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Residual stress measurement in thin films using the semi-destructive ring-core drilling method using Focused Ion Beam

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Abstract

In the present study, residual stress evaluation in thin films was achieved using a semi-destructive trench-cutting method. Focused Ion Beam (FIB) was employed to introduce the strain relief by ring-core milling, i.e. creating a trench around an "island". Either SEM or FIB imaging can be used to record sequences of images for strain change evaluation by Digital Image Correlation (DIC) analysis of micrographs. A regular array of shallow holes was drilled on a thin overlayer of Pt (~100nm) deposited on to the film prior to patterning and trenching, in order to reduce the damage introduced by the ion beam during imaging and to assist the DIC strain evaluation by adding traceable markers. Finite Element (FE) simulation was also carried out to predict the curves for strain relief as a function of milling depth, and compared with the experimental measurements, which show good agreement with each other. An empirical mathematical description of the curves was proposed that allows efficient residual stress evaluation in thin solid films.

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Keywords: residual stresses; FIB; SEM; DIC; strain relief master function

1. Introduction

Residual stresses are the stresses that persist after the original cause of deformation has been removed. Their existence in thin solid films may greatly influence their performance and durability in service. Modern engineering practice demands that they should be taken into consideration in all stages of design,

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performance prediction and assessment [1]. However, when the cause, distribution and magnitude of the residual stresses are unknown, their presence introduces a great deal of uncertainty. Large residual stresses could lead to film delamination and buckling under compression [2], or film cracking and/or decohesion under tension [3]. Therefore, in order to improve the reliability of thin film systems in mechanical and functional engineering applications, it is crucial to evaluate the residual stresses in thin films with sufficient accuracy to allow appropriately refined predictive modeling to be carried out.

Residual stresses exist across different scales [4]. Thin films typically lie in the micro- to nano- length scale, with thicknesses covering the range from several micrometers to sub-nanometer. It is well-known that at such scales, the mechanical properties of thin films are strongly influenced by both their modified structure (e.g. caused by the proximity of initerfaces and free surfaces), and by the magnitude and through-depth distribution of residual stresses [5]. However, if carefully controlled, residual stress can be used to improve the performance of thin films. This is illustrated by the use of strained semiconductors that can be used to improve transistor performance by over 80% [6], or to modify the band gap by the creation of quantum dots in strained layers.

The most commonly used techniques for residual stresses measurement in thin films can be classified into destructive and non-destructive methods. The most prominent non-destructive physical evaluation method makes use of X-ray diffraction. In thin solid film applications, $\sin^2\psi$ X-ray diffraction method is often used [7]. However, its application is generally limited to fine-grained (poly)crystalline materials [8] where Bragg's Law applies. Destructive methods, on the other hand, can be applied to a wider range of materials, including single crystals, amorphous, nanocrystalline or single crystal materials. The method relies on monitoring the changes in material deformation, either in the process of residual stress introduction e.g. coating deposition, welding, or subsequent relaxation e.g. mechanical or electro-chemical removal of material. Common destructive methods include curvature measurement (with layer deposition or removal), hole drilling, crack compliance, etc. [4]. These methods may also be applied at the microscopic level [9], provided suitable micro-machining tools can be employed, notably e.g. Focused Ion Beam (FIB) milling.

FIB milling provides an excellent tool for microscopic material removal in a controlled environment. In present study, dual-beam (FIB+SEM) system was first employed, with FIB used for surface patterning and trench milling, and Scanning Electron Microscope (SEM) for imaging. The approach allowed the examination of residual stresses in TiN PVD coating on WC/Co substrate. In order to extend the use of this approach to the cases when dual-beam equipment is not available, the use of FIB for *both* milling and imaging was developed. Thus, in second study reported here, single instrument FIB milling and imaging technique was employed to examine the residual stress state in thin BD^{TM} (Black Diamond) films. We demonstrate that stress evaluation at *sub-micron resolution* was achieved. The use of an empirical master curve based on the experimental data and FE modeling was developed in order to shed some light on the general relationship between the normalized milling depth and the relaxation strain for this type of technique applied to thin films.

2. Residual stresses measurement using dual-beam system

The motivation for using dual-beam (FIB+SEM) systems for residual stresses measurement on thin films is to take advantage of the simultaneous availability of both ion beam (easy ablation of materials) and electron beam (good imaging resolution). Hence, in the first study reported here [8], FIB was used for surface patterning and annular trench milling (FIB ring-core drilling), and SEM was used for imaging.

The sample under examination was 3.8 μm TiN CAE-PVD coating on thick WC/Co substrate. Fig.1. illustrates the FIB ring drilling configuration, showing the ring shape trench and the circular shape "island". To assist the DIC strain determination, contrast was improved by sputtering a thin layer of Pt

prior to FIB drilling, and patterning it with a regular array of shallow milled dots. The ring trench was cut incrementally so that the Pt surface was not disturbed by the FIB drilling.

Finite Element (FE) simulation of residual deformation states during incremental ring drilling was carried out. The residual stresses were created by assigning different thermal expansion coefficients to the coating and substrate parts of the model, so that the misfit could be readily generated by imposing a uniform temperature change. The ring trenching process was simulated by progressive element removal. The strain state at the sample surface was re-computed in the model after each step, so that the dependence of strain relief on the removal depth could be plotted alongside the experimental data.

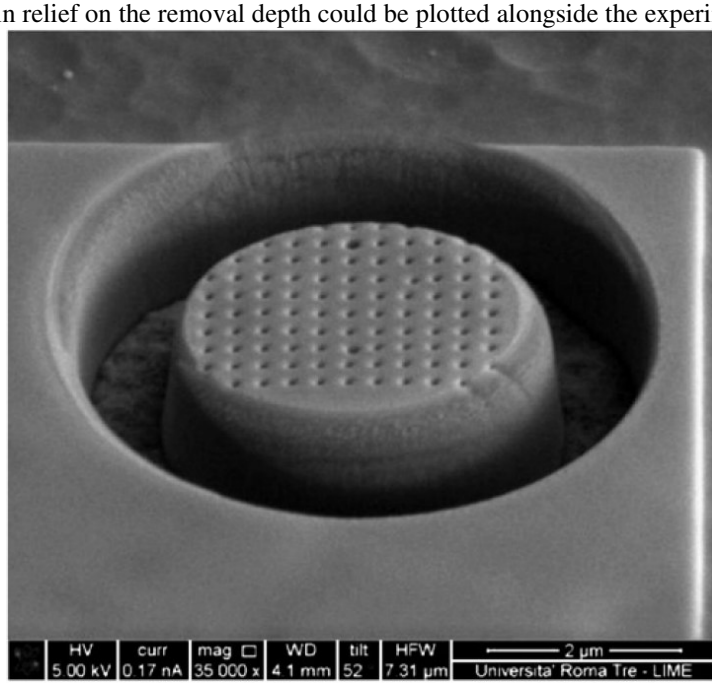

Fig. 1. Illustration of FIB ring drilling geometry with Pt sputtered surface and regular dot pattern [8].

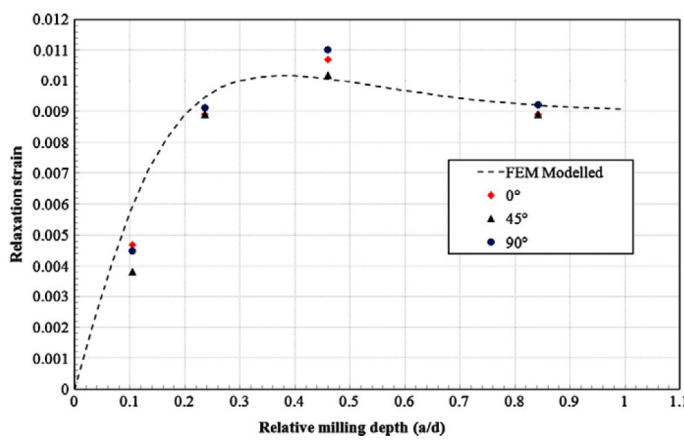

Fig. 2. Plots of Finite Element Simulation result (dashed line) with FIB ring drilling DIC measurement (markers) [8].

Fig. 2. illustrates the Finite Element simulation result (continuous curve) plotted alongside the FIB drilling-SEM imaging experimental results (markers) for the strain relaxation against the relative trench depth, i.e. trench depth *h* normalized with respect to the "island" diameter, *d*. For drilling depths exceeding the island diameter, the strain relief value approaches a constant value as the residual stress in the "island" becomes fully released [8]. Hence, the residual elastic strain value could be readily extracted (as the complete relaxation strain value when relative milling depth exceeds unity); and residual stress computed.

3. Residual stresses measurement using FIB milling and imaging

In the case described above, a dual-beam SEM-FIB system was employed. However, in many industry applications (i.e. in-line quality control instruments in the semiconductor industry), dual-beam equipment may not be readily available. It would be an advantage if both milling and imaging operations could be performed by the ion gun. With the development of high resolution imaging capabilities of FIB instruments, it has become possible to examine FIB-sectioned specimens under the ion beam. In this section, single instrument FIB milling and imaging technique is presented that allowed us to examine the residual stress state in thin BD^{TM} (Black Diamond) films in sub-micron resolution.

The samples under investigation are non-porous low-κ BD (Black Diamond^{TM}) carbon doped silicon dioxide PECVD coatings on Si wafer substrates. Due to the inherently destructive nature of ion beam treatment, damage may be introduced into the sample during FIB milling and imaging. For example, milling may cause re-deposition of removed material over the surface of interest, changing the topology of the area and hampering subsequent strain evaluation by image analysis. Moreover, even low current imaging using ion gun could cause direct damage to the sample surface, ultimately rendering attempts to carry out digital image correlation doomed to failure. Therefore, the FIB milling and imaging process was optimized so as to reduce the damage by ion beam to the sample surface during the complete test. This was achieved by adopting an outer-to-inner spiral path for the ion beam during milling, and depositing a thin (~100 nm) layer of Pt prior to patterning and trenching, providing improved resistance to ion beam damage. Using the Pt layer also assists the DIC strain evaluation, in that drilling a regular array of shallow holes (appearing dark in the image) within this layer (that appears light, due to high Z of the material) adds distinctive features to improve contrast for image correlation quality. Fig. 1a shows the sample of BD film of 1000nm thickness has a 5×5 hole pattern on the trenched island in the form of a rounded-corner square of 900×900nm^2 size. In this case it is clear that at the final stage of milling when the trench reaches the coating-substrate interface, the milling depth *h* exceeds *d*, the island's effective diameter, so that the relative milling depth, *h*/*d* exceeds the value of unity. According to Fig. 1b, this ensures that complete strain relief was attained in the experiment, and the evaluation of the residual stress state can be readily accomplished.

The condition of relative milling depth exceeding unity has, however, proved more difficult to attain for the 600×600nm^2 island configuration employed for the study of 300nm-thick BD film.

The DIC analysis of the FIB images was carried out using a Matlab® code developed at the JHU [10]. With the help of this code, the statistical average strain within the island surface can be calculated, and strain values were obtained from consecutive images with increasing cutting depth plotted as shown in Fig. 4a and 4b. Finite Element (FE) simulation was carried out to provide comparison with the experimental data.

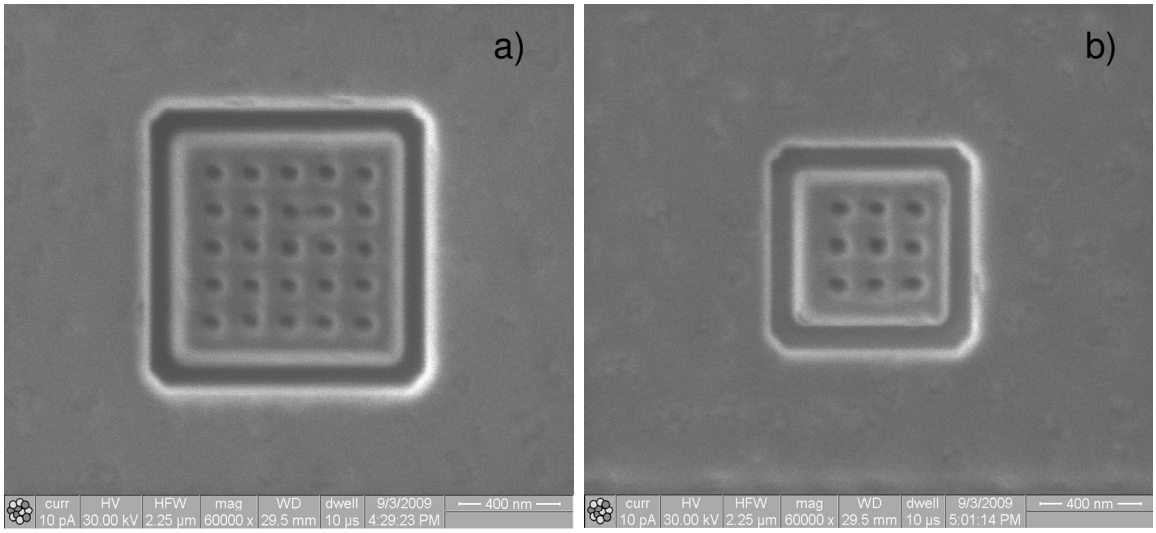

Fig. 3. a) Illustration of BD 1000nm-thick film test with a regular 5×5 pattern and rectangular island with 900nm side length; b) BD 300nm-thick film test with a regular 3×3 pattern and square island with 600nm side length (~850nm diagonal).

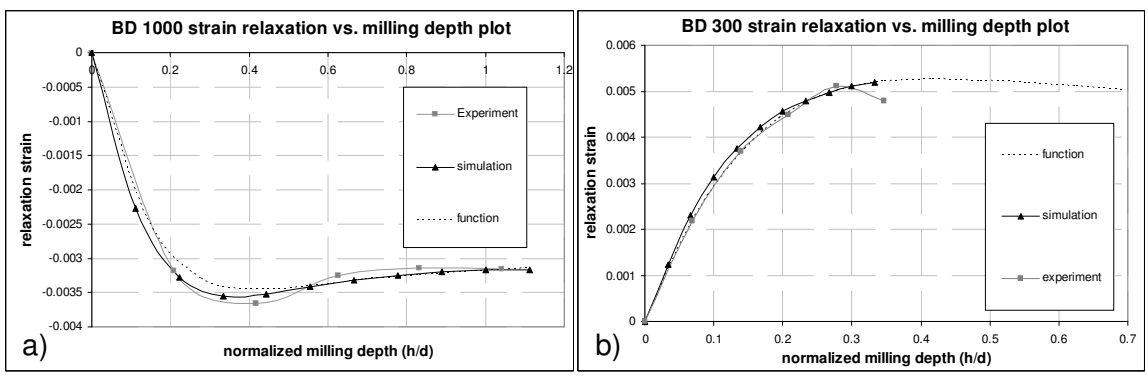

Fig. 4. a) Illustration of BD 1000nm-thick film test with the regular 5×5 patterning and square island of 900nm side length; b) BD 300nm thick-film with regular 3×3 patterning and square island of 600nm side length and ~850nm (sub-micron) diagonal.

Fig. 4a illustrates the curves of DIC measured and FE computed relaxation strain against normalized milling depth z. The experimental result matches the simulation data reasonably well. A master function curve has been proposed to link the relaxation strain with the relative milling depth (Eq. 1), where the relief strain $\Delta\varepsilon_\infty$. is the uniform residual strain value within the film. The function captures all characteristic features of the plots, providing a good match with both the experimental data and simulation curve results:

$$f(\Delta\varepsilon_\infty, z) = 1.12\Delta\varepsilon_\infty \times \frac{z}{(1+z)}\left[1+\frac{2}{(1+z^2)}\right], \qquad z = (h/0.42d) \tag{1}$$

The master function is also applicable in the situation when $h<d$, although in this case strain relief can't be fully developed because of the small film thickness and relatively coarse FIB milling resolution. In Fig. 4b, the master function fits the strain relief plots from both simulation and experiment. These were validated by independent results from different researchers [11, 12]. Therefore, the mathematical expression of the function can be seen as a convenient tool for residual strain analysis within thin films.

4. Conclusions

FIB semi-destructive method offers an efficient and practical means of residual stress evaluation at the micron to sub-micron level, which position is further strengthened by the introduction of the strain relief master curve function. This method has great potential to be widely applied to the thin film coating residual stress measurement, where conventional curvature measurement is unable to provide any meaningful spatially resolved information.

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