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NON-DESTRUCTIVE CHARACTERIZATION OF THICK DLC-FILMS

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Introduction

The purpose of this study is to characterize the microstructure of DLC films which are used now widely as the wear resistant coatings using scanning acoustic microscopy (SAM) and atomic force microscopy (AFM). The use of wear resistant coatings offers a significant improvement to wear and/or abrasion resistance even though bulk properties of the component remain the same. Preliminary wear tests on nano-structured DLC coated samples demonstrated an appreciable improvement in pitting resistance and usable life. One of the key parameters that have great impact on coating wear resistance is the microstructure; particularly, density, size and location of the defects within the coating.

Because the thickness of these wear resistant coatings is a few microns, a tool that is capable of nondestructively studying internal microstructure is a high frequency SAM [1, 2]. The images of subsurface defects inside of soft materials is easy for interpretation, since it can be made using simple ray optical approach [2, 3]. The interpretation of the images in hard and super hard solids is more complicated than for softer materials because the contrast is mainly determined by the Rayleigh surface waves [2]. For these materials, the Rayleigh waves penetrate deeper than the location of the focal points of longitudinal and shear waves. So, interpretation of the acoustical images of DLC-films requires the knowledge of their elastic properties [4]. The combination of SAM with ensemble techniques, namely AFM and optical microscopy allows to characterize the nature of the defects.

Samples

The partly transparent Cr containing DLC-coating film was approximately 3 μm thick and was deposited on a heat-treated, polished steel substrate. The coating density was then determined based on the mass and volume of the coating and it was estimated to be about 2.6×10^3 kg/m^3, with a variation of $\pm0.3\times10^3$ kg/m^3.

Acoustical visualization

A Leitz ELSAM™ scanning acoustic microscope with an operating frequency 1 GHz was utilized for getting acoustical images. This instrument can achieve a high

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spatial resolution of about one micron. Fig. 1 gives a comparison between the optical and acoustical images of the same area 240 μm × 240 μm of the DLC-film.

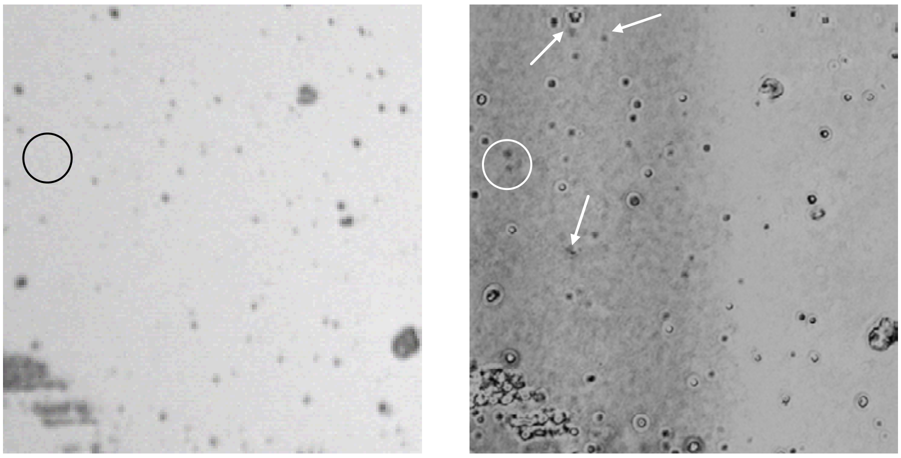

Fig.1. Optical (left) and acoustical (right) images of the partly transparent DLC film on steel substrate. The acoustical beam is focused on the top surface

Careful comparison of the images revealed that most (90%) of the micron-sized defects visible in the acoustical image (Fig. 1) could be found in the optical one (Fig. 2), though most of the defects (60%) clearly seen in SAM images have vague contrast in the optical image. Approximately 10% of all defects can be detected only by SAM (noted by arrows).

To understand the nature of the defects seen with SAM, the images were taken at different defocus position *z*. Figure 2 shows the image formed at the defocusing $z = -4$ μm. When image is taken at focus the defects are seen as small bright or dark spots; as focus moves down below the surface, the defects became brighter and wider, indicating that they locate under surface.

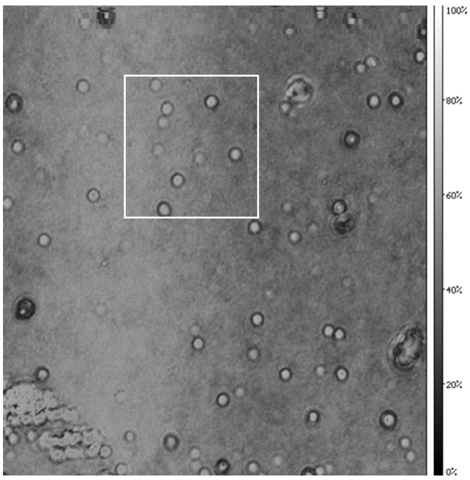

Fig. 2. Acoustic image was taken at 1.0 GHz, defocus was $z = -4$ μm. The field of view 240x240 $μm^2$

In the acoustical images shown in Figs. 1 and 2, the small circular features were most likely due to the voids in the coatings. The large non-circular features could be

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associated with the pollution and delamination in the coating.

The longitudinal sound wave velocity of this DLC film was evaluated as 5,2 km/s by the new technique combining surface Brillouin scattering measurement and laser – SAW technique [5]. So, a simple geometrical optics based model could be used for estimation of the depth of the subsurface defect. According to this model,

$$z_i = z\frac{V_{Water}}{V_{Coating}},$$

where z is defocus, or the displacement of the lens from the position where the focus is in the surface of the specimen, z_i is the depth of the focus in the coating, V_{Water} and $V_{Coating}$ are the longitduinal sound velcities in water (served as the coupling medium) and the coating, respectively. The sound velocity in water is 1.48 km/s at room temperature. The ratio of the defocus to the depth of the focus in the coating is therefore about 3,5:1. By using this ratio and comparing the signal magnitude of a defect in the images from different defocuses, the depth of the defect is estimated. For example, by comparing the 1 GHz images of specimen shown in Fig. 3, one can see that the signal magintudes of the small circular defects are maximal (brightest) in the image with a defocus of –4 μm. The defects are therefore approximately 4/3,5 or 1.1 μm deep.

AFM visualization

Atomic force microscope (Digital Instrument) was utilized for detail study of the defects inside the square on the Fig. 2. Figure 3 presents AFM images of the defect seen in optical images only. Both images show perfectly the film structure with grain size in the range of 100–200 nm. Analysis of the oval pit of 2 μm in width and 4 μm in length gave the depth of 200 nm only. Deflection mode image confirms the absence of the film discontinuity and maintenance of the grain structure inside the pit. The strong change of the cantilever deflection indicates the V-form of the pit.

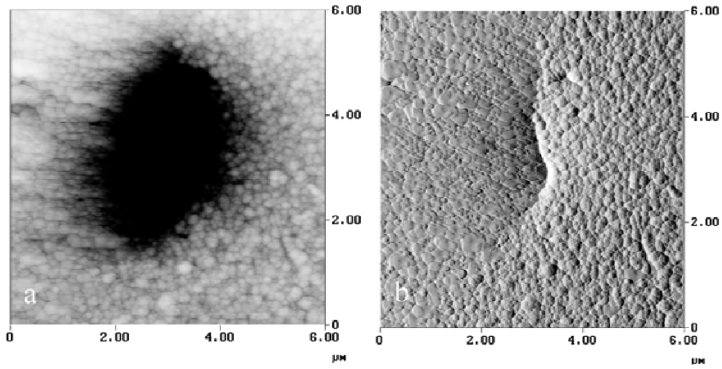

Fig. 3. AFM images of the pit in Cr-DLC film on steel: constant force mode (left) and deflection mode (right)

Figure 4 presents the AFM images of the bumps taken in the constant force mode (*a* and *c*) and in deflection mode (*b* and *d*). Such defects were seen as black dots in the optical image, but they could not be seen in acoustical microscope if their lateral size and height are smaller than sound wavelength except they are the strong scatterers of

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sound waves. Since the micron-sized bumps are presented on acoustical images with strong contrast we assume that a bump was grown above a bubble-like defect arising during deposition.

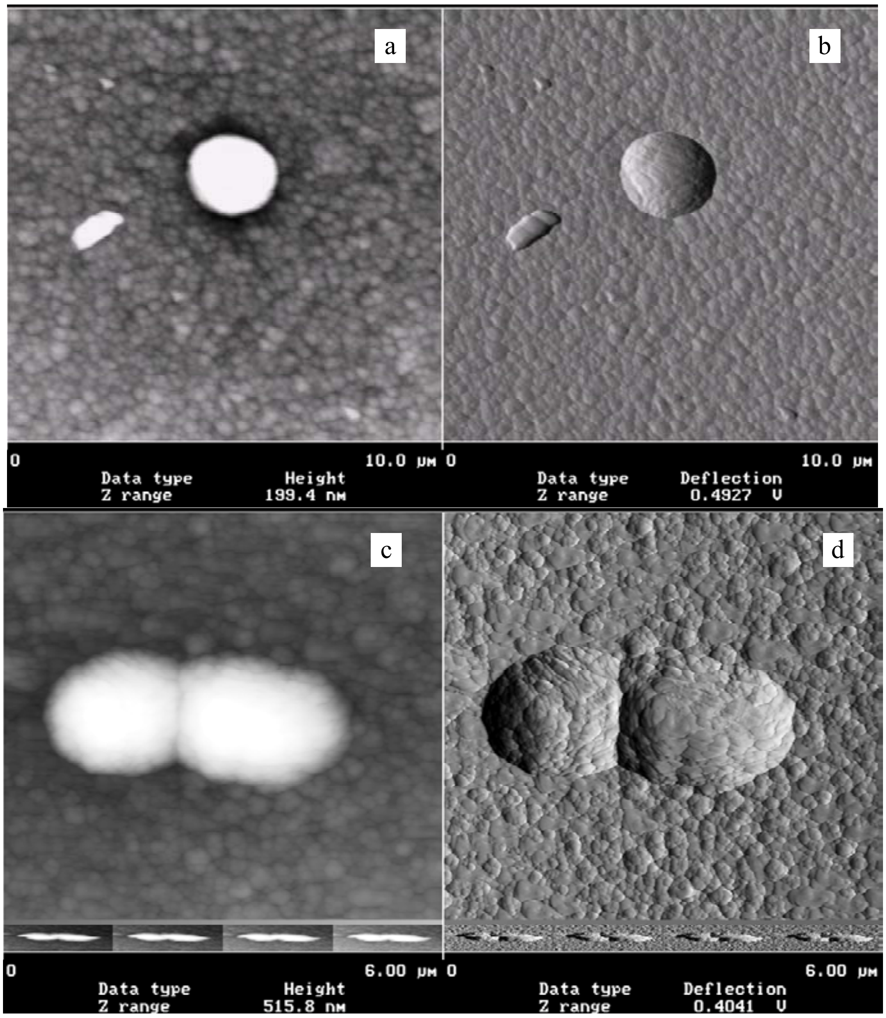

Fig. 4. AFM images of the bump defects in the DLC-films

To confirm the nature of the defects in the coating, the Cr-DLC specimen was positioned in a FIB/SEM imaging system. An ion sputtering process was applied from above the specimen to do fine milling of the coating. The specimen was then imaged with high resolution by SEM. With repeated sputtering and imaging, the 3-D microstructure of the coating was examined without removing or repositioning the specimen.

Figure 5 shows the SEM images taken in the Cr-DLC specimen after sputtering. The original SEM images were taken with a view angle of 60°. This preliminary study confirmed that small voids indeed exist in the Cr-DLC coating, especially in the region close to the substrate (Fig. 5).

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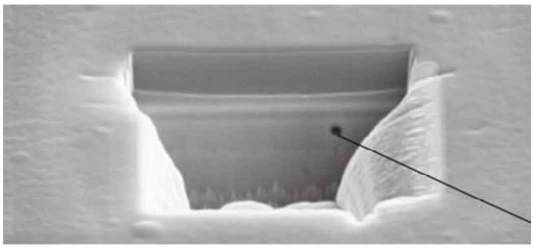

Fig. 5. SEM image of the FIB cut (groove) into the sample. Dark spot is a void

Conclusion

The investigation including the different kinds of microscopic methods seems to be very useful for nondestructive characterization of the defects in thick DLC-films. Focused ion beam milling may be used for the confirming the bubble-like character of the defects.

Acknowledgement

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