

Quantitative imaging of spin and orbital magnetic moments in subsurface ultrathin layer with soft x-ray dichroic spectroptychography

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Quantitative evaluation of magnetic moment distributions in microstructures with high spatial resolution is essential to advance the development of next-generation spintronic devices. In this study, we successfully imaged the magnetic domain structure of micropatterned subnanometer thin films by combining high-resolution and high-sensitivity imaging using soft x-ray ptychography and x-ray magnetic circular dichroism. Furthermore, by measuring hyperspectral images of magnetic thin-film patterns and applying the sum rule to absorption spectra at each pixel of the image, quantitative two-dimensional measurements of spin and orbital magnetic moments within magnetic domains were achieved. Such measurements provide a powerful evaluation method for the design of devices based on physical phenomena such as spin and orbital currents.

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Introduction. With the recent rapid progress in research on the miniaturization and application of spintronic devices [1–6], understanding the relationship between magnetism and structure with high spatial resolution has become increasingly important. To this end, x-rays are a powerful probe due to their shorter wavelength and corresponding increased spatial resolution in addition to their longer penetration depth compared to visible light. Furthermore, by tuning the photon energy and helicity, it is possible to probe specific elements and their magnetism simultaneously. These unique

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features enable the imaging of magnetic domains [7,8], domain walls [9], and skyrmionic textures [10–14] in samples ranging from buried ultrathin films to bulk specimens. By performing imaging measurements across the absorption edge of a specific element, it is possible to quantitatively detect the spin and orbital magnetic moments contributing to magnetism [15–17]. Imaging of spin and orbital magnetic moments has previously been demonstrated using x-ray photoelectron emission microscopy (PEEM) [18] and (scanning) transmission x-ray microscopy (TXM and STXM) [19,20].

Thanks to significant advancements in computational capabilities and resources, x-ray diffraction-based techniques have gained considerable popularity over the past decade [8,21,22]. One such technique is ptychography. By collecting numerous coherent diffraction patterns while varying the illumination position with a finite overlap, the real-space complex refractive index (both real and imaginary parts) can be reconstructed [23,24]. Ptychography offers several advantages over other imaging techniques: It can probe a large field of view with high spatial resolution and low acquisition time, its phase-retrieval calculations are relatively straightforward, and it is not affected by the imperfections in optical elements. The combination of x-ray magnetic dichroism and ptychography allows for rapid element-specific imaging of magnetic domains. For example, magnetic domains in 50-nm-thick amorphous SmCo_5 films [25], 1- μm -thick Fe/Gd multilayers [26], and Fe particles in magnetic bacteria and mammalian cells [27] have been imaged using soft or hard x-ray ptychography. Notably, magnetic imaging has recently advanced to three dimensions by combining tomography, enabling the reconstruction of three-dimensional magnetic vector fields [28].

However, previous studies have primarily focused on relatively thick thin films (on the order of 100 nm or thicker) or bulk specimens, and the sensitivity of ptychography for imaging ultrathin films has yet to be thoroughly evaluated. Moreover, to the best of our knowledge, ptychographic mapping of spin and orbital magnetic moments, fully leveraging the x-ray magnetic circular dichroism (XMCD) technique, remains unreported. In this study, we demonstrate the quantitative mapping of spin and orbital magnetic moments in an ultrathin subnanometer Co film using soft x-ray ptychography.

Method. Figure 1 shows an overview of the experimental setup. The ptychography measurements were conducted at beamline BL07LSU of SPring-8, which provides photon energies ranging from 250 to 2000 eV. The polarization of the incident x-rays can be fully controlled, from linear to right- or left-handed circular polarization, using a combination of eight undulators and phase shifters. The degree of circular polarization was approximately 0.93.

For the ptychography measurements, we used our custom-made apparatus called CARROT (Coherent Achromatic Rotational Reflective Optics for pTychography) [29,30], which typically achieves a resolution of about 50 nm (half-period). The x-ray beam is focused onto the sample using a total-reflection Wolter mirror. The achromatic nature of the optics allows seamless measurements across various absorption edges in the soft x-ray region [31], facilitating the mapping of spin and orbital magnetic moments. Since the total-reflection Wolter mirror does not require an order-sorting aperture, which is used in conventional Fresnel zone plate optics, we achieve a consistent large working distance of 20 mm. The detector, with a pixel size of 13.5 μm , was placed at 342 mm away from the sample. In the present study, to minimize radiation damage, the x-ray beam was defocused to a spot size of approximately 10 μm on the sample. The sample was aligned such that the x-ray beam impinged perpendicularly onto the sample surface.

The sample was scanned with a square-grid pattern at about 50 nm intervals, ensuring an overlap of approximately 95% of the illumination beam. 225 coherent diffraction patterns were recorded at each photon energy. For image reconstruction, we employed the extended ptychographic iterative engine algorithm [23].

The Co thin film was grown by electron-beam deposition on a 200-nm-thick silicon nitride membrane supported by a 0.5 mm thick silicon substrate. The thin-film stack consisted of SiN membrane (200 nm)/Ta (3 nm)/Pt (5 nm)/Co (0.6 nm)/Pt (3 nm), as shown in Fig. 2(a). The deposition rates were 0.15, 0.2, and 0.05 $\text{\AA}/\text{s}$ for the Ta, Pt, and Co layers, respectively, under a base pressure of approximately $\sim 2 \times 10^{-8}$ Pa. The Pt underlayer and overlayer impart strong

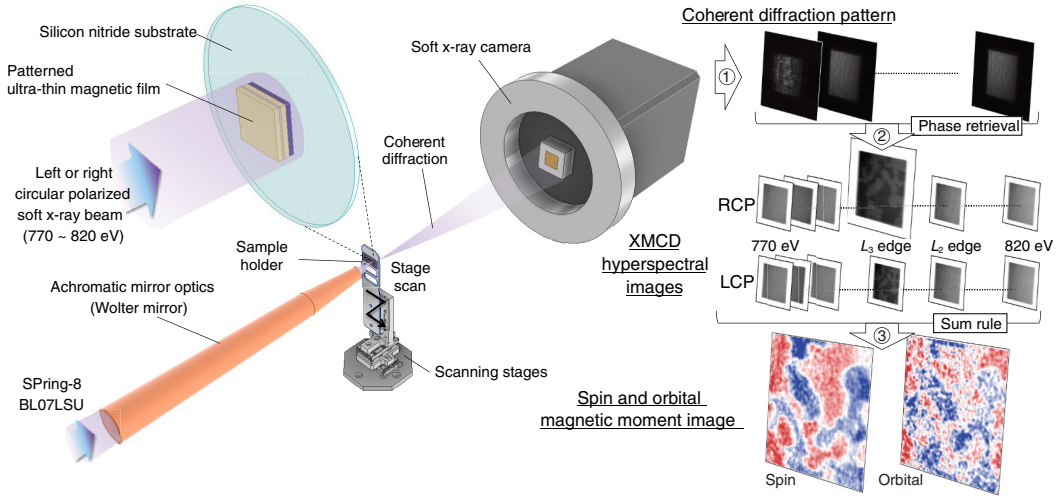


FIG. 1. Schematic diagram of the experimental concept and setup of soft x-ray dichroic spectroptychography. The soft x-ray beam is focused onto the sample using a total-reflection Wolter mirror. Forward-scattered x-rays are detected by a two-dimensional sCMOS camera positioned downstream. Collecting diffraction patterns at different sample positions by scanning piezo stages allows for ptychographic reconstruction of the real-space complex refractive index. By performing ptychography measurements while varying the photon energy across the absorption edge of a certain element, it is possible to map the spin and orbital magnetic moments using the XMCD sum rules.

perpendicular magnetic anisotropy to the Co layer [32,33], while the Pt overlayer also serves as a capping layer to prevent oxidation. The Ta adhesion layer was used to enhance the bonding of the Pt/Co/Pt multilayer to the membrane. The Co film was further patterned into $10\ \mu\text{m}$ squares using a lift-off process with high-speed electron beam lithography at the Takeda Super Clean Room facility of The University of Tokyo. This patterning improves the accuracy of phase recovery calculations compared to a uniform film lacking structural features.

Figure 2(b) shows the magnetization curve obtained using the polar magneto-optical Kerr effect (MOKE) with visible light (660 nm). The magnetization curve exhibits a squarelike hysteresis with nearly 100% remanence, indicating strong perpendicular magnetic anisotropy. Note that the ptychography measurements were performed on the as-grown samples without the application of

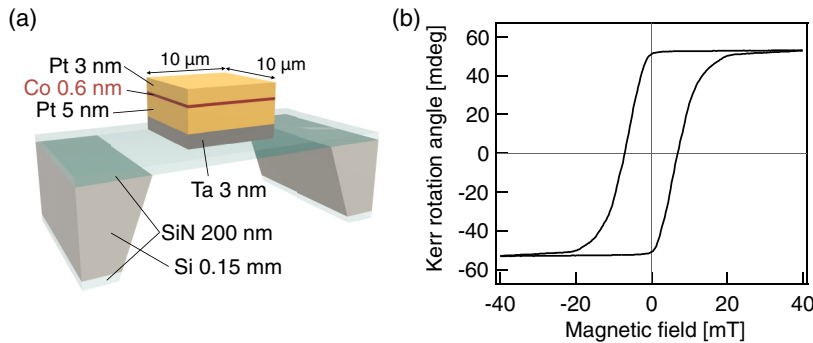


FIG. 2. (a) Schematic of the sample structure. (b) Out-of-plane magnetic hysteresis loops measured using the polar magneto-optical Kerr effect.

an external magnetic field, allowing for the visualization of perpendicular magnetic domains with domain walls, rather than a fully saturated single-domain state.

Results and discussion. Figures 3(a) and 3(b) show the x-ray absorption (optical density) maps measured with right- and left-circularly polarized x-rays at the Co L_3 absorption edge (778.8 eV), respectively. The gray 10 μm square in the center corresponds to the Co/Pt multilayer, while the black regions surrounding it represent the SiN membrane without the multilayer. Here, the transmittance map at each photon energy was normalized such that the transmission through the membrane becomes unity. The normalized transmittance map was then converted to an absorption (optical density) map by applying the natural logarithm. Clear contrast is observed in each polarization, with the contrast inverted depending on the polarization, which indicates the presence of perpendicularly magnetized domains.

For a more quantitative analysis, we subtracted a standard double-step background [34] from the x-ray absorption spectrum taken with each circular polarization at each pixel. We then calculated the sum and difference of the images with different circular polarizations, which correspond to x-ray absorption (XAS) and XMCD, as shown in Figs. 3(c) and 3(d), respectively. The XAS and XMCD images are normalized such that the average XAS intensity within the Co/Pt square is unity. The XAS image displays a uniform pattern without significant contrast, while the XMCD image clearly resolves the magnetic domains, with opposite XMCD signals indicating different magnetization orientations.

To map the spin and orbital magnetic moments, we conducted hyperspectral soft x-ray ptychography measurements across approximately 150 photon energies spanning the Co $L_{2,3}$ absorption edges (770–820 eV). The measurements, which included ~ 150 energy scans, took about 2 h for one circular polarization. Since the same measurements were performed with both right- and left-circular polarizations, the total measurement time was approximately 4 h.

Figures 3(e) and 3(f) present XAS and XMCD spectra extracted from specific pixels (50 $\times$ 50 nm^2) marked by blue and red stars in Figs. 3(c) and 3(d). The obtained XAS and XMCD spectra resemble those of hcp Co [17] or Co/Pt multilayers [35,36] and show no indication of oxidation.

While XAS spectra are nearly uniform across different positions, XMCD spectra exhibit a sign change while maintaining the same shape, confirming that the XMCD contrast in Fig. 3(d) corresponds to perpendicularly magnetized domains with opposite magnetization directions. It is worth noting that the signal fluctuation across energy scans is more pronounced than the pixel-to-pixel fluctuation. This energy-dependent fluctuation can be reduced with more precise determination of the incoming x-ray intensity and by conducting multiple energy scans. However, such fluctuations are not critical for the imaging of spin and orbital magnetic moments, as these are derived from integrals of the XAS and XMCD spectra.

Based on the hyperspectral absorption images obtained using right- and left-circularly polarized x-rays, we deduced spin and orbital magnetic moment images using the XMCD sum rules [15–17], as shown in Figs. 4(a) and 4(b), respectively. To avoid artifacts near the multilayer edges, we focused on the central region, indicated by the dashed square in Fig. 3(d), for magnetic moment mapping. The number of $3d$ holes in Co was assumed to be 7.51 [17], and the degree of circular polarization (93%) was taken into account. The real-space mapping of the spin and orbital magnetic moments revealed a domain structure similar to that observed in the XMCD intensity maps, which is more clearly seen in the images processed with an 11 $\times$ 11 pixel median filter [Figs. 4(c) and 4(d)].

To evaluate the validity of these mappings, we calculated histograms of the spin and orbital magnetic moment values for each pixel, as shown in Figs. 4(e) and 4(f). The spin magnetic moment peaks at $-1.1\mu_{\text{B}}/\text{Co}$ and $+1.3\mu_{\text{B}}/\text{Co}$, while the orbital moment shows peaks at $-0.15\mu_{\text{B}}/\text{Co}$ and $+0.05\mu_{\text{B}}/\text{Co}$.

Previous XMCD studies reported that the spin and orbital magnetic moments of Co in Pt/Co/ AlO_x multilayers range from $0.8\mu_{\text{B}}/\text{Co}$ to $1.3\mu_{\text{B}}/\text{Co}$ and $0.13\mu_{\text{B}}/\text{Co}$ to $0.18\mu_{\text{B}}/\text{Co}$, respectively, when the thickness of the Co layer varies from 0.6 to 1 nm [35]. Another study reported spin and orbital magnetic moments of $1.6\mu_{\text{B}}/\text{Co}$ and $0.2\mu_{\text{B}}/\text{Co}$, respectively [36]. The average spin and orbital magnetic moment values of $1.2\mu_{\text{B}}$ and $0.1\mu_{\text{B}}$ obtained in this study agree well with

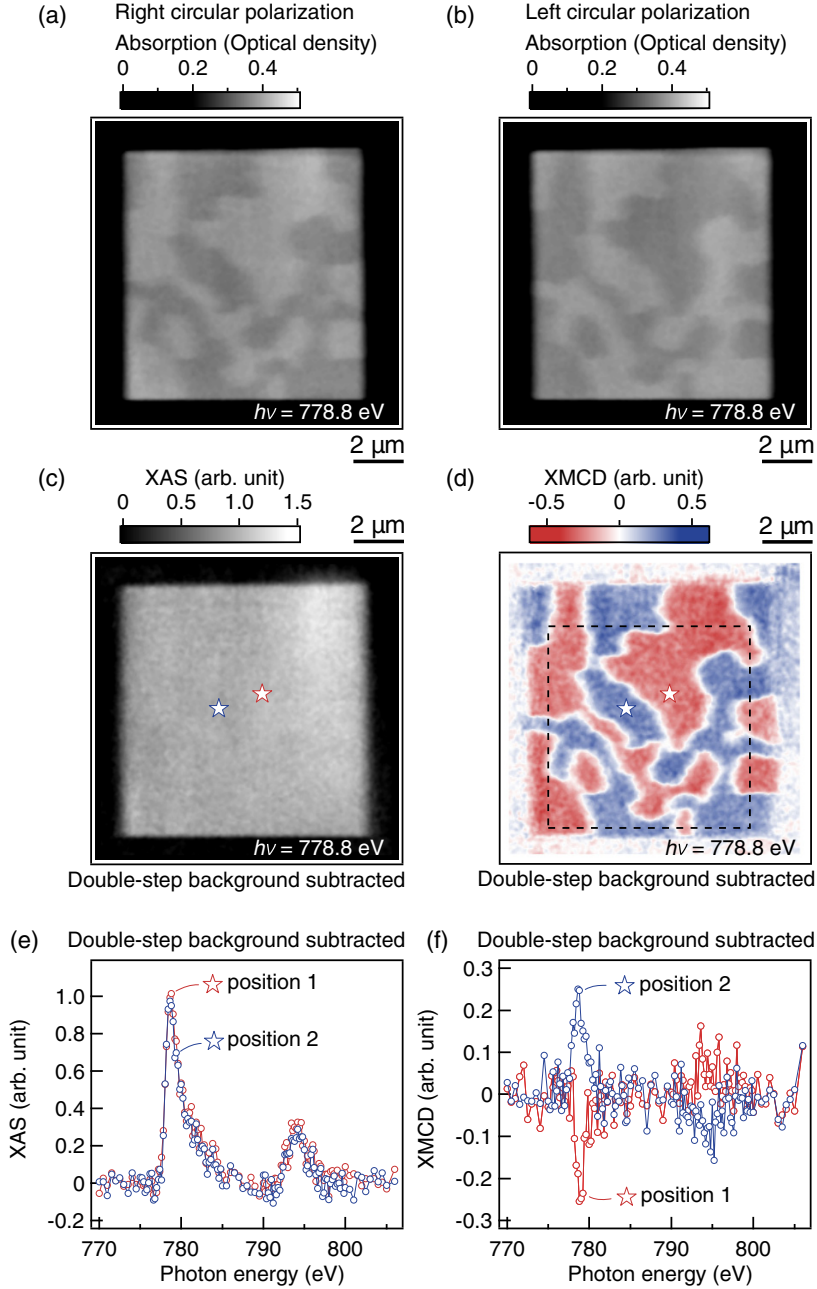


FIG. 3. Reconstructed absorption images obtained with (a) right- and (b) left-circularly polarized x-rays at the Co L_3 absorption edge (778.8 eV). (c) XAS and (d) XMCD images, derived as the sum and difference of absorption images taken with right- and left-circularly polarized x-rays. The XAS and XMCD intensities are normalized such that the average XAS intensity equals unity. The dashed square indicates the region used for the magnetic moment mapping in Fig. 4. (e), (f) Typical XAS and XMCD spectra extracted from specific pixels (1 pixel = 50×50 nm²), marked by red and blue stars in panels (c) and (d).

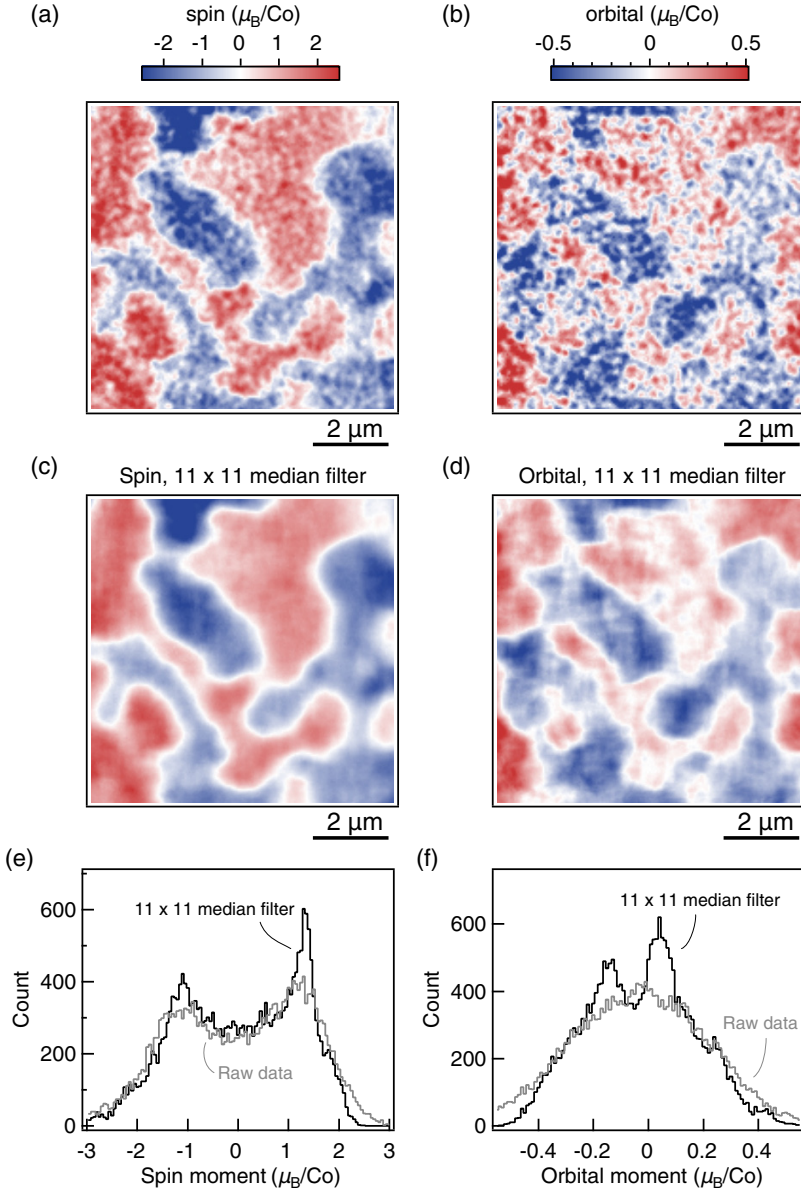


FIG. 4. (a) Spin and (b) orbital magnetic moment images. (c) Spin and (d) orbital magnetic moment images after processing with a 11×11 pixel median filter. Histogram of (e) spin and (f) orbital magnetic moments extracted from the unprocessed and processed images.

those reported in previous work, demonstrating that ptychography can accurately measure spin and orbital magnetic moments.

A deviation between the positive and negative values for both the spin and orbital magnetic moments was also observed. This could be due to a poor signal-to-noise ratio during the energy scan, which made double-step background subtraction more difficult. As noted earlier, such issues would be minimized by using thicker samples, performing energy scans multiple times over a longer period, or using the Kramers-Kronig relation for hyperspectral ptychographic reconstruction [37].

This study demonstrates that magnetic domain imaging, including separate spin and orbital magnetic moment mapping, in ultrathin ferromagnetic films (1 nm or less) can be quantitatively achieved with high spatial resolution (50 nm in the present study) using ptychography. The method presented here uniquely combines sensitivity, resolution, a large field of view, a longer working distance (20 mm), and a significant probing depth compared to previously employed techniques such as STXM and XMCD-PEEM. For instance, scanning-based techniques like STXM typically use a Fresnel zone plate to focus soft x-ray beams down to a few tens of nanometers. However, this approach necessitates a short working distance, making it challenging to measure operating devices with electrical wiring or to rotate samples for tomography to reconstruct three-dimensional magnetic moment distributions. XMCD-PEEM offers the distinct advantage of potentially achieving higher resolution, as it is not limited by the focal size or wavelength of x-rays. Nonetheless, it is restricted to probing regions within just a few nanometers from the surface, due to the limited escape depth of photoelectrons, and cannot easily accommodate the application of magnetic fields during measurements, as such fields distort electron trajectories.

X-ray ptychography addresses these challenges and holds significant promise for advancing research in magnetism and spintronics. For example, operando ptychography measurements could uncover magnetic structures within devices during operation, providing insights into the intricate interplay between spin and orbital currents and their accumulation at device edges. However, ptychography also has limitations: Thin-film samples must be grown on membrane substrates to allow x-rays to transmit through them. This requirement complicates the study of epitaxial thin films, which typically require growth on specific single-crystalline substrates. This issue might be resolved by developing reflective ptychography for the x-ray regime. The spatial resolution of 50 nm in this study—currently limited by environmental vibrations and the low-divergence illumination of the present setup [29]—is modest compared to the state of the art. However, implementing high-numerical aperture (NA) optics [38] while maintaining achromatic advantages of our system will overcome this, potentially reaching sub-10-nm resolution achieved in previous studies [25,39,40].

Conclusion. In this study, we have conducted soft x-ray dichroic spectroptychography measurements and successfully imaged the perpendicularly magnetized domains in an ultrathin subnanometer Co thin film. A quantitative mapping of spin and orbital magnetic moments was successfully obtained through the sum rule analysis performed on the hyperspectral images across Co $L_{2,3}$ absorption edges taken with right- and left-circularly polarized x-rays. Our results show that the spin and orbital magnetic moments are in good agreement with previous standard XMCD studies, confirming the validity of the technique.

The combination of high sensitivity, spatial resolution, large field of view, and deep probing capability makes ptychography a powerful tool for studying magnetic materials, especially in thin-film and multilayer systems. Furthermore, the ability to perform operando measurements opens up exciting prospects for real-time investigations of magnetic structures within actual devices under operational conditions. This study provides an avenue for advancing spintronics and magnetism research, offering detailed insights into the magnetic properties and behaviors that are critical for the development of next-generation devices.

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Data availability. The data that support the findings of this article are not publicly available upon publication because it is not technically feasible and/or the cost of preparing, depositing, and hosting the data would be prohibitive within the terms of this research project. The data are available from the authors upon reasonable request.

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