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1 **Thin-film Adhesion: A Comparative Study Between Colored Picosecond Acoustics and**
2 **Spontaneous Buckles Analysis**

3

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9

10 **Abstract**

11 This paper presents some quantitative measurements of the adhesion energy of thin WTi films
12 deposited on Si substrate. Two different techniques are applied to the same sample series. One
13 is a mechanical test based on the analysis of spontaneously formed defects. The second is based
14 on acoustic waves whose reflection at the interface between the thin-film and the substrate is
15 sensitive to the adhesion. An excellent correlation is obtained between both approaches: the
16 adhesion energy measured by buckles analysis and acoustic reflection coefficient measured by
17 picosecond acoustics. The acoustic approach offers several advantages among which a non-
18 destructive character, a compatibility with complex stacks and a sensitivity to detect adhesion
19 anomaly even if no defect is formed.

20

21 **Keywords:** adhesion; thin film; buckles; reflection coefficient; picosecond acoustics.

22

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1 **1. Introduction**

2 Industry and especially microelectronics push towards the nanoscale by fabricating devices
3 based on more and more complex stacks of thin films in which are mixed heterogeneous
4 materials. For example, a radio-frequency filter in a mobile phone is built on several resonators
5 each of them being made of more than 15 layers stacked together. Metals, dielectrics and
6 semiconductors are mixed together in a very complex multilayer and adhesion becomes a
7 crucial issue for device reliability [1]-[4].

8 Numerous methods have been proposed to test adhesion [5]-[7] (either in a qualitative manner
9 (yes/no test, like tape test [8]) or quantitatively by measuring an adhesion energy at the specific
10 interface, for example scratch testing [9][10], four point bend testing [11][12] stressed
11 overlayers [13]-[15], and nanoindentation [16]-[18]. While these approaches are well-known
12 and successfully measure adhesion energies, a requirement of all of the methods is delamination
13 of the interface must occur. Thus, the methods are destructive and not always compatible with
14 complex material stacks. For the four point bending method, multi-layer stacks can be
15 evaluated, but require a sandwich sample geometry that can be difficult to make and only the
16 weakest interface can be measured. Stressed overlayers are often limited as well and can be
17 combined with nanoindentation and scratch methods [18]. Nanoindentation and scratch
18 methods require access to proper instrumentation and also is somewhat a trial and error process
19 to induce delamination of the desired interface. A non-destructive method that can also be used
20 to quantify the adhesion energy of multiple interfaces would be useful for hard to delaminate
21 interfaces.

22 Acoustic waves can also be used to investigate adhesion and, more importantly, in a non-
23 destructive manner. For more than 30 years, picosecond acoustics (PA) has opened the field of
24 thin and ultra-thin layers to acoustics [19]. Similar to a sonar but at the nanoscale and based on
25 ultrafast laser pulses, PA has found many applications especially to the thickness control of

1 complex stacks [20][21]. It has also been proposed as a tool for detecting adhesion defects [22].
2 Indeed, a poor adhesion affects the reflection of acoustic waves at the concerned interface so
3 that by measuring the reflection coefficient one can rank samples or compare places along the
4 sample surface from the adhesion point of view.

5 To go further one needs to have a direct comparison between the acoustic adhesion number
6 (namely the reflection coefficient) and the adhesion energy measured with a mechanical model.
7 In this paper we apply two much different adhesion techniques to a same set of samples: one is
8 a variant of PA designed in the following as Colored Picosecond Acoustics (APiC) [23]; one is
9 a mechanical adhesion measurement based on an analysis of buckles defects that appear
10 spontaneously on the sample surface. From such a comparison, we demonstrate that APiC
11 technique can provide semi-quantitative adhesion measurements that are local and non-
12 destructive.

13

14 **2. Experimental details**

15 2.1. Samples description

16 The study is performed on a series of three similar WTi films with approximately 20% Ti
17 content and that were sputter deposited using a single WTi target in industrial sputtering
18 machine to a thickness of 250 nm. The films were sputtered onto Si substrates with a native
19 SiO₂ oxide of a few nanometers (approximately 2-5 nm). Due to the high compressive residual
20 stress, all films spontaneously delaminated forming the well-known telephone cord buckle
21 shape. Such defects are used to deduce the adhesion energy of the WTi layer on the Si substrate
22 following the pioneered work of Hutchinson and Suo [24].

23

24 2.2. Measuring adhesion energy using buckles

1 Confocal laser scanning microscopy (CLSM, Olympus LEXT 4100 OLS) was used to image
 2 the buckles for further analysis. CLSM uses a laser and precise stage movement to create laser
 3 intensity images, quantitative height images, and focused resolved optical light images. From
 4 the height images, (Figure 1 a-c) the buckle heights, δ , and buckle widths, $2b$, can be measured
 5 (Figure 1d). The buckle dimensions with the film thickness, h , and elastic properties (Young's
 6 Modulus E , Poisson ratio ν) of the film were then used to calculate the critical buckling stress,
 7 σ_b , and the driving stress, σ_d (Eqn. 1 and 2) following the model of Hutchinson and Suo [24],

$$8 \quad \sigma_b = \frac{\pi^2 E}{12(1-\nu^2)} \left(\frac{h}{b}\right)^2 \quad (1)$$

$$11 \quad \sigma_d = \sigma_b \left[\frac{3}{4} \left(\frac{\delta}{h}\right)^2 + 1 \right]. \quad (2)$$

12
 13 For WTi, $E = 322$ GPa was measured using nanoindentation with a Berkovich tip ($R = 150$
 14 nm) and $\nu = 0.288$ was determined with a rule of mixtures [25]. Indents were performed
 15 using the full available load range of a Hysitron TriboScope Nanoindenter, namely $100 \mu\text{N}$ to
 16 $10,000 \mu\text{N}$, which corresponds to depths between 50 nm and 450 nm. Then the measured
 17 stiffness versus the contact depths were plotted and described with the model from Li and
 18 Vlassak [26] to reach the 322 GPa elastic modulus for the WTi. From the calculated stresses,
 19 the mixed mode adhesion energy, $\Gamma(\Psi)$, was determined with Eqn. 3,

$$20 \quad \Gamma(\Psi) = \left[\frac{(1-\nu^2)h}{2E} \right] (\sigma_d - \sigma_b)(\sigma_d + 3\sigma_b), \quad (3)$$

21 where Ψ is the phase angle of loading.

22 It should be noted that the Hutchinson and Suo model is for straight-sided buckles and not
 23 telephone cord shaped buckles. While several groups have provided new adhesion models for
 24 telephone cord buckles [12] [27] [28], it is still acceptable to use the Hutchinson and Suo model
 25 for telephone cord buckles, especially when the buckle dimensions are measured at the point of
 26 inflection as demonstrated in Figure 1b. At this point the buckle cross-section is symmetric and
 27 can be modeled as a straight-sided buckle [28].

1

2 2.3. APiC experimental details

3 Basically, APiC is an ultrafast laser technology that implements a pulse-echo technique at the
 4 nanoscale [29]. The light absorption of a first laser light pulse (the pump) leads to the emission
 5 of a short acoustic pulse. It propagates in the film at the sound velocity and is partially reflected
 6 toward the surface when it reaches the film/substrate interface. The returning echo is optically
 7 detected using another laser pulse (the probe) time-delayed with respect to the pump pulse.

8 By detecting successive acoustic echoes, one can first measure the time-of-flight and deduce
 9 the film thickness from the longitudinal sound velocity. A schematic view of the experimental
 10 setup is given in Fig. 2. From the successive echo amplitude, one extracts the acoustic reflection
 11 coefficient. The portion of the strain pulse that is reflected at an interface is governed by the
 12 ratio between the acoustic impedances of both materials. The acoustic impedance of a given
 13 material (Z) is the product of the mass density by the sound velocity. And when an acoustic
 14 wave reaches an interface, the expected reflection coefficient is given by:

$$15 \quad R = \frac{(Z_2 - Z_1)}{(Z_2 + Z_1)} \quad (4)$$

16 where R is the reflection coefficient, and Z_1 and Z_2 are the respective acoustic impedances of
 17 the two materials [22].

18 In the present case, the WTi material has a very high acoustic impedance compared to silicon
 19 which means that a significant part of the acoustic pulse is reflected at the interface with the
 20 substrate. Assuming a sound velocity of 5350 m/s and a mass density of 15.3 g.cm⁻³, one obtains
 21 $Z_1 = 81.9 \cdot 10^6$ kg.m⁻².s⁻¹ and $R = -0,61$ at the interface with a Si substrate ($Z_2 = 19.7 \cdot 10^6$ kg.m⁻².s⁻¹).

22 The negative sign is related to the fact that the second medium has a lower impedance than the
 23 first one. In the following we ignore the sign of R and only focus on its magnitude $|R|$. The
 24 poorer the adhesion is, the higher $|R|$ will be. The extreme case is a total delamination, then the
 25 degree of reflection is close to -1.

1 Experimentally, APiC measurements were carried on using a commercial tunable Ti:Sapphire
2 oscillator and a conventional pump and probe scheme at normal incidence. The laser produces
3 120 fs optical pulses at a repetition rate of 80 MHz tunable between 690 and 1050 nm [30].
4 Using second harmonic generation in a β -BaB₂O₄ crystal, blue laser beam is generated to serve
5 as a probe which is time-delayed with respect to the pump using a mechanical delay line. Both
6 pump and probe beams are focused on the same point at the sample surface using a microscope
7 objective x20. The ratio between the pump and probe intensities is close to 1000:1. To improve
8 the signal-to-noise ratio, the pump beam is chopped using an acousto-optic modulator and the
9 output of the photodiode, which monitors the reflected probe, is amplified through a lock-in
10 scheme.

11 The focused spot size at the sample surface is close to 1-2 μ m in diameter which means that
12 APiC measurement is made locally. The sample is fixed on a 100 mm XY translation stage
13 which offers the opportunity to control the place where the measurement is performed. This
14 way complete mapping the sample response can be obtained with a XY resolution better than
15 0.5 μ m along surfaces as large as 100 mm x 100 mm.

16

17 **3. Results and discussion**

18 3.1. Adhesion from defect analysis results

19 In order to evaluate the interface adhesion energy, at least 30 measurements of buckles were
20 made on each WTi sample. Table I summarizes the adhesion results and illustrates that Sample
21 WTi-3 had a slightly higher adhesion compared to Samples WTi-1 and WTi-2. The values are
22 also similar to previous studies of WTi films on Si and dielectric films [31] [32]. The
23 differences in the mixed mode adhesion energies between the three samples can be considered
24 small. WTi-1 and WTi-2 are quite close and when the standard deviations are taken into
25 account, all values overlap. A possible reason for WTi-3 to have a slightly larger adhesion

1 energy is that this sample came from a different radius on the wafer, for example closer to the
2 wafer edge, where the average buckle dimensions (Table I) are slightly smaller, leading to the
3 higher adhesion energies.

4

5 3.2. APiC results

6 Figure 2 presents the transient reflectivity measured on the three WTi samples. A sudden
7 change in the reflectivity is visible at $t = 0$. At such a time-delay, the pump laser is exciting the
8 thin metallic film. Light is first absorbed by electrons in the metal which leads to strong change
9 in the optical reflectivity. About 1 ps later photoexcited electrons go back to their original states
10 transferring the excess of energy to the lattice as heat and the reflectivity goes down. The very
11 sharp peak around $t=0$ is thus the electronic response of the metal film. As the sample is heated
12 it reflects light differently and a step is detected between $t<0$ and $t>0$. This is the thermal
13 response of the sample which slowly decreases as the sample cools down. The acoustic
14 contribution is clearly visible here as a series of sharp signals about every 100 ps superimposed
15 to the thermal decrease. Each of them is an acoustic echo detected at the WTi surface and
16 corresponds to a certain number of round-trips in the WTi film. From the precise time-delay
17 and from the sound velocity of WTi (5350 m/s) the thickness is deduced. As visible in Fig.2,
18 sample WTi-2 is found to be slightly thicker than others (about 5%).

19 From the same data, the amplitude of the successive echoes can be analyzed to extract the
20 acoustic reflection coefficient. The acoustic reflection coefficient is obtained through the
21 exponential decay of the successive echoes. In the present case the reflection coefficient is
22 obtained using the 3 first echoes.

23 APiC performs a very local measurement as the laser spot size is in the range 1-2 μm . To have
24 a more global picture of each sample, it is reproduced at more than 500 points along the sample
25 surface, but only at unbuckled places. This way a statistic is produced of both film thickness

1 and reflection coefficient. Mean value and standard deviation of thickness and reflection
2 coefficient are given in Table 1.

3 If the film thickness is found not to vary from one point to another (deviation less than 2 nm
4 i.e. less than 0.8%), significant variations are observed on the reflection coefficient (from 7 to
5 14%). This conclusion is more visible in Fig. 3 where the distribution of the reflection
6 coefficient is compared for the three samples. Sample WTi-1 presents a sharp distribution
7 centered around a quite high value (0.55). Sample WTi-2 presents a slightly larger distribution
8 centered around a lower value (0.53). That suggests a better adhesion compared to WTi-1. The
9 most interesting result is obtained on sample WTi-3 on which a very large distribution is
10 obtained. That corresponds to the combination of two distributions: one is centered around low
11 values (typ. 0.44) and a second centered around 0.52. From that we suspect that sample WTi-3
12 presents various places along its surface, some of them being well adherent to the substrate and
13 others comparable to other samples.

14

15 3.3. Correlation between buckles analysis and APiC

16 We now confront the two techniques results. This is done graphically in Fig.4 where the
17 adhesion energy measured using telephone cord buckles is plotted as a function of the reflection
18 coefficient measured by APiC. The mean values are well-aligned along a line that represents
19 the expected tendency: as explained before, the higher the adhesion energy is, the lower the
20 acoustic reflection is expected to be at the interface.

21 Error bars indicate the deviations obtained using both techniques. It's important to note that an
22 error here does not only corresponds to a measurement error but also the variation of the
23 measured quantity along the surface. This is true for both techniques. For buckles analysis,
24 more than 30 defects are analyzed per sample to extract the adhesion energy. For APiC,
25 reflection coefficient is measured at various places at the sample surface. That way two

1 contrasted regions from the adhesion point of view have been identified on sample WTi-3 which
2 gives a large deviation and error bar. But an important difference between both techniques is
3 that to extract adhesion energy from buckles, the zone must have delaminated there. At a good
4 place, where no delamination occurs, no defect is visible and adhesion energy cannot be
5 extracted following the Hutshinson and Suo method. On the contrary, APiC is applied where
6 the film is still adherent to the substrate and can thus probe well and poorly bonded regions.
7 For that reason, the buckle technique cannot confirm the well bonded region identified on
8 sample WTi-3.

9 In order to determine if such a place is or not a perfect bonded zone, one may compare the
10 reflection coefficient to the theoretical value expected for a perfect WTi/Si interface. One notes
11 that measured reflection coefficients are significantly lower than expected from Eq. (4). This is
12 due to acoustic attenuation that affects the strain pulse amplitude during its propagation in the
13 WTi layer. The role of attenuation is confirmed by the broadening of the echo as its number
14 increases as attenuation preferably affects the high frequency content of the acoustic pulse.
15 Echo amplitude thus decreases for two main mechanism: transmission of acoustic energy to the
16 substrate at the interface WTi/Si and attenuation during propagation in WTi. Both effects must
17 be considered in a numerical modeling of the acoustic signal to compare quantitatively the
18 measured reflection coefficient to the theoretical value. Doing that for WTi on Si and assuming
19 a standard attenuation value for a metal (exact attenuation in the WTi alloy is not perfectly
20 known), one shows that the reflection coefficient must be corrected by a factor 1.3 to decorrelate
21 the attenuation effect. A reflection coefficient of 0.43 ± 0.05 then corresponds to reflection
22 coefficient of 0.56 ± 0.06 , a value close to the theoretical value (0.61) that supports a perfect
23 bonding there.

24

25 **5. Conclusion**

1 We presented a comparative study between two much different techniques able to test adhesion
2 of a thin film on its substrate. First adhesion energy is deduced from an analysis of telephone
3 cord buckle defects that appear spontaneously at the sample surface as the metal film is highly
4 stressed. Second, we use ultra-high acoustic waves emitted and detected by femtosecond laser
5 to measure their reflection at the concerned interface. An excellent correlation is obtained
6 between results obtained on a series of three similar samples. This work confirms that acoustic
7 waves may provide an alternative approach to measure the adhesion of thin films. The APiC
8 technique on which such acoustic measurements are based, is a full optical measurement that
9 means with no contact and no destruction of the sample. One may also point that its time-
10 resolved character let it identify the successive interfaces when the layer is stacked in a complex
11 ensemble. As demonstrated here, the APiC technique is very local, the zone to be tested being
12 the focused laser spot size. Thanks to that, adhesion can be compared from one place to another.
13 As shown here, APiC can also access to adhesion at places where no defect exists. An anomaly
14 on acoustic reflection can be detected well before a delamination occurs.

15

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Figure captions

Fig. 1. Representative CLSM height images of three WTi films: (a) WTi-1, (b) WTi-2, and (c) WTi-3. (d) Example of buckle measurements, δ and $2b$, of the white line marked in (b).

Fig. 1. Transient response of the 3 WTi samples sputtered on a Si substrate. A series of short acoustic echoes is easily detected. From that we extract the film thickness and the acoustic reflection coefficient at the interface between WTi and Si.

Fig. 2. Statistics of the reflection coefficient (absolute value) measured on each WTi sample at more than 500 points.

Fig. 3. Correlation between adhesion energy measured through buckles analysis and acoustic reflection coefficient measured using picosecond acoustics. The dash line shows the expected tendency for a perfect correlation, the higher adhesion energy is the lower the reflection coefficient is expected to be.

1 **TABLE 1**

2
3 Table I: Summary of buckle dimensions, evaluated buckle stress and adhesion energies using
4 the spontaneous telephone cord buckles and a thickness of 250 nm. Additionally, the measured
5 thicknesses and the acoustic reflection coefficient using APiC.
6

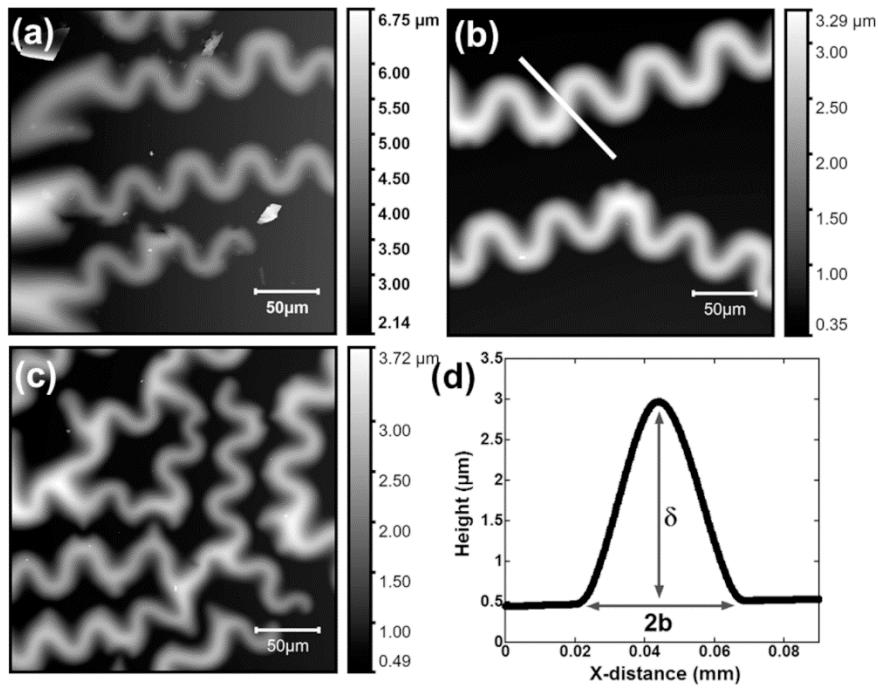
Sample	Ave. Half buckle width, b (μm)	Ave. Buckle Height, δ (μm)	Buckle Stress, σ_b (MPa)	Mixed Mode Adhesion Energy, Γ(Ψ) Jm⁻²	Thickness from APiC, h (nm)	Reflection Coefficient
WTi-1	18.5 ± 2.5	1.93 ± 0.3	55.6 ± 14.5	2.23 ± 0.56	256.8 ± 2.0	0.55 ± 0.04
WTi-2	20.5 ± 2.1	2.22 ± 0.2	43.1 ± 09.0	2.35 ± 0.18	270.2 ± 1.8	0.53 ± 0.05
WTi-3	15.4 ± 1.8	1.70 ± 0.2	79.7 ± 20.1	2.84 ± 0.53	259.5 ± 1.4	0.49 ± 0.07

7

1 FIGURE 1

2

3



4

5

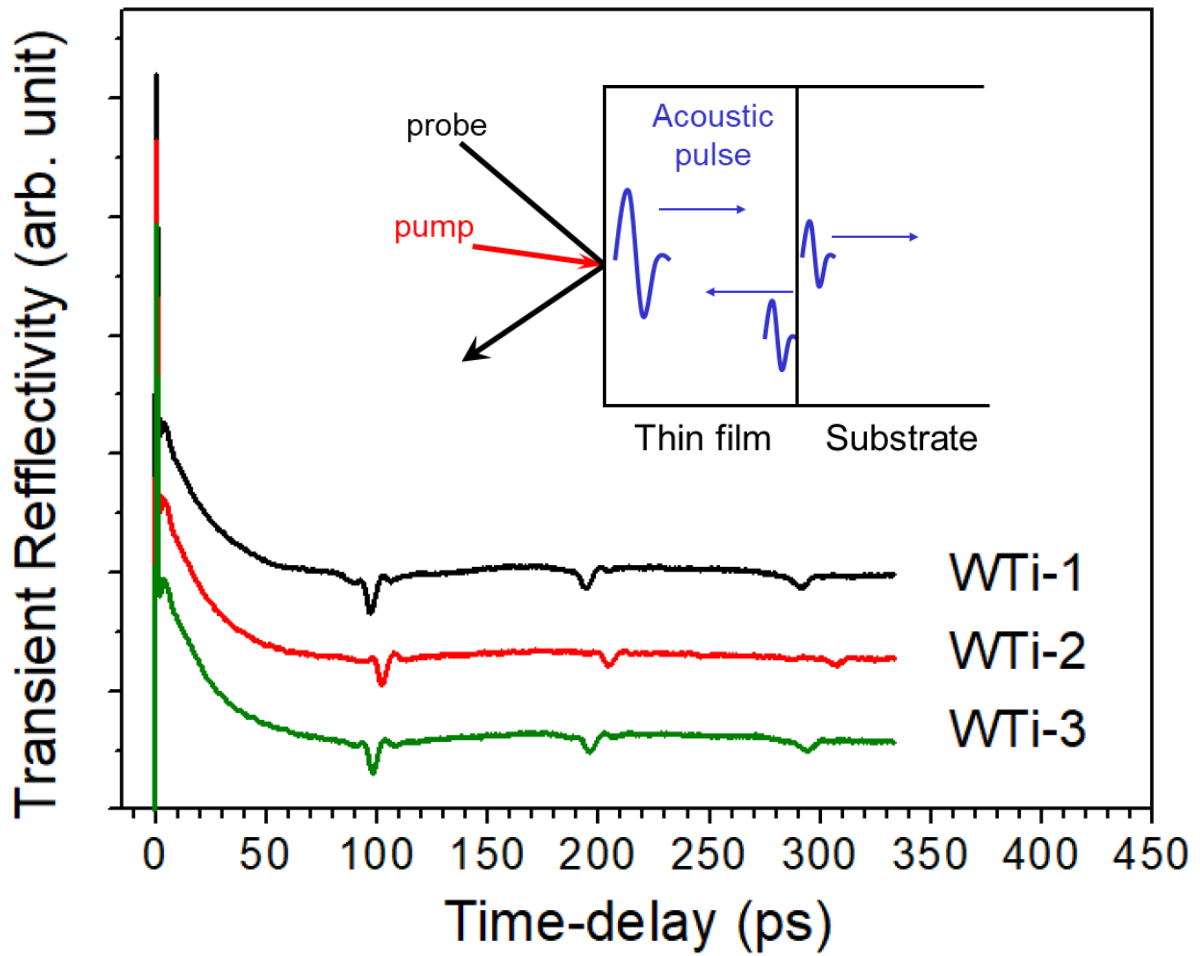
6 Fig. 1. Representative CLSM height images of three WTi films: (a) WTi-1, (b) WTi-2, and (c)

7 WTi-3. (d) Example of buckle measurements, δ and $2b$, of the white line marked in (b).

8

1 **FIGURE 2**

2



3

4

5 Fig. 2 Transient response of the 3 WTi samples sputtered on a Si substrate. A series of short

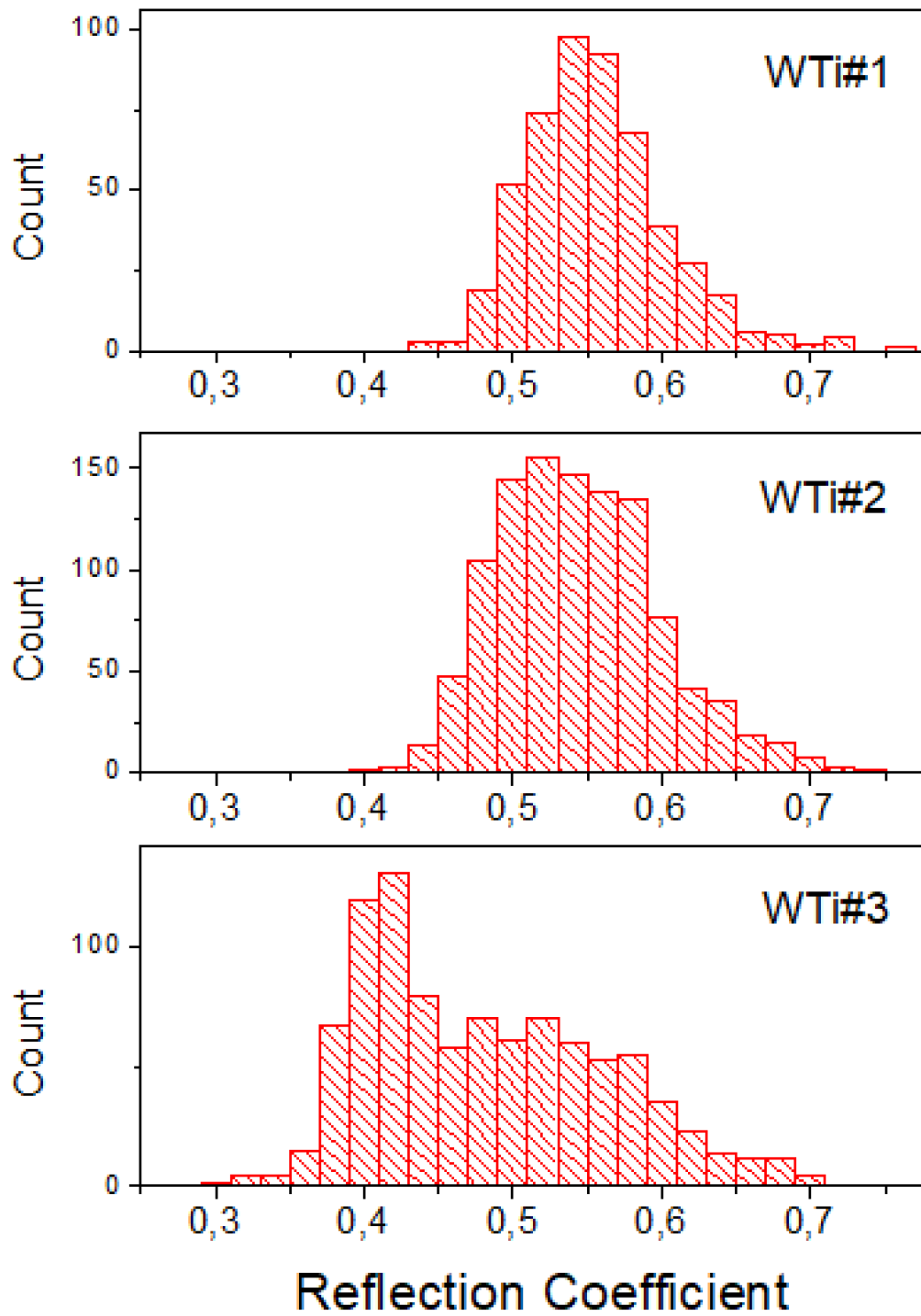
6 acoustic echoes is easily detected. From that we extract the film thickness and the acoustic

7 reflection coefficient at the interface between WTi and Si.

8

1 **FIGURE 3**

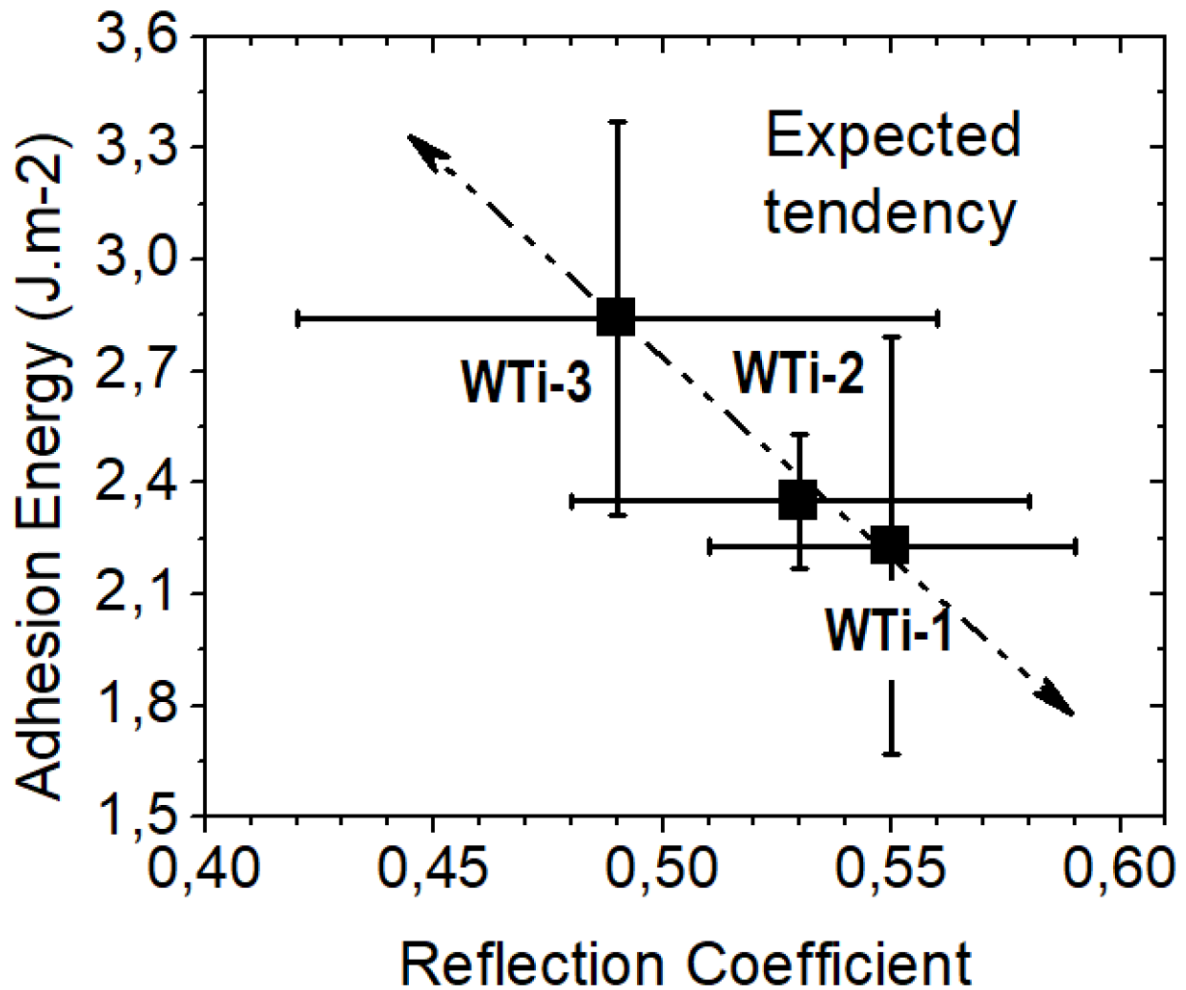
2



3

4

5 Fig. 3 Statistics of the reflection coefficient (absolute value) measured on each WTi sample at
6 more than 500 points.

1 **FIGURE 4**2
34
5

6 Fig. 4 Correlation between adhesion energy measured through buckles analysis and acoustic
 7 reflection coefficient measured using picosecond acoustics. The dash line shows the expected
 8 tendency for a perfect correlation, the higher adhesion energy is the lower the reflection
 9 coefficient is expected to be.

10