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Micro-computed tomography and laser micro-ablation on altered pyrite in lapis lazuli to enhance provenance investigation: a new methodology and its application to archaeological cases

Marta Magalini[1,2], **Laura Guidorzi**[1,2,a], **Alessandro Re**[1,2], **Francesca Tansella**[1,2], **Federico Picollo**[1,2], **Sofia Sturari**[1,2], **Pietro Aprà**[2], **Georgina Herrmann**[3], **Randall Law**[4], **Quentin Lemasson**[5,6], **Laurent Pichon**[5,6], **Brice Moignard**[5,6], **Claire Pacheco**[5,6], **Alessandro Lo Giudice**[1,2]

[1] Dipartimento di Fisica, Università di Torino, Via Pietro Giuria 1, Turin, Italy
[2] INFN Sezione di Torino, Via Pietro Giuria 1, Turin, Italy
[3] University College London (UCL), Gower Street, London WC1E 6BT, UK
[4] Department of Anthropology, University of Wisconsin, 5240 Sewell Social Sciences Building, 1180 Observatory Drive, Madison, USA
[5] Centre de Recherche et de Restauration des Musées de France, C2RMF, 14 quai François Mitterrand, Paris, France
[6] UAR 3506 Lab-BC (CNRS, Ministère de la Culture, Chimie ParisTech), 14 quai François Mitterrand, Paris, France

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Abstract This work presents an upgrade to the methodology adopted to investigate the provenance of the raw lapis lazuli material used in antiquity for carving precious artefacts. Samples from archaeological excavation contexts frequently display superficial degradation processes affecting the crystals of the mineral phases useful for provenance attribution (especially pyrite). To address this issue, an innovative workflow has been developed, centred on the application of X-ray micro-computed tomography (μ-CT) and micro-ablation treatments with a pulsed laser source prior to investigation with ion beam analysis (IBA). High-resolution μ-CT is employed to evaluate the alteration state of pyrite crystals within the entire volume of the lapis lazuli rock, and, if required, to identify the most suitable crystals on the surface for subsequent laser treatment. The micro-ablation procedure aims to create a small breach in the superficial altered layer (the irradiated areas are approximately $65 \times 65\ \mu m^2$), thereby exposing the preserved crystal beneath and allowing for the analysis of its trace element contents with IBA. The methodology of the workflow is presented, together with its first application to archaeological lapis lazuli material: three precious beads from the ancient Royal Cemetery of Ur (Mesopotamia, 3rd millennium BCE). The results are complemented by the application of a provenance protocol already validated that proved, for the first time using a micro-invasive analytical approach, a match between the Afghan quarry district and the raw material used to carve these beads.

1 Introduction

Lapis lazuli is a semi-precious stone that has been used since the 5th millennium BCE for the carving of valuable items and decorative objects, including jewels, amulets, statuettes and inlays. Archaeological findings made by this rock have been discovered in a vast territory, from the ancient Middle East and Egypt to Central Asia, especially in sites of the 3rd millennium BCE, when the greatest popularity of this material occurred. Regarding the sources of the rock, due to the very restricted geological environment necessary for its formation, there are only few sites on Earth where lapis lazuli can be extracted. Although the Badakhshan quarries in Afghanistan are widely considered to be the only sources of lapis lazuli in ancient times, others have been cited or suggested [1–5]. Obtaining information on the provenance of the raw material of archaeological artefacts could, therefore, provide very interesting indications on the most active mining areas at a given time, the distances travelled to trade this material and, indirectly, the socio-economic links between the populations that traded it.

Investigating the lapis lazuli provenance is a complex challenge due to the high heterogeneity of this rock, which consists of an aggregate of different minerals (more than 30 phases in lapis lazuli have been documented in the literature [6, 7]). The most characteristic mineral is lazurite, an alumino-silicate of the complex feldspathoid sodalite group, which confers the typical blue colour that has fascinated many populations throughout antiquity. Additionally, minerals, such as pyrite, diopside, phlogopite, calcite, K-feldspar and numerous others, can occur with varying abundances. The characterisation of the compositional and luminescence properties of some of these mineral phases (in particular, diopside, wollastonite and pyrite) with ion beam analysis (IBA) has demonstrated to be an effective method for differentiating rocks from different extraction sites [8, 9]. The same procedure is being

[a] e-mail: laura.guidorzi@unito.it (corresponding author)

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