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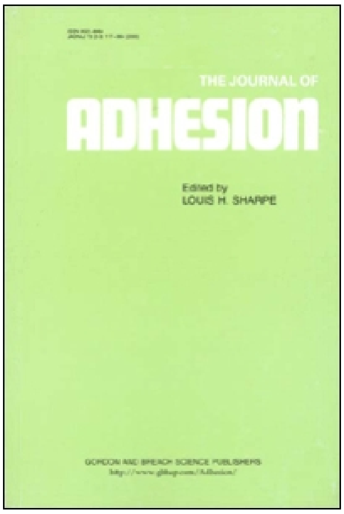

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Ravi Sharma[a]; Jong-Kai Lin[a]; James Drye[a]; Scott Lindsey[b]
[a] Motorola Inc., Phoenix, AZ, U.S.A. [b] Motorola Inc., Schaumburg, IL, U.S.A.

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A New Method to Evaluate Thin Film Adhesion*

RAVI SHARMA, JONG-KAI LIN** and JAMES DRYE

Motorola Inc., SPS, MD: AZ01-B136, 5005 E. McDowell Road, Phoenix, AZ 85008, U.S.A.

SCOTT LINDSEY

Motorola Inc., CMRC, MS: F31, Room 1014, 1301 E. Algonquin Road, Schaumburg, IL 60196, U.S.A.

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This paper discusses a new method of evaluating thin film adhesion both qualitatively and quantitatively *via* a combination of peeling and image processing techniques. The adhesion of thin film on the entire substrate can be quickly evaluated and quantified to a continuous response variable which is superior to a discrete response variable as described in the ASTM D3359-78 publication. Feasibility of this technique has been demonstrated through a gauge capability study which resulted in 2.7% P/T (precision to tolerance) ratio at six sigma standard deviation for a tolerance of 100. Experimental results using the proposed method to evaluate the process/property relationship of aluminum films as deposited onto various dielectric substrates such as polyimide, silicon dioxide, and silicon nitride have been obtained and have been shown to agree with conventional stud pull test results. The estimated cycle time to evaluate thin film adhesion is five minutes per 4-inch size wafer once the sample is prepared. This short process cycle time and proven reliability show that there is merit in implementing this technique both in the laboratory for process development and in the factory for statistical process control of products.

KEY WORDS adhesion; adhesion measurement; aluminum; thin film; pull test; peel test; image processing.

I. INTRODUCTION

Various methods to test thin film adhesion have been reported in the literature and reviewed by Mittal[1] and Valli.[2] The most frequently used methods are the pull test[3] or peel test.[4] The pull test[3] uses an adhesive cement/solder/epoxy to bond a stud to the film surface, and applies a tensile force to pull the film off its substrate. The adhesion is then characterized by the pull strength in force per unit area. Some disadvantages of the pull test include: non-uniform stress distribution or stress concentration over the contact area during the pulling process; interaction between

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**Corresponding author.

film and adhesive; annealing effect during soldering/epoxy curing; additional localized film stress when adhering the stud to the film surface; and misalignment of stud with respect to the film surface could create both tensile and shear forces during pulling process. The peel test[4] uses a backing film to thicken the original thin film so that it can be held by a grip for peeling. Part of the film to be peel-tested has to be free standing to initiate the peeling process. The peel test eliminates most of the problems associated with the pull test. However, the analysis of the adhesive force by the peel test is complex. It depends on the angle of peel, the rate of peel, the width of test sample, etc. Since it requires failure to initiate part of a free-standing film, the application is limited to relatively thick films with poor adhesion to the substrate. Both methods are time consuming for sample preparation and testing. Often, multiple testing of films from the same substrate has to be performed to ensure adequate representation of film adhesion throughout the substrate. For rapid engineering evaluation of thin film adhesion in both manufacturing and laboratory environments, short test cycle time is required. The proposed new test method uses a modified ASTM[5] tape peeling test and an image processing technique. This technique quantifies the adhesion value as a continuous variable instead of a discrete one as described in the ASTM D3359-78 publication. The adhesion of a thin film on the entire substrate surface can be quickly evaluated. In addition, the results obtained here show excellent agreement with the conventional stud pull test.

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II. THEORY OF OPERATION

An image of thin film adhesion sample must first be digitally recorded. This can be accomplished by an image scanner or a high resolution video camera used in conjunction with a frame grabber interface to the computer. If there is a high contrast between the adhering film and the substrate, a commercially-available image scanner for scanning text and photographs is adequate. If the contrast between the film and the substrate cannot be resolved by the image scanner, a high resolution video camera can be used with lighting control to enhance the contrast. A resolution of 300 points per inch is adequate for accurate image analysis. The image is scanned with sixteen levels of gray scale, 0 being white and 16 being black. The image can be saved on the computer hard disk in a Tagged-Image File Format (TIFF) for future use. The TIFF is used to exchange documents between different application software and different computer platforms.

The image processing program reads in the TIFF file and displays it in the form of a gray scale image on the computer monitor. Next, the gray scale image is converted into a binary image by adjusting the threshold level of the thin film patterns such that they are one color and the substrate background is another color. The image is now separated into two groups of data, one that represents the thin film patterns and one that does not. The image is calibrated to a known distance such as the diameter of a wafer. The image is stored in computer memory such that each square inch contains $(300)^2$ points. The image program determines how many data points are in the thin film pattern group and thus determines the area of the adhering film. The percent of thin film adhering can be determined by comparing the area of the adhering film with the area of an unpeeled sample.

III. EXPERIMENTAL PROCEDURES

Sample Preparation

The thin film with desired pattern and feature size can be prepared by either depositing through a shadow mask or depositing as a sheet film onto selected substrates followed by a photolithographic masking and etching process. The latter resembles closely the device manufacturing environment and the feature size can be much smaller than the shadow mask is able to achieve. In this paper, three millimeter diameter metal film dots were used for testing. After the thin film pattern was generated, a wafer dicing saw was used to saw through parts of the pattern. For consistent results, it was necessary to keep the saw spindle speed and feed rate constant. The thin film adhesion was evaluated by a tape peeling test. To perform the peeling test, an adhesive tape (3M 6200 permanent mending tape) was firmly pressed onto the metal/substrate surface on a row of metal dots and was subsequently peeled off the surface, similar to the way it is described in the ASTM publication D3359-78.[5] Four attempts were made to peel the film off the substrate in directions both parallel and perpendicular to the saw cuts ($\pm$X and $\pm$Y). Figure 1 shows an example of an adhesion test sample which has an array of thin film dots on a wafer. The saw cuts and the peeling directions are indicated by the straight lines and the arrows, respectively.

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Image Processing

After the peeling test, the film/substrate was put on an image scanner capable of creating 16-levels of gray scale and a resolution of 300 points per inch. A software

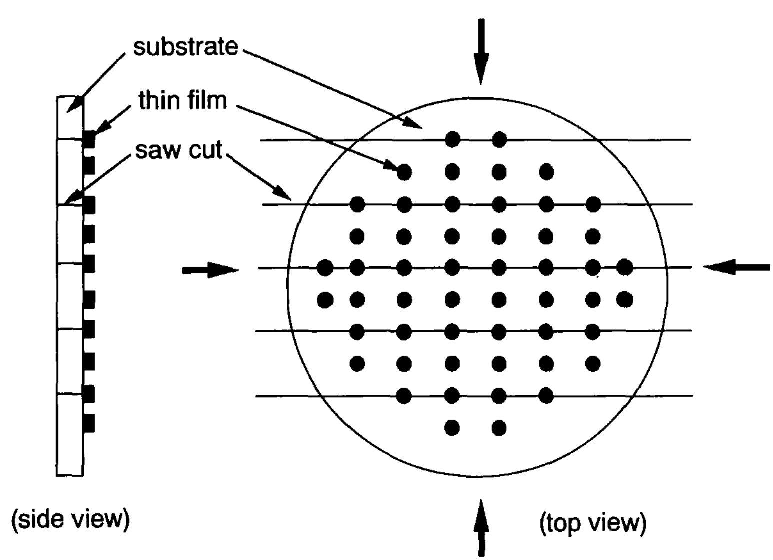

FIGURE 1 Schematics of a thin film adhesion test structure. (Arrows indicate the direction of peeling action during peel test.)

called "DeskScan" was used for scanning the film/substrate and creating a TIFF image file for analysis. The program "Image," which is available from the National Technical Information Service, was then used for image processing to calculate the area of the remaining films on the substrate. To calculate the film area, a threshold value at which all the deposited films turns into a red color while the substrate remains black has to be set. This was done by moving a cursor up and down a grey scale which represents different levels of image brightness. The areas where films survived peeling are now recognized by the image processor as the area with the red color. The areas where the films were peeled off are recognized as black. The area measurement was done by counting the number of points for each test pattern and a calibration measurement by selecting a feature with known dimension from the image.

Quantification of Adhesion Data

The area measurement and the percentage of films remained after peel testing were done on both sawed and unsawed thin film dots. The resulting adhesion scores were represented on a relative scale from 0 to 100. A score of 0 is assigned to completely delaminated film while a score of 100 is assigned to a film which shows no delamination. The calculation of adhesion score can be divided into two parts: those films which were sawed and those which were not sawed before the peeling test. Arbitrarily, one can choose to weigh the sawed and unsawed films differently in calculating the adhesion score. Since sawing before peeling is a more aggressive test, it is advisable to give more weight to the sawed films than the unsawed ones for the calculation. The worst case would be that considering only the sawed films. In this paper, the adhesion score were calculated by choosing 70% weight for the sawed film and 30% weight for the unsawed films. *I.e.*, the adhesion score was calculated by: (0.7 times percentage of remaining sawed film) plus (0.3 times percentage of remaining unsawed film).

Pull Test Measurements

The pull test was performed using a Sebastion-V (Quad Group; Spokane, WA, USA) testing unit which was connected to a Macintosh computer equipped with an A/D board for data analysis. Pre-epoxy coated aluminum studs with a diameter of 2.69 mm were obtained from the Quad Group (Spokane, WA). The aluminum metallized wafers were cut into approximately 1 cm square pieces. The studs were attached to the substrate using specially-designed clips to assure a normal and uniform force. The epoxy on the studs was cured by placing the samples into an oven heated to 150°C. The assemblies were removed after 60 to 70 minutes. This procedure was required to produce uniform adhesive thickness between the stud and the substrate. The variation in lots of epoxy coated studs was monitored by attaching several studs to 6.35 mm copper coupons. Measurements were performed at two scan speeds (8 lbs/sec and 16 lbs/sec) with at least five samples at each speed. The adhesion values from both speeds were then averaged together.

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TABLE I
Adhesion scores of samples used for gauge capability study

Sample ID	Operator A Trial #1	Operator A Trial #2	Operator A Range	Operator B Trial #1	Operator B Trial #2	Operator B Range
1	99.4984	99.5163	0.0179	99.2329	99.6515	0.4186
2	96.8253	96.8253	0.0000	96.7717	96.3661	0.4057
3	95.9080	95.9080	0.0000	95.8338	95.9559	0.1221
4	95.8220	95.8184	0.0036	97.6951	97.7057	0.0105
5	100.000	100.000	0.0000	100.000	100.000	0.0000
6	95.3526	95.3526	0.0000	99.5637	95.3968	4.1670
7	99.1078	99.1185	0.0107	98.6440	96.4786	2.1654
8	98.5452	98.5703	0.0251	98.5204	99.9238	1.4034
9	99.8710	99.8567	0.0143	99.6219	98.5007	1.1212
10	98.7280	98.7100	0.0179	98.6622	98.5914	0.0707

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IV. GAUGE CAPABILITY STUDY

To perform gauge capability study on the image scanner and the image processing technique, ten aluminum films were deposited onto polyimide substrates. The aluminum thin film patterns were created using a photolithographic process to minimize sample-to-sample variation of the dot dimension. Each sample was measured twice by two operators. The measured area was normalized to 100% for the maximum reading. The adhesion scores are summarized in Table I.

Gauge capability calculation[6] resulted in standard deviations of 0.4424 for repeatability and 0.0905 for reproducibility. The repeatability accounts for the equipment variation and the reproducibility accounts for the operator's variation during measurement. The total standard deviation of repeatability and reproducibility (R & R) was 0.4515. The precision to tolerance ratio (P/T ratio) was obtained by:

$$\frac{(\text{standard deviation of R \& R}) \times (\text{bandwidth of standard deviation})}{(\text{tolerance})}$$

The P/T ratios for various sigma bandwidths for tolerances of 5, 10, and 100 are summarized in Table II. These data indicate that, to evaluate films with 0% to 100% delamination (*i.e.*, tolerance = 100), the P/T ratio is as good as 2.7% at six sigma bandwidth which is an excellent gauge capability.

TABLE II
Precision-to-tolerance ratios of the gauge study

Tolerance	100			10			5		
Bandwidth	1 sigma	2 sigma	6 sigma	1 sigma	2 sigma	6 sigma	1 sigma	2 sigma	6 sigma
P/T Ratio	0.45%	0.90%	2.71%	4.52%	9.03%	27.09%	9.03%	18.06%	54.18%

V. DISCUSSION AND APPLICATIONS

Since the adhesion of thin films with and without a fracture surface can be very different, it is necessary to evaluate the thin film adhesion in both situations. In this paper, the sawed film was given more weight than the unsawed film (70% vs. 30%) since the sawed film represents a worse case than the unsawed film. For a robust process, one would like to evaluate the adhesion for the worst case (*i.e.*, all films sawed). However, for process development, tests of adhesion on both sawed and unsawed films gives one more detailed information on process-property relationships.

When using an image processing technique, it is important that there is good image contrast between film and substrate. Aluminum/polyimide, aluminum/silicon nitride, and aluminum/silicon dioxide usually work very well. Some image enhancement techniques such as using auxiliary camera or lighting may be employed for samples which do not have good contrast between film and substrate. Depending on the resolution of the image processing equipment, microscopic inspection of the sample may also be necessary to catch minor delamination.

The size and shape of the test structure do not affect the test result since the adhesion scores are calculated and normalized to a scale of 0 to 100. One should, however, choose a test structure whose feature size is much larger than the width of the saw blade to avoid excessive mechanical damage to the test structure due to sawing.

This technique has been used to study the adhesion of aluminum film to polyimide, silicon dioxide, and silicon nitride substrates. It has been demonstrated to be a very quick and effective method for process evaluation. Some results are shown in Figures 2, 3, and 4. The engineering time needed to evaluate one sample is about five minutes once the sample is made.

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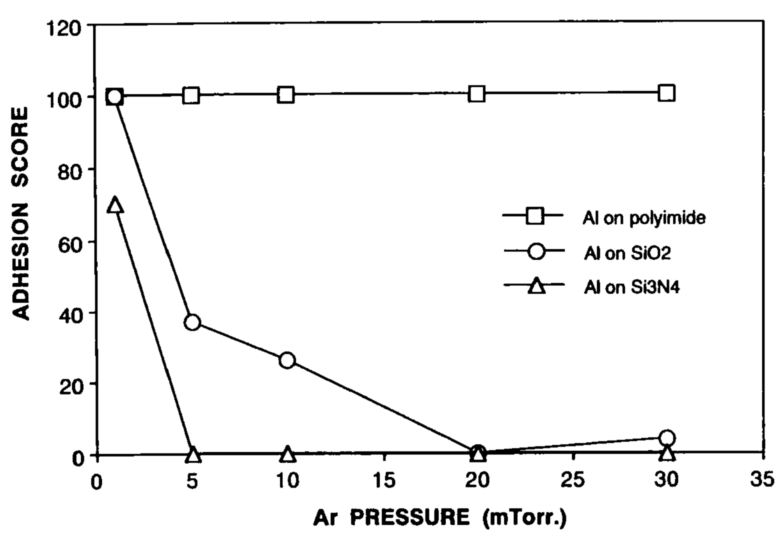

FIGURE 2 Adhesion of Al films as a function of deposition pressure. (Film thickness = 2.0 μm–2.3 μm; no substrate heating.)

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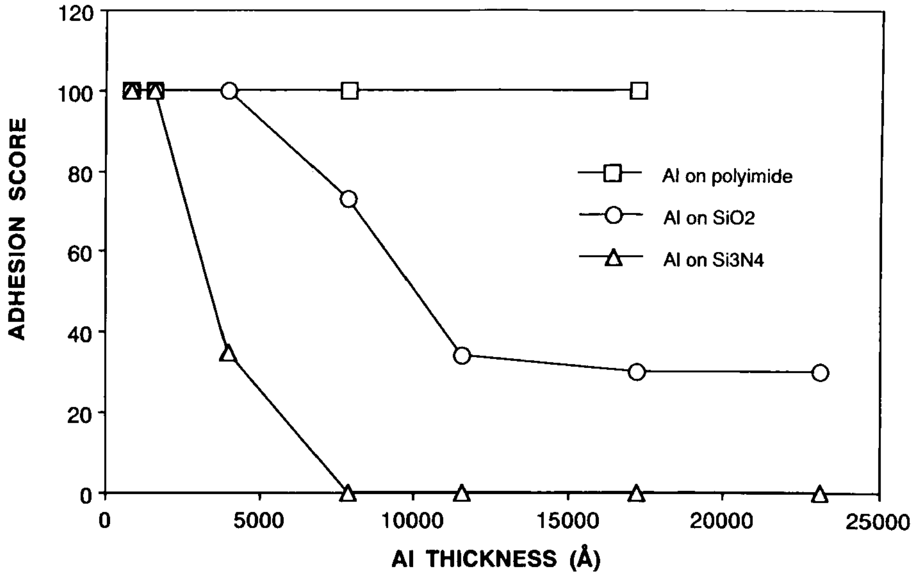

FIGURE 3 Adhesion of Al films as a function of film thickness. (No substrate heating, deposition pressure = 10 mTorr Ar.)

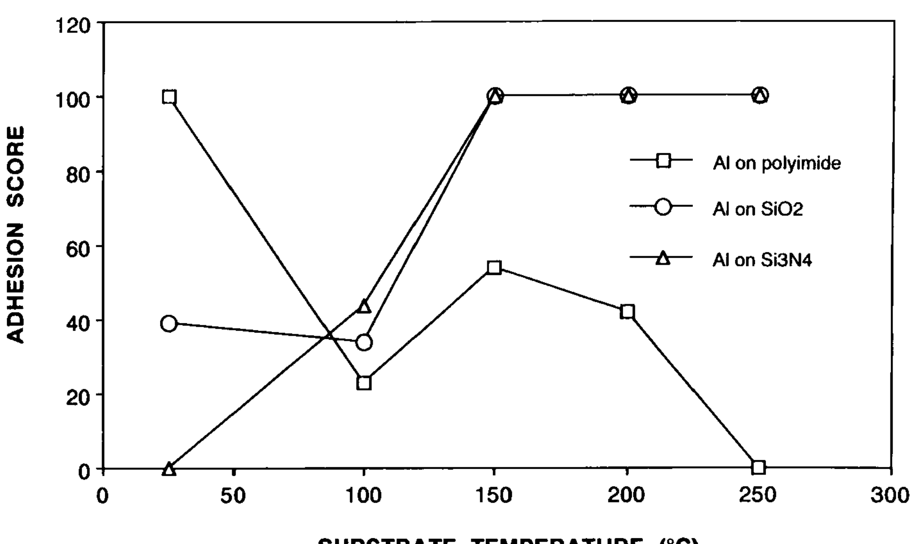

FIGURE 4 Adhesion of Al films as a function of substrate temperature. (Deposition pressure = 10 mTorr Ar, Thickness = 1.7 μm–1.9 μm.)

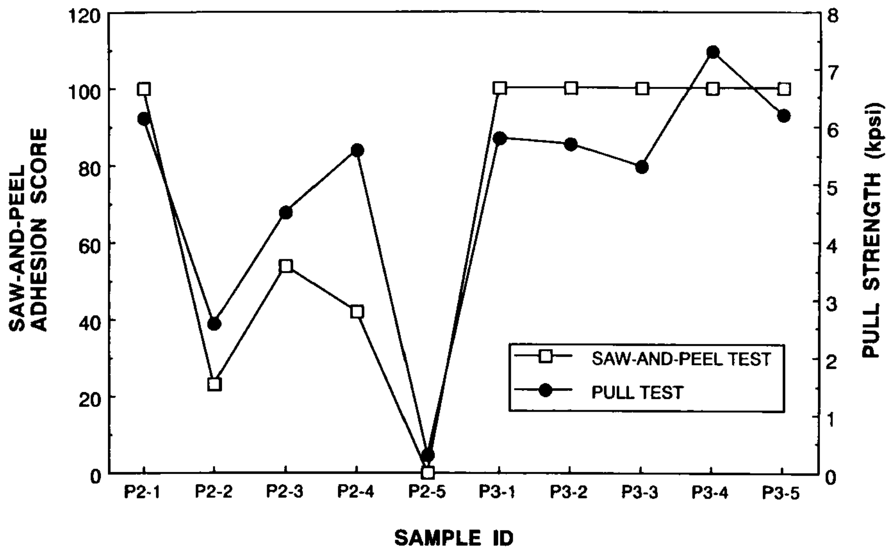

FIGURE 5 Run-to-run correlations between stud-pull strength and saw-and-peel test scores.

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In order to compare the results presented here with a more conventional test method, the aluminum-coated polyimide samples were also characterized using a stud pull test. The results from both types of adhesion measurements are given in Figures 5 and 6. In Figure 5, the scale for the adhesion score is given on the left coordinate, while the pull strength scale (in kpsi) is given on the right. In Figure 6, the correlation between the adhesion strength of pull test and the adhesion score of saw-and-peel test are plotted. The adhesion score of 100 represents the upper limit

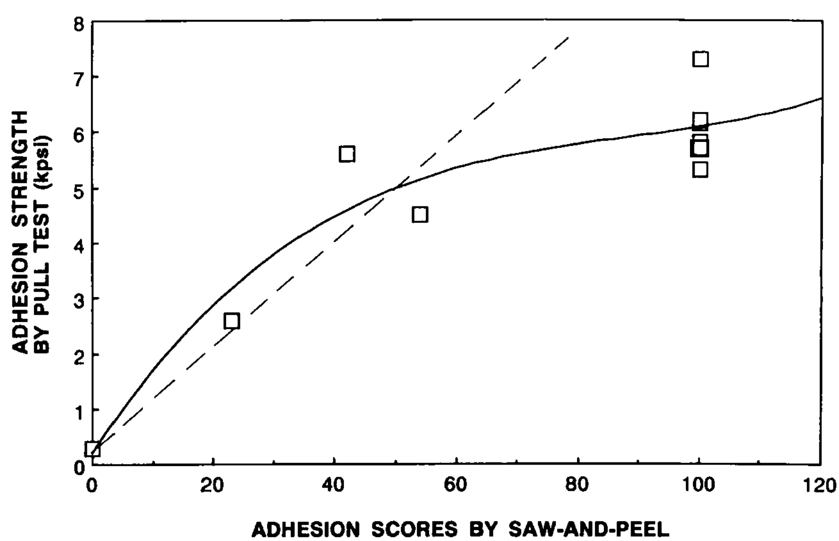

FIGURE 6 Correlation between saw-and-peel adhesion score and stud-pull adhesion strength.

of the saw-and-peel test. Beyond this value, the failure occurs at the adhesive tape/aluminum film interface rather than the aluminum/substrate interface. It is seen that the two measurement techniques show reasonably good agreement. The differences can be explained by considering the failure mechanisms of the individual die during the pull test. First, most of the stud-pulled samples failed at either the epoxy/aluminum interface or through silicon failure. Thus these values represent lower limits of the adhesion values for the aluminum/substrate interface. Further, since the majority of these failures occurred between five and seven kpsi, it is not possible to distinguish differences in adhesion in this range (even though there appear to be differences from the data). The qualitative agreement is apparent regardless of these differences and is sufficient to illustrate the comparative measurements.

VI. CONCLUSION

A new method has been developed to evaluate the adhesion of thin films to substrates efficiently and reliably by a combination of tape peeling and image processing techniques. Adhesion is quantified to a continuous response variable which is superior to a discrete response variable for thin film deposition process development. Feasibility of this technique has been demonstrated through a gauge capability study which resulted in 2.7% P/T ratio at six sigma standard deviation for a tolerance of 100. Satisfactory results have been obtained when using this method to study the process/property relationship of aluminum films as deposited on various dielectric substrates. The technique has been shown to give results which agree with those of a conventional technique—the stud pull test.

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