Development of surface plasmon resonance microscope with gratings for nanocomposite dispersion state observation

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Abstract

A surface plasmon resonance (SPR) microscope with a diffraction grating for observing the nanocomposite dispersion state is proposed. SPR microscopes can precisely measure nanocomposite materials because of their high sensitivity to refractive index variations. However, because the incident light is oblique to the sample, image distortion is a problem. Therefore, a diffraction grating is used to improve the quality. We eliminated this image distortion using high order diffracted light that is parallel to the sample.

1. Introduction

Nanocomposite materials comprise two or more phases containing different nano order compositions or structures. They have high tensile strength, heat resistance and coercive force. However, they tend to form aggregates with large specific surface. The aggregates severely affect the performance of these nanocomposite materials. To evaluate the properties of nanocomposite materials, it is necessary to observe them in the dispersed state [1].

The refractive index depends on the nanoparticle aggregates. To measure the refractive index of nanoparticles, a surface plasmon resonance (SPR) microscope was proposed [2]. SPR depends on the dielectric constant and geometry of the sample. For known sample geometry the refractive index can be determined. Furthermore, because SPR depends on light intensity, an optical setup is particularly useful for measurements. Therefore, the SPR microscope has been widely used to measure the refractive index of materials. However, in the current generation of SPR microscopes, the illumination light incident at the resonance angle gives rise to image distortion. To overcome this problem, we used a diffraction grating to vertically diffract the reflected light from the sample. Using a high-order diffracted light for imaging; the imaging device is set parallel to the object plane, thereby eliminating image distortion. In this paper, we reports the refractive index of a nanocomposite material measured using a SPR microscope using the diffraction grating.

2. SPR microscope with a diffraction grating

Figure 1 shows the SPR microscope with a diffraction grating. The SPR generating part comprises a 6.0-mm-thick glass substrate with a 50-nm-thin gold layer deposited on the substrate surface by vacuum evaporation; the sample is placed on this gold layer. The diffraction grating is optically coupled to the side of the glass substrate using immersion oil. The thin gold layer is illuminated through the other side of the substrate at a fixed incidence angle by a laser diode (LD; wavelength = 670 nm). The SPR excitation from p-polarized light depends on the refractive index of the sample. Thus, the intensity of the reflected light depends on the difference in the refractive index. The reflected light from the sample is vertically diffracted by the diffraction grating, which is of the blazed type, with a grating period of 833 nm and a blaze angle of 36.52°. The diffraction grating period is designed to generate second-order diffraction light vertical to the grating. Finally, the sample surface is imaged with a CMOS camera through an objective lens and an imaging lens. Thus, the undistorted image of the nanocomposite dispersion state is obtained using the contrast of the high-order reflected light.

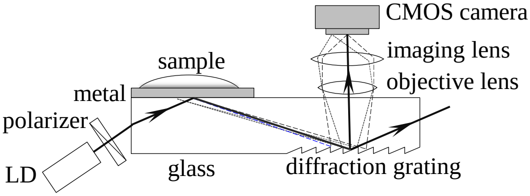

Fig.1: Optical setup of the SPR microscope using with a diffraction grating.

3. Aggregation of nanocomposite materials

To test the setup, we constructed an SPR microscope with a commercially available diffraction grating. First, the image distortion was evaluated. Figure 2 shows the experimental results for the evaluation of the image distortion. Figure 2 (a) shows the lattice pattern with a line width of 0.97 mm +/- 0.01 mm on a PET film. Figures 2 (b) and (c) show the images obtained by the conventional and proposed methods, respectively. As shown in Fig. 2 (b), the obtained image is distorted owing to the compression of the line width. In contrast, the proposed method produces an undistorted image. The length of the dashed XY line measured with both methods using the number of pixels was 0.30 mm +/- 0.01 mm and 0.97 mm +/- 0.04 mm, respectively. Clearly, the proposed method improves the aspect ratio.

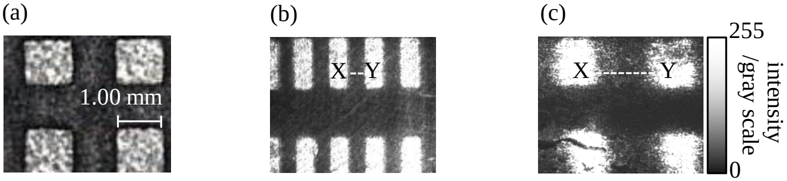

Fig. 2: Image distortion: (a) sample, (b) image obtained with the conventional method, and (c) image obtained with the proposed method.

For calibration, we used a sample with known refractive index. Figure 3 shows the dependence of the reflectance on the refractive index of a sucrose aqueous solution. We varied the refractive index by altering the sucrose concentration. The reflectance increased as the refractive index increased from 1.33 to 1.38, and remained constant between 1.38 and 1.45. These values for reflectance suggests the generation of SPR between the refractive index values of 1.33 and 1.38. However, above 1.38, the reflection is constant because SPR is not generated. Thus, in the range of 1.33 to 1.38, the dispersion state can be observed using the SPR microscope.

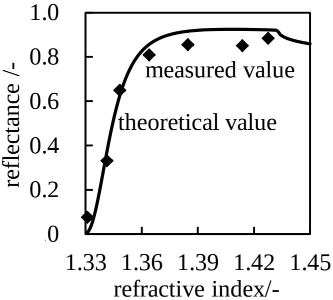

Fig. 3: Reflectance as a function of refractive index.

To observe the aggregation process of nanocomposite materials, we used a sample of water and SiO_2 nanoparticles with diameter between 10 and 20 nm (ST-30, Nissan Chemical Industries, Ltd.). The degree of nanoparticle aggregation depends on the pH levels. Thus, aggregation of the nanoparticles was controlled by the dropsies addition of distilled water. Figure 4 shows the observed images of the nanocomposite. Figure 4 (a) shows the image before adding distilled water. The intensity of the image is uniform because the nanoparticles are uniformly dispersed. Figure 4 (b) shows the image 3580 s after distilled water was added. The resulting intensity distribution is due to the change in resonance because of the nanoparticle aggregation. The refractive index distribution was calculated using the relation between reflectance and refractive index. Figure 4 (c) shows the distribution of the refractive index after 3580 s. The area with high light intensity is an aggregation area because its refractive index is close to the refractive index of SiO_2. The area with low light intensity has a refractive index close to that of water. Therefore, we have developed the SPR microscope with the diffraction grating that observes the aggregation process of the nanocomposite material by video rate.

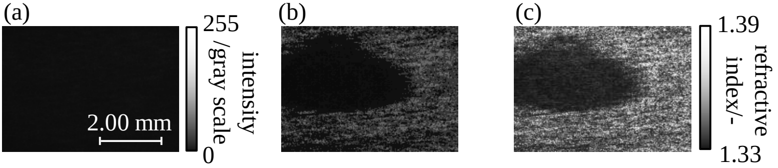

Fig. 4: Light intensity dependence of the dispersion state: (a) 0 s, (b) 3580 s, and (c) refractive index distribution.

4. Conclusions

An SPR microscope with the diffraction grating for observing the dispersion state of nanocomposites was proposed and described. The dependence of the reflectance on the refractive index was calibrated by measuring a sample of known refractive index. As a result, for refractive index between 1.33 and 1.38, the dispersion state can be observed using the SPR microscope. Additionally, the aggregation process in nanocomposite materials was observed by video rate.

References:

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