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THESIS

**THE MEASUREMENT OF ADHESION
AT FILM-SUBSTRATE INTERFACES
USING A CONSTANT DEPTH SCRATCH TEST**

by

John C. Campbell

December, 1994

Thesis Advisor: Indranath Dutta

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THE MEASUREMENT OF ADHESION AT FILM-SUBSTRATE INTERFACES USING A CONSTANT DEPTH SCRATCH TEST

by

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Lieutenant, United States Navy
B.A., Cornell University, 1987

Submitted in partial fulfillment
of the requirements for the degree of

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ABSTRACT

By using a constant depth scratch test, the interfacial shear strength of various thin film/substrate interfaces can be determined. The ability to quantitatively ascertain when thin film debonding occurs has become especially important in the fields of electronics, optics, and protective coatings. A new model and experimental apparatus have been developed in order to more accurately determine thin film interfacial shear strength. While other tests are either qualitative in nature or experimentally difficult, the constant depth scratch test, which utilizes a Vickers microindenter for scratching a film/substrate system to debond the interface, produces quantitative results that are based upon a simple model. Since the depth is maintained constant during scratching, the complexity of the analytical formulation is reduced considerably, enabling the calculation of a numerical value for shear stress. Tests were conducted on chromium films on glass, gold thin films on aluminum nitride, and diamond films on aluminum nitride in order to determine and compare various interfacial shear strengths.

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I. INTRODUCTION

The ability to determine the interfacial shear strength between films and substrates has become very important in recent years. In the fields of electronic packaging, microelectronics, optics, and magnetics, thin films deposited on substrates are widely utilized. As electronic devices become progressively smaller and more complex, the usage of multilayer films on ceramic or other types of substrates has greatly increased. The revolution in integrated circuits and computer chips is based upon ensuring that the conductive thin films between the circuits remain adhered to the substrate. Thus, poor adhesion of the film can cause reliability problems, which could lead to failure of the microelectronic components. In some cases, it is necessary to purposely design interfaces to fail. Currently, the Department of Defense is trying to produce films that would debond after a certain time period for use in electronic components of military weapons designed for export. This would ensure that the weapons would have a definite shelf life. In addition, films have also been widely used as protective and decorative coatings. Protective coatings can be used to protect the substrate from abrasion, mechanical erosion, and damage. Decorative coatings are often used to enhance the commercial viability of a product. Thus, films deposited on substrates have become important for a wide array of applications; therefore, the necessity to determine film adhesion has also become extremely critical as well.

Despite the importance of film adhesion, few standard test techniques are available for quantitatively determining the shear strength of the film/substrate interface. Although numerous tests have been proposed, most are suitable only for qualitative evaluation of adhesion, or are experimentally very complex. Due to this problem, D. P. Lascurain [Ref. 1] and D. Secor [Ref. 2] introduced a constant depth scratch test for the quantification of the interfacial adhesion of film-substrate systems.

The purpose of this thesis is to continue the research started by Lascurain [Ref. 1] and Secor [Ref. 2] in developing a constant depth scratch test. Lascurain developed the original experimental apparatus and theoretical model for thin film debonding. Later, Secor modified the apparatus and incorporated the film damage mechanism of forward lateral flaking into the theoretical model, and produced preliminary experimental data using chromium thin films on glass. He also developed theoretical formulations for the use of a conical indenter instead of a Vickers pyramidal indenter.

The present thesis has two main objectives. The first objective is to complete and refine the experimental setup to enhance the reproducibility of the test results. The second objective is to demonstrate the applicability of the test to a number of different thin film/substrate systems. To this end, the interfacial shear strengths of chromium films on glass, gold thin films on aluminum nitride, and polycrystalline diamond films on aluminum nitride will be evaluated and compared.

II. TYPES OF ADHESION TESTS

In order to understand the importance of developing a new test to determine film adhesion, a review of the existing test methods is necessary. In the last thirty years, numerous adhesion tests have been proposed and studied. All of these tests are limited in their ability to effectively and quantitatively find the interfacial shear strength of film/substrate interfaces. The majority of the tests can be divided into three separate categories. The first category produces qualitative comparisons of film adhesion. The second category only qualitatively measures the interfacial film/substrate tensile strength. Most films, however, do not debond due to tensile forces, but instead interfacial shear caused by either thermal stresses or differential strains lead to film adhesion failure [Ref. 3]. To solve this problem, the last category of tests attempt to quantify the interfacial shear strength.

A. QUALITATIVE ADHESION TESTS

One of the first qualitative tests developed was the adhesion tape or collodion test, which was proposed in 1935 by Strong [Ref. 4]. This test involved applying a piece of sticky tape to the film and then removing it. By examining the tape surface for film, which had been removed from the substrate, a relative judgement of film adhesion could be made; however, this test was restricted to films with weak interfacial shear strengths [Ref. 4, 5]. Another test, called the File Test, employed a file, which could be run across the film to see if any of the film could be removed. This test was found not to work on films which were either soft or relatively thin [Ref. 6]. In another test, called the heat and quench test, the film and substrate were placed in an oven and heated to a temperature below the melting point of the film/substrate [Refs. 6,7]. Next, the film/substrate were quenched. If any debonding of the film was noted after quenching, then, adhesion was determined to be unsatisfactory. The debonding was caused by thermal stresses, which developed during quenching. All of the above tests are only qualitative in nature, and produce no direct way to quantify film adhesion. Therefore, they are limited in their ability to accurately compare different film/substrate systems.

B. MEASUREMENT OF INTERFACIAL TENSILE STRENGTH

Two different tests have been proposed to determine film adhesion by measuring the tensile strength of the interface. The pull-off test uses a wire or column which is either soldered or glued to the substrate [Refs. 7-10]. By pulling on the wire or column until the film debonds, the force of debonding can be recorded. Based on this force, the normal interfacial shear strength can be calculated. More recently, laser spallation has been used to find the normal interfacial shear strength [Refs. 7, 9]. A pulsating laser beam is directed at the substrate side of the film/substrate sample. The laser beam creates a compressive shock wave, which is transmitted through the substrate, to the film interface. At a particular laser strength, the film will debond indicating that the normal interfacial shear stress has been exceeded. The second method is especially well suited for the accurate determination of the normal interfacial shear strength. However, since shear failure is predominant in a wide array of coating substrate systems, a separate means of determining the interfacial shear strength is crucial.

C. MEASUREMENT OF INTERFACIAL SHEAR STRENGTH

The tests to measure interfacial shear strength can be broadly classified into three categories: the peel test, the indentation test, and the scratch test [Ref. 3]. The peel test is based upon the concept of removing the film from the substrate by peeling it at a constant angle [Refs. 6, 11]. The force necessary to peel the film is measured and then translated into a shear strength by a model theoretical equation. This test is extremely difficult to perform since the film must be peeled at a constant angle. If the film is brittle, it will break, and the test will not work. Thus, the film must be very ductile for this test to work correctly. Likewise, the test will not work on films less than 100 micrometers since the film will fracture instead of peeling.

Another test using an indenter has been proposed for finding the interfacial shear strength [Refs. 12-16]. The film is indented using a spherical, Vickers, or conical indenter. The vertical load for indentation is increased until the film debonds from the substrate around indenter periphery. By measuring the vertical load at which debonding occurs, the interfacial shear stress can be determined. There are several problems associated with the indentation test. The biggest problem is determining the exact moment when debonding occurs. If the substrate is transparent, then the exact moment of debonding can be observed by using a microscope under the sample. However, most substrates are not transparent, but are opaque. To solve this problem, acoustic emissions have been used to determine the exact moment of debonding. Several researchers have found it difficult to use acoustic emissions, because these signals can be masked by other noise generated from the deformation of the film and substrate during debonding [Ref. 17]. Thus, there are limitations in using this method.

Lastly, the scratch test has been proposed as a method for finding the interfacial shear strength. This test utilizes an indenter or stylus which is used to penetrate the film/substrate. The film/substrate is translated at a constant speed relative to the indenter producing a scratch. While the scratching is occurring, the vertical load of the indenter is increased at a constant rate until the film debonds. In 1950, Heavens constructed the first scratch test by using a diamond stylus with a constant increasing load to remove chromium films from glass substrates [Ref. 18]. Heavens was able to determine the critical load at which the film was removed leaving a clean track. Benjamin and Weaver continued his research and attempted to form a mathematical model for film adhesion [Ref. 19]. By using the scratch test, Karnowsky and Estil found that contamination of the substrate reduced adhesion strength of metal oxides [Ref. 20]. Weaver developed the theory of the importance of friction between the stylus and the film/substrate [Ref. 21]. By recording the tangential friction force between the indenter and the film, the critical load corresponding to film debonding could be determined [Refs. 17, 22, 23]. Butler et al. concluded that film debonding during scratching was more complicated than simply trying to quantify the stress required to remove clean tracks of film [Ref. 24]. Using a scratch test, different modes of film failure were studied by Hedenqvist et al. [Ref. 25] and by Bull [Ref. 26]. As the indenter scratched the film, compressive stresses were shown to form in front of the indenter causing the film to buckle in semi-circular flakes.

Hedenqvist et al. identified this type of film failure as forward lateral flaking [Ref. 25]. Maan and Van Groenou [Ref. 27] studied the Vickers indenter orientation during scratching, and they showed that by aligning the indenter in a leading plane orientation the compressive stresses would occur only along that plane reducing the difficulty in resolving horizontal stresses. More recently, a horizontal cell has been used to measure the tangential force along with the vertical force during scratching. Wu working at the IBM Research Division developed a more complex scratch tester, which incorporated two load cells that would accurately measure the vertical and tangential forces during scratching [Refs. 28, 29]. The interfacial shear strength could then be determined from a concentration of the vertical load and the horizontal force, which are measured as the scratch occurs. Utilizing a continuous increasing load microscratch test, Venkataraman, Kohlstedt, and Gerberich were able to quantitatively measure the strain energy release rate and the interfacial fracture resistance of carbon and nickel films on either silicon, NiO, or Al_2O_3 substrates [Ref. 30]. This enabled a quantitative measurement of adhesion. Despite these successes in quantitatively measuring shear strength, most versions of the scratch test only make a qualitative assessment of interfacial adhesion. A numerical value of the interfacial shear strength has been difficult to obtain.

There are several problems associated with the increasing load scratch test [Ref. 3]. The first problem is the same as with the indentation test. If the substrate is transparent, a microscope can be used to determine when debonding occurs. But, since few substrates are transparent, it becomes more difficult. Thus, detecting the moment of debonding is difficult. Either acoustic or time-load plots are used to detect debonding; however, with these it is extremely difficult to determine the exact time of debonding. In addition, there is a problem with junction growth, which occurs during scratching [Ref. 31]. The constant increasing vertical load leads to junction growth, which refers to the increase in the contact area between the microscopic irregularities on the surface of the film and the indenter. This junction growth causes the indenter to ride up and down during the scratching process [Ref. 31]. This makes the depth of penetration of the indenter vary continuously in an unpredictable fashion, rendering modelling of the process difficult. For thin films, this may result in significant error in the calculated interfacial shear strength. Thus, there are three basic problems with the current scratch test [Ref. 3]. These are: the exact detection of debonding, junction growth, and difficulty in modelling the test so that exact shear stress values can be calculated.

D. SUMMARY OF LIMITATIONS OF PRESENT TESTS

All of the currently available tests for determining the adhesion of films on substrates have serious limitations. Most of the early tests were only qualitative, and they produced results which were inadequate for effectively quantifying film adhesion. Other tests were then proposed to produce quantitative adhesion values for tensile interfacial strengths. Since most interfaces fail under shear, and the shear strength does not necessarily correlate with normal strength, separate means of measuring interfacial shear strength are crucial. Only three tests have been proposed for the measurement of the interfacial shear stress, which are: the peel test, the indentation test, and the constant increasing load scratch test. However, the currently available versions of all three of these tests are limited in their ability to effectively and accurately quantify the shear strength of the interface. Therefore, no current test exists to reliably quantify and compare the interfacial shear strengths for various film substrate systems.

For this reason, Lascurain [Ref. 1] proposed a new type of scratch test. This test was intended to solve the problem of detecting the exact moment of debonding, while also minimizing junction growth related problems. Since the indenter is kept at a constant depth, a more simplified geometric situation exists for modelling. The geometry of the scratch remains fixed. This reduces the complexity of the modelling process enabling the calculation of a numerical value for the shear stress.

III. CONSTANT DEPTH SCRATCH TEST APPROACH

To alleviate the problems associated with the other types of adhesion tests, the constant scratch depth method utilizes a new and distinct approach. This approach focuses on scratching the film/substrate while maintaining a constant depth. The analytical treatment of the test can be divided into two separate parts. Both of these parts when added together produce the total shear strength of the film interface.

The first part comprises indentation of the film/substrate. The film/substrate is penetrated by a diamond indenter up to the point just before the film debonds. As the indenter penetrates the film, shear stresses develop around the indenter at the film interface. This is similar to the indentation test, except that in the constant depth scratch test, the film/substrate is not penetrated to the point when debonding occurs. The second part of the test involves scratching the film/substrate horizontally. As the film is scratched, additional shear stresses develop at the interface just ahead of the indenter. These stresses along with the shear stresses developed from the indentation, cause debonding of the film from the substrate. The depth of the scratch is maintained constant as the sample is moved horizontally relative to the indenter. During scratching, the vertical and horizontal forces are measured as functions of the indenter on the sample. The measured vertical and horizontal forces along with the mechanical properties of the film and substrate can then be plugged into a theoretical model, which will be explained in more detail later on, and a numerical value for the shear strength can be calculated.

Following the test, photographs are taken of the scratch, and the morphology of the scratch is correlated by position with the horizontal and vertical load plots. This approach allows calculation of the interfacial shear strength in any given locale of the sample. The local interfacial shear strength varies significantly in most film/substrate systems, an analysis of specific areas is possible.

IV. THEORETICAL MODEL

In order to calculate the shear strength, a theoretical model was developed. The experimentally measured vertical and horizontal forces, the film/substrate mechanical properties, and the physical characteristics of the scratch were then inserted into appropriate equations. Lascurain [Ref. 1] proposed the initial model, and Secor modified it to incorporate circular forward lateral flaking [Ref. 2]. The theoretical model applied only to the debonding that occurred after plastic deformation of both film and substrate. This is known as Type III debonding. It did not fully consider other types of debonding in which only the film is plastically deformed prior to film adhesion failure (Type II). In this thesis, the model has been expanded to include Type II debonding. The model has also received other modifications to account for film buckling, and to account for non-circular forward lateral flaking .

The theoretical model is composed of two separate parts. The first part models the indentation of the film/substrate. The second part models the scratching phase of the experiment. As previously mentioned, the interfacial shear strength is a combination of the shear stresses generated during the indentation, and during the scratching. In order to understand the theoretical model calculations, an analysis of the types of debonding, the derivation of the indentation and scratch equations, and the effect of forward lateral flaking must be undertaken.

A. TYPES OF DEBONDING

Depending upon its strength, the interface may fail via one of three modes under indentation [Refs. 12, 14]. These modes are referred to as: Type I, Type II, and Type III. When a film, which was deposited on a substrate, is vertically loaded by an indenter, shear stresses develop at the film/substrate interface around the periphery of the indenter. If the shear stress of the interface is exceeded, the film will debond from the substrate. In Type I debonding, failure occurs after the elastic deformation of the film. This debonding is characterized by weak interfaces, and is not very practical for most industrial or military applications. Due to the limited practicality of Type I debonding, no theoretical model has been produced or considered.

In Type II debonding, failure occurs after plastic deformation of the film. In this type of debonding, the indenter penetrates a substantial fraction of the film thickness. Film/substrate systems which fail in Type II debonding have intermediate interfacial shear strengths.

Type III debonding requires the plastic deformation of the film and the substrate. The film is entirely penetrated, and the substrate is only partially penetrated. This type of debonding is associated with strong interfaces. Excessive shear stress is required before the interfacial shear strength is exceeded and the film debonds.

Both Type II and Type III debonding have been assimilated into the theoretical model. Different film/substrate systems fail in different modes. Some will fail in Type II, while others will fail in Type III. Type II and Type III debonding configurations with their respective indentations as seen from the sample surface are shown in Figure 4-1.

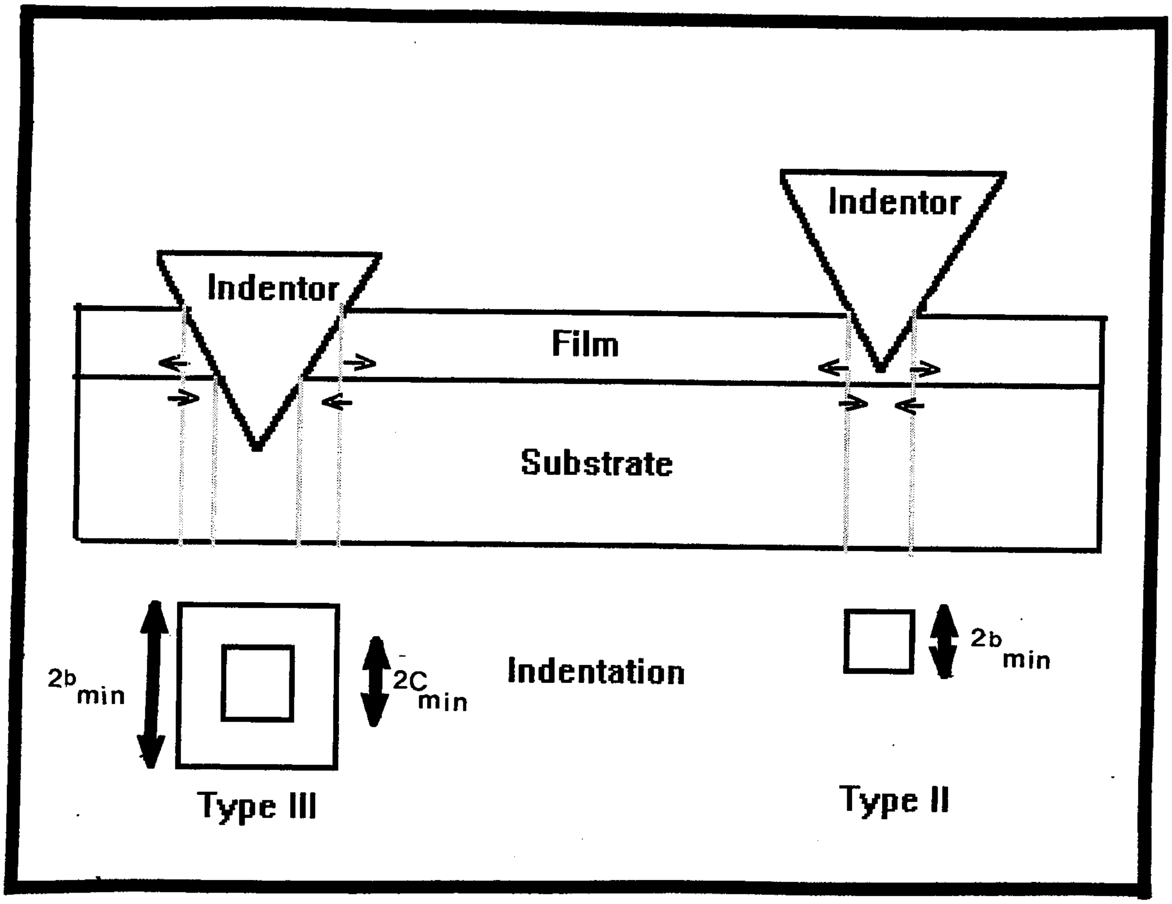

Figure 4-1: Type III and Type II configurations [Refs. 12-15].

It is critical to determine if it is necessary to penetrate the substrate to cause debonding. For this, an initial survey of the indentation failure type needs to be conducted for any given sample before subjecting it to the constant depth scratch test. Indentations to different depths (measured by a microproximitor) followed by microscopic observation can be used to determine whether the film undergoes Type II or Type III debonding. Once the approximate penetration depth required for debonding is determined, the sample is indented to a depth smaller than that required for indentation debonding, and then scratched. Following completion of the scratch, the scratch track can be examined for marks or abrasion of the substrate. The presence of which can confirm Type III configuration, whereas the absence of scratch marks on the substrate would indicate Type II failure. The effect of Type II and Type III debonding on the theoretical equations will be discussed in more detail in later sections.

B. SHEAR STRESS DEVELOPED FROM INDENTATION

A shear stress develops at the interface around the Vickers indenter when a film-substrate pair is indented. This shear stress was first quantified by Matthewson, who developed a mechanical model and equation using a spherical indenter [Refs. 12,14] Ritter et al. [Refs. 13, 15] adapted Matthewson's equation for the use of a Vickers pyramid indenter. Using Matthewson's derivation for a spherical indenter, they derived the following equation for shear stress generated at the interface along the periphery of a Vickers indenter:

$$\tau_{ihv} = \frac{\sigma_r}{\dfrac{k_1'(z)}{\Phi k_1(z)} + \dfrac{\nu t}{\sqrt{2}b_{min}\Phi^2}} \tag{4-1}$$

where: σ_r is the radial radial stress generated in the film by the indenter, t is the film thickness, b_{min} is one half the width of the Vickers indenter imprint on top of the film (see Figure 4-1), υ is the Poisson's ratio for the film, $\Phi = [6(1-\upsilon)/(4+\upsilon)]^{1/2}$, $z = \sqrt{2}b_{min} / t$, $k_1(z)$ is a modified Bessel function of the second kind of the first order, and $k_1'(z)$ is the derivative of the modified Bessel function with respect to z. It should be noted that in their original work Ritter et al. [Refs. 13, 15] used b, which is half the indenter *diagonal* imprint on top of the film, in their equations vice b_{min} , which is half the indenter *width* imprint on top of the film. By using the Pythagorean Theorem, it can be shown that $b = \sqrt{2}b_{min}$; therefore, b has been replaced by $\sqrt{2}b_{min}$ in the equations. Lascurain modified the equation by showing that $\sigma_r = -0.6875H_f$, where H_f is the indentation pressure on the film [Ref. 1]. Ritter et al. showed that the film indentation pressure for Types II and III are given by:

1) for Type III debonding:

$$H_f = \frac{W - 4c_{min}^2 H_s}{4(b_{min}^2 - c_{min}^2)} \tag{4-2}$$

2) for Type II debonding:

$$H_f = \frac{W}{4b_{min}^2} \tag{4-3}$$

where: W is the vertical load of indentation, H_s is the hardness of the substrate, and c_{min} is one half the width of the Vickers indenter imprint at the film interface [Ref. 15]. These two equations are based upon the assumption that the load W is supported by either the projected area of film (A_f) and substrate (A_s) under the indenter for Type III, or by only the projected area of the film (A_f) for Type II. Using this assumption, they derived the above equations from the following relationships: $W=A_fH_f+A_sH_s$ for Type III, and $W=A_fH_f$ for Type II. Secor modified the film indentation pressure to take into account the fact that it differs for scratching than for indenting [Ref. 2]. He showed that while scratching only three-quarters of the surface area of the indenter is actually in contact with the film/substrate; therefore, the load W is supported by only three quarters of the projected area. Thus, he slightly altered the Type III indentation hardness equation to account for this change. The modified equation is:

$$H_f = \frac{W - 3c_{\min}^2 H_s}{3(b_{\min}^2 - c_{\min}^2)} \tag{4-4}$$

Since this problem also applies to Type II indentation hardness, this same idea can be used to modify equation 4-3. The proposed equation for Type II film indentation pressure is:

$$H_f = \frac{W}{3b_{\min}^2} \tag{4-5}$$

The above model does not consider the effect of residual stresses, which may be induced in the film during deposition / coating, and which may significantly effect the calculated interfacial shear stress values. For polyimides films on soda-lime glass substrates, this deviation has been estimated by Ritter et al. [Ref. 15] to be about 15 %. In order to accurately determine the indentation shear stress, the term σ_r in Equation 4-1 must be modified to incorporate the residual stress (σ_{res}) at the film interface as follows:

1) for Type III debonding:

$$\tau_{ihv}^{III} = \frac{-0.6875\dfrac{[W - 3c_{\min}^2 H_s]}{3(b_{\min}^2 - c_{\min}^2)} + \sigma_{res}}{\dfrac{k_1'(z)}{\Phi k_1(z)} + \dfrac{\nu t}{\sqrt{2}b_{\min}\Phi^2}} \tag{4-6}$$

2) for Type II debonding:

$$\tau_{ihv}^{II}=\frac{-0.6875\left[\frac{W}{3b_{\min}^{2}}\right]+\sigma_{res}}{\frac{k_1'(z)}{\Phi k_1(z)}+\frac{\nu t}{\sqrt{2}b_{\min}\Phi^2}} \tag{4-7}$$

where σ_{res} = + *ve* for tensile in-plane stresses ($\sigma_{xx} \approx \sigma_{yy}$), and - *ve* for compressive in-plane stresses.

The above two equations are used to calculate the maximum shear stress developed at the film-substrate interface by indenting the sample. Depending on whether the given film/substrate system is subjected to Type II or Type III indentation, the correct equation must be chosen. During scratching, flaking may occur in front of the indenter. The film ahead of the indenter may undergo forward lateral flaking due to compression. If this occurs τ_{ihv} (Eqns 4-6 or 4-7), which represent a maximum value of the interfacial shear stress along the periphery of the indenter, has to be averaged over the area of the flake in order to obtain an average value of shear stress generated over the area of the interface that debonds at any given instant. This will be discussed in more detail in later sections.

The above represents the part of the total shear stress at the interface that is generated due to the vertical applied load only. The other part of the total shear stress (due to the horizontal force that is developed during scratching) has to be calculated separately, as follows.

C. SHEAR STRESS DEVELOPED FROM SCRATCHING

As the film-substrate pair is scratched, an additional shear stress develops at the interface due to the applied horizontal force (F_h). This force F_h is measured experimentally, and represents the force necessary to move the sample horizontally relative to the indenter. Since the translation stage is moving at a constant speed, a force balance equation can be utilized to determine F_{ieff} (the shear force generated at the interface ahead of the indenter due to the horizontal force).

A force balance equation has already been derived for Type III debonding. Secor and Lascurain have proposed the following force equation :

$$F_h = F_{s/ind} + P_s + F_{f/ind} + P_f + F_{ieff} \qquad \textbf{(4-8)}$$

where $F_{s/ind}$ is the shear force that acts between the indenter and the substrate, P_s is the force associated with ploughing the substrate, $F_{f/ind}$ is the shear force that acts between the film and the indenter, P_f is the force associated with ploughing the film, and F_{ieff} is the interfacial shear force at the film-substrate boundary [Refs. 1, 2, 5, 19]. However, a careful analysis of this equation suggests that the terms $F_{s/ind}$ & $F_{f/ind}$ may be redundant since the ploughing terms, which equal the indentation hardness of the phase times the projected area of the indenter on it, already incorporate shearing of the phases along the sides of the indenter. Therefore, the $F_{s/ind}$ and the $F_{f/ind}$ can be removed from Equation 4-8. The modified equation becomes:

$$F_h = P_s + P_f + F_{ieff} \qquad \textbf{(4-9)}$$

The terms P_s & P_f can be derived from the overall geometry and mechanics of the film/substrate system. In [Refs. 1, 2, 19] the force required for ploughing of the substrate was shown to be:

$$P_s = H_s\, c_{min}^2 \cot(\theta / 2) \qquad \textbf{(4-10)}$$

where: H_s is the hardness of the substrate, θ is 136^0, and $c_{min}^2 \cot(\theta / 2)$ is the area of contact between the leading edge of the indenter and the substrate projected on the vertical plane normal to the scratch direction. Likewise, the force required for ploughing of the film was also shown to be:

$$P_f = H_f\, (b_{min} + c_{min})t \qquad \textbf{(4-11)}$$

where: H_f is the indentation hardness of the film, and $(b_{min} + c_{min})t$ is the area of contact between the leading edge of the indenter and the film projected on the vertical plane normal to the scratch direction. Although the scratching process represents a dynamic situation, it can be treated as a static problem at any given instant in time. Since the translation speed remains constant, there are no problems associated with acceleration. By considering the problem as a static one, it can easily be solved by the force balance equation. Since all of the other terms in the force balance equation are either measured (such as F_h) or can be calculated (P_s & P_f), the F_{ieff} term, which is the interfacial shear force, can be determined. The interfacial shear stress (τ_{ieff}) can be found by dividing the F_{ieff} term by the area over which it acts at the film substrate interface. This area can be thought to represent the footprint of the indenter at the interface for the case when the indenter cuts through the film cleanly, or to equal the area of a flake if the film undergoes forward lateral flaking. Since $2c_{min}$ has been defined as the width of one side of the Vickers indenter at the interface, the projected area, therefore, is $4c_{min}^2$. The interfacial shear stress can then be calculated from the following equation:

$$\tau_{ieff} = F_{ieff} / A \quad \text{or in this particular case} \quad \tau_{ieff} = F_{ieff} / 4c_{min}^2 \qquad \textbf{(4-12)}$$

This depicts the interfacial shear stress caused by scratching for Type III debonding in which no flaking or buckling of the film occurs. This represents the case where the film is debonded in a nice clean scratch track created by the indenter.

This force balance equation must be revised if flaking or buckling of the film takes place. This phenomena has been observed by other researchers, and has been labelled as forward lateral flaking (FLF) [Refs. 25, 26]. Forward lateral flaking results from compressive stresses, which cause the film to buckle in front of the indenter. Secor determined that the area of the quasi-circular flakes could be approximated as:

$$A_{flf}=\Pi b_{max}^2-b_{max}^2\sin^{-1}\left[\frac{b_{min}}{b_{max}}\right]+b_{min}b_{max}\cos\left[\sin^{-1}\frac{b_{min}}{b_{max}}\right] \qquad \textbf{(4-13)}$$

where b_{max} is the flake radius, and b_{min} equals one half the width of the indenter at the film surface [Ref. 2]. The area for the quasi-circular flakes was modelled assuming that flaking occurred in front of the indenter as shown in Figure 4-2.

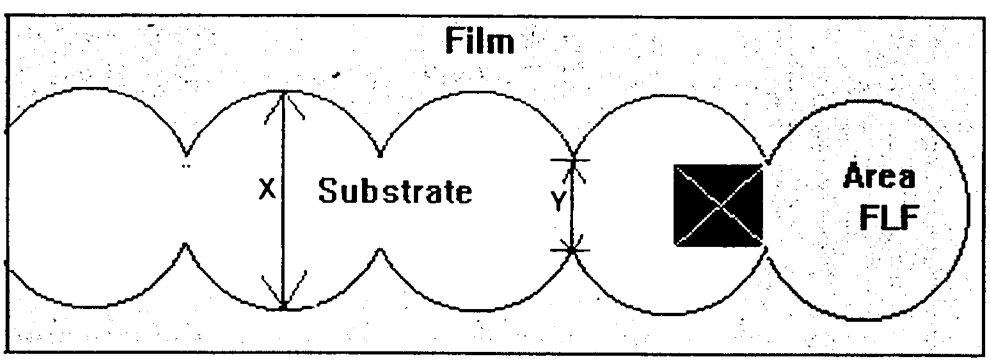

Figure 4-2: Model (forward circular flaking) where $x=2b_{max}$ and $y=2b_{min}$ [Ref. 2].

However, observations of actual scratches show that circular flaking around the indenter is not limited to this model. Other forms of circular flaking in front of the indenter (Figures 4-3 and 4-4) have been noted necessitating changes in the A_{flf} .

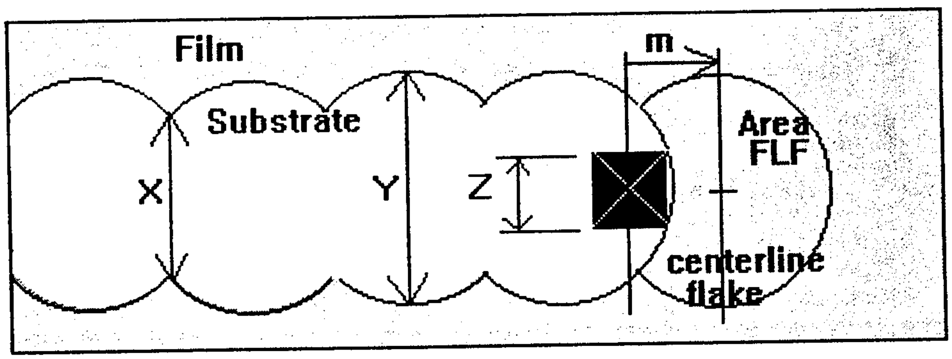

Figure 4-3: Forward lateral flaking observed in chromium films on glass and other film/substrate systems with fairly strong interfacial shear strengths, where $x=2b_{med}$, $y=2b_{max}$, $z=2b_{min}$, and m is the distance measured from the center of the indenter to the center of flake .

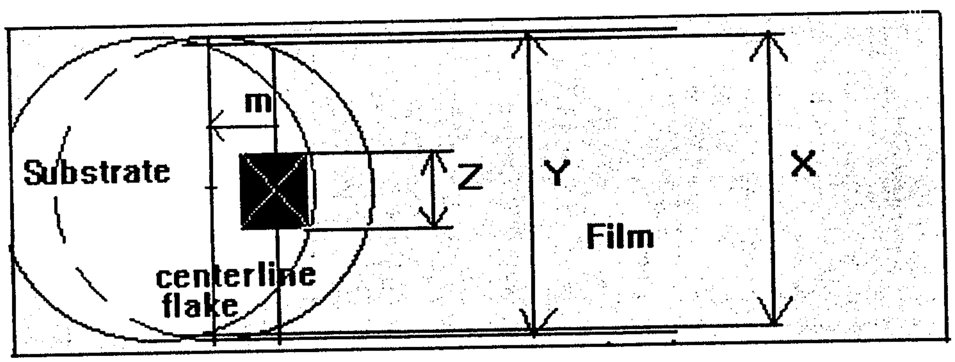

Figure 4-4: Forward lateral flaking observed in gold films on aluminum nitride, where x, y, z, and m are defined as previous figure. This type of flaking can be associated with weak film-substrate interfaces.

A more generalized form for A_{flf} can be derived to replace Equation 4-13 for determining the area of circular forward lateral flakes. This equation is as follows (see Appendix A for derivation):

$$A_{flf}=\Pi b_{\max}^{2}-2\left[b_{\max}^{2}\sin^{-1}\left(\frac{b_{med}}{b_{\max}}\right)-b_{med}b_{\max}\cos\left(\sin^{-1}\frac{b_{med}}{b_{\max}}\right)\right] \tag{4-14}$$

where b_{med} is the intermediate value for flaking as shown in Figures 4-3 & 4-4. For Type III debonding with circular forward lateral flaking, the average shear stress on the interface over the area of the flake ahead of the indenter, due to the horizontal force only, is:

$$\overline{\tau_{ieff}}=F_{ieff}/A_{flf} \tag{4-15}$$

In addition, as the film buckles and flakes, the force for ploughing of the film (P_f) must be replaced by a new buckling term (B_f). The film buckling can be modelled as simple plate buckling with one fixed end. Depending on the length of the buckled region of the film ahead of the indenter, the buckling may occur elastically or plastically. The critical stress for elastic buckling, which is based upon the standard plate buckling formula, may be expressed as follows [Ref. 32], as derived in Appendix B:

$$\sigma_{cr}^{buckle}=\left[\frac{\pi^{2}E}{(1-\upsilon^{2})}\right]\left[\frac{1}{12(L/t)^{2}}\right] \tag{4-16}$$

where: σ_{cr}^{buckle} is the critical buckling stress for the film, E is the elastic modulus of the film, υ is Poisson's ratio, t is the thickness of the film, and L is the length of the buckled film in front of the indenter. The length of buckle in front of the indenter can be derived geometrically from the measured values of b_{min} and b_{max}, which have already been defined.

The equation for the length of buckle is :

$$L=\sqrt{b_{max}^{2}-b^{2}}+b_{max} \quad \textbf{(4-17)}$$

From Euler's formula for buckling, the maximum σ_{cr}^{buckle} can not exceed the compressive yield stress (σ_{yield}) of the material [Ref. 33]. This value is often described as the *block compression allowable.* The yield stress of the film, therefore, determines the maximum permissible critical buckling stress. The value for σ_{cr}^{buckle} follows a curve which reaches a maximum at the yield strength. It is, therefore, impossible to have a critical buckling stress greater than the yield stress, since at the yield stress the material will have already buckled. The yield stress for the film can be approximated from the following [Ref. 33]:

$$\sigma_{yield} = H_f / 3.2 \quad \textbf{(4-18)}$$

If the value for the calculated critical buckling stress (Equation 4-16) is less than the film yield strength, σ_{cr}^{buckle} should be used to determine the film buckling. Likewise, if the value calculated for the critical buckling stress is more than the yield strength, σ_{yield} should be used to determine the film buckling. Using either σ_{yield} or σ_{cr}^{buckle}, the film buckling term (B_f) can be calculated. The term B_f is derived from the film buckling stress, and the projected area of front face of the indenter on the film:

$$B_f= \sigma_{cr} (b_{min} + c_{min})t \quad \textbf{(4-19)}$$

where: σ_{cr} is either σ_{yield} or σ_{cr}^{buckle} depending on the situation, and $(b_{min} + c_{min})t$ is the projected area of the leading face of the indenter and the film projected on the vertical plane normal to the scratch direction. Using B_f , the force balance equation can be changed for the Type III configuration to reflect forward lateral flaking:

$$F_h = P_s + B_f + F_{ieff} \tag{4-20}$$

When circular forward lateral flaking occurs in Type III debonding, the above equation should be used along with the area of the forward lateral flake (A_{flf}) to determine the average interfacial shear stress; hence it can be determined from $\bar{\tau}_{ieff} = F_{ieff} / A_{flf}$.

Since some film-substrate systems debond in Type II, the model must be expanded to include Type II debonding. If no flaking is occurring and the scratch track is clean, the Type II balance of force equation becomes:

$$F_h = P_f + F_{ieff} \tag{4-21}$$

This equation is similar to Equation 4-9, which applies to Type III debonding, except that the ploughing of the substrate term (P_s) has been removed. Since the substrate is not penetrated in Type II, ploughing of the substrate will not occur. The term for ploughing of the film is the same as that for Type III. The interfacial shear stress is then calculated using Equation 4-12.

In Type II, quasi-circular forward lateral flaking can also take place; therefore, the model must be expanded to account for this occurrence as well. The FLF balance of force equation becomes:

$$F_h = B_f + F_{ieff} \tag{4-22}$$

Here, B_f is the same as that for Type III with the exception that the projected area of the indenter leading edge, instead of being $(b_{min} + c_{min})t$, becomes $(b_{min} + c_{min})D$, where D is the film penetration depth. This is because in Type II configuration the entire thickness of the film is not penetrated; hence only the penetration depth (D) vice the film thickness

(t) is utilized. The equation for the Type II buckling term is modified as follows:

$$B_f = \sigma_{cr} (b_{min} + c_{min})D \tag{4-23}$$

The area of the forward lateral flake can be determined from Equation 4-14, and the average interfacial shear stress ($\bar{\tau}_{ieff}$) can be found by applying Equation 4-15.

An examination of the scratching of various film-substrates reveal that other forms of forward lateral flaking may occur other than in quasi-circular shapes. In particular, it was shown that flakes of approximately rectangular shape can develop in front of an indenter. Both Type II and Type III equations for FLF are still applicable except the area of the flake must be modified from quasi-circular to rectangular. The area of the rectangular flake can be determined by measuring its width, and then by measuring its length. The average interfacial shear stress is then determined as previously shown in Equation 4-15.

In summary, as the film/substrate is scratched, a shear stress develops at the interface. A force balance equation can be used to determine this interfacial shear stress. Depending on how the film debonds, different force balance equations must be used. The equation is different for Type II and Type III debonding. It also differs if forward lateral flaking occurs ($\bar{\tau}_{ieff}$) as opposed to no flaking at all (τ_{ieff}). In addition, the force equation for forward lateral flaking depends upon the shape of the flake. Thus, the force balance problem is complex, and consideration must be taken to ensure that the correct equation is used to determine either τ_{ieff} or $\bar{\tau}_{ieff}$. Likewise, other damage mechanisms besides forward lateral flaking may occur in other samples; hence, it may be necessary in the future to derive additional force balance equations.

D. EFFECT OF FORWARD LATERAL FLAKING

If forward lateral flaking occurs it must be taken into consideration when finding both the indentation shear stress (τ_{ihv}) and scratch shear stress (τ_{ieff}). The average values of τ_{ihv} ($\overline{\tau_{ihv}}$) and τ_{ieff} ($\overline{\tau_{ieff}}$) over the area of the interface under the flake can then be added together to determine the interfacial shear strength ($\overline{\tau_i}$). In the previous section, the effect of forward lateral flaking on deriving balance of force equations for finding $\overline{\tau_{ieff}}$ has been discussed in detail. Forward lateral flaking also slightly affects indentation shear stress. As was already stated, the value for τ_{ihv} must be averaged over the flake area if FLF is occurring. This would produce a mean value of τ_{ihv} over the area of the interface under the flake. The following equation was shown by Secor to be the average τ_{ihv} over quasi-circular flake areas [Ref. 2]:

$$\overline{\tau_{ihv}} = \frac{1}{A_{flf}} \int \tau_{ihv}(r)\, dA_{flf} \tag{4-24}$$

The function $\tau_{ihv}(r)$ reaches a maximum at the indenter, and decreases to a minimum at the far edge of the flake in the scratch direction. Due to this relationship, he showed that $\tau_{ihv}(r)$ varies according to the following equation:

$$\tau_{ihv}(r) = \sqrt{2} b_{\min} \frac{\tau_{ihv}}{r} \tag{4-25}$$

Inserting $\tau_{ihv}(r)$ and applying the limits of integration, the following equation for the average τ_{ihv} is derived as (adapted from [Ref. 2]):

$$\overline{\tau_{ihv}} = \frac{2}{A_{flf}} \sqrt{2} b_{\min} \tau_{ihv} \int_0^{\lambda_{\max}} \int_{\sqrt{2} b_{\min}}^{r_{\max}} \frac{1}{r}\, r\, dr\, d\lambda \tag{4-26}$$

where: m is the distance from center of the flake to the center of the indenter, b_{max} is the flake radius, b_{min} is one half the width of the indenter imprint on the film surface, λ is the angle used for integrating over the quasi-circular flake region, and r is the radius used for integrating over the quasi-circular flake region. As previously mentioned, the area of the forward lateral flake needs to be modified to account for flaking as shown in Figures 4-3, and 4-4. The value of m is given by the following equation:

$$m=b_{min}-\sqrt{b_{max}^2-b_{min}^2}+2\sqrt{b_{max}^2-b_{med}^2} \tag{4-27}$$

The value for λ, which is the angle used for integrating over the quasi-circular region, varies from zero to λ_{max}, and r, which is the radius used for integrating over the quasi-circular region, varies from $\sqrt{2}b_{min}$ to r_{max}, where λ_{max} and r_{max} are given by:

1) when m is + *ve* (the center of the flake is in front of the indenter when flaking occurs):

$$\lambda_{max}=\tan^{-1}\left(\frac{b_{med}}{b_{min}-\sqrt{b_{max}^2-b_{min}^2}+\sqrt{b_{max}^2-b_{med}^2}}\right)$$

$$r_{max}=m\cos\lambda+\sqrt{m^2\cos^2\lambda-m^2+b_{max}^2} \tag{4-28}$$

2) when m is - *ve* (the indenter has already passed the center of the flake when flaking occurs):

$$r_{max}=m\cos\lambda-\sqrt{m^2\cos^2\lambda-m^2+b_{max}^2}$$

$$\lambda_{max}=\pi+\tan^{-1}\left(\frac{b_{med}}{b_{min}-\sqrt{b_{max}^2-b_{min}^2}+\sqrt{b_{max}^2-b_{med}^2}}\right) \tag{4-29}$$

Figures 4-2 & 4-3 show the type of circular flaking when m is *+ve*, and Figure 4-4 shows the type of circular flaking when m is *-ve*. By simplifying Equation 4-26 and by inserting the maximum limits of integration, the equations for determining the average τ_{ihv} are as follows:

1) for flaking as shown in Figures 4-2 & 4-3 (m is *+ve*):

$$\overline{\tau_{ihv}}=\frac{2}{A_{flf}}\sqrt{2}b_{min}\tau_{ihv}\int_0^{\tan^{-1}\left(\frac{b_{med}}{b_{min}-\sqrt{b_{max}^2-b_{min}^2}+\sqrt{b_{max}^2-b_{med}^2}}\right)}\left(m\cos\lambda+\sqrt{m^2\cos^2\lambda-m^2+b_{max}^2}-\sqrt{2}b_{min}\right)d\lambda \tag{4-30}$$

2) for flaking as shown in Figure 4-4 (m is *-ve*):

$$\overline{\tau_{ihv}}=\frac{2}{A_{flf}}\sqrt{2}b_{min}\tau_{ihv}\int_0^{\pi+\tan^{-1}\left(\frac{b_{med}}{b_{min}-\sqrt{b_{max}^2-b_{min}^2}+\sqrt{b_{max}^2-b_{med}^2}}\right)}\left(m\cos\lambda-\sqrt{m^2\cos^2\lambda-m^2+b_{max}^2}-\sqrt{2}b_{min}\right)d\lambda \tag{4-31}$$

The derivation of the above equations is discussed in detail in Appendix C. It should be noted that either Equation 4-6 for Type III indentation shear stress or Equation 4-7 for Type II indentation shear stress is inserted into the above equations for τ_{ihv}. The average indentation shear stress can be calculated using this equation; however, this only applies for quasi-circular flaking, and not for other modes of forward lateral flaking.

If rectangular flaking occurs, τ_{ihv} must also be averaged over the flaking area. The average value was approximated using the following equation:

$$\overline{\tau_{ihv}}=\frac{1}{L}\int\tau_{ihv}(z)\,dz \tag{4-32}$$

where L is the length of the flake measured from the indenter to the to end of the flake in the scratch direction. Since the function τ_{ihv} (z) reaches a maximum at the indenter, and decreases to a minimum at the far edge of the flake in the scratch direction, an equation similar to Equation 4-25 can be produced:

$$\tau_{ihv}(z) = \sqrt{2}b_{\min}\frac{\tau_{ihv}}{z} \tag{4-33}$$

With the limits of integration added, the following equation can be derived:

$$\overline{\tau_{ihv}} = \left[\frac{\sqrt{2}b_{\min}\tau_{ihv}}{L}\right]\int_{\sqrt{2}b}^{L+\sqrt{2}b}\frac{1}{z}\,dz \tag{4-34}$$

The above equation can be rewritten as:

$$\overline{\tau_{ihv}} = \left[\frac{\sqrt{2}b_{\min}\tau_{ihv}}{L}\right]\left[\ln(L+\sqrt{2}b_{\min}) - \ln(\sqrt{2}b_{\min})\right] \tag{4-35}$$

As more studies are done on different film-substrate systems, different types, and shapes for forward lateral flaking may become apparent; therefore, other models for averaging τ_{ihv} over the flake area may need to be developed. It must also be remembered that when using these equations, they are only a model approximation of the flaking areas, since most flaking is neither completely circular nor rectangular. The flaking shape areas can be slightly irregular, and care must be taken to ensure that the correct formula over which to average τ_{ihv} is used.

E. SUMMARY OF THEORETICAL MODEL

The mean interfacial shear strength ($\overline{\tau_i}$) of the film/substrate pair, which is over the area of the region ahead of the indenter undergoing forward lateral flaking, is determined by adding the average shear stress ($\overline{\tau_{ihv}}$) developed at the interface under the flake due to the vertical load only, and the average shear stress ($\overline{\tau_{ieff}}$) developed in the same region due to the horizontal force. The equation for the mean interfacial shear strength is [Ref. 2]:

$$\bar{\tau_i} = \bar{\tau}_{ihv} + \bar{\tau}_{ieff} \qquad \textbf{(4-36)}$$

If no flaking is occurring, the values for τ_{ihv} and τ_{ieff} are not averaged, and the interfacial shear strength is determined from the following equation [Ref. 1]:

$$\tau_i = \tau_{ihv} + \tau_{ieff} \qquad \textbf{(4-37)}$$

The test is based upon indenting the sample up to the point just before debonding. This indentation creates a shear stress at the film/substrate interface. During subsequent scratching of the sample with the indenter, an additional shear stress is developed at the film interface. Together, these two shear components add up to equal the interfacial shear strength. By using this method, the problems associated with the other previously developed scratch tests can be avoided. Likewise, due to the constant depth, a more simplified geometric situation exists enabling an actual value for either $\bar{\tau_i}$ or τ_i depending upon the debonding situation to be calculated using either Equation 4-36 or Equation 4-37 respectively. The effects of forward lateral flaking and Type II / Type III debonding must be taken into consideration when determining the interfacial shear strength.

V. EXPERIMENTAL APPARATUS

A. EXPERIMENTAL SET UP

One of the objectives of this thesis was to modify the experimental apparatus constructed earlier [Refs. 1,2] to yield better reproducibility of results. A schematic of the modified apparatus is shown in Figures 5-1 & 5-2.

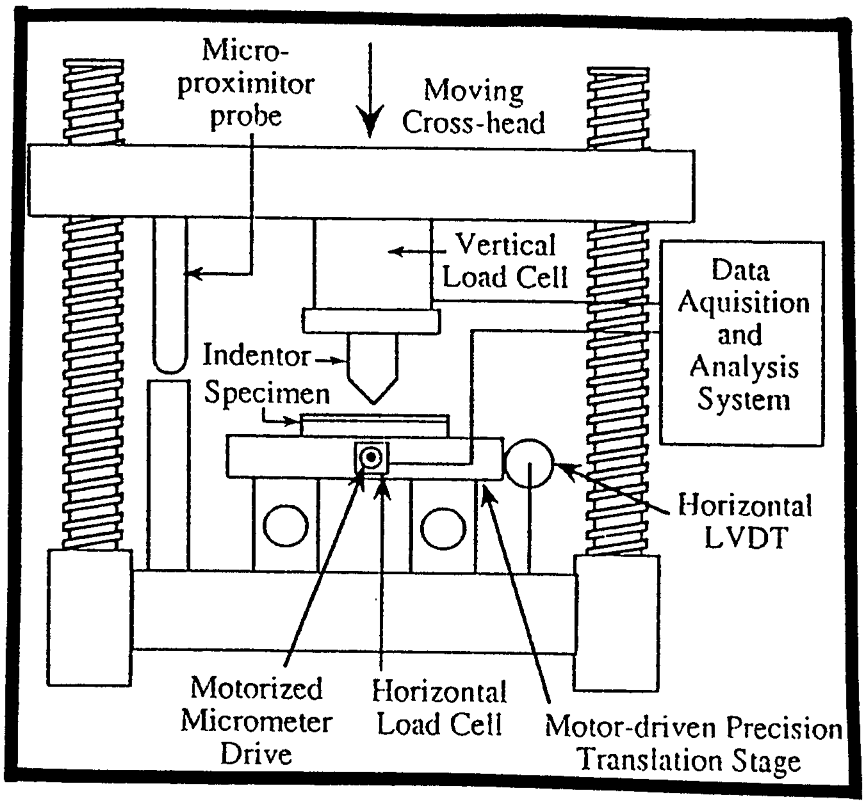

Figure 5-1: Experimental apparatus as seen from the front view [Adapted Ref. 2].

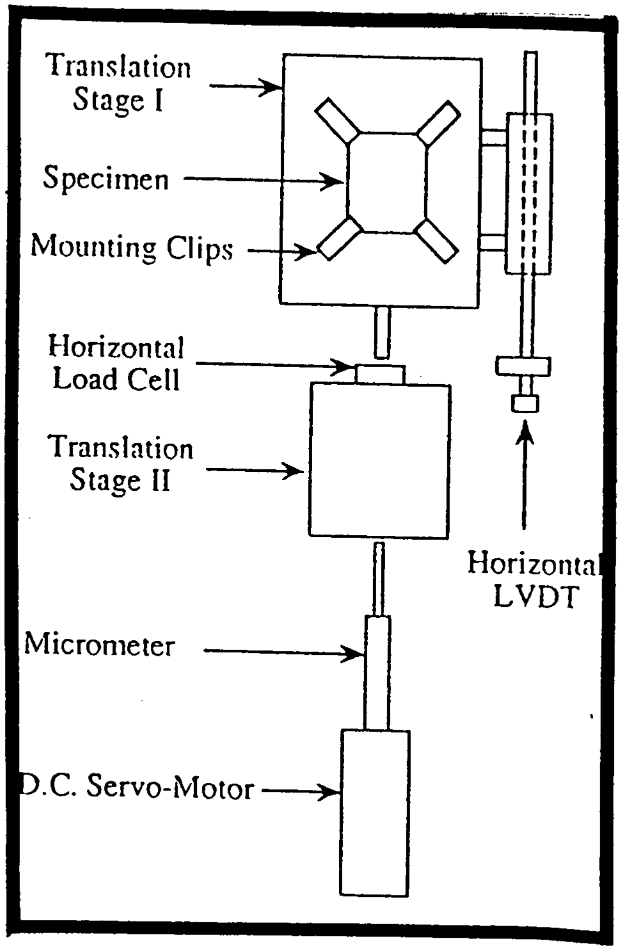

Figure 5-2: Top view schematic of experimental apparatus [Adapted Ref. 2].

To maintain the rigidity necessary for constant depth scratching, Lascurain chose a universal test frame (Instron^{tm}) as the experimental platform [Ref. 1]. The Instron ensures that little if any vertical movement or vibration of the cross-head occurs during scratching and indenting. In order for the test to produce accurate shear stress data, the distance between the indenter and sample must be rigidly maintained. The indenter and vertical load cell are mounted to the cross-head of the test frame. The use of an Instron for film/substrate indentation has been widely utilized by others for exactly the same reasons [Ref. 15]. During testing, the cross-head is manually lowered so that the indenter can penetrate the sample. In order to achieve finer penetration depth control for the indenter, an external gear arrangement with a reduction ratio of 60 to 1 reduction was added to the manual control for lowering the Instron cross-head, so that one complete turn of the control knob resulted in a cross-head movement of 0.003430 millimeters. This allows the operator extremely precise vertical movement control, which is important when penetrating thin films.

The horizontal translation stage on which the sample is mounted during scratching was modified to yield greater reproducibility. Two high precision roller bearing stages were used in tandem (Figure 5-2). One was used to mount the sample holder, which was comprised of a micrometer controlled X-Y translation stage, and on the other, a horizontal load cell was mounted. Earlier tests had shown that mounting the load cell on the same translation table as the sample stage resulted in poor baseline repeatability due to varying tension in the load cell wire, which altered the measured values of F_h . By isolating the load cell from the sample's translation stage, this problem was eliminated. During scratching, a micrometer head attached to a DC servo motor is utilized to push the first translation stage, containing the horizontal load cell, at a constant speed (0.008121 mm/sec). When the first translation stage touches the second translation stage, which is directly in front of it, it causes the second stage to move forward at the same rate. The sample, which is mounted on the second stage, then begins to be scratched by the indenter as the translation stages move forward in unison. The load cell, which is situated between the two translation stages , measures the total force required to move the sample stage horizontally. The measured force, thus, includes the frictional forces that need to be overcome to move the stage, as well as that required to scratch the sample. Consequently, for each test, a baseline representing the horizontal force required to overcome the frictional resistance to the movement of the stage is subtracted off the total force data. This is discussed further subsequently.

Two load cells are utilized in the experimental set up. Both cells are designed to measure compressive loads through strain changes in an internal Wheatstone bridge. The vertical cell is attached to the indenter, which in turn is connected to the Instron cross-head. This cell measures the vertical force (W), which is necessary in determining indentation shear stress and indentation hardness of the film. The horizontal cell measures the force necessary to move the sample during scratching. This force (F_h) is necessary in calculating either τ_{ieff} or $\overline{\tau_{ieff}}$. During scratching, it was found that the vertical load reading was affected slightly by a moment causing a small rise in measured load as the indenter pushed horizontally by the moving sample. The change in the vertical force reading was found to be a definable function of the horizontal force. The functionality was determined, and was utilized to correct the vertical force (W) for the moment effect.

A linear variable differential transducer (LVDT) is attached to the sample translation stage to measure horizontal displacement as a function of time. This enables the vertical (W) and horizontal (F_h) forces to be accurately correlated with the scratch distance as a function of time. To measure the penetration depth of the indenter into the film/substrate, a microproximitor probe, which is attached to the cross-head, is used. The probe functions by generating an eddy current between itself and an adjustable flat surface attached to the sample platform. The changes in this induced electrical field, which correlate with the vertical separation between the probe and the flat surface, are utilized to measure the penetration depth.

Four different measurements are made during scratching. The voltages form the horizontal load cell, vertical load cell, LVDT, and microproximitor probe are recorded by a multi-channel 5½ digit voltmeter / data acquisition control unit (HP 3852), which is interfaced with a personal computer via a IEEE-488 bus. Several programs in BASCIA have been written to control the data acquisition, to store revelant experimental information, and to calculate the interfacial shear strength form the measured data. These are discussed subsequently.

After completing the scratch test, an optical microscope with photographic capabilities is used to examine and obtain a photographic record of the scratch track. The width of the indenter imprint on the surface (b_{min}), flake radius for circular forward lateral flaking, and the length and width of the flake for rectangular forward lateral flaking are subsequently measured from the photographs of the scratch (1000 times magnification) using a pair of digital vertical calipers.

In order to ensure the reliability of the experimental apparatus, several tests were conducted. Since roller bearings are used in the translation stages, it is critical to ensure that the force necessary to push them is repeatable. Several tests on the unloaded translation stages have been conducted, and they show that the roller bearings have a high precision and the force necessary to push them is reproducible. Since the scratch test involves indenting film/substrates at varying loads, it is also important to determine the frictional effects, if any, that varying vertical forces have on the measured horizontal force. During scratching of a sample with a vertically applied force of W, the total measured horizontal force is given by:

$$F_h^{total} = F_h^0 + \mu W + F_h \qquad \textbf{(5.1)}$$

where: F_h^0 equals the force required to move the horizontal stage without an applied vertical force, μ is friction coefficient of the roller bearings, and F_h equals the ploughing/shearing/buckling force required during scratching of the sample. As shown by Figure 5-3, different weights have little effect on the force necessary to move the sample translation stage. This suggests that the coefficient of friction (μ) is extremely small, and that Equation 5-1 can be modified to:

$$F_h^{total} \approx F_h^0 + F_h \qquad \textbf{(5-2)}$$

Therefore, prior to lowering the indenter and penetrating the film/substrate system for scratching, a baseline is run to determine F_h^0 as a function of table position which can then be subtracted from the horizontal force measured during scratching to yield F_h . Lastly, to ensure that constant depth is maintained during scratching, several tests have been run to confirm that the distance between the indenter and sample platform remain constant. This was done by scratching glass to observe the width of the scratch track and vertical load changes with scratch distance. The platform was then properly shimmed in order to ensure that the indenter penetration depth remains constant during scratching.

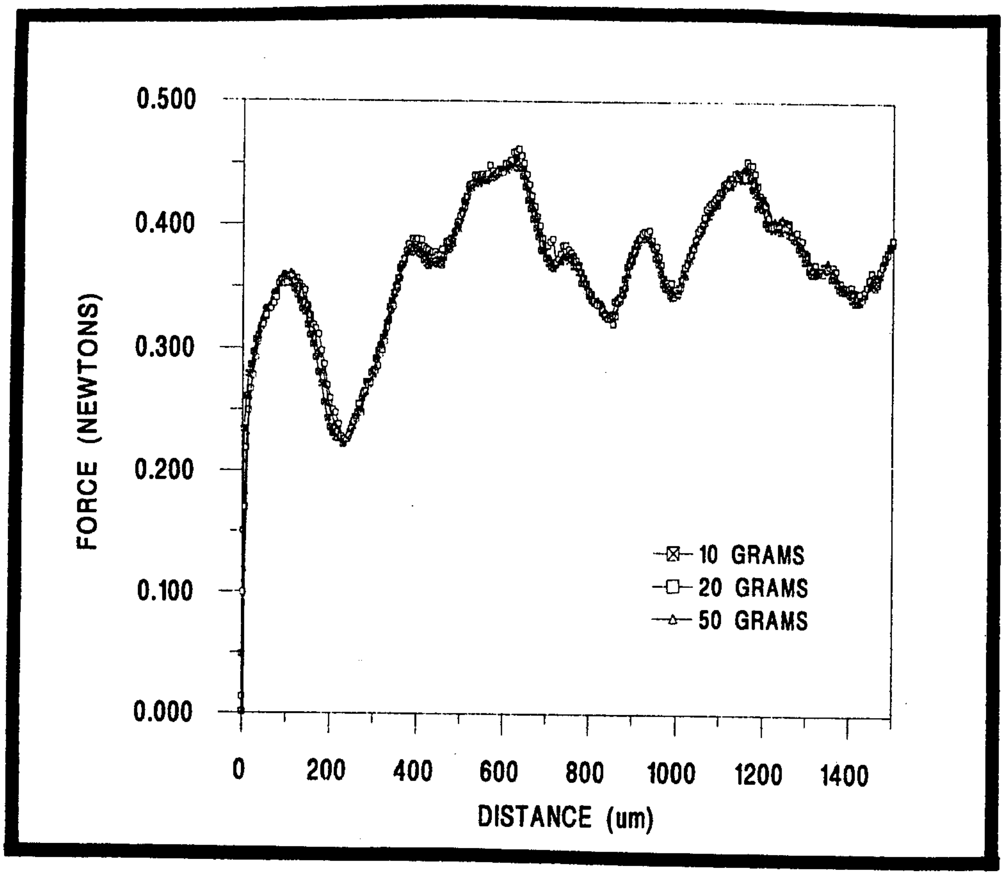

Figure 5-3: There different runs with applied loads (10, 20, and 50 grams) showing the force necessary to move the sample translation stage.

B. EXPERIMENTAL PROCEDURE

The method for conducting the scratch test is relatively simple and straight forward. The test method can be divided into seven different segments. These segments are:

1) sample preparation
2) baseline generation
3) determination of microproximitor probe reference voltage
4) penetration of film/substrate
5) actual scratching of sample
6) analysis of the scratch track morphology (photo microscopy)
7) final calculation of shear strength.

To accomplish these tasks, five different BASIC programs (see Appendix D) for an IBM personal computer were developed.

Before beginning the scratch test, the sample and test apparatus were properly prepared. The samples were carefully cleaned with soapy distilled water to remove any debris on the film surface. Extreme care was taken to ensure that the film was not damaged during cleaning. After cleaning the sample, it was rinsed thoroughly with distilled water, followed by ethanol, and was dried using a low heat source blower-dryer. It is important to ensure that the film surface is clean since foreign particles on the surface can affect the scratching process. The sample is then sprayed with a light coat of 10WD-40 oil. The oil helps reduce adverse effects related to friction (such as heating, rise in F_h due to excessive yielding and shearing of sample aspertities with the indenter, etc.) during scratching and improve test repeatability [Ref. 7]. Since the oil is fluid, it has no effect on the measured vertical force (W). Besides sample preparation, the experimental apparatus was carefully prepared for the test as well. The indenter diamond tip was thoroughly cleaned to remove any particulate debris from prior scratches adhering to the diamond tip. The diamond tip was also checked for correct alignment to ensure that one face of the diamond pyramid was oriented along the scratch direction.

The sample was then mounted in the experimental apparatus. Before lowering the indenter to penetrate the sample, a baseline representing the variation F_h^0 with table position was run. Two programs were used in conjunction with the experimental apparatus for determining F_h^0 (see Appendix D for programs) . The first program was *INITIAL1.BAS*, which found the initial horizontal load cell and LVDT values by averaging ten voltage readings obtained from the Hewlett-Packard 3852 DAQ / Control Acquisition Unit. These values were then inserted into the second program (*COLLECT1.BAS*), which was used to collect the horizontal and LVDT voltage readings during sample stage translation. In the *COLLECT1.BAS* program, the initial readings of horizontal force and LVDT displacement were then subtracted off the measured values obtained during sample stage movement. This baseline data set was then saved as BASE.DAT for later use.

After completing the baseline, the translation stages were pushed back to the initial start position. Stoppers were installed in the apparatus to ensure that on each test the start and end positions remain exactly the same to ensure repeatability. The Instron crosshead, on which the indenter is attached, was then slowly lowered manually by using the external fine adjustment control. During lowering, the program *INDENT1.BAS* was utilized to monitor the change in the vertical voltage load cell (W). The program was designed to make the computer beep when it recorded a voltage rise of .0001 volts in the cell output (an applied load of 0.2308 grams) indicating that the indenter has touched the sample surface. Upon hearing the beep, the lowering of the indenter was halted immediately, and the voltage reading of the microproximitor probe at the time of the beep was recorded. Since the vertical voltage was observed to rise rapidly relative to the sampling rate upon touching, the indenter would typically slightly penetrate the sample before the beeping occurred. To circumvent this problem and establish an accurate reference microproximitor voltage corresponding to the condition when the indenter just touched the sample surface, the microprox voltage two sampling points prior to the initial beep was assumed to be the initial value. It was experimentally determined that this made the instantaneous voltage at the exact moment of initial contact between the indenter and sample fairly reproducible.

With the reference voltage (microproximity zero setting) determined, the program *Pent1.BAS* was used to calculate the microproximitor voltage change necessary for the desired penetration depth. The indenter was then carefully lowered further with the fine external manual control to obtain the desired penetration depth. Upon reaching this depth, the program *Pent1.BAS* caused the IBM computer to produce an audible beep. Upon hearing this beep, the lowering the indenter was halted. After the microproximitor voltage settled out, the program displayed the actual penetration depth achieved during indentation in micrometers.

If the penetration depth matches the desired depth, the next phase of the test can then be performed. The next phase of the test involved performing the actual scratch test. The motor for moving the translation stages was started once again, and the film/substrate pair began to be scratched by the indenter. As during baseline generation, the program *INITIAL1.BAS* was used to obtain the initial voltage values from the microprox, horizontal load cell, vertical load cell, and the LVDT, which were then inserted into the program *COLLECT1.BAS*. During scratching, *COLLECT1.BAS* was used to record the voltage changes of the vertical and horizontal load cells, LVDT, and microproximitor probe. The scratch data was then recorded in a data file named RES.DAT.

After completing the scratch, the indenter was raised, and sample was removed from the experimental apparatus. Using a microscope, several photographs were taken of the scratch track along its length. The pictures were aligned so that scratch distance could be measured, and so that individual regions along the track could be accurately correlated to the horizontal and vertical load plots recorded during the test. Using a micrometer, the width of the scratch track (b_{min}) was measured. If forward lateral flaking occurred, the radii of individual flakes were also measured. These measured values were then correlated with the load plots.

The program *EVALUAT.BAS* was used to calculate the interfacial shear strength by using the horizontal/vertical load plots and the measured values obtained from examining the morphology of the scratch track. Before using this program, it was ensured that both the baseline and result data have exactly the same number of initial sampling points. Even though the stopper ensures that the film/substrate translation distance is the same, it does not ensure that the number of acquisition data points prior to translation stage movement is the same. This is due to the fact that the data acquisition system is started manually. Therefore, there could be a slight time offset between the baseline and result data. Secor [Ref. 2] noted that this offset existed and showed that by plotting the BASE and RESULT data together verse time a correction could be made. In Figure 5-4 this horizontal offset can clearly be seen. By counting the number of offset points, the BASE curve can be shifted so that it starts at exactly the same point as the RESULT curve. Using the program *EVALUAT.BAS*, the BASE data file is shifted by the horizontal offset, and then subtracted off the RESULT data file. As previously mentioned, the vertical force (W) must also be corrected for moment. The program automatically makes this correction.

The two load plots (W with moment correction, and F_h with the baseline subtracted) were then used to calculate the interfacial shear strength. The mechanical properties of the film and substrate, the track width (b_{min}), and the flake radius (when FLF occurred) were entered into *EVALUAT.BAS*. The program was designed so that the operator can choose between the Type II and Type III configurations for the calculations. It also allows the operator the choice of imputing whether debonding occurs by forward lateral flaking or no forward lateral flaking. It further subdivides forward lateral flaking into circular or rectangular. The interfacial shear strength (τ_i) was then determined by adding together the calculated τ_{ihv} and τ_{ieff} shear stresses.

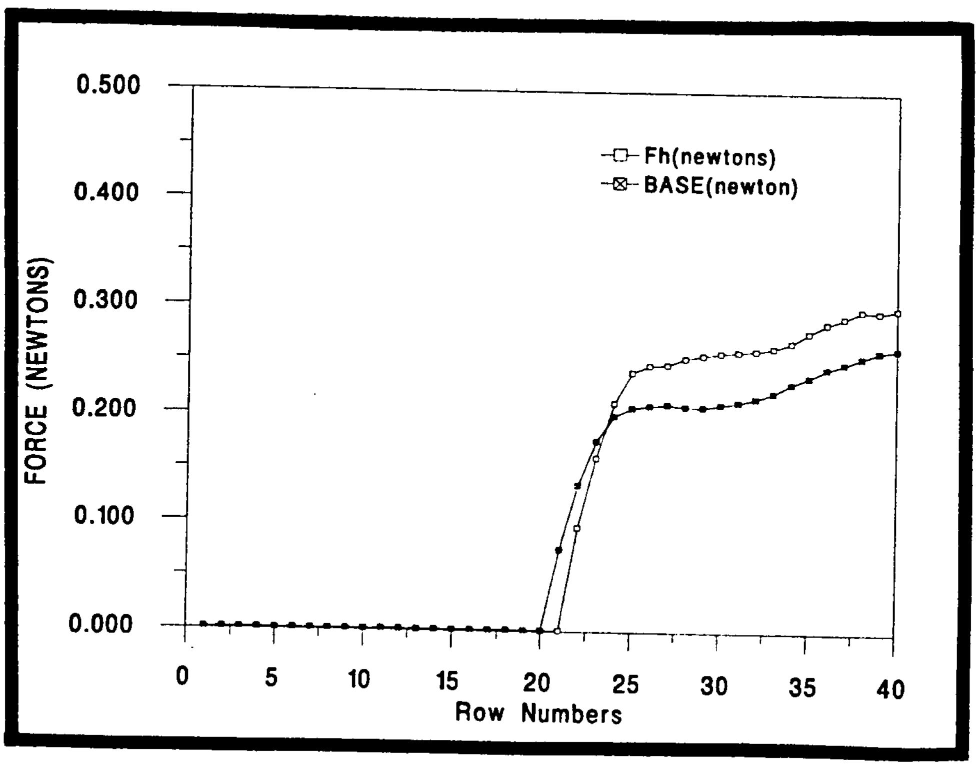

Figure 5-4: This graph shows the offset between the baseline and the result data. For this particular example the offset is equal to one data point. The horizontal axis (row numbers) represents individual data acquisition points.

VI. RESULTS AND DISCUSSION

Three different film/substrate systems were tested using the constant depth scratch test. They are listed below in Table I.

SAMPLE	SUBSTRATE	FILM	FILM THICKNESS	DEPOSITION CONDITION
1	**Soda Lime Glass**	**Cr**	**2554Å**	**Thermal Evaporation, 5×10^{-1} Torr, Substrate baked at 300°C**
2	**Soda Lime Glass**	**Cr**	**3436Å**	**Thermal Evaporation, 5×10^{-1} Torr, Substrate baked at 300°C**
3	**AlN**	**Au**	**3280Å**	**Thermal Evaporation, 5×10^{-1} Torr, Substrate baked at 300°C**
4	**AlN, coated with 64Å Cr**	**Au**	**3280Å Au + 64Å Cr**	**Thermal Evaporation, 5×10^{-1} Torr, Substrate baked at 300°C**
5	**AlN**	**Polycrystalline Diamond**	**15-20µm**	**Microwave plasma assisted chemical vapor deposition from CH_4/H_2 (Diamond crystallite size of 5µm)**

Table I: Film/substrate samples used for testing.

Each sample was scratched several times to determine the appropriate penetration depth of the indenter into the sample that gave debonding of the film from the substrate. This was visually apparent in the form of flaking of the film from the substrate. Typical appearances of the scratch for each sample are shown in Figures 6-1 to 6-10. A typical plot of the vertical and horizontal forces (W & F_h) during scratching is shown in Figure 6-11 for sample #1.

Following collection of data, the results were analyzed in regions along the scratch track that resulted in distinct flaking indicative of certain interfacial debonding. These regions were identified from a series of photographs (1000×magnification) along the scratch track, starting from the beginning of the scratch. The distance of the desired region for analysis was measured from the photographs, and the data corresponding to this distance were analyzed. Table II, on the following page, lists the input data, along with the results of the analysis for the various samples. For each sample, at least two regions were analyzed. The location of these regions from the beginning of the scratch are also indicated in Table II. Figures 6-1 to 6-10 show the appearance of the scratch corresponding to these regions. Forward lateral flaking was observed in samples 1-4, and the mean interfacial shear strength ($\bar{\tau_i}$) was calculated over the flake area in front of the indenter. In sample 5 (polycrystalline diamond on AlN), no flaking was observed and τ_i was calculated. Samples 1, 2, and 5 debonded in the Type III configuration, while samples 3 & 4 debonded in the Type II configuration. As shown in Table II, there can be significant variation in the measured values of $\bar{\tau_i}$ or τ_i . It should be noted that the residual shear stress induced in the film during deposition was not taken into account when calculating $\bar{\tau_i}$ or τ_i for the above samples (residual shear stresses were assumed to be zero). As shown in Equations 4-6 & 4-7, the values for τ_{ihv} (and hence $\bar{\tau_{ihv}}$) could be somewhat different if residual stresses were determined and used in the calculation. A tensile σ_r would reduce the calculated value of τ_{ihv} (and hence $\bar{\tau_i}$ and τ_i), whereas a compressive σ_r would increase $\bar{\tau_i}$ and τ_i .

Sample	Distance (μm)	b_{min} (μm)	b_{max} (μm)	b_{med} (μm)	W (N)	F_h (N)	Depth μm	H_s (GPa)	υ_f	E_f (GPa)	t (μm)	$\bar{\tau}_{ihv}$ or τ_{ihv}	$\bar{\tau}_{ieff}$ or τ_{ieff}	$\bar{\tau}_i$ or τ_i
1	264 to 305	3.99	9.96	4.00	0.2133	0.0470	1.62	5.7	0.31*	215.82**	0.2554	861	100	961
	508 to 528	3.99	9.50	4.25	0.2027	0.0396						661	68	729
2	81 to 150	4.07	8.25	4.15	0.2050	0.0317	1.64	5.7	0.31*	215.82*	0.3436	638	49	687
	912 to 962	4.07	8.18	6.79	0.2286	0.0339						898	28	926
3	313 to 324	2.53	7.50	7.06	0.066	0.0157	0.24	7.35**	0.42*	78.28*	0.3280	37	49	86
	91 to 113	2.15	10.10	2.20	0.040	0.020						10	53	63
4	31 to 38	2.03	3.80	-	0.050	0.0066	0.21	7.35**	0.42*	78.28*	0.3344	909	46	955
	1180 to 1210	2.13	8.22	4.30	0.0577	0.0167						515	65	580
5	24 to 38	47.15	-	-	10.211	1.106	19.0	7.35**	0.47*	112.82*	15.0	725	529	1254

Table II: Data from samples used to calculate interfacial shear strength (τ_i or $\bar{\tau}_i$); (H_s = hardness of substrate, υ_f = Poisson's ratio of film, E_f = Elastic modulus of film, t = film thickness); (*is obtained from [Refs. 34-36], **is obtained from [Refs. 37,38]).

Figure 6-1: Optical photograph (1000 × magnification) of sample 1 (Cr / soda-lime glass) which includes the region 264-305 μm. This particular region is shown above.

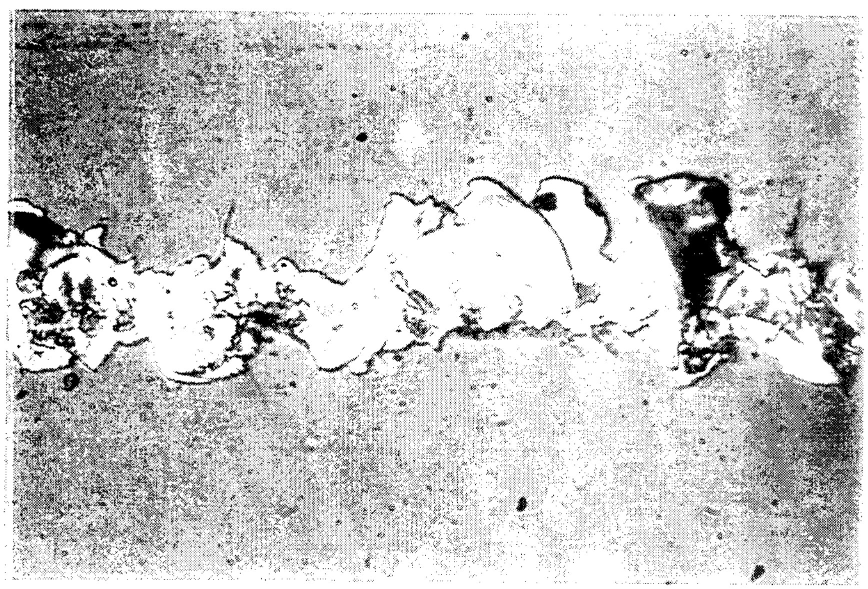

Figure 6-2: Optical photograph (1000 × magnification) of sample 1 (Cr / soda-lime glass) which includes the region 508-528 μm. This particular region is shown above.

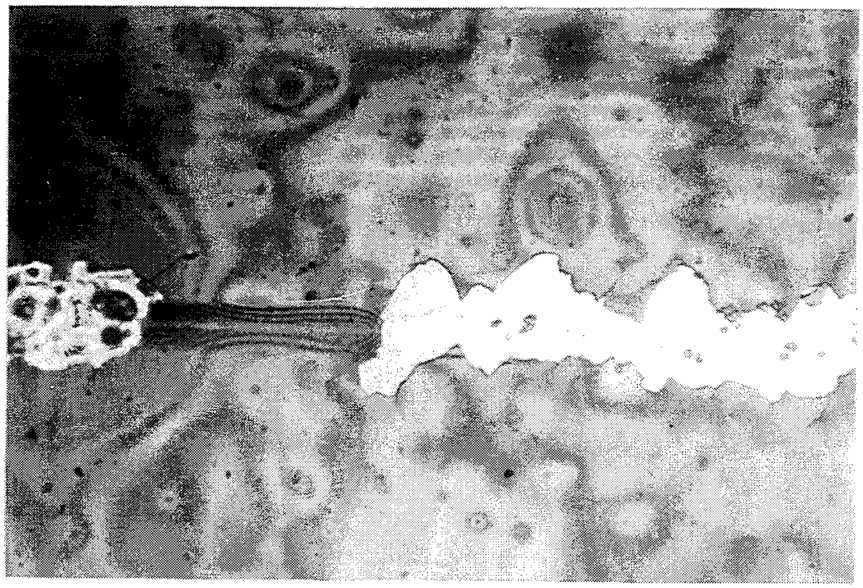

Figure 6-3: Optical photograph (1000 × magnification) of sample 2 (Cr / soda-lime glass) which includes the region 81-150 μm. This particular region is shown above.

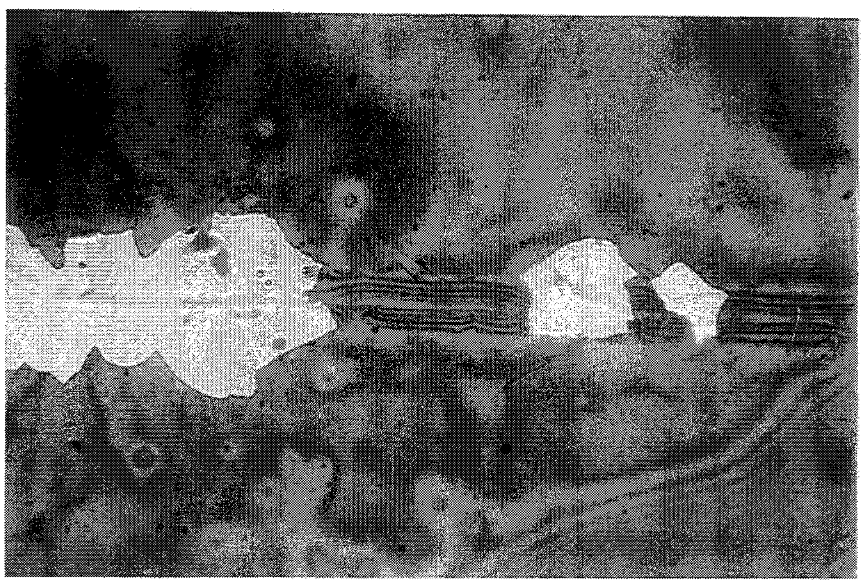

Figure 6-4: Optical photograph (1000 × magnification) of sample 2 (Cr / soda-lime glass) which includes the region 912-962 μm. This particular region is shown above.

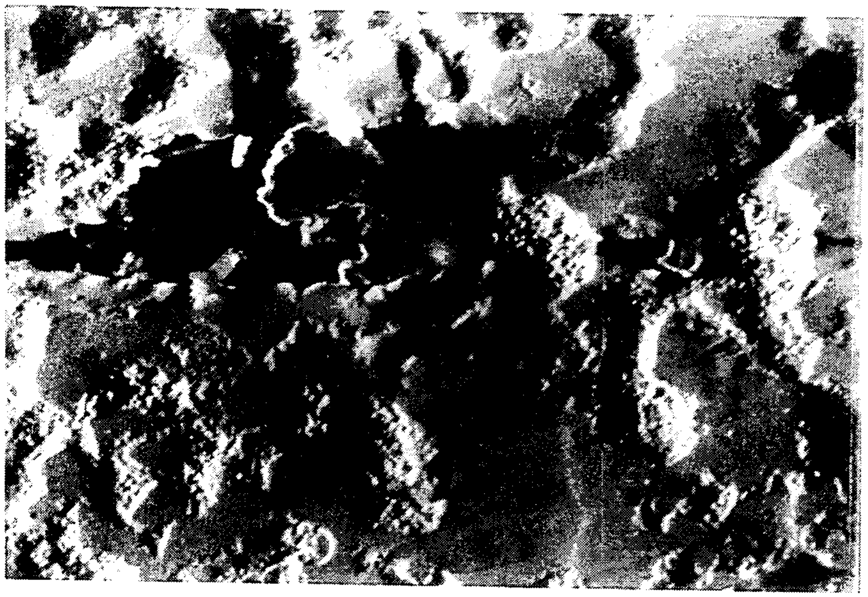

Figure 6-5: SEM micrograph (1003 × magnification) of sample 3 (Au / AlN) which includes the region 313-324 μm. This particular region is shown above.

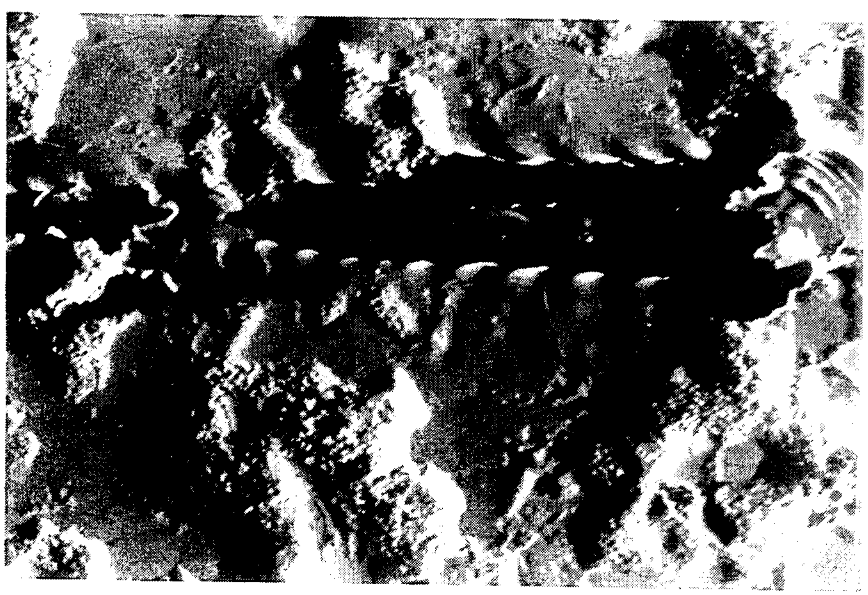

Figure 6-6: SEM micrograph (1003 × magnification) of sample 3 (Au / AlN) which includes the region 91-113 μm. This particular region is shown above.

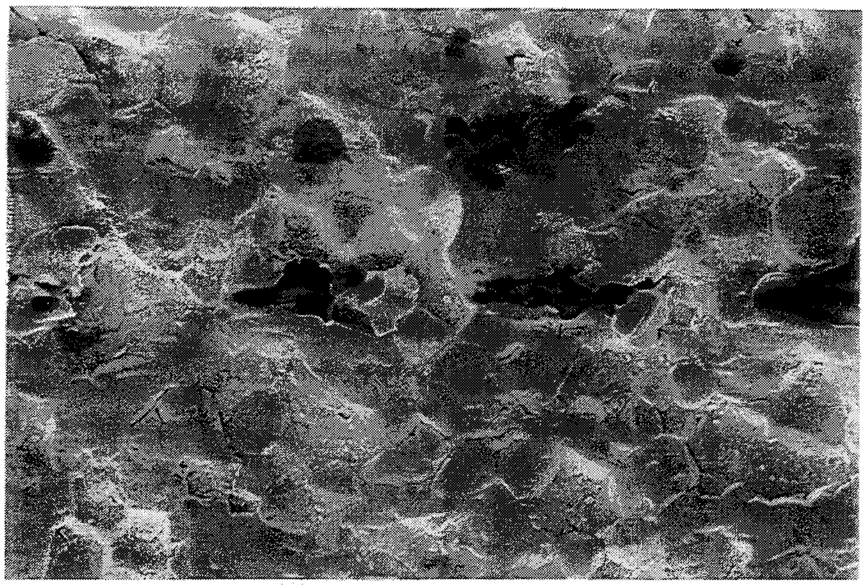

Figure 6-7: SEM micrograph (1003 × magnification) of sample 4 (Au-Cr layer / AlN) which includes the region 31-38 μm. This particular region is shown above.

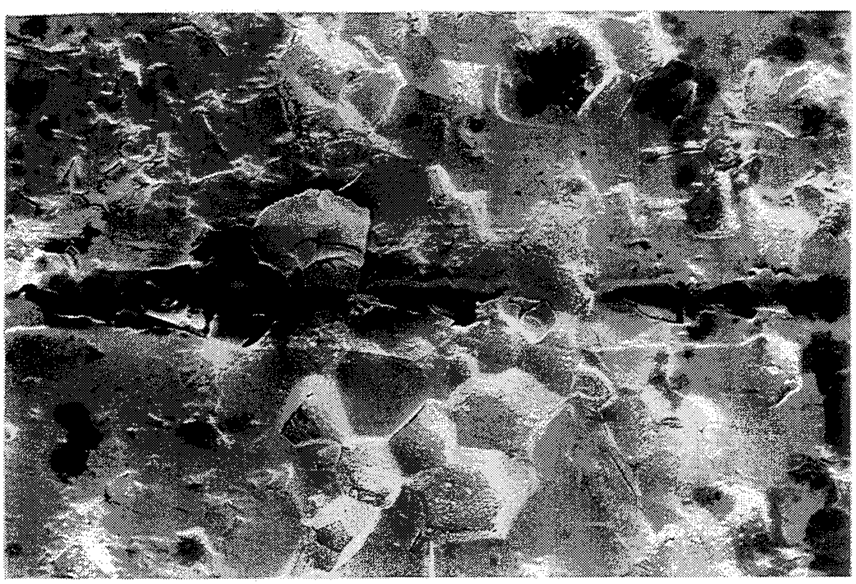

Figure 6-8: SEM micrograph (1003 × magnification) of sample 3 (Au-Cr layer / AlN) which includes the region 1180-1210 μm. This particular region is shown above.

Figure 6-9: Optical photograph (800 × magnification) of sample 5 (Diamond / AlN) which includes the region 24-38μm. This particular region is shown above.

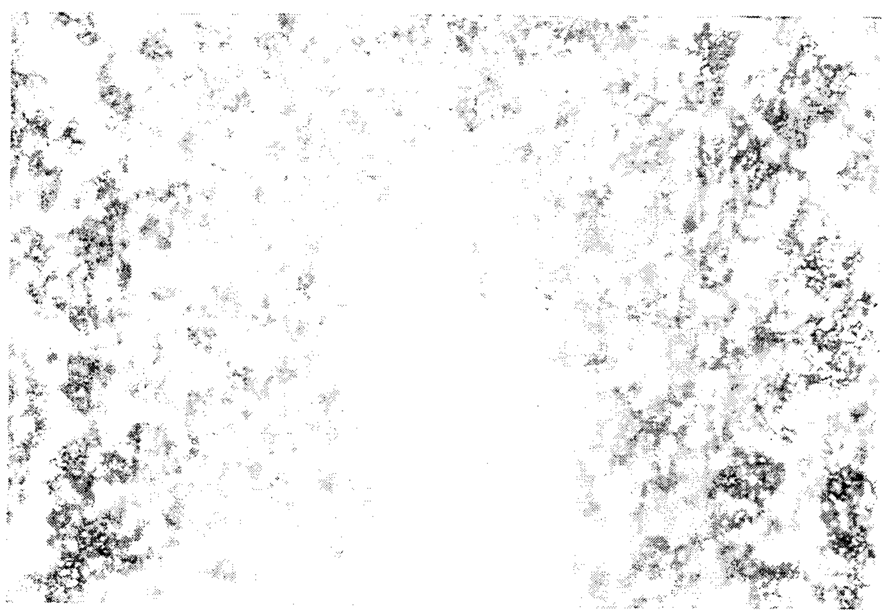

Figure 6-10: Optical photograph (800 × magnification) of sample 5 (Diamond / AlN) which includes the region 24-38μm. This particular region is shown above.

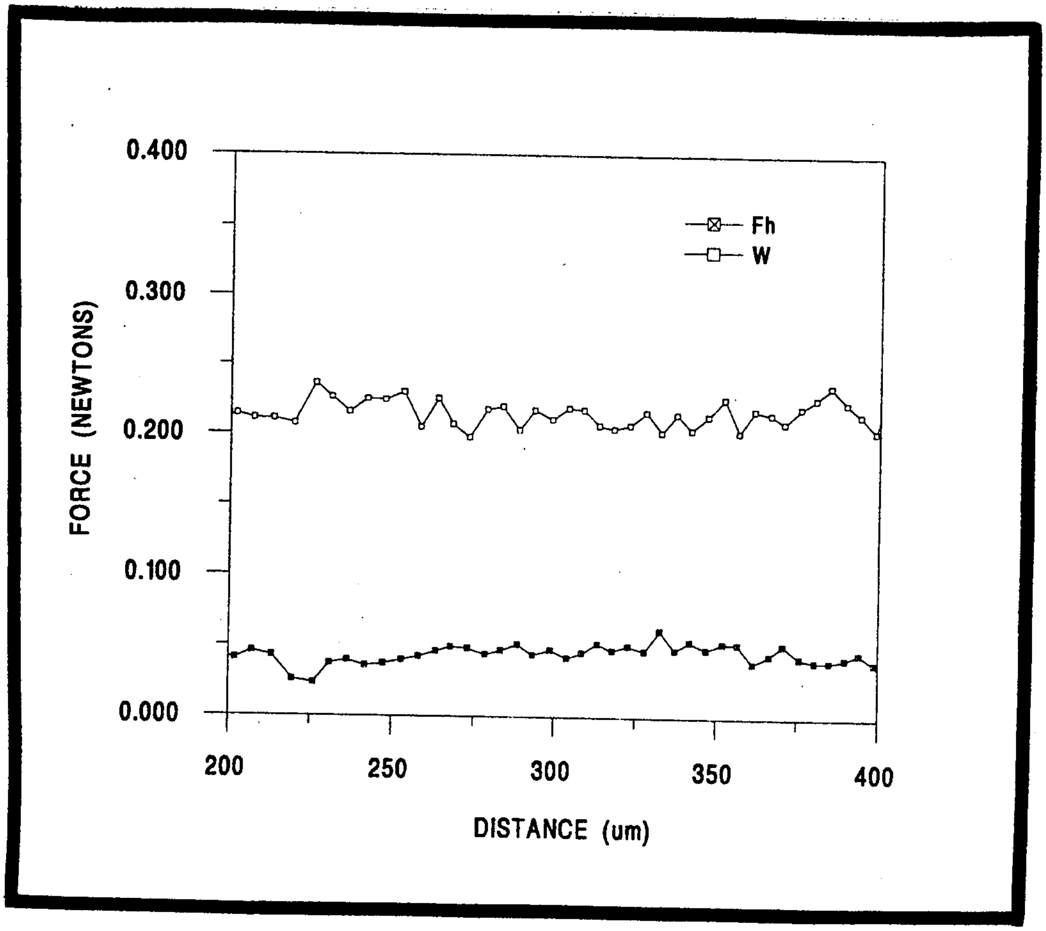

Figure 6-11: A plot of the vertical and horizontal forces (W & F_h) for sample 1 (Cr /soda lime glass).

VII. CONCLUSION AND RECOMMENDATIONS

By using the constant depth scratch test, a quantitative value for the adhesion of chromium films on glass, gold films on aluminum nitride, and polycrystalline diamond films on aluminum nitride were determined. While other tests are either only qualitative or experimentally difficult, the constant depth scratch test is simple to perform and produces quantitative results that are based upon a geometrically simple mathematical model. From the above results, the applicability of this test to a wide range of film/substrate systems seems plausible. Further testing is necessary to verify its validity.

There are several possible modifications to the experimental apparatus that would improve the accuracy and simplify the operation of the test. Instead of conducting preliminary scratch tests at different penetration depths in order to determine the necessary depth for flaking, a continuously increasing load scratch test similar to that used by Venkataraman, Kohlstedt, and Gerberich [Ref. 30] could be incorporated into the present test. This would provide a mechanism for easily determining the necessary penetration depth. It has been shown in [Ref. 30] that a sudden drop in the vertical load during the continuously increasing load test corresponds to the start of flaking. By correlating this drop with the microproximitor, an accurate penetration depth could be found. The constant depth scratch test could then be performed using this depth.

Another possible modification to the apparatus is the replacement of the microproximitor with a device that would measure the changes in penetration depth during scratching. The microproximitor determines the penetration depth at the start of the scratch test, but does not determine the depth while scratching is occurring. Since most film/substrate surfaces are not flat, it is necessary to account for the effect of sample surface roughness. A laser based optical measuring device is a possible candidate for this.

APPENDIX A - DERIVATION OF FORWARD LATERAL FLAKE AREA

The area of a circular forward lateral flake can be calculated by the following equation (adapted from Ref. 2):

$$A_{flf}=\Pi b_{\max}^{2}-2\left[b_{\max}^{2}\sin^{-1}\left(\frac{b_{med}}{b_{\max}}\right)-b_{med}b_{\max}\cos\left(\sin^{-1}\frac{b_{med}}{b_{\max}}\right)\right] \tag{A-1}$$

The shaded area in Figure A-1 is the circular flake removed in front of the indenter during scratching. The shaded area (A_{flf}) can be determined by using simple geometry in conjunction with the measured parameters b_{max} and b_{med}.

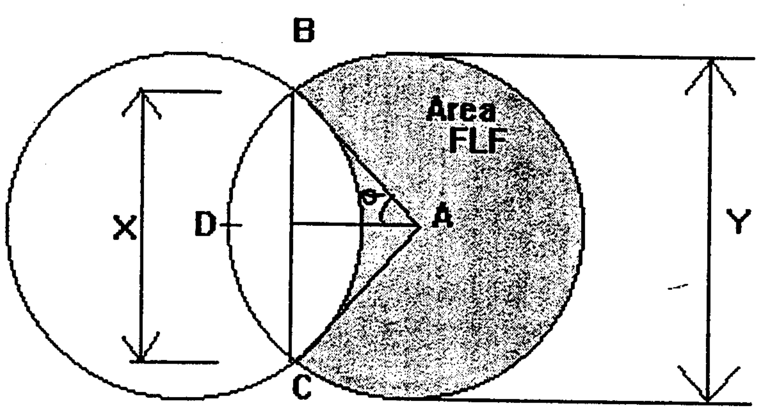

Figure A-1: Shaded area is region removed during forward lateral flaking, where $x = 2b_{med}$ and $y = 2b_{max}$.

The shaded area can be determined by the following:

$$A_{flf} = A_{circle} - 2\,(A_{circular\ sector\ (BACDB)} - A_{triangle(BACB)})$$

where:

$$A_{circle} = \pi \, b_{max}^{\;2}$$

$$A_{circular\ \ sector\ (BACDB)} = \theta \, b_{max}^{\;2}$$

$$A_{triangle(BACB)} = b_{med} \; b_{max} \; \cos\theta \qquad [\, \theta = \sin^{-1} (\, b_{med} \; / \; b_{max}) \,]$$

Therefore, plugging these values into the above equation leads to Equation A-1.

APPENDIX B - DERIVATION OF CRITICAL BUCKLING STRESS (σ_{CR})

The critical stress for film buckling is modelled as elastic plate buckling with one fixed end. Simple plate buckling with no applied moment ($w \neq f(y)$) is defined as [Ref. 32]:

$$\frac{EI}{1-\nu^2}\frac{\partial^4 w}{\partial x^4}+\sigma_x t\frac{\partial^2 w}{\partial x^2}=0 \tag{B-1}$$

where: E is the Elastic Modulus, υ is Poison's Ratio, I is the moment of inertia ($I = t^3/12$), t is the film thickness, x is the scratch direction, w is component of displacement perpendicular to the scratch direction, and σ_x t is the force applied along the x direction. Letting $\partial^2 w/\partial x^2 = Y$, the above equation can be rewritten as follows:

$$\frac{EI}{1-\nu^2}\frac{\partial^2 Y}{\partial x^2}+\sigma_x tY=0 \tag{B-2}$$

The characteristic solution is in the form:

$$Y=C_1\sin\sqrt{\frac{\sigma_x t}{[\frac{EI}{1-\nu^2}]}} \tag{B-3}$$

where C_1 is a constant of integration. Applying the boundary conditions [Y(0) = 0 and Y(L) = 0], the characteristic values of the critical load are:

$$(\sigma t)_{cr}=n^2\pi^2\frac{EI}{1-\nu^2}\frac{1}{L^2} \tag{B-4}$$

Since the lowest Euler buckling load occurs for n = 1, the critical buckling film stress is as follows:

$$\sigma_{cr}=\pi^2\frac{EI}{1-\nu^2}\frac{1}{12\left(\frac{L}{t}\right)^2} \tag{B-5}$$

APPENDIX C - EQUATION FOR AVERAGING τ_{ihv} OVER CIRCULAR FLF

For circular flaking, τ_{ihv} must be averaged over the flake area by using either Equation 4-30 or Equation 4-31 depending upon the type of circular flaking present. The flaking region can be defined using the standard equation for a circle that is offset from the origin [Ref. 39]:

$$a^2 = d^2 + r^2 - 2rd\cos(\lambda-\beta) \qquad \textbf{(C-1)}$$

as shown in the following figure:

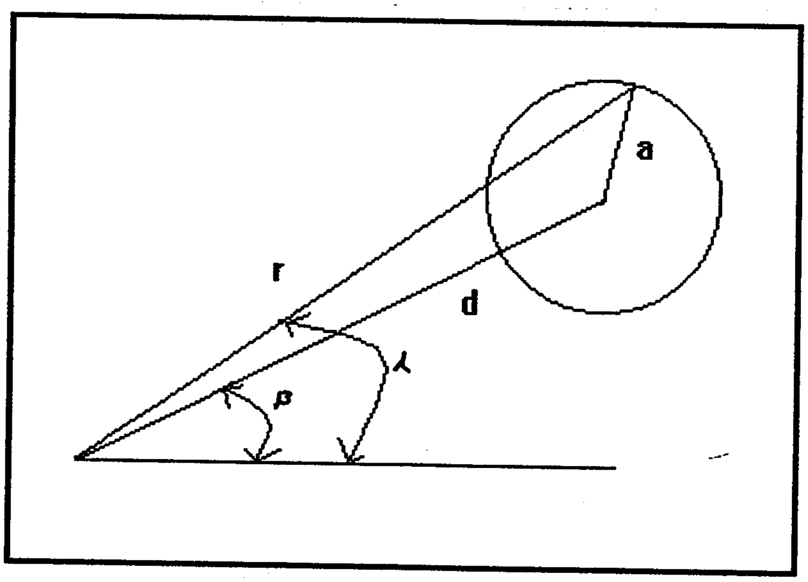

Figure C-1: Geometrical relationship of a circle offset from the origin

Using the above mathematical equation and applying it to indenter scratching, the angle β is equal to zero, b_{max} equals a, and m (the distance from the center of the indenter to the center of the circular flake) equals d. Equation C-1 can, therefore, be modified for scratching as follows [Ref. 2]:

$$b_{max}^2 = m^2 + r^2 - 2mr\cos\lambda \qquad \textbf{(C-2)}$$

Using the quadratic equation, the value for r can be determined from the above equation:

$$r = m\cos\lambda \pm \sqrt{m^2\cos^2\lambda - m^2 + b_{max}} \qquad \textbf{(C-3)}$$

The value for m can be determined geometrically from the following figure:

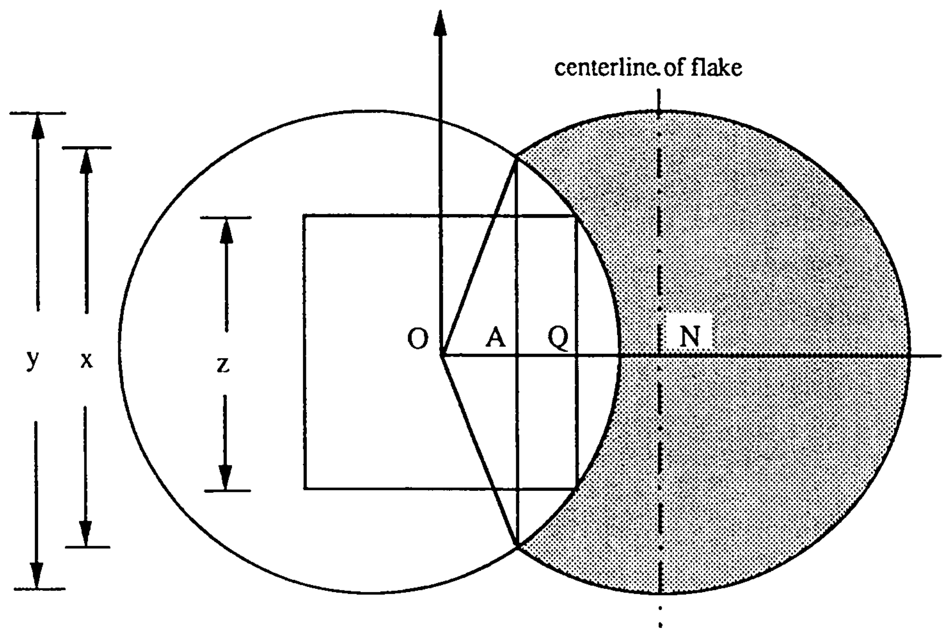

Figure C-2: Geometrical relationship between the indenter and forward lateral flaking, where: $x = 2b_{med}$, $y = 2b_{max}$, and $z = 2b_{min}$.

The derivation of m is as follows:

$$m = OA + AN \qquad \textbf{(C-4)}$$

where:

$AN = (b_{max}^2 - b_{med}^2)^{1/2}$ derived using the Pythagorean Theorem

$OA = OQ - AQ = b_{min} - [(b_{max}^2 - b_{min}^2)^{1/2} - (b_{max}^2 - b_{med}^2)^{1/2}]$

Inserting the values for AN and OA into Equation C-4, the following equation for m is produced (see Equation 4-27):

$$m = b_{\min} - \sqrt{b_{\max}^2 - b_{\min}^2} + 2\sqrt{b_{\max}^2 - b_{med}^2}$$

(C-5)

The limit for λ can be determined geometrically from $\tan\lambda = b_{med} / OA$.

If m is *+ve* then the upper limits for integrating over the circular area:

$$\lambda_{\max} = \tan^{-1}\left(\frac{b_{med}}{b_{\min} - \sqrt{b_{\max}^2 - b_{\min}^2} + \sqrt{b_{\max}^2 - b_{med}^2}}\right)$$

$$r_{\max} = m\cos\lambda + \sqrt{m^2\cos^2\lambda - m^2 + b_{\max}^2}$$

(C-6)

If m is *-ve* then the upper limits for integrating over the circular area:

$$\lambda_{\max} = \pi + \tan^{-1}\left(\frac{b_{med}}{b_{\min} - \sqrt{b_{\max}^2 - b_{\min}^2} + \sqrt{b_{\max}^2 - b_{med}^2}}\right)$$

$$r_{\max} = m\cos\lambda - \sqrt{m^2\cos^2\lambda - m^2 + b_{\max}^2}$$

(C-7)

The lower limit for r is $\sqrt{2}b_{min}$, which is the radius of a circle formed around the indenter corners (see Figure C-2). The lower limit for integrating λ is zero.

The average τ_{ihv} can be determined by integrating the following equation over the flake area [Ref. 2] (see Equation 4-24) :

$$\overline{\tau_{ihv}} = \frac{1}{A_{flf}} \int \tau_{ihv}(r)\, dA_{flf}$$

(C-8)

By applying the above limits of integration for r and λ, Equations 4-30 and 4-31 can be derived.

APPENDIX D - EXPERIMENTAL APPARATUS COMPUTER PROGRAMS

```
1000 '*************************************************************************************
1010  '
1020  '                    PROGRAM INITIAL1.BAS
1030  '
1040  '                    REVISION DATE  27 JULY 94
1050 '*************************************************************************************
1060  'THIS PROGRAM IS USED TO FIND THE INITIAL VALUES FOR HORIZONTAL  LOAD
1070 'CELL, VERTICAL LOAD CELL, LVDT, AND MICROPORIX VOLTAGE.  DATA ACQUISITION
1080  'PART OF PROGRAM WRITTEN BY TOM CHRISTIAN
1110  'PROGRAM WRITTEN BY  LT CAMPBELL
1120 '*************************************************************************************
1130 PRINT "PROGRAM DESIGNED TO FIND INITIAL VALUES OF LOAD CELLS, MICROPROX, AND
LVDT ."
1131 PRINT "THE INITIAL VALUES ARE FOUND BY AVERAGING TEN READINGS."
1132 PRINT "THESE READINGS ARE OBTAINED FROM THE DATA ACQUISITION / CONTROL UNIT"
1140 CLEAR
1150 DEF SEG = &HD000
1160 GPIB = 0: FLG% = 0: BRD% = 0: DUMMY% = 0'CLEAR VARIABLES
1170  '
1180 '*************************************************************************************
1190  'SETUP METRABYTE INTERFACE BOARD
1200 INIT$ = "SYSCON MAD=3,CIC=1,NOB=1,BA0=&H300"
1210  'SYSCON     IS SET UP COMMAND TO INITIALIZE AND CONFIGURE BOARD
1220  'MAD        IS MY ADDRESS EQUALS THREE
1230  'CIC        IS CONTROLLER IN CHARGE EQUALS ONE
1240  'NOB        IS NUMBER OF BOARDS EQUALS ONE
1250  'BA0        IS BASE I/O ADDRESS OF BOARD
1260 CONF$ = "CONFIG MTA,LISTEN=9"   'PREPARE DEVICE 9 AS LISTENER
1270 REMOT$ = "REMOTE 9"             'PLACE DEVICE 9 IN REMOTE MODE
1280 CMDOUT$ = "OUTPUT 9[$E]         'BEGINNING OF OUTPUT STRING"
1290 CMDIN$ = "ENTER 9[$,0,18]      'INPUT STRING FOR 19 CHARACTERS"
1300 CLR$ = "CLEAR 9"               'CLEAR DEVICE 9
1310 DAT$ = SPACE$(20)              'DIMENSION MEMORY FOR DATA
1320  '
1330 '*************************************************************************************
1340  'PREPARE METRABYTE INTERFACE BOARD
1350  'CALL INTERPRETER IN ROM TO ACCESS BOARD AND RETURN FLAG INFO
1360 CMD$ = INIT$
1370 GOSUB 3280
1380 CMD$ = CONF$
1390 GOSUB 3280
1400 CMD$ = CLR$
1410 GOSUB 3280
1420 CMD$ = REMOT$
1430 GOSUB 3280
```

```
1440  '
1450 '*************************************************************************************
1460  'SETUP 3852 VOLTMETER ETC
1470 MSG$ = "RESET 000"           'RESET VOLTMETER IN SLOT 0
1480 GOSUB 3310
1490 MSG$ = "USE 000"             'USE VOLTMETER FOR MEASUREMENTS
1500 GOSUB 3310
1510 MSG$ = "CONF DCV"            'CONFIGURE FOR DC VOLTS
1520 GOSUB 3310
1530  '
1540 '*************************************************************************************
1870  'SETUP ARRAYS FOR DATA STORAGE
1880 DIM VOLTS(20, 4)  'PREPARE AN ARRAY FOR RAW DATA
1940  '
1950 '*************************************************************************************
1960  'BEGIN TO TAKE RAW DATA
1961 SUMA = 0.0
1962 SUMB = 0.0
1963 SUMC = 0.0
1964 SUMD = 0.0
1990 FOR NO = 1 TO 20          'USE NESTED LOOPS FOR SCANS AND CHANNELS
2000 FOR CH = 0 TO 3           'USE FOR/NEXT LOOP AS CHANNELS
2010 J = 205 + CH              'CHANNELS = 205 PLUS CH
2020 CHNL$ = STR$(J)              'COMMAND TO 3852 MUST BE IN STRINGS
2030 MSG$ = "MEAS DCV," + CHNL$    'MEASURE VOLTAGE ON CHANNEL 205 PLUS CH
2040 GOSUB 3310
2050 GOSUB 3340                'CALL ENTER ROUTINE
2060 VOLTS(NO, CH) = VAL(DAT$)     'DATA IS RETURNED IN DAT$
2260 NEXT CH
2270 NEXT NO
300 MSG$ = "BEEP"              'MAKE 3852 BEEP WHEN DONE
2310 GOSUB 3310
2320 CMD$ = CLR$               'CLEAR AND RESET DEVICE 9
2330 GOSUB 3280
2350 '*************************************************************************************
2351 '  THE LAST 10 READINGS FOR LDVT, HORIZONTAL, VERTICAL, AND MICROPROX ARE
2352  '  EACH SUMMED.
2400 FOR NO = 11 TO 20
2411 SUMA = SUMA + VOLTS(NO,0)
2412 SUMB = SUMB + VOLTS(NO,1)
2413 SUMC = SUMC + VOLTS(NO,2)
2414 SUMD = SUMD + VOLTS(NO,3)
2530 NEXT NO
2531  '  EACH SUM IS DIVIDED BY 10 (THE TOTAL NUMBER ADDED)
2540 LVDT = SUMA  / 10
2550 HCM = SUMB / 10
2560 MP = SUMC / 10
2570 VCM = SUMD / 10
2580 PRINT "*********************************************************************************"
2590 PRINT " LVDT(V)  FH(V)   MICROPROX (V)    W(V)
2670 FOR NO = 1 TO 20
2680 FOR CH = 0 TO 3
```

```
2700 PRINT USING "####.#######"; VOLTS(NO,CH);
2710 PRINT CHR$(32);
2720 NEXT CH
2730 PRINT " "
2740 NEXT NO
2745 PRINT ""
2750 PRINT "*********************************************************************************"
2760 PRINT "LVDT INITIAL AVERAGE VALUE  = "; LVDT ; "VOLTS"
2761 PRINT " "
2780 PRINT "HORIZONTAL LOAD CELL AVERAGE INITIAL VALUE ="; HCM; "VOLTS"
2781 PRINT " "
2785 PRINT "MICROPROX INITIAL AVERAGE VALUE ="; MP; "VOLTS"
2786 PRINT " "
2790 PRINT "VERTICAL LOAD CELL AVERAGE INITIAL VALUE =" ;  VCM ;  "VOLTS"
2800 PRINT  "************************************************************************** "
3270 END
3280 CALL GPIB(CMD$, DUMMY%, FLG%, BRD%)
3290 IF FLG% <> 0 THEN PRINT " ERROR IN "; CMD$; VAR$; "FLAG = HEX "; HEX$(FLG%)
3300 RETURN
3310 CALL GPIB(CMDOUT$, MSG$, FLG%, BRD%)
3320 IF FLG% <> 0 THEN PRINT " ERROR IN "; MSG$; "FLAG = HEX "; HEX$(FLG%)
3330 RETURN
3340 CALL GPIB(CMDIN$, DAT$, FLG%, BRD%)
3350 IF FLG% <> 0 THEN PRINT " DATA INPUT ERROR FLAG = HEX "; HEX$(FLG%)
3360 RETURN
```

```
1000 '*****************************************************************************************
1010  '
1020  '                    PROGRAM INDENT1BAS
1030  '
1040  '                    DATE 02 JULY 1994
1050 '*****************************************************************************************
1060  'PROGRAM USED TO FIND WHEN INDENTER PENETRATES SUBSTRATE
1070 'TO DETERMINE ZERO VALUE FOR MICROPROXIMITY TRANSDUCER.  DATA ACQUISITION
1075  'PART OF PROGRAM WRITTEN BY TOM CHRISTIAN
1080  'PROGRAM WRITTEN BY LT CAMPBELL
1120  '****************************************************************************************
1125 PRINT "PROGRAM DESIGNED TO TELL WHEN INDENTER IS TOUCHING FILM"
1126 PRINT "BY USING THIS PROGRAM, THE ZERO (REFERENCE) SETTING OF MICROPROX CAN
BE FOUND"
1127 PRINT "*********************************************************************************"
1128 PRINT ""
1130 PRINT "ENSURE MICROPROX VOLTMETER READS -24 VOLTS"
1140 CLEAR
1150 DEF SEG = &HD000
1160 GPIB = 0: FLG% = 0: BRD% = 0: DUMMY% = 0'CLEAR VARIABLES
1170  '
1180 '*****************************************************************************************
1190  'SETUP METRABYTE INTERFACE BOARD
1200 INIT$ = "SYSCON MAD=3,CIC=1,NOB=1,BA0=&H300"
```

```
1210  'SYSCON    IS SET UP COMMAND TO INITIALIZE AND CONFIGURE BOARD
1220  'MAD       IS MY ADDRESS EQUALS THREE
1230  'CIC       IS CONTROLLER IN CHARGE EQUALS ONE
1240  'NOB       IS NUMBER OF BOARDS EQUALS ONE
1250  'BA0       IS BASE I/O ADDRESS OF BOARD
1260 CONF$ = "CONFIG MTA,LISTEN=9"   'PREPARE DEVICE 9 AS LISTENER
1270 REMOT$ = "REMOTE 9"             'PLACE DEVICE 9 IN REMOTE MODE
1280 CMDOUT$ = "OUTPUT 9[$E]         'BEGINNING OF OUTPUT STRING"
1290 CMDIN$ = "ENTER 9[$,0,18]       'INPUT STRING FOR 19 CHARACTERS"
1300 CLR$ = "CLEAR 9"                'CLEAR DEVICE 9
1310 DAT$ = SPACE$(20)               'DIMENSION MEMORY FOR DATA
1320  '
1330 '**************************************************************************************
1340  'PREPARE METRABYTE INTERFACE BOARD
1350  'CALL INTERPRETER IN ROM TO ACCESS BOARD AND RETURN FLAG INFO
1360 CMD$ = INIT$
1370 CALL GPIB(CMD$,DUMMY%,FLG%,BRD%)
1380 CMD$ = CONF$
1390 CALL GPIB(CMD$,DUMMY%,FLG%,BRD%)
1400 CMD$ = CLR$
1410 CALL GPIB(CMD$,DUMMY%,FLG%,BRD%)
1420 CMD$ = REMOT$
1430 CALL GPIB(CMD$,DUMMY%,FLG%,BRD%)
1440  '
1450 '**************************************************************************************
1460  'SETUP 3852 VOLTMETER ETC
1470 MSG$ = "RESET 000"              'RESET VOLTMETER IN SLOT 0
1480 CALL GPIB(CMDOUT$,MSG$,FLG%,BRD%)
1490 MSG$ = "USE 000"                'USE VOLTMETER FOR MEASUREMENTS
1500 CALL GPIB(CMDOUT$,MSG$,FLG%,BRD%)
1510 MSG$ = "CONF DCV"               'CONFIGURE FOR DC VOLTS
1520 CALL GPIB(CMDOUT$,MSG$,FLG%,BRD%)
1540 '**************************************************************************************
1550 TIME = 1000
1870  'SETUP ARRAYS FOR DATA STORAGE
1900 DIM  VOLTS(TIME,2)        'PREPARE AN ARRAY FOR VERTICAL LOAD
1901 DIM  W(TIME)
1902 DIM  MP(TIME)
1950 '**************************************************************************************
1951  INPUT "ENTER INITIAL VOLTAGE OF VERTICAL LOAD CELL (VOLTS) ",VCM
1952 PRINT "*********************************************************************************"
1960  'BEGIN TO TAKE RAW DATA
1961 COUNT = 0
1990 FOR NO = 1 TO TIME          'USE NESTED LOOPS FOR SCANS AND CHANNELS
1991 FOR CH = 0 TO 1
2010 J = 207+ CH
2020 CHNL$ = STR$(J)             'COMMAND TO 3852 MUST BE IN STRINGS
2030 MSG$ = "MEAS DCV," + CHNL$    'MEASURE VOLTAGE ON CHANNEL 208
2040 CALL GPIB(CMDOUT$,MSG$,FLG%,BRD%)
2050 CALL GPIB(CMDIN$,DAT$,FLG%,BRD%)
2060 VOLTS(NO,CH) = VAL(DAT$)      'DATA IS RETURNED IN DAT$
2061 IF CH = 1 THEN GOTO 2170
```

```
2062 IF CH = 0 THEN  GOTO 3391
2170 IF ABS(VOLTS(NO,CH) - VCM) < (.0001 ) THEN GOTO  3391
2171 COUNT = COUNT + 1
2172 IF COUNT = 1 THEN  DEP = NO
2173 IF COUNT > (10) THEN GOTO 2303
2180 PRINT "INDENTER TOUCHING FILM: VERTICAL=";VOLTS(NO,1); "VOLTS; MICROPROX = ";
VOLTS(NO,0); "VOLTS"
2292 BEEP
2300 MSG$ = "BEEP"              'MAKE 3852 BEEP WHEN DONE
2302 GOTO 3391
2303 IF COUNT < (60) THEN GOTO 3391
2304 PRINT ""
2305 PRINT "**********************************************************************************"
2306 PRINT "THE VERTICAL VOLTAGE IS";  VOLTS(DEP-1,1); "VOLTS"
2308 PRINT "MICROPROXIMITY ZERO SETTING (REFERENCE SETTING) IS "; VOLTS(DEP-1,0);
"VOLTS"
2310 SCANS= NO
2315 CALL GPIB(CMDOUT$,MSG$,FLG%,BRD%)
2316 CMD$=CLR$
2330 CALL GPIB(CMD$,DUMMY%,FLG%,BRD%)
2342 PRINT "**********************************************************************************"
2343 INPUT "WOULD YOU LIKE TO SEE RAW DATA?",QUES$
2344 IF QUES$="N" OR  QUES$="n" THEN GOTO 2441
2350' PRINT DATA
2352 PRINT "PROX(VDC)       W(VDC)"
2363 FOR NO = 1 TO SCANS
2374 FOR  CH = 0 TO 1
2385 PRINT USING "####.######";VOLTS(NO,CH);
2396 PRINT CHR$(9);
2407 NEXT CH
2418 PRINT ""
2429 NEXT NO
2430 PRINT ""
2441 INPUT "DO YOU WISH TO SAVE RAW VOLTAGE DATA (Y/N)?",QUES$
2452 IF QUES$ = "N" OR QUES$ ="n" THEN GOTO 4500
2463 INPUT "ENTER THE NAME OF THE DATA FILE TO CREATE (IE IND##.DAT)",FILE$
2474 OPEN FILE$ FOR OUTPUT AS 1
2485 FOR NO = 1 TO SCANS
2496 FOR CH = 0 TO 1
2507 PRINT #1, USING "####.######"; VOLTS(NO,CH);
2518 PRINT #1, CHR$(9);
2529 NEXT CH
2530 PRINT #1,""
2541 NEXT NO
2552 CLOSE #1
2560 END
3391 NEXT CH
3392 NEXT NO
3450 PRINT "PROGRAM AND TEST MUST BE RESTARTED.  SCAN TIME HAS EXPIRED"
3500 BEEP
3550 BEEP
4500 END
```

```
1000 '*************************************************************************************
1010  '
1020  '                    PROGRAM PENT1.BAS
1030  '
1040  '                    DATE 03 JULY 1994
1050 '*************************************************************************************
1060  'PROGRAM USED WHEN PENETRATING FILM OR FILM/SUBSTRATE
1070  'MICROPROXIMITY PROBE VALUES AND VERTICAL LOAD VALUES WILL.
1080  'BE MEASURED.  PRIMARY ROUTINES FOR METRABYTE IEEE-488 BOARD TO HP-3852
1090 'DATA ACQUISITION UNIT WRITTEN BY TOM CHRISTIAN. BY USING THIS PROGRAM, ONE
1095 'IS ABLE TO ACCURATELY PENETRATE THE REQUIRED THICKNESS FOR TYPE II OR TYPE
1098   'III DEBONDING.  PROGRAM WRITTEN BY LT CAMPBELL.
1120  '*************************************************************************************
1130 PRINT "PROGRAM ENABLES ONE TO PENETRATE TO DESIRED THICKNESS"
1131 PRINT "ENSURE MICROPROX VOLTMETER IS SET AT  -24 VOLTS"
1140 CLEAR
1150 DEF SEG = &HD000
1160 GPIB = 0: FLG% = 0: BRD% = 0: DUMMY% = 0'CLEAR VARIABLES
1170  '
1180 '*************************************************************************************
1190  'SETUP METRABYTE INTERFACE BOARD
1200 INIT$ = "SYSCON MAD=3,CIC=1,NOB=1,BA0=&H300"
1210  'SYSCON    IS SET UP COMMAND TO INITIALIZE AND CONFIGURE BOARD
1220  'MAD       IS MY ADDRESS EQUALS THREE
1230  'CIC       IS CONTROLLER IN CHARGE EQUALS ONE
1240  'NOB       IS NUMBER OF BOARDS EQUALS ONE
1250  'BA0       IS BASE I/O ADDRESS OF BOARD
1260 CONF$ = "CONFIG MTA,LISTEN=9"  'PREPARE DEVICE 9 AS LISTENER
1270 REMOT$ = "REMOTE 9"            'PLACE DEVICE 9 IN REMOTE MODE
1280 CMDOUT$ = "OUTPUT 9[$E]        'BEGINNING OF OUTPUT STRING"
1290 CMDIN$ = "ENTER 9[$,0,18]      'INPUT STRING FOR 19 CHARACTERS"
1300 CLR$ = "CLEAR 9"               'CLEAR DEVICE 9
1310 DAT$ = SPACE$(20)              'DIMENSION MEMORY FOR DATA
1320  '
1330 '*************************************************************************************
1340  'PREPARE METRABYTE INTERFACE BOARD
1350  'CALL INTERPRETER IN ROM TO ACCESS BOARD AND RETURN FLAG INFO
1360 CMD$ = INIT$
1370 CALL GPIB(CMD$,DUMMY%,FLG%,BRD%)
1380 CMD$ = CONF$
1390 CALL GPIB(CMD$,DUMMY%,FLG%,BRD%)
1400 CMD$ = CLR$
1410 CALL GPIB(CMD$,DUMMY%,FLG%,BRD%)
1420 CMD$ = REMOT$
1430 CALL GPIB(CMD$,DUMMY%,FLG%,BRD%)
1440  '
1450 '*************************************************************************************
1460  'SETUP 3852 VOLTMETER ETC
1470 MSG$ = "RESET 000"             'RESET VOLTMETER IN SLOT 0
1480 CALL GPIB(CMDOUT$,MSG$,FLG%,BRD%)
```

```
1490 MSG$ = "USE 000"              'USE VOLTMETER FOR MEASUREMENTS
1500 CALL GPIB(CMDOUT$,MSG$,FLG%,BRD%)
1510 MSG$ = "CONF DCV"             'CONFIGURE FOR DC VOLTS
1520 CALL GPIB(CMDOUT$,MSG$,FLG%,BRD%)
1521 TIME = 1000
1540 '************************************************************************************
1870  'SETUP ARRAYS FOR DATA STORAGE
1900 DIM  VOLTS(TIME,2)        'PREPARE VARIOUS ARRAYS
1901 DIM  W(TIME)
1902 DIM  MP(TIME)
1903 DIM  MPC(TIME)
1940 '************************************************************************************
1941  PRINT  "MICROPROX VOLTAGE MUST BE ENTERED WITH A NEGATIVE SIGN"
1942  PRINT ""
1943  INPUT "ENTER ZERO SETTING OF MICROPROXIMITY PROBE(VOLTS)   ", MPP
1944  PRINT ""
1945  PRINT "ENSURE MICROPROX VOLTAGE IS BETWEEN  -5.0 VOLTS AND -21.0 VOLTS"
1946  PRINT "ONLY IN THIS RANGE (-5.0 TO -21.0) ARE THE VALUES FOR MICROPROX LINEAR"
1954 PRINT "*****************************************************************************"
1955 INPUT "ENTER THICKNESS (AN) IN WHICH YOU WANT INDENTER TO PENETRATE   ", FT
1956 FT = FT/10000!              'CONVERTS ANGSTROMS TO MICROMETERS
1957 MEASD = (FT + .170919)/(.275754)     ' CALIBRATION CURVE BETWEEN MEAS / ACTUAL
1958 DEPTH = .040 * MEASD    ' CONVERSION SCALE FACTOR 40mV / MICROMETER
1959 PRINT ""
1960 PRINT "DEPTH TO PENETRATE  IN VOLTAGE  FOR MICROPROX IS"; DEPTH; "VOLTS"
1961 PRINT ""
1962 INPUT "ENTER INITIAL VOLTAGE OF VERTICAL LOAD CELL  ",  VCM
1963 PRINT "*****************************************************************************"
1970  'BEGIN TO TAKE RAW DATA
1980 COUNT = 0
1990 FOR NO = 1 TO TIME         'USE NESTED LOOPS FOR SCANS AND CHANNELS
1991 FOR CH = 0 TO 1
2010 J = 208 - CH
2020 CHNL$ = STR$(J)            'COMMAND TO 3852 MUST BE IN STRINGS
2030 MSG$ = "MEAS DCV," + CHNL$     'MEASURE VOLTAGE ON CHANNEL 208
2040 CALL GPIB(CMDOUT$,MSG$,FLG%,BRD%)
2050 CALL GPIB(CMDIN$,DAT$,FLG%,BRD%)
2060 VOLTS(NO,CH) = VAL(DAT$)     'DATA IS RETURNED IN DAT$
2061 IF VOLTS(NO,0) > (.432)  THEN GOTO 6000  ' PROTECTS VERT CELL FROM MAX LOAD
2062 IF CH = 1 THEN GOTO 2170
2063 IF CH = 0 THEN  GOTO 3391
2170 IF ABS(VOLTS(NO,CH) - MPP) <  DEPTH  THEN GOTO  3391
2171 COUNT = COUNT + 1
2172 IF COUNT > (3) THEN GOTO 2302
2180 PRINT "REQUIRED THICKNESS PENETRATED "; VOLTS(NO,1); "VOLTS (MICROPROX)"
2291 BEEP
2300 MSG$ = "BEEP"               'MAKE 3852 BEEP WHEN DONE
2301 GOTO 3391
2302 IF COUNT < (53) THEN GOTO 3391 ' THIS WILL GIVE DATA TO SHOW STABILIZATION
2303 PRINT "*****************************************************************************"
2305 SCANS = NO
2306 PRINT "THE VERTICAL VOLTAGE IS"; VOLTS(NO,0); "VOLTS"
```

```
2308 PRINT "MICROPROXIMITY VOLTAGE IS "; VOLTS(NO,1); "VOLTS"
2310 PRINT "**********************************************************************************"
2311 PENT = (ABS(VOLTS(NO,1)-MPP)/.040) 'CALCULATES MEASURED PENETRATION DEPTH
2312 ACTUAL = -.170919 + .275754*PENT     ' USES CALIBRATION  CURVE FOR ACTUAL
2313 PRINT "ACTUAL TOTAL DEPTH PENETRATED IS" ;ACTUAL; "MICROMETERS"
2315 PRINT "**********************************************************************************"
2316 CALL GPIB(CMDOUT$,MSG$,FLG%,BRD%)
2320 CMD$ = CLR$                    'CLEAR AND RESET DEVICE 9
2330 CALL GPIB(CMD$,DUMMY%,FLG%,BRD%)
2331'*****************************************************************************************'
2332 INPUT "WOULD YOU LIKE TO SEE RAW DATA?",QUES$
2333 IF QUES$="N" OR  QUES$="n"  THEN GOTO 2441
2342 ' PRINT DATA
2352 PRINT "PROX(VDC)       W(VDC)"
2363 FOR NO = 1 TO SCANS
2374 FOR  CH = 0 TO 1
2385 PRINT USING "####.######";VOLTS(NO,CH);
2396 PRINT CHR$(9);
2407 NEXT CH
2418 PRINT ""
2429 NEXT NO
2430 PRINT ""
2441 INPUT "DO YOU WISH TO SAVE RAW VOLTAGE DATA (Y/N)?",QUES$
2452 IF QUES$ = "N" OR QUES$ ="n"  THEN GOTO  2556
2463 INPUT "ENTER THE NAME OF THE DATA FILE TO CREATE (IE PENTR##.DAT)",FILE$
2474 OPEN FILE$ FOR OUTPUT AS 1
2485 FOR NO = 1 TO SCANS
2496 FOR CH = 0 TO 1
2507 PRINT #1, USING "####.######"; VOLTS(NO,CH);
2518 PRINT #1, CHR$(9);
2529 NEXT CH
2530 PRINT #1,""
2541 NEXT NO
2552 CLOSE #1
2553'*****************************************************************************************
2554 'CONVERSION OF RAW DATA
2556 FOR NO = 1 TO SCANS
2557 FOR CH = 0 TO 1
2558 IF CH = 1 THEN MP(NO) = ABS(VOLTS(NO,CH) - MPP) / .040
2559 MPC(NO) =  -.170919 + .27575 * MP(NO)        ' CALIBRATION CURVE MEAS / ACTUAL
2560 'CONVERTS VOLTS FROM MICROPROX TO MICROMETERS FOR DISTANCE
2561 IF CH = 0 THEN W(NO) = 7692.3076# * (VOLTS(NO,CH) - VCM - .00007) * .00981
2565 'CONVERTS VOLTS VERTICAL FORCE TO NEWTONS
2567 NEXT CH
2569 NEXT NO
2571'*****************************************************************************************
2573 ' MAKE DATA FILE AND PRINT RESULTS TO SCREEN
2575 PRINT ""
2580 PRINT " MICROPROX (MICROMETER)     VERTICAL FORCE (N)"
2585 FOR NO = 1 TO SCANS
2590 PRINT USING "####.######"; W(NO); MPC(NO);
2595 PRINT ""
```

```
2600 NEXT NO
2610 PRINT ""
2615 PRINT "ENSURE THE FOLLOWING DATA IS RECORDED"
2620 PRINT ""
2630'*************************************************************************************
2635 'MAKE DATA FILE FOR RESULTS
2640 INPUT "DO YOU WANT TO SAVE THE RESULTS DATA (Y/N)  ",QUES$
2645 IF QUES$="N" OR QUES$="n"  THEN GOTO 3500
2650 INPUT "ENTER NAME OF DATA FILE TO CREATE (IE PENT.DAT) ",FILE$
2655 OPEN FILE$ FOR OUTPUT AS 1
2660 FOR NO = 1 TO SCANS
2665 PRINT #1, USING "####.######"; W(NO);
2670 PRINT #1, CHR$(9);
2675 PRINT #1, USING "####.######";MP(NO);
2680 PRINT #1, CHR$(9);
2685 PRINT #1, ""
2690 NEXT NO
2700 CLOSE #1
2760 END
3391 NEXT CH
3392 NEXT NO
3400 PRINT "PROGRAM AND TEST MUST BE RESTARTED.  SCAN TIME HAS EXPIRED"
3410 BEEP
3500 END
6000 BEEP
6001 PRINT "MAX VERTICAL FORCE HAS BEEN EXCEEDED"
6002 PRINT "CONTINUED LOAD ON VERTICAL CELL WILL DAMAGE CELL"
6003 BEEP
6004 END
```

```
1000 '*************************************************************************************
1010  '
1020  '                    PROGRAM COLLECT1.BAS
1030  '
1040  '                    REVISION DATE  940911
1050 '*************************************************************************************
1060  'THIS PROGRAM IS USED DURING A SCRATCH TEST TO COLLECT DATA TO BE USED TO
1070  'DETERMINE THE INTERFACIAL SHEAR STRENGTH AT THIN FILM-CERAMIC BOUNDARIES
1080  'IN REAL TIME.  PRIMARY ROUTINES FOR METRABYTE IEEE-488 BOARD TO HP-3852
1090  'DATA ACQUISITION UNIT (WRITTEN BY TOM CHRISTIAN) USE 44702A HIGH SPEED
1100   'VOLTMETER AND 447711A FET MUX CARD.  PORTIONS OF THIS PROGRAM WERE
1110   ' WRITTEN BY DAVE  LASCURAIN.  MODIFIED DAN SECOR AND JOHN CAMPBELL.
1120 '*************************************************************************************
1121 PRINT "THIS PROGRAM IS USED DURING AN ACTUAL SCRATCH TEST TO COLLECT DATA
VIA THE
1122 PRINT "3852A DATA ACQUISITION/CONTROL UNIT"
1123 PRINT ""
1130 PRINT "ENSURE ALL VOLTMETERS ARE ON BEFORE START OF TEST"
1131 PRINT "ENSURE THE MICROPROX VOLTMETER IS SET AT -24 VOLTS"
```

```
1132 PRINT ""
1133 PRINT "MOTOR SHOULD BE OFF AND IN STAND-BY CONDITION"
1140 CLEAR
1150 DEF SEG = &HD000
1160 GPIB = 0: FLG% = 0: BRD% = 0: DUMMY% = 0'CLEAR VARIABLES
1170  '
1180 '**************************************************************************************
1190  'SETUP METRABYTE INTERFACE BOARD
1200 INIT$ = "SYSCON MAD=3,CIC=1,NOB=1,BA0=&H300"
1210  'SYSCON     IS SET UP COMMAND TO INITIALIZE AND CONFIGURE BOARD
1220  'MAD        IS MY ADDRESS EQUALS THREE
1230  'CIC        IS CONTROLLER IN CHARGE EQUALS ONE
1240  'NOB        IS NUMBER OF BOARDS EQUALS ONE
1250  'BA0        IS BASE I/O ADDRESS OF BOARD
1260 CONF$ = "CONFIG MTA,LISTEN=9"  'PREPARE DEVICE 9 AS LISTENER
1270 REMOT$ = "REMOTE 9"            'PLACE DEVICE 9 IN REMOTE MODE
1280 CMDOUT$ = "OUTPUT 9[$E]        'BEGINNING OF OUTPUT STRING"
1290 CMDIN$ = "ENTER 9[$,0,18]      'INPUT STRING FOR 19 CHARACTERS"
1300 CLR$ = "CLEAR 9"               'CLEAR DEVICE 9
1310 DAT$ = SPACE$(20)              'DIMENSION MEMORY FOR DATA
1320  '
1330 '**************************************************************************************
1340  'PREPARE METRABYTE INTERFACE BOARD
1350  'CALL INTERPRETER IN ROM TO ACCESS BOARD AND RETURN FLAG INFO
1360 CMD$ = INIT$
1370 GOSUB 3280
1380 CMD$ = CONF$
1390 GOSUB 3280
1400 CMD$ = CLR$
1410 GOSUB 3280
1420 CMD$ = REMOT$
1430 GOSUB 3280
1440  '
1450 '**************************************************************************************
1460  'SETUP 3852 VOLTMETER ETC
1470 MSG$ = "RESET 000"             'RESET VOLTMETER IN SLOT 0
1480 GOSUB 3310
1490 MSG$ = "USE 000"               'USE VOLTMETER FOR MEASUREMENTS
1500 GOSUB 3310
1510 MSG$ = "CONF DCV"              'CONFIGURE FOR DC VOLTS
1520 GOSUB 3310
1530  '
1540 '**************************************************************************************
1541 PRINT ""
1550  'DETERMINE INITIAL OFFSET VALUES.  INITIAL OFF SET VALUES  ON INSTRUMENTS
1560  'WITHOUT ANY LOADS APPLIED ARE SUBTRACTED FROM TRUE LOAD READINGS
1570  'MEASURED DURING THE SCRATCH TEST.
1571 PRINT "THE INITIAL VOLTAGE IS THE VOLTAGE BEFORE ANY LOAD HAS BEEN APPLIED."
1572 PRINT "THE INITIAL VOLTAGE OF THE LVDT IS THE VOLTAGE PRIOR TO MOVEMENT OF"
1573 PRINT  "THE TRANSLATION STAGE."
1580 PRINT " "
1620 PRINT "VOLTAGE OF LVDT AND MICROPROX MUST BE ENTERED WITH A NEGATIVE SIGN."
```

```
1650 PRINT " "
1660 INPUT "ENTER INITIAL VOLTAGE OF TABLE LVDT (volts)  ", VCM
1690 INPUT "ENTER INITIAL VOLTAGE OF HORIZONTAL LOAD CELL (volts) ", VHORZ
1700 INPUT "ENTER INITIAL VOLTAGE OF VERTICAL LOAD CELL (volts) ", VVERT
1701 INPUT "ENTER THE CURRENT VOLTAGE OF THE MICROPROX (volts)  ", VMP
1710 PRINT " "
1720 PRINT "INITIAL VOLTAGE OF TABLE LVDT (volts) = ", VCM
1730 PRINT "INITIAL VOLTAGE OF HORIZONTAL LOAD CELL (volts) = ", VHORZ
1740 PRINT "INITIAL VOLTAGE OF VERTICAL LOAD CELL (volts) = ", VVERT
1741 PRINT "CURRENT VOLTAGE OF MICROPROX (volts) =  ",VMP
1750 PRINT " "
1760 INPUT "DO YOU AGREE WITH THESE VALUES IN VOLTS?  (Y/N)  ", QUES$
1770 IF QUES$ = "n" OR QUES$ = "N" GOTO 1660
1780 INPUT "HOW MANY CHANNELS DO YOU WISH TO USE (MAX IS 4)  ", ADSTOP
1790 ADSTOP = ADSTOP - 1
1800 IF ADSTOP > 4 THEN GOTO 1780
1810 PRINT "PROGRAM RUNS AUTOMATICALLY AFTER THE NEXT INPUT.  START THE"
1820 PRINT "TABLE MOTOR IMMEDIATELY AFTER THE PROGRAM COMMENCES."
1830 INPUT "HOW MANY TIMES DO YOU WISH TO SAMPLE THESE CHANNELS  ", SCANS
1840 PRINT "WARNING IF SCREEN SAVER COMES ON PROGRAM STOPS TAKING DATA!!"
1841 PRINT " "
1842 PRINT "    LVDT(vdc)   Fh(vdc)   PROX(vdc)      W(vdc)"
1850  '
1860 '**************************************************************************************
1870  'SETUP ARRAYS FOR DATA STORAGE
1880 DIM VOLTS(SCANS, ADSTOP)  'PREPARE AN ARRAY FOR RAW DATA
1890 DIM RESULTS(SCANS, ADSTOP) 'PREPARE AN ARRAY FOR RESULTS
1900 DIM W(SCANS)          'PREPARE AN ARRAY FOR VERTICAL LOAD (NEWTONS)
1910 DIM FH(SCANS)         'PREPARE AN ARRAY FOR HORIZONTAL LOAD (NEWTONS)
1920 DIM CM(SCANS)         'PREPARE AN ARRAY FOR DISTANCE (MICROMETER)
1930 DIM MICP(SCANS)       'PREPARE AN ARRAY FOR MICROPROX (MICROMETER)
1931 DIM MICPC(SCANS)   ' PREPARE AN ARRAY FOR MICROPROX CALIBRATION CURVE
1940  '
1950 '**************************************************************************************
1960  'BEGIN TO TAKE RAW DATA
1970 STRTIM = TIMER                'GET START TIME
1980 ALARM = 0                     'RESET FALSE ALARM COUNT TO ZERO
1990 FOR NO = 1 TO SCANS           'USE NESTED LOOPS FOR SCANS AND CHANNELS
2000 FOR CH = 0 TO ADSTOP          'USE FOR/NEXT LOOP AS CHANNELS
2010 J = 205 + CH                  'CHANNELS = 205 PLUS CH
2020 CHNL$ = STR$(J)               'COMMAND TO 3852 MUST BE IN STRINGS
2030 MSG$ = "MEAS DCV," + CHNL$    'MEASURE VOLTAGE ON CHANNEL 205 PLUS CH
2040 GOSUB 3310
2050 GOSUB 3340                    'CALL ENTER ROUTINE
2060 VOLTS(NO, CH) = VAL(DAT$)     'DATA IS RETURNED IN DAT$
2070  'AND CONVERTED TO REAL NUMBER
2080  'THE NEXT 18 LINES DETERMINE IF THE HORIZONTAL OR VERTICAL LOAD CELL
2090  'HAS BEEN EXCEEDED.  IF SO, AN ALARM SOUNDS.  A COMPUTER GLITCH MAY CAUSE
2100  'A FALSE ALARM, BUT IF THIS HAPPENS THREE TIMES, THE PROGRAM IS ENDED.
2110 IF CH = 0 OR CH = 2 GOTO 2260
2120 IF CH = 1 THEN GOTO 2140
2130 IF CH = 3 THEN GOTO 2170
```

```
2140 IF VOLTS(NO, CH) < (.0117 + VHORZ) THEN GOTO 2260
2150 PRINT "HORIZONTAL FORCE EXCEEDED  GRAMS!!!!!"
2160 GOTO 2190
2170 IF VOLTS(NO, CH) < (.3509) THEN GOTO 2260
2180 PRINT "VERTICAL FORCE + PRE-LOAD EXCEEDED GRAMS!!!!!"
2190 FOR CNT = 1 TO 12
2200 BEEP
2210 NEXT CNT
2220 ALARM = ALARM + 1
2230 IF ALARM < 3 THEN GOTO 2260
2240 PRINT "LOAD CELL RANGE EXCEEDED; PROGRAM ENDED"
2241 SCANS  =  NO
2242 PRINT " "
2243 PRINT "TOTAL SCANS BEFORE LOAD CELL EXCEEDED =  ", SCANS
2244 PRINT " "
2245 INPUT "DO YOU WISH TO SAVE THE DATA (Y/N)",QUES$
2246 IF QUES$ = "N" OR QUES$ = "n" THEN GOTO 3270  'THIS TAKES YOU TO END
2250 GOTO 2280
2260 NEXT CH
2265 PRINT USING "####.#######"; VOLTS(NO,0) ;VOLTS(NO,1) ; VOLTS(NO, 2); VOLTS(NO,3);
2266 PRINT CHR$(32);
2267 PRINT ""
2268 PRINT ""
2270 NEXT NO
2280 STPTIM = TIMER                'GET STOP TIME
2290 TOTIME = STPTIM - STRTIM      'CALCULATE ELAPSED TIME
2300 MSG$ = "BEEP"                 'MAKE 3852 BEEP WHEN DONE
2310 GOSUB 3310
2320 CMD$ = CLR$                   'CLEAR AND RESET DEVICE 9
2330 GOSUB 3280
2340  '
2350 '**************************************************************************************
2360  'PRINT RAW DATA.  THE MEASURED VALUE DURING THE SCRATCH TEST HAS THE
2370  'INITIAL OFF-SET VALUE SUBTRACTED OFF IN THE STEPS BELOW.
2380 PRINT " "
2390 PRINT "   DIST (vdc)   FH (vdc)   PROX(vdc)     W (vdc)"
2400 FOR NO = 1 TO SCANS
2410 FOR CH = 0 TO ADSTOP
2420 IF CH = 0 THEN VOLTS(NO, CH) = ABS(VOLTS(NO, CH) - VCM)
2430 CM0 = VOLTS(1, 0)
2440 IF CH = 0 THEN VOLTS(NO, CH) = VOLTS(NO, CH) - CM0
2450  'THESE LAST TWO LINES SET THE POSITION OF THE FIRST DATA POINT AT
2460  'ZERO.  ALL OTHER POINTS ARE NOW WRT THE FIRST DATA POINT.
2470 IF CH = 1 THEN VOLTS(NO, CH) = VOLTS(NO, CH) - VHORZ
2471 IF CH = 2 THEN VOLTS(NO, CH) = VOLTS(NO, CH) - VMP
2480 IF CH = 3 THEN VOLTS(NO, CH) = VOLTS(NO, CH) - VVERT
2490 PRINT USING "####.######"; VOLTS(NO, CH);
2500 PRINT CHR$(32);
2510 NEXT CH
2520 PRINT " "
2530 NEXT NO
2540 PRINT " "
```

```
2570 PRINT "TOTAL ELAPSED FOR DATA ACQUISITION = ";
2580 PRINT USING "###.##"; TOTIME;
2590 PRINT "    SECONDS"
2600 PRINT "THE INITIAL OFF-SETS HAVE BEEN SUBTRACTED FROM EACH"
2610 PRINT "RESPECTIVE OUTPUT"
2620 PRINT " "
2630 INPUT "DO YOU WISH TO SAVE THE RAW DATA  (Y/N)  ", QUES$
2640 IF QUES$ = "N" OR QUES$ = "n" GOTO 2780
2650 INPUT "ENTER THE NAME OF THE DATA FILE TO CREATE (IE RAW##.DAT)  ", FILE$
2660 OPEN FILE$ FOR OUTPUT AS 1
2670 FOR NO = 1 TO SCANS
2680 FOR CH = 0 TO ADSTOP
2690 PRINT #1, USING "####.######"; VOLTS(NO, CH);  'REMEMBER THAT OFF SET
2700  'VALUES WERE SUBTRACTED
2710 PRINT #1, CHR$(9);
2720 NEXT CH
2730 PRINT #1, " "
2740 NEXT NO
2750 CLOSE #1
2760  '
2770 '*************************************************************************************
2780  'CONVERSION OF RAW DATA
2790 FOR NO = 1 TO SCANS
2800 FOR CH = 0 TO ADSTOP
2810 IF CH = 0 THEN CM(NO) = 1326.7878# * VOLTS(NO, CH)
2820  'CONVERTS VOLTS TO DISTANCE (MICROMETERS)
2830 IF CH = 1 THEN FH(NO) = (25536.97882# * VOLTS(NO, CH) + .132112)* .00981
2840  'CONVERTS VOLTS TO GRAM FORCE HORZ AND THEN TO NEWTONS
2850 IF CH = 2 THEN MICP(NO) = VOLTS(NO,CH) / .040     ' CALIBRATION  MICROPROX 40mV /
MICROMETER
2851 MICPC(NO) = -.170919 + .275754*MICP(NO)   'CALIBRATION CURVE FOR
2852    'ACTUAL VS MEASURED VERTICAL DISTANCE FOR MICROPROX ( MICROMETERS)
2870 IF CH = 3 THEN W(NO) = 7692.30769# * (VOLTS(NO, CH) - .00007) * .00981
2880  'CONVERTS VOLTS TO GRAM FORCE VERT AND THEN TO NEWTONS
2890 NEXT CH
2900 NEXT NO
2910  '
2920 '*************************************************************************************
2930  'PRINT RESULTS DATA
2940 PRINT " "
2950 PRINT "DIST (MICRO)      FH (N)      PROX(MICRO)         W(N)"
2960 FOR NO = 1 TO SCANS
2961 PRINT USING "####.######"; CM(NO);
2962 PRINT CHR$(9);
2963 PRINT USING "####.######"; FH(NO);
2964 PRINT CHR$(9);
2965 PRINT USING "####.######"; MICPC(NO);
2966 PRINT CHR$(9);
2967 PRINT USING "####.######"; W(NO);
2970 PRINT  " "
2990 NEXT NO
3000 PRINT " "
```

```
3010 PRINT "!!ENSURE THE FOLLOWING DATA IS RECORDED!!"
3020 PRINT "TOTAL ELAPSED TIME (sec) FOR DATA ACQUISITION = ", TOTIME
3030 PRINT " "
3040 PRINT " "
3050  '
3060 '***************************************************************************************
3070  'GENERATE RESULT DATA FILE
3080 INPUT "DO YOU WISH TO SAVE THE RESULTS DATA  (Y/N)  ", QUES$
3090 IF QUES$ = "N" OR QUES$ = "n" GOTO 3250
3100 INPUT "ENTER NAME OF THE DATA FILE TO CREATE (IE RESULT##.DAT)  ", FILE$
3110 OPEN FILE$ FOR OUTPUT AS 1
3120 FOR NO = 1 TO SCANS
3130 PRINT #1, USING "####.######"; CM(NO);
3140 PRINT #1, CHR$(9);
3150 PRINT #1, USING "###.######"; FH(NO);
3160 PRINT #1, CHR$(9);
3170 PRINT #1, USING "###.##"; MICPC(NO);
3180 PRINT #1, CHR$(9);
3190 PRINT #1, USING "###.######"; W(NO);
3200 PRINT #1, " "
3210 NEXT NO
3220 CLOSE #1
3230  '
3240 '***************************************************************************************
3250 INPUT "WOULD YOU LIKE TO TRY ANOTHER RUN  (Y/N)  ", QUES2$
3260 IF QUES2$ = "Y" OR QUES2$ = "y" GOTO 1140
3270 END
3280 CALL GPIB(CMD$, DUMMY%, FLG%, BRD%)
3290 IF FLG% <> 0 THEN PRINT " ERROR IN "; CMD$; VAR$; "FLAG = HEX "; HEX$(FLG%)
3300 RETURN
3310 CALL GPIB(CMDOUT$, MSG$, FLG%, BRD%)
3320 IF FLG% <> 0 THEN PRINT " ERROR IN "; MSG$; "FLAG = HEX "; HEX$(FLG%)
3330 RETURN
3340 CALL GPIB(CMDIN$, DAT$, FLG%, BRD%)
3350 IF FLG% <> 0 THEN PRINT " DATA INPUT ERROR FLAG = HEX "; HEX$(FLG%)
3360 RETURN
```

```
1000 '***************************************************************************************
1010  '
1020  '                    PROGRAM EVAL10.BAS
1030  '
1040  '                          * * *
1050  '                    VICKER'S INDENTER
1060  '                          * * *
1070  '
1080  '                   REVISION DATE  941112
1090 '***************************************************************************************
1100  'THIS VERSION OF EVAL10.BAS (original) HAS BEEN MODIFIED TO ACCOUNT FOR
1110  'FOR TYPE II AND TYPE III DEBONDING.  THE PROGRAM IS DIVIDED INTO THREE
1111  'SUBSECTIONS (CIRCULAR FORWARD LATERAL FLAKING FOR TYPE II AND TYPE III
1115  'DEBONDING,  RECTANGULAR FORWARD LATERAL FLAKING FOR TYPE II AND
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1120 'TYPE III DEBONDING, AND NO FORWARD LATERAL FLAKING FOR TYPE III DEBONDING).
1125  'THE MODELS FOR FORWARD LATERAL FLAKING HAVE BEEN MODIFIED
1130  'TO ACCOUNT FOR BUCKLING OF THE FILM.  (PROGRAM WRITTEN BY LASCURAIN,
1140  'SECOR, CAMPBELL)
1150  '
1160 '***************************************************************************************
1170  'DIMENSIONALIZE ARRAYS
1180 DIM W(325)              'ARRAY FOR VERTICAL LOAD
1190 DIM FH(325)             'ARRAY FOR HORIZONTAL LOAD
1200 DIM HOR(325)            'ARRAY FOR MODIFIED HORIZONTAL LOAD
1201 DIM LVDT(325)           ' ARRAY FOR LVDT
1210 DIM BASE(350)           'ARRAY FOR BASELINE
1220 DIM SHEAR(325, 4)       'ARRAY FOR DATA PRINTING; CHANGE 3 BACK TO 4 WHEN
1230                                   'USING DISTANCE COLUMN
1231 DIM TIHVM(325)          'ARRAY FOR MAX RITTER SHEAR STRESS
1240 DIM TIHV(325)           'ARRAY FOR RITTER SHEAR STRESS
1250 DIM TIEFF(325)          'ARRAY FOR BENJAMIN SHEAR STRESS
1280 DIM A(325, 4)           'ARRAY FOR HOLDING FIRST BASELINE DATA
1300 DIM C(325, 4)           'ARRAY FOR HOLDING RESULT DATA
1310 DIM L(325)              'ARRAY FOR LAMDA VALUES FOR INTEGRATION
1320 DIM R(325)              'ARRAY FOR RADIUS VALUES FOR INTEGRATION
1330 DIM F(325)              'ARRAY FOR INTERMEDIATE INTEGRATION
1340 DIM T(325)
1345 DIM HFILM(325)
1350  '
1360 '***************************************************************************************
1370  'INPUT RESULT FILE TO BE ANALYZED
1380 INPUT "ENTER THE DATE OF THIS EVALUATION (YYMMDD).  ", DATE
1390 INPUT "WHICH RESULT##.DAT IS TO BE EVALUATED?  ", FILE$
1400 INPUT "HOW MANY DATA SETS ARE IN THE RESULT FILE (MAX IS 300)?  ", SCANS
1410 OPEN FILE$ FOR INPUT AS 3
1420 FOR NO = 1 TO SCANS
1430 FOR CH = 0 TO 3
1440 INPUT #3, C(NO, CH)
1445 IF CH = 0 THEN LVDT(NO) = C(NO,CH)
1450 IF CH = 1 THEN FH(NO) = C(NO, CH)
1460 IF CH = 3 THEN W(NO) = C(NO, CH)
1470 NEXT CH
1480 NEXT NO
1490 CLOSE #3
1500 PRINT " "
1510  '
1520 '***************************************************************************************
1530  'INPUT BASELINE DATA.
1580 INPUT "ENTER THE BASELINE FILE TO BE USED.  (BASE##AorB.DAT)  ", FILE$
1590 OPEN FILE$ FOR INPUT AS 1
1600 FOR NO = 1 TO SCANS
1610 FOR CH = 0 TO 3
1620 INPUT #1, A(NO, CH)
1630 IF CH = 1 THEN BASE(NO+30) = A(NO,CH)
1640 NEXT CH
1650 NEXT NO
```

```
1660 CLOSE #1
1661 FOR NO = 1 TO 30
1662 BASE(NO) = 0
1663 NEXT NO
1810 PRINT " "
1820  '
1830 '************************************************************************************
1840  'ENSURE BOTH RESULT FILE AND BASE DATA FILE HAVE THE SAME ZERO
1850  'START POINT
1860 PRINT "ENTER THE HORIZONTAL SHIFT FOR THE BASELINE (MAX 30 POINTS)."
1861 PRINT "IF MORE POINTS ARE DESIRED THE PROGRAM MUST BE MODIFIED"
1870 INPUT "SLIDING THE BASELINE FH COLUMN UP / (DOWN) REQUIRES A + /(-) NUMBER. ",
HOFF
1930   'CALCULATE THE NET HORIZONTAL FORCE VALUES
1940 FOR NO = 1 TO SCANS
1950 HOR(NO) = FH(NO) - BASE(NO + 30 + HOFF)  'SUBTRACT OFF BASELINE FROM HORIZONTAL
1990 NEXT NO
2000 PRINT " "
2010 PRINT " "
2020   ' SAVE TO DATA FILE THE RESULTS - THE BASELINE
2021 INPUT "DO YOU WISH TO SAVE THE RESULTS FILE MINUS THE BASELINE (Y/N)?  ",QUES$
2022 IF QUES$="N" OR QUES$="n" THEN GOTO 2043
2023 INPUT "ENTER THE NAME OF THE DATA FILE TO CREATE (IE RESB##.DAT)  ", FILE$
2024 OPEN FILE$ FOR OUTPUT AS 1
2025 FOR NO =1 TO SCANS
2026 PRINT #1, USING "####.######"; LVDT(NO);
2027 PRINT #1, CHR$(9);
2028 PRINT #1, USING "####.######"; HOR(NO);
2029 PRINT #1, CHR$(9);
2030 PRINT #1, USING "####.######"; W(NO);
2031 PRINT #1, CHR$(9);
2032    'FH(NO) AND BASE(NO+HOFF) WILL BE SENT TO DATA FILE AS WELL TO CONFIRM
2033       ' THAT THE ZERO OFF SET OF HORIZONTAL AND BASELINE DATA HAS BEEN
CORRECTLY PERFORMED
2034 PRINT #1, USING "####.######"; BASE(NO + 30 + HOFF);
2035 PRINT #1, CHR$(9);
2036 PRINT #1, USING "####.######"; FH(NO);
2037 PRINT #1,""
2038 NEXT NO
2040 CLOSE #1
2041 PRINT "DATA HAS BEEN RECORDED"
2042'*************************************************************************************
2043 PRINT ""
2044 'MOMENT EFFECT FROM INDENTER MUST BE SUBTRACTED OFF VERTICAL
2045 PRINT "MOMENT FROM INDENTER MUST BE SUBTRACTED OFF VERTICAL"
2046 FOR NO = 1 TO SCANS
2047 '    CALIBRATION CURVE  FOR MOMENT (FHCOR VS WCOR)
2048 IF HOR(NO) < .224  THEN W(NO) = W(NO) - (.00686 + 1.72871* HOR(NO))
2049 '    THERE IS  ALSO A MAX VERTICAL CORRECTION FOR MOMENT
2050 IF HOR(NO) >= .224  THEN W(NO) = W(NO) - .314
2051 NEXT NO
2052'*************************************************************************************
```

```
2053   ' SAVE TO DATA FILE (MOMENT CORRECTION MADE TO VERTICAL)
2054 INPUT "DO YOU WISH TO SAVE THE DATA FILE FOR MOMENT CORRECTION (Y/N)? ",QUES$
2055 IF QUES$="N" OR QUES$="n" THEN GOTO 2068
2056 INPUT "ENTER THE NAME OF THE DATA FILE TO CREATE (IE RESM##.DAT)  ", FILE$
2057 OPEN FILE$ FOR OUTPUT AS 1
2058 FOR NO =1 TO SCANS
2059 PRINT #1, USING "####.######"; LVDT(NO);
2060 PRINT #1, CHR$(9);
2061 PRINT #1, USING "####.######"; HOR(NO);
2062 PRINT #1, CHR$(9);
2063 PRINT #1, USING "####.######"; W(NO);
2064 PRINT #1,""
2065 NEXT NO
2066 CLOSE #1
2067 PRINT "DATA HAS BEEN RECORDED"
2068 PRINT "
2069 INPUT "DO YOU WISH TO INPUT DATA INTO MODEL EQUATIONS (CONTINUE PROGRAM)
(Y/N)?  ",QUES$
2070 IF QUES$="N" OR QUES$="n" THEN GOTO 4550
2071 PRINT " ********************************************************************************"
2072  ' DATA IS NOW IMPUTED INTO MODEL EQUATIONS FOR TIHV AND HOR
2073  PRINT " "
2074   'INPUT THE SCRATCH TRACK DATA AND MATERIAL PROPERTIES
2076 INPUT "ENTER BMIN, THE HALF TRACK MIN WIDTH (um)  ", BMIN
2077 INPUT "ENTER T, THE FILM THICKNESS (um) (0.4)  ", T
2080 INPUT "ENTER NU, THE POISSONS RATIO OF FILM (Cr BULK = 0.21)  ", NU
2081 INPUT "ENTER E, THE ELASTIC MODULUS FOR THE FILM (GPa)  ", E
2082 E = E*1000000000
2084 BMIN = BMIN / 1000000!'CONVERTS HALF TRACK MIN WIDTH (um) TO (m)
2086 T = T / 1000000!  'CONVERTS FILM THICKNESS (um) TO (m)
2087 DEPTH = .28564 * SQR(2) * BMIN     'PENETRATION DEPTH OF INDENTER
2088 PI = 3.14159265359#
2090 THETA = (136 / 180 * PI)        'CONVERTS VICKERS ANGLE TO RADIANS
2091 '
2092'*************************************************************************************
2093 'CALCULATE HFILM FOR TYPE II AND TYPE III
2094'*************************************************************************************
2095 '
2104 INPUT "ENTER 2 FOR TYPE II DEBONDING / ENTER 3 FOR TYPE III DEBONDING.  ",VALU$
2105 IF VALU$ = "2" THEN GOTO 2125
2106            ' TYPE III RITTER MODEL FOR HFILM
2107 PRINT " "
2108 PRINT "RITTER'S MODEL FOR TYPE III DEBONDING WILL BE USED TO DETERMINE TIHV"
2109 PRINT " "
2110 INPUT "ENTER THE SUBSTRATE HARDNESS (GPa)   ",HS
2111 HS = HS *1000000000
2112 INPUT "ENTER THE RESIDUAL STRESS IN FILM (+/- FOR TENSION/COMPR) IN MPa    ",
SIGMARES
2113 SIGMARES = SIGMARES*1000000
2114 C = BMIN - (T * TAN(THETA / 2)) 'HALF TRACK WIDTH OF
2120   'INDENTER/SUBSTRATE INTERFACE
2121 FOR NO = 1 TO SCANS
```

```
2122 HFILM(NO) = (W(NO) - 3*HS * C^2) / (3*(BMIN^2 - C^2))     ' (N/m^2)
2123 NEXT NO
2124 GOTO 2180
2125              ' TYPE II RITTER MODEL FOR HFILM
2126 PRINT " "
2127 PRINT "RITTER'S MODEL FOR TYPE II DEBONDING WILL BE USED TO DETERMINE TIHV"
2128 PRINT " "
2129 FOR NO = 1 TO SCANS
2130 HFILM(NO) = W(NO) / (3*(BMIN^2))
2131 NEXT NO
2132 '
2133'*****************************************************************************************
2134 '  PRINT PARAMETERS TO SCREEN
2135'*****************************************************************************************
2180 FOR CNT = 1 TO 10
2190 PRINT " "
2200 NEXT CNT
2210 PRINT "DATE OF EVALUATION IS ", DATE
2240 PRINT "THE HALF TRACK MIN WIDTH (um) BMIN = ", BMIN * 1000000
2270 PRINT "FILM THICKNESS (um) T= ", T * 1000000
2320 PRINT "POISSONS RATIO OF FILM  NU=  ", NU
2321 PRINT "ELASTIC MODULUS (Pa) E =  ", E
2325 PRINT "INDENTATION HARDNESS OF SUBSTRATE (N/m^2) HS= ", HS
2330   '
2340'*****************************************************************************************
2341 'CALCULATE Tihv(max)   (MODIFIED RITTER MAX VALUE)
2342'*****************************************************************************************
2350  'DETERMINE VARIOUS CONSTANTS AND PARAMETERS REQUIRED FOR SHEAR VALUES.
2400 PHI = SQR(6 * (1 - NU) / (4 + NU))      'USED IN BESSEL FUNC APPROX
2440 Z = SQR(2) * PHI * BMIN / T        'ARGUMENT FOR BESSEL FUNC
2450 PRINT "PHI = ", PHI, " ", "Z = ", Z
2451 MU = 4
2452 '      BESSD = APPROX DERIVATIVE OF BESSEL SECOND KIND FIRST ORDER
2453 '      BESS = APPROX  BESSEL SECOND KIND FIRST ORDER
2454 BESSED = -(1 + (NU+3)/(8*Z) + ((NU-1)*(NU+15))/(128*Z^2))
2455 BESS = 1 + (NU-1)/(8*Z) + ((NU-1)*(NU-9))/(128*Z^2)
2456 DENOM = BESSED/(BESS*PHI) + (NU*T)/(SQR(2)*BMIN*PHI^2)
2460 FOR NO = 1 TO SCANS
2461 TIHVM(NO) = -.6875*HFILM(NO) / DENOM
2465 ' THIS GIVES THE MAXIMUM VERTICAL SHEAR STRESS FROM MOD RITTER'S EQN
2466 NEXT NO
2467 '
2470'*****************************************************************************************
2475 ' NEXT SECTION FOR DETERMINING FLF (CIRCULAR / RECTANGULAR) OR NO FLF
2476'*****************************************************************************************
2477 '
2478 INPUT "ENTER 1 FOR FORWARD LATERAL FLAKING OR 2 FOR NO FORWARD LATERAL
FLAKING.  " ,QUES$
2479 IF QUES$="2" THEN GOTO 3525   ' GOING TO THIS LINE NUMBER MODEL NO FLF USED
2480 PRINT " "
2485 PRINT "THE FORWARD LATERAL FLAKING MODEL  (FLF) WILL BE USED"
2487 PRINT " "
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2490 '
2491 INPUT "ENTER 1 FOR CIRCULAR FLF OR ENTER 2 FOR RECTANGULAR FLF  ",QUES$
2492 IF QUES$="2" THEN GOTO 3205  'GOING TO THIS LINE NUMBER REC FLF USED
2493 PRINT " "
2494 PRINT "THE CIRCULAR FORWARD LATERAL FLAKING MODEL WILL BE USED"
2496 PRINT " "
2497 '
2498'*****************************************************************************************
2500  ' THE MODEL FOR CIRCULAR FORWARD LATERAL FLAKING (TYPE II / TYPE III)
2501'*****************************************************************************************
2502 '
2503 INPUT "ENTER BMAX, THE HALF FLAKE DIAMETER (um)   ", BMAX
2504 BMAX = BMAX / 1000000!'CONVERTS BMAX FROM (um) TO (m)
2505 INPUT "ENTER BINT, THE FLAKE INTERMEDIATE VALUE (um)  ",BINT
2506 BINT = BINT / 1000000!'CONVERTS BINT FROM (um) TO (m)
2507 RATIO1 = BMIN / BMAX   'USED FOR CALCULATING INV SIN
2508 ALFA = ATN(RATIO1 / SQR(-RATIO1 * RATIO1 + 1))  'THIS IS THE COMPUTER'S
2509   'WAY OF CALCULATING INV SIN(RATIO).  ALFA IS THE ANGLE BETWEEN THE
2510   'HORIZONTAL AND THE LINE BETWEEN THE CENTER OF THE FLAKE CIRCLE AND THE
2511   'CORNER OF THE INDENTER.
2512 AREA =  PI * BMAX ^ 2  - 2*(( BMAX ^ 2 * ALFA) + (BMAX * BMIN * COS(ALFA)))  'THIS IS
THE
2513   'AREA (m^2) IN FRONT OF THE INDENTER OVER WHICH THE SHEAR STRESSES ACT.
2514 PRINT "AREA OF FLAKING IN FRONT OF THE INDENTER (um^2) =  ", AREA
2515'*****************************************************************************************
2516 'CALCULATE Tihv FOR CIRCULAR FLAKING (FLF)
2517'*****************************************************************************************
2518   'DOUBLE INTEGRATION OF SHEAR OVER THE AREA OF FLAKE CIRCLE.
2519 FOR NO = 1 TO SCANS
2520 M =  BMIN - SQR( BMAX^2 - BMIN^2) + 2*(SQR(BMAX^2 - BINT^2))
2521            'DISTANCE FROM CENTER OF INDENTER TO CENTER  'OF FLAKE CIRCLE
2522 IF M < 0 THEN GOTO 2530
2523 UPLAM = ATN(BINT / (BMIN - SQR(BMAX^2 - BMIN^2) + SQR( BMAX^2 - BINT^2)))
2524          'UPPER LIMIT OF LAMDA IF M IS GREATER OR EQUAL TO ZERO
2525 GOTO 2540
2530 UPLAM =  PI + ATN(BINT / (BMIN - SQR(BMAX^2 - BMIN^2) + SQR( BMAX^2 - BINT^2)))
2531          'UPPER LIMIT OF LAMDA IF M IS LESS THAN ZERO
2540 E1 = (M ^ 2 - BMAX ^ 2)        'COMBINING TWO CONSTANTS FOR FUTURE USE
2550 N = 8                         'NUMBER OF INTERVALS FOR SIMPSONS RULE
2570 LOWLAM = 0                    'LOWER LIMIT OF INTEGRATION WRT LAMDA.
2580  'LAMDA IS THE ANGLE FORMED BY THE HORIZONTAL AND A LINE FROM THE INDENTER
2590   'CENTER TO AN APPROX TANGENT OF THE FLAKE CIRCLE.
2610   'INTEGRATION WRT LAMDA
2620 LAMINT = (UPLAM - LOWLAM) / N  'LAMDA INTERVAL SIZE
2630 FOR I = 1 TO N + 1
2640 L(I) = LOWLAM + (I - 1) * LAMINT
2650 LOWRAD = SQR(2) * BMIN     'LOWER LIMIT OF INTEGRATION WRT RADIUS
2651 IF M < 0 THEN GOTO 2662
2660 UPRAD = M * COS(L(I)) + SQR(ABS(M ^ 2 * (COS(L(I))) ^ 2 - E1))   'UPPER RADIUS (M
POSITIVE)
2661 GOTO 2670
2662 UPRAD = M * COS(L(I)) - SQR(ABS(M ^ 2 * (COS(L(I))) ^ 2 - E1))   'UPPER RADIUS (M
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NEGATIVE)
2670  'LIMIT OF INTEGRATION WRT RADIUS.  AS THE PROGRAM APPROACHES THE UPPER
2680  'LIMIT OF THE VARIABLE R, THE SQR(xxx) APPROACHES ZERO.  HOWEVER, DUE TO
2690  'COMPUTER ACCURACY, xxx MIGHT BE COMPUTED AS A NEGATIVE NUMBER TO THE
2700  'ORDER 10E-18.  THIS IS ESSENTIALLY ZERO, BUT THE COMPUTER ERRORS ON
2710  'TAKING THE SQUARE OF A NEGATIVE NUMBER, HENCE THE ABS VALUE IS USED.
2720 RADINT = (UPRAD - LOWRAD) / N
2730 FOR H = 1 TO N + 1
2740 R(H) = LOWRAD + (H - 1) * RADINT
2750  'THE FOLLOWING  IS THE EQUATION WHICH IS BEHIND THE DOUBLE INTEGRAL
2770 T(H) = 2*SQR(2)*BMIN*TIHVM(NO)
2830 NEXT H
2840 SUMEVE = 0
2850 FOR H = 2 TO N STEP 2
2860 SUMEVE = SUMEVE + T(H)
2870 NEXT H
2880 SUMODD = 0
2890 FOR H = 3 TO N - 1 STEP 2
2900 SUMODD = SUMODD + T(H)
2910 NEXT H
2920 F(I) = (RADINT / 3) * (T(1) + 4 * SUMEVE + 2 * SUMODD + T(N + 1))
2930 NEXT I
2940 SUMEVE = 0
2950 FOR H = 2 TO N STEP 2
2960 SUMEVE = SUMEVE + F(H)
2970 NEXT H
2980 SUMODD = 0
2990 FOR H = 3 TO N - 1 STEP 2
3000 SUMODD = SUMODD + F(H)
3010 NEXT H
3020  '
3030 '**************************************************************************************
3035 ' FIND THIV (VERTICAL COMPONENT FOR SHEAR STRESS FROM MODIFIED RITTER'S EQN)
3040 TIHV(NO) = ((LAMINT / 3) * (F(1) + 4 * SUMEVE + 2 * SUMODD + F(N + 1))) / AREA
3045 '
3046'**************************************************************************************
3047 'CALCULATE Tieff FOR CIRCULAR FLAKING (FLF)
3048'**************************************************************************************
3049 '
3052 '  THE BUCKLING OF THE FILM MUST BE CONSIDERED IN THE PLOUGHING TERM FOR
HORIZONTAL
3053 '  BALANCE OF FORCES
3054 LENGTH =  SQR( BMAX^2 - BMIN^2) + BMAX  ' THIS IS THE LENGTH OF FILM BUCKLE
3055 SIGMACR = ((PI^2)*E)/((1 - NU^2) * 12 * ((LENGTH/T)^2))
3056  '  WHERE  SIGMACR IS THE CRITICAL  BUCKLING STRESS FOR THE FILM
3057  SIGMAY = HFILM(NO)/ 3.2
3058 '  WHERE SIGMAY IS THE YIELD STRENGTH OF THE FILM
3059 IF SIGMACR < SIGMAY THEN SIGMA = SIGMACR
3060 IF SIGMACR > = SIGMAY THEN SIGMA = SIGMAY
3061 ' THE ABOVE TWO LINES SHOW THAT THE MAX BUCKLING STRESS MUST BE YIELD
STRENGTH
3062 '
```

```
3063 '        FIND Tieff (SHEAR STRESS FOR HORIZONTAL COMPONENT)
3064 SHS = (2 * C ^ 2 * HS) / (5.5 * SIN(THETA / 2))          ' SHEAR OF SUBSTRATE
3065 PS = (C ^ 2 * HS) /(TAN(THETA / 2))          ' PLOUGHING OF SUBSTRATE
3066 BFILM = SIGMA*(BMIN+C)*T        'BUCKLING OF FILM FOR TYPE III (THICKNESS OF FILM
USED)
3067 BFILM2 = SIGMA*(BMIN+C)*DEPTH   'BUCKLING OF FILM FOR TYPE II (PENETRATION
DEPTH USED)
3068 IF VALU$ = "3" THEN TIEFF(NO) = (1 / AREA) * (HOR(NO) - PS - BFILM)   ' TYPE III DEBOND
3069 IF VALU$ = "2" THEN TIEFF(NO) = (1 / AREA) * (HOR(NO) - BFILM2)   ' TYPE II DEBOND
3071 ' THE ABOVE TWO LINES ARE HORIZONTAL FORCE BALANCE FOR TYPE III AND TYPE II
3072 '
3073  'UNITS FOR BOTH TIHV AND TIEFF ARE (N/m^2)
3080 TIHV(NO) = TIHV(NO) * .000001   'CONVERTS TIHV (N/m^2) TO (MPa)
3090 TIEFF(NO) = TIEFF(NO) * .000001 'CONVERTS TIEFF (N/m^2) TO (MPa)
3100 NEXT NO
3110 GOTO 3840  'THIS LINE NUMBER GOES TO SECTION TO PRINT DATA
3120'
3200'*************************************************************************************
3203'*************************************************************************************
3204 '
3205'*************************************************************************************
3206 '  MODEL FOR RECTANGULAR FORWARD LATERAL FLAKING (TYPE II / TYPE III)
3208'*************************************************************************************
3210 '
3220 PRINT " "
3230 PRINT "MODEL FOR RECTANGULAR FORWARD LATERAL FLAKING (FLF) WILL BE USED"
3235 PRINT " "
3236 PRINT "BMAX IS THE HALF DIAMETER OF FLAKE PERPENDICULAR TO SCRATCH
DIRECTION"
3237 INPUT "ENTER BMAX, THE HALF FLAKE DIAMETER  (um)  ", BMAX
3238 BMAX = BMAX / 1000000!       'CONVERTS HALF TRACK MAX WIDTH (um) TO (m)
3239 PRINT " "
3240 PRINT "THE LENGTH OF THE FLAKE IS MEASURED PARALLEL TO THE SCRATCH
DIRECTION"
3245 PRINT "THE LENGTH IS FROM THE INDENTER TO THE END OF THE FLAKE"
3246 INPUT "ENTER THE LENGTH OF THE FLAKE (um) ", LENGTH2
3250 LENGTH2 = LENGTH2 / 1000000
3255 FOR NO = 1 TO SCANS
3256'*************************************************************************************
3257 ' CALCULATE Tihv FOR RECTANGULAR FLAKING (FLF)
3258'*************************************************************************************
3259  '   THE NEXT LINE IS THE SOLUTION TO A CLOSED FORM INTEGRAL FOR TIHV
3260  '    THIS IS SOLUTION TO INTEGRAL : SQR(2)* Bmin * Tihv max / Z  WHERE  THE LIMITS
3261  '         OF Z ARE FROM  [LENGTH2 + SQR(2) * Bmin]  TO  [ SQR(2) * Bmin]
3262 TIHV(NO) = ((SQR(2) * BMIN *TIHVM(NO)) * ( LOG(LENGTH2 + SQR(2) * BMIN) - LOG(SQR(2)
* BMIN))) / LENGTH2
3263 '
3265 ' ************************************************************************************
3270 ' CALCULATE Tieff FOR RECTANGULAR FLAKING (FLF)
3275'*************************************************************************************
3280 '
3285 '   THE BUCKLING OF THE FILM MUST BE CONSIDERED IN THE PLOUGHING
```

```
3290 '   TERM FOR HORIZONTAL BALANCE OF FORCES
3300 '
3310 AREA2 = LENGTH2 * BMAX       'THIS IS THE AREA (m^2) IN FRONT OF INDENTER
3315  ' OVER WHICH THE SHEAR STRESSES ACT
3355 SIGMACR = ((PI^2)*E)/((1 - NU^2) * 12 * ((LENGTH2/T)^2))
3356  '  WHERE  SIGMACR IS THE CRITICAL  BUCKLING STRESS FOR THE FILM
3357  SIGMAY = HFILM(NO)/ 3.2
3358 '   WHERE SIGMAY IS THE YIELD STRENGTH OF THE FILM
3359 IF SIGMACR < SIGMAY THEN SIGMA = SIGMACR
3360 IF SIGMACR > = SIGMAY THEN SIGMA = SIGMAY
3361 ' THE ABOVE TWO LINES SHOW THAT THE MAX BUCKLING STRESS MUST BE YIELD
STRENGTH
3362 '
3363 '        FIND Tieff (SHEAR STRESS FOR HORIZONTAL COMPONENT)
3364 SHS = (2 * C ^ 2 * HS) / (5.5 * SIN(THETA / 2))          ' SHEAR OF SUBSTRATE (NOT USED IN
EQUATION)
3365 PS = (C ^ 2 * HS) /(TAN(THETA / 2))            ' PLOUGHING OF SUBSTRATE
3366 BFILM = SIGMA*(BMIN+C)*T         'BUCKLING OF FILM FOR TYPE III (THICKNESS OF FILM
USED)
3367 BFILM2 = SIGMA*(BMIN+C)*DEPTH    'BUCKLING OF FILM FOR TYPE II (PENETRATION
DEPTH USED)
3368 '
3369 IF VALU$ = "3" THEN TIEFF(NO) = (1 / AREA2) * (HOR(NO) - PS - BFILM)  'TYPE III DEBOND
3370 IF VALU$ = "2" THEN TIEFF(NO) = (1 / AREA2) * (HOR(NO) - BFILM2)  'TYPE II DEBOND
3371 '
3372 ' THE ABOVE THREE LINES ARE HORIZONTAL FORCE BALANCE FOR TYPE III AND TYPE
II
3374                  'UNITS FOR BOTH TIHV AND TIEFF ARE (N/m^2)
3380 TIHV(NO) = TIHV(NO) * .000001       'CONVERTS TIHV (N/m^2) TO (MPa)
3390 TIEFF(NO) = TIEFF(NO) * .000001           'CONVERTS TIEFF (N/m^2) TO (MPa)
3400 NEXT NO
3401 PRINT "AREA OF FLAKING IN FRONT OF THE INDENTER (m^2) =  ", AREA2
3410 GOTO 3840    ' THIS LINE NUMBER GOES TO SECTION TO PRINT DATA
3411 '
3500'***************************************************************************************
3515'***************************************************************************************
3516 '
3520'***************************************************************************************

3525 ' MODEL FOR NO FORWARD LATERAL FLAKING (TYPE II / TYPE III)
3526'***************************************************************************************
3530 '
3535 PRINT " "
3540 PRINT "THE MODEL FOR NO FORWARD LATERAL FLAKING WILL BE USED"
3545 PRINT " "
3550 FOR NO = 1 TO SCANS
3555'***************************************************************************************
3560 ' CALCULATE Tihv FOR NO FORWARD LATERAL FLAKING
3565'***************************************************************************************
3570 TIHV(NO) = TIHVM(NO)
3575 '
3580'***************************************************************************************
```

```
3585 'CALCULATE Tieff FOR NO FORWARD LATERAL FLAKING
3590'*************************************************************************************
3600 C = BMIN - (T * TAN(THETA/2))
3601 PS = (C^2 * HS) / (TAN(THETA/2)) ' PLOUGHING OF SUBSTRATE
3602 PF = HFILM(NO) *((BMIN+C)*T)   ' PLOUGHING OF FILM
3603 AREA3 = 3*C^2   'AREA OF SCRATCH AT INTERFACE (EQN 2.26 SECOR THESIS)
3604 '
3605 IF VALU$ = "3" THEN TIEFF(NO) = (1 / AREA3) * (HOR(NO) - PS - PF)   ' TYPE III DEBOND
3606 IF VALU$ = "2" THEN TIEFF(NO) = (1 / AREA3) * (HOR(NO) - PF)   ' TYPE II DEBOND
3607 '
3620 '  THE ABOVE EQUATION (TIEFF) WAS DEVELOPED BY LASCURAIN (PAGE 97 OF THESIS)
3621 '  MODIFIED BY SECOR FOR CHANGE IN ORIENTATION INDENTER (PAGE 23 - 26 OF THESIS)
3622 '     THE TERMS FOR SHEARING OF SUBSTRATE AND SHEARING  OF FILM HAVE BEEN
3623 '      BEEN REMOVED FROM ORIGINAL EQUATION
3630              'UNITS FOR BOTH TIHV AND TIEFF ARE (N/m^2)
3640 TIHV(NO) = TIHV(NO) * .000001      'CONVERTS TIHV (N/m^2) TO (MPa)
3650 TIEFF(NO) = TIEFF(NO) * .000001          'CONVERTS TIEFF (N/m^2) TO (MPa)
3660 NEXT NO
3661 PRINT "AREA OF FLAKING IN FRONT OF THE INDENTER (m^2) =  ", AREA3
3680 '
3800'*************************************************************************************
3810'*************************************************************************************
3811 '
3812'*************************************************************************************
3840  'PRINT SHEAR DATA
3845'*************************************************************************************
3846 '
3850  'TACT IS THE TOTAL INTERFACIAL SHEAR STRENGTH BASED ON THE HORIZONTAL AND
3860  'VERTICAL FORCES
3870 FOR NO = 1 TO SCANS
3880 FOR CH = 0 TO 3
3881 IF CH = 0 THEN SHEAR(NO, CH) = LVDT(NO)
3900 IF CH = 1 THEN SHEAR(NO, CH) = TIHV(NO)
3910 IF CH = 2 THEN SHEAR(NO, CH) = TIEFF(NO)
3920 IF CH = 3 THEN SHEAR(NO, CH) = TIEFF(NO) + TIHV(NO)   'BOTH TIEFF AND TIHV
3930  'ACT OVER THE SAME AREA; AREAS CANCEL OUT IN FORCE BALANCE EQUATION.
3940 NEXT CH
3950 NEXT NO
3960 PRINT " "
3970 PRINT "          LVDT      TIHV (MPa)  TIEFF (MPa)  TACT (MPa)"
3980 FOR NO = 1 TO SCANS
3990 FOR CH = 0 TO 3
4300 PRINT USING "#######.####"; SHEAR(NO, CH); 'NOTE THE SUM IS THE ACTUAL
4310 PRINT CHR$(32);    '               SHEAR STRESS, TACT
4320 NEXT CH
4330 PRINT " "
4340 NEXT NO
4350 PRINT " "
4360 BEEP
4370  '
4380 '*************************************************************************************
4390  'GENERATE SHEAR DATA FILE
```

```
4400 INPUT "DO YOU WISH TO SAVE THE SHEAR DATA  (Y/N)  ", QUES$
4410 IF QUES$ = "N" OR QUES$ = "n" THEN GOTO 4550
4420 INPUT "ENTER NAME OF DATA FILE TO BUILD (IE SHEAR##.DAT)  ", FINAL$
4430 OPEN FINAL$ FOR OUTPUT AS 1
4440 FOR NO = 1 TO SCANS
4450 FOR CH = 0 TO 3
4460 PRINT #1, USING "#######.####"; SHEAR(NO, CH);
4470 PRINT #1, CHR$(9);
4480 NEXT CH
4490 PRINT #1, " "
4500 NEXT NO
4510 CLOSE #1
4520 PRINT " "
4530  '
4540 '*******************************************************************************************
4550 END
```

LIST OF REFERENCES

1. Lascurain, D. P., *A Scratch Test for the measurement of Adhesion at Thin Film Ceramic Interfaces*, Master's Thesis, Naval Postgraduate School, Monterey, California, March 1993.

2. Secor, Daniel E., *A Constant Depth Scratch Test For the Measurement of Adhesion At Film-Substrate Interfaces*, Master's Thesis, Naval Postgraduate School, Monterey, California, March 1994.

3. Dutta, I., Lascurain, D. P., and Secor, E.D., "Measurement of Adhesion at Film-Substrate Interfaces by a Constant Depth Scratch Testing," Department of Mechanical Engineering, Naval Postgraduate School, Monterey, California.

4. Strong, J. , *Review of Science Instruments*, no. 16, p. 143, 1935.

5. Benjamin, P., and Weaver, C., "The Adhesion of Metals to Crystal Faces," Proceedings of the Royal Society of London, v. A274, pp. 267 - 273, 1963.

6. *Annual Book of ASTM 6 Standards, Standard Test Methods for Adhesion of Metallic Coatings*, v. 205, p. 327, 1991.

7. Valli, J., " A Review of Adhesion Test Methods for Thin Hard Coatings," *Journal of Vacuum Science and Technology*, v. A4, no. 6, pp. 3007-3014, November / December 1986

8. Osaka, T., Nagata, H., Nakajima, E., and Koiwa, I., "Metalization of AlN Ceramics by Electroless Ni - P Plating," *Journal of the Electrochemical Society: Solid - State Science and Technology*, v.133, pp. 2345-2349, November 1986.

9. Sachse, W., Kim, K. Y., and Conway, H. D., "Determination of the Bond Strength of a Metal Coating on a Substrate," *Materials Research Symposium Proceedings*, v. 154, pp. 293-303, 1989.

10. Kuwahara, K., Hirota, H., and Umemeto, N., in *Adhesion Measurement of Thin Films, Thick Films, and Bulk Coatings,* K.L. Mittal, ed., ASTM STP 640, ASTM, Philadelphia, p. 198, 1978.

11. Aravas, N., Kim, K.S., Loukis, M.J., "On the Mechanics of Adhesion Testing of Flexible Films," *Material Science and Engineering*, v. A107, pp. 159-168, 1989.

12. Matthewson, M. J., "Adhesion Measurements of Thin Films by Indentation," *Applied Physics Letters,* v. 49, No. 21, pp. 1426-1428, 24 November 1986.

13. Lin, M. R., Ritter, J. E., Rosenfield, L., and Lardner, T. J., "*Measuring the Interfacial Shear Strength of Thin Polymeric Coatings on Glass*," Proceedings of the Materials Research Society Symposium, v. 130. pp. 237-242, 1989.

14. Matthewson, M.J., "Axi-Symmetric Contact on Thin Compliant Coatings," *Journal of the Mechanics and Physics of Solids*, v. 29, no. 2, pp. 89-113, 1981.

15. Ritter, J. E., Lardner, T. J., Rosenfeld, L., and Lin, M. R., "Measurement of Adhesion of Thin Polymer Coatings By Indentation," *Journal of Applied Physics*, v. 66, no. 8, pp. 3626-3634, 15 October 1989.

16. Pharr, G. M., Oliver, W. C., "Measurement of Thin Film Mechanical Properties Using Nanoindentation," *MRS Bulletin*, v. XVII, no. 7, July 1992.

17. Sekler, J. Steinman, P. A., and Hinterman, H. E., "The Scratch Test: Differential Critical Load Determination Techniques," *Surface and Coatings Technology*, v. 36, pp. 519-529, 1988.

18. Heavens, O.S., "Some Factors Influencing the Adhesion of Films Produced by Vacuum Evaporation," *Journal of Physics and Radium*, v. 11, pp. 355-360, 1950.

19. Benjamin, P. and Weaver, C., "Measurement of Adhesion of Thin Films," *Proceedings of the Royal Society of London*, v. A254, pp. 177-183, 1960.

20. Karnowsky, M.M., and Estill, M.B.,"Scratch Test for Measuring Adherence of thin Films to Oxide Substrates," *The Review of Scientific Instruments,* v. 35, no. 10, pp. 1324-1325, October 1964.

21. Weaver, C., "Adhesion of Thin films," *Journal of Vacuum Science Technology*, v. 12, no. 1, pp. 18-25, 1977.

22. Valli, J. and Mäkelä, U., "Applications of the Scratch Test Methods for Coating Adhesion Assessment," *Wear*, v. 115, pp. 215-221, 1987.

23. Valli, J., Mäkelä, U., Matthews, A., and Murama, V., "TiN Coating Adhesion Studies Using the Scratch Test Method," *Journal of Vacuum Science and Technology*, v. A3, no. 6, pp. 2411-2414.

24. Butler, D. W., Stoddart, C. T. H., and Stewart, P. R., "The Stylus or Scratch Method for Thin Film Adhesion Measurements: Some Observations and Comments," *Journal of Physics D: Applied Physics*, v. 3, pp. 877-833, 1970.

25. Hedenquist, P., Olsson, M., and Jacobson, S., "Failure Mode Analysis of TiN - Coated High Speed Steel: in situ Scratch Adhesion Testing in the Scanning Electron Microscope," *Surface and Coatings Technology*, v. 41, pp. 31-49, 1990.

26. Bull, S. J., "Failure Modes in Scratch Adhesion Testing," *Surface and Coatings Technology*, v. 50, pp. 25-32, 1991.

27. Maan, N., and Groenou, A. B., "Low Speed Scratch Experiments on Steel," *Wear*, v. 42, pp. 365-390, 1977.

28. Wu, T. W., "Microscratch and Load Relaxation Tests for Ultra-Thin Films," IBM Research Division, and Almaden Research Center, *Materials Research Society*, pp 407, 1991.

29. Wu, T. W., Burn, R. A., Chen, M. M., and Alesopoulos, P.S., "Micro-Indentation and Micro-Scratch Tests on Sub-Micron Carbon Films," IBM Research Division, and Almaden Research Center, *Materials Research Society*, v. 130, 1989.

30. Venkataraman, Chancer K., Kohlstedt, David L., and Gerberich, William W., "Metal -Ceramic Interfacial Fracture Resistance Using the Continuous Microscratch Technique," *Thin Solid Films*, v. 223, pp. 269-275, 1993.

31. Tabor, D. "Junction Growth In Metallic Friction: The Role of Combined Stresses and Surface Contamination," Research Laboratory for the Physics and Chemistry of Solids, Department of Physics, Cambridge University, 1958.

32. F. Black, *Buckling Strength of Metal Structures*, (New York: McGraw Hill , 1952), pp. 4-8, 305-307.

33. Logan, Daryl L,. *Mechanics of Materials*, (New York: Harper Collier Publishers Inc., 1991), p. 579.

34. Smithells, C. J., *Metals Reference Book*, 2nd edition, (London: Butterworths Scientific Publications, 1955).

35. Hunington, H. B., *Sold State Physics*, v. 7, p. 214, 1958.

36. C. D. Hodgman, ed., *Handbook of Chemistry and Physics,* 43rd edition, (Cleveland: Chemical Rubber Publishing Co., 1961).

37. Brady, G., and Clauser, H. R., *Materials Handbook*, (New York: McGraw Hill, 1977), p. 652.

38. Tabor, D., "The Physical Meaning of Indentation and Scratch Hardness," *British Journal of Applied Physics*, v. 7, 1956.

39. Thomas, G. B., Jr., *Calculus and Analytic Geometry*, p. 368, Addison-Wesley Publishing Co., 1968.

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