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Understanding the manufacturing techniques of Renaissance armour: a coupling between metallographic and SR-XRD investigations

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From the late Middle Ages onwards, complete suit of armour is a highly manufactured product, composed of a multitude of ferrous alloys plates skillfully articulated to protect its wearer. Studying the nature of metal used to make such pieces is of great interest for historical research, especially for the understanding of manufacturing techniques, including how materials were produced and traded by ancient societies. To decipher the structure of alloys, metallography is a very efficient technique but required to take samples on the artefacts. Furthermore, to be representative of the ancient ferrous metals that are very heterogeneous, it is in many cases necessary to get several samples on the same piece. As sampling possibilities are limited in case of museum artefact, non-invasive techniques as X-ray diffraction (XRD) are well suited to identify the different phases, characteristic of ancient ferrous alloys, such as ferrite and cementite (Fe₃C). Nevertheless, such experiment presents analytical challenge due to the complex shape of armours. A specific methodology was therefore developed to conduct XRD measurements under synchrotron radiation at the SOLEIL-DiffAbs beamline to study fifteen pieces of armour, all stamped with the mark of Valentin Siebenbürger's workshop, armourer in Nuremberg at the beginning of the 16th c. This was a unique opportunity to assess the nature of the metal of artefacts made in a same workshop, thanks to the collaboration of the Musée de l'Armée (Paris, France) and the Musée des Beaux-Arts of Rennes (Rennes, France). The study presents and discusses the methodology developed for XRD measurements to determine the spatial distribution of mineral phases in ferrous alloys directly on large and complete museum armour pieces. Thanks to the very high brightness of the synchrotron source and the fast acquisition time using the pixel-hybrid 2D detector CIRPAD, it was possible to multiply the analyses on the same object in order to estimate the variability of the alloys used (carbon content, heat treatments...) at the scale of separate plates. Heterogeneities related to the nature of the metal, revealed by variations in the intensity and shape of certain peaks (as for cementite and ferrite phases), were highlighted. These results were coupled and discussed with metallographic investigations performed on a few samples and raise new questions about the manufacturing processes of the armours.