

Advancements in x-ray microscopy for the non-destructive metallurgical characterization of mesoscale components

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ABSTRACT

KEYWORDS

X-Ray Micro-Computed Tomography, Synchrotron, Coherent Imaging, Ptychography, Holography.

The metallurgical characteristics of mesoscopic samples are critically important for ensuring the safety, reliability, and performance of everything from jet engines to medical implants, and for designing new alternatives. To monitor real-time failure of components, a non-destructive analytical technique that provides high-resolution, three-dimensional visualization of a sample's internal microstructure comprising grains, pores, and micro-cracks is crucial. Conventional optical-based characterization techniques, while valuable for surface and compositional analysis, cannot visualize the true internal structure in three dimensions. Here, the use of X-ray microscopy (XRM) as a non-destructive method to analyze and visualize internal architectures of mesoscopic samples at nanoscale resolution is explored. XRM simultaneously helps in mapping a material's physical structure, crystal orientation, and chemical composition —providing the complete picture needed to create the next generation of advanced materials. The recent advances in detector and focusing technologies derived from synchrotron research have enhanced laboratory-based XRM systems, achieving image resolutions that surpass traditional micro-CT and expanding their applicability for advanced material development and diagnostics.

1. Introduction

To ensure the safety, reliability, and performance of everything from jet engines to medical implants, as well as for designing new components, it is critically important to study the metallurgical characteristics of mesoscopic samples. For doing so, real-time monitoring of a component being stressed or heated is crucial, making the X-ray microscopy technique an indispensable tool in non-destructive testing. X-ray microscopy (XRM) scans can reveal the true 3D internal structure, which dictates strength, toughness, and failure, and can accurately map the complex 3D shape of internal grains, pores, and micro-cracks.

For decades, a significant resolution gap has existed in materials characterization. Optical microscopy, which is ideal for in situ studies and offers excellent contrast for various properties, is fundamentally limited by the diffraction of light to resolutions of several hundred nanometers.

Conversely, Transmission Electron Microscopy (TEM) provides atomic-scale resolution but is limited to extremely thin samples, typically under 100 nm, and requires high-vacuum environments.

XRM decisively fills the void. 3D XRM, also known as X-ray micro-computed tomography (micro-CT), is a non-destructive analytical technique that provides high-resolution, three-dimensional visualization of a sample's internal microstructure. XRM can achieve spatial resolutions from the submicron down to the nanoscale. By using wavelengths shorter than UV light but with far greater penetration power than electrons, XRM can investigate the internal structure of mesoscale samples, defined as a system that exists on an intermediate scale, between microscopic and macroscopic, that is “too thick for electron microscopy” (Falcone et al., 2011).

While electron microscopes offer extremely high resolution, they are generally limited to visualizing a sample's surface; thus, XRM was developed to overcome these limitations of other microscopy techniques. To see internal features, these traditional methods require

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the sample to be physically and destructively sectioned, which can damage the specimen, alter its internal structures, and make 3D reconstruction from 2D slices inaccurate. 3D XRM uses the deep penetration power of X-rays to image the entire volume of a sample without requiring any physical slicing. The non-destructive quality of XRM is its primary advantage, as it allows for the internal structure to be quantified in its native state and preserves the sample for future studies.

The spectacular revival and rapid development of XRM are inextricably linked to the pioneering research at synchrotron facilities, which use brilliant X-ray beams from particle accelerators for unmatched resolution and contrast. While synchrotron science continues to probe materials at ever-finer scales, the detector and focusing technologies developed there are now enhancing commercially available, lab-based XRM systems. These lab-based systems are key to advanced 3D imaging and tomography, offering resolutions far beyond traditional micro-CT. They provide non-destructive 3D imaging across a wide range of length scales, from millimeters down to nanometers. A unique strength of these systems is their ability to perform in situ studies of how a sample's microstructure changes over time, known as four-dimensional (4D) imaging. Consequently, XRM is rapidly being adopted for a growing number of research and industrial applications, including the study of batteries, advanced alloys, biological samples, and geological materials.

The methodologies and applications of modern X-ray microscopy, an advanced characterization tool, is reviewed based on the findings of leading research in the field. The primary instrumentation approaches, contrasting traditional lens-based optics with the new directions of lensless, coherent imaging, are discussed initially. The specific hard X-ray techniques that are making a significant impact in materials science are explored, along with a detailed case study on diagnosing and detecting failure in faulty devices.

2. Methodologies: The Optics of X-ray Vision

The primary challenge encountered by X-ray microscopy has always been the fact that X-rays have a refractive index very close to unity for all materials. Thus making it impractical to use conventional, refraction-based lenses (like those in an optical microscope). The field has therefore

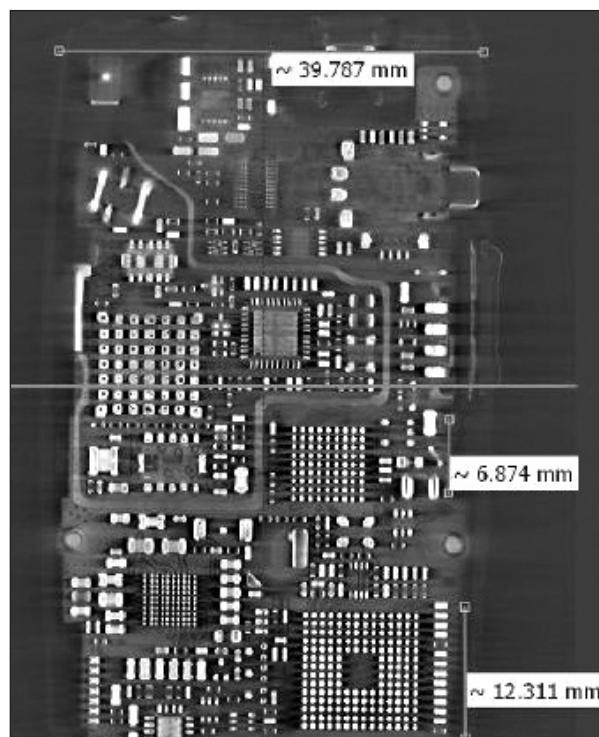


Fig. 1. Virtual cross-sectional view of a printed circuit board in a mobile handset.

evolved along two major paths: one using specialized X-ray optics and one that cleverly bypasses the need for lenses altogether.

2.1 Lens-based microscopy: full - field and scanning

The most established forms of XRM are Scanning Transmission X-ray Microscopy (STXRM) and Full-Field Transmission X-ray Microscopy (TXRM). These instruments typically rely on Fresnel zone plates—circular diffraction gratings that focus X-rays—to achieve high resolution in the 25–50 nm range (Falcone et al., 2011), with some soft X-ray microscopes pushing this to better than 15 nm (Chao et al., 2005).

These microscopes consist of an X-ray source (usually a tube) and a detector. The sample is placed between them and illuminated. Although X-rays pass through the object, they are absorbed to different degrees by different materials based on local density, and the absorption contrast produces the image on the detector. The detector is typically a scintillator, which converts X-rays in-to visible light, combined with a CCD to detect that light. By using the latest scintillators tuned for sensitivity, glass lenses for optical magnification, and modern CCDs with small pixels, the setup can achieve

imaging resolutions of 500 nm and voxel sizes as small as 40 nm.

Even higher resolutions, defined as the smallest features resolvable in the image, can be achieved by using advanced X-ray optics. X-rays from the source first pass through a capillary condenser to evenly illuminate the sample. After passing through the object, the transmitted X-rays are then focused by a Fresnel zone plate, which acts as an objective lens to produce a magnified image. Optionally, the X-rays can also pass through a Zernike phase ring to create an edge-enhancing phase contrast effect before hitting the detector. With these unique X-ray optical elements, the setup can achieve resolutions as low as 50 nm.

However, zone plates are not the only option. A significant advancement has been the development of High-Resolution Transmission X-ray Microscopy (HRTXRM), a technique based on parabolic compound refractive lenses (Bosak et al., 2010). The powerful and immediate benefit of the lens-based approach is the ability to retrieve both the high-resolution Fraunhofer diffraction pattern (reciprocal space information) and real-space images simultaneously, all within the same experimental setup. It allows researchers to see the structure and understand its underlying crystallographic properties, such as defects and stacking faults. The lens-based method has achieved resolutions of 100 nm and has been demonstrated using both soft (10 - 20 keV) and high-energy (up to 50 keV) X-rays, proving its versatility.

2.2 New directions: lensless and coherent imaging

While optic-based methods are mature, a new frontier in XRM aims to bypass the physical limitations and aberrations of lenses. The lensless techniques leverage the high coherence of modern synchrotron sources. Figure 1 demonstrates the practical utility of this approach through a virtual cross-sectional view of a multi-layered printed circuit board (PCB) within a mobile handset. This non-destructive visualization allows for the precise inspection of internal solder bumps, via-alignments, and hidden metallurgical defects within the dense mesoscale assembly without requiring physical sectioning.

Coherent Diffractive Imaging (CDI) generate the diffraction pattern resulting when a coherent X-ray beam illuminates a sample, which is

recorded by a detector. A computer then uses an iterative algorithm to reconstruct the phase of the scattered wave, ultimately generating an image of the sample (Schroer et al., 2008). In theory, CDI's resolution is limited only by the wavelength of the X-ray and the quality of the diffraction data, offering a pathway to near-atomic resolution (Ai et al., 2025; Liu et al., (2025). However, the non-crystalline specimens (like biological cells) produce much weaker and more diffuse scattering patterns, and making reconstruction difficult continues to be its primary challenge. Ptychography is a powerful and rapidly developing scanning variant of CDI. Instead of illuminating the whole sample at once, a focused beam is scanned across the sample in a series of overlapping positions (Adams & Barbante, 2015). By combining the many diffraction patterns from these overlapping spots, ptychography can robustly reconstruct an image with high resolution (sub-10 nm) and is less susceptible to the issues of traditional CDI (Falcone et al., 2011; Hitchcock et al., 2024). Whereas the holography technique reconstructs an image by capturing the interference pattern between X-rays scattered by the sample and a known reference wave (McNulty, 1994). The resulting image provides rich detail, including both absorption and phase contrast, making it suitable for studying a wide range of objects like biological specimens, nanomaterials, crystal structures, and microelectronic defects (Shi et al., 2021; Tegze & Faigel, 1996).

These lensless methods yield significant advantages by using powerful algorithms to reconstruct an image from scattered diffraction patterns. The computational method not only achieves higher resolution but also provides quantitative phase and absorption data, all while offering the potential for a larger field of view. They are central to the future of XRM, promising to push the boundaries of resolution while minimizing the complex fabrication requirements of X-ray optics.

3. Advanced Characterization Capabilities in Materials Science

The true power of modern X-ray microscopy, particularly using hard X-rays (multi-keV), lies in its multi-modal capabilities. A single synchrotron-based experiment is able to provide a collection of complementary information about a sample, far beyond a simple image (Holt et al., 2013).

3.1 3D Tomographic visualization

High-energy X-rays are ideal for non-destructive 3D imaging, as they can penetrate thicker samples. A full 3D internal structure can be computationally reconstructed (Withers et al. (2021) by rotating a sample and collecting a series of 2D projection images. The tomographic capability of XRM is invaluable for understanding complex, heterogeneous systems (see Figure 2), such as porous materials, composites, or microelectronic devices, without the need for destructive physical slicing.

3.2 Spectroscopic elemental and chemical mapping

The distribution and chemical state of elements within a sample Ade et al. (1992) can be identified via spectroscopic elemental and chemical mapping with X-ray microscopy. Instead of detecting emitted X-rays, the elemental mapping method works by precisely tuning the energy of the incident X-ray beam. When the beam's energy matches a specific absorption edge of an element, that element suddenly absorbs X-rays much more strongly. By scanning the beam and mapping the absorption, researchers can see exactly where that element is located. More advanced techniques, like X-ray Absorption Near Edge Structure (XANES) or Scanning Transmission X-ray Spectromicroscopy (STXRM), analyze the fine structure of the absorption edge. The presence of the element and its chemical state, such as its oxidation state (e.g., Fe^{2+} vs. Fe^{3+}) or its local coordination environment, are revealed in the fingerprint, allowing scientists to map chemical reactions, identify phases, and understand functional properties at the nanoscale.

3.3 Microdiffraction-based structural analysis

The wave nature of X-rays is exploited to probe the local crystallographic structure. Researchers have demonstrated that the collection of diffraction patterns from individual grains or regions of interest is possible when the X-ray beam is focused to a nanoscale spot in the HRTXRM technique Bosak et al. (2010) using the hard X-ray (Holt et al., 2013). The microdiffraction can map crystal phase, orientation, lattice strain, and the presence of defects. It is a critical tool for understanding how materials deform, fail, or grow, linking nanoscale structure to macroscale mechanical and electronic properties. The ability to combine the diffraction data with the real-

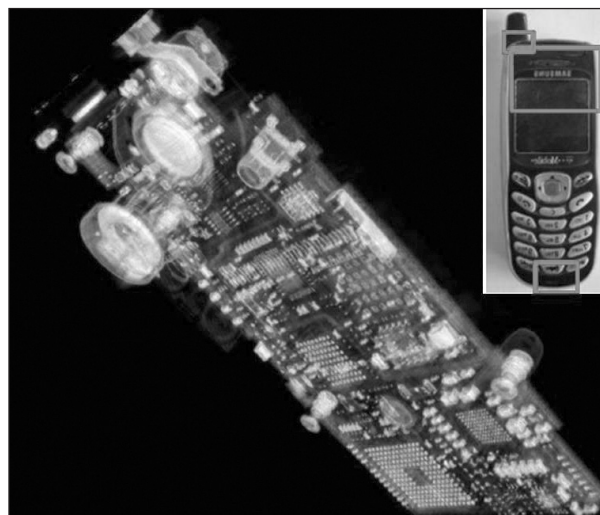


Fig. 2. 3D tomographic view of a mobile handset (shown in the inset).

space imaging in a single setup (as in HRTXRM) is a particularly potent combination.

4. Application Case Study

4.1 Diagnosing a faulty mobile handset charging port with 3D XRM

A mobile handset with a non-functional charging port was analyzed using high-resolution 3D X-ray Microscopy (XRM). The primary imaging challenge was to non-destructively visualize the internal, subsurface morphology of an electronic component. A tomographic scan was performed, rotating the intact sample 360 degrees while acquiring thousands of 2D X-ray projections.

The key diagnostic process involved the multi-planar re-slicing of the 3D volume, generating virtual 2D cross-sections from three orthogonal perspectives. The XRM's absorption contrast mechanism provided excellent material differentiation, clearly distinguishing the high-density metal pins and solder, which allowed for a precise, slice-by-slice inspection of the critical interfaces between the contact pins and the main circuit board.

As illustrated in Figure.3, the virtual slices exposed fine micro-cracks propagating through the internal polymer housing, which correlated with a visible misalignment of the contact pins. From an imaging standpoint, the functional failure was definitively linked to these morphological defects: the XRM data clearly showed a loss of electrical continuity caused by severe voiding and crack-induced pin misalignment, all of

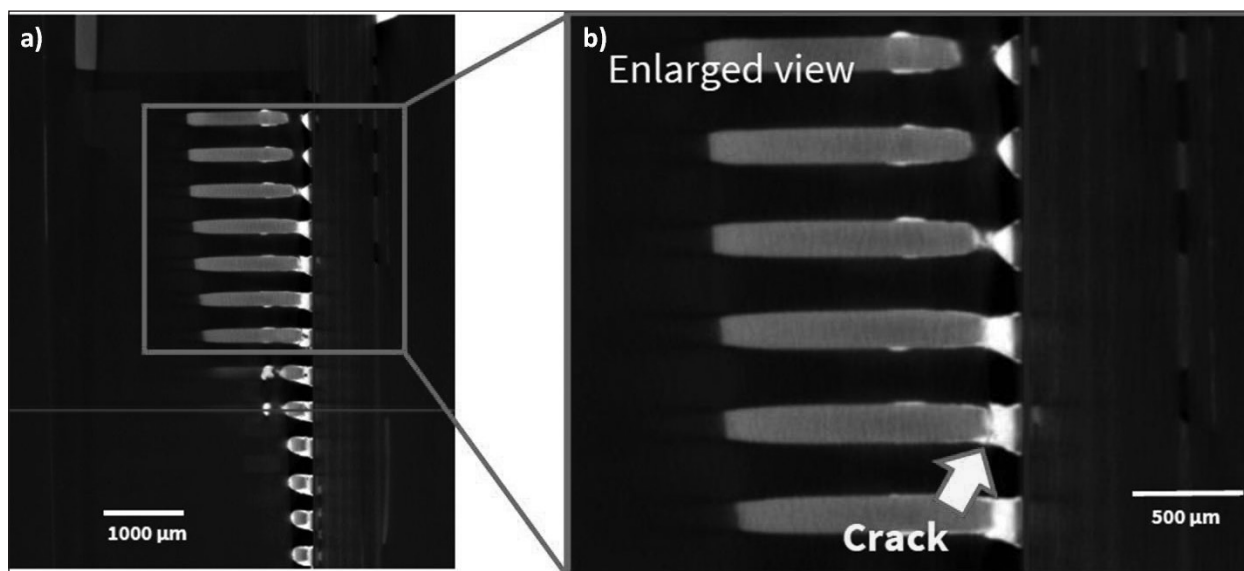


Fig. 3. Voids and cracks were observed in the charging port of a mobile handset by slicing through 3D volume in three orthogonal perspectives.

which were impossible to detect without non-destructive 3D visualization.

4.2 Exposing the cause of lithium-ion battery failure in real-time

While a high-capacity cathode material showed exceptional performance initially, its ability to hold a charge—its capacity—faded rapidly after just 50 charge/discharge cycles (Cognigni et al., 2023). The traditional diagnostic method was to examine the battery after it had already failed, and the destructive process of cutting the battery open often altered or destroyed the very evidence they needed.

To overcome this, the team adopted non-destructive 3D X-ray microscopy (XRM). Initially, a custom battery cell that was transparent to X-rays and could operate inside the microscope was constructed. The battery was charged and discharged inside the XRM, with the microscope taking a full 3D scan every 30 minutes. The 4D dataset allowed them to watch the battery's internal structure evolve over time.

At the macro-scale, the images showed the cathode particles expanding and contracting as expected, but some particles were expanding far more than others, creating immense physical stress. At around cycle 30, the scans captured a microscopic crack forming in one of these oversized particles. The team then used the XRM's high-resolution nano-CT mode to zoom in on the single cracked particle. The high-resolution scan

revealed that the new crack had exposed the raw cathode material to the liquid electrolyte, triggering an unwanted chemical reaction that consumed the electrolyte, formed gas bubbles, and, most critically, electrically isolated the cracked fragment of active material. Hence, the isolated material could no longer hold a charge, directly causing the capacity fade.

By using non-destructive, multiscale XRM, the true root cause was pinpointed: the failure was mechanical (particle cracking), not purely chemical. The initial crack caused the gas and electrolyte breakdown. The manufacturing process needed to be refined to produce smaller, more uniform particles that could better withstand the stress of cycling. XRM allows scientists to move beyond static analysis and directly observe the inner workings of complex systems in real time, accelerating the development of safer, more powerful energy storage.

5. Technical Infrastructure and Capabilities of the Sigray Eclipse XRM at CMTI

To support the metallurgical characterization of mesoscale components in the Indian manufacturing sector, a state-of-the-art Sigray XRM Eclipse 900 system (Figure.4) has been established at the Sensor Technology Development Centre of Central Manufacturing Technology Institute (CMTI), Bengaluru. With a sub-micron spatial resolution of 300 nm this facility provides a non-destructive alternative to traditional cross-sectional analysis, allowing for 3D visualization of

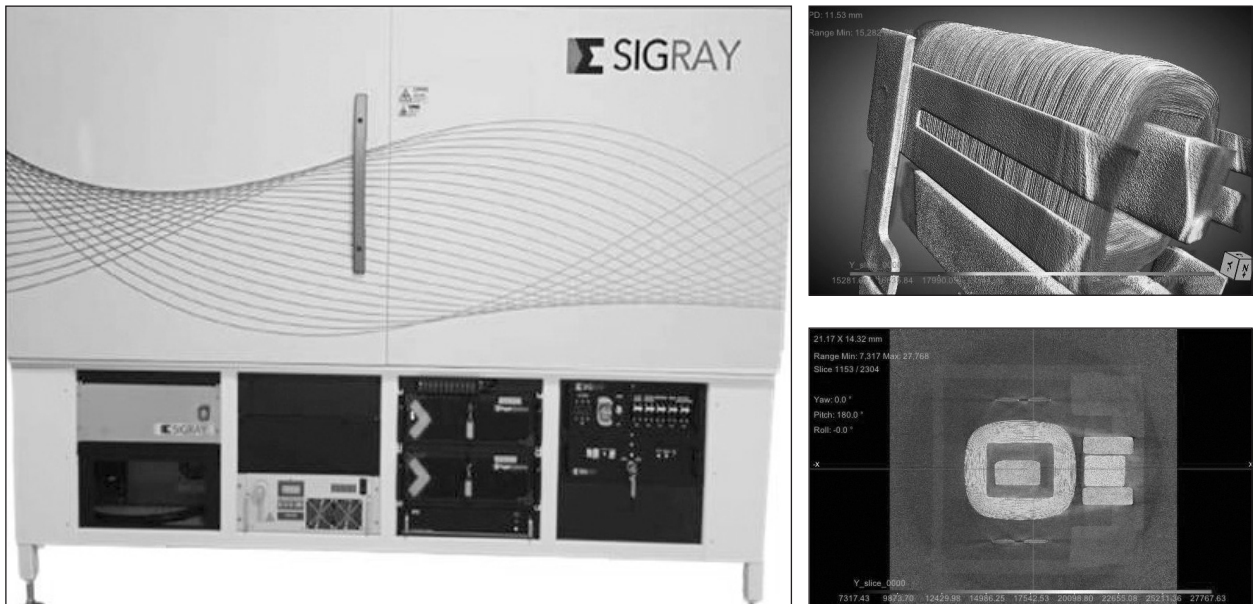


Fig. 4. The Sigray Eclipse 3D XRM (left) installed at CMTI and reconstructed high-resolution image of a relay revealing its internal structure (right).

Table 1

Technical specifications of sigray eclipse 3D XRM installed at CMTI.

Parameter	Specifications
System Model	Sigray Eclipse XRM
Spatial Resolution	Sub-micron (up to 0.3 μm)
Tube voltage range	40 – 160 keV, power 16 W
Modes	Absorption Contrast Imaging, and Phase Contrast Imaging
Applications	Semiconductor and battery failure analysis, study of biological and polymer samples

internal defects, porosity, and grain boundaries in high-density materials. The technical specifications of the equipment are given in Table.1.

The reconstructed image of a relay (Figure.4, right) reveals the intricate internal copper windings and contact geometry without the need for mechanical cross-sectioning. Unlike traditional micro-CT, the Eclipse XRM-900 utilizes a lensless, projection-based architecture. This allows for sub-micron spatial resolution even at larger working distances, which is critical for imaging encapsulated components like relays or in-situ battery cells. The system's high-flux, multi-target X-ray source provides the necessary contrast to distinguish between high-density metallic windings and lower-density polymer housings, facilitating a comprehensive metallurgical assessment.

6. Challenges and Future Prospects

Despite its rapid progress, X-ray microscopy still faces significant challenges that define its future research directions (Jacobsen, 2016). The requirement for higher resolution often requires a higher X-ray dose, which can damage radiation-sensitive materials (like polymers or biological specimens) before they can be adequately measured (Falcone et al., 2011; Howells et al., 2009). Developing new, more dose-efficient imaging methods (like ptychography) and cryogenic sample environments is a critical priority. The hard X-ray techniques described by Holt et al. (2013)—especially 3D tomography and ptychography—generate massive datasets. The development of high-speed detectors and, crucially, high-performance computing and

robust algorithms for data analysis and image reconstruction is essential to manage the data firehose. While source brightness has improved, probing nanoscale volumes still results in a weak signal and remains a fundamental challenge, especially for spectroscopy and diffraction techniques that rely on observing weak interference patterns or fine structures (Holt et al., 2013).

Looking ahead, the evolution of X-ray microscopy is increasingly defined by the integration of the fourth dimension: time. With modern detectors, the short acquisition times of techniques like HRTXRM allow for the method to be extended to time-resolved studies (Bosak et al. (2010) enabling scientists to move beyond static pictures and watch processes like crystal growth, chemical reactions, or material deformation as they happen. Real-time monitoring combined with 3D mapping of both real and reciprocal space represents the next frontier for X-ray microscopy as the ultimate in situ characterization tool.

7. Conclusion

X-ray microscopy is no longer a niche technique but a mature and essential pillar of modern materials characterization. Successfully bridging the gap left by optical and electron methods, XRM provides a unique combination of nanoscale resolution, high penetration power, and rich analytical contrast. Lens-based techniques like HRTXRM unlock the complex structures of mesoscopic materials and offer unprecedented, simultaneous access to both real-space images and reciprocal space diffraction. In parallel, new lensless directions like CDI and ptychography are pushing the boundaries of spatial resolution, driving toward the sub-10 nm regime. When these imaging capabilities are combined with the power of hard X-ray analytical methods—such as 3D tomography, chemical spectroscopy, and microdiffraction—X-ray microscopy becomes a unified tool. A single instrument allowing researchers to visualize the internal 3D structure of a material, map its elemental and chemical composition, and probe its local crystallographic strain and defects represents a paradigm shift in metallurgical characterization. As synchrotrons become brighter and data analysis more powerful, X-ray microscopy is set to keep transforming our understanding of the nano- and mesoscale world.

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