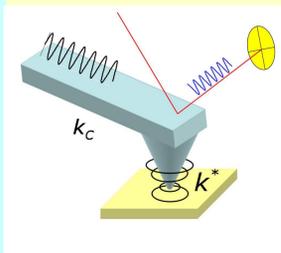


Introduction

In the last few years, various scanning probe microscopy based techniques have been developed to accurately interrogate the mechanical response of materials at the nanoscale. Such measurements are critical for mechanical characterization of discrete and integrated nanoscale building blocks of high-performance electronics. In this work, by using contact-resonance atomic force microscopy (CR-AFM), nanoscale contact stiffness measurements were performed on TiN films and TiN/interlayer dielectric (ILD) structures. From these measurements, the elastic moduli of thick TiN films were determined by using the Hertz contact model. In the case of TiN/ILD structures, a significant reduction in the elastic modulus of TiN was found. Combined CR-AFM measurements and finite element modeling (FEM) calculations were performed for TiN/ILD layered structures.

Contact Resonance AFM



The CR-AFM technique relies on the sensitivity of the resonance frequency of the AFM cantilever to the elastic properties of the material indented. A clamped-spring coupled beam model is used to convert the measured frequencies into contact stiffnesses.

$$k^*/k_c = F(f_{res}^{contact} / f_{res}^{air}).$$

The contact stiffness is determined by the contact geometry and elastic properties of the material indented:

$$f^{ref} < f^{test}$$

$$k^* = 2aE^*$$

$$M = E/(1-\nu^2)$$

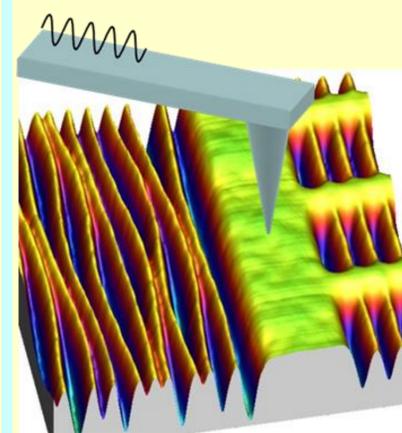
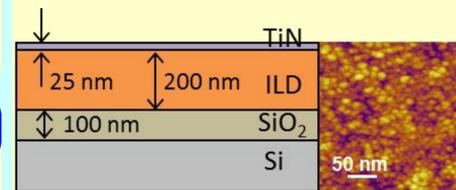
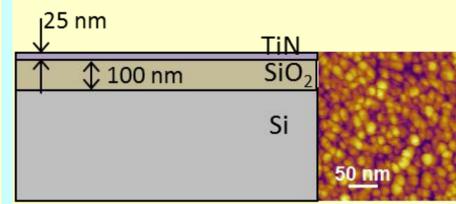
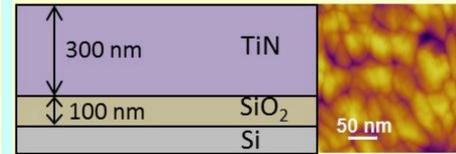
$$1/E^* = 1/M_s + 1/M_T$$

The CR-AFM measurement accuracy is greatly enhanced when measurements are made alternatively on the test material and a reference material with known elastic modulus, so the elastic modulus of the test material is determined with respect to that of the reference material.

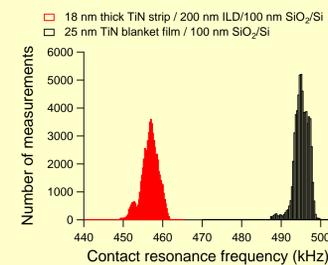
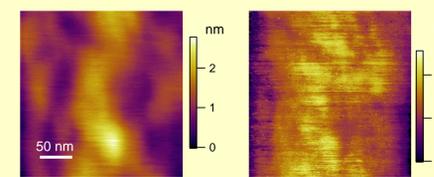
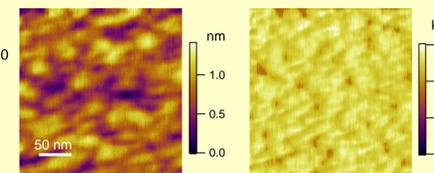
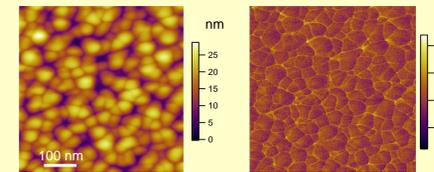
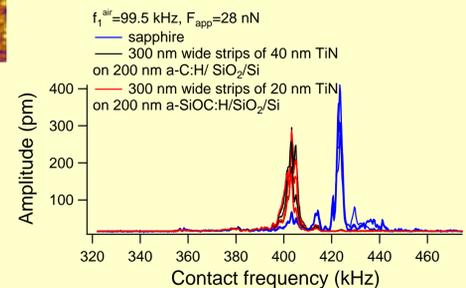
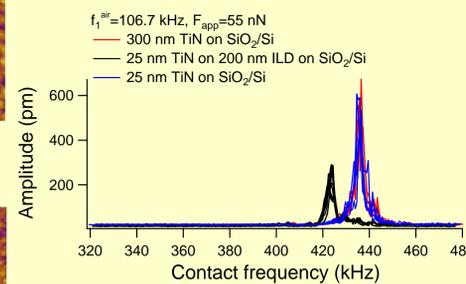
$$E_s^* = E_R^* |k_s^*/k_R^*|^{3/2}$$

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Elastic measurements on TiN blanket films and strips



Point contact-resonance AFM measurements were performed successively on TiN blanket films deposited on various substrates: 300 nm thick TiN on SiO₂/Si, 25 nm thick TiN on SiO₂/Si and 25 nm thick TiN on ILD/SiO₂/Si. While no significant difference was observed in the contact resonance frequency on TiN films on SiO₂/Si substrates, a smaller frequency shift was measured on TiN on ILD, suggesting a more compliant material in this last case.

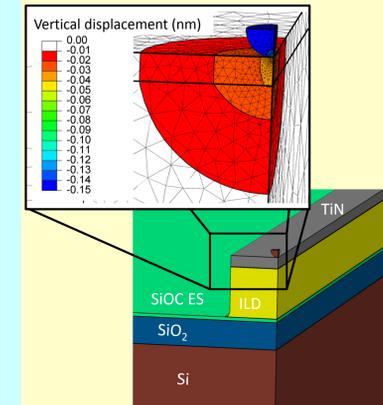


With either sapphire or 300 nm thick TiN/SiO₂/Si as references, CR-AFM measurements were performed on 300 nm wide strip TiN/ILD/SiO₂/Si layered structures. The ILD layer was either a-SiOC:H or a-C:H.

Similar shifts in the contact resonance frequency were measured on TiN blanket films and strips deposited on ILDs and both of them were consistently less than on TiN/SiO₂/Si films. In the Hertz contact model approximation, the corresponding reduction for the indentation modulus is by a factor of 4.

Sample structure	Sample size	Reference	M _{reference} (GPa)	M (GPa)
300 nm TiN/ 100 nm SiO ₂ /Si	blanket film	Si(100)	164.8	408 17
25 nm TiN/100 nm SiO ₂ /Si	blanket film	sapphire	466.5	440 20
25 nm TiN/200 nm ILD/100 nm SiO ₂ /Si	blanket film	300 nm TiN/SiO ₂ /Si	440 20	116 16
40 nm TiN/ 200 nm a-C:H/ 100 nm SiO ₂ /Si	300 nm wide strip	sapphire	466.5	108 5
18 nm TiN/200 nm a-SiOC:H/ 100 nm SiO ₂ /Si	300 nm wide strip	sapphire	466.5	109 5

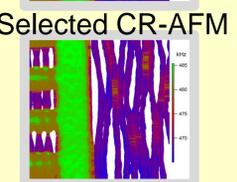
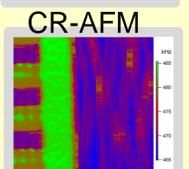
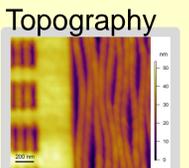
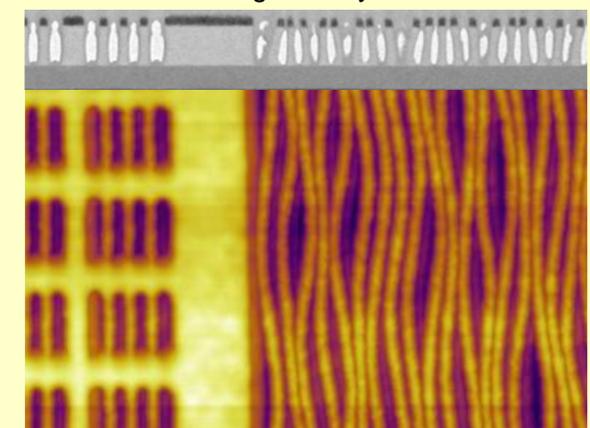
Combined FEM and CR-AFM



To consider the ILD contribution to CR-AFM measurements, FEM that mimicked the measurement conditions (tip radius, indented layered structure, applied force) was performed. With the elastic moduli ratio determined from a buckling analysis for each sample, the indentation modulus of TiN was adjusted until the simulated contact stiffness matched the measured value.

Sample	f _c (kHz) CR-AFM	E _{TiN} /E _{ILD} buckling	E _{TiN} (GPa) FEM	E _{ILD} (GPa) FEM	E _{ILD} (GPa) PeakForce-AFM
sapphire	422.9 ± 0.5	-	-	-	-
18 nm TiN/ 200 nm a-SiOC:H/ 100 nm SiO ₂ /Si	401.5 ± 0.3	23.1	230 9	5.3	5.9 0.9
40 nm TiN/ 200 nm a-C:H/ 100 nm SiO ₂ /Si	402.4 ± 0.6	25.3	155 10	3.9	-

CR-AFM mapping was performed over 40 nm narrow strips adjacent to a 300 nm wide strip to observe contact geometry effects.



Conclusions

- The elastic modulus of TiN blanket films and strips was found to be sensitive to substrate material: a more compliant TiN was measured on ILD substrates than on SiO₂ substrates.
- A methodology consisting of combined FEM calculations and CR-AFM measurements was used to separate the mechanical contributions of stiff TiN deposited on compliant ILD materials.