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Multi-disciplinary study of the carbonaceous polymorphs associated to the 4 kyr BP burnt soil-strata tracing the 4-kyr BP impact-ejecta

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The solid expertise long achieved in meteoritic mineralogy based petrography, mineralogy, crystallography and geochemistry has greatly helped to study impact-linked minerals on Earth, in terms of nature (i.e. high pressure vs. high temperature) and origin (i.e. impactor vs. target materials). In contrast, elucidating the extraterrestrial vs. terrestrial origin of carbonaceous phases and their modes of formation during impactprocesses (i.e. vapour vs. solid phase interaction) is often an analytical challenge.

We report here the strategy we have elaborated to study the complex carbonaceous polymorphs encountered in the distinctive 4 kyr BP burnt soil-strata with its unique suite of impact-ejecta debris. The research has been conducted on the most well-preserved soil records of the 4 kyr BP impact in various regions of France and Syria. In spite of their geological diversity, these contexts display the similar association in discrete pods of carbonised local vegetation, baked pedo-sediments and impact-debris

that occur either as concentrations of micro-debris, or as fist-sized blocks, or as a semicontinuous, vesicular, mammilated melt-crust. Undisturbed clods showing the close imbrication of the intact impact debris with the host baked-matrix have been carefully sampled. Organic and mineral compounds have been extracted under the binocular microscope at successive stages of water-sieving and well-controlled chemical treatment. Contact with industrial products (i.e. silver paper, abrasive paste or paper, nylon films, polymer resins, diamond paste or spray, detergent) was in general prohibited. A first examination of clods and slices using the environmental SEM-EDS under BSE has allowed to study the 3D- and 2D-geometry of the organic and mineral phases, together with their raw chemistry. Then, their mineralogy, crystallography, and their chemical, structural and isotopic compositions were accurately defined using various techniques: XRD, Raman micro-spectrometry, SEM-EDS, HRTEM, EELS, GC-IR-MS, isotopes (C, O, S, Pb, Fe, Cr), noble gas. A Renishaw's Raman structural and chemical analyser (SCA) for SEM has been used for identifying the micro-scale 3Dpattern of organic vs. mineral phases and simultaneously performing Raman analysis on carbon-containing micro-domains.

The integrated field-analytical procedure has allowed to establish that the 4 kyr BP impact-ejecta comprises a wide range carbonaceous and mineral phases associated to a micro-faunal assemblage (foraminifera, diatoms and radiolaria) from subtropical, subpolar and austral seawaters. The micro-faunal and analytical data indicate a common origin of the impact-ejecta from multiple target sources in the Southern hemisphere. The allochtonous carbonaceous polymorphs have been subdivided into (1) intact oil shales with mineral inclusions; (2) amorphous carbon in the form of droplet-clusters and vesicular vitreous carbon glass with graphitic nanotubes resulting from moderate heating of the oil shale precursor in the ejecta; (3) green carbonaceous filaments rich in carbides and with chaoite nano-platelets that were formed in the ejecta possibly from unknown terrestrial precursors and/or cosmic materials; (4) a metal/diamond/graphite/hydrocarbon association disseminated within the crystallised impact-products that indicates complex interaction of the solid, liquid and gas phases derived from the mineral and the organic terrestrial precursors and the extraterrestrial components during the ejecta emplacement.

Occurrence within the local carbonised vegetation and the baked aggregates of the oil-shale derived amorphous carbon has helped to establish a direct linkage between the explosive pulverisation of rocks yielding a metal-rich carbonaceous melt and local ignition at the host soil surface. Evidence for mixing of the allochtonous and local carbon was important to explain the few too early ¹⁴C radiocarbon ages. This unique suite of carbonaceous polymorphs offers robust criteria to solve the confusing morphological resemblance of the 4 kyr BP impact by-products with organo-mineral phases

produced by human smelting, lightening strikes, wildfires or pyrometamorphism.