High-throughput analysis of thin-film stresses using arrays of micromachined cantilever beams

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We report on a technique for making high-throughput residual stress measurements on thin films by means of micromachined cantilever beams and an array of parallel laser beams. In this technique, the film of interest is deposited onto a silicon substrate with micromachined cantilever beams. The residual stress in the film causes the beams to bend. The curvature of the beams, which is proportional to the residual stress in the film, is measured by scanning an array of parallel laser beams generated with a diffraction grating along the length of the beams. The reflections of the laser beams are captured using a digital camera. A heating stage enables measurement of the residual stress as a function of temperature. As the curvature of each beam is determined by the local stress in the film, the film stress can be mapped across the substrate. This feature makes the technique a useful tool for the combinatorial analysis of phase transformations in thin films, especially when combined with the use of films with lateral composition gradients. As an illustration, we apply the technique to evaluate the thermomechanical behavior of Fe–Pd binary alloys as a function of composition. © 2008 American Institute of Physics. [DOI: 10.1063/1.2912826]

I. INTRODUCTION

Thin films and coatings are widely used in engineering applications to impart unique combinations of magnetic, electrical, mechanical, optical, or chemical properties to a device. The mechanical behavior of a coating, while often not the primary reason why a coating is used, is critical to the reliability of the device. Indeed, a coating that peels or cracks as a result of excessive residual stresses is useless. Similarly, a coating that warps or even fractures a substrate is not desirable.

Measuring the residual stress in a film on a substrate as a function of temperature is a classic technique for obtaining information on the thermomechanical behavior of thin films.2 By using this technique, the yield stress, stiffness, and thermal expansion coefficient of a film may be measured. The technique can also provide information on the temperature at which a phase transformation takes place, on thin-film densification mechanisms such as grain growth, or more generally on any process that changes the dimensions of a coating.

The residual stress in a film can be measured using several methods, the most common of which are x-ray diffraction and the substrate curvature technique.2,3 X-ray diffraction measures the lattice strains in crystalline films. These strains can be converted into stresses if the elastic constants of the film are known. In the substrate curvature technique, the residual stress is determined by measuring the curvature of the substrate induced by the residual stress in the film.

This technique has the advantage that the residual stress can be calculated without knowledge of the mechanical properties of the film as long as the film is thin compared to the substrate. It is sufficient to know substrate properties such as thickness and modulus. The substrate curvature technique has long been applied to both complete substrates4–6 and cantilever beams.6–8

Traditionally, mapping of the thermomechanical behavior of a film as a function of composition has been accomplished by measuring the residual stress in the film as a function of temperature, one film at a time. This process is very time consuming if one is interested in measuring properties over a wide range of compositions and temperatures. Thus, we have developed a high-throughput technique that relies on the use of arrays of micromachined cantilever beams and multiple parallel laser beams to determine the film stress. First, a silicon substrate with an array of cantilever beams is fabricated using standard bulk silicon micromachining techniques. Then, a film with a lateral composition gradient is deposited onto this micromachined substrate, such that each of the cantilever beams is coated with a film of different composition. The residual stress in the film induces a curvature in the cantilever beams that is directly proportional to the local stress in the film. Since, in general, the residual stress in a film changes with composition, the curvature of the cantilever beams will vary from beam to beam. The curvature of each cantilever beam and hence the residual stress in the film coating it are measured by scanning an array of parallel laser beams along the cantilevers. This approach makes it possible to simultaneously measure the stress-
temperature behavior of a large number of films of different compositions, leading to significant savings in cost and time. Arrays of cantilever beams with compositional gradients have been used before to obtain qualitative information on phase transformations in ternary alloy systems. Here, we describe a technique to obtain quantitative information on the film stress. We apply the measurement technique to map the thermomechanical behavior of binary Fe–Pd alloy films as a function of composition.

II. STRESS MEASUREMENT PRINCIPLE

Consider a long cantilever beam coated by a film with a residual stress \( \sigma_{\text{res}} \). The stress in the film causes the cantilever beam to deflect. The change in beam curvature, \( \Delta \kappa \), is directly proportional to the residual stress in the film and is given by Stoney’s equation,

\[
\Delta \kappa = \frac{6tf(1 - \nu_b)}{E_b t_b^2} \sigma_{\text{res}}, \tag{1}
\]

provided that the film is much thinner than the beam and the width of the beam much larger than its thickness. In Eq. (1), \( E_b \) and \( \nu_b \) are Young’s modulus and Poisson’s ratio of the cantilever beam material; \( tf \) is the film thickness and \( tb \) is the beam thickness. The curvature of the cantilever beam can be measured by scanning a laser beam along its axis, as illustrated in Fig. 1, and by capturing the reflected beam on an imaging screen. The distance \( \delta \) by which the beam reflection is displaced on the imaging screen as a result of the curvature of the beam (see Fig. 1) is

\[
\frac{d\delta}{dx} = -\frac{2L}{\cos^2(\alpha + 2\beta)\cos \beta}, \tag{2}
\]

where \( \alpha \) is the incident angle of the laser beam, \( \beta \) is the angle of the local cantilever beam normal, and \( L \) is the distance between the cantilever beam and the imaging screen. For near-normal incidence of the laser beam and small deflections of the cantilever, the angles \( \alpha \) and \( \beta \) are very small, and Eq. (2) can be simplified to

\[
\frac{d\delta}{dx} = -2L \kappa. \tag{3}
\]

Thus, by measuring \( \delta \) as a function of the distance \( x \) along the cantilever beam, the curvature of the cantilever can be determined. Using Stoney’s equation, the residual stress in the film is then calculated from the change in beam curvature before and after the film is deposited onto the cantilever.

III. DESCRIPTION OF APPARATUS

The technique we have developed relies on arrays of micromachined cantilevers and multiple parallel laser beams to determine the residual stress in a film as a function of location on its substrate. Figure 2 shows a schematic diagram of the experimental apparatus used for the stress measurements. A laser beam shines through a diffractive optical element that generates a divergent array of laser beams. These beams are collimated using a lens with a long focal length and reflected off the micromachined cantilever beams. The laser beams are scanned along the long axes of the cantilevers by moving the substrate with the cantilevers on a micropositioning stage. The reflected beams are captured on a ground glass screen using a high-resolution charge coupled device (CCD) camera.

There are several ways of generating arrays of parallel laser beams to probe the micromachined cantilevers. In our apparatus, we make use of diffractive optics. A laser shines through a diffractive element that generates a square array of
laser beams with an interbeam angle $\gamma$. The diffracted laser beams are collimated using a planoconvex lens with focal length $f$. The diameter and focal length of the collimating lens are determined by the following considerations: the minimum lens diameter is set by the size of the micromachined array. Furthermore, the lens needs to be placed at a distance $f$ from the diffractive element to collimate the array. This requirement together with the interbeam angle $\gamma$ determines the distance between the laser beams in the array and hence the spacing $s$ between the micromachined cantilevers on the substrate:

$$s = 2f \tan \frac{\gamma}{2} \tag{4}$$

At first sight, any combination of $f$ and $\gamma$ satisfying Eq. (4) can be selected. In actual practice, $f$ needs to be at least as large as $L$ because the collimator lens causes the individual beams in the diffracted array to converge in focal points at a distance $f$ from the lens. If $f$ is much smaller than $L$, the diffracted beams significantly diverge before they are captured on the imaging screen. If $f$ is larger than $L$, on the other hand, the individual laser beams are nearly collimated and result in well-defined spots on the imaging screen that are readily analyzed. For a given collimating lens, the spacing between the laser beams can be adjusted by selecting diffractive elements with different interbeam angles $\gamma$. Alternately, the distance between the diffractive element and the collimating lens can be slightly adjusted.

Another technique for generating an array of parallel laser beams consists of shining a laser beam through two inclined etalons at right angles to each other. Internal reflection of the laser beam inside the etalons then results in a square array of parallel beams. This approach has the advantage that individual laser beam within the array are collimated and that the beams in the array are parallel so that no collimating lens is required. Moreover, the spacing between the beams is easily adjusted by changing the inclination of the etalons. However, because the individual beams in the array are formed by multiple internal reflections inside the etalons, the intensity of the beams in the array exponentially decreases from one side of the array to the other. It is therefore difficult to generate large arrays with many beams of uniform intensity. The diffractive optical elements we use in this work have the advantage over the etalon technique that large arrays of uniform intensity can be generated and that they are commercially available with a wide range of interbeam angles.

In the current implementation of the apparatus, we use a 3 mW laser diode with a wavelength of 635 nm. The diffractive optical element was purchased from StockerYale and generates an array of $33 \times 33$ beams with an interbeam angle of 0.38°. The array is limited to $7 \times 7$ beams by means of an aperture. The distance $L$ between the substrate with the cantilever beams and the imaging screen is 1000 mm; the focal length of the collimating lens is equal to $L$. The image on the screen is captured by a high-resolution CCD camera with a double-Gauss macroimaging lens with a 50 mm focal length to minimize spherical aberration. The camera is a Pulnix TM-1020 monochrome camera with 1008×1018 pixels and a pixel size of 9.0×9.0 μm². The images are captured at specified time intervals using an automated image acquisition program based on the commercially available LABVIEW. All optical components including the camera are mounted on a standard optical bench. Because of the simultaneous capture of all reflections in the array by the CCD camera, small movements of the laser beam array do not affect the distance between the reflections, i.e., the apparatus is virtually insensitive to small vibrations.

The substrate with the cantilever beams is placed on top of a heating stage and is covered by a flat window with an antireflection coating. The maximum temperature of the heating stage is currently 120 °C. The temperature of the heating stage is controlled to within 1 °C with a proportional-integral-derivative controller and a K-type thermocouple attached to the heating plate. During experiments, the temperature of the heating stage is continuously recorded using a LABVIEW program. The whole heating assembly is mounted on a one-axis translation stage that allows the laser beams to scan the cantilever beams. Both the optical bench and the translation stage are attached to a rigid metal frame. The entire system is enclosed in a box to allow operation of the system in ambient light conditions. A small fan is added to keep the outside of the heating assembly cool and to improve the stability of the laser beams by reducing hot air convection inside the enclosure.

IV. IMAGE AND DATA ANALYSIS

In order to calculate the curvature of the cantilever beams, the precise locations of the laser beam reflections on the imaging screen need to be determined from the images captured by the CCD camera. This is achieved by analyzing the digital images using custom software developed in a MATLAB environment.

First, the CCD images are processed to determine the approximate location of each reflection. By using binary thresholding, all images are converted from gray-scale to black-and-white images. The threshold for the conversion is calculated using Otsu’s method—an approach that chooses the threshold value to maximize interclass variance between light and dark pixels. Low-pass spatial filtering with a Gaussian kernel is applied to the images immediately prior to the thresholding operation to reduce noise in the binary image. With the intensity profile array successfully converted to a binary image, it becomes possible to define objects that correspond to sets of contiguous white pixels. The average area, i.e., number of pixels, of these objects is calculated and used to remove small remnant objects from the image—all objects under a constant user-definable multiple of the average area are dropped. The remaining objects correspond to the laser beam reflections in the image. It should be noted that identification of the reflections using this approach is highly simplified because the reflections all have approximately the same intensity as a result of the use of diffractive optics. Once the reflections have been identified, minimum bounding boxes for each reflection are computed from the binary image. These bounding boxes are relaxed by a user-definable percentage to ensure complete inclusion of the in-
RAPIDITY and reactive ion etching. In a second masking step, windows are opened in the SiN$_x$ on the back side of the substrate. Precise alignment of the patterns on the front and back of the wafers is not critical.

The cantilever beams are created by a two-step etch in an aqueous KOH solution at 85 °C. First, the back side of the silicon substrate is etched until approximately 100 μm of silicon remains. This process step determines the final thickness of the cantilever beams. Next, the front of the substrate is etched to define the cantilevers. The KOH solution is an anisotropic etchant that etches the silicon {100} planes much faster than the {111} planes. Thus, if the edges of the cantilever beams are aligned with the {110} directions in the silicon substrate, the cantilevers are bounded by the {111} planes in the silicon substrate. The result is an array of well-defined SiN$_x$-coated cantilevers, the sides of which make an angle of 54° with the surface of the substrate.

Figure 3(b) shows an optical photograph of a substrate with a 7×7 array of cantilever beams. Typical cantilever beam dimensions are 2.5 mm×4.0 mm×100 μm. The in-plane dimensions of the cantilever beams are determined mainly by the spacing and the diameter of the laser beams in the array, as well as by the scan length required for an accurate stress measurement. The thickness of the cantilevers is readily changed by adjusting the duration of the first KOH etch. The optimum cantilever thickness depends on the film thickness and the resolution required for the stress measurements. The silicon etch that defines the cantilever beam thickness is a timed etch; there is no etch stop layer. Consequently, it is important to ensure that this wet etch proceeds as uniformly as possible. This was achieved by performing the etch process in a water bath maintained at constant temperature. When the second KOH etch punches through the Si, the back side of the substrate is also exposed to the etchant and this can lead to locally nonuniform beams. This problem was eliminated by exposing both sides of the substrate to the etchant during the last few minutes of the etch process. This process resulted in a beam thickness nonuniformity of approximately 3% (one standard deviation).

VI. RESOLUTION OF THE TECHNIQUE AND STRESS CALCULATION

Through repeated measurements on various substrates, it was found that the distance between laser beam reflections on an image captured by the CCD camera could be reproduced to within 0.14 pixels (one standard deviation). This corresponds to a theoretical resolution of $5 \times 10^{-5}$ m$^{-1}$ for the curvature of a silicon substrate or to a resolution of $1 \times 10^{-3}$ m$^{-1}$ for the curvature of an individual cantilever. This curvature resolution is equivalent to a stress resolution of 0.4 MPa in a 1 μm film on a 100 μm cantilever beam.

Before quantitative stress measurements can be made, the distance $L$ between the substrate and imaging screen needs to be calibrated. This is achieved through use of a set of calibration mirrors with well-known radii of curvature. The displacements $\delta$ of the reflections are measured as a function of scan length $x$ for each of the calibration mirrors. The distance $L$ can be calculated from the slopes of the $\delta=x$ curves using Eq. (3).

\[ x = \frac{\sum P(x_p,y_p)x_p}{\sum P(x_p,y_p)}, \quad y = \frac{\sum P(x_p,y_p)y_p}{\sum P(x_p,y_p)} \]
An accurate determination of the residual stress in the films requires that the cantilever beam thickness is precisely known since the beam thickness enters Stoney’s equation quadratically. For the results presented in this paper, individual beam thicknesses were measured by focusing an optical microscope on the top and bottom surfaces of each beam. The difference in microscope height was taken to be the thickness of the beam.

Finally, it should be pointed out that Stoney’s equation is only valid in the limit of infinitely long beams, where the boundary conditions play no role. The cantilever beams used in this paper, however, have a finite length, so that the effect of the clamped boundary condition imposed by the silicon substrate needs to be evaluated. This effect was analyzed by Sader\textsuperscript{13} using an energy minimization technique. For beams with a length-to-width aspect ratio of 1.6, as the beams used in this study, the clamped boundary condition changes the curvature at the end of the beam by less 3%. Consequently, the error induced by the boundary condition was ignored in the current analysis. It is possible, however, to further reduce this error by defining narrower beams.

VII. APPLICATION: THE THERMOMECHANICAL BEHAVIOR OF Fe-Pd COATINGS

To demonstrate the technique described in this paper, a Fe-Pd coating was deposited onto a silicon substrate with cantilever beams using dc cosputtering in a vacuum system equipped with three confocal magnetrons (ATC 1800, built by AJA International). The design of this deposition system makes it straightforward to introduce lateral composition gradients in the coating by varying the power to and by changing the inclination of the individual magnetrons in the deposition system (see Fig. 4). The Fe-Pd coating was deposited on the SiN\textsubscript{x}-coated cantilevers by cosputtering from individual elemental Fe and Pd targets (see Fig. 4), which were 50.8 mm in diameter. The system base pressure was better than $2 \times 10^{-7}$ Torr, while the Ar working gas pressure was 5 mTorr. The power applied to the Fe and Pd targets was 270 and 70 W, respectively. The angle of the Fe target was fixed at 21° for uniform Fe deposition, while the angle of the Pd target was set to 16.2° to maximize the lateral nonuniformity of the Pd. During deposition, the substrate was rotated at a rate of 17 rpm to obtain a coating with an axisymmetric composition profile. The composition was analyzed by Rutherford backscattering spectrometry using a 1.7 MV General Ionex Tandetron Accelerator equipped with both a gas and a heavy ion source. The Pd content varied from approximately 22% in the center of the substrate to 30% near the edge as shown in Fig. 5(a). Because the Fe deposition was uniform across the substrate, the overall film thickness as measured with a profilometer also increased slightly from center to edge, as shown in Fig. 5(b). Figure 6 shows the variation of the residual stress as a function of Pd content in the as-deposited Fe-Pd thin-film alloy. It is evident from the figure that the residual stress values are in very good agreement with measurements on similar Fe–Pd films by Sugimura \textit{et al.}\textsuperscript{14} and that the stress increases slightly with increasing Pd content.

Before exploring the effect of the Pd content on the ther-

![FIG. 4. Schematic illustration of the confocal sputtering setup.](image4)

![FIG. 5. (Color online) (a) Variation of composition (a) and thickness (b) of the Fe-Pd coating.](image5)
momechanical properties of binary Fe-Pd alloys, the as-deposited Fe-Pd thin films were heat treated at 895 °C for 15 min in an atmosphere of Ar gas followed by a quench in flowing Ar and N₂ gas. This heat treatment results in a coating with a face-centered-cubic structure, which is normally only found at elevated temperatures. The SiNₓ on the cantilever beams prevented the formation of silicides through reaction of the Fe-Pd with the underlying silicon during the heat treatment. After the heat treatment, the curvature of the cantilever beams was measured as a function of temperature between room temperature and 120 °C by using the heating stage in the substrate curvature apparatus. Measurements were made at 10 °C intervals both on heating and cooling. Prior to each measurement, the temperature was allowed to equilibrate for at least 20 min, to ensure a uniform temperature and to minimize any curvature induced by temperature gradients.

Figure 7 shows the residual stress during the thermal cycle for various Fe-Pd compositions. At room temperature, the residual stress is tensile in all films. As the cantilevers are heated up, the residual stress changes as a result of the thermal mismatch between film and substrate. This stress change is linear and reversible during heating and cooling. For most metallic films, the thermal expansion coefficient is larger than for silicon, and one would expect the residual stress to decrease with increasing temperature. This is indeed observed for the majority of the films in Fig. 7. The stress in the film with 28.1 at. % Pd, however, shows the opposite trend. Upon heating, the residual stress increases up to 80 °C and then levels off at higher temperatures. On cooling the stress curve follows the heating curve in the reverse direction. This observation is consistent with previously published results for Fe-Pd films. The behavior at temperatures higher than 80 °C is attributed to an anomalously low value of the thermal expansion coefficient in Fe-Pd films of this composition as a result of the well-known Invar effect. The thermal expansion coefficient of Fe-Pd with this composition is similar to that of silicon and the stress is invariant on heating or cooling. The same behavior is also observed for the film with 29.4% Pd. The change in residual stress between 30 and 80 °C is associated with a martensitic transformation between the high-temperature face-centered-cubic phase and a low-temperature face-centered-tetragonal phase. In bulk Fe–Pd alloys, this martensitic transformation is known to occur well below room temperature at a narrow composition range around 30 at. % Pd. In thin films, by contrast, the transformation occurs near room temperature because of the high residual stress levels films are subjected to.

It should be noted that in the previous work by Sugimura et al., the Fe-Pd stress-temperature curves were obtained over a wide composition range by depositing, heat treating, and measuring one specimen at a time. The stress-temperature curves in this study were obtained using the parallel laser curvature system with significant savings in time and manpower.

VIII. CONCLUSIONS

We have developed a high-throughput technique that relies on arrays of micromachined cantilever beams, a special substrate curvature apparatus, and coatings with lateral composition gradients, to measure the thermomechanical behavior of alloy thin films as a function of composition. The cantilever beam arrays are fabricated using simple silicon micromachining techniques, while the curvature apparatus is constructed from inexpensive optical components that are readily available commercially. The technique is demonstrated by applying it to the binary Fe-Pd alloy system. Measurements are in good quantitative agreement with previously reported data, showing both the Invar effect and the martensitic transformation that are known to occur in this alloy system over a very narrow composition range.

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