

Metallographic DPSSL-assisted lens-less microscopy of Si-Al systems using a multi-angle planar analyzer based on a polarizing microscope rotating table and a digital image correlator/2D FFT spectrometer

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ABSTRACT

Quality control of metalworking under a microscope plays a crucial role in promoting environmental sustainability and green energy. By ensuring precise, pure and accurate metal fabrication, we can minimize material waste and energy consumption, thus reducing the overall environmental impact of manufacturing processes. This level of quality control also contributes to the longevity and reliability of components used in sustainable energy technologies, such as wind turbines and solar panels. Through meticulous inspection at the microscopic level, we can guarantee the integrity of materials, leading to more efficient and durable green energy solutions. Ultimately, the careful scrutiny of metalworking processes under a microscope supports the advancement of eco-friendly practices and the development of a more sustainable future. We offer a new technology for the quality control of metalworking and many tribological procedures leading to abrasion and wear of the product and environmental pollution with microparticles and nanoparticles of metals, bimetals, alloys, sitalls, etc. This technology is publicly available, and the tools for its implementation can be assembled and installed in any production facilities or at environmental monitoring laboratories and product quality control stations.

Keywords: Environmental tribology, surface qualimetry, sustainable development, lens-less microscopy, laser-assisted microroughness measurements, weathering, correlographic imaging, 2D FFT, 2D multi-angle laser scanning imaging

1. INTRODUCTION

To date, there are known several methods of lens-less, reflection-based dark-field microscopy and combined reflection and transmission microscopy^{1,2}, used to characterize the surface topography and its shadow-based 3D visualization³, including its multi-angle and multi-axis implementation. Lens-less reflection digital holographic microscope with a Fresnel-Bluestein transform⁴ and simpler holographic microscopes⁵ for the same purposes are also well known. The possibilities of spectral implementation of this lens-less approach, according to the data known from literature, extend from the VUV to the far IR^{6,7} and THz range^{8,9}. Lens-less ghost imaging can be implemented not only in the optical mode, but also with true thermal light¹⁰⁻¹². In the wavelength range of penetrating and corpuscular spectral bands, reflective geometry is often used in X-ray microanalysis/X-ray microtomography¹³ and reflection electron microscopy¹⁴. Therefore, lens-less microscopy in a reflection mode can be considered a universal tool for analyzing opaque and partially transparent turbid samples. When using lens-less microscopy as a reference method, the problem of aberrations and insufficient focusing is eliminated, because in conventional devices such effects are determined by the lenses (although focusing methods and algorithms in lens-less microscopy are well developed^{15,16}).

Our technology is within the trend of development of digital holographic microscopy techniques for reflection-based microimaging or microanalysis of industrial surfaces¹⁷ (although there are also another application of reflection holographic microscopy techniques including total internal reflection holographic microscopy techniques for cellular imaging and analysis of cell-substrate contacts in regenerative medicine, implantology, scaffold engineering¹⁸⁻²¹). Considering the possibility of switching between the compact lasers of different spectral ranges (in particular, with the microscope revolver turret for three to five lenses, providing installation of three to five diode lasers or diode pumped solid state lasers, as we have already implemented in one of the latest modifications of the device/installation), it is

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possible to consider this technology as multispectral or multiple-wavelength method for lens-less imaging of materials.

Among the numerous methods for characterizing materials and their damage, wear and tear using reflection holographic microscopy and off-axis reflection holographic microscopy or optical micron structure tomography^{22,23}, multispectral or multiple-wavelength methods are given special attention. There are known methods of multiwavelength digital holographic microscopy and optical setups for submicron topography measurements of reflecting specimens²⁴. However, it is rightly believed that they can be more useful for cell biology and other industries, where the range of microobjects with different absorption or transmission and phase contrast is significantly higher than in bulk materials and even in composites^{25,26} (despite the fact that for many applications of reflecting digital holographic microscopy in cell biology, purely phase contrast is almost sufficient^{27,28}). However, in our opinion, integration of the optical descriptors of the sample provided by phase and amplitude, as well as by various spectra of the sample components or compartments, is not unnecessary for most of the future applications of the considered inspection technologies within the field of high-tech qualimetry. Therefore, we are preparing in advance a publicly available lens-less toolkit for measurements in the reflection mode, which will be simple and sufficient specifically for industrial applications, and not for the unique experiments (such as application of lens-less ultramicroscopic imaging of atomic surface structures in reflection mode²⁹).

In all the above reflection digital holographic microscopy methods, by definition, the phase plays a significant role. It either requires spherical phase compensation³⁰, or is a distinctive feature of the system design (as, for example, in equipment for Phase-Shifting Digital Holographic Microscopy (PSDHM)³¹ or in equipment for plane wave illumination for correct phase analysis and alternative phase unwrapping in transmission and reflection 3D holographic microscopy³²). It is obvious that compensation of spherical aberrations is not required in the case of lens-less microscopy, since it, by definition, is an aberration-free (due to the absence of lenses) method. Therefore, we prefer lens-less microscopy, without taking into account very specific methods operating outside the optical range based on interferometry and total internal reflection, such as X-ray holographic microscopy using total-reflection mirror interferometry³³ or extreme ultraviolet imaging reflectometry³⁴.

2. MATERIALS AND METHODS

We used a homemade installation for lens-less metallographic microscopy based on a rotating table from a POLAM polarizing microscope, and the CCD sensor of the lens-less microscope was located in the central area of the table, where the center of positioning of the condenser and the Keller illuminator beam is usually located. The illumination source was a diode pumped solid state laser with a wavelength of 532 nm, powered by two AAA batteries (the selected laser wavelength is close to that used in the factory metallographic interferometers of the Uverskii system). The laser was fixed in a tripod, which allowed to change the beam angle, while the sample was located on the table of a lens-less microscope on thin submillimeter mica side carriers.

To obtain the image, it was important to position the laser on a tripod so that the laser beam entered the gap between the sensor located on the condenser holder (vertically moved using a microscrew) and the solid-state sample. At the same time, it was possible to vary the quality of the resulting image by varying the distance between the sample and the sensor, since it was different for different lasers (for example, in lens-less ceramography and lens-less plastography we used other lasers with different wavelengths and focusing features). It was also possible to observe interference phenomena and standing waves between the metal sample and the sensor or the glass surface of an anti-alias or ultraviolet filter on it, as in Lippmann photography.

The analog lens-less CCD sensor was placed on a PCB whose size exceeds the size of the sensor by no more than a millimeter. The PCB was powered from a 9 V power source, and the sensor signal was recorded through a BNC connector (or through an RCA-BNC adapter), the cable of which was output to a video mixer and an LCD display that displayed the image in real time, and then to digitization via a video tuner/video capture card. It was also possible to record video from the display using a digital camera mounted on a separate tripod. A photo of the whole installation is shown in Figure 1. Digital images could be analyzed both later and in real time in situ using the QAVIS software developed using the FFTW library and allowing direct work with the screen memory with less than a second interval. This software calculated integral spatial characteristics and integrated frequency characteristics based on 2D FFT recorded in real time.

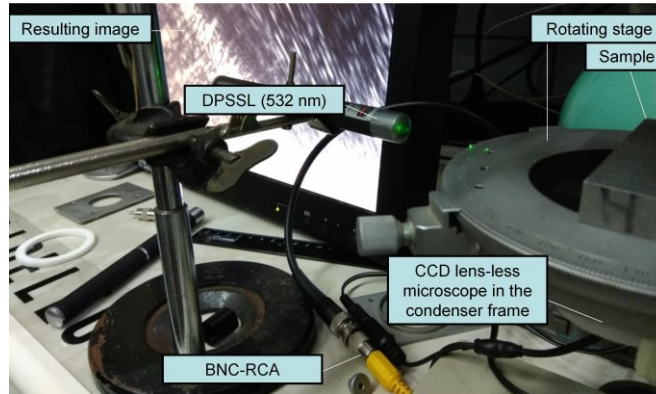


Figure 1. A simplest lens-less installation for the analysis of metals and alloys in reflected light/reflection mode. Such an installation can be reproduced at any plant for its own needs.

The structural ordering of the alloy surface was also studied (including after tribological studies with the establishment of the parameters of the grooves formed on the surface of the material, in which both longitudinal and transverse elements of order coexisted). The above-mentioned software could also operate as an image correlator/correlometer by scanning a constant-size region of interest (ROI) with the selection window in the direction of parallel grooves-the results of a tribotechnical experiment. We used similar methods to establish the parallelism of grooves in microfluidic and mesofluidic chips formed in a similar way on sittals and alloys coated with special compositions. In this case, by rotating the table, it was possible to rotate the ruffled/corrugated sample relative to the axis of the laser beam in such a way that it was possible to study all the reflections and features of both transverse and longitudinal structure/texture with different angular orientations. This was done by analyzing correlograms with the window translation along the corresponding elements of the tribotechnical or micro-/mesofluidic texture.

3. RESULTS

Examples of the measurement results are shown in Figures 2 and 3. Figure 2 shows images with superimposed correlograms of the structures with different levels of the occurrence frequency, revealed when scanning different tribotechnical grooves with a very fine (“low-angle”) rotation of the table. Figures 2a-2d show data for the texture of the alloy surface with pronounced (and perfectly revealed by interferometric methods) transverse components of the groove structure. In order to make sure that manifestations of the structure regularity observed in the correlograms are not an artifact, for example, originated from scattering/laser speckle texture, an analysis of the orderliness of the speckle texture obtained from a non-textured substrate in the far zone of a lens-less microscope is carried out and the results are shown in Figures 2f and 2g. It can be seen that diffuse zones and reflections of the parasitic exposure prevail in this control, corresponding to the laser beam propagation on the surface of either a sensor or an anti-alias filter.

This result can be confirmed by calculating and visualizing the full integral frequency characteristics (with the selection of bars corresponding to the texture features) and integral spatial characteristics. Figures 3a-3d shows integral frequency characteristics (in the form of histograms in Cartesian coordinates) and integral spatial characteristics (a sector graph in polar coordinates-a product of processing the primary 2D FFT spectrum) of the texture observed after tribotechnical processing of the initial material. One can see the pronounced longitudinal and transverse components of the surface texture. At the same time, analysis of the integral spatial characteristics indicates one prevailing narrow maximum of the diffraction pattern. This maximum corresponds to the laser beam, and its small side “shoot” corresponds to the radial components of its “low-angle” reflections. At the same time, if in the sample, due to the different elements of its structure, many regular texture components are observed, as can be seen from the red bars in the histograms of integral frequency characteristics (Figures 3a-3d), then on a “control” laser speckle pattern the software usually identifies only one such bar, due to the beam’s own texture with identical speckle elements (Figures 3e and 3f). These data correlate well with the results of direct reconstruction of 3D surface topography after tribotechnical processing using a lens-less reflective microscope of our design (Figure 4). This indicates the physical significance (reliable correlation with 3D microstructure of the material), reproducibility and cross-method validation of the results, and therefore the possibility of using the developed methodology for measuring the parameters of different solid-state materials, in particular alloys, in industrial and raw materials qualimetry.

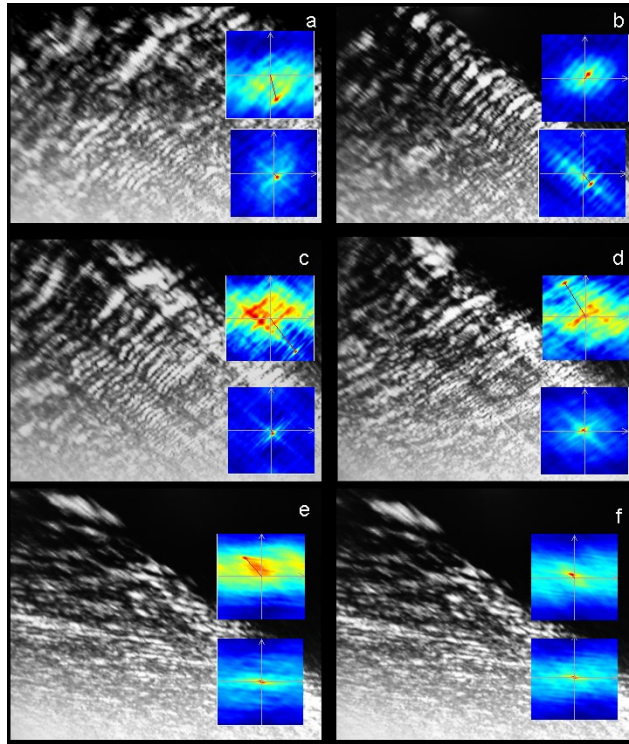


Figure 2. Lens-less imaging results and their correlograms (0.4 sec. delay): a-d: Texture of the material after a tribotechnical experiment; e, f: zero experiment-detection of the “instrument response function” of the installation using a laser speckle.

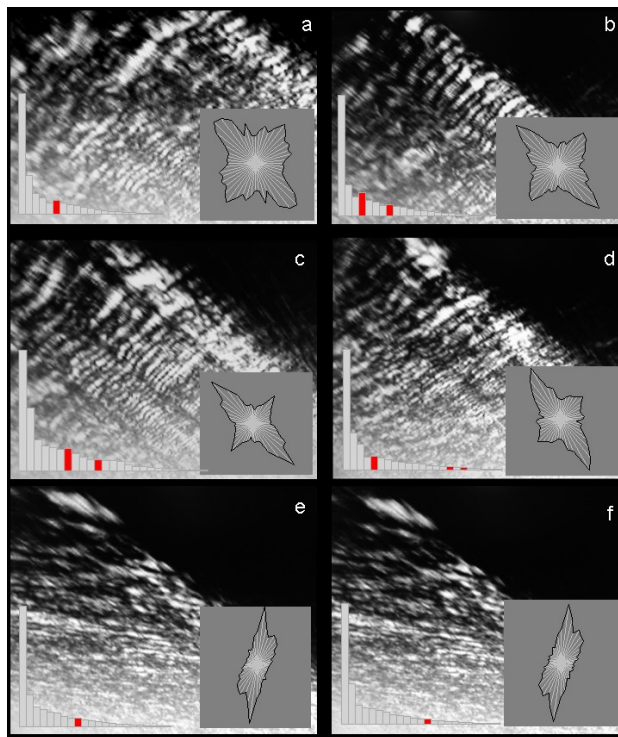


Figure 3. Laser lens-less images and their 2FFT based descriptors (IFC-Integral Frequency Characteristics; ISC-Integral Spatial Characteristics): a-d: Texture of the material after a tribotechnical experiment; e, f: zero experiment-detection of the “instrument response function” of the installation using a laser speckle.

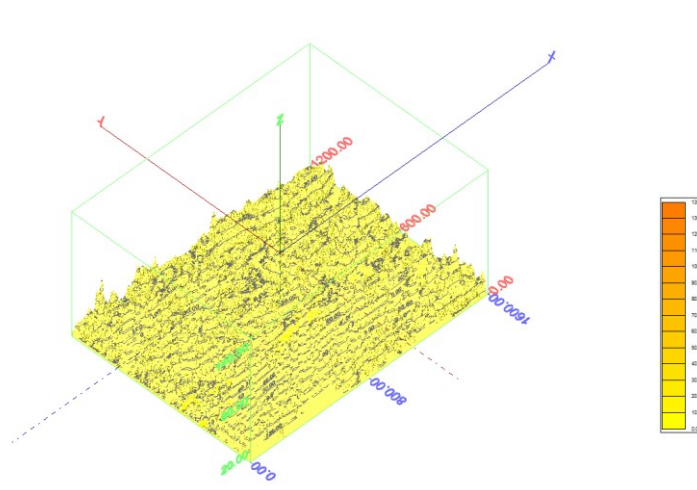


Figure 4. 3D surface topography after tribotechnical processing, reconstructed using a lens-less reflective microscope of our design.

4. DISCUSSION

The problem of creating lens-less optical systems using lasers for the purpose of opaque 3D object imaging using reflected light was actualized back in the 1960s and was associated with the early steps of holography using analogue, that is, photoemulsion media for the image registration³⁵. However, it is still relevant today in the format of lens-less reflection imaging of obliquely illuminated objects with choosing a domain for phase retrieval and ptychography³⁶. The problem of integrated transmission and total internal reflection holographic microscopy in digital implementation³⁷ has been updated and solved recently, as was the problem of simultaneous measurement of refractive index distribution and topography by integrated transmission and reflection holographic microscopy in digital implementation^{38,39}. Therefore, the work on integrating a polarizing microscope and a lens-less microscope with an additional laser input in the rotating table plane (instead of an opaque illuminator) is undoubtedly actual and rational.

REFERENCES

- [1] Imanbekova, M., Perumal, A. S., Kheireddine, S., Nicolau, D. V. and Wachsmann-Hogiu, S., "Lensless, reflection-based dark-field microscopy (RDFM) on a CMOS chip," *Biomedical Optics Express*, 11(9), 4942-4959 (2020).
- [2] Lee, M., Yaglidere, O., Ozcan, A., "Field-portable reflection and transmission microscopy based on lensless holography," *Biomedical Optics Express*, 2(9), 2721-2730 (2011).
- [3] Yuan, C., Zhai, H., Wang, X. and Wu, L., "Lensless digital holography with short-coherence light source for three-dimensional surface contouring of reflecting micro-object," *Optics Communications*, 270(2), 176-179 (2007).
- [4] Shin, S. and Yu, Y., "Lensless reflection digital holographic microscope with a Fresnel-Bluestein transform," *Journal of the Korean Physical Society*, 74, 98-101 (2019).
- [5] Adinda-Ougba, A., Kabir, B., Koukourakis, N., Mitschker, F., Gerhardt, N. C. and Hofmann, M. R., "Compact low-cost lensless digital holographic microscope for topographic measurements of microstructures in reflection geometry," *Proc. SPIE*, 9628, 200-207 (2015).
- [6] Witte, S., Tenner, V. T., Noom, D. W. and Eikema, K. S., "Lensless diffractive imaging with ultra-broadband table-top sources: from infrared to extreme-ultraviolet wavelengths," *Light: Science & Applications*, 3(3), e163 (2014).
- [7] Harada, T., Nakasuji, M., Nagata, Y., Watanabe, T. and Kinoshita, H., "Phase imaging of EUV masks using a lensless EUV microscope," *Proc. SPIE*, 8701, 357-364 (2013).
- [8] Mansourzadeh, S., Damyanov, D., Vogel, T., Wulf, F., Kohlhaas, R. B., Globisch, B., Schultze, T., Hoffmann, M., Balzer, J. C. and Saraceno, C. J., "High-power lensless THz imaging of hidden objects," *IEEE Access*, 9, 6268-6276 (2021).

- [9] Damyanov, D., Friederich, B., Yahyapour, M., Vieweg, N., Deninger, A., Kolpatzeck, K., Liu, X., Czyllwik, A., Schultze, T., Willms, I. and Balzer, J. C., "High resolution lensless terahertz imaging and ranging," *IEEE Access*, 7, 147704-147712 (2019).
- [10] Li, H., Xiong, J. and Zeng, G., "Lensless ghost imaging for moving objects," *Optical Engineering* 50(12), 127005 (2011).
- [11] Bisht, N. S., Sharma, E. K. and Kandpal, H. C., "Experimental observation of lensless ghost imaging by measuring reflected photons," *Optics and Lasers in Engineering*, 48(6), 671-675 (2010).
- [12] Chen, X. H., Liu, Q., Luo, K. H. and Wu, L. A., "Lensless ghost imaging with true thermal light," *Optics Letters*, 34(5), 695-697 (2009).
- [13] Roy, S., Parks, D., Seu, K. A., Su, R., Turner, J. J., Chao, W., Anderson, E. H., Cabrini, S. and Kevan, S. D., "Lensless X-ray imaging in reflection geometry," *Nature Photonics*, 5(4), 243-245 (2011).
- [14] Hammadi, Z. and Morin, R., "Lensless electron reflection microscopy using a coaxial point-source structure," *Ultramicroscopy*, 106(6), 480-485 (2006).
- [15] Perry, J., [Thrust# 3: Simulation and Measurement of Bio-Enabled Lensless Infrared Focusing and Spatially-Controlled Multiwavelength IR Reflection], Georgia Institute of Technology, Atlanta, (2019).
- [16] Li, Z., Zuo, C. and Chen, Q., "An auto-focusing reflection-type lens-less digital holographic microscope," *Proc. SPIE.*, 11761, 378-383 (2021).
- [17] Fernández, M. V., Gonçalves, E., Rivera, J. L. V., Ricardo, J., Abreu, M. E. F. and Vaz, M. A. P., "Development of digital holographic microscopy by reflection for analysis of surface," *Results in Physics*, 11, 182-187 (2018).
- [18] Ash III, W. M. and Kim, M. K., "Cellular imagery with total internal reflection holographic microscopy," *Proc. SPIE*, 7182, 76-83 (2009).
- [19] Mandracchia, B., Gennari, O., Marchesano, V., Paturzo, M. and Ferraro, P., "Label free imaging of cell-substrate contacts by holographic total internal reflection microscopy," *Journal of Biophotonics*, 10(9), 1163-1170 (2017).
- [20] Ash III, W. M., Clark, D., Lo, C. M. and Kim, M. K., "Total internal reflection holographic microscopy for quantitative phase characterization of cellular adhesion," *Proc. SPIE*, 7568, 11-22 (2010).
- [21] Ash III, W. M., Krzewina, L. and Kim, M. K., "Quantitative imaging of cellular adhesion by total internal reflection holographic microscopy," *Applied Optics*, 48(34), H144-H152 (2009).
- [22] Suzuki, S., "Reflection type high-speed holographic microscopy to photograph crack bifurcation," *Experimental Mechanics, Advances in Design, Testing and Analysis*, 1, 583-588 (1998).
- [23] Cheng, G., Jiang, Z., Wang, D., Ding, M. and Cui, H., "Off-axis reflection digital holographic microscopy for micron structure tomography measurement," *Proc. SPIE*, 7848, 555-561 (2010).
- [24] Montfort, F., Charrière, F., Kühn, J., Colomb, T., Cuhe, E., Emery, Y., Marquet, P. and Depeursinge, C., "Multiwavelength digital holographic microscopy for submicron topography of reflecting specimens," *Proc. SPIE* 6443, 91-96 (2007).
- [25] Kühn, J., Montfort, F., Colomb, T., Rappaz, B., Moratal, C., Pavillon, N., Marquet, P. and Depeursinge, C., "Submicrometer tomography of cells by multiple-wavelength digital holographic microscopy in reflection," *Optics Letters*, 34(5), 653-655 (2009).
- [26] Kühn, J., Montfort, F., Colomb, T., Rappaz, B., Moratal, C., Pavillon, N., Marquet, P. and Depeursinge, C., "Red blood cell tomography in reflection by multiple-wavelength digital holographic microscopy," *Proceedings of Focus on Microscopy*, (2009).
- [27] Li, Y., Qian, X., Mei, D. and Dong, K., "Study of experimental data processing method in reflecting digital holographic microscopy reconstructing phase of cells," *Journal of Yunnan University: Natural Sciences Edition*, 29(5), 465-469 (2007).
- [28] Qian, X., "Study on cells by use of reflecting digital holographic microscopy," *Acta Photonica Sinica*, 36(7), 1318 (2007).
- [29] Zhu, C., Harder, R., Diaz, A., Komanicky, V., Barbour, A., Xu, R., Huang, X., Liu, Y., Pierce, M. and You, H., "Lensless imaging of atomic surface structures using ptychography in reflection mode," *Bulletin of the American Physical Society*, 2014, Z24-003 (2014).
- [30] Weijuan, Q., Choo, C. O., Rongwei, L. T., Qiangsheng, X., Zhaomin, W., Zhenzhong, X. and Asundi, A., "Physical spherical phase compensation in reflection digital holographic microscopy," *Optics and Lasers in Engineering*, 50(4), 563-567 (2012).
- [31] Ding, H., Hu, C., Weng, J. and Zhong, J., "Phase-shifting digital holographic microscopy in reflection

- configuration,” *Zhongguo Jiguang (Chinese Journal of Lasers)*, 38(12), 201-206 (2011).
- [32] Kim, M., Hong, S., Shim, S., Soh, K., Shin, S., Son, J. Y., Lee, J. and Kim, J., “Plane wave illumination for correct phase analysis and alternative phase unwrapping in dual-type (transmission and reflection) three-dimensional digital holographic microscopy,” *Optical Engineering*, 49(5), 055801 (2010).
- [33] Suzuki, Y. and Takeuchi, A., “X-ray holographic microscopy using total-reflection mirror interferometer,” *Japanese Journal of Applied Physics* 47(11R), 8595 (2008).
- [34] Porter, C. L., [Complex Extreme Ultraviolet Imaging Reflectometry: Quantitative Lensless Imaging with Short-Wavelength Light in Reflection Geometries], Boulder: University of Colorado at Boulder, Ph.D. Thesis, 2019.
- [35] Johnston, S. F., “Telling tales: George Stroke and the historiography of holography,” *History and Technology*, 20(1), 29-51 (2004).
- [36] Artyukov, I. A., Popov, N. L. and Vinogradov, A. V., “Lensless reflection imaging of obliquely illuminated objects I: Choosing a domain for phase retrieval and ptychography,” *Symmetry*, 13(8), 1439 (2021).
- [37] Zhang, J., Ma, C., Dai, S., Di, J., Li, Y., Xi, T. and Zhao, J., “Transmission and total internal reflection integrated digital holographic microscopy,” *Optics Letters*, 41(16), 3844-3847 (2016).
- [38] Ma, C., Di, J., Zhang, J., Li, Y., Xi, T., Li, E. and Zhao, J., “Simultaneous measurement of refractive index distribution and topography by integrated transmission and reflection digital holographic microscopy,” *Applied Optics*, 55(33), 9435-9439 (2016).
- [39] Frómeta, M., Moreno, G., Ricardo, J., Arias, Y., Muramatsu, M., Gomes, L. F., Palácios, G., Palácios, F., Velázquez, H. and Valin, J. L., “Optimized setup for integral refractive index direct determination applying digital holographic microscopy by reflection and transmission,” *Optics Communications*, 387, 252-256 (2017).