

# Experimental report

22/02/2022

**Proposal:** 1-03-48

**Council:** 4/2020

**Title:** Neutron diffraction analysis of Roman coins from Hispania: methodological approach to compositional and minting characterization

**Research area:** Other...

**This proposal is a new proposal**

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**Samples:** CuSnPbAgNa

Instrument	Requested days	Allocated days	From	To
D1B	3	2	09/03/2021	11/03/2021
D20	2	0		

## Abstract:

We propose to perform a neutron diffraction study on Roman coins (Cu-Sn-bronze) selected from the August-Tiberian period emissions, as well as those of the Caligula's government; a crucial stage in the coinage production in the Iberian Peninsula. The objective of the analysis is to quantify the composition of the coin, without need to eliminate the historical patina, and to characterise the manufacturing process. Metal was worked in a forge, and beaten with a hammer or, in this case, a stamp, this causes the crystalline networks of the metals to be reoriented, generating a characteristic pattern called texture. Neutron analysis is non-destructive and provides essential data for numismatic research: bronze composition/contamination, state of conservation ( % Cuprite, Chalconatronite, Atacamite) and metallurgical data related to minting technology and manufacturing processes like whether the metal flans were hot-, cold-struck or even reheated after minted. Overall it will provide for the first time metallurgical information which in addition to classical stylistic criteria will constraint official and imitation mints during this period in Hispania.

**Experimental report 1-03-48 @ D1B\_ILL** 09/03/2021 To 11/03/2021

## **Neutron diffraction analysis of Roman coins from Hispania: methodological approach to compositional and minting characterization**

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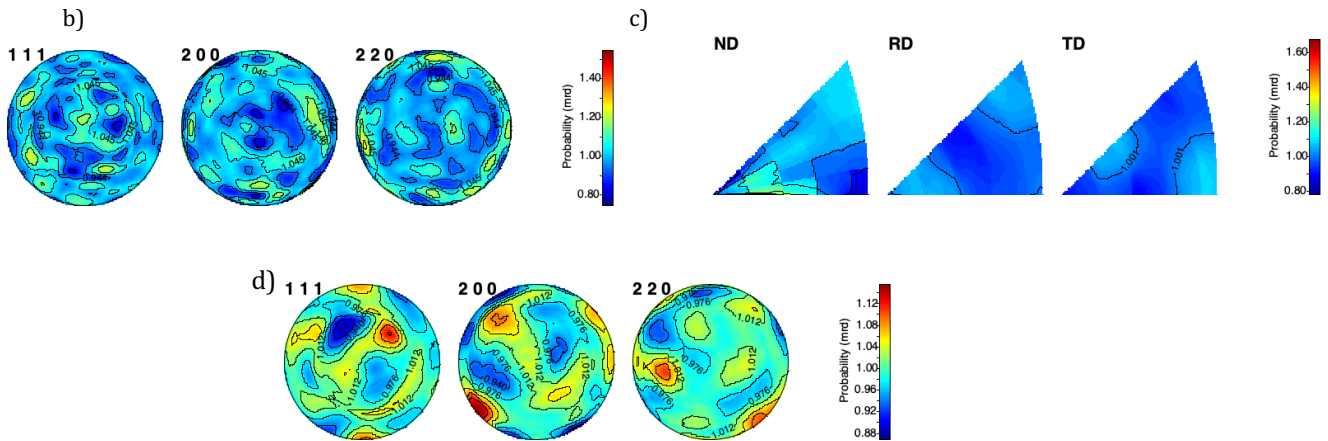
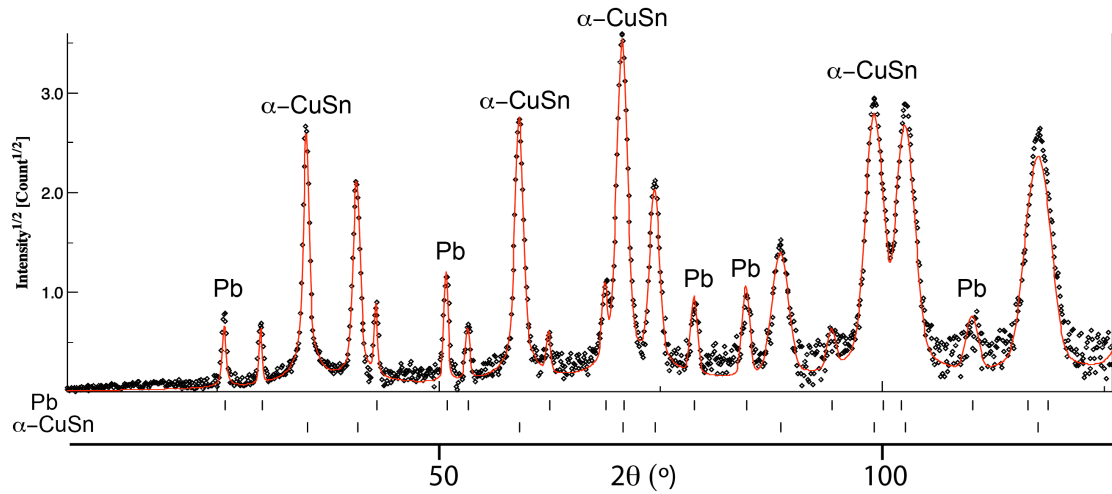
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We have conducted a neutron diffraction study on Roman coins (Cu-Sn-bronze) selected from the August-Tiberian period emissions, as well as those of the Caligula's government; this is a crucial stage in the coinage production in the Iberian Peninsula. The objective of the analysis was, on the one hand, to quantify the composition of the coin, without need to eliminate the historical patina, with an accuracy limit of 0.5-1%, [1] and by another, to characterize the manufacturing process. This last aspect is derived from the study of the preferential orientation, or texture, of the crystals of the metals that make up the coin [2, 3, 4, 5, 6, 7]. Coin manufacture results in texture and particular textural components are identified according to the process temperature and deformation associated with minting [8]. With neutron analysis we have obtained some essential data for numismatic research, including the origin of the materials (e.g. Phase composition, Pb additions/contaminations, Zn content in the case of 'official' issues), state of conservation (e.g. % Cuprite, Chalconatronite, Atacamite) and in some cases the minting technology and manufacturing processes like whether the metal flans were hot-, cold-struck or even reheated after minted (e.g. [9]). Our experiment has provided for the first time metallurgical and compositional data (e.g. Pb%), which in addition to classical stylistic criteria define official and imitation mints for this period in Hispania, defining the methodology for future investigations.

**Experiment:** The proposed experiment aims at quantify coin composition and texture in Cu-Sn-Bronze roman coins from Spain. Crystallographic preferred orientations of phases will be done using the approach already tested at ILL (e.g. 183\_d1b\_exp\_CRG-2559) or elsewhere for polycrystalline samples [11, 12]. Each coin was mounted in transmission on an specifically designed V-holder and measured with a scan grid of 10° by using 4-circle goniometer. Wavelength used was 1.28Å. Thanks to the low absorption of neutrons, acquisition time was, on average, 10 s per spectrum, resulting in 360 measured scans per sample ( $\phi$ : 0→355°;  $\chi$ : -90→0°).  $\omega$  angle was set at 45° to make use of the detector 2 $\theta$  full range (0-128°). In house standards (NAC and Si) were measured to refine experimental parameters. Texture standards were used for symmetry control. Raw data was converted into \*.FIB format with macro d1b\_2\_FIB at LAMP for latter refinement at MAUD (Lutterotti et al, 1999; Benítez Pérez, 2017). Quantitative texture analysis was done in Rietveld software package MAUD, computing ODF using E-WIMV. Due to the non-standard shape of coins, absorption models have been tested. Selected pole figures and inverse pole figures were recalculated and rotated to show the reference system (strike-plane).

In the Figure 1a, an example of total spectrum with experimental (dots) and Rietveld model (solid red line) from an Augustus coin is showed, with a 14 wt % Pb and 86%  $\alpha$ -bronze CuSn. Pole and inverse pole figures of Bronze and interstitial Pb are showed (Figs. 1b-d).

a)



Calculated pole and inverse pole figures suggest a combination of uniaxial striking and recrystallization in most of the samples, with some exceptions in which more work is needed to characterize the metallurgical process.

## Conclusion

Neutron diffraction is an excellent technique to analyze texture of metallic archeological artifacts. In particular, coins recorded metallurgical imprint both in texture and composition so that systematic analysis of large populations would be critical to identify the evolution of coinage technology in the past, including both imitation paths and modern falsifications. Our results highlight for the first time the textural variations, i.e. manufacturing variations (?), between two emissions in Iberia.

## References

- Siano et al. (2006). *Archaeometry*, 48(1), 77-96.  
 [1] Artioli et al. 2007. *Appl. Phys. A* 89, 899–908  
 [2] Corsi, et al. (2016). *Microchemical Journal*, 126, 501-508.  
 [3] Nagler et al. (2019). *Journal of analytical methods in chemistry*.  
 [4] Kocks et al. 1998. *Texture and anisotropy: preferred orientations in polycrystals and their effect on materials properties*. Cambridge university press.  
 [5] Salvemini et al. 2018. *Journal of Archaeological Science: Reports*, 20: 748–755.  
 [6] Sidot et al. 2005. *Materials Science and Engineering: A*, 393:147–156.