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Combined computed tomography and position-resolved X-ray diffraction of an intact Roman-era Egyptian portrait mummy

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Hawara Portrait Mummy 4, a Roman-era Egyptian portrait mummy, was studied with computed tomography (CT) and with CT-guided synchrotron X-ray diffraction mapping. These are the first X-ray diffraction results obtained non-invasively from objects within a mummy. The CT data showed human remains of a 5-year-old child, consistent with the female (but not the age) depicted on the portrait. Physical trauma was not evident in the skeleton. Diffraction at two different mummy-to-detector separations allowed volumetric mapping of features including wires and inclusions within the wrappings and the skull and femora. The largest uncertainty in origin determination was approximately 1.5 mm along the X-ray beam direction, and diffraction- and CT-determined positions matched. Diffraction showed that the wires were a modern dual-phase steel and showed that the $7 \times 5 \times 3$ mm inclusion ventral of the abdomen was calcite. Tracing the 00.2 and 00.4 carbonated apatite (bone's crystalline phase) reflections back to their origins produced cross-sectional maps of the skull and of femora; these maps agreed with transverse CT slices within approximately 1 mm. Coupling CT and position-resolved X-ray diffraction, therefore, offers considerable promise for non-invasive studies of mummies.

1. Introduction

For nearly a century, mummy interiors have been imaged non-invasively with X-rays, including clinical computed tomography (CT) [1,2] which maps X-ray absorptivity in three dimensions (3D). X-ray absorptivity combines density and composition [3] and can differentiate bones from soft tissue and wrappings but not many wrapping-contained ritual objects (minerals as different as jasper, malachite, lapis lazuli and calcite [4]). Magnetic resonance imaging [5] and terahertz imaging [6] also have been applied to mummified tissue, but, unlike X-ray diffraction, these techniques cannot distinguish between different crystalline materials. Although X-ray diffraction has studied materials *extracted* from mummies, e.g. [7–9], it can also identify crystalline materials within *intact* mummies, something which apparently has not been reported previously. This paper describes CT-guided, *in situ* small diameter, high-energy X-ray microbeam mapping and the identification of objects within Hawara Portrait Mummy No. 4 (HPM4).

During preparation of the exhibit 'Paint the Eyes Softer' (Block Museum, Northwestern University, Winter 2018), HPM4 was 'discovered' at Garrett-Evangelical Theological Seminary's library, a few hundred metres from the Museum. The exhibit, on portraits from Roman-era Egyptian mummies, was altered to include HPM4 [10]. Prior to conservation (August 2017), HPM4 was imaged by CT at Northwestern Memorial Hospital in order to reveal internal damage needing stabilization; to provide an accurate 3D description of the individual, the wrappings and their contents; and to furnish a 'roadmap'

Figure 1. (a) Photograph of HPM4 on the CT scanner table with axes x , y , z indicated. (b) Photograph of HPM4 on the translation table for X-ray diffraction mapping with a microbeam at APS beamline 1-ID. Axes X , Y , Z are shown, and the white covering protected the mummy surface from contamination. The green lines in the foreground are alignment lasers.

for position-resolved, high-energy X-ray diffraction (i.e. materials' phase(s), composition and crystallographic texture and assessment of bone condition). The CT data matched radiographs recorded earlier [11] and identified volumes to prioritize for diffraction study (November 2017).

Questions addressed include: how good is the CT roadmap for positioning the X-ray microbeam? What small objects can be located and identified within the much larger mummy? What depth resolution can be achieved? What information can be obtained from the bones? Although HPM4 is smaller than adult mummies, scanning it across the beam is challenging. Additionally, HPM4 was to remain supine. With only 24 h of beam time, the plan for rapid registration of CT and diffraction coordinate systems and for scanning the priority locations was established in advance. Although high-energy X-radiation deposits minuscule doses, exposures were to be minimized.

The CT data concentrate on what the human remains reveal and on details of the wrappings and other non-human contents of the mummy, i.e. on dimensions and observed contrast. Next, diffraction data are described for soil encrusting HPM4, for objects within the wrappings and for bones and teeth; supporting CT data are given in these sub-sections. Suggestions for improved methodology and for future studies conclude the paper.

2. Material and methods

Flinders Petrie's excavations of 1910–1911 uncovered HPM4 in the Fayum region of Egypt [12]. The small dimensions suggest that the mummy contains a child. Prior radiography showed a complete, intact skeleton and radiodense objects with long linear or with oval shapes [11]. Earlier CT data, images on X-ray film [13], had low contrast and only revealed gross geometry.

This study uses two different coordinate systems: x , y , z for CT data and X , Y , Z for diffraction data. The axes are close to parallel. Axes x and X are along HPM4's cranial–caudal axis (horizontal, figure 1), the origins are outside the cranial wrappings and values increase moving caudally. Axes y and Y are vertical with larger values being anterior, and z and Z are horizontal and along HPM4's mediolateral axis.

2.1. Computed tomographic scans

Before conservation and removal of the encrusting soil, CT scans of HPM4 were performed using a Siemens SOMATOM Force system (figure 1a, Northwestern Memorial Hospital, Chicago,

Table 1. Parameters for CT scans; the slices are the y – z plane, and the mummy was translated through the scanner gantry along x .

scan	number of voxels			tube voltage (kVp)
	y	z	x	
03	512	512	1164	120
04	512	512	1164	
07	512	512	1157	90
08	512	512	2891	

IL, USA) and different scanning parameters (table 1). Scan 03 produced a volume of $512 \times 512 \times 1164$ voxels (y , z , x , respectively), i.e. y – z was the slice plane s , with 0.82 mm in-plane and 1.0 mm out-of-plane voxel dimensions. Scan 07 produced a reconstructed volume of $512 \times 512 \times 1157$ voxels (0.69 mm in-plane and 1.00 mm out-of-plane dimensions). Scan 03 was used to select features for position-resolved X-ray diffraction; scan 07 data were used for 3D renderings.

Features selected for X-ray diffraction analysis were: wires in the wrappings near the skull, the skull, resin shards within the skull, teeth, cervical vertebrae, femora, inclusions A and D (which have similar contrast) and inclusion F. Table 2 gives the nominal scan 03 positions of these features.

X-ray attenuation values in scan 03 were quantified using Fiji [14]. For small diameter features, representative values were determined by interrogating multiple single points. For larger features, a box (not intersecting the boundary voxels) determined the mean linear attenuation coefficient and its standard deviation. Absorption is reported in Hounsfield units (HU), and linear attenuation coefficients expressed in a scale with distilled water equalling 0 HU and air -1000 HU [15]. Cortical bone is typically 1400 – 1700 HU and human enamel 2500 – 2800 HU [4].

Visualization and rendering (scan 07) used Amira 6.4 (FEI Visualization Sciences Group, Berlin, Germany). Segmentation of each feature of interest (skeleton, resin shards, wires and inclusion F) from the surrounding materials (mummified soft tissue and wrappings) was performed by selecting the range of attenuation values encompassing that feature. If unwanted volumes were included, manual segmentation eliminated them. The relatively highly absorbing wrapping layer (see Results, figure 3c–e) required manual segmentation.

Figure 2. (a) Schematic of the diffraction set-up at beamline 1-ID, APS. The incident X-ray microbeam passes through the mummy and is blocked by the beam stop before it hits the detector. When the incident beam traverses polycrystalline objects within the mummy, cones of diffracted intensity result and are recorded on the area detector. (b) Schematic of the complication in the transmission geometry of a long path through the mummy. A diffraction peak observed at the position of the red disc (r_1 away from the incident beam position) at detector position 1 might originate at any position between A and B. The possible diffraction angles 2θ could range between $2\theta_B$ and $2\theta_A$. The same diffraction peak recorded at r_2 (red disc) and not at the position of the open red circle would show that the beam originated from position B. The precise 2θ value determines d accurately enough to identify the material diffracting at position B. (c) Sagittal (longitudinal) CT section of HPM4 with areas scanned with diffraction indicated schematically: the red discs indicate point-by-point sampling by the X-ray beam; the arrowed lines indicate the direction of the scans. The scan labelled with the yellow asterisk is through inclusion D which cannot be seen in this sagittal section. The labels in yellow indicate features within the mummy: skull 's', resin shards 'rs', wire 'w', teeth 't', portrait 'p', soil on the outside of the mummy 'so', inclusion F 'incl F', femur 'f' and muscle surrounding the femur 'm'. The lighter the pixel in this image, the more attenuating the voxel.

Table 2. Feature locations and diffraction scan positions. The target position for a given feature based on CT dataset 03 is x_{object} ; x_{range} is the range of X positions of the diffraction scans (Y -scans) across the features; ΔX is the translation increment between Y -scans; y_{range} is the range of the Y -scans and ΔY is the translation increment between diffraction patterns. All quantities are in units of mm, and the reference positions for the CT data and diffraction data differ by an arbitrary amount.

feature	x_{object}	x_{range}	ΔX	y_{range}	ΔY
wires, shards, skull ^a	237	139–169	6	0 to 64.5	0.5
teeth	280	206–212	1	–64 to 65	1
cervical vertebrae	326	247–249	2	–64 to 65	1
inclusion D	387	303–315	3	–64 to 0	1
inclusion F	527	444–452	2	–64 to 0	1
femora	772	681–693	3	–64 to 65	1

^aThese scans should also have covered inclusion A.

2.2. X-ray diffraction scans

Conservation of HPM4 was partially complete when diffraction mapping was performed: some soil remained at undocumented locations [16]. Transmission X-ray diffraction patterns were collected at beamline 1-ID (figure 2a), the Advanced Photon Source (APS, Argonne National Laboratory) using 71.676 keV photons and a (0.25 mm)² wide microbeam. The X-ray area detector (Dexela 2923 CMOS detector; 3888 × 3072 pixels, 75 μm pixel pitch) was nominally perpendicular to and centred

on the incident X-ray beam and mounted on a translator allowing the specimen–detector separation to be varied. Ceria powder (NIST SRM-674a, 1 mm thickness) was used to determine detector position and (small) tilts relative to the incident beam [17]. Using the CT roadmap, diffraction patterns covered the objects of interest by translating HPM4 vertically and horizontally across the beam; scanning all of HPM4 was impractical (electronic supplementary material). Collecting each pattern required 4 s (detector integration time of 0.2 s to avoid saturation; readout time and motor motion with shutter closed). Diffraction patterns consisted of complete or incomplete diffraction rings and discrete diffraction spots.

An approximately 25 kg weight of HPM4 was accommodated by translators allowing approximately 1.2 m horizontal (X) and 200 mm vertical (Y) translations with precision/accuracy/reproducibility approximately 0.25 mm (X-ray beam width). Furthermore, HPM4 remained supine; figure 1b shows it positioned for diffraction mapping.

The crystal periodicities d_{hkl} (d -spacings) are determined from the angles $2\theta_{hkl}$ at which each hkl diffracted beam makes with the incident beam via Bragg's law:

$$\lambda = 2d_{hkl} \sin \theta, \quad (2.1)$$

where λ is the X-ray wavelength and θ is the Bragg (diffraction) angle (e.g. [18]). When area detectors record transmission X-ray diffraction patterns (figure 2a), the separation between diffracting volume and detector must be known. Figure 2b illustrates a complication of large paths through specimens such as HPM4. At detector position 1 (nominally 525 mm from the centre of HPM4), diffraction peaks might be from position A or B or anywhere between: the angular and d_{hkl} uncertainties are too large to be useful. Repeating the measurement at detector position 2 (400.0 mm farther than position 1) resolves the ambiguity and

Figure 3. Six CT slices (transverse sections) through HPM4; the anterior surface of the mummy is up in these images. White contrast indicates the area of higher X-ray absorption. (a) Slice through the skull ($x = 237$ mm) with labels: Inclusion A 'incl A', skull 's', skin 'sk', resin shards 'rs' and wire 'w'. An enlargement of the area around the wire is shown within the inset yellow box. (b) Slice through the head intersecting teeth 't' and the portrait 'p'. Soil on the outside of the mummy is labelled 'so' ($x = 280$ mm). (c) Slice through a cervical vertebra 'cv' with dense linen layer ℓ_1 and layer ℓ_2 identified ($x = 326$ mm). (d) Slice through the thorax with inclusion D within the wrappings with one humerus 'h' and one rib 'rb' labelled ($x = 387$ mm). (e) Slice through the abdomen with high absorption inclusion F within the abdomen's wrappings and radius and ulna labelled 'incl F' and 'r, u', respectively ($x = 527$ mm). (f) Slice through the femora 'f', muscle 'm' and high absorption particle 'q' ($x = 769$ mm). (g) Area near shoulders ($X = 340$ mm) with a layer of absorbing particles very near the body's posterior surface. The shoulders are in the upper portion of the image, and the particles run from the left to the right shoulder. (h) Relatively high absorption (1400–1500 HU), large particle (major diameter approx. 5 mm) near one shoulder ($X = 356$ mm). (i) Area near one hip (greater trochanter is in the upper right) showing an unusual hollow particle ($X = 615$ mm). The scale bar for (a–f) is shown in the lower left corner of (c), and the vertical field of view for (g–i) is 41 mm.

both identifies the depth from which the diffracted beam originates and provides accurate d_{hkl} .

The beamline's alignment lasers' intersection approximately located HPM4 relative to the X, Y position of the beam at $Z = 0$ (figure 1b). Diffraction-based alignment scans (detector position 1) registered X, Y, Z and x, y, z coordinate systems. First, an X-scan at $Y = 0$ located where the cranial wrappings began (the first diffraction spots appeared) and where the crown of the skull began (bone diffraction patterns appeared). Next, Y-scans across the skull at $X = 111$ and 100 mm ensured that the vertical translation range covered the expected positions of the resin shards, wires near the skull, teeth, femora, cervical vertebrae, inclusions F, A and D. An additional Y-scan at $X = 42$ mm covered only wrappings and confirmed the registration.

Scans across all of the regions of interest were performed at detector position 1 and then at detector position 2 (table 2 and figure 2c). The diffraction patterns were collected as a series of Y-scans (translation increment $\Delta Y = 0.5$ or 1 mm and approximately 130 patterns per Y scan) at 5–7 X positions (translation increment $\Delta X = 1, 2, 3$ or 6 mm between Y-scans, depending on the object's size measured via CT). The X positions were selected based upon the objects' distance from the crown. Between scans at detector positions 1 and 2, diffraction patterns were recorded at both detector positions of reference materials (an archeological human bone, a second metacarpal, MC2, and a steel insect pin thought to be similar to the wires inserted into the wrappings) placed beyond the cranial end of the mummy.

After detector tilt correction, the 2D polar geometry diffraction patterns (diffracted intensity versus radii r) were converted to Cartesian plots $I(r, \eta)$: circular Debye rings covering all

azimuths η transformed to lines and discrete spots at a specific η remained spots at that η . Note that constellations of diffraction spots (or patterns of closely versus widely diffraction lines) at detector position 1 are preserved (albeit stretched along r) at detector position 2; characteristically shaped diffraction spots kept their shape at both detector positions and not all features could be seen at both detector positions. Next, radii r_1 and r_2 (at the same η) were measured for a spot/line. From the radii and the known detector translation (400.0 mm), extrapolation to $r = 0$ revealed the Z position originating the diffracted intensity. With Z known, 2θ is calculated from trigonometry and d from equation (2.1). The series of d from the same X, Y, Z are characteristic of the crystallographic phase(s) present, and phase identification was done using the Powder Diffraction File (PDF) [19], a database of experimentally determined polycrystalline diffraction patterns.

3. Results

X-ray absorptivities match in CT scans 03 and 07. Differences are always less than 100 HU and, for almost all voxels, less than 25 HU. Different voxel sizes and partial voxel effects [3] produce the small differences.

The longitudinal (sagittal) CT section through HPM4 (figure 2c) shows where the diffraction scans were collected. In figure 2c, broken shards are prominent within the skull vault. Within the cervical and thoracic vertebrae and proximal femur and nearby pelvis, trabeculae are unresolved, as

Figure 4. Two 3D renderings of the mummy and enlargements of the volume of wrappings anterior of the neck. The labels are the same as in figure 3. (a) The wrappings, bone and teeth are shown semi-transparent with darker yellow shades indicating higher attenuation. Wires are rendered green, the resin shards are coloured blue and various objects within the wrapping but having higher linear attenuation coefficient values are shown in magenta. (b) The mummy from a slightly different viewing angle than in the first panel. The bones and teeth are rendered in the same yellow colour scale as in (a), the long bones (femora, tibiae and fibulae) and teeth are rendered opaque, and the front surface of the wrappings has been removed. The dense layer posterior to the skeleton (' ℓ_1 ' in figure 3c) is rendered pink and extends from shoulders to ankles and partway up the sides; two other high absorption layers in the wrappings have much smaller areas and are located anterior of the neck (' ℓ_2 ' in figure 3c) and to the side of the skull (ℓ_3), respectively, and are rendered magenta. The highly absorbing inclusions within the wrappings (above the torso and legs) appear purple. The wires are shown in green, and the white arrow shows additional wire images that were not included in (a). (c) Anterior view, enlarged volume around ℓ_2 (rendered light blue) and a second layer (light green) between ℓ_2 and the neck. (d) Posterior view of the volume seen in (c).

expected given the voxel size, and absorptivities are lower than in the adjacent cortices, reflecting the interiors' smaller partial voxel fractions of bone. On HPM4's surface, the portrait's paint and the soil are highly absorbing. Within the wrappings, inclusion F and several smaller particles (near the calf and foot) have HU values much larger than the surrounding material. In figure 2c, moderately absorbing layers within the lightly absorbing wrappings are visible anterior to the neck and between the body's dorsal surface and the mummy's surface.

Specific features studied with diffraction are shown in the CT slices of figure 3. In figure 3a, wires and inclusion A are visible within the wrappings and resin 'shards' within the cranial vault. The shards and linen are much less absorbing than the skull's cortical bone and than the most

attenuating nearby features, the wires. The teeth, portrait and encrusting soil appear in figure 3b; note the portrait paint is more attenuating than the underlying wood panel. Figure 3c shows a cervical vertebrae and layers ℓ_1 and ℓ_2 (more attenuating than the surrounding wrappings, also present in figure 3d–f). A slice through the thorax (figure 3d) shows one humerus, one rib and inclusion D. The radii and ulnae, abdomen and wrapping-embedded, highly absorbing inclusion F are labelled in figure 3e (abdomen). The femora and surrounding muscle and large particles q1 and q2 appear in figure 3f.

Figure 4 shows HPM4 in 3D with different internal features rendered in different colours. The skeleton's length is 937 mm (top of the skull to the bottom of the feet), and the wrappings add another 50 mm. The maximum mediolateral

Figure 5. (a) Rendering of the interior anterior surface of the skull vault viewed from the interior and showing the nasal perforation used for excerebration. The excerebration hole is labelled 'eh' and the nerve foramina 'nf'. (b) Teeth in 3D shown semi-transparent with the lighter pixels representing more attenuating voxels. The deciduous teeth 'dt' had not been shed at time of death, and the unerupted permanent teeth 'pt' can be seen below the deciduous teeth. (c) Photograph of the portrait after conservation (by Clare Britt for The Block Museum of Art). (d) CT rendering of the portrait before conservation. This image was produced by simultaneous segmentation of voxel values and of voxel gradient values (rendering courtesy of Ajay Limaye, Australian National University). The scales of the panels differ.

width (along *z*) equals 285 mm at the shoulders; the maximum thicknesses (along *y*) are 179 mm at the head and 202 mm at the feet. Figure 4b, discussed below, shows the posterior, high attenuation layers within the wrappings in 3D.

3.1. Human remains

The CT data show that all bones are present and well preserved and, with only minor exceptions, remain articulated in an anatomical position. Several ribs and the right femur's distal epiphysis have been displaced after death, a common occurrence when remains are moved after prolonged post-mortem intervals. The connective tissues holding bones together have degraded, including those bones or portions of bones that fuse later during development. The anterior cranial fossa is pierced, immediately left of the midline through the sphenoid body and lesser wing, creating a passageway between the nasal and cranial cavities (figure 5a). Transnasal excerebration would create such a hole and is a standard although not universal procedure in Egyptian mummification [20]. Otherwise, skeletal trauma is absent, leaving cause of death indeterminate.

Deciduous dentition is complete, although several of these teeth were displaced post-mortem, and none of the permanent teeth have erupted (figure 5b). Crown enamel of the first and second permanent molars is partially developed and can be visualized in their crypts. Standards based on large aggregates of well-characterized data [21] indicate that age at death was approximately 5 years (± 0.75 yr).

Soft tissue is well preserved, but muscles appear shrunken, likely due to dehydration during mummification

(figure 3d–f). The skin remains in close contact with the innermost layer of wrappings, consistent with tight wrapping of the desiccated body when it was prepared and with no subsequent loosening. Isolating the skin's surface from the wrappings would require painstaking manual segmentation because of the small difference in CT contrast, the close contact and the noise in the reconstruction; such subjective segmentation is not the focus here. Internal organs other than the heart have been removed, and the heart is too desiccated and collapsed for its structures to be discerned.

Male external genitalia¹ could not be seen, and some of the external soft tissue could be labia (external female genitalia). Internally, the end of the alimentary canal (rectum) is visible, but the internal urogenital organs towards the ventral side of the pelvic cavity cannot be visualized, because they are either flattened and dried together or were removed with the rest of the abdominal organs.

3.2. Mummy exterior and wrappings

When imaged, soil covered large fractions of HPM4's surface, but its thickness and coverage was quite irregular. Absorption coefficients were typically greater than 1500–1600 HU, and soil thickness reached 9 mm on the posterior thorax. Conservation prior to the diffraction studies removed much of this soil, but the location of the remaining soil was not recorded.

The portrait depicts a young adult woman but not a child (figure 5c). The portrait contains enough X-ray contrast for voxel value plus voxel gradient segmentation to reveal a CT image nearly identical with the portrait's photograph (figure 5d).

The wrapping's individual linen layers can be resolved at many locations, and the layers' mean linear attenuation coefficient is -540 ± 95 HU,² compared with -990 HU for air. Although HPM4 is tightly wrapped, the space between the linen layers and within the weave produces the relatively large standard deviation. Dense layer 1 within the linen wrappings (-65 ± 95 HU) follows the contours of the body (figure 3c) and spans the majority of the body's dorsal surface as well as its right and left sides (figure 4b). Layer ℓ_1 is continuous but contains small gaps often where the layer appeared thin in adjacent slices. At the humeral midshaft, approximately seven linen layers are found in the 10–23 mm between the mummy's skin and layer ℓ_1 , and an additional nine within the 20–28 mm separating layer ℓ_1 and HPM4's external surface. At the femoral midshaft, the skin-layer ℓ_1 separation is somewhat larger than that at the humerus (17–25 mm) with seven linen layers, and the distance between layer ℓ_1 and the external surface is 30–35 mm and includes nine layers of linen. The tibial midshaft has a skin-layer ℓ_1 separation of 22–25 mm (six linen layers) and layer-external surface separation of 27–31 mm (nine layers).

Layer ℓ_2 (figures 3c and 4b–d) is much thicker and more absorbing than ℓ_1 ; its mean linear attenuation value is 354 ± 56 HU, about the same as inclusions A and D (table 3) and other inclusions present in figure 4. Layer ℓ_2 extends over a number of slices, and a layer ℓ_3 with similar absorption and somewhat greater thickness than ℓ_2 is on one side of the thorax (figure 4a). It appears that ℓ_1 , ℓ_2 and ℓ_3 are parts of the same layer of wrapping. Enlargements of the volume surrounding ℓ_2 are shown in figure 4c,d. Figure 4c shows an anterior view of ℓ_2 ; this area of higher absorption than linen also has a 'tail' running towards feet along the body midline.

Table 3. Typical linear attenuation values of different features in HPM4. The column A/P denotes whether the mean value of the linear attenuation was obtained from an area (A) or a series of points (P). The column *N* gives the number of voxels averaged. The mean linear attenuation coefficient and its standard deviation are in HU.

feature		A/P	<i>N</i>	mean	s.d.
air (inside skull)		A	1369	−990	3
cortical bone	skull	P	10	1260	190
	femur	P	80	1485	136
cervical vertebral body		A	237	−292	235
teeth	vertebra cortex	range	1200–1400		
	dentine (crown)	P	86	2028	174
	dentine (root)			~ 1200	
	enamel	P	82	2970	130
wires				→1000	
low absorption shards		A	407	115	17
wrappings		A	3249	−537	84
		A	3249	−541	107
	dense layer ℓ_1	P	32	−64	96
	dense layer ℓ_2	A	72	354	56
inclusion A		A	150	380	59
inclusion D		A	78	336	28
inclusion F		A	39	3047	52

A smaller second layer (also more attenuating than linen) lies underneath ℓ_2 (green) in figure 4c and is seen in its entirety in the posterior view (figure 4d).

A layer of highly absorbing, millimetre-sized particles lie very close to the posterior surface of the body (figure 3g–i). Absorption values range from 100 to 1500 HU, and the particles are densely packed near the shoulders (figure 3g,h) but much fewer in number below the rest of the torso and legs. If smaller particles are present in these slices, partial volume effects may limit their visibility. Near the hips and in the same layer of wrappings, a large diameter (5–6 mm), hollow object is present (figure 3i). The path used to access tissue from the left shoulder for a biopsy performed in 2000 was visible (data not shown, see [11]).

The diffraction data of figure 6a,b plot azimuth η vertically and detector radius r horizontally and show two constellations of discrete diffraction spots from the same position on HPM4 but different detector positions (yellow ellipses). These diffraction spots are clearly related and trace back to HPM4's surface. Every discrete diffraction spot traced back to its origin, originated at the mummy's surface. The d -spacings of these discrete spots are consistent with quartz, low, synthetic (PDF 00-033-00161). At position X where Y scans were expected to traverse layers ℓ_1 – ℓ_3 , no diffraction spots or lines could be traced back to these layers' positions.

3.3. Wires

Thirty-six thin, linear and highly absorbing features appear within the wrappings and connect to HPM4's surface. These linear features are concentrated near the ends of the mummy where linen curves the most (11 surrounding the head and neck, 20 around the feet and five near the torso). These objects' lengths and diameters average 33.7 ± 3.7 and

less than 1 mm, respectively. Linear attenuation coefficients approach 1000 HU, with contrast typically spread over two or more voxels. These observations suggest that the linear objects are straight wires or specimen pins with sub-voxel diameters (see Discussion).

Figure 6a,b shows diffraction patterns from X, Y = 163, 4.5 mm for detector positions 1 and 2. A pattern from the reference specimen pin is shown for comparison (figure 6c). Background is high at small r , drops monotonically with increasing r and is much higher for all of the patterns from the mummy than for the reference specimen in air. In figure 6a,b, three diffraction lines (1–3) are visible and possess strong azimuthal intensity variation (see also electronic supplementary material), characteristic of the crystallographic texture of formed metals (e.g. [13]); where the first two lines have appreciable intensity, the peaks are three to five times more intense than the adjacent background. Low background allows observation of three additional lines in figure 6c (reference pin), and azimuthal intensity variation matches that in figure 6a,b. For figure 6a–c, the diffraction line at the second lowest radius has maximum azimuthal intensities three or four times larger than the azimuthal minima, and the minima are only slightly above background. At X differing by 6 mm from that in figure 6a,b, matching azimuthally modulated diffraction lines were observed at a different Y position, consistent with a linear object oriented obliquely to the incident beam.

Patterns matching that of figure 6a,b were noted at two other positions, one at X, Y = 163, 21 mm, i.e. the same X but 16.5 mm lower than figure 6a,b and one near the torso. The diffraction lines persisted at only over $\Delta Y = 0.5$ mm. In anticipation of the phase identification below, source of the azimuthally modulated diffraction lines varying intensity are termed wires.

Figure 6. Diffraction patterns of wires and of the skull (bone) transformed to Cartesian coordinates (radius on detector on the horizontal axis and azimuth on the vertical axis; the lighter the pixel, the greater the diffracted intensity). Patterns (a–e) cover 360° in 1° azimuthal bins vertically, patterns (a–d) cover 1250 detector pixels ($@ 74.8 \mu\text{m}$) horizontally and the enlargement (e) covers 200 detector pixels horizontally. Diffraction angles increase from left to right, and the lighter the pixel, the higher the diffracted intensity. All labels are explained in the text. (a) Diffraction pattern from a wire within the mummy wrappings at detector position 1. (b) Diffraction pattern from the same wire as in (a) but at detector position 2. (c) Diffraction pattern from a reference specimen pin at detector position 2; note that the pin was positioned somewhat farther from the detector than the mummy. (d) Diffraction pattern from the skull. (e) Enlargement of the portion of the diffraction pattern in (d) containing the first two diffraction lines of bone (i.e. the skull). Scalloping at the upper and lower right corners of (a–c) and on the left side of (d) and (c) represents azimuths that were not covered by the detector.

The six diffraction lines of the reference specimen pin extrapolate to the same origin with a standard deviation in Z of 1 mm; the origins determined for wires within the mummy have similar precision. For $X = 163 \text{ mm}$, the origin of wire at $Y = 4.5 \text{ mm}$ was $Z = 599 \text{ mm}$ and that at $Y = 21 \text{ mm}$ was $Z = 418 \text{ mm}$, i.e. positional differences $\Delta Y = 16.5 \text{ mm}$ and $\Delta Z = 181 \text{ mm}$. In CT dataset 03, the wires in slice 244 have $\Delta y = 13 \text{ mm}$ and $\Delta z = 184 \text{ mm}$ (figure 7a), and in nearby slice 234, $\Delta y = 16 \text{ mm}$ and $\Delta z = 193 \text{ mm}$. The positional differences between CT and X-ray diffraction datasets are attributable to differences in mummy alignment. The wires' d -spacings (reflections 1–3, figure 6a–c) agreed with those from the reference

specimen pin, a modern dual-phase steel (table 4) and those from bcc and fcc iron–carbon phases (table 4).

3.4. Skull

The skull's cruciform eminence (inner, posterior surface) can be seen in figure 3a (below and slightly left of label 'rs'). The skull's sides reach thicknesses up to 3–4 mm, but the bone is typically thinner than this. At 10 transects of the posterior skull (figure 3a), the maximum attenuation averaged $1250 \pm 190 \text{ HU}$.

In diffraction scans of the skull, the X-ray beam traversed both the left and right sides, and these were far enough apart that their diffracted beams separated on the X-ray detector. Figure 6d shows a typical diffraction pattern from the skull with L1 and H1 being 00.2 cAp diffraction lines from the two sides of the skull and L2 and H2 being the cAp unresolved quadruplet lines; figure 6e enlarges part of the pattern. Lines L1 and H1 show strong azimuthal modulation of intensity (maxima approaching 6200 cts and minima 1300 counts lower and 600 cts above background), and lines L2 and H2 somewhat weaker intensities and modulation. Diffracted intensities are quite a bit lower for cAp than for the wire, a characteristic of bone. Figure 7b plots the origins of the 00.2 diffracted beams (L1 and H1) for different Y at $X = 100 \text{ mm}$, and the skull's shape matches in figure 7b and a, including left- and right-side asymmetry. The maximum lateral skull width from diffraction is 135 mm; in figure 7a, the maximum width is 134 mm.

3.5. Low absorption shards

Low absorption shards are prominent within the skull vault and have very uniform attenuation ($115 \pm 17 \text{ HU}$), considerably greater than air ($-990 \pm 3 \text{ HU}$), linen or dense linen layer $\ell 1$ (table 3), and uniform thickness of 10–11 mm. Virtual assembly of the shards [23] shows that they are an endocast of the region surrounding the cruciform eminence. When the endocast separated from the bone, small pieces of bone (linear attenuation values matching nearby cortical bone) remained attached to the shards (inset in figure 7c).

3.6. Teeth

Contrast from the tooth crown dentine averages $2028 \pm 174 \text{ HU}$, values for root dentine are approximately 1200 HU and for tooth enamel average $2970 \pm 130 \text{ HU}$. There is no evidence of extraordinary tooth wear or of *in vivo* damage. Diffraction patterns from scans across the teeth show complex variation in crystallographic texture: figure 8a,b shows patterns separated by 1 mm and trends are not evident. At each position, the X-ray beam encountered two or more teeth and bone and multiple cAp containing phases (enamel, dentine, cementum and bone) with different textures. Analysis of the many overlapping reflections would require finer spatial sampling at more detector positions; therefore, diffraction analysis was not pursued further.

3.7. Cervical vertebrae

The trabeculae within the (cervical) vertebral bodies cannot be resolved (figure 3c), and the mean attenuation value $-292 \pm 235 \text{ HU}$ reflects the significant partial volume effect. The cortical bone of the vertebral arch has attenuation

Figure 7. CT slices and plots of origins of diffracted intensity. (a) CT slice through the skull ($x = 244$ mm) with separations between the wires indicated. (b) Origins of the 00.2 diffracted beams (L1 and H1) for the Y scan at $X = 100$ mm. Note the Z axis is reflected relative to (a). (c) CT slice through the skull showing resin shards with an enlargement (inset upper right) showing small fragments of bone (yellow arrowheads) attached to the resin. (d) Origins of 00.2 diffracted beams (L1 and H1 in figure 8d) for the femora. The corresponding CT slice appears in figure 3f.

coefficients of 1200–1400 HU. The authors were unable to identify diffraction from the cervical vertebrae.

3.8. Inclusions A and D

Inclusions A and D had comparable contrast: 380 ± 59 and 336 ± 28 HU, respectively (table 3). Diffraction scans crossing inclusions A and D did not detect diffracted intensities.

3.9. Inclusion F

Figure 3e shows inclusion F in the wrappings on the ventral side of the abdomen at the level of lumbar vertebra 4 (L4). Inclusion F is highly attenuating (3055 ± 24 HU, near the scan's maximum). The three principal diameters of the nominally ellipsoidal inclusion F were 7, 5, 3 mm along the x , z , y axes, respectively.

The diffraction patterns of inclusion F (figure 8c) contained six diffraction lines (numbered 1–6 from lower to higher r , see intensity versus radius plot of electronic supplementary material, figure S3) with uniform azimuthal intensities (see electronic supplementary material, figure S4). Peak 1 is the most intense averaging 5150 ± 650 cts pixel⁻¹ versus background of 2200–2300 cts pixel⁻¹. Diffraction patterns from inclusion F were observed over 7 mm translation along X and 5 mm along Y . A total of 28 extrapolations could be used to determine the Z positions originating diffraction (combinations of different X , Y positions and peaks 1–4). The mean depth was $Z = 545.5 \pm 1.7$ mm. Six extrapolations were outliers and were at inclusion F's edges; disregarding

these outliers yields $Z = 545.8 \pm 0.7$ mm. This origin matches inclusion F's position from CT.

The d -spacings of the first four diffraction lines of inclusion F match those of calcite (PDF 00-005-0586; table 4). The experimental values of d_{104} are systematically lower than those of the PDF card, averaging $(d_{PDF} - d_{104})/d_{PDF} \sim 4.5 \times 10^{-3}$, about five times larger than the measured standard deviations $\sigma \Delta d_{\sigma}/d = 9 \times 10^{-4}$ (table 4). Magnesium is a common substitution in natural calcite, and d_{104} for $\text{Ca}_{1-x}\text{Mg}_x\text{CO}_3$ with $x = 0.03$ (PDF 00-086-0306) is shown for comparison in table 4 and for other compositions in electronic supplementary material.

3.10. Femora

In figure 3f, the cortical thickness is approximately 3 mm, and the medial–lateral and anterior–posterior outer diameters average 15–16 and 13 mm, respectively. The anterior–posterior gap between femora is approximately 5 mm. Linear attenuation coefficients approach 1500 ± 135 HU. Figure 3f also shows three highly attenuating inclusions within the wrappings: plate-like q1 $8 \times 3 \times 6$ mm, q2 measuring up to 3 mm and a third unlabelled particle (out of Y-scan range). Attenuation of both q1 and q2 exceeds 2000 HU.

A typical femoral diffraction pattern (figure 8d) contains split cAp 00.2 reflections (L1 and H1) from the bone's medial and lateral sides and split cAp 00.4 reflections (L1' and H1'). Splitting of the cAp quadruplet reflection cannot be resolved. The dashed line indicates the femur's longitudinal axis, and azimuthal intensities of 00.2 and 00.4 are strongly peaked

Table 4. Mean *d*-spacing (s.d.) for most intense diffraction peaks from different objects and compared with PDF reference patterns.

	peaks (Å)						
phase		d_1	d_2	d_3	d_4	d_5	d_6
	Y (mm)						
wire	4.5	2.090	2.049	1.812			
	21.0	2.094	2.053	1.819			
specimen pin		2.090	2.045	1.809	1.449	1.281	1.183
BCC ^a			2.0268		1.4332		1.1702
<i>hk.l</i>			110		220		211
FCC ^b		2.08		1.80		1.27	
<i>hk.l</i>		111		200		220	
inclusion F		3.019 (0.003)	2.483 (0.003)	2.273 (0.001)	2.086 (0.002)		
calcite ^c		3.035	2.495	2.285	2.093		
<i>hk.l</i>		1.04	11.0	11.3	20.2		
Ca _{0.94} Mg _{0.06} CO ₃ ^d		3.025					
bone (skull)		3.430					
bone (femur)			1.728				
hAp ^e		3.4325	1.716				
<i>hk.l</i>		00.2	00.4				

^aPDF 00-006-0696 for ferrite.^bPDF 00-031-0619 update with values from 00-033-0397 for austenite.^cPDF 00-005-0586 for calcite.^dPDF 00-086-0306 for Ca_{0.97}Mg_{0.03}CO₃.^ePDF 00-086-1201 for synthetic hAp.

along this direction (figure 8e). The maximum 00.2 femur diffracted intensities have pixel values lower than those from the skull (4300–4500 cts versus 6200), and the femur 00.2 minima were only 1000 cts lower than the maxima and were comparable with background. The background from the surrounding wrappings is too high to allow much analysis beyond determination of the 00.2 and 00.4 peak positions. Figure 8f shows that much higher quality diffraction patterns can be obtained from similar aged bones (archeological human MC2) when the mummy is not contributing background: the ratio of 00.2 peak to the adjacent background intensity is 1.9 for the MC2 and 1.1 for the HPM4 femur.

For $X = 687$ mm, bone diffraction patterns appear at $9 \leq Y$ (mm) ≤ 20 (femur 1) and at $26 \leq Y$ (mm) ≤ 38 (femur 2). Diffraction shows anterior–posterior femoral diameters of 11 and 13 mm and a 6 mm gap between bones. Splitting of cAp 00.2 and 00.4 is absent at $Y = 9, 10, 19$ and 20 mm and at $Y = 26, 27, 36, 37$ and 38 mm; double peaks are between these Y positions. Evidently, separations of ΔZ approximately 5 mm are required before separate objects (medial, lateral cortices) can be resolved. The 00.4 cAp *d*-spacings were about what is expected (table 4). Figure 7d shows the positions originating the 00.2 cAp diffraction lines; this cross-section can be compared with the matching CT slice in figure 3f. The separation of the femora's medial surfaces is 43.5 mm from diffraction and 42.5 mm from CT.

Diffraction patterns from a position below femur 2 match that of inclusion F and are from calcite. This position matches the CT position of inclusion q1 (figure 3f).

4. Discussion

The CT-derived 3D roadmap was essential to the diffraction study. Without this information, covering HPM4's sagittal (longitudinal) cross-section (e.g. figure 2c; electronic supplementary material, figure S1) would require up to 19 days (at 4 s pattern⁻¹, see electronic supplementary material), and only 24 h was available (with set-up, 16 h for data collection). The X-ray beam position was less than 1 mm from the alignment laser positions, but registration of CT and diffraction coordinate systems based on HPM4's exterior is approximate and rotations were expected between the placement of HPM4 in the CT scanner and in the diffraction set-up. Collecting a series of Y -scans at closely spaced X insured sampling of the objects of interest and diffraction-based measurement of dimensions of features like inclusion F.

4.1. Age and sex assessment

Assessing the biological profile of the person within HPM4 requires estimates of biological sex, age at death, ancestry and stature. The height of the skeleton is 937 mm, and, as the mummy remains in articulation, this is the likely stature, suggesting that the remains are a child's. This inference is corroborated by the degrees of skeletal and dental growth and development. The vast majority of the bones or portions of bones that fuse during development remains separate. Skeletal growth can be affected by environmental factors (malnutrition, illness and other stressors) and genetic factors;

Figure 8. Diffraction patterns with lighter pixels representing greater diffracted intensities. Patterns (a–e) cover 360° in 1° azimuthal bins vertically and (a–d) cover 600 detector pixels (@ $74.8 \mu\text{m}$) horizontally and (e) cover 200 detector pixels horizontally. (a) Diffraction pattern from teeth. (b) Diffraction pattern from a tooth position $\Delta Y = 1 \text{ mm}$ away from that in (a). (c) Diffraction pattern from inclusion F. The first six diffraction lines are labelled '1–6'. These lines index as calcite (PDF 00-05-586), see also table 4, with $hkl = 10.4, 11.0, 11.3, 20.2, 01.8$ and 11.6 , respectively. (d) Diffraction pattern from a femur with 00.2 cAp lines from the two sides of the femur labelled 'L1' and 'H1', the quadruplet reflection labelled 'q' and the 00.4 cAp labelled 'L1'' and 'H1''. The horizontal dashed line (black) shows the femur's longitudinal orientation. (e) Enlargement of area of (d) containing the cAp 00.2 diffraction lines. (f) Diffraction line profile at the position of the dashed line in figure 8d (avoiding as many as possible of the sharp, intense diffraction spots) compared with a diffraction line profile for an archaeological bone specimen (recorded immediately before the mummy scans but without the mummy in the beam). (top) Pattern from one of the mummy's femora (labelled 'femur') with cAp 00.2 reflections L1 and H1 and quadruplet q indicated. (bottom) Pattern from an archaeological human second metacarpal bone (labelled 'MC2') with 00.2 and quadruplet peaks indicated. The vertical arrowheads indicate possible splitting of reflections from the two sides of the diaphysis.

therefore, stature- and skeletal development-based measures of age at death can be biased. Dental development provides a much better age indicator because it is under tight genetic control in modern human populations and is largely unaffected by environmental factors [24]. The complete deciduous dentition is present, but none of the permanent teeth have erupted (figure 5b). The developing crown enamel can be seen in the crypts of the first and second permanent molars. Age at death of 5 ± 0.75 years estimated from [21] is consistent with height and bone development.

Ancestral affiliation and biological sex are difficult if not impossible to determine from juvenile human skeletal remains. Craniofacial morphology of adult skeletal remains routinely provides robust estimates of ancestry, but these techniques cannot yet be applied to subadults (but see [25]). Therefore, ancestral affiliation was not estimated here. In adults, the pelvis is most frequently used for estimating sex, but, because changes to pelvic morphology arise due to pubertal hormonal surges, these sex-based differences are not appreciable during childhood [26]. Methods involving other skeletal areas, such as the skull and postcrania, are population-specific, and no juvenile reference sample exists for HPM4.

Soft tissue is still largely present in HPM4, and CT images might be used to determine sex via the most obvious visual

cue, presence or absence of external genitalia. Male external genitalia were not seen, and some of the external soft tissue could be labia (external female genitalia). Internal urogenital organs could not be visualized. X-ray imaging supports, therefore, what is seen in the portrait, namely the subject was biological female.

4.2. Wrappings and encrusting soil

Soil on the outside of HPM4 complicated diffraction analysis. X-ray diffraction was performed midway through conservation (i.e. soil removal) but where soil was removed was not recorded (R. C. Sabino 2018, personal communication, see [16]). The density of diffraction spots attributable to HPM4's surface (soil) varied considerably with position (e.g. figure 6a,b versus figure 6d and figure 8a versus figure 8b–d) and may be due to sampling conserved versus pre-conserved regions.

Most linen layers possessed mean linear attenuation coefficients of $-540 \pm 95 \text{ HU}$. The contrast of dense layer 1 averaged $-65 \pm 95 \text{ HU}$, consistent with linen impregnated with pitch (resin shards' contrast was $115 \pm 17 \text{ HU}$). The gaps in layer $\ell 1$ (see Results) could be real or be areas small contrast differences and finite resolution made the layer 'disappear'. The amount of linen in HPM4 and the resources it represents were discussed elsewhere [11].

4.3. Wires

Figure 7a shows a CT slice through the skull ($x = 244$ mm) and the separations between the two wires outside the skull were $\Delta y = 13$ mm and $\Delta z = 184$ mm for this slice and $\Delta y = 16$ mm and $\Delta z = 193$ mm for $x = 234$ mm. From diffraction, the two wires were separated by $\Delta Y = 16.5$ mm and $\Delta Z = 181$ mm. Given X – Y – Z was not perfectly parallel to x – y – z (relocation of HPM4), agreement is reasonable.

X-ray diffraction identified the wires as a modern dual-phase steel: diffraction patterns from wires within HPM4 were identical with those of the reference specimen pin, including identical strong characteristic azimuthal intensity variation. The wires and pin contain austenite and ferrite phases (e.g. PDF 00-006-0696 for ferrite and 00-033-0397 for austenite), and such wire-based repair is commonly observed in CT scans of Egyptian mummies [27].

The wires were concentrated where the wrappings curve the most, suggesting that the wires were inserted to stabilize HPM4's outer layers. The wire lengths averaged 33.7 ± 3.7 mm, and this agrees with 37 mm lengths of commercial specimen pins (whose lengths include a nylon head and angle ground point [28]). In HPM4, the wires' images spread across one or two voxels, and contrast only approached 1000 HU whereas a full voxel of steel would exceed 3000 HU [29]. The wire diameters, therefore, must be smaller than the in-plane voxel dimension (0.82 mm). A commercial 0.5 mm diameter pin could occupy up to 29% of the voxel, and given that linear attenuation scales linearly with partial volume [30], one expects attenuation values approaching 1000 HU. The CT data are thus consistent with 0.5 mm diameter steel specimen pins.

4.4. Low absorption shards

The resin shards (solid pitch) within the skull had absorption values of 115 ± 17 HU, matching published values for resin in mummies (71 ± 23.4 HU; [4]). The shards are an unusual endocast. Resin solidified in skulls of most Egyptian mummies with the body lying supine (e.g. [20,31,32]) although that of Tutankhamun shows two orientations [2]. In HPM4, the uniform thickness fragments follow the skull's internal contours, suggesting that the body was tilted continuously while the resin solidified.

4.5. Inclusion F

Diffraction shows that inclusion F consists of calcite. From CT, its absorption coefficient is greater than 3000 HU, the scan's maximum, somewhat higher than 2650 HU in the literature [4]. Based solely on attenuation, one might identify the inclusion as garnet or malachite [4], but different scanner calibrations and/or reconstruction software settings could disproportionately affect upper extremes of contrast.

Diffraction mapping and CT locate inclusion F at the same position within HPM4: at the L4 level within the wrapping ventral to the abdomen. The CT and diffraction diameters also match. From the 28 extrapolations (different reflections, different X , Y), the standard deviation of Z origin of calcite diffraction was ± 1.7 mm, and some spread in Z origin is expected, given inclusion F's thickness. With extrapolation of only two detector positions, a few origin outliers can be expected; similar variability appears for the skull and femoral origins, see below. Origin determinations with only two detector positions can be quite accurate and precise, therefore, provided that multiple extrapolations are used.

Scarabs have been observed by CT of intact Egyptian mummies [27,33]; two mummies have high absorption scarabs, somewhat larger than inclusion F and with somewhat lower absorptivity, on the abdomen at the lumbar vertebral levels [27]. Scarabs recovered from within the pelvis of skeletons from this era had dimensions only slightly larger than inclusion F [34]; a scarab positioned above L4 would be expected to fall within the pelvis. Calcite amulets are unusual but not unheard of in mummies, e.g. [35]. The positioning of inclusion F (level of L4, above the abdomen) is consistent with placement to protect (spiritually) the site of evisceration [20]. Inclusion F's long axis is parallel to the cranio-caudal axis, and this might argue for purposeful introduction.

On the other hand, other highly absorbing particles exist elsewhere within the wrappings. Inclusions q1 and q2 have contrast approximately 2000 HU, and X-ray diffraction identifies q1 as calcite. Two of the three diameters of inclusion q1 are a bit larger than F, but q1 is plate-like rather than ellipsoidal. One suspects that q1, q2 and several other inclusions with similar contrast are limestone debris, mainly calcite with admixture of variable amounts of other, less absorbing mineral phases.

Inclusion F's diffraction lines contain many sharp, closely spaced spots with minimal radial broadening; this indicates fine grains with micrometre sizes. For the multiple peaks and multiple positions of inclusion F, d -spacing precision can be determined. For d -spacing standard deviations Δd_σ (table 4), $\Delta d_\sigma/d = 9 \times 10^{-4}$ for this calcite. The d -spacing difference between pure calcite (d_{PDF} no Mg) and the present experiment averages $\Delta d_{\text{PDF}}/d \sim 4.5 \times 10^{-3}$, five times larger than $\Delta d_\sigma/d$. Ancient Egyptian limestone quarries were dolomitic (i.e. containing some to significant amounts of Mg) [36], and the same would be true of more decorative forms of calcite. The composition $\text{Ca}_{1-x}\text{Mg}_x\text{CO}_3$ of inclusion F appears, therefore, to be $0.03 < x < 0.06$ and closer to the lower value.

4.6. Other inclusions, cervical vertebrae and teeth

Diffraction was not observed from inclusions A and D. Contrast from A and D (380 and 336 HU, respectively) is slightly greater than that of the resin shards. A small amount of mineral probably produces the contrast, but diffraction is too faint to be seen above background. Now that conservation is complete, it might be possible to find signal from A and D, but much longer counting time may be required to detect the signal.

Diffraction from the bone in the cervical vertebrae was not observed. There is enough cAp to produce detectable 00.2 peaks, but the density is lower than in the femora or skull and the mineral spread over Z approximately 10 mm. The resulting radial intensity spread on the detector submerged the vertebra's signal within the background.

Diffraction patterns from teeth (and the surrounding bone) present the opposite challenge from the cervical vertebrae: the multiple diffraction arcs change too rapidly between positions, and arcs from different depths merge. If 3D diffraction mapping of the teeth and associated bone were a priority, much smaller lateral mapping steps and more detector separations would be required.

4.7. Skull and femora

The CT contrast from the skull and femora cortical bone is what one would expect. The skull cross-section from diffraction agrees with CT (figure 7b is reflected relative to figure 7a).

Diffraction and CT also agree for cross-sectional femoral dimensions and for relative femora positions. For the skull, a pair of points have anomalous origins, and for the femora some scatter in origins is present. Both effects probably result from low peak intensity plus high background intensity (figure 8f) and use of only two detector positions. At the anterior and posterior of each femur, a pair of origins are stacked on top of each other; this results from resolving only one origin where the beam passes tangentially through the cortex.

Figure 8f compares 1D diffraction profiles (intensity versus radius) of a femur within HPM4 with an archaeological MC2 (displaced along Y from HPM4). Background is very high for the former and drops monotonically, characteristic of semi-crystalline and non-crystalline materials such as textiles (i.e. the linen wrappings); non-resolved, lower intensity soil diffraction also contributes. Background is relatively flat and much lower than the peaks for the latter. Much longer counting times would improve the cAp patterns relative to the background, but risk of radiation damage, even with high-energy, monochromatic X-radiation which interacts little with specimens, would need to be carefully evaluated.

4.8. X-ray damage

Clinical CT scans of mummies pose little danger of X-ray-induced damage because patient long-term safety requires low doses. As the investigators expected, visible damage to HPM4 was absent after diffraction mapping, i.e. at the first level of concern in cultural heritage studies [37]. Molecular-level damage in mummies is a concern because it may interfere with subsequent DNA extraction. The X-ray dose to bone in HPM4 is estimated at 317 Gy and that to muscle at 84 Gy (see electronic supplementary material for details). It is unclear whether these doses may lead to measurable changes in DNA, but this could be quantified in fragmentary mummies and mummified tissue. On the other hand, CT guidance allowed the irradiated volume to be minimized. Even in densely sampled volumes (e.g. that covering inclusion F, 1 mm² grid sampling), only 1/16 of the volume was irradiated by 72 keV X-rays.

4.9. Improvements and future prospects

The objects studied in HPM4 were sparse and well-spaced. Diffraction from femora (hollow cylinders of bone) indicated that an object's diffraction pattern could be resolved once they were 5 mm or more apart along Z . Even a dozen objects lined up along Z should not be problematic. However, structures with 'diffuse' distribution of mass (i.e. cervical vertebrae) would be difficult to see.

Beamline 1-ID, APS, can record *in situ* radiographs with a horizontal field of view of 2 mm, and stitching to cover larger areas is routine. Because of dose considerations, synchrotron radiographic mapping of HPM4 was avoided and probably should be avoided in the future. On the other hand, collection of a few properly oriented, high-resolution (1–2 μ m pixels) synchrotron radiographs (phase contrast and sharp edge demarcation dominate at this energy) might reveal surface features of inclusion F consistent with a scarab. These radiographs could be recorded covering only the wrappings and not the body.

Sometimes, diffraction spots visible at detector position 1 cannot be seen at detector position 2 even though other matching spots are present. Given translations covering hundreds of

millimetres (X) and multiple Y scans, mechanical inaccuracies could sum to move a micrometre-sized crystal out of the beam by the time patterns at detector 2 positions were recorded. For the vast majority of diffraction lines and spots that are recorded at both detector positions, machine learning approaches might be developed to make it practical to identify the 3D position and d -spacing of all of these features.

The present study shows the value of CT-guided, 3D X-ray diffraction mapping, specifically, X-ray diffraction location and identification of specific crystalline materials (calcite and austenitic–ferritic steel), something not possible with other non-invasive modalities including CT, MRI or terahertz imaging. Furthermore, the diffraction data identified the calcite's Mg content and processing-related crystallographic texture of the wires. Future *in situ* X-ray diffraction analysis of metal or ceramic artefacts (faience) within mummy wrappings may provide important links to studies of *ex situ* mummy artefacts (scarabs and amulets), in particular, manufacturing-related characteristics. Were pathologies such as kidney or gall stones or heart calcifications located by CT, targeted *in situ* X-ray diffraction could identify what crystallographic phases are present, e.g. cholesterol, struvite, apatite, calcite and aragonite in gall stones [9]. It is certainly impractical to scan many mummies at APS (or similar synchrotron radiation sources), but key questions could be answered.

Data accessibility. The CT datasets are hosted at the IMPACT Radiological Mummy database [1] curated at the University of Western Ontario: <https://impactdb.uwo.ca/IMPACTdb/Index.html>. The high-energy X-ray diffraction patterns have been deposited at the Materials Data Facility: <https://materialsdatafacility.org/>.

Authors' contributions. S.R.S. designed the study; obtained loan of the mummy and obtained beam time for X-ray diffraction analyses; supervised CT data collection; contributed to CT data analysis; selected volumes for X-ray diffraction analysis; participated in X-ray diffraction data collection; performed X-ray diffraction analysis; wrote the original manuscript and prepared figures; edited the manuscript. M.K.S. contributed to CT data analysis; performed forensic anthropology analysis; produced 3D renderings; reviewed and edited the manuscript. J.D.A. supervised the set-up for X-ray diffraction experiments; performed X-ray diffraction data collection; performed X-ray diffraction data analysis; reviewed and edited the manuscript.

Competing interests. We declare we have no competing interests.

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Endnotes

¹CT data of the mummy of a 5-year-old boy revealed a penis [22], so one expects scans of HPM4 should reveal this organ, if present.

²Whenever quantities other than age are given followed by ' $\pm$ ', the second number is the standard deviation.

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