JOURNAL OF NANO- AND ELECTRONIC PHYSICS
Vol. 10 No 4, 04007(7pp) (2018)
ЖУРНАЛ НАНО- ТА ЕЛЕКТРОННОЇ ФІЗИКИ
Том 10 № 4, 04007(7cc) (2018)

Optimizing Combination of Parameters for Pull-off Adhesion Testing through Design of Experiment Study

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(Received 05 January 2018; revised manuscript received 08 August 2018; published online 25 August 2018)

A minimum of adhesion strength of thin films and coatings to substrates is required for their durable applications. Practical Adhesion is measure of that strength required to pull-off coating or film from the substrate. A suitable method to measure this adhesion strength has to be one that allows quantitative repeatable hence reliable results. There are many methods and techniques to measure this practical adhesion. However, none inclusively quantifies adhesion with repeatable results. Pull-Off method involves application of tensile forces and delivers quantitative results. Here we report optimizations made in combination of parameters for Pull-Off adhesion testing. Optimized combination of adhesive mixing ratio, time for hardening (curing), time to test after hardening and applied force rate was achieved through detailed Design of Experiment study. Achieved combination delivered results which were quantitative, repeatable, consistent and uniform and allowed application of method to a variety of coatings and films with enhanced reliability. The improvements here in reported are applicable to majority of thin films and coating systems delivering some standardized parameters combination for pull-off method.

Keywords: Adhesion Strength, Pull-Off Adhesion Testing, Design of Experiment, Thin Films.

DOI: 10.21272/jnep.10(4).04007 PACS numbers: 00.06. 07.05.Fb

1. INTRODUCTION

Irrespective of later use, structural, mechanical and functional properties of films and coatings depend upon their adhesion to substrate. This requires to measure adhesion strength of films and coatings for quality control to meet industry standards and product specifications. Substrates provide support to films and this support depends on adhesion between film and substrate. A good adhesion will make sure durable performance of films however poor adhesion makes film wearing-off rapid [1]. There are two forms of strength, cohesive (within film or substrate) and adhesive (at coating – substrate interface). There are many methods and techniques to determine adhesion strength. Selection of technique depends on individual requirements and credibility of instrument. There are many adhesion tests meeting standards set by American Society for Testing and Materials (ASTM). More than 200 adhesion measurement techniques and methods [2] are available. As per the dimensions of specimen, test methods are usually "macro" and "micro [3]. In case of measuring practical adhesion, desirable characteristics to meet by an ideal test are described in [4] however there is not a single instrument that can meet all these requirements. There is almost always a scatter in measured values among different bonding tests [5] and not any test is believed to give accurate results [6-7]. This is because of dissimilar interfaces that exist for huge variety of film and coating systems [2]. Most commonly used methods for the purpose of measuring quantitative practical adhesion of thin films and coatings are indentation [8] and scratch [9-10] tests. In addition to these conventional methods, recent works detail laser spallation technique [11], centrifugation [12] and possible application of atomic force microscopy (AFM) in measurement of adhesion for 2D nanomaterials [13]. However, among these different tests, pull-off test provides quantitative adhesion measurement. The test complies with ASTM D4541 standard [14] wherein a loading fixture (dolly) is fixed using an adhesive (glue) to the upper surface of multilayer films and coatings. A tensile force is applied either by mechanical (twist by hand), hydraulic (oil) or pneumatic (air) pressure. The weakest plane within the system will fail and the strength can be measured in Mega Pascal's (MPa) or pounds per square inches (psi). Notwithstanding, the measured strength depends on instrument used and results for different devices may vary. Also same coatings on different substrates may give different strength values. Here we report Design of Experiment (DoE) study carried to optimize combination of test instrument parameters for Pull-Off method to measure practical adhesion strength for system of multiple layers. The test instrument utilized in this study was PosiTest AT Pull-Off Tester from DeFelsko Manufacturers, USA. This tester complies with different international standards like ASTM D4541/D7234, ISO 4624/16276-1, and AS/NZS 1580.408.5 and can measure bond strengths as low as 3.5MPa (500 psi) and as high as 70MPa (10000 psi). The study helped to reach an optimized combination of parameters for instrument. The applied set of parameters delivered repeatable, reliable and consistent values. The combination of optimized

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set of parameters were later used to measure quantitative values of adhesion strength of different multilayer stacks reported elsewhere.

2. EPERIMENTAL DETAILS

In this study multi-layer stacks were deposited by Chemical Vapor Deposition (CVD). These multi-layer stacks were deposited in different sequences and received different treatments during and after deposition. Graphitic layers of 300 nm thickness were obtained by thermal activation of C_2H_4 and CH_4 precursors. Standard Si wafers of 8 inch diameter and 700 μm thickness were used as substrates. The deposited graphite layers had (100) lattice orientation. The substrate temperature during deposition was from 400 to 900 °C. The rate of deposition was about 0.5-20 nm/min. The Si wafer substrates were cleaned before putting them into the deposition chamber. Details of the layer combinations and different pre- and post-deposition treatments are provided elsewhere. The layer stacks were characterized for the adhesion strengths and for cohesion among layers in a stack. The Pull-Off Adhesion Testing method was used to measure the adhesion strengths. However, measured values had large scatter among them which reduced their reliability. Due to lack of repeatability and homogeneity in the obtained results, a Design of Experiment (DoE) study was conducted to obtain optimized parameters for test instrument. Optimized parameters were then utilized and resulted in repeatable homogeneous results for adhesion measurements. Here we report details of DoE and obtained parameters and their effectiveness in enhancing workability of instrument.

2.1 Preparation for Adhesion Testing

The system to measure the adhesion strength of layer stacks comprised the following components: Al plate used as base to place Si substrate wafer containing layer stacks on it, dolly to be attached onto the stack. Two component epoxy paste adhesive Araldite® 2011 from Huntsman Advanced Materials, Switzerland used to glue different system components. Araldite glue comprised two components 2011A and 2011B. Other details of the glue are available on manufacturer's web site.

2.2.1 Sample Preparation

Step wise preparation of samples for adhesion testing are detailed.

1. The heating plate was turned on keeping the temperature fixed at 80 °C.
2. The Al plates were cleaned by the cleaning paper and placed on the heating plate one by one.
3. Dollies were cleaned and placed on the heating plate.
4. All plates and dollies were left for heating for spread of glue within the components for better and even contact. This also removed any moisture on the surfaces of dolly or plate.
5. The glue was prepared by mixing equal proportions of two constituents. The mixing was manual and proportions were operator dependent.
6. A strip of 3 cm width was cut from the middle of the wafer containing coatings. The strip was further cut into small pieces of about 3 x 3 cm^2 discarding the centre piece of strip.
7. Pieces were glued onto the Al plate and "Dolly" was fixed on it, while the system was placed on the heating plate.
8. Stop watch was turned on to start counting down for two hours while the heating plate was kept at 80 °C.
9. The PosiTest AT Tester instrument was used to measure the adhesion strength of the coatings after hardening time elapsed.

Figures 1 to 3 schematically describe the steps in preparation of samples for adhesion testing.

3. DESIGN OF EXPERIMENT STUDY

The spread in obtained adhesion strength values from 1st run of measurements and variation in system response created doubt about the capability of the instrument and testing method to produce repeating and reproducible results. Also the results did not help to reach a solid conclusion about which layers and treatments' combination is capable of delivering graphite films with desired adhesion strengths. So Design of Experiment (DoE) study was scheduled by taking into account different factors which could influence the measurements. This was to optimize the testing method and to check the instruments' capability for producing consistent results. The details about results before and after the study in first and second run of measurements respectively are described.

Design of Experiment (DoE) is a study where systematic steps are taken ahead of time to ensure that the appropriate data will be obtained by observing not only influence of the individual elements but also of their mutual interaction on the end results, which will permit an objective analysis and will lead to valid inferences regarding the stated problem.

3.1 1st Run of DoE Study

The DoE was primarily planned to obtain an optimum combination of testing parameters for the adhesion test to have repeatability in results so that measured values carry greater reliability in them. Factors taken into consideration were: 1) glue mixing ratio, 2) time for hardening (curing), 3) time to test after hardening, 4) applied force rate.

Keeping glue mixing ratio as a variable to influence adhesion strength may seem unreal for bonding process. But it is suggested that tensile strength of glue does affect adhesion property [15]. To start with, a simple system comprising dolly and Al base plate of 3 mm thickness was used and arranged as shown in Figure 4 and a matrix of runs was scheduled. The matrix of runs is given in table 1 detailing different factors considered. The corresponding strength values obtained with mean, median and standard deviation are displayed in Table 2.

Values obtained against each run are reproduced in Table 2. The values were however, not consistent and homogeneous. Even values for the same run did not

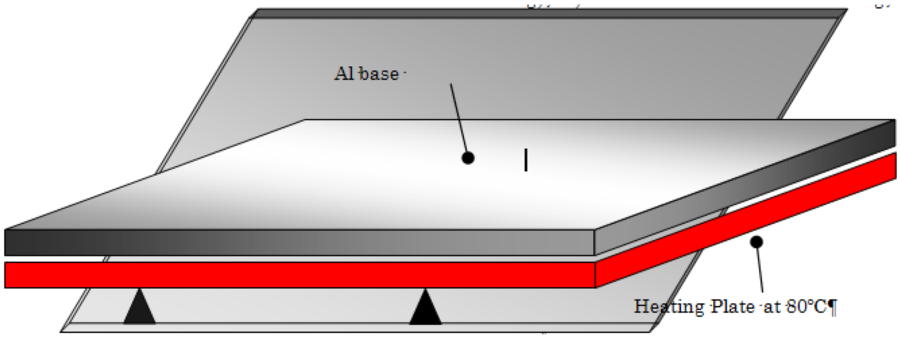

Fig. 1 – Placing Al base plate on heating plate

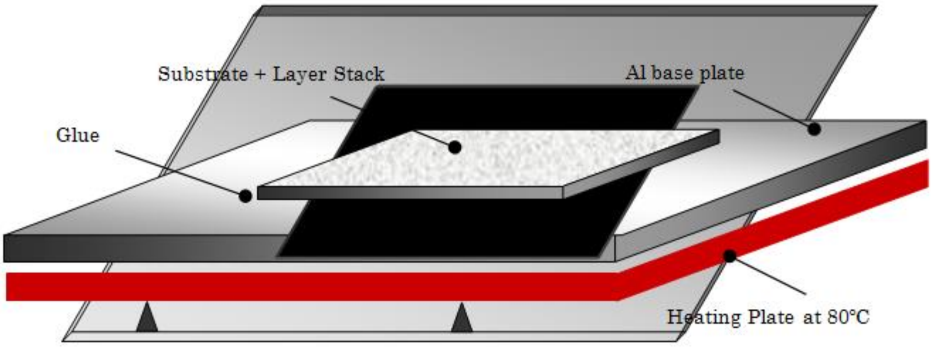

Fig. 2 – Gluing one by one, wafer sample containing layer stack to the Al base plate

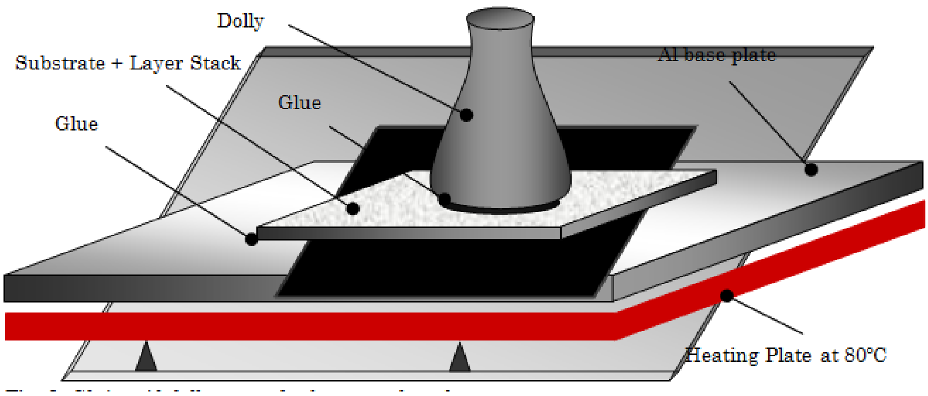

Fig. 3 – Gluing Al dolly on to the layer stack and system kept for hardening before adhesion testing

show any repeatability in them. For the run 4 and 5 where the mixing ratio of glue and hardener is 25:75, the values obtained could be regarded as repeatable or reproducible. However, the strength values measured are very low as more of hardener is used than glue. So although results are reproducible they are of no practical consideration.

To further clarify run 1 was performed again but with abrasive cleaning before isopropanol cleaning of the dolly and Al base plate. The rest of the factors remained the same. The values obtained in this case for first run of Table 1 are given in Table 3.

The minimum bond strength values obtained in case of abrasive cleaning increased a little as compared to the only isopropanol cleaning but the problem of lack of reproducibility remained. The mean and median values increased slightly in case of abrasive cleaning. It was also noteworthy that standard deviation in case of abrasive cleaning reduced although a little but indicating a positive effect of abrasive cleaning in bringing consistency in the strength values.

3.1.1 Analysis: Matrix of Runs

The strength values obtained for matrix of runs for a simple system comprising dolly, glue and Al plate substrate were utilized to reach at better combination of these factors.

3.1.1.1 Runs vs. Strength Values

The obtained strength values for all the runs shown in Table 2 where mean and medians for all the runs are compared for run 1 and after redoing it with abrasive cleaning indicated that abrasive cleaning had positive influence on bond strengths.

3.1.1.2 Glue Composition vs. Strength Values

The adhesive used for gluing dolly to wafer and wafer to base plate was Araldite 2011. The composition of glue (Araldite 2011A) and hardener (Araldite 2011B) was varied for different runs. Three different compositions used were A: B in 50:50, A: B in 75:25 and A: B in 25:75. Observing from the mixing ratio point of view, better bond strength values were obtained for the ratio 50:50 as shown in table 2 & 3.

3.1.1.3 Hardening Time vs. Strength Values

The system was put to different times of hardening. The samples were placed on a heating plate fixed at 80 °C and let for hardening for different times. Hardening for times of 120, 150 and 180 minutes were carried out and its influence on bond strength values was analyzed. Overall situation showed better strengths for hardening time of 2 hours as seen from table 2 & 3.

3.1.1.4 Time to Test vs. Strength Values

The samples after hardening were tested for bond strength values at different times, immediately after hardening time of two hours, after 8 hours from start of hardening and after 16 hours from start of hardening. Tables 2 and 3 detail the strength values obtained for three different times to test. From the mean and median values, it was inferred that waiting longer than testing immediately after hardening time of two hours results better adhesion strengths.

3.1.1.5 Force Rate vs. Strength Values

The samples were tested for strength values at different applied force rates to observe possible influence. Less than 50 psi/s, more than 50 psi/s and more than 100 psi/s were three applied force rates. The values obtained at applied force rates of more than 50 psi/s showed higher strength values. Tables 2 and 3 provide direct comparison of applied force rates vs. strength values.

3.1.1.6 Conclusions for 1st Run of DoE Study

Analyzing the strength values obtained for different runs against the parameters of test instrument, matrix of runs helped to reach a better combination of testing parameters. The following values were selected for the next testing.

1. glue: hardener ratio 50:50
2. hardening time 2 hours
3. time to test ≥ 4 hours
4. force rate > 50 psi/s

Although the strength values obtained in the first step of DoE were quite high, the problem of lack of repeatability in the results remained.

Table 1 – Matrix of runs for test parameters

Factor/Run	Glue composition (glue: hardener)	Hardening (curing) time at 80 °C	Time to test after curing	Applied force rate
1	50:50	120 min	immediately	< 50 psi/s
2	75:25	150 min	Immediately	< 50 psi/s
3	75:25	180 min	Immediately	< 50 psi/s
4	25:75	180 min	Immediately	< 50 psi/s
5	25:75	150 min	Immediately	< 50 psi/s
6	50:50	120 min	Immediately	< 50 psi/s
7	50:50	150 min	8 hours	< 50 psi/s
8	50:50	120 min	Immediately	< 50 psi/s
9	50:50	120 min	Immediately	> 100 psi/s
10	50:50	120 min	16 hours	< 50 psi/s

Table 2 – Values obtained for matrix of runs

Run	Strength values in MPa										Mean	Median	Std. deviation
1	13.29	7.48	14.91	7.79	8.09	9.23	11.96	12.38	9.7	11.57	10.7	10.6	2.5
2	8.39	8.37	8.64	12.18	8.76	6.86	7.39	7.74	6.41	8.43	8.3	8.4	1.6
3	8.43	8.51	9.7	9.44	10.86	3.93	8.31	7.82	11.45	7.68	8.6	8.5	2.1
4	1.59	1.74	1.58	1.66	1.75	1.81	1.62	1.62	1.68	1.62	1.7	1.7	0.1
5	1.39	1.5	1.41	1.14	1.47	1.45	0.99	1.45	1.42	1.46	1.4	1.4	0.2
6	9.41	13.75	9.22	14.38	11.05	8.18	9.12	9.59	9.48	12.1	10.6	9.6	2.1
7	10.59	10.14	12.16	14.98	11.79	11.1	12.39	7.25			11.3	11.4	2.2
8	14.01	8.42	12.16	10.64	7.3	13.47	9.37	11.09	11.98	10.78	11	11	2.1
9	12.0	9.89	9.8	10.61	9.33	9.92	8.86	8.99	9.92	9.57	9.9	9.8	1
10	15.12	12.38	13.08	9.07	9.21	11.69	7.29	14.83	8.15	7.92	10.9	10.4	3

Table 3 – Values for first run of table 1 with abrasive cleaning of dolly and Al base plate

Strength values in MPa										Mean	Median	Std. deviation
9.5	14.87	9.98	14.28	13.79	10.74	9.79	9.72	11.05	13.97	11.8	11.9	2

3.2 2nd Run of DoE Study

Along with different test instrument parameters, an observation was made regarding base plate used to glue samples on. Al base plate served as substrate for the multilayer film stack. Substrate plays a role in affecting critical load bearing of the thin film stacks [7, 16]. Warping in the plate during the 1st run of DoE study was suspected to be the reason of inconsistency in the values and system response. Uneven distribution of the tensile stress resulted due to low thickness of base plate. This produced non-uniform pull on the dolly [17]. After reaching optimized values of different parameters for the test setup (method), a change of the Al base plate was planned to make it sustain higher tensile force.

3.2.1 Dolly-glue-Al base Plate System

Observing the system components, Al base plate (3 mm) was replaced by thicker one. The tests were performed using the same system of dolly-glue-Al base plate (6 mm) with the testing parameters selected in 3.1.1.6. The results were very encouraging as the strength values obtained were very high and also there was observable repeatability in the values as shown in Table 4. This was further tested for more samples with an Al base plate of 4 mm thickness and the recycled dollies, both abrasive cleaned. The strength values obtained were much higher than with the 3 mm Al base plates but less than with 6 mm Al base plates. Also the force rate was more than 50 psi/s and testing was done after normal hardening time of 2 hours at 80 °C. These results supported the conclusion drawn about the possible influence of the base plate. 6 mm plate was therefore chosen for the next tests. Also the effect of abrasive cleaning in increasing the strength values as well as consistency was again confirmed.

3.2.2 Dolly-Si Wafer-Al Base Plate System

However, to further check the method and instrument repeatability, more tests were carried out with 6 mm thickness Al base plate adding Si wafer between dolly and plate. For comparison, thin (3 mm) Al plates were also used, side by side. Keeping in view the positive influence of abrasive cleaning on strength values and consistency, Al base plates & dollies were abrasive cleaned and new and recycled dollies were used simultaneously. Test results for system comprising dolly, Si wafer and Al base plate are shown in Table 6 where all values are in MPa. If a comparison is made according to the behavior of the system in Table 6, it is observed that it was every time glue that was going-off in case of thick plates with new and recycled dollies. Only one time the breakage of silicon wafer has occurred in case of recycled dolly on thick plate. This has occurred at a value much lower than all other values and indicates stronger cohesion between dolly and glue. This was considered to be due to smaller size of silicon wafer sample glued to the Al plate. The actuator was not covering the whole Si wafer sample. Therefore the force applied being uneven on the wafer sample could be the reason of breakage at lower strength values. In case of thin Al plates, again strong cohesion between dolly and glue was evident and most of the time it was silicon substrate breakage occurring signifying the influence of base plate. Although the cohesion between dolly and glue was good enough, the thinner Al base plate could not support the higher applied tensile force and silicon substrate breakage resulted. This was prominent in case of recycled dollies & 3 mm base plate and further signifies the role of base plate substrate in providing strength to the wafer.

The possible role of the base plate in providing equal tensile stress to the deposited coatings and films during the adhesion test was depicted by the comparison of obtained values for different thickness plates.

3.3 Conclusions for DoE Study - 2nd Step

The results obtained showed strong influence of the base plate as the values are doubled for thicker plates (6 mm) compared to thinner ones (3 mm). Same was the case for recycled dollies, where again the strength values were higher in case of thicker base plates than with the thinner ones as shown in table 6. Additionally a comparison of the new and recycled dollies showed that bond strengths in case of new dollies are higher than with recycled ones considering thick Al base plate. However, comparing thinner Al base plates, the values are higher for recycled dollies.

But noteworthy was the fact that in case of new dollies on thin Al base plates, it was most of the time glue that is going-off whereas in case of recycled dollies on thin Al base plates, it was most of the time Si wafer breakage. It is argued that in case of thin Al base plates with recycled dollies, the cohesion between Si wafer and plate is not enough to bear the applied pressure and support of the thin plate was not enough to strengthen the system. The same was opposite in case of new dollies where strength was lower between glue and Si wafer. This could be attributed to the irregular effect of thinner base plates as evidenced from high variation in values already obtained for all the wafers tested.

3.4 Optimized Parameters for Testing

From both runs of the DoE study, the following optimized parameters were chosen for the adhesion strength measurements in case of multilayer stacks. Values obtained using the selected test parameters were very consistent and repeatable as exampled in results and discussions section.

1. glue:hardener ratio 50:50
2. hardening time 2 hours
3. time to test ≥ 4 hours
4. force rate > 50 psi/s
5. Al base plate 6 mm thickness

Table 4 – Strength values and system response in case of 6 mm Al base plate

With abrasive cleaning (values in MPa)	20,2	23,63	23,91	23,98
Without abrasive cleaning (values in MPa)	17,67	9,78	16,54	7,46

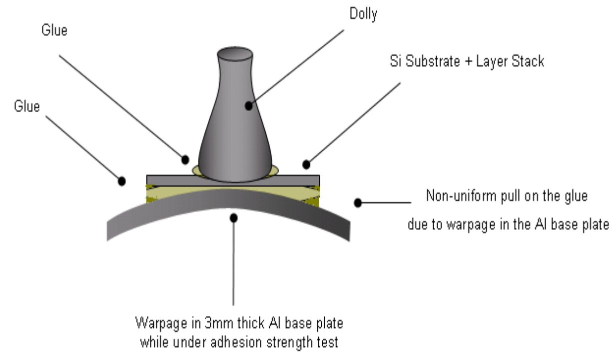

Fig. 4 – Warp in Al base plate while under pull-off test causing uneven stress on glue between substrate and Al base plate

Table 5 – Run 8 performed using 4 mm Al base plate

Run 8 with 4 mm Al base plate (values in MPa)	19,96	17,18	18,09	21,82

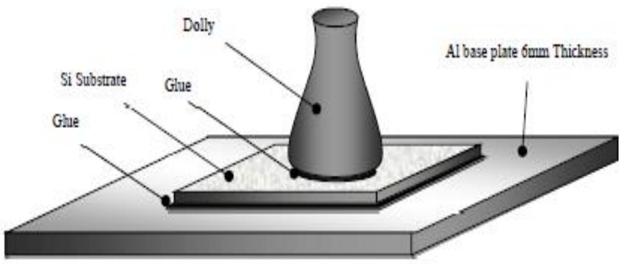

Fig. 5 – System comprising dolly-Si wafer-Al plate

4. RESULTS AND DISCUSSIONS

In the first run of measurements, at least four samples were taken from each wafer. However, due to lack of repeatability and consistency in the obtained results, a Design of Experiment (DoE) study was scheduled to reach an optimized combination of testing parameters. The tests made with these selected parameters provided quite consistent and repeatable results. This increased the reliability of the obtained adhesion strength values. Below we present a comparison of results obtained before and after DoE study. The bond strength values obtained for different wafer samples were quite low and these values & system response in the first run of the pull-off adhesion test showed spread among them. This spread in values was some times less and on other times it was very high. Also the behavior of the glue, layer stacks or substrate seemed to lack consistency and had no apparent pattern. The values obtained in case of one of the wafers before DoE study are reproduced in Table 7 for reference and example.

Considering other system components in the test, if one time the glue was strong enough to resist the applied tensile stress. It was however, going-off at a very low value at another time for the same sample. Although all the samples have different layer stacks, behavior of glue was not reproducible even for samples from same wafer. Nevertheless, it was observed that in all other cases, the glue has shown a resistance up to at least the value of 4 MPa for all the other samples of all the wafers. For a number of times, the value was even higher than 6 MPa. It supports to say that failure in the system was most of the time adhesive rather than cohesive. It was observed that only 31 % of the times the glue has went-off as compared to the 69 % of the times where either a layer stack has went-off or Si substrate breakage has occurred. For some of the times both the layer and glue had went-off half-half. This supports that the strengths obtained are of adhesive type although there is no good reproducibility or repeatability in the values. There were cases where both layer and glue have went-off and also there were times where the Si substrate breakage has occurred indicating greater local cohesion among dolly, glue and layer stacks.

From the results, capability of the testing method & instrument to produce repeatable & reproducible results came under doubt. Therefore, it was decided to check the reproducibility of the testing method & instrument. For this a matrix of runs was scheduled and starting from a simple system, repeatability of the instrument was checked. The system components were gradually changed from simple Al base plate and dolly to inclusion of simple silicon wafer. Wafers already tested containing graphite and other layers were tested again with abrasive cleaning and with selected parameters from matrix of runs. In-use 3 mm Al base plate was replaced by thicker 6 mm plate and tests were performed. The values were much higher and consistent than those of earlier obtained for the same wafer samples as shown in the Table 8.

Table 6 – Testing results for 3 mm and 6 mm Al base plates with new and recycled dollies

Thick substrate (6 mm) new dolly	14,05 glue-off	14,04 glue-off	17,4 glue-off	17 glue-off
Thin substrate (3 mm) new dolly	7,23 glue-off	8,83 glue-off	9,34 silicon breakage	7,88 glue-off
Thick substrate (6 mm) recycled dolly	13,38 glue-off	12,13 glue-off	13,21 glue-off	10,74 silicon breakage
Thin substrate (3 mm) recycled dolly	10,75 silicon breakage	9,45 glue-off	9,01 silicon breakage	8,54 silicon breakage

A direct comparison of results obtained before and after DoE study helped to conclude further the differences caused by selected parameters in strength values and system response. A comparison of values for 3 other wafers in table 9 further elaborates the difference caused by selected parameters from DoE study. Fifty wafers with layer stacks prepared in the first run of experiments were all characterized for the adhesion strengths before and after DoE study with optimized combination of parameters. The obtained values confirmed the effectiveness of optimized test parameters for adhesion strength characterization of multilayer stacks.

Table 7 – Values obtained before optimization of test instrument parameters

12.3 MPa	Glue-off
5.3 MPa	Film-off
5.75 MPa	Film-off with glue
8.43 MPa	Glue-off with substrate breakage

Table 8 – Values obtained for sample of Table 7 after DoE study for optimization of test parameters

23.58 MPa	Not pulled-off
21.72 MPa	Substrate broken
23.45 MPa	Film-off with glue
23.53 MPa	Glue-off with glue

Table 9 – Values obtained before and after DoE study for some sample wafers

3 mm Al base plate		Wafer	6 mm Al base plate	
System response	Values (MPa)		Values (MPa)	System response
glue-off	10.64	1	22.96	not pulled-off
glue-off	5.37	1	23.71	not pulled-off
film-off	5.29	1	23.24	not pulled-off
film-off	2.46	1	20.84	film off
film-off	5.59	2	23.71	not pulled-off
film-off	5.66	2	22.66	film off
film-off	6.49	2	22.6	not pulled-off
film-off	3.97	2	23.69	not pulled-off
film-off	3.97	3	20.09	film off
flue-off	8.93	3	23.61	not pulled-off
film-off with glue	6.01	3	23.58	not pulled-off
film-off	4.4	3	23.58	not pulled-off

5. CONCLUSIONS

Although there are many methods and techniques available, quantitative adhesion strength measurement has the advantage that a "numerical" value is obtained. The quantitative value can provide better idea about "in-service" capability of the different thin film stack systems. Quantitative adhesion strength measurement has been optimized for a particular measuring system. Design of Experiment study has resulted in a combination of parameters that could be applied to varied systems with confidence and enhanced consistency and reproducibility in results.

ACKNOWLEDGEMENT

The work was carried out at Infineon Technologies, Villach, Austria against a "Praktikum" position won by the principal author as a master student of University Institute of Advanced Studies, Pavia, Italy. Thanks are due to the anonymous experts who performed CVD at Infineon Technologies. Very special thanks to Norrie Habets in Quality Assurance at Infineon for his great help to prepare figures.

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