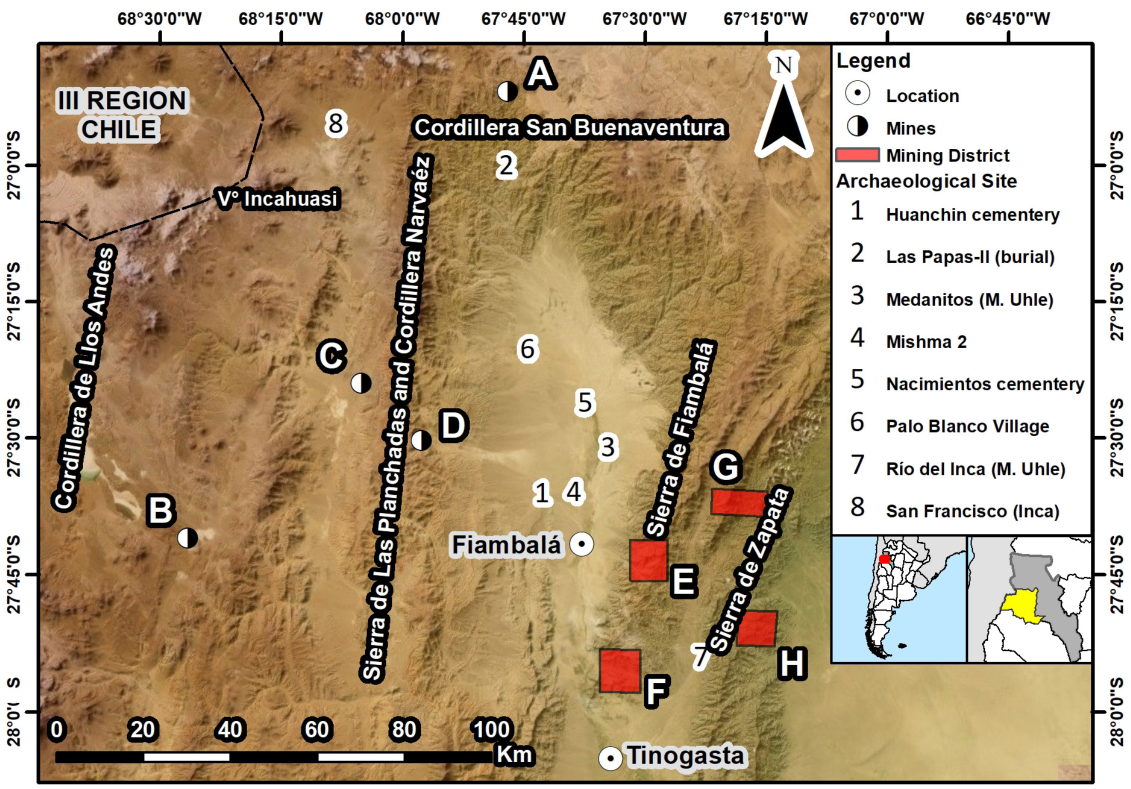

Figure 1: Archaeological sites, mines and mining districts in the Chaschuil and Fiambalá regions. A= La Hoyada district; B= Aparejo mine; C= Lampaya mine; D= Cerros de Las Minas mine; E= Los Ratones district; F= El Salto-Agua de Los Mineros district; G= El Fraile district; H= Sierra de Zapata district.

Table 1: Metalliferous resources in western Tinogasta, NW Argentina.

Resources and location	Height (masl)	Groups and/or mines	Minerals	Fig 1
La Hoyada district, Cordillera San Buenaventura	4200	Descubridora group (deep veins) Rosario group (upper veins)	Polymetallic deposit Ag-Pb-Zn-Cu-Au	A
Los Aparejos, Chaschuil transitional Puna	4200	Aparejo mine	Cu	B
Lampaya, Chaschuil Valley, Cazadero Grande	3700	Lampaya mine	Cu	C
Narváez Cordillera and Zanjón de Apocango, Colorado province	c3000	Cerro de las Minas mine	Cu	D
Los Árboles–Los Ratones district, Sierras de Fiambalá	2200-3100	Pachamama (Pb-Cu) Los Ratones (Pb-Zn) Buena Suerte (W) La Franca (ex Las Termas) (U)	Sn-W with Pb-Zn and Pb-Cu-U	E
El Salto–Agua de Los Mineros district, Sierras de Fiambalá	1500-2500	Andacollo and Tres Sargentos (W) Vil Achain (Sn) La Argentina (Fe)	W, Sn, Fe	F
El Fraile district, Sierras de Fiambalá	3300-3800	Progreso Argentino Las Champas	Sn	G

The mineral resources of the western Tinogasta regions of Chaschuil and Fiambalá are described in geological maps, including the Paso San Francisco sheet 2769-IV (Seggiaro *et al* 2006), Chaschuil leaf 13 b (Turner 1967) and Fiambalá sheet 13 c and 2769-II (Rubiolo *et al* 2003) and their mining description (Tezón 1957). The known mineral resources are shown in Figure 1 and Table 1.

- In the southern Puna, the polymetallic deposit of the La Hoyada district (Fig 1A) is important. Its mineral veins were mainly formed by fissure-filling processes along faults and fractures in andesitic rocks. The district is composed of two mining groups, Descubridora and Rosario, which occur at different depths with characteristic mineral associations (Table 1). In the Spanish Colonial Period, work was carried out on the gold-enriched veins of the Rosario Group; it was not until the 19th century that underground reconnaissance and exploitation began on the Descubridora Group. This latter group has a wide distribution of oxidised minerals such as cerussite, copper carbonates and limonites. The average metal contents of the analysed samples for the Descubridora Group are 2.78% Cu, 4.8% Pb, 0.62% Zn, 343ppm Ag, 0.85ppm Au and 900ppm Bi, and for the Rosario Group: 1.73% Cu, 1.41% Pb, 0.62% Zn, 1150ppm Ag and 0.47ppm Au.
- In the transitional Puna of Chaschuil lie the copper mines of Los Aparejos and Lampaya (Fig 1B-1C). The former is a skarn-type deposit 3-30m thick with copper mineral lenses found in association with other minerals (garnet, quartz, calcite, epidote, chalcopyrite, magnetite and pyrite). Lampaya is an old sloping shaft 8m long, with vein mineralization made up of chalcopyrite, malachite, and azurite patina (Turner 1967; Seggiaro *et al* 2006).
- The Cerro de Las Minas copper mine (Fig 1D) is located within the granite of the Narvaéz Formation, at the confluence between the Zanjón de Apocango and the Cordillera. Turner (1967) reported that she was unable to undertake a complete survey of the area although she did clarify that there were other copper deposits there.
- The Sierra de Fiambalá contains the largest metal resources in the area, especially tin, copper, lead, iron, and tungsten. In this sense, the Los Arboles-Los Ratones, El Salto-Agua de Los Mineros and El Fraile districts stand out (Rubiolo *et al* 2003; Tezón 1957; Fig 1E-1G). The geological data also mentions mineral districts in the Sierras de Zapata, Department of Belén (Fig 1H), which are adjacent to our research area and known to be rich in tungsten and tin (San Ramón and San Antonio mines). Tin occurs in the form of cassiterite (oxide), stannite and hexastannite (sulphides), although stannite is more common. They

consist of vein deposits of different sizes and large alluvial accumulations produced by erosion of the veins. In particular, the Pachamama mine has copper sulphides (chalcocite, chalcopyrite), iron sulphides and galena as primary minerals, while the secondary minerals present are malachite, azurite, haematite, cerussite, and other alteration minerals. The minimum copper grade of the deposit is around 20%, occurring in non-superficial veins of considerable thickness (Catalano 1944).

Generally the commercial operation and exploitation of the minerals in these mines, mainly tin and copper, dates to the beginning of the 20th century. It consisted of open-cast mines with galleries, chimneys and flues, or inclined galleries (Rubiolo *et al* 2003). The work was carried out *a pirquén*, a type of mine leasing involving cash payment or a share of production. This system coexisted with the more traditional *pirquén* industry that was carried out by a miner or group with little capital investment or technology (Godoy Orellana 2016).

Metal objects from western Tinogasta

Archaeological excavations in Catamarca province, NW Argentina, rarely uncover many metal objects, nor are they common in either private or museum collections (González 1977; González and Goretti 2012). We have registered, surveyed and inventoried – in keeping with current law – metal objects from collections held by local people (Pereira, Pereyra, Quintar, Castro), as well as others held at a variety of museums, such as the Hombre de Fiambalá and Tullio Robaudi Museum (Tinogasta, Catamarca), the Inca Huasi Museum (La Rioja), the Jesús María National Jesuit Museum (Córdoba), and the Berlin Ethnological Museum (Germany) (Ratto 2015; Ratto and Basile 2020). Additional pieces and by-products of mining activity were collected from archaeological excavations carried out at sites in the Chaschuil and Fiambalá-Abaucán regions from the 1960s to the present (Sempé 1976; Ratto 2013; Ratto *et al* 2018). Other museums hold metal objects whose descriptions refer to Tinogasta, as in the case of the very elaborate metal axes found in the Adam Quiroga Museum, Catamarca (González 2008, 71, fig 10); but, since we cannot precisely fix their place of origin, we do not to consider them here.

Generally, there are restrictions on taking samples, altering the surface, and/or transferring pieces from collections when engaging in archaeometallurgical studies. Therefore, our analysis was restricted to noting the objects' macroscopic characteristics. In some cases, it was possible to reconstruct the associated contexts of the objects because of an existing written or oral record of its discovery (Ratto and Basile 2020).

Metal objects from private and museum collections

In the Museo del Hombre de Fiambalá there is an Inca metal statuette dressed in a variety of textile garments from the Incahuasi Volcano ceremonial site (Fig 1; 6638m above sea level). In the Robaudi Museum are metal objects, mainly chisels, needles and tweezers, which were recovered from fields around La Troya, 25 km south of Fiambalá (Fig 1), which are the product of unsystematic collections carried out by Tullio Robaudi during the 1960s and 1970s.

However, most of the metal objects are in provincial museums and museums in other countries. The Inca Huasi Museum (La Rioja) holds the materials from the Huanchín Prehispanic cemetery (Fig 1,1) excavated by the Franciscan priest Gómez (1953), who reported that the metal pieces were part of the grave goods, along with pottery, bone, pyrographed gourds, textiles and other finds. Sempé (1976) examined these objects, noting their dimensions and raw materials. He highlighted those made of copper (pectorals, axes, bracelets, knuckledusters, awl, chisel, needle), those of gold (plates, bracelet and headband fragment), and a sceptre, 575mm long, made of an alloy of Cu, Au, Ag and Sn. In contrast, at the Nacimientos cemetery (Fig 1,5), excavated by the Jesuit Dreidemie (1953), metal pieces were sparse with only two needles and few copper beads uncovered. These materials are stored at the Museo Nacional de Jesús María, Cordoba.

Outside Argentina, the Uhle Collection of the Ethnological Museum of Berlin (Germany) has metal pieces from Tinogasta collected during Max Uhle's short sojourn in western Tinogasta in 1893 (Ratto 2015). This assemblage was collected by Uhle from surveys and excavations at different locations and includes various types of materials, including metal objects, whole and fragmented, as well as by-products of metallurgy (slag, metal and sheets). The collection includes:

- Material from excavations at Medanitos (Fig 1,3) at a site described as a 'place with stone walls' where he recovered a copper *tumi* knife, of possible Inca affiliation, in addition to slag and metal sheets of the same metal, and particularly a possible Hispano-indigenous silver ingot (Figs 2B, 2F). These discoveries were important because in Medanitos we have the 'Indian town of Abaucán'. Abaucán was considered the hub of a complex social network formed by local populations, the Inca conquest, the people mobilized by the empire and the subsequent Spanish colonial conquest (Ratto and Boixadós 2012). At the El Sunchal site (Fig 1,3), Uhle recovered a fragment of an inverted-face bell, possibly made of bronze (Fig 2G). This type of object was highly significant within Andean religion (González 2008).

- From the site of Río del Inca (Fig 1,7) comes the largest quantity and diversity of materials (ceramic, lithic, bone, shell, metal) collected (Uhle 1912; Ratto 2015). The site is located at a strategic point of communication between western Tinogasta and the eastern valleys located beyond Cuesta de Zapata. Among the metals, copper and silver stand out, but there is also iron, a clear indicator of a post-Spanish conquest context. The finished objects were made of copper (needle, chisel, tweezers, ring, hoop, nose ornament), but there are also sheets of copper, silver and gold (Figs 2C-2E), and remains of copper, silver and iron slag. Uhle (1912) states that metallurgical activities were carried out at Río del Inca during the Prehispanic period and continued after the Spanish conquest. Río del Inca had both a residential and productive function.
- Other metal objects were in the hands of local collectors, the result of unsystematic collections carried out across different parts of the region (Figs 2A, 2H-2K). These included copper objects such as needles, tweezers, nose ornaments, combs, and ornaments from Palo Blanco (Fig 1,6; Quintar Collection) and Saujil funerary contexts (Pereira and Pereyra Collections, CP and CPy, respectively; Fig 1,3), and an Inca *topu* from the settlement site of Mishma (Fig 1,4; Castro Collection) (Ratto and Basile 2020).

It is striking that the pieces in these collections, whether public or private, date exclusively to later period when there was an Inca and/or Spanish presence in the region.

Figure 2: Metal finds from archaeological collections: A) needle (CP-03-1-1); B) possible silver ingot (EMB,VC1402; C) gold sheet (EMB,VC1629b); D) gold sheet (EMB,VC1629a); E) copper sheet (EMB,VC1505); F) tumi (EMB,VC1406); G) bell fragment (EMB,VC1393); H) possible earring (CP-03-5-2); I) possible nose ornament fragment (CP-03-5-4); J) hair comb ornament fragment (CP-03-5-3; cf González 2008, fig 4:64); K) nose ornament fragment (CP-03-5-6). Scale 100mm.

Metal objects analysed

The history of academic archaeological research in Tinogasta began with the surveys and excavations conducted by A R González and M C Sempé in the 1960s. Research resumed almost 35 years later under the aegis of the Chaschuil Archaeological Project starting in the mid-2000s (González and Sempé 1975; Sempé 1976; Ratto 2013). Fieldwork at different archaeological sites in the region yielded metal objects, although always in much lesser quantities than the abundant ceramic and lithic material recovered. These sites included the housing nuclei and midden of the Palo Blanco settlement site (1st–11th centuries CE); Burial LP-II at Las Papas (14th century CE); the San Francisco-Inca site (15th and 16th century CE); and the Mishma-2 site at Mishma (15th century CE). Each one of these sites refer to a different moment and socio-political organization within the socio-environmental history of western Tinogasta, dating from the 1st to the 15th century CE. The sites include residential contexts of different functions and occupational intensity, as well as funerary settings (Table 2).

The Palo Blanco settlement site is located at an elevation of 1,900masl in the northern sector of the Fiambalá Valley (Fig 1,6). It is composed of six housing units built using a rammed-earth technique; the wall layout form right-angled, regular-shaped adjoining enclosures, with internal open spaces enclosed by a perimeter wall (Sempé 1976; Ratto *et al* 2019a). The settlement emerged between the 1st and 10th century CE and displays the prevalence of repeated collective actions both in its architecture and in its artifact assemblages. In social terms, we inferred that these enclosures were inhabited by extended families that continually reproduced their practices throughout this 1st millennium, with an absence of a centralized political system (Ratto *et al* 2019a). In the settlement we recovered the following metal objects: a burin chisel, tweezers and a needle belong to different moments in the history of the settlement development (Table 2, Figs 3A-3C). Sempé (1976) also recovered fragments of foundry moulds in the housing core, dating to the 7th-8th centuries CE.

Burial LP-II is located at an elevation of 2,870 masl, on the periphery of the Las Papas settlement, in the middle of the San Buenaventura Cordillera (Fig 1,2). It is a stone-lined circular chamber tomb (cist) containing a young adult male of between 20-35 years of age. This individual was buried together with many objects of different materials including ceramic, lithic, bone, metal, wood, and textile (Ratto *et al* 2019b). The radiometric date of the site and the techno-morpho-decorative characteristics of the ceramic pieces places this tomb within the context of late (pre-Inca) societies that were later impacted by the arrival of the empire. The metal object uncovered was either a knuckleduster or tensioner (Fig 3D). It was employed by inserting the hand into the hole and grasping the piece in ones palm, so that the back of the fist was covered by the curved or dorsal sector of the piece (González and Regueiro 1969; González 2006). We know that it was gripped in this manner given that several similar examples have been found with the leather preserved around what would be the grip area (Table 2).

The San Francisco site is located at an elevation of 4,000masl in Chaschuil's transitional puna (Fig 1,8). Its architecture is Inca, composed of two compound perimeter enclosures. Abundant ceramic and bone assemblages as well as a metal flat-tipped chisel were recovered from excavation (Fig 3E). The pottery pieces found were for storage and food-serving (*aribalos*, aribaloid, and duck effigy plates) used in the consumption and sharing of food and alcoholic beverages in state-sponsored ceremonies (Orgaz *et al* 2007; Lantos *et al* 2015; Miyano *et al* 2017). The site's location is linked to the Incahuasi Volcano summit ascent route (6638masl), where a high-altitude Inca sanctuary with offerings was

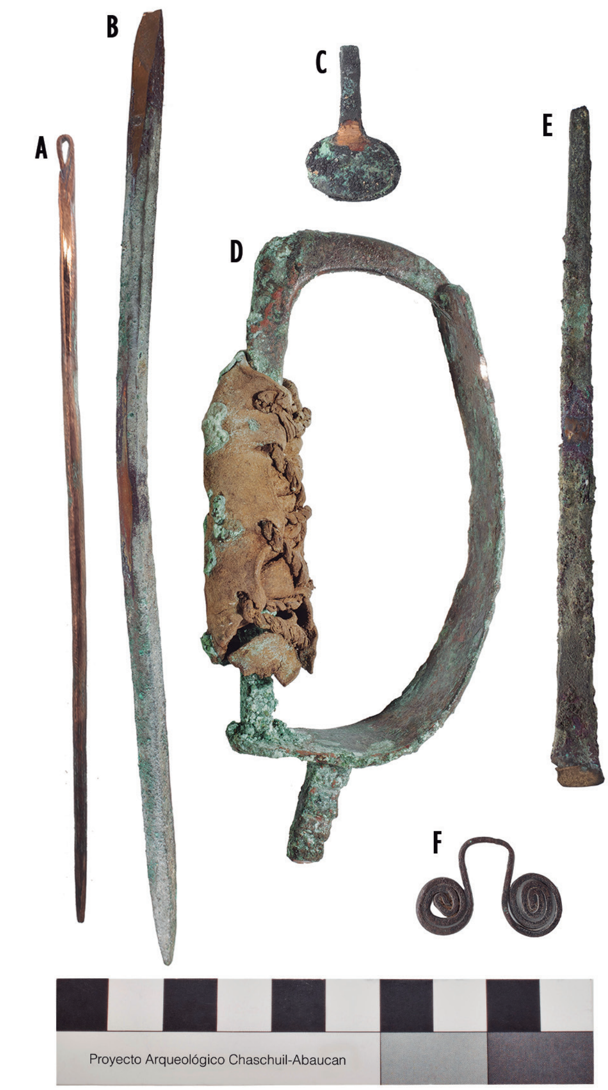

Figure 3: Metal objects studied in this paper: A) needle, B) burin chisel, and C) tweezers, from Palo Blanco settlement; D) knuckleduster from Burial LP-II; E) flat and smooth-tipped chisel from the San Francisco Inca site; F) nose ring from the Mishma-2 site. Scale 100mm.

located (Orgaz and Ratto 2015). Finally, the Mishma-2 site, Mishma archaeological site, was situated 1,700 masl in the Zanjón de Apocango within the Fiambalá valley (Fig 1,4). It was excavated by Sempé (1976) who, given the nature of the abundant and diverse archaeological evidence uncovered (fire-pits, pre-Inca ceramic, domestic camelid bones, macro-plant remains and a piece of metal), interpreted it as the residence of an extended family. Radiometric dating places the site in the period of Inca presence in the region (Table 2). The metallic object found was a piece of double coiled wire which functioned as a nose ornament (Fig 3F).

Minerals and metals from western Tinogasta

As we have shown, in western Tinogasta copper is found in mines located in the highlands (Los

Aparejos, La Lampaya, Cerro de Las Minas) and in the mesothermal valley (Pachamama), the latter located within the Los Ratones Mining district (Fig 1,E). These mines are spatially distant from one another (Fig 1B-1D). Gold and silver ore are ubiquitous in the La Hoyada polymetalliferous district, Cordillera de San Buenaventura (Fig 1A). Tin is much more widely dispersed throughout the Fiambalá and Zapata Sierras, in one case tin was found in association with copper. This mountainous area constitutes the natural corridor between western Tinogasta and the eastern valleys (Belén). It is important to note that the Río de Inca site was the only one with evidence for metallurgical production and it was located in a place between these two mountainous areas, closely associated to the presence of copper and tin (Fig 1,7).

The objects recovered from the region were predominantly of copper or copper alloyed with tin. Those objects in both public and private archaeological collections from funerary contexts had diverse forms and functions, including pectorals, bracelets, knuckledusters, nose ornaments, ornaments, needles, tweezers, and chisels. The association of these objects with other materials dates them to the Late and Inca Period. The exception to this pattern were the residential and/or metallurgical production sites excavated by Max Uhle, which dated to the period of social mixing between local communities, the Inca and Spanish colonial conquerors. In contrast, metal objects recovered systematically through archaeological study belong to varied socio-political productive societies (1st and 15th century CE) where copper continued to dominate. These objects came from funerary and multi-functional residential contexts, although they are the same types of objects found in the collections described above, namely needles, chisels, tweezers, knuckledusters, and nose ornaments. In the case of Palo Blanco, the settlement site dating to the first millennium, evidence of refractories in the shape of crucibles were uncovered suggesting small-scale *in situ* work.

Analytical methodology

A sample of six objects were analysed (Table 2). Analysis consisted of a visual inspection of the objects' surfaces, some of which were covered with greenish corrosion products. Subsequently, a metallographic, microstructural analysis was undertaken, either on the surface (after removing the patina, polishing the surface as set out in ASTM E3-11 (2017), and etching it with a suitable reagent, as recommended by Caron *et al* 2004) or on a small fragment cut from the object. Images were generated using a scanning electron microscope (SEM). The chemical composition of the objects was determined using energy dispersive Xray spectrometry (EDS). For this purpose, we used a 20x Arcano stereoscopic magnifying glass; a Carl Zeiss inverted stage reflection optical microscope (Axio Vert A1 MAT model) with a Carl Zeiss camera (model ERc5s); a JEOL JSM6510LV SEM equipped with an EDS probe (model X-MaxN-50mm^2); and a Philips 505 SEM with an EDAX CDU-UTW probe. These studies allowed us to characterise the construction methods by revealing the sample microstructure and the composition of the objects (Table 3).

Table 2: Provenance and dating of the objects analysed

Settlement or archaeological site	Metal object	Sector and provenance	C-14 dates (years BP) and calibrated (CE)	Other metallurgical evidence
Aldea de Palo Blanco settlement (1900m asl)	Burin chisel 4418	Midden south of NH-4, Strat D-F	1940±60 BP 44-203 CE	none
	Needle 3616	NH-1, Hab. A	Median = 1704±69 BP 252-467 CE	none
	Tweezers 3632	NH-5, Hab. 2	Median = 1392±46 BP 643-764 CE	mould, slag
Las Papas-II burial	Knuckleduster DA05-13/115	Grave	720±50 BP 1281-1384 CE	none
San Francisco-Inca	Chisel 5953	Structure 12	570±50 BP 1392-1442 CE	none
Mishma settlement	Nose ornament 5954	Mishma-2	500±50 BP 1410-1462 CE	mould, slag (settlement level)

Note: The C-14 dates were calibrated using Program Calib 7.0.4 curve Shcal13.
Calibrated dates are all given at one sigma.

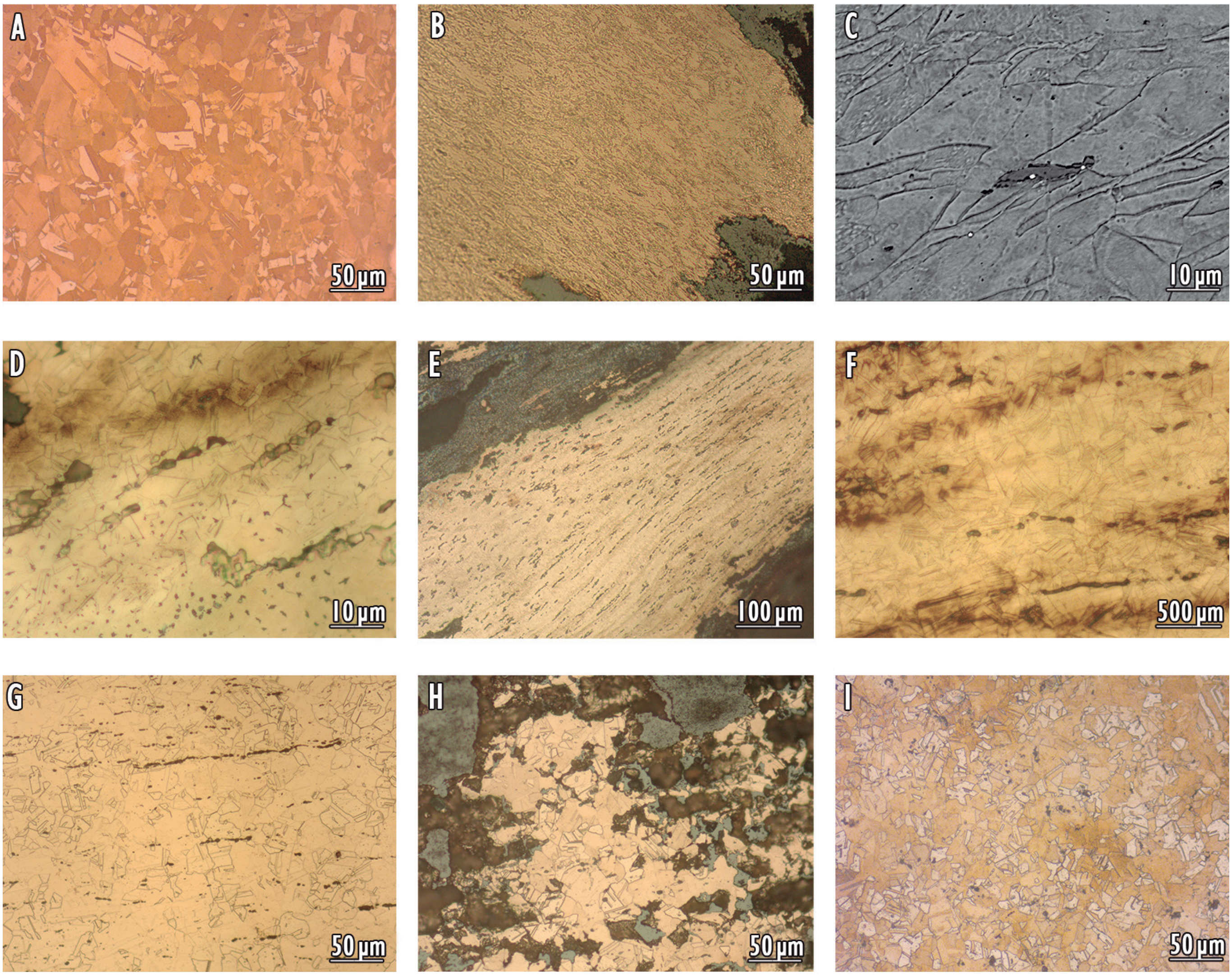

Figure 4: Metallographic sections etched using NH_3OH, H_2O_2 solution. All except C are optical images. Burin chisel 4418: A) centre, B) tip, C) tip (BSE image). Needle 3616: D) body, E) body-eyelet, F) eyelet. Tweezers 3632: G) handle and jaw, H) corroded area and I) non-corroded area.

Results

Samples from Aldea de Palo Blanco

The settlement yielded three pieces for analysis: a needle (3616, Fig 3A), a burin chisel (4418, Fig 3B) and a pair of tweezers (3632, Fig 3C). They dated to different periods in the life cycle of the settlement, the first two were from the 1st-5th centuries while the last dated to the 7th-8th centuries (Tables 2 and 3).

The burin chisel was covered by a small layer of greenish corrosion products. It had a total length of 180mm, curved slightly possibly due to use, and was rectangular in section (6.2 x 5.3mm at the centre), tapering towards both ends (2 x 3.5mm at its thinnest); it weighs 46.5g. The microstructure of the central area presented equiaxed solid solution grains of mixed size with recrystallization twins (Fig 4A), while the tip presented elongated grains consistent with a cold plastic deformation process (Fig 4B). Elongated grains with deformed recrystallization twins and non-metallic inclusions were observed on the object's tip (Fig 4C). The base metal composition was copper, while that of the inclusions was copper sulphide.

The needle was roughly circular in section and had a reddish-brown metallic surface with no apparent corrosion products. It was 149.0mm long, weighed 8.5g, had a maximum diameter of 2.9mm, tapering towards the tip where it had an average diameter of 1.3mm. The microstructure was a solid solution of equiaxed grains with longitudinally-aligned dark-grey non-metallic inclusions. The grain size varied, being larger in the body (Fig 4D), medium-sized in the eyelet section (Fig 4F) and smaller in the body-eyelet joint area (Fig 4E). The grains had recrystallisation twins, which indicated that it was manufactured using

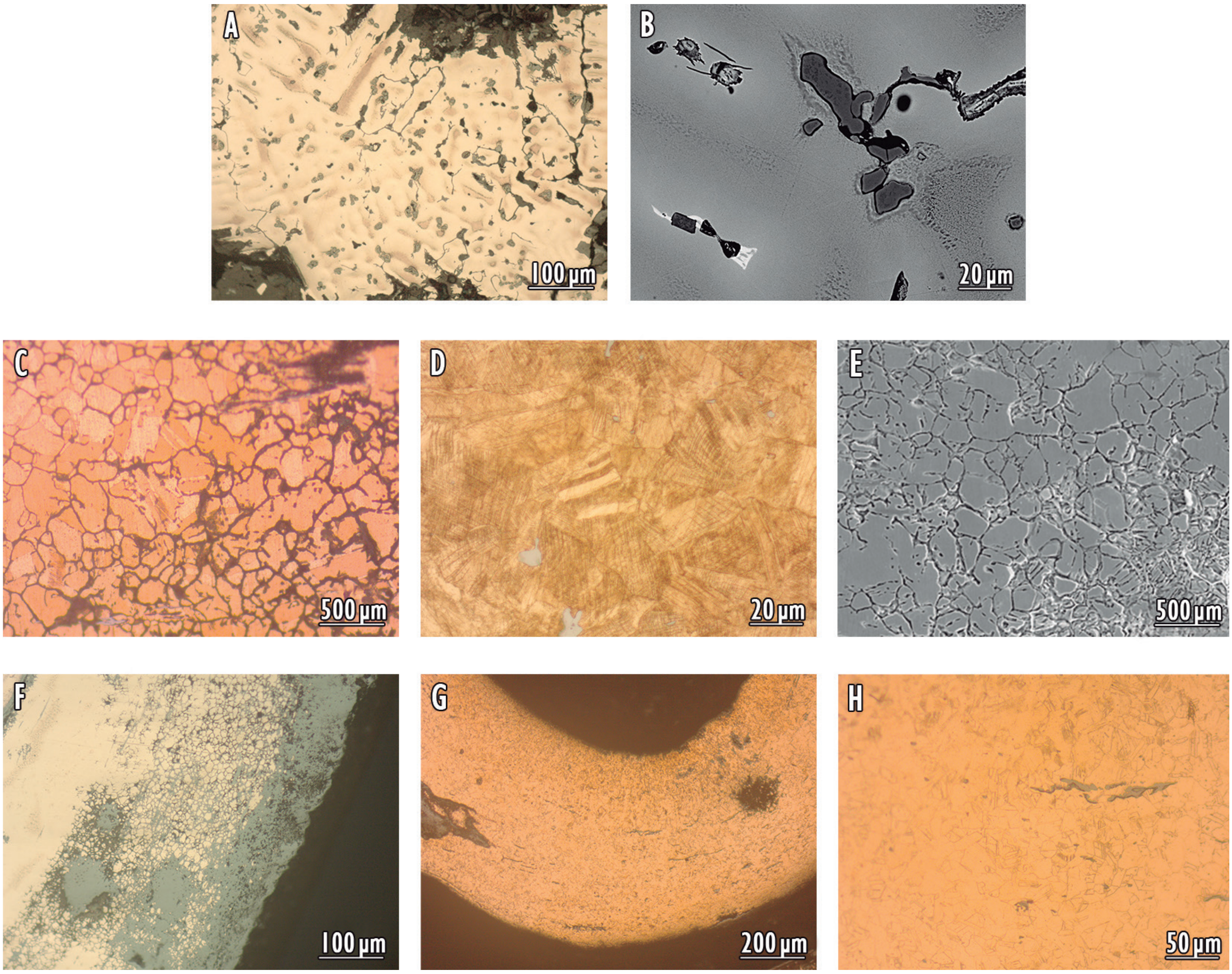

Figure 5: Metallographic sections etched using NH_3OH, H_2O_2 solution. All except B and E are optical images. Knuckleduster DA5-13/115: A) As-cast dendritic microstructure with secondary phases, B) inclusion and the base metal (BSE image); Chisel 5953: C) centre, D) tip; E) tip (SE image); Nose ornament 5954: F) end-area of one of the spirals showing corrosion, (G) and (H) microstructure of the revealed piece.

Table 3: Chemical composition of bulk metal and inclusions (normalised wt% EDS analyses)

Object	**No**	**Composition**										**Fig**
		Cu	**Sn**	**As**	**Bi**	**Al**	**Si**	**Pb**	**Sb**	**S**	**Fe**	
Burin chisel	4418	98.5				1.2	0.3					3a, 4a-c
		78				*<1*				*21.3*		
Needle	3616	91.5		5.5	2.3	0.7						3b, 4d-f
		48.3		*25.3*	*22.7*				*3.7*			
Tweezers	3632	98.5				1.15	0.35					3c, 4g-i
		17.5						*82.0*			*<1*	
Knuckleduster	DA5-13/115	90	9.7								<1	3d, 5a-b
		77.0								*20.5*	*2.5*	
Chisel	5953	91.5	8.5									3e, 5c-e
		82.5	*4.5*							*13*		
Nose ornament	5954	95	4.5				<1					3f, 5f-h
		87.2	*3.9*				*0.8*			*7.3*	*0.8*	

Notes: The data for the inclusions are shown in italics below that for the bulk metal.
The upper three samples are 1st-10th century and the lower three are 13th-15th century.

a process of cold plastic deformation with subsequent recrystallization, or by alternate stages of deformation and heating. The EDS analysis showed the needle was made of copper with 5.5% As; lower quantities of Bi and Al were also detected, the latter most likely the result of sample contamination during the preparation stage as polishing was carried out using alumina suspension. All non-metallic inclusions were complex, with high concentrations of As and Bi, and a small amount of Sb, which were considered products of the reaction of these elements with Cu or with each other. While Sb was only detected in inclusions, Bi was also detected in the bulk metal (but only because the analysis was carried out near an inclusion). Both of them are usually a minor component of copper minerals, and Bi is considered deleterious because it causes embrittlement of the metallic product. Sb and Bi were only detected at the trace level, in small amounts in relation to the alloying elements; we therefore considered their presence to have been unintentional.

The surface of the tweezers was covered with corrosion products, greenish on the outside, with a reddish-brown internal substrate adjacent to the surface of the metal itself. This was concentrated around the finger support and fold section of the piece. The artifact weighed 2.5g and had a total length of 28.8mm, composed of oval shaped jaws (largest radius = 1.6mm) and rectangular handles 5.2mm in length and 1.44mm average thickness. The microstructure was made up of equiaxed grains and recrystallization twins without subsequent plastic deformation. The grain size throughout the entire piece was homogeneous (40-50µm; Figs 4G-4I). There were non-metallic inclusions aligned in the longitudinal direction of the handles. In some areas there was advanced intergranular corrosion with dark grey corrosion products. The SEM-EDS analysis of the object revealed that it was made of unalloyed copper with inclusions with high lead (74.4wt%) and oxygen (9.4wt%) contents in addition to traces of iron (0.6wt%).

Samples from the 13th-15th centuries

The three pieces analysed were the knuckleduster (DA5-13/115, Fig 3D), the flat-ended chisel (5953, Fig 3E) and the nose ring (5954, Fig 3F). These three pieces came from sites with different functions and locations (Tables 2 and 3).

The knuckleduster surface was completely covered by a thick layer of greenish oxides. Its total length was 117mm, and the palm area of rectangular section was covered with treated leather and a textile cord; the dorsal section had a thickness of 1.8mm along its most curved area. The microstructure of the piece consisted of a solid solution with dendritic growth (as-cast condition) and chemical micro-segregation (Fig 5A). It had grey non-metallic inclusions that contained Cu sulphide (Figs 5A5B) and showed intergranular corrosion. The object was made of a Cu-Sn alloy with a tin content of around 10%, with local variations in composition due to chemical micro-segregation.

The chisel had a rectangular section, was 124.8mm long and weighed 15.2g; its surface was covered by a thin layer of greenish oxides. The microstructure along the central zone presented equiaxed grains and recrystallization twins, without evidence of final cold plastic deformation; it also had elongated grey-coloured inclusions and intergranular corrosion (Fig 5C). At the end it showed recrystallized grains with growth twins and slip bands, the product of cold plastic deformation, in all likelihood the result of a manufacturing process or percussion work events (Fig 5D). SEM-EDS results indicated that it was made of bronze and that the non-metallic inclusions were sulphidic (Fig 5E).

The nose ornament came from one of the sites connecting the mesothermal valley with Chaschuil's transitional Puna. Its surface was dull and covered with dark green corrosion products, it weighed 1.37g and consisting of a rectangular-sectioned wire (1.1 x 1.2mm) wound into two linked spiral parts that formed counter-revolved, flat, three-coiled structures with an approximate diameter of 10mm. Intergranular corrosion was detected in the external zone of the spiral (Fig 5F). The microstructure corresponded to a solid solution, with equiaxed grains, slightly deformed in the folds of the turns (Figs 5G-5H). The grains also had recrystallization twins associated with heat treatment following a cold forming process. A few light-grey non-metallic inclusions were observed. According to the EDS analysis results, the piece was made of tin bronze with traces of Si (Table 3). The corroded areas also contained oxygen (14.6wt%) associated with other corrosion products including Si (2.1wt%), Fe (0.4wt%), As (1.2wt%) and Cl (1.2wt%). These last four elements were detected only in the corroded area and are not considered alloy components.

Discussion

The six objects analysed were probably manufactured using local copper or copper alloyed with arsenic or tin. These alloys are the most widespread ones within NW Argentina but it is interesting that they occur at different times in the Prehispanic history of western Tinogasta.

The results of the metallographic and SEM-EDS analysis performed on the burin chisel, the needle and the tweezers show differences in composition and manufacture throughout the 1st millennium, possibly related to the function of the pieces. The burin chisel and the needle were made using cold plastic deformation by which a ductile metal acquires hardness and strength as it is worked. In the case of the needle, it had subsequent recrystallisation, or it was subjected to alternate deformation and heating phases. However, they differ in the composition of the metals from which they were manufactured. The needle was made with arsenical copper, while the chisel and the tweezers with unalloyed copper. In this sense, it is interesting to note that within the broad timespan of the 1st millennium CE, the needle and the chisel belong to the first half, prior to 500 CE while the tweezers date to the 7th-8th centuries. Therefore, we can tentatively suggest that there was a functional knowledge of alloys from an early period. In this regard, the amount of As detected in the needle was crucial in lending it greater strength as the As contributes to relative hardening of the alloy when it is present above 3.7% (Scott 2011). The elements present in the piece (Sb, Bi and As) may have been contained within the naturally occurring Cu minerals (Liddel 1945), and in our region Bi and Sb are present in the La Hoyada mining district (Table 1; Fig 1A).

The Cu-As alloy used for the needle, and its subsequent hardening, was probably intentional given the need for a sharp point (Ziobrowski *et al* 1996). This means that its manufacture may be non-local since there is no record of arsenical copper in our region. On the other hand the tweezers and the burin chisel have compositions compatible with the copper mineral resources of western Tinogasta. The curvature in the body of the burin chisel may be a product of its use, since its hardness and strength was low in comparison to the flat and smooth-ended chisel manufactured with Cu-Sn alloy at a later date (see below).

In contrast, the objects dating to the 13th-15th centuries CE present distinct characteristics regardless of their use, their recovery contexts, or the different socio-political organizations within which they existed and materialized their relationships. The first difference, with respect to the previous millennium, is the use of Cu-Sn alloys for the analysed pieces. The presence of tin-bronze is a constant within the metallurgy of NW Argentina for this period. Although its use originates during the earlier Middle Period (5th-10th centuries CE), its use peaks between the 10th and 15th centuries CE. Later, the skills and artisanship of these local metallurgists were exploited by Inca administrators for state purposes (González 2008).

In the case of the knuckleduster, our analysis indicated that it was manufactured by casting as a single piece of bronze without subsequent plastic deformation. The raw material employed sulphidic Cu. Due to its shape, the absence of flash lines at the plane of symmetry, and the uniformity of the filling, we do not rule out the possibility of it being cast by the lost wax method. In this regard, there is consensus among researchers that metal bells (Fig 2D) in the region were also made using this technique (González 2006; 2008). In contrast, the flat and smooth-ended chisel and the nose ornament were created using plastic deformation with thermally-activated recrystallization, employing a Cu-Sn alloy where the presence of sulphide inclusions may indicate the use of sulphidic raw materials such as chalcocite (Cu_2S) or chalcopyrite ($CuFeS_2$) (Oudbashi and Hasanpour 2016), both present in mineral deposits from the region.

The assemblages from later periods prior to the Spanish conquest could also have been manufactured with this Cu-Sn alloy, given the colouring presented by some objects, such as the *tumi* and bell (Figs 2A, 2D). In other cases, we can only compare in relation to the artefact's dimensions and formal characteristics, such as the chisels, needles, and nose ornament from the Pereira Collection (Figs 2E, 2I, 2K). It is intriguing that none of the chisels in the collection shows deformations of the body, indicating that they may date to the Late-Inca Period when we have the presence of the Cu-Sn alloy that conveys greater hardness to the objects, as is the case of the flat and smooth-ended chisel from the San Francisco site (Fig 3E). Additionally, both the LP-II knuckleduster and the one from the Huanchin cemetery (in the Inca Huasi Museum) were from regional funerary contexts, albeit located more than 100km apart.

The physical presence of tin in the region is not trivial. In other regions of NW Argentina, for example in the Quebrada de Humahuaca (Jujuy), it is argued that the supply of tin was secured through the use of camelid caravans that brought the mineral from other highland areas (Angiorama 2006). In this sense, González (2002) maintains that the metallurgists of the Rincón Chico workshop, Yocavil Valley (Catamarca), obtained the tin required for the manufacture of bronze objects from the Fiambalá and Zapata mountain ranges, more than 140-170km from their workshop. The supply of raw materials, especially tin, was most certainly a major factor linking the different sectors of the Valladolid area of Catamarca, such as Yocavil and Fiambalá. This exchange of raw materials can be traced through other types of evidence that highlight these interregional connections, such as the presence of pottery from the Yocavil area in regional funerary contexts (Dreidemie 1953; Ratto and Basile 2020).

We know that the complexity and variety of the social processes involved in the production and circulation of metal objects during the Prehispanic period requires a study of each socio-historical context. Nevertheless, sometimes the general characteristics and particularities of one area are relevant to understanding the wider pattern. In the case of western Tinogasta, despite the intense survey work undertaken, no tunnels, galleries and/or trenches were identified in association with tools related to copper and tin mining activity. However, these Prehispanic features may have been destroyed by subsequent reworking of the mines during the first half of the 20th century, as well as through collapse or landslides (Turner 1967). Faced with this unknown, we must once again rely on the Max Uhle collection and his field-books. In these he reported on the importance of the Río del Inca metallurgical site for the Inca and Hispano-indigenous period, and on the wide range of metals present (copper, silver and gold sheets, copper slag, silver and iron) in addition to the copper objects. Furthermore, the site is located at a strategic position both for the supply of Cu and Sn minerals and to facilitate onward links with the eastern valleys (Belén). The artefactual quantity and richness of the materials in the collection give a clear indication of the importance that this site held in the past (Ratto 2015). The scale of this site is a clear counterpoint to other sites of more limited overall area such as Mishma-2 and Palo Blanco. The latter, dating post-500 CE, is associated with metal objects, moulds and slag (Sempé 1976). Both sites may demonstrate a family scale of metallurgical production.

Conclusion

The number of samples analysed using archaeometric methods was small; however, it allowed us to address metallurgical activity over a wide time range, from the 1st to the 15th centuries CE. In this period, we have the development of agropastoral village communities during the 1st millennium CE, followed by late pre-Inca societies that were then impacted by the Inca Empire. It is within this complex framework that the Spanish conquest unfolds.

In the 1st millennium we find common objects relating to work (needle and chisel) and personal care (tweezers). These were made using unalloyed copper or, as in the case of the needle, with a small amount of added As, suggesting that it was not an object of local manufacture. In the first centuries of the 2nd millennium CE the objects are also common and relate to personal possessions, such as the knuckledusters and nose ornaments, as well as work tools (chisel) uncovered at funerary and residential sites. All the pieces were made by plastic deformation and thermally activated recrystallization, except for the knuckleduster, which was probably a lost wax casting.

Our studies have allowed us to present an initial interpretation of the metallurgical technology of western Tinogasta, a region where the use of copper predominated over that of silver or gold. Combining the objects from collections, their associated written records, the survey of the region's economic geology, and archaeometric analyses allowed us to define the production system of metal objects across the different socio-political contexts in which they existed. This approach also made it possible to outline the role western Tinogasta played in the development of metallurgy in the Catamarca area of Valladolid. In this sense, Tinogasta would have been a major supplier of tin, a raw material of vital importance for the artisans who manufactured elaborate metal objects with bronze, such as those in the Yocavil Valley.

Research in NW Argentina demonstrates that it was in the central and eastern valleys of the province of Catamarca where systematic experimentation in the working of copper and its alloys began. This specialisation was undertaken by village-level societies during the 1st millennium CE. In turn, this was a situation closely related to processes of social inequality, demand for prestigious goods and the development of cultic-religious practices in which metals held a prominent place (González 2008). It is possible that western Tinogasta was the main supplier of tin, and that this metal was the key to the development of complex of social, economic, and political relations.

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