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Micro-Line Investigation in Titanium Dioxide Thin Film Coated on TiNi Alloy by Indirect Microscopy

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Abstract: Titanium Dioxide thin film was synthesized on Titanium Nickelide alloy by using sol-gel spin coating method. Nowadays, it is a big issue to investigate the micro crack line in any thin film through the optical microscope for character the adhesion. The super-resolution microscope is better for evaluating the adhesion. The surface morphology of the film was studied using Scanning Electron Microscopy (SEM). Recently, only expensive and inconvenient non-optical method such as SEM (Scanning Electron Microscope) and TEM (Transmission Electron Microscope) are available for high resolution services. Here, we are reporting on the investigations of the crack line in titanium dioxide thin film coated on titanium nickelide alloy by Superresolution Parameter Indirect Microscopy Imaging. Furthermore, we found that the crack line can be seen in PIMI images whereas in SEM the crack line not seen clearly even the magnification of SEM is 10000X, which have been demonstrated by comparing the line intensity across the crack.

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1.Introduction

Nowadays, very few methods have been developed for the investigation of micro scratch in a thin film. High resolution microscope is required for the investigation of indentation, cracks and morphology for the qualitative assessment of any thin film. It is very important to know the observation of tiny cracks in any thin films. The TiO₂ thin film has been selected for this report, because the film is very thin and effective as compare to other thin film. NiTi alloy coated on TiO₂ thin film has become an important orthopedic, cardiovascular and other implantation material. Because of their high specific strength, good corrosion resistance, good toughness, and excellent bio-compatibility, titanium, nickel and its alloys have found widespread applications in many engineering industries such as aerospace, chemical, marine and biomedical industry [1-3]. TiO₂ thin film has been studied intensively due to their high hardness and good resistance to wear, corrosion and oxidation. Many studies have been evaluated the properties of Ti based Ti-Cr-B-N thin film [4-5]; however, none to date have investigated the crack line of the NiTi based TiO₂ thin film. Usually it has examined by traditional microscope and SEM, due to more expensive, time taken and more

possibility to damage the sample, prefer the optical microscope. In this way, we proposed a new imaging method (PIMI). To overcome the difficulties in optical resolution improvement, many efforts on optical microscopy have been made, including optimizing SiO₂/Si substrate to improve contrast built on Fresnel reflection [6-7], using contrast spectroscopy and improved refractivity to built empirical thickness-contrast relationship for variable thickness quantification [8-10]. Recently we have reported the light scattering by sub wavelength Cu₂O particle [11], and application of PIMI method of sensing the scattering signals from a sub micrometer particle [12]. In this work, we have proposed a new application of PIMI, the material of NiTi alloy coated on TiO₂ Thin film. The crack line properties of Titanium Oxide thin film was studied by Superresolution Parameter Indirect Microscopy Imaging. The resolution of the crack line has been calculated. The details are given in the following sections.

2. Basic Theory of PIMI:

As a substitute of utilizing the direct optical field intensity, in which the resolution is basically limited by the optical diffraction, PIMI indirectly obtains the contrast built by the indirect optical wave parameters obtained from modulated optical field. In this paper, these optical wave parameters are confined to polarization parameters that define polarization status, including polarization phase, orientation, degree of polarizations, and depolarization field intensity. By establishing the interaction between the structural anisotropy and the far field intensity disparity, we can ultimately obtain multiple images with contrasts built using the above polarization parameter varieties. All these images, though which can be used independently and have different spatial resolving power, are compensative to each other to reveal more detailed metrological and structural information about the SUM (Sample Under Measurements). Using the geometric optical coordination system, polarization orientations illustrated and the Jones model [14-17], one can find that during polarization sweeping, the pixel intensity, I_i , depending on the vector sum of two wave fronts of different polarization states from the intrinsic structural anisotropy of the SUM, varies with input polarization angle α . This dependence can be described by Eq. (1) in a simplified form.

$$\frac{I_i}{I_{dp}} = a_0 + a_1 \sin 2\alpha + a_2 \cos 2\alpha \quad (1)$$

where $a_0 = 1/2$, $a_1 = \cos 2\phi \sin \delta$, $a_2 = \sin 2\phi \sin \delta$

I_{dp} is the average intensities over all polarization status transmitted through or reflected from each field points of the SUM, so its value least affected by scatterings near field change of polarization status, and $\sin \delta$ is the sine of phase difference between two orthogonal polarization states, ϕ is the polarization angle of the slow axis. The field intensities at individual pixel of the CCD matrix were recorded while extensive the polarization which is achieved by turning a linear polarization disc in a control component. The intensity variation with parameters, such as the polarization phase and angles were fit to Eq. (1). From the derived equation, the values of the parameters such as I_{dp} , $\sin \delta$ and ϕ can be

extracted. From the above polarization parameters, using the relationship between Jones's and Mueller's model, the Stokes parameters, S_0 , S_1 , S_2 and S_3 can be further calculated with Eq. (2).

$$\begin{pmatrix} S_0 \\ S_1 \\ S_2 \\ S_3 \end{pmatrix} = \begin{pmatrix} E_x E_x^* + E_y E_y^* \\ E_x E_x^* - E_y E_y^* \\ E_x E_y^* - E_y E_x^* \\ i(E_x E_y^* - E_y E_x^*) \end{pmatrix} = \begin{pmatrix} I_{dp}(1 + \sin \delta) \\ I_{dp}(1 + \sin \delta) \cos 2\phi \\ \sqrt{2} I_{dp}(1 + \sin \delta) \sqrt{\sin 2\phi} \cos \delta \\ \sqrt{2} I_{dp}(1 + \sin \delta) \sqrt{\sin 2\phi} \sin \delta \end{pmatrix} \quad (2)$$

Where E_x and E_y are the x and y optical field components, $i = \sqrt{-1}$, * represent the complex conjugate, S_0 , S_1 , S_2 , S_3 are stokes parameters along with the depolarization intensity, I_{dp} , phase difference, $\sin \delta$, and polarization angle, ϕ can be retained to each pixel coordinates in the sensor space to form individual parameter maps, or indirect images as we call them, with the contrasts not built by the optical intensity but the spatial polarization parameters related to near field characteristics.

3. Experimental Setup:

With help of the above theory, we have developed an indirect polarization microscope with modules including the conventional microscope light path, polarized illumination, polarization control of the incident light, optical image field detection matrix (CCD) and control the analysis interface. The PIMI system illustrated in Fig. 1, was built by using an Olympus reflection microscopic system BX51M as the basic optical microscopic path, and inserting into it with polarization-modulation mechanics with the angle precision of 0.05 degree.

Fig.1: Modulated polarization parameter imaging system.

We have already discussed in the previous section, PIMI obtained optical images indirectly by analyzing optical intensity dependence on polarization parameters, and then achieve sample's property of measurement information. During measurements, the incident light with spectrum line width(<20 nm) is modulated by a polarizer with a precision of 0.05 degree, and as such the errors due to the structure deal are really censored. As we have shown, PIMI enables one to obtain optical images with much higher resolution than that obtained by conventional optical microscopes at the material, graphene [13]. In a conventional optical microscope, its resolution is ultimately limited by the system's diffraction. PIMI however adopts modulated scanning on one or more polarization parameters, such as the angle of polarization perpendicular to the optical axis, while continually measuring the optical field strength variation at all images point represented by pixel in the CCD matrix.

4: Results and Discussion:

In the results section, one can see in Fig.2, the image of conventional microscopy is represented by I_{00} , Fig.2(a), which has been compared with the PIMI (Fig. 2 (b-c)) and SEM (Fig.2 (d-e)) images. From the images, it is very easy to identify the spot area between them, shown in white frame of the above figs. There is a crack line in the red sphere, one can see the difference between PIMI image ($\sin\delta$) Fig.2 (b) and SEM image (Fig.2 (e)). The crack line is looking very clear in the PIMI image but in the SEM image, the crack line is not seen more accurately, even the magnification of SEM is very high (30000X). Furthermore, from Fig 2(d), in SEM image cannot ensure whether there is a crack line whose magnification is 10000X, while the PIMI system ensure the crack line which has only 100X magnification with the Numerical Aperture (NA) of 0.9. To make the image visibility comparable to our results applies orientation certainty level (OCL) and mean square error (MSE) [18, 19] to the direct and indirect images. We have found that the image contrasts of some of the polarization parameter such as phase difference ($\sin\delta$) and (Φ) are big difference as the direct imaging image (I_{00}). One can see in Fig.3, the image contrast of $\sin\delta$ and ϕ are more better as compared to the direct image, which can shown in table:1

Fig.2 Bright field images taken by the modulated polarization parameter imaging system using 100X

objective both compare with SEM and PIMI (a I_{00} ; b $\sin\delta$; c Φ ; d & e SEM)

Fig.3: (a) Bright field images taken by the modulated polarization parameter imaging system using 100X objective both compare with PIMI (I_{00} , $\sin\delta$, Φ) indicate the line.(b)Variation of Intensity with Pixel of (I_{00} , $\sin\delta$, Φ) from a TiO_2 film on TiNi alloy.

Table:1 Contrast of Images

S.N.	OCL	MSE	RMSE	OCL/RMSE	Contrast
I_{00}	0.3307	1.11E-04	0.01053	31.40	0.01
$\sin \delta$	0.3729	0.0011	0.03316	11.24	0.03
Φ	0.4223	9.98E-04	0.03159	13.36	0.05

Further, investigation of crack line on the thin film, we have drawn a red line very near to the spot in all

PIMI images, which can be shown in Fig3 (a). With help of line data, we have drawn a comparative plot between all PIMI parameter (I_{00} , $\sin\delta$, Φ). The images contrast have also calculated, which can be shown in (table:1). This curve is showing the information of signal from the different PIMI parameters. The image contrast of ϕ and $\sin\delta$ are higher than the contrast of I_{00} . From the table:1, one can see that the contrast ratio between I_{00} : $\sin\delta$: Φ is 0.01:0.03:0.05. On behalf of our report, one can say that PIMI is able to get the more information than conventional optical microscopy. For the support in our report, we have drawn the SEM data curve compared with PIMI parameter data (I_{00} , $\sin\delta$, Φ) shown in Fig.4. The SEM data curve can be obtained by using the matlab code. With help of Origin, we can draw the SEM data curve between position and intensity. From the curve, one can see that peak signal in PIMI (Φ) images is better as compare to SEM peak signal even the magnification of SEM is 10000X, which have already mentioned in the circle. The first peaks are very close and like the same in PIMI (Φ) images, but the second peak is not like the first peak. With help of above results, one can easily find the exact position of the line near the crack.

Fig.4 Variation of position / intensity between PIMI parameter ($(I_{00}, \sin\delta, \Phi)$) and SEM images with different magnifications (10000X,30000X).

From above analysis, it can help the reconstruction of samples, image processing of PIMI and recovering 3D image from polarization parameter, and more material measurement in the material science. With the help of high resolution optical microscope, the crack line can be observed clearly, which can also help for evaluate the film adhesion more efficiency.

Conclusion:

In this report, we have been discussed some of the most exciting and promising research directions in the field of crack investigation on thin film through PIMI. With help of PIMI system, we can investigate the small crack on the thin film, which was not resolved by conventional microscope and even the SEM, and it can be used to evaluate the adhesion accurately. This method can also be useful for the inspection of the morphology, crack, scratch on the nano-films.

Compliance with Ethical Standards: The authors declare that, they have no conflict of interest.

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Caption List

Fig.1:Modulated polarization parameter imaging system.

Fig.2: Bright field images taken by the modulated polarization parameter imaging system using 100X objective both compare with SEM and PIMI (a I_{00} ; b $\sin\delta$; c Φ ; d & e SEM)

Fig.3: Bright field images taken by the modulated polarization parameter imaging system using 100X objective both compare with PIMI (I_{00} ; indicate the line.(b)Variation of Intensity with Pixel image quality and resolution analysis results of (I_{00} , $\sin\delta$, Φ) from a TiO₂ film on TiNi

Fig.4: Variation of position/ intensity between PIMI parameter ($(I_{00}, \sin\delta, \Phi)$) and SEM images with different magnifications (10000X,30000X).

Table 1: Contrast of Images.