Nanoscale Visualization of Elastic Inhomogeneities at TiN Coatings Using Ultrasonic Force Microscopy

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Received: 26 May 2009 / Accepted: 18 August 2009 / Published online: 16 September 2009 © to the authors 2009

Abstract Ultrasonic force microscopy has been applied to the characterization of titanium nitride coatings deposited by physical vapor deposition dc magnetron sputtering on stainless steel substrates. The titanium nitride layers exhibit a rich variety of elastic contrast in the ultrasonic force microscopy images. Nanoscale inhomogeneities in stiffness on the titanium nitride films have been attributed to softer substoichiometric titanium nitride species and/or trapped subsurface gas. The results show that increasing the sputtering power at the Ti cathode increases the elastic homogeneity of the titanium nitride layers on the nanometer scale. Ultrasonic force microscopy elastic mapping on titanium nitride layers demonstrates the capability of the technique to provide information of high value for the engineering of improved coatings.

Keywords PVD nanostructured coatings · TiN · Ultrasonic force microscopy · Scanning probe microscopy · Nanomechanics

Introduction

The technological relevance of titanium nitride (TiN) deposited by Physical vapor deposition (PVD) is reflected in its wide range of applications, from hard protective coatings in cutting tool industry to biomaterial in implantable devices [1, 2]. In such applications, phenomena such as cracking, wear and corrosion, among others, depend essentially on surface and subsurface features, e.g., microstructure, stress distribution, elastic discontinuities, defects and chemical composition [3–8].

Scanning acoustic microscopy (SAM) constitutes an outstanding tool to observe subsurface features such as elastic discontinuities in thin film materials. When an acoustic microscope is operated in imaging mode (qualitative mode), the image contrast provides a clear distinction of elastic gradients in the surface structure; nevertheless, the resolution is limited to the microscopic level at most [9–12].

Recently, a new family of scanning probe microscopy (SPM) techniques based on the use of atomic force microscopy (AFM) with ultrasound excitation has been proposed [13, 14]. It has been demonstrated that these procedures provide a valuable means for the characterization of dynamic elastic, viscoelastic and adhesive material properties, and permit to obtain subsurface information. Among them, the technique of ultrasonic force microscopy (UFM) [15–18] relies in the so-called “mechanical-diode” effect, in which a cantilever tip is in contact with the sample surface, and normal ultrasonic vibration is excited at the tip-sample contact. If the excitation frequency is high enough, or is not coincident with a high-order cantilever contact resonance, the cantilever will not be able to linearly follow the surface vibration due to its inertia. Nevertheless, if the ultrasonic excitation amplitude is sufficiently high
that the tip-sample distance is modulated within the non-linear tip-sample force interaction regime, the cantilever experiences a static force during the time that the ultrasonic excitation is acting. This force is called “the ultrasonic force”, and it can be understood as the net force that acts upon the cantilever during a complete ultrasonic cycle, due to the nonlinearity of the tip-sample interaction force. The cantilever behaves then as a mechanical diode, and it deflects when the tip-sample contact vibrates at ultrasonic frequencies of sufficiently high amplitude. The magnitude of the ultrasonic force, or of the ultrasonic-force-induced additional cantilever deflection (UFM signal), is dependent on the details of the tip-sample interaction force, and hence on material properties such as elasticity and adhesion. In this way, surface and/or subsurface nanoscale elastic discontinuities and stress fields can be easily detected with UFM.

Earlier reports have presented a continuum mechanic description of the tip-sample interaction of the UFM response using the Johnson–Kendall–Roberts (JKR) model, demonstrating that with this technique it is -in principle- possible to measure absolute stiffness values of nanoscale contacts, and effectively differentiate materials with distinct elastic constants [17, 19]. Also, methods to obtain information about the work of adhesion and the adhesion hysteresis at the tip-sample contact using UFM have been proposed [20, 21]. UFM has been successfully applied to the study of nanometer-sized Ge islands epitaxially grown on a Si (100) substrate [22]. Nanoscale mapping of these islands revealed variations in the UFM contrast, which were attributed to local variations in elasticity. More recently, Cuberes et al. [23] applied UFM to investigate the elastic nanostructure of individual Sb particles. In that study, the UFM images also revealed variations in the particle stiffness, attributed to locally strained regions within the Sb nanoparticles.

In this article, the results of an UFM investigation consisting in nanoscale elastic mapping are presented, along with X-ray Diffraction (XRD) and scanning electron microscopy (SEM) analysis of magnetron sputtered TiN films produced by varying the sputtering power applied to the Ti cathode. The aim of this investigation is to test the potential of UFM for nanoscale mapping of hard coatings and assess the elastic quality and possible origin of the UFM response (elastic discontinuities) in the TiN films.

Experimental Details

Preparation of TiN Coatings

TiN coatings were prepared by dc magnetron sputtering onto polished AISI 304 stainless steel (SS) discs in a vacuum chamber at room temperature using a water-cooled Ti target. SS-AISI-304 is commonly used in chemical, marine, food processing and hospital surgical equipments, etc. due to its good chemical and mechanical properties, and it is expected that good-quality deposited PVD-TiN coatings will further improve its surface properties. Depositions were carried out varying the power at the cathode $W_S = 100, 150$ and $200$ W in a N$_2$ and Ar atmosphere with a N$_2$:Ar ratio of 50% and a total pressure of 1.3 Pa with grounded substrates during 60 min, for all experiments. The discharge was started using a pure Ar atmosphere yielding a titanium layer of about 500 nm. After that, the N$_2$:Ar ratio was fixed, and the TiN layer was deposited without interruption.

Characterization of TiN Coatings

The coated samples were characterized by XRD in a symmetric $\theta-2\theta$ Bragg–Brentano configuration using a Philips X’Pert diffractometer with Cu K$_\alpha$ radiation in order to observe the developed crystallographic orientations. Elastic mapping at the nanoscale was performed with AFM–UFM, using a commercial AFM system (Nanotec) modified as shown in Fig. 1a [14]. Olympus rectangular Silicon Nitride cantilevers (spring constant of 0.6 N m$^{-1}$, with a pyramid-like shaped tip) were used for the...
measurements. Sample UFM mode (S-UFM) was implemented by exciting the ultrasonic vibration at the tip-TiN sample contact using a piezotransducer bonded with polycrystalline salol at the back of the coated stainless steel disc. The modulated ultrasonic vibration at the piezo was excited using an arbitrary waveform generator (Agilent 33220A). The ultrasonic-induced cantilever response—dependent on the local material properties—was detected at the ultrasonic modulation frequency by means of a lock-in amplifier. Figure 1b shows a typical UFM curve obtained by recording the ultrasonic-induced cantilever deflection (UFM signal) when the tip is in contact with the sample surface with a set-point force of $\approx 70$ nN, and an ultrasonic signal of 4 MHz is excited at the tip-sample contact, being its amplitude linearly varied from 0 up to a maximum amplitude $A_m$ of 8 V $pp$ (piezo excitation voltage). To record an UFM image, the triangular-shaped signal in Fig. 1b is periodically excited, and the resulting UFM response is detected by means of a lock-in amplifier. A higher UFM signal is usually indicative of a stiffer area; nevertheless, adhesion also plays a fundamental role in the UFM response.

The cantilever response to the ultrasonic force (UFM signal) $F_{ult}$, is given by [15–17]:

$$F_{ult}(h_{eq}, A) = \frac{1}{T_{ult}} \int_{0}^{T_{ult}} F\left[h_{eq} - A \cos(\omega t)\right] dt$$

(1)

being $A$ the ultrasonic excitation amplitude, $\omega$ the ultrasonic frequency, $T_{ult}$ the ultrasonic time period, $h_{eq}$ corresponds to the quasi-static equilibrium position reached by the tip in the presence of ultrasonic vibration. $F_{ult}$ is responsible of the ultrasonic deflection (or UFM response) of the cantilever. In the presence of ultrasound, due to the nonlinearity of the tip-sample force, the tip moves from an initial position $h_o$ to a quasi-static equilibrium position (UFM deflection) $h_{eq}$, which is larger the higher the ultrasonic excitation amplitude, as can be seen in Fig. 1b. Quantitative analysis of the UFM data requires an accurate calibration of the system and in most cases a better understanding of the dynamic tip-sample interactions [24].

Our AFM–UFM set-up (Fig. 1a) allows us to simultaneously record the AFM image in contact mode (topography) and the UFM image (elastic mapping) of a same TiN area. UFM imaging was stable in all the analyzed samples, and the recorded images showed no sign of deterioration in time. From the topographic images recorded in AFM contact mode, it is possible to determine the root-mean-square (RMS) roughness at each of the sample surfaces. The sample surface structure was also investigated by SEM, and the coating thicknesses were obtained from SEM cross sectional views. The grain size was measured both with AFM and SEM, obtaining consistent results.

**Results and Discussion**

Crystallographic Orientations

XRD patterns from TiN deposited onto SS-AISI 304 as function of the sputtering power applied to the cathode are shown in Fig. 2a. The TiN coatings were polycrystalline and exhibited diffraction peaks related to the cubic $\delta$-NaCl structure. The XRD patterns show the (200) (characteristic of the [100] orientation [25]) and (111) reflections of the TiN films. (002) and (101) peaks from the hcp $\alpha$-Ti phase of the layer deposited in a pure argon atmosphere, and (111), (110) and (200) reflections from the SS substrate can also be noticed in the XRD pattern since the X-ray penetration depth is larger than the thickness of our deposited TiN coatings (see Table 1). Figure 2b illustrates a schematic representation of the $\delta$-TiN/$\alpha$-Ti/SS304 system with the TiN grains growing in a specific direction.

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Fig. 2 a XRD patterns ($\theta$-2$\theta$ Bragg–Brentano scan) of TiN deposited on SS304 with different sputtering power ($W_s$) and b schematic representation of the $\delta$-TiN/$\alpha$-Ti/SS304 system with the TiN grains growing in a specific direction.
scheme also shows an α-Ti droplet. It has been demonstrated that α-Ti droplets can incorporate in TiN films in the solid state from the Ti target [26]. Nevertheless, to the best of our knowledge, nothing has been stated regarding the volume and distribution of α-Ti droplets contained in TiN films. These kinds of defects will be described later in this document. In the XRD pattern from Fig. 2a, it can be also noticed that the peaks from TiN are shifted toward lower diffractions angles with respect to their nominal positions. This indicates that the coating is under stress. This is a persistent observation in PVD-TiN thin films, commonly attributed to the fact that growth defects cause lattice distortion [27].

In order to estimate the degree of preferred orientation in our coatings, the texture coefficient $T_C$ has been evaluated. $T_C$ is defined as $T_C = I_200/(I_{111} + I_{200})$ and $T_C = I_{111}/(I_{111} + I_200)$ [28], where $I$ is the integrated intensity for the hkl planes. The outcomes are shown in Table 1. The (200) plane, with $T_C (200) \approx 0.8$ is the preferred orientation for all the sputtering power $W_S$ values studied here. These results demonstrate that a power increase at the cathode has only a subtle influence on the change of preferred orientation in the coatings. The surface energy of TiN is the lowest for the (001) orientation (81 meV Å$^{-2}$ for TiN (001) and 75 and 364 meV Å$^{-2}$ for the N and Ti-terminated TiN(111) surfaces [29]), which means that a (001) growth texture should develop in the first growth stages. Changes in texture upon the growth of TiN films (>1 μm thickness) have been observed in other studies and have been related to strain energy minimization, with lower-strained grains growing at the expense of those more highly strained [30, 31]. Pelleg et al. [32] and Oh and Je [33] have argued that since the biaxial elastic modulus along the (111) direction ($E_{111} = 418$ GPa) is lower than along the (002), ($E_{002} = 556$) the texture should change from (001) to (111) as the film thickness increases, in order to minimize the strain energy term. Nevertheless, in our case, even with film thicknesses >1 μm, the (002) orientation is the one preferred (see Table 1). Numerous reports in the literature underline the importance of kinetic issues in the development of a specific texture in TiN coatings [25, 27, 34–36]. In this respect, aspects such as anisotropy in adatom mobility and surface diffusion can play a decisive role. The composition of the gas mixture strongly influences the eventual crystallographic texture adopted by the TiN films. In our current study, with a used composition of N$_2$:Ar ratio of 50%, an effective dissociation of N$_2$ is expected. In these conditions, a continuous source of atomic N is available near the surface. Chemisorption N atoms will alter the diffusion of Ti, enhance the TiN surface nucleation rate and lower the chemical potential of the (100) surface, leading to a preferential growth of the [100] grains. Such atomistic processes have been previously proposed by Gall et al. [29] and Mahieu et al. [36] to explain the growth of [100] TiN grains.

Table 1 Influence of sputtering power $W_S$ on texture coefficient $T_C$, film thickness, grain size and surface root-mean-square (RMS) roughness of TiN thin films

| $W_S$ (W) | $T_C$ (200) | $T_C$ (111) | Film thickness (μm) | Grain size (nm) | RMS-AFM roughness (nm) |
|----------|------------|------------|---------------------|----------------|-----------------------|
| 100      | 0.80       | 0.20       | 1.7 ± 0.11          | 225 ± 39       | 25.2 ± 1.2            |
| 150      | 0.79       | 0.21       | 2.1 ± 0.13          | 297 ± 57       | 33.1 ± 1.1            |
| 200      | 0.83       | 0.17       | 2.9 ± 0.09          | 203 ± 74       | 23.5 ± 1.7            |

The absence of reflections of ε-Ti$_2$N or any known titanium oxide in the XRD patterns demonstrates that if present those phases are in quantities below the detection sensitivity of our technique. According to the Ti–N phase diagram, ε-Ti$_2$N forms at temperatures below 1050 °C in the range of 3 at. % N to 41 at. % N [37, 38]. Nevertheless, sputtering is a nonequilibrium process. The nonappearance of the ε-Ti$_2$N phase in our TiN films may be due to the quite low ratio $T_s/T_m \approx 0.03$ (substrate temperature $T_s \approx 100$ °C; melting temperature $T_m \approx 2949$ °C). This assumption is supported by the experiments described by Kiran et al. [3]. In [3], TiN, layers with $0.4 < x \leq 0.5$ were deposited at $T_s \approx 80$ °C with RF magnetron sputtering. XRD results only showed a pure TiN phase in the diffraction pattern. After annealing the samples at 500 °C, the ε-Ti$_2$N clearly appeared in the diffraction patterns. In that case, annealing was required (and sufficient) to form the ε-Ti$_2$N phase, stable at 500 °C in the mentioned nitrogen concentration range.

TiN Surface Structure

Figure 3 shows SEM and AFM topography images and SEM cross sectional view of the TiN samples deposited varying the sputtering power $W_S = 100$ W (a–c), 150 W (a’–c’) and 200 W (a”–c”). The RMS roughness, thickness and grain size data of all TiN film samples are given in Table 1. At the lowest power applied to the Ti target, $W_S = 100$ W (Fig. 3a–c), the TiN exhibits a columnar structure with a surface roughness of 25.2 ± 1.2 nm. Voids and boundaries throughout the film thickness have often been observed in columnar TiN films, and their formation
has been attributed to low mobility of the impinging atoms and to preferential trapping of diffusing surface atoms at low-energy orientations of already nucleated grains (atomic shadowing effect) during film growth [29, 39, 40]. It is observed that both the surface roughness and the grain size of the TiN films increase when increasing the sputtering power up to 150 W and then decrease when further increasing it to $W_s = 200$ W (see Fig. 3 and Table 1); in this latter case, the columnar film becomes thicker and denser. When increasing the sputtering power, the total energy and Ti fluxes supplied to the growing film increases [41], and as a result the mobility and migration of adsorbed atoms over the surface will be increased. For a sufficiently high sputtering power, these effects are expected to lead to films with higher packing density, more uniform grains and hence less surface roughness [39].

Nanoscale Elastic Mapping

The AFM and UFM images of the TiN film generated over the SS substrate with $W_s = 100$ W are shown in Figs. 4 and 5. Figure 4a, b were simultaneously recorded over a $(5 \times 5)$ $\mu$m$^2$ surface area. In Fig. 4a the TiN surface exhibits a protruding droplet (indicated by the arrow) surrounded by a topographically smooth and sinking area. Similar protruding droplets have been observed by SEM, being typically found randomly distributed on PVD-TiN coating surfaces [26]. The corresponding UFM image (Fig. 4b) reveals nanoscale differences in stiffness at the surface or near subsurface region of the TiN layer. Strictly, the UFM contrast is dependent on both stiffness and adhesion. Nevertheless, significant differences in surface energy of TiN grains are not expected in our films (see section “Crystallographic orientations” and “TiN surface structure”). Since a smaller Young’s modulus causes a smaller UFM response [16], the darker areas in Fig. 4b can be attributed to softer regions. Also, the influence of the topographic features on the contact stiffness (via a modification of the tip-sample contact area) must be taken into account in the analysis of the UFM contrast. To this purpose, higher resolution images were recorded over the area marked by a dotted square in Fig. 4a, b and are displayed in
Fig. 4  TiN film obtained at $W_s = 100$ W: a Topography in AFM contact mode. Surface area: $(5 \times 5)\ \mu$m$^2$; Grey-scale range: 466 nm. b UFM image simultaneously recorded with (a). c Topography in AFM contact mode recorded over the square region in (a). Surface area: $(1 \times 1)\ \mu$m$^2$ Grey-scale range: 72 nm. d UFM image simultaneously recorded with (c), over the region squared in (b). e Topographic and f elastic profile along the lines indicated in (c) and (d), respectively.

Fig. 5  a AFM topographic image. Surface area: $(500 \times 500)\ \text{nm}^2$ Grey-scale range: 58 nm. b Derivative image of (a). c UFM image simultaneously recorded with (a), over the region near to the darker grain (iii) in 4d.

Fig. 4c (AFM topography) and Fig. 4d (UFM). Figure 4e,f corresponds to the topographic and elastic profiles along the lines in Fig. 4c, d, respectively. Arrows in the images and in the profiles have been used to identify specific grains, labeled by $i$, $ii$ and $iii$ (see Fig. 4c–f). The grains type $i$ are at different heights over the surface, but nevertheless give rise to a similar UFM response. As clearly noticeable from the elastic profile in Fig. 4f, grains type $i$ appear stiffer than those at their surroundings. Grains type $ii$ display a similar contact stiffness, about 33% lower than that of the $i$ grains. Remarkably, the grain type $iii$ (Fig. 4e) exhibits a notable reduction in stiffness (78%) with respect to the type $i$ grains, and it is not possible to associate any particular feature in the topography to this UFM response. The softer TiN regions in the Fig. 4b, d are attributed to the presence of substoichiometric impurities. Sputtered
coatings often show compositional fluctuations due to variations in molecular impingement rates. Changes in the Ti:N ratio may lead to the formation of substoichiometric TiN upon the substrate surface [3, 42]. Recently, Kiran et al. [3] identified the presence of TiN$_x$ in TiN films using optical and electrical methods. Nevertheless, the presence of substoichiometric impurities is not apparent in the XRD patterns in Fig. 2a. In case TiN$_x$ is present, the appearance of TiN$_x$-related XRD peaks would be expected, since the TiN$_x$ species preserve the $\delta$-NaCl structure over a wide range of composition, $0.42 \leq x \leq 1.2$ [43]. Still, it is possible that the sensitivity XRD is insufficient to disclose small traces of TiN substoichiometric species located at or near the very surface of TiN films. On the other hand, it is well known that the chemical composition of sputtered TiN strongly influences the measured values of the Young Modulus $E$. Variations in $E$ ranging from $\frac{\sigma}{\varepsilon}$ & strongly influences the measured values of the Young 

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Grain boundary collapse may give rise to the formation of grooves such as those apparent in Fig. 5a, c. From Fig. 5c, it is also noticeable that on the grains type $i$ in Fig. 4d, the brighter contrast is due to the presence of stiffer stripes. These cannot be related to any topographic feature in Fig. 5a, b and probably originate from subsurface defects. Stiffness in these grains may be associated to the trapped impurities at the subsurface region such as oxygen and/or argon atoms might explain the differences in stiffness in these grains. Results in the literature demonstrate that such impurities may indeed be present [8, 26, 44], and they are expected to induce local lattice strain, hinder the dislocation movement, and thus enhance the local stiffness and strength.

Figure 6 shows topographic contact-mode AFM and UFM images of TiN coatings generated with $W_S = 150$ W. Here, the UFM image (Fig. 6b) also shows nanoscale elastic inhomogeneities in the TiN layer. Apparently, substoichiometric regions still form in the TiN film when the $W_S$ is increased. Nevertheless, in this case, regions with darker contrast in the UFM image (attributed to the presence of those softer substoichiometric impurities) appear in less proportion than in the coatings generated with $W_S = 100$ W (Fig. 4b). Higher resolution images ($\mu m^2$) of the area marked by a dotted square in Fig. 6a, b are displayed in Fig. 6c (AFM), Fig. 6d (D-AFM) and Fig. 6e (UFM). No feature related to the UFM contrast in Fig. 6e is apparent from Fig. 6c or Fig. 6d, which allows us to discard any topographic influence. The UFM image in Fig. 6e also shows a TiN structure with stiffer grooves within some grains (see the corresponding encircled area in Fig. 6c–e).

Figure 7 shows topographic contact-mode AFM (Fig. 7a) and simultaneously recorded UFM (Fig. 7b) images of TiN coatings generated with $W_S = 200$ W. As can be seen, a further increase in the sputtering power up to 200 W generates a more elastically homogeneous surface. Here, softer UFM regions (some marked with arrows in Fig. 7a, b) appear in less proportion than in the cases of TiN coatings produced with less sputtering power. As mentioned earlier, the increase in $W_S$ from 100 to 200 W increases the total energy and Ti fluxes supplied to the growing film. Hence, the formation of substoichiometric and defective regions is expected to decrease, since the availability of the species and their mobility increases. As a result, the surface coverage will be more effective.

Conclusions

In this work, UFM has been applied to nanoscale elastic mapping of PVD-TiN coatings with a lateral resolution of $\approx 5$ nm.
The UFM image contrast lateral reveals nanoscale inhomogeneities in stiffness on the TiN films prepared with different sputtering power. Those have been explained as due to the presence of softer substoichiometric TiN and/or trapped subsurface gas within the films.

According to XRD analysis, the TiN coatings preferentially grow in the (200) orientation, even though some TiN grains exhibit a (111) orientation. The presence of substoichiometric TiN phases or titanium oxides is not evident from the XRD data. When increasing the sputtering power, the TiN coatings become thicker, denser, flatter, and—according to the UFM study—more elastically homogenous. These characteristics have been attributed to a higher availability and enhanced surface/bulk diffusivity of Ti and N species.

The UFM data provide evidence of surface tensions related to grain boundaries collapse and subsequent formation of grooves generated because of grain coarsening during grain growth and film thickening.

In service operation of engineering elements coated with PVD-TiN films, the presence of impurities and structural defects that give rise to elastic discontinuities leads to detriment of the mechanical properties and of the protection against corrosion. Nanoscale elastic mapping of nanostructured hard coatings can be used for indentifying weak structural regions, and constitutes a novel tool of high value for the improvement of quality and design of thin films.

**Acknowledgments** Funding from the National Science and Technology Council of Mexico (CONACYT) and the Junta de Castilla-La Mancha (JCCM) in Spain, under grant 004Eo.38467U and project PCI-08-0092 respectively, are gratefully acknowledged. J. A. H thanks the National Science and Technology Council of Mexico, CONACYT for financial support for a three-month stay in the Laboratory of Nanotechnology in Almadén, Spain.
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