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High-resolution scanning near-field optical nanotomography: A technique for 3D multimodal nanoscale characterization of nano-biophotonic materials

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Abstract

An instrumental system and the corresponding experimental technique for multiparametric nanoscale 3D characterization of a wide range of composite nanomaterials have been developed. This system makes it possible to obtain 3D data on the chemical composition of a material by optical methods in the confocal and near-field optical microspectroscopy modes (fluorescence and Raman) with a lateral resolution up to 50 nm, as well as data on the 3D morphology and spatial distribution of mechanical, electrical, and other characteristics of the material in the scanning probe microscopy mode with a resolution of about 10 nm on the X and Y axes and several angstroms on the Z axis, for a single area of the sample (100 μm × 100 μm × 3 mm). The nanoscale 3D pattern of the distribution of these characteristics is obtained by sequentially examining nanomaterial layers at a step of 20 nm along the Z axis and a total depth of Z-scanning of 3 mm.

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Keywords: Correlative microscopy, scanning probe microscopy, nanotomography, 3D structural analysis, near-field optical microscopy.

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1. Introduction

Explosive development of correlative microscopy in the past decade is accounted for by the necessity of multiparametric nanoscale 3D characterization of both novel hybrid nanomaterials and biological samples (Caplan et al. (2011); Feofanov et al. (1995); Sharonov et al. (1993)). Correlative microscopy is essentially a combination of several microscopic and spectroscopic techniques in examining a single area of a sample. This allows obtaining important mutually complementary data on the morphology, chemical composition, and local optical, mechanical, and electrical characteristics. Examples of the most prominent achievements in this field are optical microscopy combined with electron tomography into correlative light and electron microscopy (CLEM) (Mironov and Beznoussenko (2009); Spiegelhalter et al. (2010)); scanning probe microscopy (SPM) combined with optical microspectroscopy (OM) (Colombelli et al. (2009); Labernadie et al. (2010)); focused ion beam (FIB) etching combined with scanning electron

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microscopy (SEM) to make sequential slices and scan surfaces (the "slice-and-view" technique) (Al-Abboodi et al. (2013); Holzer et al. (2004)); and SPM combined with ultra-microtomography (UMT) into scanning probe nanotomography (SPNT) (Efimov et al. (2007); Efimov et al. (2012)).

Obviously, all data obtained by these methods can be subdivided into morphological (SPP, SEM, and OM) and analytical (optical spectroscopy) data, which substantially differ from each other in spatial resolution: the resolution of the standard OM (≥ 300 nm) is tens of times worse than those of SEM and SPM (several nanometers). Modern near-field techniques, such as scanning near-field optical microscopy (SNOM) and tip-enhanced Raman microspectroscopy (TERS), provide data with a resolution close to 10 nm, but they are unsuitable for 3D analysis (Betzig et al. (1991); Zenhausern et al. (1994); Pettinger et al. (2004); Amenabar et al. (2013)). Thus, development of an approach combining morphological and analytical techniques that have comparable spatial resolutions and allow for 3D analysis is a topical task in correlative microscopy.

2. Experimental part

In this study, we have developed an experimental approach to multiparametric nanoscale 3D characterization of materials with the use of a combination of several near-field microscopic techniques. The study was based on our earlier results of combining SPNT with OM (Mochalov et al. (2013)). The method developed allows optical confocal microspectroscopy and SPM analyses to be performed for a single area of a sample. In particular, we employed this integrated approach to carry out the first study where near-field microscopy was used to analyze the morphology and optical properties of the nanohybrid materials based on cholesteric liquid-crystal matrices doped with fluorescent nanocrystals (NCs) that we engineered earlier (Bobrovsky et al. (2012)).

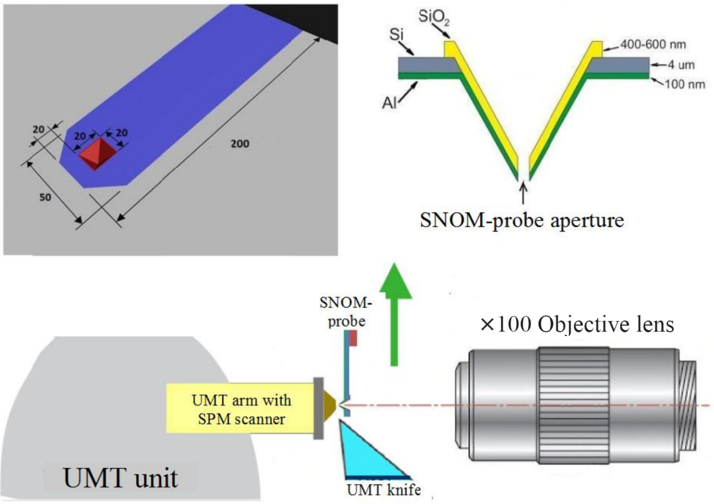

Fig. 1. Schematic presentation of the method combining SPM, UMT, and SNOM; the near-field signal is formed using a hollow cantilever with a nanosized aperture at the tip.

The principle of the method is reconstruction of the 3D structure and optical characteristics of the object examined with a nanometer spatial resolution by means of confocal and near-field OM and SPM, with the sample ultra-microtomed into thin (~20-nm) slices. The method employs the "slice and view" technique, where the 3D morphology and distribution of optical (e.g., fluorescent) characteristics are reconstructed from sequential 2D scans in the SPM and/or OM modes. An important advantage of the "slice and view" technique is that the SPM and/or OM scanning(s) is/are performed over the fresh blockface after each sequential microtome section, which allows the X–Y coordinates of the fields of scanning to be precisely matched.

A high spatial resolution is ensured by the SNOM technique. The near-field area is formed about the tip of a special SNOM probe, which is a hollow cantilever with a nanosized aperture at the tip. In the case shown in Fig. 1, the spatial resolution of fluorescence gathering is about 100 nm. A precision positioning system ensures identical positioning of probes of different types to obtain spectral data (SNOM and TERS) and information on the geometric parameters of the sample at a high resolution.

The procedure of obtaining data for reconstructing spectral and geometric 3D information consists of several steps.

(1) The primary slice made with the UMT knife.