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Picosecond Acoustics Versus Tape Adhesion Test: Confrontation on a Series of Similar Samples With a Variable Adhesion

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Abstract

A large number of methods have been developed to characterize the adhesion of a thin film to a substrate. Among them, tape test is still one of the most popular methods. Acoustic waves and especially ultra-high frequency acoustic waves are also sensitive to adhesion defects as they affect the way acoustic waves are reflected at such an interface. In this work, we first prepare a series of identical thin-film samples with a variable adhesion at the interface between the film and its substrate. To modulate adhesion of some samples, an ultrathin gold layer is deposited on the substrate before thin-film sputtering. Si+ ion implantation is also used to reinforce locally gold adhesion. The samples are then characterized using two much different techniques: picosecond acoustics (PA) for measuring thickness and acoustic reflection coefficient (R) at the interface and tape test to have an independent evaluation of the adhesion through the peeled area (P). On the samples series, R is found to vary from -0.46 the expected value for a perfectly bonded Ni/Si interface to almost -0.83 not far from -1 which corresponds to delamination. An excellent correlation is found between R and P: high |R| value samples are easily peeled off and P is large. That work demonstrates the capability of PA to perform adhesion test in a totally non-destructive manner and furthermore locally.

Keywords: adhesion; thin film; reflection coefficient; picosecond acoustics.

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1. Introduction

Thin-film adhesion is a critical issue for any high technology industry. Many devices like mobile phone, solar cell or space mirrors are based on more and more complex stacks of thinner and thinner layers. Various materials are mixed together so that the cohesion of the whole is not always ensured. To be able to control adhesion at such a scale is a real challenge. A large number of methods have been developed to characterize the adhesion of a thin film to a substrate[1][2][3]. Following Mittal, one can divide the adhesion techniques in three categories: qualitative, quantitative and semi-quantitative[2]. In a qualitative method the measurement gives a go/no go type of result. Among such methods, tape test is still one of the most popular methods[4][5][6][7][8]. A quantitative method provides the value of the adhesion energy in J/m². Finally in the semi-quantitative class one finds all the techniques that provide a number related to adhesion but which is not the adhesion energy itself. This is the case of acoustic techniques for which the number is the acoustic reflection coefficient. Acoustic waves and especially ultra-high frequency acoustic waves are also sensitive to adhesion defects as they affect the way acoustic waves are reflected at the concerned interface. By scanning the acoustic reflection coefficient along the sample surface, such waves can be used to detect interface defects. As layers become thinner and thinner, higher and higher frequency acoustic waves are needed to keep a good sensitivity to interface defects.

Picosecond Acoustics (PA) that uses a femtosecond laser to excite and detect ultra-short acoustic pulses, is the perfect technique for making physical acoustic studies at the nanoscale: thickness, elasticity, hypersound attenuation, etc[9][10][10][12][12][13][14]. As a full optical technique, PA is a totally nondestructive method. As for any acoustic technique, PA is also sensitive to adhesion through the measurement of the acoustic reflection coefficient at the interface. A few published reports have shown such a capability. Tas et al. used PA to map the adhesion changes in a thin gold layer induced by ion implantation[15]. Devos et al. show that

PA can also be applied to stacks as complex as a high reflectivity mirror made of more than 50 layers to detect adhesion issues at a deeply buried interface[16].

In this work, we demonstrate that PA can be used to quantify adhesion at an interface between a thin-film and its substrate For that, we first elaborate a series of similar thin-film samples but differing by the adhesion at the interface with the substrate. PA is first used to characterize the magnitude of the acoustic reflection at such an interface. The series of sample reveals a large variation of the reflection coefficient suggesting that some samples are perfectly bonded whereas others are about to be delaminated. Such a result is then confirmed by a destructive test based on peeling by a tape test. Using four different strengths, we find a perfect correlation between acoustic results and behaviors under peeling. Finally local character measurement of PA is demonstrated on a sample prepared in such a way that a part of the film is well bonded to the substrate whereas the other is very weak.

2. Experimental details

2.1. Samples description

An important point is that both measurement methods can be applied to the same samples: first PA which is nondestructive then tape test. Each sample must be compatible with both methods with a well-controlled, contrasted and reproducible adhesion. Layers of a few hundreds of nanometers are preferred to limit the impact of acoustic attenuation which can be very strong in the hypersonic range. It is also important to prepare samples with dimension in the order of a few centimeters as four different tapes must be sticked on each sample.

Samples are made of a 200 nm layer of either nickel or a nickel/chromium alloy (90% Ni and 10% Cr) sputtered onto a (100) silicon wafer. For some samples and in order to weaken the adhesion, a 20 nm gold layer is deposited on silicon before the metal deposition. Indeed, gold is known to be poorly adherent to silicon[15]. The gold layer is chosen to be very thin compare to the metal layer so that it can be seen as a fragile interface layer. Adhesion of a thin-film is also dependent on the deposition technique. For that reason we prepare the gold layer using either evaporation or sputtering.

Table 2 presents the details for the 8 samples. Samples (#1 & #2) are made of a thin pure Ni layer deposited on Si. All the other samples have a gold layer in between metal and the substrate. Deposition technique used for gold is designated as a E for Evaporation or S for Sputtering.

Another good reason to choose gold as a fragile layer is that its adhesion to silicon can be enhanced by ion implantation[17][18][19]. A last sample labelled #I is similar to others but is made in such a way that adhesion is expected to be very contrasted between two regions. A gold film is first evaporated onto a Si wafer. Then the sample is implanted with 110 keV Si^+ ions with a dose of 10^{16} ions/cm² but a part of the sample is protected by a mask. Finally, a NiCr film is sputtered over the gold. The resulting sample is made of the same layers but with an important variation on its adhesion along its surface.

2.2. Picosecond acoustic setup

In this work, the picosecond acoustic measurements are performed using a two-color pump-probe setup with a commercial femtosecond laser operating around 800 nm[20].

The principle of such a measurement is illustrated in Fig. 1. The laser output is split in two parts, the pump and the probe. The pump beam is focused at the sample surface where it's absorbed and the resulting local heating leads to a strain pulse that propagates in the layer at the sound velocity. When such a pulse reaches the interface with the substrate, a part of the pulse is transmitted to silicon and the rest is reflected towards the surface.

The second part of the laser, the probe beam, is first frequency doubled using a β-BaB_2O_4 nonlinear crystal. The resulting blue beam is then focused at the sample surface at the same place but time-delayed with respect to the pump thanks to a mechanical delay-line. The probe light reflected by the sample is monitored by a photodiode. To improve the signal-to-noise ratio, the pump beam is chopped using an acousto-optic modulator and the output of the photodiode is amplified through a lock-in scheme.

As the reflected acoustic pulse reaches back the free surface, the probe light reflection is slightly affected through the photo-elastic mechanism and an acoustic echo is detected as a sudden change in the transient reflectivity of the sample[21].

The zone that is measured using the PA technique is defined by the spot size of the laser beams. In our setup both beams are focused through a same x20 microscope objective leading to a spot size of 1-2 µm. PA measurement is thus very local and we repeat it over a large part of the surface to compare the resulting statistical results with the macroscopic measurements.

Such a two-color configuration has been chosen following previous published work of one of the authors as photo-elastic detection is reinforced using a blue probe in Ni material[16].

2.3. Mechanical testing of adhesion

Adhesion is also tested using a tape test to peel off each sample studied. We work with four different clear adhesive tapes with increasing strengths from the 3M company. Prior to the adhesion tests, the adhesion capability of each tape is measured according to the ASTM D330/D3330M - 04 "Standard Test Method for peel adhesion of pressure-sensitive tape". We carry out 180° peel tests on a reference plate with a defined roughness and after systematic cleaning. The plate used is always the same to limit the differences between the measurements. Controlled pressure is applied to the tape as it is placed on the plate. This test is carried out on a tensile test machine. Five measurements are performed for each tape. Results are given in Table 1 where tapes are numbered from Tape 1 to Tape 4 according to the adhesion strength.

Originally the tape test as a qualitative method is a binary test, good or not, depending on the film is still glued to its substrate after the test. But following the pioneer work of Kondo, the peeling can be quantified in terms of percent peeled area P, defined by the ratio between the peeled off area and the studied area[22].

Each tape covers a quarter of the sample surface and stays on for 60 s to assure a good adhesion between the surface and the tape which is then pulled off vertically to the substrate. It is important for the sample to stay still as a small tilt of the pull direction may cause a moment[22][23]. We obtain 0 % if the film is not peeled off, 100 % if totally peeled off and a number between 0 & 100 for a partial peeled off.

3. Results and discussion

3.1. Protocol description

As shown on the PA signal reproduced in Fig. 1, several acoustic echoes are clearly detected in a thin Ni film deposited on a Si substrate. Each echo arrives at the surface with a time-of-flight (tof) which is a multiple of the ratio between the film thickness (t) and the longitudinal sound velocity (v) inside the material. Assuming a sound velocity of 6050 m/s for Ni, one expects the first echo near 66 ps relative a first round-trip in the Ni layer. Knowing the sound velocity, the PA measurement can conversely be used to measure accurately the film thickness. On samples with a gold layer at the interface between metal and silicon, the gold layer thickness can be extracted from a detailed analysis of the echo shape and assuming the sound velocity in gold.

Such a series of echoes can then be used to extract the acoustic reflection coefficient R at the interface between Ni and Si. The portion of the strain pulse that is reflected at an interface is governed by the ratio between the acoustic impedances of both materials. The acoustic impedance of a given material (Z) is the product of the mass density by the sound velocity. And when an acoustic wave reaches an interface, the expected reflection coefficient of the strain field is given by:

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

where R is the reflection coefficient, and Z_1 and Z_2 are the respective acoustic impedances of the two materials.

In the present case, Ni or NiCr has a high acoustic impedance compared to Si which means that a significant part of the acoustic pulse is reflected at the interface with the substrate. Assuming a sound velocity of 6050 m/s and a mass density of 8.9 g.cm^{-3}, one obtains Z_1=53.8 10^6 kg.m^{-2}s^{-1} and R = -0.465 at a perfect interface with a Si substrate (Z_2=19.7 10^6 kg. m^{-2}s^{-1}) [24]. The negative sign is related to the fact that the second medium has a lower impedance than the first one. In the following we ignore the sign of R and only focus on its magnitude $|R|$. The poorer the adhesion is, the higher $|R|$ will be, the extreme case $|R|=1$ corresponding to a delamination.

R is extracted from the experimental signal by fitting the successive echo amplitudes with an exponential decay. Indeed, the first echo amplitude is related to R, the second to R^2 and more generally, the nth order echo to R^n.

Another mechanism affects the echo amplitude. As the strain pulse propagates in the metal layer, it experiences acoustic attenuation. Such an effect cannot be neglected in a thin metal film due to the ultra-high frequency involved in picosecond acoustics, typically a few 10 GHz in the present case. As attenuation is worse at higher frequency, it induces first a loss of the highest frequencies of the pulse and then an enlargement of the echo. To separate both effects (reflection at interface and attenuation), we perform a numerical modeling of the signal with our home made acousto-optic software (details can be found in Ref. [25]). From that, sound attenuation in Ni and NiCr is calibrated (6.10^{-3} nm^{-1} THz^{-2}, no difference between Ni and NiCr) and then the reflection coefficient can be derived easily from the exponential decay. As the sample series is composed of similar 200 nm thick Ni samples, attenuation is not a critical parameter for comparing samples. It only affects the absolute value of the reflection coefficient. For example a perfect Ni/Si interface, reflection coefficient R=-0.46 but due to attenuation R would be found 40% smaller (close to -0.32).

3.2. Acoustic adhesion results

On each sample, we first make 50 PA measurements uniformly distributed in a 25 mm^2 square. At each point, acoustic time of flight is used to extract the film thickness of the nickel layer and of the gold layer if present. Results are compiled in Tab. 2. Except for #2, the thickness is found to be very close to the nominal thickness (200~nm).

Then we extract the exponential decay of the amplitude of successive echoes to deduce the acoustic reflection coefficient R at the interface between the metal film and the Si substrate. It is found to vary from 0.31 to 0.56. Such measured values are affected by sound attenuation that also decreases the amplitude of the successive echoes as the pulse propagates in Ni.

By considering acoustic attenuation inside Ni, the lowest reflection coefficient corresponds to $|R|$=0.47 and the largest to $|R|$=0.83. One should note that the lowest value is very close to expected value for a perfect Ni/Si interface (0.465). In other words sample #1 can be considered as a perfect Ni/Si sample from the adhesion point of view. On the contrary, sample #8 with a $|R|$=0.83 is not far from delamination ($|R|$= 1).

As expected, samples containing a gold layer present a larger R value and then a worse adhesion is suspected. The complete acoustic reflection results, the mean value and the standard deviation of the 50 measurements, are plotted as a bar graph in Fig.2. Sample numbering has been made so that from #1 to #8, the reflection coefficient increases gradually. One clearly sees that we have obtained a gradual series of similar samples but differing by reflection coefficient.

3.3. Confrontation with Tape Test

After acoustic measurements, the samples are tested with tapes of various strengths. The stronger the tape force is, the easiest the layer is peeled off. For some samples, the metal layer is totally peeled off after one test which cannot be continued with a stronger tape. The final result of a tape test is a number related to the area that has been peeled off.

Fig. 3 presents for each tape, the correlation between the acoustic reflection coefficient and the peeled-off surface. As some samples were totally peeled off after one test, no further tape test can be performed after: this is the case for #8 after Tape 1 or #7 after Tape 3. For each tape, one observes a similar curve: low reflection coefficient samples resist to the test and high R value samples do not. The tape test gives usually binary results but for some of the layers we obtain a partial peel-off that goes perfectly with the reflection coefficient value. It creates 3 peel-off classes for the 3 reflection coefficient classes obtain between 0.31 and 0.56. As expected, the stronger the tape is, the more samples fail to the test. The gold layer at the interface is confirmed to weaken the bonding between Ni and its substrate: high reflection coefficients are measured and the Ni layer is easily peeled-off. The worst case appears to be the combination between an evaporated gold film and the sputtering of NiCr (sample #8). Adhesion is so weak that all the layer is removed after the first tape test.

Such an excellent correlation between both techniques finishes to demonstrate that acoustic reflection coefficient can be used to evaluate the adhesion of the thin-film on its substrate.

After the tape test, all the samples were damaged except #1. In order to confirm what happened during the peel-off test, an acoustic measurement is performed on the tape after the test with the strongest tape on Sample #2. About 50% of the layer has been removed from the substrate and is now glued on the tape itself. In Fig. 4, are compared the signals measured on the sample before the tape test and on the tape after. One retrieves a similar response dominated by a series of acoustic echoes. That first confirms the Ni nature of the tested layer in both cases. One also notes that the time of flight is the same which indicates that the whole Ni layer has been peeled-off from the substrate. One also remarks a clear difference between the reflection coefficient. As expected, the Ni film is almost suspended on the tape so that acoustic energy is confined in the metal film and the amplitude of the successive echoes decreases slowly through sound attenuation.

3.4. Local adhesion measurements

We now focus on sample #I made of a similar stack of layers (Ni 200 nm and Au 20 nm on silicon) but part of which has been exposed to an ion beam implantation. As only one part of the sample surface has been exposed, the final sample is thus expected to present two areas differing by adhesion. Here we take advantage of the local character of the acoustic measurement to map the reflection coefficient in both zones. Approximately 300 points of measurement are made on a 6x2 mm^2 zone which falls on both areas.

In Fig. 5 we show the corresponding map of the acoustic reflection coefficient. The two areas are clearly distinguished: 0.32 and 0.58 for the implanted and not implanted zone respectively. The zone that has been exposed to the ion beam presents a low reflection coefficient attesting for a good adhesion of the Ni film to the substrate. As previously reported in literature, the ion implantation reinforced the adhesion of the gold layer on silicon. On the contrary, at the not exposed places, adhesion is very poor, as expected due to the gold interface layer.

We finally perform a tape test with the lowest peel-off force. Only the not implanted area is peeled off as shown in Fig. 5b. The border between both areas is visible on both the acoustic map and the picture. The darker area is the one that was peeled off with a clear presence of gold layer on the tape.

4. Conclusion

We elaborated a series of similar samples but differing by the adhesion at the interface between a thin metal film and a silicon substrate. For that we combined various deposition techniques and the addition or not of an ultra-thin gold layer at the interface to weaken adhesion. Then samples are first tested using an ultra-high frequency acoustic technique. Based on a femtosecond laser such a technique offers a way to measure the acoustic reflection coefficient at the interface between the film and its substrate in a totally non-destructive manner. Then the same samples are tested using various strengths tapes. A perfect correlation is found between the two approaches: a sample with a high reflection coefficient value is fragile from the adhesion point of view and conversely a sample with a reflection coefficient close to the theoretical value does resist to any pullout test.

Picosecond acoustics combines several advantages: non-destructive, quantitative as the reflection coefficient is a number and local since the measurement is made on a area defined by the laser spot size, here 1-2 µm. Such a spatial capability is demonstrated here by studying the adhesion of a sample which adhesion has been reinforced at a specific zone. The acoustic image reproduces the mask geometry that has served to protect the unexposed zone. All the results presented here are concerned with samples made of a single layer deposited on a substrate. In further studies we would like to emphasize another advantage of the technique which is its capability to separate the successive layers in a complex stack. Such a capability is directly related to the time-resolved nature of the PA technique: the echoes issued from successive layers of a stack are detected at separate times of flight. From the adhesion point of view, such a capability is very useful to identify which interface is responsible of failure of a stack.

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Figure captions

Fig. 1. Principle of acoustic measurement at the nanoscale using a full optical setup and typical signal measured on a 200 nm thick Ni layer deposited on silicon. A femtosecond laser is split in two parts, the pump excites a short acoustic pulse at the sample surface; the probe is time-delayed with respect to the pump thanks to mechanical delay-line at specific delays . Tof means Time of flight.

Fig. 2. Mean value of the acoustic reflection coefficient measured at various places along the surface for the different samples using the picosecond acoustic technique. The larger the reflection coefficient is, the worse the adhesion is expected to be.

Fig. 3. Correlation between acoustic measurements and tape tests. Samples are successively tested using stronger tapes. The peeled surface is used to quantify the tape test. Inset: bar graph of the 8 samples color indicates effect. Green means no peeling. Orange partial peeling. Red full peeling. White means no tape test done (sample already destroyed).

Fig. 4. Acoustic measurements on the pulled off layer on the tape. The measured time of flight confirms that the whole layer has been peeled off.

Fig. 5. a) Mapping of acoustic reflection coefficient on and out the implanted zone of sample #I. A clear contrast is obtained confirming the reinforcement of adhesion induced by ion implantation. b) Tape test on the implanted sample: not implanted area is completely peeled off whereas implanted region does resist.

TABLE 1

Table 1: Calibration of the four tapes used in this work to evaluate thin-film adhesion. Adhesion strength is measured following a protocole described in the text. For each tape, five identical measurements are performed and the resulting mean values and standard deviations are given in the table.

Tape label	Reference	Adhesion strength (N/cm)	
		Mean value	Standard Deviation
Tape 1	3M 7100127554	1.56	0.04
Tape 2	3M 7000048101	1.87	0.29
Tape 3	3M 79303252	5.46	0.97
Tape 4	3M 7100117144	9.51	0.85

TABLE 2

Table 2: Samples description and results of acoustic measurements. For each sample, the thickness of the main layer (made of Ni or NiCr) and the thickness of Au layer are extracted from the acoustic time-of-flight. Acoustic reflection coefficient is measured form the decrease of the successive echoes. For the sample #I, two zones are distinguished depending on the location of the measurement (implanted or not zone).

Sample	Main layer		Gold interface layer		Acoustic Results
	Nature	Thickness (nm)	Deposition Technique	Thickness (nm)	Reflection Coefficient Mean value ± Std deviation
#1	Ni	206.1	-	-	0.31 ± 0.04
#2	Ni	184.0	-	-	0.38 ± 0.05
#3	Ni	202.2	E	20.5	0.42 ± 0.07
#4	Ni	207.4	E	22.1	0.46 ± 0.07
#5	Ni	204.6	E	24.5	0.48 ± 0.07
#6	Ni	206.4	S	24.3	0.52 ± 0.06
#7	NiCr	205.2	E	23.2	0.55 ± 0.06
#8	NiCr	204.0	E	24.7	0.56 ± 0.04
#I	NiCr	202.3	E	27.0	0.32 ± 0.06 / 0.58 ± 0.04

FIGURE 1

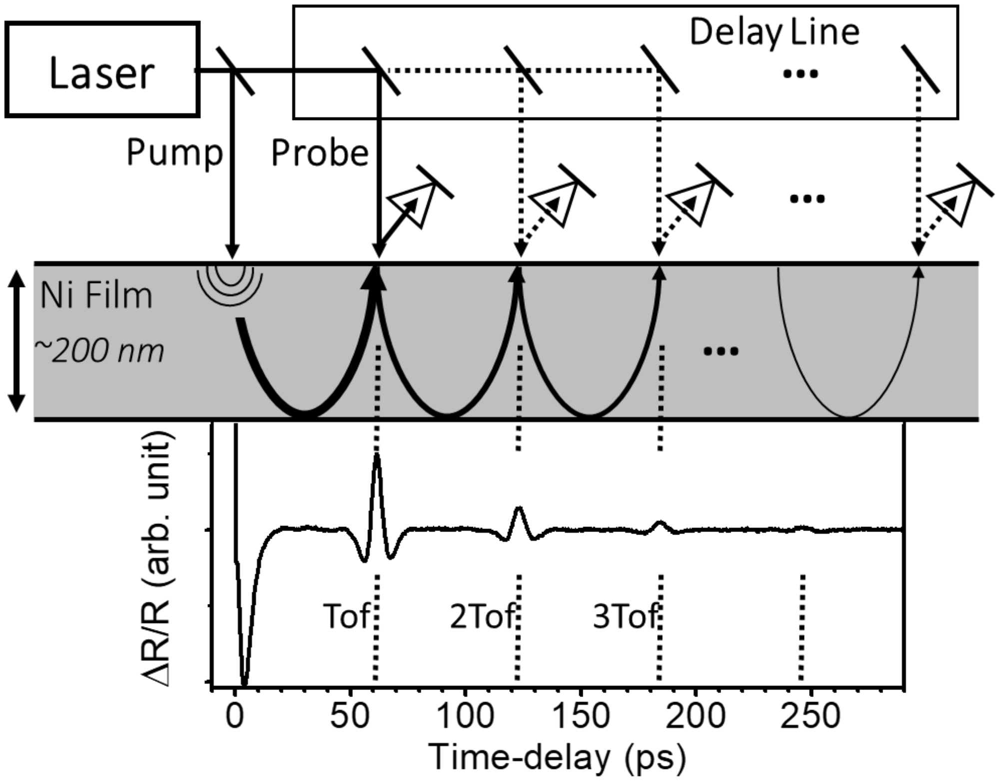

Fig. 1. Principle of acoustic measurement at the nanoscale using a full optical setup and typical signal measured on a 200 nm thick Ni layer deposited on silicon. A femtosecond laser is split in two parts, the pump excites a short acoustic pulse at the sample surface; the probe is time-delayed with respect to the pump thanks to mechanical delay-line at specific delays . Tof means Time of flight.

FIGURE 2

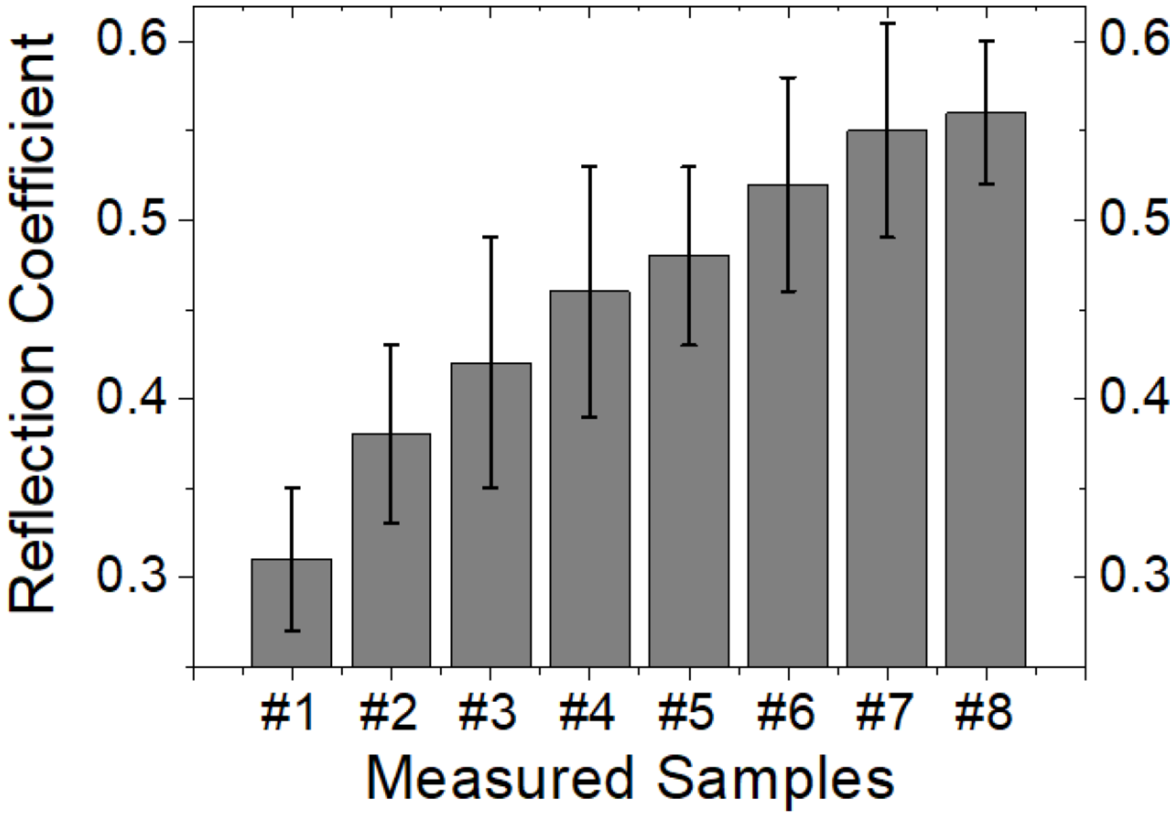

Fig. 2 Mean value of the acoustic reflection coefficient measured at various places along the surface for the different samples using the picosecond acoustic technique. The larger the reflection coefficient is, the worse the adhesion is expected to be.

FIGURE 3

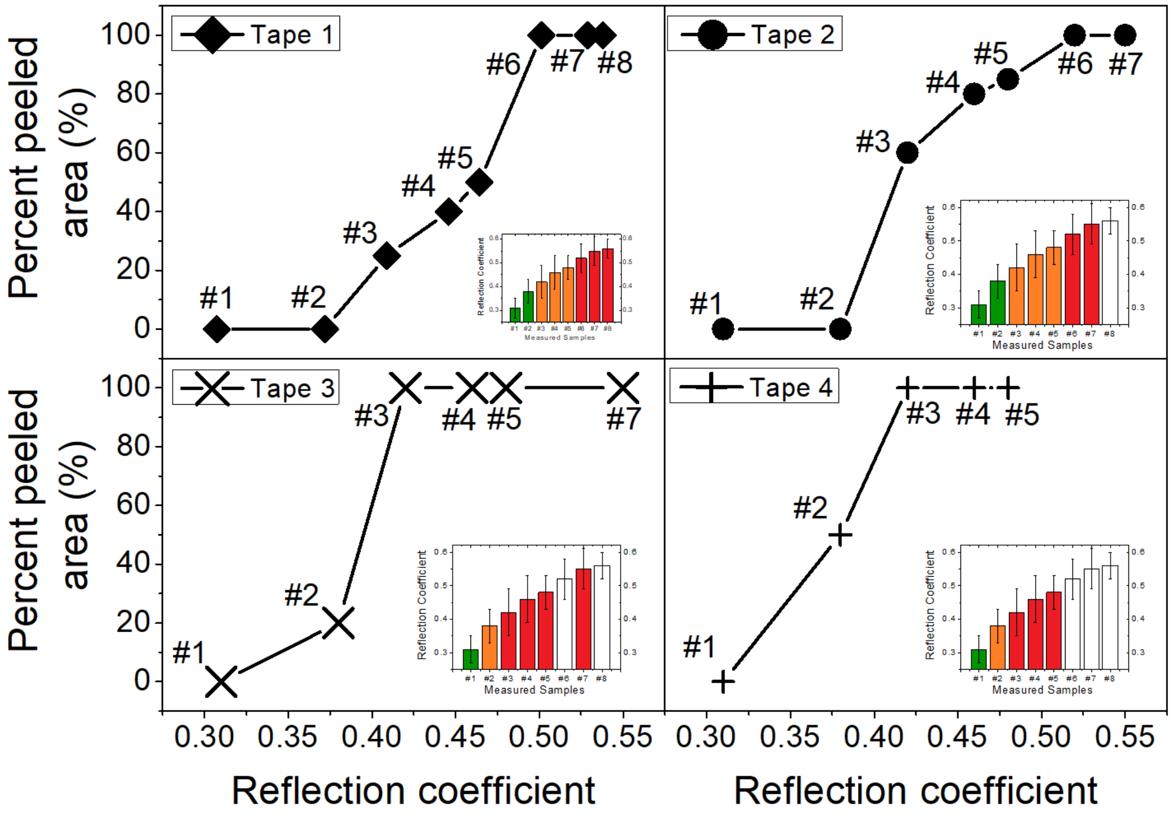

Fig. 3 Correlation between acoustic measurements and tape tests. Samples are successively tested using tapes with stronger and stronger strength. The peeled surface is used to quantify the test. Inset: bar graph of the 8 samples color indicates tape effect. Green means no peeling. Orange partial peeling. Red full peeling. White means no tape test done (sample already destroyed).

FIGURE 4

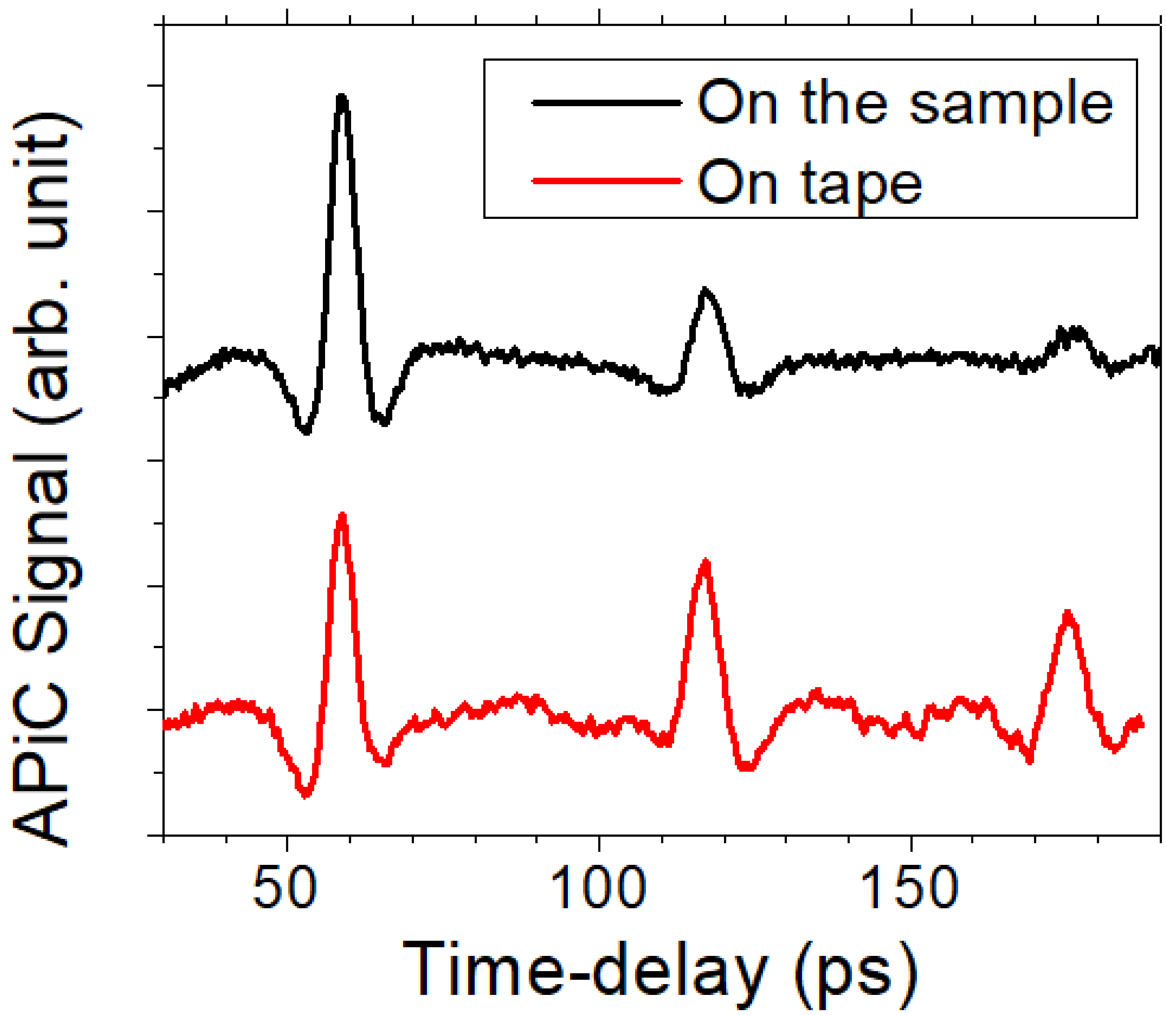

Fig. 4 Comparison between an acoustic measurement on the sample and on the tape after the peeled off test. The measured time of flight confirms that the whole layer has been peeled off.

FIGURE 5

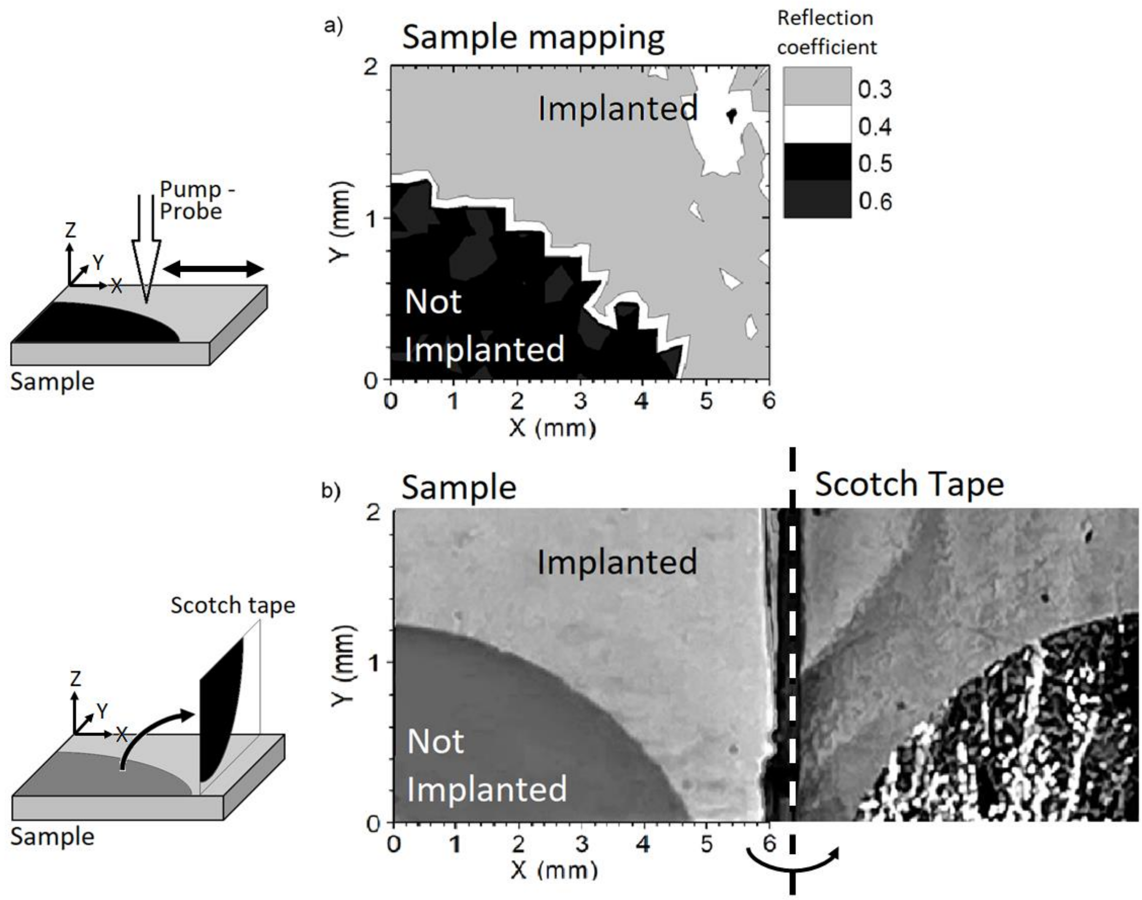

Fig. 5 – a) Mapping of acoustic reflection coefficient on and out the implanted area. A clear contrast is obtained between on and out. The non-implanted area presents a high reflection coefficient which means a poorer adhesion. That confirms the reinforcement of adhesion induced by ion implantation. b) Picture of the sample and picture of the tape after the adhesion test on the implanted sample. The sample image reveals an optical contrast as the not implanted zone area is totally peeled off whereas the implanted region does resist. The Ni film is now visible on the tape itself.