

Improved instrumental techniques, including isotopic analysis, applicable to the characterization of unusual materials with potential relevance to aerospace forensics

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ABSTRACT

The problem of precise characterization, analysis, and eventual identification of unknown materials arises in many fields and takes many forms, depending on the nature of the substances under study. In the first part of this paper we review common, modern mass spectrometry techniques applied to such studies. We also give an overview of improvements made to these technologies in recent years by Silicon Valley companies and other teams focused on precise biomedical research dependent upon sensitive techniques, yet applicable to a wide range of non-biological materials. In the second and third parts of the paper we review practical experiences applying these techniques to the simplest case of the characterization of solid materials (as opposed to liquids or gases) and comparing our results with previously undertaken isotopic analysis. In particular, we describe our correlations of that analysis with the patterns described by witnesses in a well-documented, still-unexplained incident, initially thought to be of aerospace origin, which gave rise to the deposition of unknown material, and by the investigators who handled it in the field and the laboratory. The lessons from this specific investigation are applicable to a wider range of issues in reverse engineering of complex, esoteric materials, and aerospace forensics.

1. Introduction

The problem of precise characterization, analysis, and eventual identification of unknown materials arises in many fields – ranging from archaeology to meteoritics, medicine, law enforcement, nuclear forensics, space exploration and national (US and foreign) intelligence agencies. In materials analysis of metals and other inorganics, the approach can take many forms, depending on the nature of the substances under study. In general, one starts with basic constituents such as elemental composition, moving onto studies of internal structure if any.

In the first part of this report, we review common analytic procedures, including modern mass spectrometry, X-Ray spectroscopy, and certain other techniques that are often initially applied for such studies. We also provide an overview of improvements made to one of these technologies in recent years by investigators that focused on precise biomedical research with sensitive techniques yet are still applicable to a

wide range of non-biological materials. This development was driven by that fact that most materials are not ‘uniform’ in their composition and therefore techniques that allow for 2D and 3D analysis of materials are key to both understanding as well as quality control.

For example, an insight brought from the biological research arena is that spatial patterns (for instance, of cells) in a tissue are critical determinants of outcomes in disease states (whether certain immune cell types are within a given radius of a tumor or pathogen). Spatial component organization is key as well in design of microelectronic circuitry. As such, in the analysis of novel materials whose function will depend on local changes in elemental composition (wiring or semiconductors), the use of spatially sensitive instrumentation adds great value. In addition, while isotopes of elements are often considered equal in functional attributes for most purposes, subtle distinctions in properties of isotopes relative to spin states of nuclei, utility in quantum entanglement systems, and even pharmaceutical effects have been apparent in recent years (detailed below).

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In the second and third parts of this report we review practical experience applying some of these techniques to a simple case of the characterization of a solid material (as opposed to liquids or gases) while comparing our results with previously undertaken isotopic and elemental analysis.

We describe correlations of that analysis with the patterns described by witnesses in a well-documented, still-unexplained incident, initially thought to be of aerospace origin, which gave rise to the deposition of an unknown material, and by the investigators who handled it in the field and in the laboratory. The lessons from this specific investigation are applicable to a wider range of issues in reverse engineering of complex, esoteric materials, and aerospace forensics.

We apply these insights to a case of a material derived from an unidentified aerial object as observed by multiple independent witnesses. We apply the standard, and new, techniques as outlined herein as an example of how the analysis of such materials can be addressed. We detail the elemental and spatial differences in elemental and isotopic composition of subsamples from the parent sample CB_JV-1 at 50 nm resolution. The results show differences in the elemental distribution based on 5 subsample grains, suggesting the parent sample is inhomogeneous. For each of the subsamples the elemental composition was homogeneous to a depth of ~50 nm. Notably, there were no significant isotopic differences from terrestrial normal in the subsamples, and thus the overall sample could have been made with terrestrial-derived materials. That said, the CB_JV-1 sample itself remains of unknown provenance or function.

2. Basic approaches for the initial characterization of unknown materials

There are multiple technologies for determining the chemical makeup of complex materials. The analytic approach that is to be taken is largely determined by the general nature of the material under analysis. With materials of completely unknown makeup, and starting from the smallest scale, one can begin with identifying the elements, such as hydrogen, carbon, iron, copper, and so on, and determining their relative abundances within a sample. While for most analytic purposes the

isotopes of a given element are not relevant, the ratios of the stable isotopes in an analytic sample can also be determined using sufficiently sensitive mass spectrometry instrumentation.

If one is interested in local chemical bonding structures, alloys, nanomaterials and organic constituents, then more complex analytic approaches are warranted such as nuclear magnetic resonance ("NMR"), electron microscopy, scanning transmission electron microscopy (STEM), X-ray diffraction XRD, back-scatter, among others. An overview of a retinue of currently available analytic procedures, and their utility, is shown in Fig. 1.

We will detail the principal approaches used in the current analysis, with a focus on mass spectrometry, with the understanding that more advanced analysis is warranted by the initial findings on the current case and several others under investigation by the authors.

2.1. Materials analysis techniques using ICP-MS and SIMS

The two principal techniques we have applied so far in our investigative work with unknown materials are known as ICP-MS (Inductively Coupled Plasma Mass Spectrometry) and SIMS (Secondary Ion Mass Spectrometry). Both techniques are based on determination of the masses of the atomic components of a material. They operate principally by ionizing the atoms in such material and passing those ions into an instrument that determines their mass, usually by time of flight ("TOF"), magnetic sector, or by other means that separate them based on mass and charge of the atom.

These two techniques have distinct, and important, applications. ICP-MS generally accomplishes isotopic measurements of materials dissolved in a liquid, typically in monoatomic form. SIMS can undertake similar measurements in two and three dimensions in an intact material with resolutions down to at least the 25 nm per axis scale, and under certain circumstances, at 5 nm per axis scale. This latter capability might be key in understanding how elements and isotopes are spatially distributed throughout a given material. While we document here some significant technical uses for both technologies, it is also important to recognize their limitations in specific analytical settings, given the varied nature of the samples under study. The approaches discussed

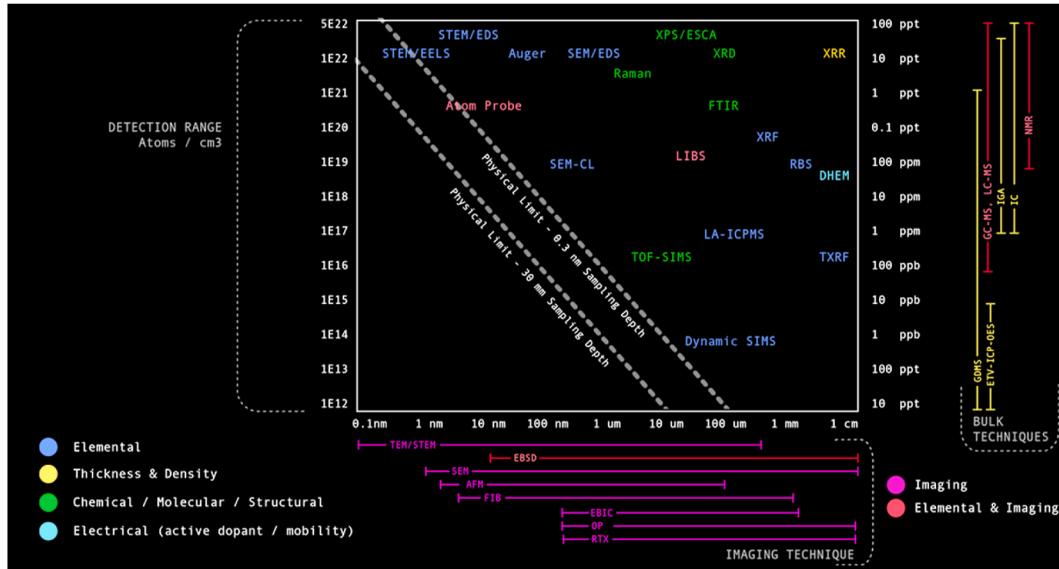


Fig. 1. Overview of techniques for materials analysis. The color legend represents the functional utility of the technique. The center of the graph provides the names of techniques often used in materials analysis. The X axis represents the scale of size of the same in xy dimensions and shows in purple the general techniques and the range across which they are useful. The Y axis left hand side represents the numbers of atoms measurable in cubic centimeters as a metric. The Y axis on the right side represents the same data but as a measure of sensitivity in terms of parts per thousand (ppt top), parts per million (ppm), parts per billion (ppb), and parts per trillion (ppt bottom). On the right side are the instruments and techniques and the range across which they are sensitive. Adapted from <https://www.eag.com/techniques/>. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

have a long history and acceptance in analysis of metals and alloys [1–3], organic and inorganic materials [4,5], to detail everything from isotopic constituents [6], materials structure, and material contaminants [1–3,5,7,8]. Given this resolution limit, neither approach allows for the direct determination of the molecular or local atomic structure of any given material. Atomic localization requires measurements on the scale of 2–5 angstroms (where 1 angstrom = 0.1 nanometer) and can be carried out by procedures such as X ray diffraction [9] or atom probe tomography [10], which are additional specific techniques useful in analysis.

The result of such an analytic procedure is a table of the relative ratios of the elements, and their isotopes, across a given mass range. Depending on the instruments, that can include everything from hydrogen up through the heaviest of the elements. With the most sensitive instruments and appropriate calibration, one can tell the difference between, say, ⁶⁴Zinc (34 neutrons and 30 protons, or as the linear formula 64Zn34) and ⁶⁴Nickel (36 neutrons and 28 protons, or as the linear formula 64Ni36). ⁶⁴Zn has an atomic mass of 63.729145 and ⁶⁴Ni has a mass of 63.9279679 — a difference of 0.1988 AMU (Atomic Mass Units).

Instruments capable of measuring the composition of materials to such accuracy are useful in a variety of contexts. For instance, stable (non-radioactive) isotopes are used as tracers that can serve as atomic barcodes indicating the origin of materials. Similarly, small differences in certain isotope ratios of given elements can determine the origin of a gemstone (e.g., African, or South American), or whether a meteorite had an origin from the inner solar system, outer, or beyond [11,12]. Isotope abundances can also be used to trace manufacturing processes and more recently is used in tagging of monetary currencies to prevent counterfeiting.

Interestingly, isotopes can influence a variety of important biological, chemical, and electronic functions. From a clinical perspective, Lithium—widely recognized for its effect in psychiatry—has been shown to have profoundly different effects upon the nursing behavior of female rats and on the depth perception of said nursed pups, depending upon whether it is provided as ⁶Li or ⁷Li [13]. In materials utilization, Silicon-28, or ²⁸Si, is valuable in quantum computing due to its lack of a nuclear spin—which for certain applications facilitates a lower quantum decoherence effect [14]. In some cases, nuclear spin of the atom can be ‘programmed’ using isotopes of certain elements, such as for NMR labeling, where ²H, ¹³C, ¹⁴N, ¹⁵N, ¹⁷O among others are used in clinical imaging. Varying the isotope constituency of carbon in a boron-doped diamond has demonstrated changes in the superconductivity transition temperature of this material. The behavior of light, reflected as a change in optical emission spectra, can be altered in transition metal dichalcogenides wherein isotopically pure ⁸⁶W⁸⁰Se₂ bilayer single crystals had a distinct (and useful) phonon-assisted indirect excitonic transition when compared to the natural isotope abundance counterpart [15]. And most recently, cubic-boron nitride, when made specifically of only ¹⁰B or ¹¹B, was found to give markedly increased thermal conductivity when compared to the material derived from a mixture ¹⁰B and ¹¹B [16]. Thus, while these effects might be subtle, more precise control of the way a material is engineered might lead to important discoveries that change the way we will eventually design advanced products with unique and powerful properties.

Beyond NMR use of isotopes, there are other aspects of materials function that isotopes can affect. These areas are spintronics, spin-mechtronics, and spin-plasmonics. Together these are termed “spin physics” and exploit the quantum mechanical properties of elementary particles and nuclei, where the isotope can drive the differential behavior of its nuclei and electron. Spintronics is short for spin transport electronics and uses the spin of an electron along with its magnetic moment to drive electron transport in materials.

For instance, researchers at IBM recently [17] used atomic force microscopy to control the magnetic properties of single atoms. They accomplish this via use of the spin of the electrons that orbit the nuclei

under study. In one experiment they were able to detect the spin of the nucleus that distinguished ⁵⁶Fe from ⁵⁷Fe when they were sitting in a bilayer of MgO. In a second experiment they detected the electron parametric resonances of the ⁴⁷Ti versus ⁴⁸Ti isotopes of titanium (Fig. 2). In addition, spintronics is already used in crystalline insulators [18]. A related phenomenon is termed spin mechatronics wherein the magnetic or acoustical or optical properties of a materials are determined. Yet another phenomena is termed acoustic phonons and can be used, for instance, to measure superconductivity in rhombohedral tri-layer graphene [19]. Spintronics has been previously investigated in US government analysis of unconventional craft in the Defense Intelligence Reference Documents produced under the Advanced Aerospace Weapon System Applications Program (AAWSAP) program (see item #24 Metallic Spintronics [20]). Finally, spinplasmonics combines spintronics and plasmonics where one uses electron spin (again, which can be driven by the isotope of a given nucleus and surrounding materials) to create novel materials that can be used as storage devices, light emitting devices, and electronic switches with low power consumption.

The latter examples are not meant to be a comprehensive review of the utility of quantum mechanical functions that can be exploited via usage of given elements and their surroundings. The associated fields are only just recently developing methodologies to detect their existence, derive their functionalities, and then exploit them for practical gain. But they underscore a growing realization that isotopes of different elements can offer novel electronic properties for materials design.

2.2. Inductively coupled plasma mass spectrometry (ICP-MS)

ICP-MS operates by passing a small portions of the material under study through a super-hot (i.e., several thousand Kelvin ≈ surface temperature of the sun) argon plasma, to atomize and ionize it [1,2]. Most ICP-MS instruments require an experimental sample to be completely dissolved in liquid. If the material is not already dissolved, this can be accomplished by grinding the material into a powder and then dissolving it in one or more acids (such as nitric or hydrochloric acid). Alternatively, the materials can be dispersed by intense pulse application of lasers or electric discharge, then passed into the plasma, and then into the mass spectrometer.

Other approaches, such as the “CyTOF” mass spectrometer available in one of the labs [21] used in this study, allow for introduction of the

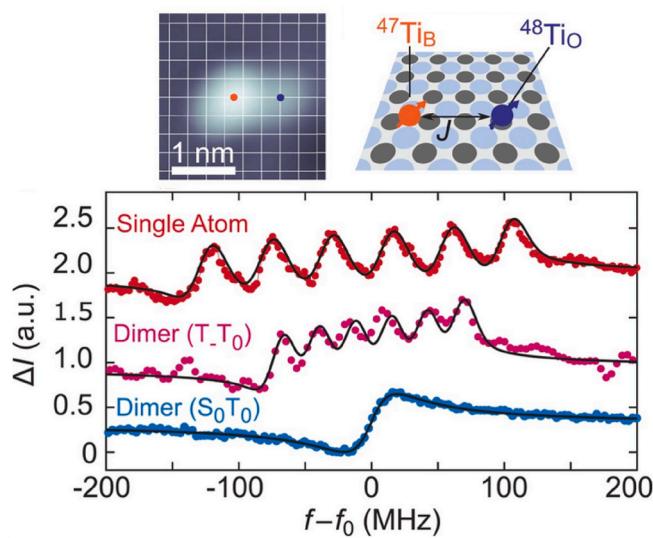


Fig. 2. The local spintronic environment of a given paramagnetic isotope is governed by its surrounding atoms wherein a ⁴⁷Ti atom was placed proximal to a ⁴⁸Ti. The bottom graph shows that the dimer of the two distinct isotopes show a different coupling strength for ⁴⁷Ti than for the ⁴⁷Ti atom alone. See Ref. [17] for details.

materials into the mass spectrometer as mono-disperse colloids (particles up to 10 µm across, about the size of a cell) which then are atomized and ionized for mass determination of the particle's atomic constituents.

Those particles retain information about local structure and content compared to ICP-MS, which homogenizes an experimental sample by dissolving it in acid. This can be a significant advantage because it allows local isotopic content to be determined relative to other atoms in the immediate vicinity. ICP-MS instruments are manufactured by a variety of vendors (Thermo Fisher, Bruker, etc.) with a heavy focus by purveyors to the fields of biomedical analytics, geological, and nano-materials composition and engineering.

2.3. Secondary ion mass spectrometry ("SIMS"): Nano-Sims and "MIBI" technology

SIMS allows for materials analysis 'in situ'. It can raster across a target material, ablate in an XY plane down to generally 5–1000 nm with a Z depth of as little as 5 nm. With such rastering one can collect both 2D and 3D information on the content [5,22,23].

The instrument works by accelerating a PRIMARY beam of particles (usually charged ions) to hit the target, ablate it, and atomize the target into secondary ions, via the kinetic energy of the primary ions. These secondary ions are electromagnetically directed into a mass spectrometer. The primary ion beam can vary from helium, other elements, and heavier objects such as buckyballs (60 carbon regular truncated icosahedron)—with different ion beams able to ionize or fragment given materials with different efficiencies [3,4].

Traditional SIMS is exemplified by the NanoSIMS (Supplementary Fig. 1) we have used in preliminary testing of materials at the Stanford Shared NanoSIMS Facility. That instrument (Cameca NanoSIMS 50L), however, is limited in that it can only measure 7 isotopes at a time. The device is extraordinarily sensitive and capable of distinguishing isotopes of different elements with near identical mass and is accomplished using a magnetic sector device [24]. In addition to the Zn and Ni isotopes noted above, many other isotopes have an amu that requires highly accurate instrumentation. For instance, ⁵⁴Fe and ⁵⁴Cr weigh respectively, 53.9396 and 53.9389 atomic mass units, which means the device must be capable of a sensitivity in the parts per million range to correctly call the difference. In such a device the resulting secondary ions generated from the investigation material is passed through a strong electromagnetic field. Atoms of different mass not only move and arrive at a detector according to their mass (with lighter elements arriving first), but they are separated in 2D space according to their ionization (i.e., charge). Together these can be used to place detectors along a track on the instrument to capture ions at specific, expected locales calculated based on mass and charge. The detectors can be bulky and only a limited number can be linearly placed on the Cameca device, and that limits the numbers of isotopes that can be measured at a given time. Alternative equipment as also applied here for secondary ion mass spectroscopy is known as "MIBI" for "Multiplexed Ion Beam Imaging." It provides isotope analysis and spatial resolution within the material. The "MIBI" instrument (Supplementary Fig. 2) represents an advance that allows us to read across the entire mass range AND it has all the advantages of SIMS, as well as allowing for 3D structure determination of nanometer scale objects using isotopes as tags [25].

The caveat applicable to SIMS of any type is still relevant, however: The particle beam (oxygen plasma, cesium, or gold) determines the material that can be analyzed and the efficiency of elemental ionization [3,4]. Critically for the applications here, isotopes of a given element are released at the same ionization efficiency, but different elements might have vastly different ionization efficiencies. That is, one cannot compare the molar ratios between different elements in an experimental sample, but comparisons of isotopes within a given element is valid. Therefore, the MIBI instrument used in this report is not quantitative between elements, due to difference in efficiencies of ionization, while it is quantitative for isotopes intra-element. For instance, Nickel has 5 stable

isotopes. Any measurement of materials containing Nickel isotopes by MIBI, or any SIMS instrument is perfectly valid although there are potential artefacts due to oxide, hydride, and other diatomics.

MIBI was designed for biologic measurements of proteins via metal chelated antibodies and permits routine imaging of up to 50 proteins simultaneously as opposed to only two or three with currently available instruments, with resolution on par with light microscopy, but also enabled to accomplish 3D determinations [22,23].

2.4. Energy dispersive spectroscopy (EDS) and related materials analysis systems

While both spectrometry techniques noted above are destructive, EDS enables relative ratios of elements to be measured in a material. In EDS, the experimental sample is bombarded with a charged particle beam (electrons or protons), or X-rays (a related technique is called EDX, energy dispersive X-ray spectroscopy). Each element, depending on its electron shell composition, will emit X rays with a distinct signature. Comparison to prior known standards allows for multiple elements to be distinguished.

The value of EDS to the analysis of materials, especially when using SIMS or ICP-MS data, is that it allows for one to rule out certain diatomic species that might form during ion generation. Since both SIMS and ICP-MS can form oxide and hydride diatomics, EDS allows for correction or filtering of values.

Related to EDS, but slightly different in detection modes is X-ray fluorescence microscopy (XRF). XRF can detect elements at parts per million limits, whereas EDS generally can only detect elements at the 0.1% lev.

2.5. Scanning transmission electron microscopy

With scanning transmission electron microscopy (STEM), beams of electrons are focused on an ultra-thin material wherein the electrons are either scattered and detected as such (thereby indicating the presence of an atom) or the detector can be an EDS or EDX, which can provide the elemental composition of said atom. This device is used for nanometer and sub nanometer determination of structures of materials, can be non-destructive, has been adapted in limited cases to sub-angstrom scale capable of determining inter-atomic distances. The reader is directed to Ref. [26] for an overview of additional techniques relevant to the use of STEM.

2.6. Raman

Raman spectroscopy is yet another technique for measuring constituent atoms and molecules. Generally, the technique is used to create signatures of molecules from the scattering of photons from a material. Photons impinging on a material interact with the vibrational components of that material (which can also include phonons). Recently, certain nanomaterials (such as gold or other metal nanoparticles) have been shown to 'amplify' the Raman signature to a level useable as standardized detectors. Usually, however, the signatures are complex and complex materials give back spectra that are too difficult to deconstruct. However, as perhaps noted by the fact that phonon vibrations are dependent on nuclear structure, Raman signatures can also be affected by the isotopes found in a molecule. The reader is directed to Refs. [27,28] for further reading.

3. Investigating unknown material collected in the field: A case study

Elemental and isotopic analysis has proven of value in the study of material recovered from disintegrating aeronautical or astronomical objects, or from residue deposited on earth following an observation of an unknown phenomenon. In such cases it is essential to distinguish

between natural and manufactured material and, in the latter case, to obtain information about the process that led to the creation of the recovered sample, and its potential purpose.

While geologists and astronomers are keenly interested in the analysis of meteorites or comet residue, where isotopic ratios differing from terrestrial norms may give clues to the distribution of elements in the solar system (and even our galaxy) across space and time [11,12], other scientists have focused on materials suggesting industrial or military processes oriented to propulsion or other purposes of aerial or space navigation.

3.1. Overview of the incident at Council Bluffs, Iowa (17 December 1977)

An event that took place in Council Bluffs, Iowa, on the evening of Saturday, December 17, 1977, provides an interesting opportunity to compare testing methodologies suitable for the analysis of material recovered from aerial objects. The Council Bluffs case has been previously reported in detail by Jacques Vallée [29] and in a book by Professor Peter Sturrock of Stanford University's Plasma Research Institute [30].

On that day, at 19:45 CST (0145 GMT) a red, luminous mass was observed by two Council Bluffs residents as it fell to earth near the northern city limits, slow enough to enable its approximate trajectory to be noted. A bright flash was seen, followed by flames 8–10 feet high. When they reached the scene, the witnesses found an area covered by molten metal that glowed red-orange, igniting the grass. Police and a fireman arrived within 15 min and saw the mass (with estimates ranging from 35 to 55 pounds) "running, boiling down to the edges of the levee," in an area of about 4 feet by 6 feet. The central portion remained warm to the touch for approximately 2 h. No cratering was noticed.

The site is a public area ("Big Lake Park") in that suburb of Omaha, Nebraska, located on the southeastern shore of the Missouri River in Iowa. This section of the park, known as "Gilbert's Pond," lies across the river from Eppley airport. The weather at the time was overcast with a 2500 feet ceiling, visibility 10 miles, temperature 32 °F. Wind was from the WNW at 16 mph, with gusts up to about 25 mph.

Given the physical evidence (a representative fragment is shown in Fig. 3), the availability of cooperating witnesses, and the proximity of a major airport, the investigation was immediately oriented to the potential for an industrial accident, an aeronautical malfunction, or a meteorite. After ascertaining that the material was not radioactive, two chemical analyses were rapidly conducted, one at the Iowa State University laboratory and the other at Griffin Pipe Products Company. Both agreed with the determination that the material was a metallic alloy, chiefly iron with small amounts of nickel and chromium. The report indicated that the micro-structure suggested the studied sample was carbon steel that was cast, subsequently re-heated to about 1000° and cooled at an intermediate rate, so that it resembled wrought iron [31] (wrought iron is an alloy containing low carbon content in the 0.08% or less level). No pictures of the 'microstructure' were provided in the historical report. The problem with that determination was that nothing accounted for the way a large amount of that very heavy and extremely hot material could have been deposited in that area in a molten state, following the clear observation of a luminous object in the sky, (the purported source of the material) which remained unidentified.

Therefore, the investigation went into a second, more specific phase of analysis along several hypotheses. First, there was an abundance of valuable eyewitness testimony that had to be reconsidered, which will be reviewed below.

3.2. Initial eyewitnesses and official reaction: First "on the spot" evidence

As mentioned above, the first observers were "two Council Bluffs residents," and their report was soon confirmed by independent people. Investigators were able to gather testimony from no less than 11



Fig. 3. Material samples recovered at Council Bluffs (scale in inches).

witnesses in separate groups, all within an hour of the incident. Unusual in such cases of anomalous phenomena, the key witnesses could be positively located and identified.

The first report came at 7:45 p.m. from 17-year-old Kenny Drake and his nephew, 12-year-old Randy James, who were driving on North 16th street. Kenny's wife Carol, 16 years old, was also in the car. All three saw a "reddish ball" at an estimated altitude of 500–600 feet. It fell "straight down" and impacted within Big Lake Park (see map). There was a bright flash and what appeared to be "flames shooting into the air, 8–10 feet high." While these witnesses only observed the dropping object, others (see below) had observed a stationary flashing object that was in the area from where the reddish ball originated.

Mr. Robert Allen, who had served in the Air Force and wrote a regular astronomy column for a local paper, investigated the site the next day and confirmed "there was a clear line of sight from the impact point to (their) location." As the molten material formed a large hot mass about four by six feet wide (Fig. 4 and Supplementary Fig. 3 for 4 original Polaroids), igniting the nearby grass, Drake and James drove to a local store and called 911. The call was given to Jack Moore, Assistant fire chief for the Council Bluffs fire department, who responded in his personal fire car. No fire-fighting crews were required, however, while the police, who had intercepted the call, dispatched a cruiser car to the scene, driven by Assistant chief Moore. He requested an officer from the Identification section to join him with a camera to photograph the material that was "running, boiling down the edges of the levee. The center of it was way too hot to touch." The material would remain warm for another 2 h, despite the freezing air and ground temperature. The time was about 8pm.

Officer Dennis Murphy arrived and took several pictures with both a Polaroid color camera and a 35 mm SLR camera equipped with color film. (Note: Four Polaroids shown in Supplementary Fig. 3, original Polaroid photographs are in the authors' case file). The recount of the incident by Drake and James before the officers did not deviate from the story they first told. Checks were made with both Eppley Airfield and Offutt Air Force Base, Nebraska, "to ascertain if any aircraft might have



Fig. 4. Enhanced Original Polaroid of #3 of 4 Police photographs showing deposited material (grey and green area). (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

reported equipment or engine failure or loss," with negative response from both.

Arriving on the scene the next morning, Robert Allen had no difficulty locating the site, noting there was no cratering—which he had expected given that he believed there would have been an impact. He

gathered several samples of the ground, and pieces of the object, including the ones now in the authors' custody, were collected and catalogued. But an analysis of the reports disclosed another interesting fact: Early witnesses had observed a hovering object, separate from the glowing, falling mass.

3.3. Corroborating witnesses

As the first three witnesses were watching the glowing mass, a small car stopped and four young men, about 18 years old, asked if they had seen "that thing fall out of the sky," after which they drove away (Fig. 6, original map, point #1). A few minutes later, when Kenny Drake was making the 911 phone call, authorities were also contacted by a middle-aged couple who were travelling north on 16th street (Fig. 5, point #2).

They reported seeing a bright red mass "rocket to the ground near Big Lake". They refused to be identified on the record. At that point, no less than nine witnesses in three separate groups had watched the incident as it happened. Two more would soon come forward.

Further south on Broadway Avenue, 24-year-old Mike Moore and his wife Criss had been driving east toward downtown, crossing the 16th street intersection when she first saw the object, described as round with "red lights blinking in sequence around the periphery." Mr. Moore saw it when they reached the Broadway viaduct (Fig. 6, point #3), as a "bright-red thing at treetop level." He described it as "a big round thing hovering in the sky, below treetops. *It was hovering. It wasn't moving.*" Mike Moore was at that time a parts man at an auto dealership. His wife Criss, also 24 years old, was a legal secretary. Supplementary Fig. 4 shows a circa 1970s map of the area.

Given the amount of detail and the consistency of the observations, supported by the evidence of the mass of material, five hypotheses had

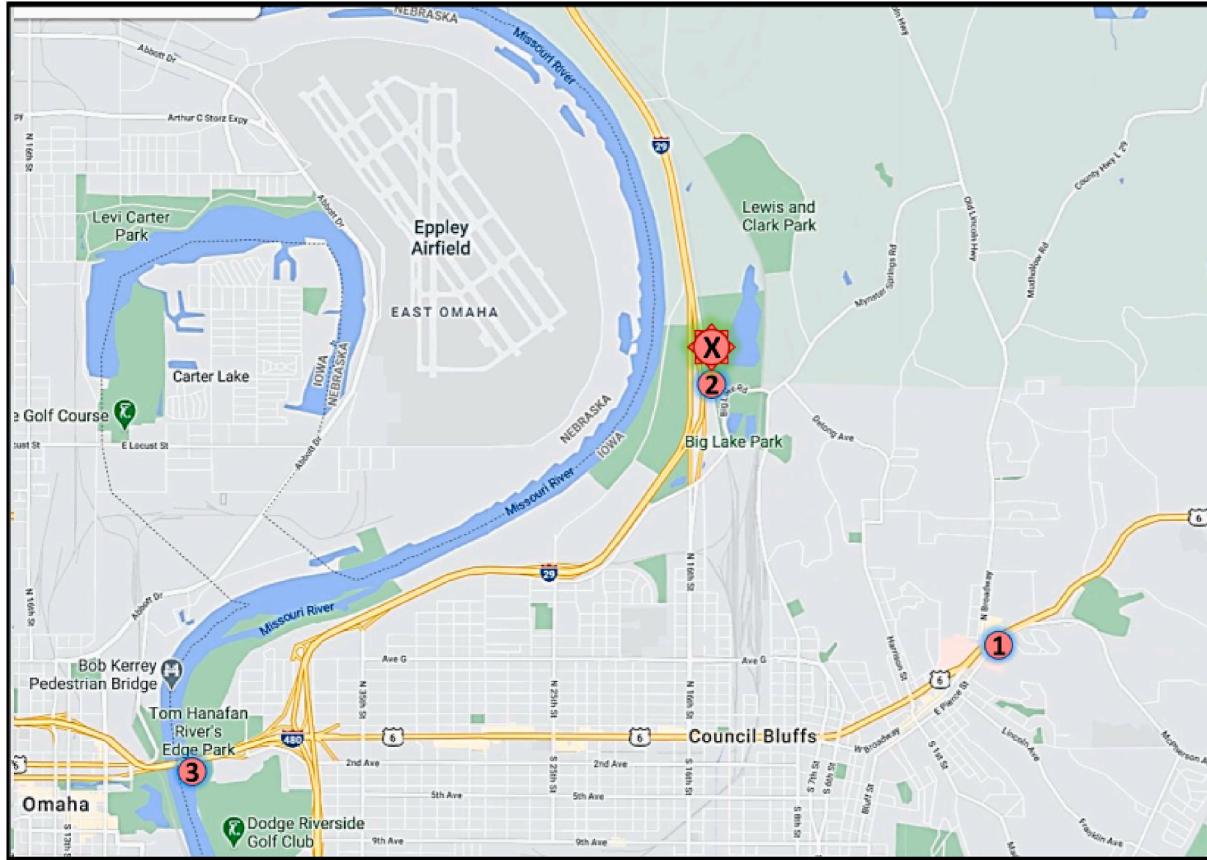


Fig. 5. Contemporary map of Council Bluffs, with the site of impact (X) and main witness locations as numbered. Numbered circles indicate positions of witnesses, Starred X with red arrow represents the site of material as it was discovered. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

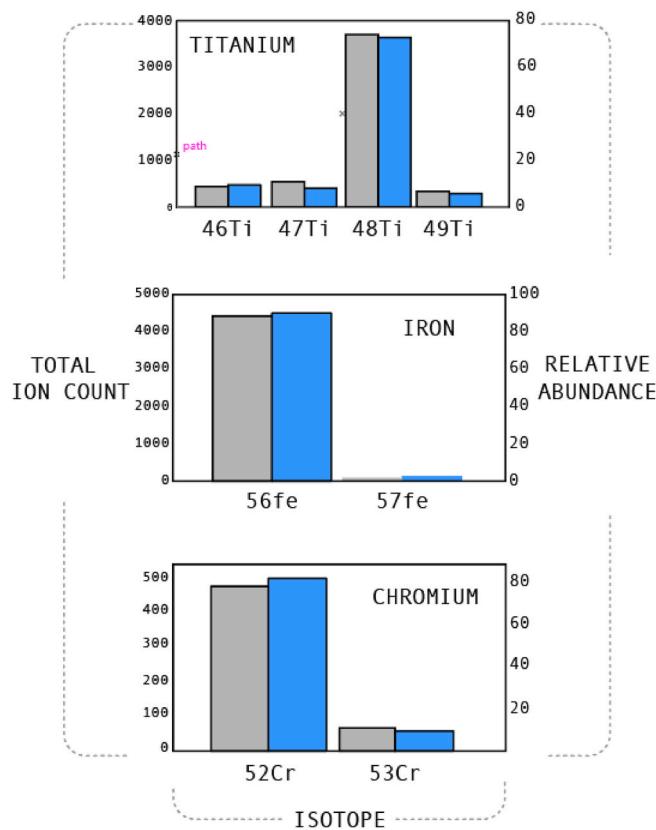


Fig. 6. Isotope abundances for (a) Titanium; (b) Iron; (c) Chromium as determined by NanoSIMs on two fragments of the Council Bluffs material. The grey and the blue bars represent readings from each of two subsamples tested. The left Y axis shows the total ion count for each sample, and the right Y axis is scaled to represent percentage abundance for the isotopes of each metal. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

to be seriously investigated.

3.4. Reentering satellite debris hypothesis

The US Air Force reacted rapidly to Robert Allen's request for their advice. The detailed report he had submitted to them proposed: "It is our theory that (the material) was a piece of space debris which re-entered the earth's atmosphere and was not completely destroyed during reentry. Measurements taken at the impact point would indicate the object was travelling from the southwest to the northeast on a heading of 109–110°."

In response, the Office of the Secretary wrote that its scientists "were very impressed by the thorough documentation of the events and the analysis presented in (the) report," yet they did not feel that the material was space debris for four reasons:

- (a) Reentering spacecraft debris does not impact the earth's surface in a molten state. The aerodynamic heating that usually causes the spacecraft to completely burn up during reentry takes place in the upper layers of the atmosphere before the maximum dynamic pressure is reached. If the material were hot enough to be in a molten condition as it passes through this maximum deceleration point, which probably reaches 10 g's, it would separate like a fluid. In that case, it would have cooled while descending through the lower atmosphere and would have been solid at earth impact – not in a molten state.
- (b) The mass of material was estimated by police to weight 35 to 40 pounds, yet there was no crater or noticeable indentation (Fig. 4).

Space debris having a reasonable ballistic coefficient in the 100–300 pound per square foot range can be expected to hit at a velocity of 200–400 miles per hour and would make at least some impression in the ground and would have splattered well beyond the original site—no such dispersal was noted by any witness.

- (c) The reported visual sighting was at an estimated altitude of only 500 or 600 feet. Reentering space debris is not glowing or hovering at this low altitude.
- (d) There are no structure indications in any of the debris samples. This is very unlikely for space debris from human satellites or missile launches.

Colonel Charles Senn concluded: "*The Air Force does not feel that additional investigation or analysis is warranted.*" While helpful in dismissing the satellite hypothesis, this report left Mr. Allen and the Council Bluffs police department with an unsolved problem, and four remaining hypotheses (see Supplementary Fig. 5 for a copy of the letter).

3.5. Meteoric impact hypothesis

The idea that the material might be the result of a meteoric impact seemed initially attractive. Meteorites of all dimensions are routinely picked up on the surface of the earth. In this case, however, examination of the site and primary chemical analysis quickly eliminated this hypothesis. First, there was no cratering. Perhaps a glancing impact would have accounted for this?

Two additional determinative lines of argument eliminate an iron meteorite fall as the source of the Council Bluffs material. First, there are the aerodynamic forces of passage through the atmosphere interacting with the mechanical properties of the meteorite that determine at what speed and in what form meteorites can impact the Earth's surface. The total mass of the Council Bluffs material (≈ 18 kg) would have formed a bolide of only about 8 cm radius. Computer modeling shows that at typical meteorite atmospheric entry speeds (≈ 11 –30 km/s) an iron meteorite of this size would probably survive to the surface without breakup but would have a terminal velocity of ≤ 100 m/s [32]. An impact velocity of 100 m/s corresponds to a specific energy of 5 kJ/kg, while the specific energy required to melt slag iron (latent heat of fusion) ≈ 209 kJ/kg [33,34]. Thus, an iron meteorite of this size would arrive at the ground with far too little kinetic energy to become molten. Second, iron meteorites are found to contain between 5 and 40% Nickel [35], whereas the Council Bluffs material had only trace amounts of Nickel.

Thus, the material did not meet the physical requirements for a meteorite. It remained too long in a molten state, and "spectrographic analysis did not disclose metal components that should be an integral part of meteoritic material."

3.6. Fallen equipment from aircraft hypothesis

Because of the proximity of Eppley Airfield, it was thought that the incident might have been associated with "a piece of equipment or metal which fell from an aircraft landing at Eppley on the runway heading 320°." The initial check with Eppley, however, indicated no pilot report of equipment loss or failure.

Next, a check of all airlines operating into Eppley Field indicated no arrivals using the runway that would bring any aircraft near the impact site. Furthermore, no equipment or metal would fall at a speed necessary to bring the material to incandescent or molten state. An aircraft using that runway would have had to descend rapidly over the Richmond Gordman Store from which impact was observed by the first two witnesses, and at that point it would be at 500–1000 feet altitude only.

Having eliminated the hypotheses of reentering space debris, meteorite or aircraft equipment, the investigators were left with the possibility that the case might be the result of a hoax. There were two ways in which one might imagine that the material might have been deposited as

a joke.

3.7. Is this a hoax perpetrated using thermite?

When the material was examined at the laboratory of Professor Frank Kayser (University of Iowa at Ames) he observed that: "One could prepare a rather similar sample in the following way: One would collect the splatters from a casting or welding operation involving carbon steel. One would transport this to the position where the material was found. One would then surround the metal with thermite powder and ignite the latter. This would heat the metal to the 900–1000-degree C range, and the thermite reaction would generate the iron-aluminum oxide sponge. A cooling rate appropriate to 'wrought iron' microstructure could be achieved by spraying water on the mixture."

The problem with that hypothesis was that the material was in a molten state when witnesses arrived. It had not solidified and could not be picked up. The ground was frozen to a depth of at least 4 inches and the air temperature was 32° F. Thus, "*had someone tried to cool the material in an intermediate state, there would have been considerable ice in the immediate vicinity caused by the water used to cool the material.*"

Finally, a check made with both chemical and construction firms in the Council Bluffs-Omaha area disclosed no source of thermite. Thermite is readily made from powdered iron oxide III and aluminum powder which can react as: $\text{Fe}_2\text{O}_3 + 2 \text{ Al} \rightarrow 2 \text{ Fe} + \text{Al}_2\text{O}_3$. While it is not impossible to rule this out, this does not explain how thirty pounds of iron (with other elements such as titanium) was reacted in midair and dropped visibly in sight of multiple witnesses and how no visible melting of nearby earth was evident to have created the heat to melt iron at that temperature.

3.8. Is the incident a hoax perpetrated by pouring molten metal on the ground?

Another, more radical hypothesis assumed that persons unknown (perhaps the first two witnesses) had simply brought molten material to the site and poured it on the ground. Accordingly, a check was made with every metal firm in the Metropolitan area with the remotest possibility of maintaining metal in a molten state. This included all foundries, pipe manufacturers, metal fabricators, etc.

The only facility with such capability and equipment in Council Bluffs was the Griffin Pipe Products Company. Mr. Linton Stewart, works manager, stated they "*dropped the bottom from their cupolas*" on Friday afternoon and did no pouring whatsoever until early on Monday.

Katelman's Foundry used to have facilities for pouring molten metal at one time but had not done so in three years. Paxton-Mitchell Steel in Omaha stated that "*in order to melt the material and then transport the molten mass to the Big Lake area would require considerable equipment.*" They added: "*The melting point of some of the components is in the range of 2400–2500 degrees F. Once molten, the metal would have to be transported in a brick oven of at least 6 inches thick lining, which would necessitate a large truck to transport the oven. And it would be necessary to keep the metal at the required melting point.*"

The investigators concluded that:

"The material associated with the object in Big Lake Park was not manufactured, transported or deposited in the area in order to perpetrate a hoax, by any of the foundries in the metropolitan area. Primary witnesses do not have the equipment or experience to meet all the prerequisites of perpetrating a hoax. In all interviews and discussions with investigator, police, and news media, their recounts of the events have not deviated since the initial conversations."

4. Advanced analytical methods applicable to exceptional physical events

The material recovered at Council Bluffs consists of three components: solid metal, slag, and white ash inclusions in the slag. In the previous section we have mentioned the results of the analysis at the Ames Laboratory, which examined all three of those by X-Ray fluorescence, electron beam microprobe, and emission spectroscopy, under the supervision of Mr. Edward DeKalb.

4.1. Early tests of chemical composition

The metal was found to be chiefly iron, with less than 1% of alloying metals such as nickel and chromium. The slag was a foam material containing metallic iron and aluminum with smaller amounts of magnesium, silicon, and titanium, "probably present as their oxides." The white ash inclusions were "principally calcium with some magnesium, again probably as oxides."

The first test, run on 21 December 1977 (within four days of the incident) by lab technician Jack Coan (at Griffin Pipe Products), was run on two exposures of the metal sample using a "Spectro-Comp" instrument that could identify 18 elements spectroscopically. It found carbon at 0.70%, manganese at 0.56%, silicon at 0.52% and other elements as traces only.

The more comprehensive Ames report (Table 1) confirmed the fact that the material was not of meteoritic origin, for "in that case one would expect a much higher nickel content" [31]. The authors added that man-made space hardware would also be unlikely as a source: "Such hardware usually involved alloys of much higher strength-to-weight ratio, containing high amounts of nickel, chromium and titanium. Our first judgment was that the material resembled cast iron." Interestingly, carbon steels are usually configured to contain between 0.05 and 3.8% by weight of carbon. Given that carbon is about 4.5-fold lighter than iron, and assuming the material is principally iron, then by percent composition the carbon should be present at between 0.23 and 17%. This was not represented in any of the data from the Ames lab report.

Next, Professor Frank Kayser, of the metallurgy division at University of Iowa, in Ames Iowa, examined the metal sample metallographically and reported it was most likely a carbon steel, confirming the judgment of the Griffin Pipe Products Company. He pointed out that "despite the name, carbon steels contain less carbon (about 1.0–1.2%), than cast iron (about 4%)." From the microstructure he judged that the metal was cast, subsequently reheated to 900–1000° C, and cooled at an intermediate rate so that it resembled wrought iron (see Supplementary Fig. 6).

These were the observations available to us when we undertook the study of the isotopic ratios in the main components of the metal samples.

4.2. Secondary ion mass spectrometry: Initial isotopic results

A surviving piece of the original material was retained by JV and provided for the experiments herein (hence termed CB_JV-1 for clarity). The CB_JV-1 sample is approximately 2 inches across in the XYZ dimensions. Note that while this sample is claimed to be derived from the same samples used in Table 1 (those samples are no longer available), it is distinct and uniquely measured in this report.

The isotopic abundances of two subsample grains from the parent Council Bluffs metal CB_JV-1 sample, as run by the authors on a Nano-SIMS machine (manufactured by Thomson-Syseca) yielded the graphs shown for the relative distribution of isotopes of Titanium (^{46}Ti , ^{47}Ti , ^{48}Ti and ^{49}Ti). The results are consistent with terrestrial values (Fig. 6a). We also found unsurprising ratios for Iron (^{56}Fe and ^{57}Fe) and for Chromium (^{52}Cr and ^{53}Cr) (Fig. 6b and c).

From an analytical viewpoint it is important to note that error bars are often not included in such analyses since, due to local oxidation conditions on the sample, or other impurities, sputtering efficiencies, charge fractionation, sample bias setting, and other issues can lead to

Table 1

Table of results from Laboratory of Professor Frank Kayser (University of Iowa at Ames). Per written notes (see Supp Figure 3) these are qualitative estimates based on spectrography. Given the sample is principally iron, we interpret » 1 to be a convention used by the analyst relate that the amplification of the spectrometer was set to maximum to measure the trace elements.

	metal-1	metal-2	metal-3	metal 4	slag-1	slag-2	ash
Iron	>>1.00	>>1.00	>>1.00	>>1.00	>0.1	>0.1	0.20
Magnesium	0.30	0.30	0.30	0.30	>0.1	0.10	0.10
Tantalum	<0.40	<0.40	<0.40	<0.40	0.40	0.40	<0.40
Titanium	0.25	0.05	0.05	0.05	0.10	0.10	<0.1
Molybdenum	0.20	0.20	0.20	0.20	<0.05	<0.05	<0.05
Aluminum	0.15	0.03	0.03	0.01	>>0.10	>0.10	0.03
Silicon	0.10	0.20	0.20	0.20	<0.10	<< 0.1	<0.10
Manganese	0.10	0.10	0.10	0.02	0.50	0.10	0.10
Tungsten	<0.10	0.10	0.10	0.10	0.10	0.10	<0.10
Trace elements:							
Copper	0.08	0.08	0.08	0.08	0.02	0.02	0.01
Chromium	0.02	0.01	0.02	0.01	0.10	0.01	<0.01
Nickel	0.04	0.04	0.04	0.04	<0.005	0.01	0.01
Calcium	<0.01	0.01	<0.01	0.01	0.10	0.25	>>0.1
Cobalt	<0.01	0.01	0.01	0.01	<0.10	<0.10	<0.10

significant systematic effects. In the case of the sample analyzed here (see below) we addressed this in part by measuring different areas of the sample to access a sense of the homogeneity (or heterogeneity) of the material composition (see below).

On this basis our initial conclusion was that sample components were consistent with a terrestrial origin. Since NanoSIMS is limited to measuring 8 elements or isotopes concurrently, we turned to a different instrument capable of measuring a broader range of isotopes. Therefore, to further validate this result, and add a spatial depth analysis, we subsequently ran the samples again using the recently developed MIBI instrument.

4.3. Multiplexed ion beam imaging: Advanced results

Five subsample grains from the parent CB_JV-1 sample measuring approximately 100 µm across and 50 µm thick were chosen for analysis, focusing on grains that had minimal apparent oxidation. Samples were embedded on carbon conductive stickers and placed on a gold-coated slide.

The conductivity of the embedding substrate (i.e., conductive carbon and gold) is necessary to minimize charge buildup from the primary ion beam on the samples during the SIMS run. The primary ion beam was set to a low sample bias which minimizes the contribution of diatomics such as hydrides, oxides, or diatomics of the main sample constituents (Mg–Mg, Al–Mg, etc.).

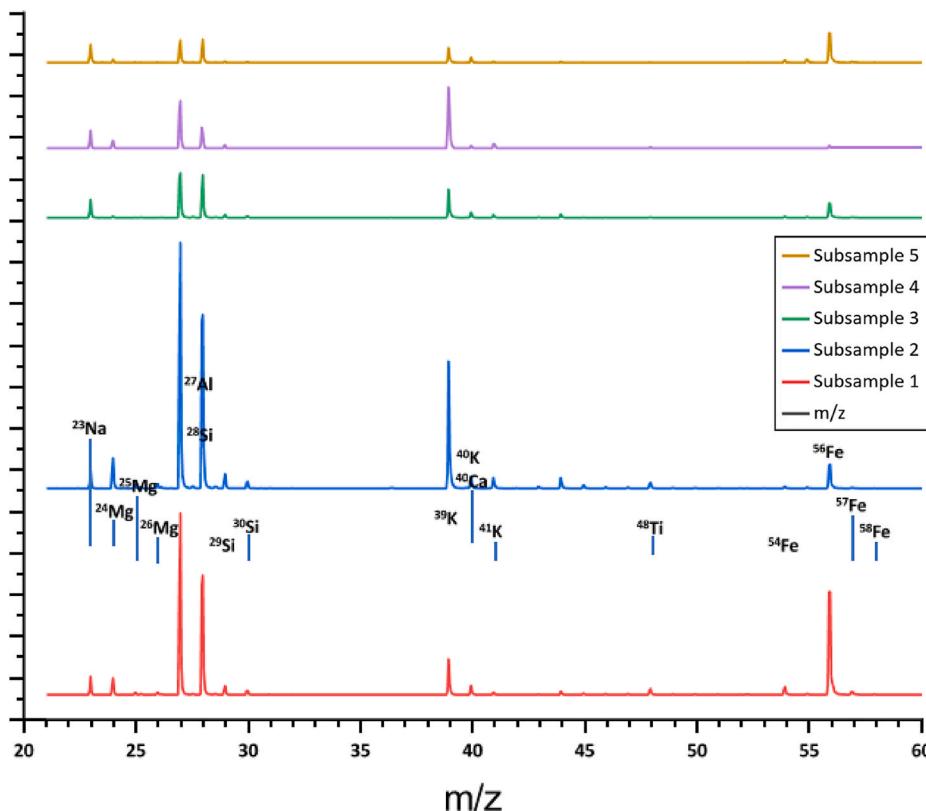


Fig. 7. Relative isotopic ionization efficiencies comparing 5 subsamples of the original parent sample. Peak heights and isotope are shown in the bottom panel for subsample 5.

Ten depths measurements were collected (from each of 5 subsample grains from the parent CB_JV-1 sample in the series). Each depth was approximately 1 nm in the Z dimension. Raw data for the entire run is in supplemental materials as Excel spreadsheets. The measurements for each of the ten depths for each of the grains (subsamples) 1 through 5 were summed and are shown in Fig. 7 as raw values between 20 and 60 mass units. While the subsamples showed similar elemental content, the ratios of the prominent elements differed slightly, suggesting the sample grains arose from an inhomogeneous parent sample. For instance, note in S10 (subsample 5) that the primary Fe peak cluster is greater than the Mg cluster and those of Na, Al, and Si. However, the Fe cluster is significantly lower relative to the other peaks in subsamples 1 through 4. The differences could be due to local variations in oxidation of the sample. For instance, if Fe is oxidized in the other samples, it would be subject to diatomic filtering of Fe–O due to instrument settings. Supporting this is the observation that samples 2 and 4, which had the lowest ion counts across the mass range (meaning they were slightly oxidized and perhaps not well ionized during analysis) also had the lowest Fe peak cluster ratio relative to the other elements as detected in the samples.

In Fig. 8A (left panel) are shown the principal isotope counts for the most abundant elements between mass 20 and mass 60 for samples 1, 3, and 5 (samples 2 and 4 are not presented here due to the low ion counts). These principal elements were Na, Mg, Al, Si, Ti, Fe, and Mn. Ti is not considered here in the analysis due to the presence of Ca and K in the sample, whose weights cannot be discriminated from the trace amounts of isotopes of Ti. In addition, ^{23}Na | ^{23}Na and ^{23}Na | ^{24}Mg diatomics similarly overlap with the Ti peaks. High peaks such as Ca, K, and Ni are not included in this analysis due to the possible interference with significant diatomics. Other mass spectrometry approaches, such as ICP-MS specifically targeted to this mass range will be needed to verify this result but would require the complete destruction of the sample.

All isotope ratios were similar between the samples and did not show any statistically significant deviations from expected terrestrial normal except for ^{57}Fe . In examining the ion values around ^{57}Fe it is apparent that this slightly high value of ^{57}Fe in the samples can be attributed to a minor contribution of a diatomic peak around 57.05 (Fig. 9B, right panel). Since ^{56}Fe is 91.72% of terrestrial Fe, this is likely to be caused by a minor contamination of Fe-hydride diatomic ions from contaminating C–H or O–H sources.

We examined the depth profile of all samples for the given elemental cluster set. Again, no variations from isotope frequencies were observed (data not shown). A representative profile from subsample 3 is shown in Supplementary Fig. 6. As can be noted there is a slight increase in the ionization efficiency with increasing depth which might be expected as oxidation layers are removed during continued sample ablation.

While there were no significant differences noted in the measured isotope ratios that could not be explained by artifactual interferences, we did observe significant differences in the homogeneity of the

elements compared across the 5 subsample grains from the parent sample CB_JV-1 (Fig. 9). Note that the relative amount of detectable Al varies up to 2-fold across the subsamples, Fe up to 10-fold, and Mg up to 20-fold. While this can be explained potentially by relative oxidation levels of the individual constituents altering the ionization efficiencies, the samples chosen did not exhibit significant surface oxidation and further over the depths of the samples (about 200–500 nm), there were no apparent differences in the relative detection of these 4 elements. Therefore, it might be reasonable to conclude that the sample was inhomogeneous (incompletely mixed) across its totality for reasons yet to be determined.

Examination of the entire mass spectrum suggests several potential trace elements are present in the sample. The most significant is ^{72}Ge with a mass of 71.922. However, again, the peak from the collected data is at 71.83, suggesting again a diatomic from sample elemental constituents is the explanation.

5. Speculative conclusions

We have outlined current trends in advanced materials analysis, as applied to solid samples collected in the field, with a view to determine their nature, structure, and potential purpose. Our experience with the Council Bluffs case study shows how difficult such a determination can be, even when abundant evidence is collected within minutes of an event, supported by reliable testimony from multiple witnesses and in well-defined meteorological conditions.

5.1. Operation Morning Light

After their initial dismissal, the US Air Force again took a special interest in this case because of another “crash” that took place in northern Canada at 11:53 a.m. GMT on January 24, 1978, only five weeks after the unidentified observation and the recovery of metallic material at Council Bluffs.

The Canadian crash was the result of a malfunction aboard Cosmos 954, a soviet reconnaissance satellite designed to observe ocean traffic, including surface and undersea vessels. It was powered by a liquid sodium-potassium thermionic converter driven by a nuclear reactor containing 45 kilograms of uranium-235 [36]. Soviet authorities secretly warned the United States that control of the satellite had been lost and that the system intended to propel the spent reactor core into a safe disposal orbit had failed. The science correspondent of the London *Daily Mirror*, Arthur Smith, pointed out the satellite was the fifteenth of its type to be launched by Russia [37,38]. The effort to recover radioactive material from Cosmos 954 was dubbed *Operation Morning Light*. Thus, it was natural for US and Canadian authorities to revive their lagging interest in Council Bluffs and to investigate a potential connection.

Given the analytical results and the instrumental procedures

Isotope	Counts			Ratio			Natural
	Subsample 1	Subsample 3	Subsample 5	Subsample 1	Subsample 3	Subsample 5	
Na(23)	193,232	760,011	3,678,992	100.0	100.0	100.0	100.00
Mg(24)	212,261	54,861	529450	77.70	77.29	78.32	78.90
Mg(25)	27,923	7,622	69623	10.22	10.74	10.30	10.00
Mg(26)	32,990	8,501	76953	12.08	11.98	11.38	11.10
Al(27)	2,516,178	2,585,313	5,225,295	100.0	100.0	100.0	100.00
Si(28)	1,972,739	2,112,325	5,012,265	90.7	89.9	91.4	92.23
Si(29)	129,486	146,147	286,544	6.0	6.2	5.2	4.67
Si(30)	72,565	91,367	183,495	3.3	3.9	3.3	3.10
Fe(54)	124,234	64,637	599,774	5.9	6.2	6.2	5.80
Mn(55)	16,591	40,926	998,997	100.0	100.0	100.0	100.00
Fe(56)	1,897,430	928,102	8,728,626	90.7	89.7	90.1	91.72
Fe(57)	63,258	37,071	318,315	3.0	3.6	3.3	2.20
Fe(58)	7,906	4,534	38,122	0.4	0.4	0.4	0.28

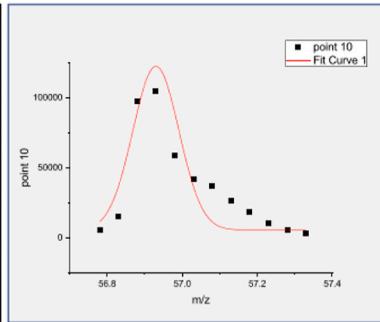


Fig. 8. A. Table of primary elements and their isotopes with ion counts for subsamples 1, 3, and 5. Relative ratios of isotopes are calculated for each subsample and the natural ratio is shown in the final Table column. B. Ion counts for the masses around ^{57}Fe showing a diatomic signature as represented by the black squares that indicate a non-Poisson distribution in the curve, suggesting a second minor peak.

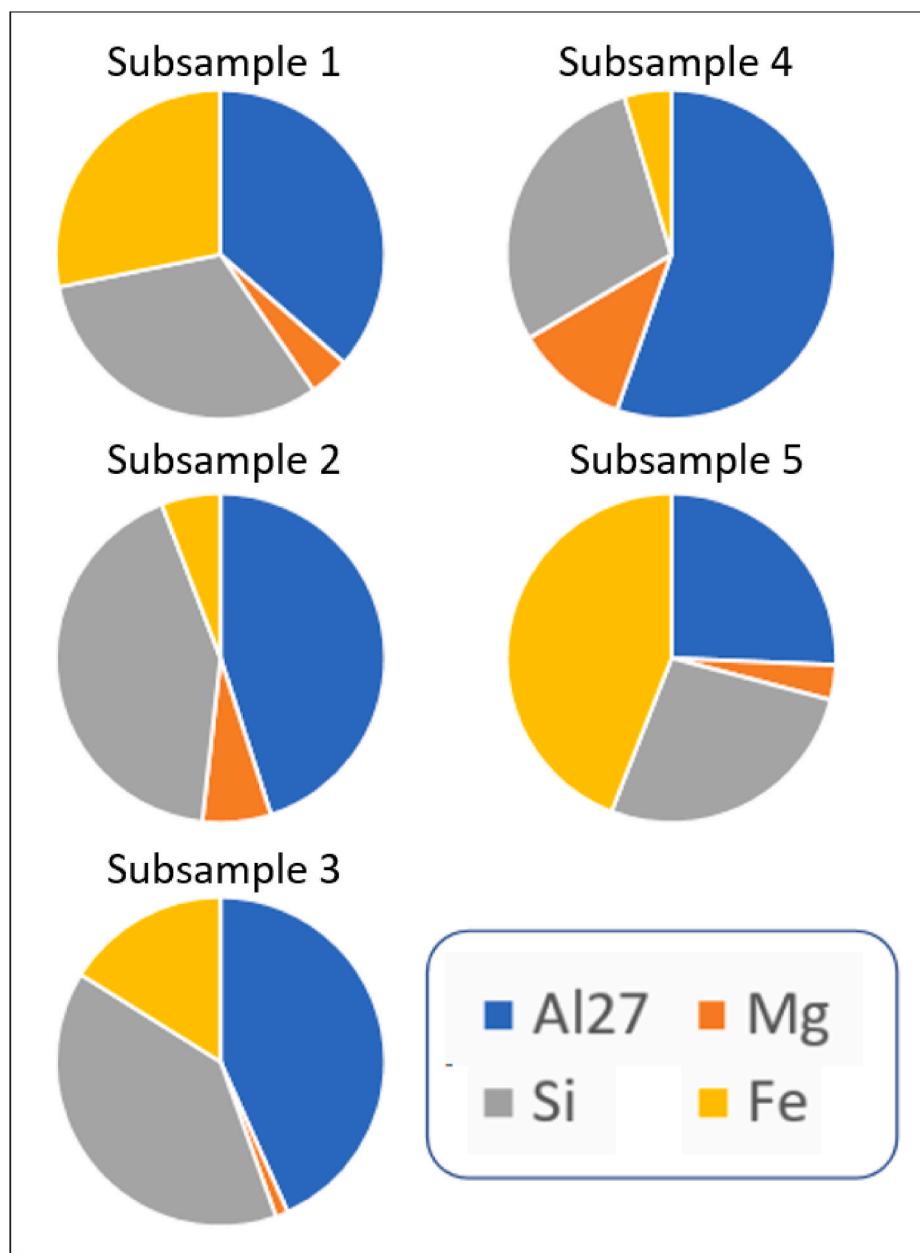


Fig. 9. Relative ratios of the elements for each of the subsamples indicating inhomogeneous elemental distribution.

described above, it soon became clear there was no connection between the two events. The origin of the materials collected at Council Bluffs remained a tantalizing mystery, leading to speculation about more esoteric theories about the hovering object with rotating lights independently described by two of the witnesses. One is led to ask, what would be the requirements to determine whether such materials were not man-made, but originated with an unknown source, either in the solar neighborhood, or elsewhere in the galaxy? This includes the possibility that the material was engineered in a purely conventional manner by humans for yet purposes unknown.

5.2. Potential significance of advanced material design

The fact that two witnesses (Mike and Criss Moore) saw a round object hovering ("not falling") with "red lights blinking around the periphery" is intriguing. Such an object might have ejected the mass of material observed by the other witnesses and recovered by police.

Even in such a speculative area, the scientific steps to be taken are

not fundamentally different from those we have outlined above. One would have to ask, does the catalogue of elements found in the material cause us to believe it does not come from our planet? The materials from Council Bluff show no evidence suggesting it was been engineered or designed. The material would not be expected to form naturally, and as shown does have unusual inhomogeneity. While we cannot divine the purpose, that does not mean it could not be created by methods even extant in the 1970s.

A definitive study of the isotope ratios would be part of any investigation, while keeping in mind that such ratios should be expected to only vary by a small amount (a few percentage points) across our neighborhood of the galaxy. Large differences in isotope ratios, on the other hand, while they would be interesting, would not, by themselves, "prove" an outer space origin: They would only mean that the material had been re-engineered at the isotopic level (as major nations have been doing for the last 70 years, since the Manhattan Project, in separating uranium isotopes to make atom bombs).

5.3. Liquid metal, MHD and advanced flying vehicles

One aspect of the material is that it was claimed to have dropped from an aerial vehicle. Could liquid metal be part of some propulsion or power generation system? In the words of J. R. Bumby of the University of Durham, "the high conductivity of liquid metals makes them an attractive means of current collection for homopolar machines." [39]. A homopolar machine, when implying a motor of some kind is wherein the electrical polarity of the conductor in the system and magnetic field poles do not change, as with the first such electric motors built by Faraday as far back as 1821 [40]. He goes on to cite several such techniques, both superconducting and non-superconducting, that have been built [41–43] and one that is commercially available [44,45]. No such device has been reported to date, so such ideas remain highly speculative.

Similarly, liquid metal designs have been proposed for magneto-hydrodynamic (MHD) generators for the decomposition of toxic wastes *and for superconducting airborne platforms* [46]. However, the composition of the liquids used in such machines is radically different from the list of elements in the Council Bluffs materials. As noted by Busby, "At room temperature the only pure liquid metal is mercury, although at slightly higher temperature gallium (29.8 °C) and sodium (97.8 °C) become liquid." Again, there is no current physics that could explain the use of liquid iron with impurities in any of the aforementioned technologies. Actual machines are using sodium-potassium and gallium-indium mixtures as current conductors, thus minimizing wear and friction. The latter is liquid above 15.7 °C. None of these elements has been identified so far among the samples we have reviewed.

A different approach has been proposed by J. Roser in correspondence with one of the authors: He hypothesized a nuclear design as a potential power plant, utilizing direct energy conversion rather than a heat-driven mechanical prime mover: "A closed cycle MHD generator with a liquid metal working fluid with no vapor staging pumping could be configured in a torus or circular shape and would make very little noise due to the lack of moving parts," he wrote, adding that the nuclear process known as beta decay might allow the design to extract a surplus of power in the form of free electrons.

Assuming a working fluid of aluminum-27 plus some percentage of phosphorus-31 (solitary stable isotopes of their respective elements) Roser speculated that *depleted fluid might need to be occasionally ejected*: "This discarded material would contain Al-27, P-11, iron from the original melt or housing erosion, plus isotopes of nuclei close to aluminum and phosphorus such as Mg, Na, Si and S." (Iron and Silicon were indeed found in our Council Bluffs samples, but the other elements were not present). Roser suggests further isotopic analysis to determine if it reveals anomalous isotopes (such as ^{32}Si with half-life of 280 years) which would indicate a nuclear-based power source. The latter isotope was not detected in the Council Bluffs materials. Should the Council Bluffs material be determined to be engineered for a function we don't currently understand, it remains that our physics are as yet insufficient to explain the purpose of such a material.

6. Summary

Because of the clarity, abundance and timeliness of the testimony, and the open attitude of local police and fire investigators in the Council Bluffs case, we were able to investigate the event with a high degree of certainty as to chain of custody around the material obtained. While this study verified the prior findings in terms of elemental composition and "natural" isotope content, we additionally found that there was local homogeneity of the samples to the degree measured, but considerable diversity in the elemental ratios across the subsamples. This implies that whatever the sample's origins, it was incompletely mixed at the time of deposition.

The Council Bluffs case is one of many—the last several decades has recorded numerous cases wherein materials were claimed to be dropped

from unknown aerial objects. It has not been possible, or financially feasible to date, to bring full range of current materials analysis capabilities to bear. Deep metallurgical analysis by specifically trained analysts will need to be consulted for each different material as warranted. Recently, news reports have suggested the presence of other aerial craft of unknown provenance witnessed by Navy pilots concurrently detected with electronic sensors and visual identification [47–49]. While the data collected about this set of events does not include any material evidence, the day might come when materials from such events are available to be examined. Approaches that maximally provide information down to the atomic scale are now applicable to such studies, including such devices as atom probes and cryo-electron microscopy. Since technology has considerably improved since 1977, we are now better prepared to analyze such events, with a view to bringing previously unidentified episodes into the scope of practical and reproducible science. The objective is to provide data in an open-source manner so that others might replicate the analytic approaches or divine a testable hypothesis of why and how such materials are deposited or left behind. To many this will feel incomplete or insufficient, but this would be a premature conclusion. In the case of Council Bluffs, however, the data is verifiable... it is only the origin and nature of the material (and the phenomenon in general) that remains open.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.paerosci.2021.100788>.

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