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Laboratory evidence for co-condensed oxygen- and carbon-rich meteoritic stardust from nova outbursts

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Although their parent stars no longer exist, the isotopic and chemical compositions and microstructure of individual stardust grains identified in meteorites provide unique constraints on dust formation and thermodynamic conditions in stellar outflows[1–5]. Novae are stellar explosions that take place in the hydrogen-rich envelope accreted onto the surface of a white dwarf in a close binary system[6]. The energy released by a suite of nuclear processes operating in the envelope powers a thermonuclear runaway, resulting in the ejection of processed material into the interstellar medium. Spectral fitting of features observed in the infrared spectra of dust-forming novae provided evidence of the co-condensation of both carbonaceous and silicate dust in stellar outflows within 50 to 100 days after explosion[7–9]. Although novae appear as prolific producers of both carbon- and oxygen-rich dust, very few presolar grains that can be attributed to novae have been found in meteorites thus far[10–16]. Here, we report the identification of an oxygen-rich inclusion, composed of both silicate and oxide nanoparticles, inside a graphite spherule that originated in the ejecta of a low-mass carbon- and oxygen-rich (CO) nova. This observation establishes laboratory evidence of the co-condensation of oxygen- and carbon-rich dust in nova outbursts and is consistent with large-scale transport and mixing of materials between chemically distinct clumps in the nova ejecta.

Novae are traditionally classified as neon or non-neon, reflecting the composition of the white dwarf that hosts the explosion, that is, oxygen- and neon-rich (ONe) or carbon- and oxygen-rich (CO), respectively[6]. Recently, a uniquely ^{13}C- and ^{14}N-rich presolar graphite grain was identified in situ in the primitive carbonaceous chondrite, LaPaz Icefield (LAP) 031117. This grain, called LAP-149, is about 1 µm wide (Fig. 1) with a croissant-like shape (Supplementary Fig. 1a) and is characterized by one of the largest ^{13}C enrichments ($^{12}C/^{13}C = 1.41 \pm 0.01$) ever measured in any circumstellar graphite[17]. Comparisons of its carbon, nitrogen, silicon and sulfur isotopic compositions with stellar nucleosynthesis model calculations and equilibrium dust-condensation models suggest an origin in the ejecta of a low-mass (0.6 solar masses) CO nova[17]. Unlike all other putative nova grains (for example, SiC, silicates and oxides) that are only qualitatively consistent with direct nova model predictions, the isotopic compositions of LAP-149 match both quantitatively and qualitatively CO nova model predictions, providing strong evidence for graphite condensation in nova ejecta[17]. However, both $^{16}O/^{17}O$ and $^{16}O/^{18}O$ ratios are within solar values for LAP-149. This oxygen isotopic composition is inconsistent with CO nova models, which predict both large enrichments in ^{17}O and depletions in ^{18}O ($^{16}O/^{17}O$ at least 7.5 times lower than solar and $^{16}O/^{18}O$ at least 60 times higher than solar)[6,12].

A recent study re-examined the isotopic ratios produced by thermonuclear runaways on CO white dwarfs, using a Monte Carlo technique that involves a random sampling over the most relevant nova model parameters[18]. It identified, among all the previously reported nova grain candidates, including LAP-149, those that are most likely to have formed in CO novae[18]. Iliadis et al.[18] assumed a grain to have a CO nova origin if all of its measured isotopic ratios are in quantitative agreement with the model predictions (without assuming any dilution of the ejecta). Their new calculations identified six grains deemed most likely to have originated in CO novae[18]. We note that they are all SiC, for which the oxygen isotopic compositions were not measured. In comparison, all the grains with the lowest likelihood of a CO nova origin are those for which oxygen isotopic ratios were measured[18]. In other words, there is a systematic discrepancy in the oxygen isotopic compositions between all putative nova grains, including oxygen-rich grains (silicates and oxides), and stellar nucleosynthetic models[17,18]. It has been suggested that this discrepancy might reflect equilibration of oxygen isotopic compositions by parent-body alteration or laboratory processing (for example, acid dissolution), or mixing of the nova ejecta with solar composition material[10,12,13]. However, in the case of LAP-149, the NanoSIMS (Nanoscale Secondary Ion Mass Spectrometry) and Auger nanoprobe data did not show any sign of oxygen diffusion or an isotopic gradient into the grain, and LAP-149 was identified in situ in a thin section of a minimally altered carbonaceous chondrite, thus excluding any possible isotopic dilution due to laboratory processing[17,19]. Furthermore, chemical maps of an electron-transparent section of LAP-149, obtained by collecting energy-dispersive X-ray spectra (EDS) using scanning transmission electron microscopy (STEM), do not show any sign of oxygen diffusion inside the

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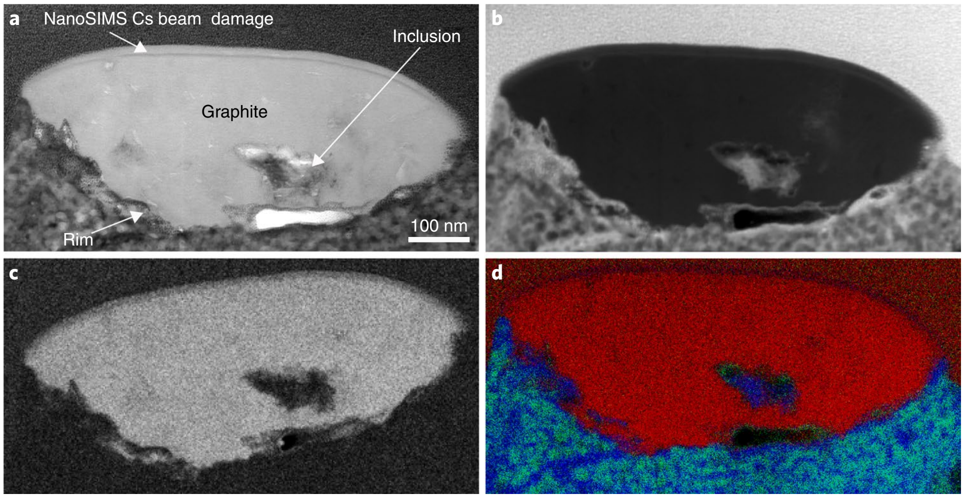

Fig. 1 | STEM data of LAP-149. a,**b**, Bright-field (**a**) and annular dark-field (**b**) images of LAP-149. **c**, EELS three-window C-K edge map of LAP-149, showing the carbon distribution. **d**, False-colour composite EDS elemental maps (carbon, red; oxygen, blue; silicon, green). The scale bar in **a** applies to all of the panels.

graphite spherule (Fig. 1). The fact that oxygen diffusion from surrounding matrix material and oxygen isotopic equilibration from laboratory treatments and/or alteration processes can be definitely excluded for LAP-149 shows that additional computational models and astronomical observation of nova ejecta are required to reconcile laboratory measurements of all putative nova grains with model predictions for oxygen isotopes.

STEM phase-contrast lattice-fringe imaging of LAP-149 and a presolar graphite shows that LAP-149 is composed of nanocrystalline graphite with an interplanar distance (d_{002} spacings = 0.34 nm, where '002' is the Miller index) consistent with graphite (Supplementary Fig. 2). This observation is also consistent with the comparison of the electron energy-loss spectroscopy (EELS) C-K near-edge structure of LAP-149 and two other presolar graphite grains (Fig. 2).

High-resolution secondary electron, bright-field and annular dark-field imaging show the presence of an inclusion inside LAP-149, measuring approximately 170 nm × 70 nm (Fig. 1). Both the EELS maps and EDS spectrum images show that the inclusion is composed of oxygen-rich material (Figs. 1 and 3). Furthermore, the heterogeneous distribution of silicon and oxygen across the inclusion suggests that it is an aggregate composed of silicate and oxide grains. Electron-nanodiffraction patterns of distinct regions in the inclusion show that the silicate and oxide grains are nanocrystalline aggregates (Fig. 4). The EDS data reveal that the oxides are iron- and aluminium-rich, and the silicates are ferromagnesian in composition (Fig. 3).

On the basis of spatial relationships, this inclusion must have formed before the host graphite, LAP-149, in the same nova ejecta, as it is completely surrounded by the graphite grain (Figs. 1 and 4). In addition, the inclusion could not have filled a crack or cavity in the graphite spherule, because it progressively appeared during the focused ion beam (FIB) thinning process on both sides of the section, confirming that the inclusion was entirely enclosed inside its host graphite. Different types of refractory inclusions have been reported inside presolar SiC and graphite grains[1,20], but, to our knowledge, not silicate and oxide grains inside a carbonaceous grain. This observation is not consistent with traditional equilibrium thermodynamic calculations of dust condensation in circumstellar environments that predict the formation of carbonaceous grains in carbon-rich stellar envelopes with carbon/oxygen > 1 and silicate and oxide dust in oxygen-rich envelopes with carbon/oxygen < 1. Instead, our detection of nanocrystalline silicate and oxide

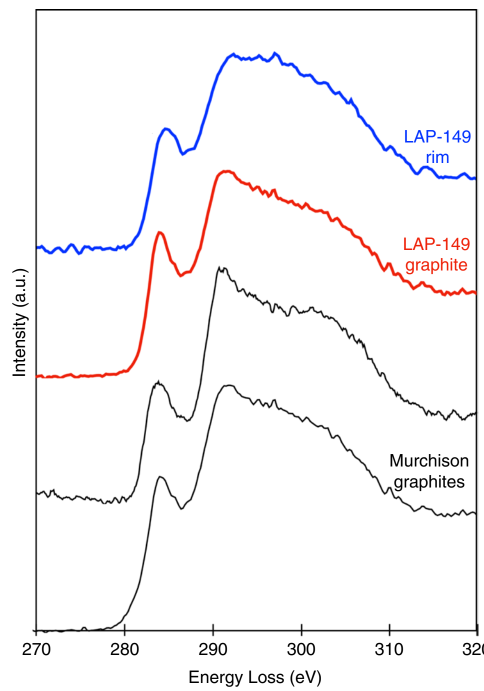

Fig. 2 | Energy-loss near-edge structure of presolar graphite grains. Comparison of the C-K edge energy-loss near-edge structure of LAP-149 (red), the carbonaceous material in its bottom coating layer (blue) and two Murchison presolar graphite grains (black: upper, KFC1b2-gr1; lower, KFC1b2-303).

grains inside a graphite spherule is consistent with astronomical observations over periods of several years in CO nova ejecta that report production of both carbon-rich (for example, graphite, SiC and amorphous carbon) and oxygen-rich (for example, silicates and oxides) dust[7–9]. The coexistence of both carbon- and oxygen-rich material also provides clues on the condensation conditions (that is, kinetic versus equilibrium) and dust mixing in an explosive environment, and reduces the critical importance of the carbon/oxygen ratio as the main criterion.

The nanocrystalline silicate and oxide inclusions in LAP-149 provide ground-truth laboratory evidence of the co-condensation

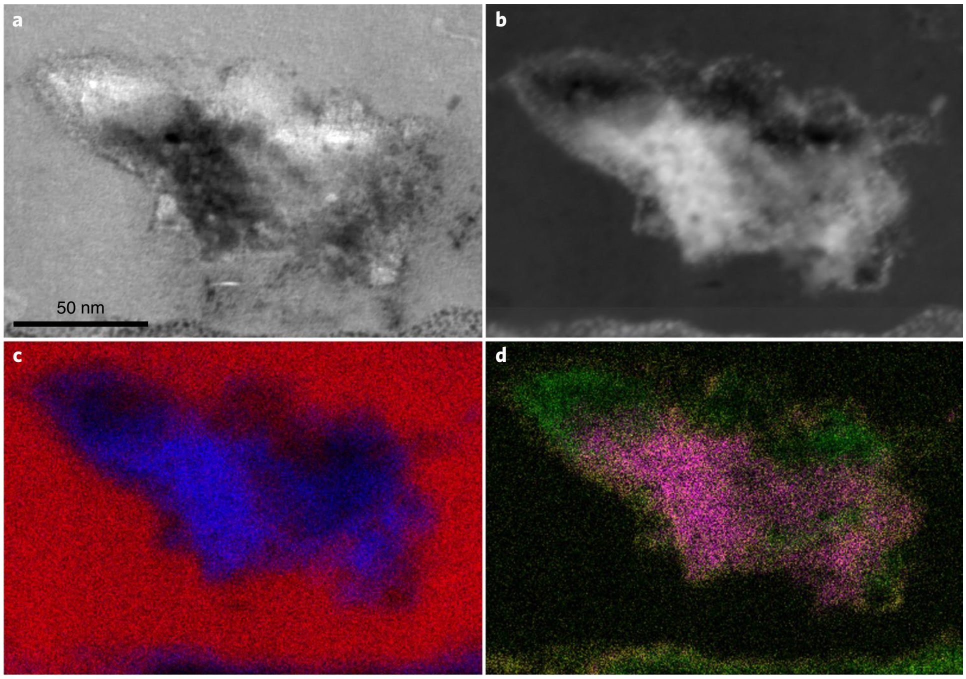

Fig. 3 | STEM imaging and EDS data of the inclusion inside LAP-149. a,**b**, STEM bright-field (**a**) and annular dark-field (**b**) images of the inclusion inside LAP-149. **c**,**d**, EDS false-colour composite elemental maps of the inclusion inside LAP-149 (oxygen, blue; carbon, red (**c**); iron, pink; silicon, green; magnesium, yellow (**d**)). The scale bar in **a** applies to all of the panels.

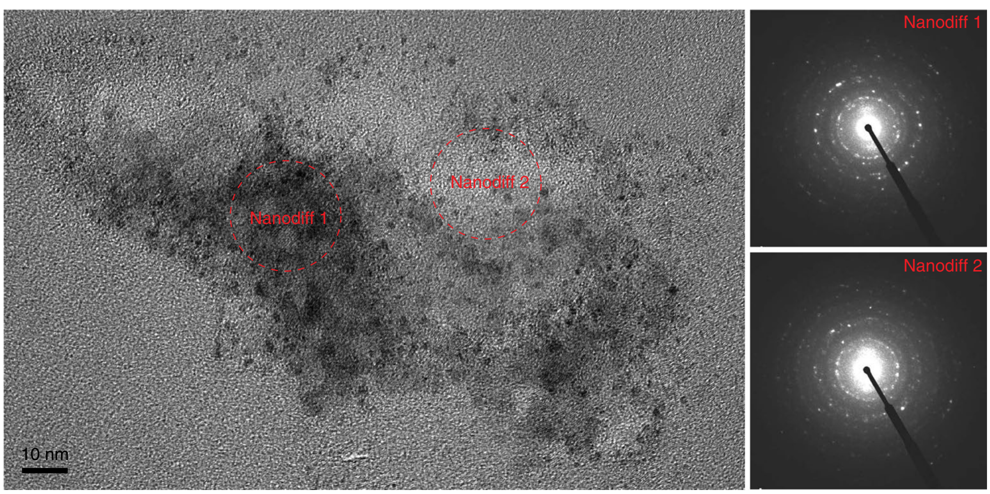

Fig. 4 | TEM imaging and electron-nanodiffraction data of the inclusion inside LAP-149. 'Nanodiff 1' and 'Nanodiff 2' show the diffraction patterns of the nanocrystalline oxides and silicates, respectively.

of oxygen-rich (silicate and oxide) and carbon-rich (for example, graphite) grains in CO nova outbursts. These observations thus suggest that either the nova ejecta have temporal or spatial heterogeneities with distinct clumps or layers of carbon- and oxygen-rich material, or the nova outbursts are substantially asymmetric with different ejecta compositions in different directions[21,22]. Such asymmetries were previously observed. For example, multi-wavelength spectroscopic observations of several novae, such as observations of the GK Persei nova ejecta with the Hubble Space Telescope, the Chandra X-ray Observatory and the Very Large Array, provide evidence of the asymmetric structure of the nova ejecta and show that clumps of material are ejected during nova outbursts[23]. The chemistry of nova outbursts depends on characteristics of the binary star system, such as the nature of the white dwarf core (CO- or ONe-rich) and the composition of the gas mixture between the white dwarf surface and the accreted material from the envelope of the companion star[24].

If our observation reflects temporal variations in the nova ejecta, LAP-149 and its silicate-oxide inclusion could have formed in the same clump either under equilibrium, with variation of the carbon/oxygen ratio over time, or under kinetic conditions where all the oxygen is not tied up in CO gas, allowing the concurrent condensation of both oxygen- and carbon-rich dust grains. However, this latter hypothesis is not consistent with astronomical observations of dust condensation in nova ejecta that suggested that carbon- and oxygen-rich dust formed in different parts of the nova outburst, and carbon-rich grains formed first before the condensation of silicates[7–9]. LAP-149 and its inclusion thus probably formed in distinct clumps of the nova ejecta (with oxygen- and carbon-rich compositions, respectively). In this case, the observation of the silicate-oxide inclusion inside LAP-149 indicates dust mixing and transport between distinct clumps in the nova ejecta.

Although observations of most CO nova ejecta are consistent with formation of silicates after carbonaceous dust in the ejecta[7–9], Smith et al.[21] provided evidence that the silicate emission observed in the ejecta of Nova Herculis 1991 could reflect a light echo from silicate grains formed in a previous nova outburst that was then mixed with a fraction of the new ejecta. In this scenario, the nanocrystalline silicate-oxide inclusion observed in LAP-149 could have formed in a previous nova eruption and was then mixed into a carbon-rich region of the new outburst, where graphite condensed around it. However, it is not clear whether this scenario can be directly applied to our observation, as Nova Herculis 1991 is a ONe nova, and whether such mixing would be possible, given the long recurrence times (about 1 Myr) between two consecutive outbursts for a CO nova of 0.6 solar masses.

Either way, our data provide evidence that both carbon- and oxygen-rich dust and large-scale transport could have occurred in a circumstellar environment. This result goes against the currently accepted picture of carbon- and oxygen-rich dust grain condensation under different conditions and therefore suggests that the dynamics of novae should be re-examined.

All of the current nucleosynthesis model predictions for novae rely on one-dimensional simulations. Recent preliminary work on three-dimensional hydrodynamic simulations of the interaction between the nova ejecta, accretion disk and stellar companion suggests that, within one hour of the nova outburst (before the condensation of dust grains), a fraction of the ejecta might collide with the accretion disk and/or the envelope of the companion star[25]. Other three-dimensional simulations suggest that turbulent convection might be responsible for the observation of highly fragmented and heterogeneous nova ejecta and could explain the transport of heterogeneous material across the ejecta[26,27]. Such processes would affect only a small fraction of the ejecta and could provide an explanation for the transport and mixing of material between carbon- and oxygen-rich clumps in the nova ejecta[26,27]. In this context, our discovery of an oxygen-rich inclusion, composed of nanocrystalline silicate and oxide grains, in a presolar graphite spherule further supports inhomogeneities, mixing and transport of dust in the ejecta and is consistent with a formation model where the nanocrystalline iron- and aluminium-rich oxides and ferromagnesian silicates condensed first in a previous nova outburst or in an oxygen-rich region of the ejecta, and the silicate-oxide inclusion was then transported to a carbon-rich region of the ejecta, where nanocrystalline graphite condensed around it.

Bright-field and annular dark-field images also reveal the presence of two different rims on the top and bottom surface of the grain (Fig. 1). Whereas the top layer reflects Cs^+ ion beam damage due to the NanoSIMS measurements, the rim on the bottom surface of the grain, in direct contact with the surrounding matrix material, was not exposed to either the NanoSIMS analyses or any other laboratory processing. EDS mapping shows that this rim is composed of a mixture of carbonaceous material and oxygen-rich material with an elemental composition consistent with a ferromagnesian silicate (Supplementary Fig. 3). EELS measurements of the carbonaceous material in the rim indicate that it is mostly composed of aromatic amorphous carbon (Fig. 2). Because LAP-149 was identified in a pristine (unequilibrated) carbonaceous chondrite that experienced only minimal secondary processing, such as aqueous alteration and thermal metamorphism, the rim does not reflect secondary processing on the meteorite parent body[19]. Instead, the rim probably reflects processes in the nova ejecta or grain surface processing in the interstellar medium. Although irradiation of the graphite spherule in the interstellar medium could explain the formation of a rim composed of amorphous carbon on the surface of the graphite grain, it would not explain the mixture of both amorphous carbon and ferromagnesian silicate material. Another possibility is that the rim formed during processing in the parent nova environment. In addition to SiC, graphite and silicates, infrared spectroscopic observations of nova ejecta have suggested the presence of amorphous carbon and aromatic organic nanoparticles[7,9,22]. The surface coating on LAP-149 could then reflect the condensation of amorphous carbon and ferromagnesian silicate nanoparticles on the surface of the grain within the nova outburst.

Methods

An electron-transparent cross-section of grain LAP-149 was prepared following previously described FIB techniques[28], using a dual-beam Helios NanoLab 660 FIB/scanning electron microscope (Supplementary Figs. 1 and 4). In brief, platinum fiducial markers were first deposited on top of the grain using electron-beam deposition. Using ion beam deposition, a protective C strap (or capping layer) was then deposited on the top surface of the cross-section to mitigate any potential damage (for example, Ga^+ ion implantation and amorphization) during the sputtering process. The section was then cut and extracted from the meteorite thin section using an EasyLift micromanipulator system. Finally, the section was mounted onto the transmission electron microscopy (TEM) grid and progressively thinned to electron transparency (<100 nm), using a range of accelerating voltages and current. Final ion polishing was performed at 5 keV and 0.68 nA to remove any potential surface damage caused by higher-voltage milling.

We then carried out coordinated high-resolution STEM imaging (secondary electron, bright field and annular dark field), EDS and EELS measurements of the grain, using a 30 kV Hitachi SU9000 scanning electron microscope/STEM. The SU9000 is equipped with an Oxford Instruments X-Max 100LE EDS detector and Hitachi EELS system. All measurements were carried out with a 30 kV accelerating voltage. For comparison, we also acquired EELS spectra of two presolar graphite grains (KFC1b2-gr1 and KFC1b2-303) extracted from the Murchison meteorites. Hole-count EDS spectra acquired in the vacuum just above the FIB section show Cu and Al system peaks (Supplementary Fig. 5), confirming that all other elements identified in the sample are indigenous to the grain. On the basis of the EELS low-loss spectrum (zero-loss and plasmon peaks) of LAP-149, we estimated a t/λ ratio of about 0.55 (where t corresponds to the sample thickness and λ is the local inelastic mean free path), corresponding to a thickness of about 35 nm. This estimate was calculated using the Microscopy and Microanalysis Tool Set[29]. We tested the accuracy of this method by measuring the t/λ ratio of microtome sections of cyanoacrylate standard with a known thickness (30 nm).

Using a Gatan Be double-tilt high-resolution holder, we also acquired additional EDS and electron nanodiffraction patterns of the O-rich inclusion in LAP-149, using a Hitachi HF5000 200 keV STEM/TEM to obtain further information on its nanoscale microstructure and to confirm the presence of Al in the inclusion (Supplementary Fig. 6).

Finally, we carried out additional NanoSIMS ion raster imaging of carbon and oxygen isotopes (^{12}C, ^{13}C, ^{16}O, ^{17}O and ^{18}O) of the FIB section of LAP-149, using a focused Cs^+ primary beam of ~0.3 pA that was rastered over the section (covering a surface of 6 μm × 6 μm and 256 pixels × 256 pixels) with a dwell time of 1 ms per pixel. Unfortunately, despite using a low primary ion beam current, the FIB section started to fold onto itself as soon as it was exposed to the Cs^+ primary ion beam, so we were not able to obtain isotopic measurements of the inclusion. This was probably due to the extremely low thickness (35 nm) of the section.

Data availability

The data that support the plots within this paper and other findings of this study are available from the corresponding author upon reasonable request.

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Author contributions

P.H. prepared the FIB sample cross-section, carried out the TEM and NanoSIMS measurements and wrote the manuscript. J.Y.H., K.K., T.S., A.M. and T.J.Z. helped with the TEM analyses. T.J.Z., S.A., K.L. and J.J. contributed to the data interpretation. All authors contributed to the manuscript.

Competing interests

The authors declare no competing interests.

Additional information

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