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WEIBULL ANALYSIS APPLIED TO THE PULL ADHESION TEST
AND FRACTURE OF A METAL-CERAMIC INTERFACE*

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ABSTRACT

Various adhesion tests have been developed to measure the mechanical bonding of thin coatings deposited on substrates. In the pull test, pins that have been bonded to the coating under test are pulled with increasing force normal to the coating until the coating is pulled from the substrate. For many systems, large scatter in the data is often observed due to uncontrolled defects in the interface and the brittle nature of the pull test.

In this study, the applicability of Weibull statistics to the analysis of adhesion of Ag films to vacuum sputter-cleaned zirconia was examined. Data were obtained for smooth and rough substrates for various levels of adhesion. A good fit of the data to the Weibull distribution was observed. The Weibull modulus was found to depend on the roughness of the substrate, but was insensitive to the adhesion strength.

INTRODUCTION

Various test methods, such as the scratch, microscratch (1), pull, magnetic hammer (2), laser-pulse spallation (3), and peel (4) have been developed to measure the adhesion of thin films to substrates. Each method possesses certain advantages and disadvantages (5-7).

Scratch-testing is a widely used technique for testing hard coatings and has been analyzed in detail (6-9). In the scratch test, a small-diameter diamond stylus is moved across the coating. An increasing normal force is applied, and the coating is judged to have failed when disruption or

cracking of the coating occurs. Large scatter in the data is often observed, and this scatter can be attributed to the brittle nature of the hard coating and interface. Catastrophic failure occurs when the increasing normal and traction forces initiate cracks at defects in the coating or in the coating/substrate interface.

Weibull statistics are widely used for the characterization of many engineering materials, perhaps most commonly for fracture of brittle materials such as glass and ceramics. For brittle fracture in particular, the Weibull statistics are easily analyzed and can be used to characterize material quality and strength.

Bull and Rickerby used Weibull statistics to analyze the results of a series of scratch tests on hard TiN (10). Although hard films are commonly thought to fail during the scratch test at a "critical load" that is more or less constant, the authors observed a spectrum of failures above and below the critical load and found that the failure distribution, which is related to the interfacial flaw-size distribution, could be analyzed in terms of the Weibull distribution. Substrate surface effects were found to influence the Weibull parameters, with roughness significantly affecting the Weibull modulus. For TiN with a Ti underlayer, the Weibull modulus and critical load were functions of the interface thickness, but were not correlated with abrasion resistance (11).

In a similar approach, Weibull statistics were used to analyze 4-point-bend fracture-strength measurements of several types of bones (12). Weibull statistics fitted the fracture data for the three bone types studied. The statistics were also a function of the type of bone, and it was suggested that the defect distribution in the bones was such as to allow the Weibull statistics to discriminate among the bone types.

In the pin-pull method of testing adhesion, pins or studs are bonded to the film to be tested. The pins are pulled with an increasing force normal to the substrate until the pin is pulled from the substrate. Failure at the film/substrate interface occurs catastrophically, in a manner similar to the sudden tensile fracture of ceramics. The adhesion strength is taken to be the force required to pull the film from the substrate divided by the area of the pin. It should be emphasized that this measured adhesion strength cannot be directly associated with molecular bonding between the film

and substrate. Although the adhesion strength has dimensions of stress, it is an experimental measurement that may be influenced by experimental parameters, such as pin size, misalignment, presence of defects in the interface, and is not easily related to any intrinsic properties of the interface. It is most useful for comparison rather than a way of arriving at fundamental properties of interfaces. Despite this limitation, it is perhaps the most easy and inexpensive method for characterizing the adhesion of non-brittle films.

Large scatter is often present in the data. Because of this, several tests are performed on each specimen, and mean values are commonly reported as a measure of adhesion. It is assumed that fracture initiates from flaws or impurities at the interface between film and substrate. These weak areas concentrate the applied load and serve as initiation sites for cracks. The random nature and distribution of the weak areas lead to widely varying values of load that leads to film failure.

Interfacial flaws due to particles or gross contamination can be minimized by careful solvent or sputter-cleaning of the substrates prior to film deposition. However, if the interface chemistry is such that only moderate adhesion is measured, it is clear that a distribution of areas of poor bonding must exist. For this case, it was hypothesized that the distribution of pull-off forces could be analyzed by Weibull statistics.

To investigate this hypothesis, we prepared several specimens under conditions designed to produce intermediate adhesion between film and substrate. An intermediate adhesion was chosen to avoid confounding effects. At very low adhesion strengths, the stresses required to pull the film from the substrate are difficult to measure with any accuracy. For adhesion in the "Scotch-tape-test" regime, a pin can be easily broken off during handling or testing. At high adhesion strengths, however, failure of the bonding agent occurs, and the pull-off force is that required to break the bonding agent, not the interface. Such bonding failure is, however, easy to detect. Thus for a study of distribution of failure stresses, deposition conditions were chosen to produce intermediate adhesion strengths, i.e., $\approx 5\text{-}50$ MPa. Pull tests were conducted and the resulting data were analyzed by Weibull statistics.

Specimen cleaning by sputtering in vacuum is critical for enhancing the adhesion of Ag films deposited on different substrates (13, 14). The results of adhesion tests of Ag deposited on zirconia using various sputtering treatments were reported previously and served as a guideline (15).

WEIBULL STATISTICS

Weibull proposed a general distribution function for characterizing measured parameters of various physical systems, one example being the failure strength of materials (16). He assumed that the function can be used to characterize a wide variety of physical systems regardless of the physical underpinnings of the particular system. Weibull showed that the function works well for modeling such disparate phenomena as the strength of steel, the size distribution of fly ash, the strength of cotton, and the length of marine animals. The method does not seem to have been applied to the pin-pull test.

The Weibull method fits the data to a three-parameter distribution function (two parameters for brittle fracture). The "Weibull plot" is an aid to obtaining these parameters. The Weibull method has been found to work in a wide variety of physical systems, in many cases better than any other known distribution function (16), despite its phenomenological origin.

In his original report, Weibull stated that he chose the distribution function in large part for its simplicity (16). The Weibull plot is thus not directly related to a physical model as is the Arrhenius plot. The Arrhenius plot allows the rapid determination of an activation energy on which a physical phenomenon is assumed to depend; the Weibull plot allows the determination of parameters that characterize a probability distribution.

Weibull defined $P(x)$ as the probability of choosing at random a sample that exhibits a characteristic less than or equal to x . Applied to fracture, $P(x)$ is the probability that an element under test will support a load x . The probability of failure can be written as

$$P(x) = \frac{n_x}{N+1}$$

where n_x is the ranking of a specimen that failed at strength x when the data pairs are sorted in increasing order of x , and N is the total number of tests. For any system, $P(x)$ can always be written as

$$P(x) = 1 - e^{-\varphi(x)}$$

where φ is a suitably chosen function of the variable x . Weibull proposed that the empirical function

$$\varphi(x) = \frac{(x-x_u)^m}{x_o}$$

will satisfy the requirements in many physical systems. Thus,

$$P(x) = 1 - \exp\left[-\frac{(x-x_u)^m}{x_o}\right].$$

The parameter x_u is the minimum stress for failure, and for brittle fracture, x_u is found to be essentially zero. In the case of ductile failure, such as occurs in steels, Weibull demonstrated that x_u can be significant. The previous expression can be written as

$$\ln\left[\ln\left(\frac{1}{1-P(x)}\right)\right] = m \ln(x-x_u) - \ln(x_o).$$

If x_u is assumed to be zero, $\ln(x)$ can be graphed as a function of $\ln(\ln\{1/[1-P(x)]\})$ the slope of the line is m , and the y intercept is $-\ln(x_o)$.

Other workers utilize a modified function which can be obtained from the original by moving the exponent m outside the parentheses (10, 12). The equation is then

$$P(x) = 1 - \exp\left[-\frac{(x-x_u)}{x_o}\right]^m$$

so

$$\ln\left[\ln\left(\frac{1}{1-P(x)}\right)\right] = m \ln(x-x_u) - m \ln(x_o).$$

This form of the Weibull equation does not alter the meaning of the exponent m and is advantageous because x_o has the dimensions of stress; x_o is now the stress at which the probability of failure is 63.2%. The Weibull modulus m is a measure of the spread of the data, x_u can be thought of as a minimum stress for fracture, and x_o is a characteristic stress. The Weibull modulus

is a measure of the range of flaw sizes present, not the absolute flaw size. If failure is not brittle (or if the Weibull method is used to model other physical systems), x_u may be nonzero, and a graph of $\ln(x)$ against $\ln(-\ln[1-P(x)])$ will not yield a straight line. In this case, the parameters must be determined by varying x_u until a straight line is obtained, or by using other methods. If x_u is zero, or is reasonably close to zero, $\ln(x)$ can be plotted as a function of $\ln(-\ln[1-P(x)])$; the slope of the line is m , and the y intercept is $-m \ln(x_0)$.

EXPERIMENT

Zirconia substrates were coated with Ag to a thickness of 1 μm in a vacuum chamber equipped with an electron-beam-heated evaporation source. Specimens were polished commercial polycrystalline ZrO_2 , 25.4 x 50.8 x 6.36 mm, and were either coated in the as-received condition, or were roughened prior to coating by grinding with 400-grit SiC abrasive papers. The evaporation rate of the Ag was 0.78 $\text{nm}\cdot\text{s}^{-1}$, as measured and controlled with the aid of a quartz-crystal rate monitor. Coating was done with the specimens at ambient temperatures, and temperature increases during deposition were typically a few degrees Celsius. During sputter-cleaning and coating, specimens were rotated at a rate of 1 revolution $\cdot\text{s}^{-1}$. Typical base pressures in the cryopumped system were 10^{-5} Pa. Specimens were cleaned with ethanol and dried with hot air in preparation for coating.

Energetic ions for sputter-cleaning were generated by a Kaufman-type ion source. Ion kinetic energy was 500 eV and the current density was nominally 150 $\mu\text{A cm}^{-2}$, measured with a Faraday cup. The ion beam was neutralized with an external filament to minimize charging of the insulating substrates. Specimens were sputter-cleaned with Ar^+ ions, the ion beam was turned off, and Ag was allowed to immediately (<0.5 s) deposit on the cleaned specimen. Gas flow into the ion source was UHP argon or a 1:1 mixture of Ar and O_2 . The total gas flow rate was $1.67 \times 10^{-7} \text{ m}^3\cdot\text{s}^{-1}$ (10 sccm), which produced a dynamic chamber pressure of 1×10^{-2} Pa.

The pull tests were conducted with a commercial pull-test machine (Sebastian V). A schematic diagram of the pull geometry is shown in Fig. 1. Tapered Al pins (2.69 mm head diameter) were bonded to the film with an epoxy that was cured on a hot plate for 1800 s at 150°C. To minimize

misalignment of the pulling force, the specimen platform was modified so that it could tilt slightly to equalize pulling forces. We took the "adhesion strength" x to be the force required to pull a pin from the specimen divided by the area of the pin. At least 30 pull tests were done sequentially on each specimen.

EXPERIMENTAL RESULTS

Adhesion-strength data were obtained from a number of smooth and rough specimens. A Weibull distribution of failure strengths was found: graphing of $\ln(-\ln[1-P(x)])$ as a function of $\ln(x)$, where x has units of MPa allowed m and x_0 to be were obtained by a least-squares fit of the data to a straight line.

Smooth Interface

Fig. 2a is a graph of P as a function of x for a smooth zirconia specimen sputter-cleaned with $2.8 \times 10^{20} \text{ Ar}^+ \cdot \text{m}^{-2}$ before coating with Ag. The arrow marks x_0 , the stress at which the probability of failure is 63.2%. Fig. 2b shows the associated Weibull distribution curve. A good fit of the data to a straight line is seen, although a slight positive curvature might be discernible. The Weibull modulus is 2.04, and the characteristic stress is 38.8 MPa. For comparison, mean and median adhesion strengths were 33.7 and 33.1 MPa, respectively.

A smooth zirconia specimen was sputter-cleaned with $2.8 \times 10^{19} \text{ Ar}^+ \cdot \text{m}^{-2}$ to produce a lower mean adhesion. Fig. 3 shows the Weibull distribution curve. A moderately good fit of the data to a straight line is seen, with a slight negative curvature discernible. The Weibull modulus is 2.07, essentially the same as for the stronger case, and the characteristic stress is 23.9 MPa. Mean and median adhesion strengths were 21.1 and 18.7 MPa, respectively.

In order to compare these results to fracture of a brittle homogeneous material, a series of pin-pull tests was conducted on small pieces of glass microscope slides. In this case, the bonding agent was stronger than the glass, and during testing round sections of glass were pulled from the surface. Fig. 4 shows the Weibull distribution curve for these tests. An excellent fit of the data to a straight line is observed. The Weibull modulus is 1.99 (again essentially the same as for the Ag

films on smooth zirconia), and the characteristic stress is 46.15 MPa. Mean and median adhesion strengths were 40.2 and 41.3 MPa, respectively.

Rough Interface

Fig. 5 shows the Weibull distribution curve for a rough zirconia specimen sputter-cleaned with $1.95 \times 10^{20} \text{ Ar}^+ \cdot \text{m}^{-2}$. A good fit of the data to a straight line is seen. The Weibull modulus is somewhat lower at 1.7, and the characteristic stress is 34.7 MPa. Mean and median adhesion strengths were 30.8 MPa and 27.2 MPa, respectively.

Fig. 6 shows the Weibull distribution curve for a rough zirconia specimen sputter-cleaned with $9.5 \times 10^{19} \text{ Ar}^+ \cdot \text{m}^{-2}$ to produce less adhesion than that of the specimen in Fig. 5. Deviation from a straight line is seen. For this particular specimen, examination of a graph of the original data in the sequence that they were obtained revealed that the measured adhesion strengths improved after repeated testing. The data showing the rise in adhesion strengths were discarded, and the data obtained prior to onset of noticeable adhesion increase (the first 18 tests) were replotted and are shown as an inset to Fig. 6. The data now fit well to a straight line. The Weibull modulus was 1.6, again essentially the same as for the stronger specimen, and the characteristic stress was 6.95 MPa. Mean and median adhesion strengths were 6.04 and 6.33 MPa, respectively.

DISCUSSION

Specimens coated without undergoing sputter-cleaning exhibited insignificant film adhesion. Data could not be obtained from these specimens because the bonded pins fell off with the coating attached to them at the slightest touch during handling. This low adhesion shows that some sputter-cleaning is necessary to produce film-to-substrate bonding, and the force necessary to tear the circular edge of the metal film is insignificant compared to typical pull forces.

Weibull Modulus

A smooth surface minimizes any contributions to adhesion from mechanical interlocking of the film with the substrate. The calculated Weibull moduli of the glass and the smooth Ag/zirconia specimens are all very close to 2.0. For these specimens, the modulus was apparently not a strong function of mean interfacial adhesion, if at all. The Weibull moduli are quite low, and reflect the

large scatter present in the data, indicating a large range of interfacial flaw sizes. For comparison, Weibull moduli between 2 and 5 were seen for scratch testing of TiN (10), and between 15 and 40 for TiN with a Ti interlayer (11). The Weibull modulus for bone, however, was 3.1 (12).

The similarity of the Weibull moduli from Figs. 2-4 suggests that the Ag/zirconia interface is brittle and would fracture in a manner similar to that of glass. The glass is expected to exhibit brittle fracture. The exponent of 2.0 is equivalent to saying that the probability of the film remaining intact decreases with load in a Gaussian fashion.

For rough specimens, the Weibull modulus was reduced from ≈ 2.0 to ≈ 1.7 or 1.6. The Weibull modulus is a measure of the range of flaw sizes present, not the absolute flaw size. The reduction in Weibull modulus for rough surfaces is indicative that the distribution of flaw sizes is broader. Although a rough surface may provide more opportunity for mechanical interlocking of the film and may exhibit greater actual strength, the Weibull modulus could be lower due to the wider range of flaw sizes. This could be explained if the rough surface, as a result of the irregular surface presented to the ion beam, is not sputter-cleaned as uniformly as the smooth. The specimen with $m = 1.6$ was found to have a larger average surface roughness than the specimen with $m = 1.7$, although this may be fortuitous. This dependence of Weibull modulus on roughness is similar to that observed for scratch tests of TiN (10), although the physical reason for this trend is not apparent in either case. Without a quantitative model of interface failure, it is unclear what this parameter implies about flaw size or distribution.

One could argue that the lower Weibull moduli may be attributed simply to the presence of more contamination at the interface, and not to the presence of roughness. Although in two instances a lower Weibull modulus is accompanied by a lower mean strength (Figs. 6 and 2 or 3), in another case the opposite is true (Figs. 5 and 3).

Artifacts

Several experimental artifacts can produce a nonlinear Weibull distribution. For an inverted pull-test geometry, induced torques due to the weight of the specimen can affect the measured pull-off force. If the operator does not carefully support a specimen, forces at one edge of the pin are

generated, and the measured failure stress will be less than the true failure stress. In this case, if all measured normal forces are uniformly low, a positive curvature will be obtained. This effect may explain the slight positive curvature in Fig. 2b.

That the presence of surface roughness would modify the statistics is perhaps not surprising. The more complicated topography of the rough surface could serve to broaden the range of flaw sizes present due to nonuniform cleaning. This would result in a lower Weibull modulus. However, the contribution of mechanical interlocking to adhesion may have led to a more complicated frequency distribution of the data. A knee between linear portions of the graph with different slopes indicates a bimodal distribution (16). Some of the deviations from Weibull behavior for the fracture of bones was explained in this manner (12), but this effect does not seem to have been observed in the present work.

Another cause for deviation from linearity is the presence of a threshold stress. For nonzero x_u , a graph of $\ln(-\ln[1-P(x)])$ as a function of $\ln(x)$ exhibits negative curvature. This curvature was observed for data from a specimen prepared using O_2 -ion bombardment to obtain very good film adhesion. Failure occurred when the epoxy bonding agent broke. A very tightly grouped set of strengths were obtained (85-98 MPa), and the Weibull distribution curve was nonlinear. When $\ln(-\ln[1-P(x)])$ was graphed as a function of $\ln(x-x_u)$, where x_u was chosen to be 83 MPa, the graph was essentially linear. The parameter x_u can be considered a minimum stress for failure, below which the failure does not occur.

The threshold stress x_u , the Weibull modulus m , and the characteristic stress x_0 could be determined by iteratively fitting the Weibull equation to the data. However, for the data obtained, a unique determination of the three parameters was not possible. Depending on initial starting conditions we obtained nonunique sets of parameters that all fitted the data extremely well. Unless the scatter in the data is extremely small, the three parameters seem to offer too many degrees of freedom. Outlying points, of questionable accuracy, also exert a large influence and make curve-fitting imprecise.

The increase in adhesion after repeated testing (Fig. 6 specimen) is thought to arise from the exposure to repeated heating and cooling. It may be recalled that to obtain these data, a pin is bonded to the specimen using a 150°C epoxy cure for 1800 s, the pin is pulled to failure, and the process is repeated. Thus, final data were obtained from a specimen that has been exposed to many more heating and cooling cycles than the initial data. This adhesion increase may have involved a chemical change in the contamination present at the Ag/zirconia interface. It is not known why the other specimens did not exhibit this effect, but because it occurred only with the specimen exhibiting the poorest adhesion, (6 MPa) additional contamination may have been present causing sensitivity.

The data in Fig. 3, which exhibited a slight nonlinearity, were examined. No aging effect was seen for the duration of those tests. Thus, the slight negative curvature exhibited in Fig. 3 cannot be attributed to this cause.

Characteristic Stress

The parameter x_0 is the stress at which the probability of failure is 63.2%. As expected, x_0 was always slightly higher than either the mean strength or the median strength, in which case the probability of failure is 50%.

SUMMARY AND CONCLUSIONS

The goal of this work was to determine if pull-test data from Ag deposited on zirconia could be analyzed successfully with Weibull statistics. For the Ag/zirconia film system, the data adequately fitted the Weibull distribution. The system exhibited brittle fracture with a zero Weibull threshold stress. The Weibull moduli were low and were a function of surface roughness, ≈ 2.0 for smooth specimens and ≈ 1.7 for rough specimens, but were apparently independent of adhesion strength. These results show that a similarity exists between fracture behavior measured by the pull test, the scratch test, and the tensile test, and that probabilistic analysis can successfully be applied to the pull test as a tool to investigate the nature of film/substrate failure.

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FIGURE CAPTIONS:

Fig. 1. Schematic diagram of pull-test geometry.

Fig. 2. (a) Probability distribution curve using Weibull statistics for smooth specimen sputter-cleaned with $2.8 \times 10^{20} \text{ Ar}^+ \cdot \text{m}^{-2}$. (b) Weibull distribution curve of same data.

Fig. 3. Weibull distribution curve for smooth specimen sputter-cleaned with $2.8 \times 10^{19} \text{ Ar}^+ \cdot \text{m}^{-2}$.

Fig. 4. Weibull distribution curve for glass tests.

Fig. 5. Weibull distribution curve for rough specimen sputter-cleaned with $1.95 \times 10^{20} \text{ Ar}^+ \cdot \text{m}^{-2}$.

Fig. 6. Weibull distribution curve for rough specimen sputter-cleaned with $9.5 \times 10^{19} \text{ Ar}^+ \cdot \text{m}^{-2}$.
Inset: Weibull distribution curve with subset of data discarded.

Fig. 1

Fig. 2a

Fig. 2b

Fig. 3

Fig. 4

Fig.5

Fig.6

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