Mechanical Strength and Tribological Behavior of Ion-Beam-Deposited Boron Nitride Films on Nonmetallic Substrates

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MECHANICAL STRENGTH AND TRIBOLOGICAL BEHAVIOR OF ION-BEAM-DEPOSITED BORON NITRIDE FILMS ON NONMETALLIC SUBSTRATES*

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SUMMARY

An investigation was conducted to examine the mechanical strength and tribological properties of boron nitride (BN) films ion-beam-deposited on silicon (Si), fused silica (SiO_2), gallium arsenide (GaAs), and indium phosphide (InP) substrates in sliding contact with a diamond pin under a load. The results of the investigation indicate that BN films on nonmetallic substrates, like metal films on metallic substrates, deform elastically and plastically in the interfacial region when in contact with a diamond pin. However, unlike metal films and substrates, BN films on nonmetallic substrates can fracture when they are critically loaded. Not only does the yield pressure (hardness) of Si and SiO_2 substrates increase by a factor of 2 in the presence of a BN film, but the critical load needed to fracture increases as well. The presence of films on the brittle substrates can arrest crack formation. The BN film reduces adhesion and friction in the sliding contact. BN adheres to

E-3439

Si and SiO_2 and forms a good quality film, while it adheres poorly to GaAs and InP. The interfacial adhesive strengths were 1 GPa for a BN film on Si and appreciably higher than 1 GPa for a BN film on SiO_2.

INTRODUCTION

Advanced propulsion systems such as gas turbines and adiabatic diesel engines are designed to achieve high efficiency through higher operating temperatures and rotational speeds [1,2]. These conditions lead to two primary problems. First, bearings and other load-carrying contacts are required to operate over a temperature range that exceeds the capability of conventional liquid lubricants: that is, their thermal oxidation limits [3,4]. None of the liquid lubricants available today are useful beyond 350 °C. Obvious candidate materials for fulfilling these needs are inorganic compounds. Indeed, conventional solid lubricants (such as CaF_2/BaF_2 binary eutectic coatings and PbO/SiO_2 coatings in an oxidizing atmosphere, as well as MoS_2 and graphite in a nonoxidizing atmosphere) have been used at high temperatures [2]. The use of ceramics (both in bulk and coating forms) in a vacuum or space environment has tribological benefits, because of the low adhesion of ceramics in contact with themselves or with other solids such as metals [5]. Further, at high temperatures in a vacuum environment, heating a ceramic such as silicon carbide can result in the graphitization of the ceramic surface with the graphite functioning to reduce adhesion and friction. A lubricating film is, therefore, provided from the ceramic material itself [6,7].

Secondly, at very high rotating speeds centrifugal forces from the rolling elements lead to extensive overload of the rotating mechanical components. One solution to this problem is to reduce the density of the material used for the rolling elements and thereby decrease the centrifugal loading. An attractive solution is to use ceramics for the rotating mechanical components [8]. For example, silicon nitride has a density approximately 40 percent that of tool

2

steels; therefore, fundamental and focused research to develop high-temperature ceramics for use as components in mechanical systems is a logical approach to meet the stringent requirements of advanced engine technology.

Ion-beam-deposited boron nitride (BN) films are relatively new materials and are promising ceramics for use not only as high-temperature, wear-resistant, solid lubricating films, but also as adequate gate dielectrics for high-speed III-V semiconductor devices and integrated circuits. This is the most difficult and crucial application of an insulator film [9,10].

The present work continues the ongoing tribological studies with ion-beam-deposited BN films. The objectives of this study were to investigate the tribological properties and mechanical strength (interfacial adhesive and intrinsic cohesive strength) of hard and brittle BN films deposited on silicon-based nonmetallic materials. This study was initiated with well-defined, pure silicon (Si) and fused silica (quartz, SiO_2) substrates rather than complicated structural ceramic substrates such as silicon nitride and silicon carbide. It was aimed at gaining a more fundamental understanding of adhesion between BN and silicon-based materials.

Single-pass scratch experiments were conducted to examine the adhesion, friction, and deformation behavior of the BN films deposited on Si {100} plane and SiO_2 in sliding contact with a diamond pin in room-temperature air at a relative humidity of 45 percent. Comparative scratch experiments were also conducted with BN films deposited on gallium arsenide (GaAs) and indium phosphide (InP) {100} planes sliding in the <110> directions.

MATERIALS

Thin films containing BN have been synthesized by Spire Corporation using an ion beam extracted from a borazine ($B_3N_3H_6$) plasma [11]. The substrates used for scratch experiments were Si, SiO_2, GaAs, and InP (Table I).

3

APPARATUS

The ion source used to deposit BN films is described in reference 11.

The friction and wear apparatus used in this investigation was capable of scratching the surface of a BN film with a rounded diamond pin (a Rockwell cone diamond with a tip radius of 0.2 mm). The apparatus was the Revetest, a commercially purchased device [12-14]. A load was applied by placing deadweights on a pan on top of a rod. The other end of the rod contained the diamond pin. A piezoelectric accelerometer mounted just above the diamond pin detected the acoustic emission released as the coating film was disturbed.

XPS and AES/SIMS systems were also used for chemical composition analyses of the BN films.

EXPERIMENTAL PROCEDURE

The experiments were conducted in a laboratory air atmosphere. The diamond pin surfaces were scrubbed with levigated alumina (1- and 1/4-μm particle diameters) and were rinsed with tap water, then with distilled water, and finally with 200-proof ethyl alcohol before each single-pass sliding experiment. The BN films were also rinsed with 200-proof ethyl alcohol before use. After the surfaces of the pin and the BN film had been dried with nitrogen gas, the specimens were placed in the friction and wear apparatus. The specimen surfaces were then brought into contact and loaded, and the friction experiment was begun. The time in contact before sliding was 30 sec. Each sliding experiment consisted of a single pass with a total sliding distance of approximately 10 mm at a sliding velocity of 12 mm/min. The acoustic emission signal was continuously monitored during sliding, and the wear tracks formed on the BN films were examined by scanning electron and optical microscopes.

RESULTS AND DISCUSSION

Microstructure and Chemical Composition

It was found that the ion-beam-deposited BN films were predominantly amorphous. In addition to a structureless amorphous phase, however, there was a crystalline phase with a size range of 8 to 30 nm. That is, the films were not completely amorphous; properties characteristic of hexagonal BN were detected. This finding agrees with electron microscopy and optical band gap observations [3,4,15].

The XPS and AES/SIM analyses revealed that the BN films were nonstoichiometric, with a B/N ratio of approximately 2, and that they contained small amounts of oxides and carbides in addition to BN [3,4,5,15].

Friction and Wear

When a diamond hemisphere slides on a BN film deposited on a nonmetallic substrate, the surface damage is indicated in two main features. First, the diamond indents and slides on the BN film without itself suffering permanent deformation, but it causes permanent deformation in the BN film - the scratch has the typical grooved appearance characteristic of metals or other ductile materials. Secondly, in spite of this gross plastic deformation, the sliding action also produces visible microscopic cracking of the BN film and the substrate. Microscopic fragmentation of the BN film is occasionally observed in and near the region of contact with the diamond.

Acoustic emissions were released and detected when the intrinsic cohesive bonds in the BN film and/or in the substrate, as well as adhesive bonds between the BN film and the substrate, were broken with a new surface being created. The pattern and intensity of the acoustic emission depend on the nature of the disturbance: plastic flow, cracking, or flaking of fragments.

5

Figure 1 presents typical acoustic emission traces for a BN film deposited
on a nonmetallic substrate. When the BN film surface is brought into contact
with the diamond pin under a small load, which is lower than the critical loads
needed to fracture intrinsic cohesive bonds in the BN film on the substrate
(Table II), no acoustic emission is detected (Fig. 1(a)). After the diamond
had passed over the surface only once, scanning electron microscopic examination
of the wear track indicated that a permanent groove was formed in the BN film,
much like in metallic films under similar conditions [16]. However, the BN film
was not observed to crack with sliding. An increase in load to or above the
critical loads needed to fracture the intrinsic cohesive bonds in the BN film
and in the substrate, however, resulted in a small amount of cracking in and
near the plastically deformed groove. The acoustic emission trace indicated
evidence of a fluctuating acoustic emission signal output (Fig. 1(b)).
Acoustic emission was observed when the sliding appeared to involve small
amounts of cracking in addition to plastic flow. Such acoustic emission is
due to the release of elastic energy when cracks propagate in the BN film and
in the substrate.

When a much higher load was applied to the BN film, the sliding action
produced (in addition to plastic flow) locally gross surface and subsurface
fracturing in the film, in the substrate, and at the interface between the BN
film and the substrate. In such cases the acoustic emission traces are
primarily characterized by chevron-shaped behavior (Fig. 1(c)).

Coefficient of Friction

The coefficients of friction measured in laboratory air for BN films on
the Si, SiO_2, GaAs, and InP substrates are presented in Fig. 2, together with
comparative data for uncoated Si, SiO_2, GaAs, and InP. The coefficients of
friction depend on the type of nonmetallic substrate on which the BN was
deposited as well as on the applied normal load.

The coefficient of friction is lowest with BN films deposited on Si and SiO$_2$ substrates at temperatures of 200 and 350 °C. Good quality films were obtained on Si and SiO$_2$ substrates at these deposition temperatures.

The coefficients of friction for BN films on doped GaAs and Sn-doped InP substrates are relatively higher and are almost the same as those for the uncoated surfaces of GaAs and InP substrates (Figs. 2(c) and (d)). Optical microscopic examination of the wear tracks on the BN films deposited on doped GaAs and InP substrates clearly revealed that the sliding action caused breakthrough of the film in the contact area.

The breakthrough of the BN films is due to poor interfacial adhesive bonds existing between the film and the substrate in and near the diamond contact region. The removal of the BN films induced direct contact of the diamond pin with the doped GaAs and InP substrates. It, therefore, resulted in a higher coefficient of friction.

The BN film adhered poorly to the undoped InP substrate (Fig. 2(d)). However, it did provide relatively lower coefficients of friction than were obtained for uncoated InP. Optical microscopic examination of the wear tracks of the BN film deposited on the undoped InP clearly revealed that the sliding action of the diamond burnished the BN film to the substrate. When this occurs, the diamond does not entirely break through the BN film in the contact area and does not come in direct contact with the undoped InP substrate. The coefficient of friction is not constant and depends on the applied normal load in the ranges indicated in Fig. 2.

There are generally three main observations to be made from the data. First, below a certain load, depending on the substrate, the coefficient of friction decreases steadily as the load is increased. The sliding action with the diamond on the BN film produces predominantly elastic deformation accompanied by a permanent groove. The real area of contact, therefore, is

7

not generally proportional to the load, and there is a corresponding deviation from Amontons' Law. For a first approximation for the load range investigated, the relation between coefficient of friction μ and load W is given by $\mu = kW^{1/3}$ [17,18]. The friction is a function of the shear strength of the elastic contact area.

Second, at higher loads, the friction behavior is modified. There is a very small but definite increase in coefficient of friction corresponding to the increased amount of mean yield pressure P_m in the contact area. The wear track generally consists of plastic deformation and slight cracking of the BN film and substrate. At higher loads, the increase in coefficient of friction with load is due mainly to plastic grooving. This is in direct contrast to the decrease in coefficient of friction with load observed at small loads. The plowing process, which generates plastic grooves and cracking in the BN film, absorbs an additional amount of frictional energy.

Third, with the BN film on Te-doped GaAs, with the BN film on Sn-doped InP, and with the uncoated InP (Figs. 2(c) and (d)), the coefficient of friction increased drastically above a specific load depending on the material. The sliding action with these surfaces produces catastrophic gross surface and subsurface fracturing as well as plastic deformation. Under such conditions wear debris particles and large fracture pits were observed. The area of the fracture pit was a few times larger than that of the plastically deformed groove.

The drastic increase in friction is due to the catastrophic gross surface and subsurface fracturing of the BN film and the substrate. Most of the frictional energy is dissipated in the fracturing process during sliding.

8

Yield Pressure and Meyer's Law

Scratch measurements were conducted with a BN film deposited on the substrates, starting from the smallest loads at which the scratches were visible by optical and scanning electron microscopy.

The width D and height H of a groove with some amount of deformed BN piled up along its sides are defined in Fig. 3. The widths of the grooves reported herein were obtained by averaging the widths from 10 or more measurements of optical or scanning-electron microscope observations. Mean contact pressure (yield pressure) P during sliding may then be defined by $P = W/A_s$, where W is the applied load and A_s is the projected contact area given by $A_s = \pi D^2/8$ (only the front half of the pin is in contact with the flat) [15,19].

The yield pressure over the contact area gradually increased until the deformation passed to a fully plastic state, as presented in Figs. 4(a) to (d). The mean contact pressure at a fully plastic state P_m is proportional to and approximately 25 percent greater than the measured Vickers hardness (Tables II and III).

The yield pressure of Si and SiO_2 increased by a factor of 2 with the presence of BN films (Figs. 4(a) and (b)). When the load exceeds a certain critical value (Fig. 4(a)), the sliding action with the diamond on the BN film deposited on Si produces locally gross surface and subsurface fracturing in the BN film and in the Si substrate. The portion FF in Fig. 4(a) represents the condition of severe fracture, and the yield pressure falls with increasing load.

There was no gross failure of interfacial adhesive bonds between the BN film and the SiO_2 substrate observed over the entire load range (Fig. 4(b)). Therefore, the yield pressure at the fully plastic state remained constant.

The yield pressures for BN films on doped GaAs and on InP are the same as those for the uncoated surfaces of doped GaAs and InP (Figs. 4(c) and (d)). The BN films adhered poorly to the GaAs and InP substrates, and the breakthrough and removal of the BN films induced direct contact of the diamond pin with the doped GaAs and InP substrates, as described earlier. Therefore, the yield pressure of the doped GaAs and InP did not increase with the presence of BN films.

The relation between the load and the width of the resulting scratch may be expressed with a number derived from empirical relations [20]. When the width of the resulting scratch D for BN films is plotted against the load W on logarithmic coordinates, this is expressed as $W = kD^n$ (i.e., Meyer's law, as typically presented in Fig. 5.).

Figures 5(a) to (d) present data for scratch widths obtained for BN films on Si, SiO_2, GaAs, and InP substrates. Comparative data for uncoated Si, SiO_2, GaAs, and InP are also presented. The portion LM for BN films or L'M' for uncoated materials is gradually curved but is considered to be composed of approximately straight portions of transitional slopes. For example, the transitional slopes are 2.6 and 2.4 for the BN film on Si.

The portion MN for BN films, or M'N' for uncoated materials, is a straight line with a slope of 2. It is evident that either MN or M'N' is the range over which Meyer's law is valid for BN films on nonmetallic substrates and for uncoated nonmetallic materials. Here the Meyer's index n is a constant and has a value of 2. Thus, the BN films on nonmetallic substrates behave plastically much like metals when they are brought into contact with hard solids, such as diamond.

10

Adhesion and Shear Strength

Silicon. - With uncoated Si (Table III), the critical load to fracture cohesive bonds in the Si was 5 N in sliding contact. With the BN film deposited on the Si substrate, however, cracking occurred at a load of 7 N (Table II). The BN film on the Si plays an important role in surface fracture. The presence of BN film is very effective in increasing the critical load needed to initiate fracture in Si. A similar trend was observed with good quality BN films on SiO_2, as well as with poorly adhered films on Zn-doped GaAs and undoped InP (Tables II and III).

The failure of the BN film on the Si substrate did not induce fracture of interfacial adhesive bonds between the BN film and the substrate. The critical loads to fracture the interfacial adhesive bonds were 11 N for the BN film deposited on Si at 200 °C and 8 N for that at 350 °C (Table IV).

Benjamin and Weaver [21] derived the following expressions for scratch adhesion in terms of shearing stress S produced at the coating-substrate interface by the plastic deformation, the hardness of the substrate (yield pressure at fully plastic state P_m), the critical load applied on the pin W_c, and the tip radius of the pin R:

$$S = K \left(\frac{W_c P_m}{\pi R^2} \right)^{1/2} \tag{1}$$

$$S \simeq \frac{2W_c}{\pi DR} \tag{2}$$

These relations allow for the calculation of the shear strength (i.e., the adhesion strength of the interfacial bonds) [19,21]. The results are presented in Table IV. The values of the critical loads were obtained and confirmed not only by optical and SEM microscopy of the scratches, but also by the acoustic emission technique.

Fused Silica. - Hertzian cracks were developed in the BN film deposited on SiO_2 as well as in the uncoated SiO_2 at the specific loads indicated in Tables II and III, respectively. The diamond traversing a BN film surface generated intermittent, circular hertzian cracks in the BN film and in the SiO_2 substrate just outside the mutual contact zone between the loaded diamond and BN film. The cracks intersected each other during sliding.

Although very local detachment of the BN film was observed in areas where the cracks intersected, no gross failure of interfacial adhesive bonds between the BN film and the SiO_2 substrate was observed, even at loads to 15 N. Therefore, the actual value of the interfacial adhesive strength between the BN film and SiO_2 substrate is larger than those for the BN film on the Si substrate indicated in Table IV. This relative high adhesion is attributable to the strong chemical bond formed between BN and SiO_2.

CONCLUSIONS

As a result of single-pass scratch experiments conducted with ion-beam-deposited boron nitride (BN) films on silicon (Si), fused silica (SiO_2), gallium arsenide (GaAs), and indium phosphide (InP) substrates in sliding contact with a diamond pin, the following conclusions were drawn:

1. BN films and nonmetallic substrates, like metal films and substrates, deform elastically and plastically in the interfacial region between two solids in contact under a load. However, unlike metallic films and substrates, BN films and nonmetallic substrates can fracture when they are critically loaded.

2. Not only the yield pressure (hardness) of Si and SiO_2 increased by a factor of 2 with BN films, but the critical load needed to fracture these materials also increased. The BN films arrested crack formation.

3. The BN films reduced adhesion and friction in sliding contact. The coefficients of friction for BN films on Si and SiO_2 were less than 0.1 when sliding against diamond.

12

4. BN adhered to Si and SiO_2 and formed good quality films, but it adhered poorly to GaAs and InP. The interfacial adhesive strengths were 1 GPa for BN film on Si and appreciably higher than 1 GPa for BN film on SiO_2.

5. The relation between the load W and the width of the plastically deformed groove D for BN films is expressed by $W = kD^n$ (i.e., the classical Meyer's law).

6. Acoustical measurements of the critical load required to fracture a BN film on a nonmetallic substrate agree well with those detected by optical and SEM microscopy of the scratches.

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TABLE I. - SUBSTRATES

| Material | Doping | | Resistivity, Ω-cm | Sliding | | Primary cleavage plane |
	Dopant	Concen-tration, cm^{-3}		Plane	Direction	
Si	--	------	0.01 to 0.02	{100}	<110>	{111}
SiO$_2$ (quartz)	--	------	------------	-----	-----	-----
p-type GaAs	Zn	3x10^{17}	0.12	{100}	<110>	{110}
n-type GaAs	Te	3x10^{17}	------------	{100}	<110>	{110}
InP	--	------	------------	{100}	<110>	{110}
n-type InP	Sn	3x10^{17}	0.010	{100}	<110>	{110}

TABLE II. - YIELD PRESSURE OF AND CRITICAL NORMAL LOAD
TO FRACTURE BORON NITRIDE FILM COATED ON
NONMETALLIC SUBSTRATE

Substrate and film	Yield pressure at fully plastic state P_m, GPa	Vickers hardness H_v, GPa	Ratio P_m/H_v	Critical normal load to fracture, N
Si coated with BN at 200 °C	24.0	18.8	1.28	7
Si coated with BN at 350 °C	21.9	17.5	1.25	7
SiO$_2$ coated with BN at 200 °C	23.9	----	----	3
SiO$_2$ coated with BN at 350 °C	21.2	17.2	1.23	3
Zn-doped, p-type GaAs coated with BN	7.15	5.72	1.25	3
Te-doped, n-type GaAs coated with BN	7.05	5.65	1.25	3
InP coated with BN	4.51	3.66	1.23	2
Sn-doped, n-type InP coated with BN	4.54	3.61	1.26	1

TABLE III. - YIELD PRESSURE OF AND CRITICAL NORMAL
LOAD TO FRACTURE NONMETALLIC MATERIALS

Material	Yield pressure at fully plastic state P_m, GPa	Vickers hardness H_v, GPa	Ratio P_m/H_v	Critical normal load to fracture, N
Si	12.6	9.89	1.27	5
SiO_2	12.4	----	----	1
Zn-doped, p-type GaAs	6.94	5.61	1.24	2
Te-doped, n-type GaAs	----	5.68	----	-
InP	----	----	----	1
Sn-doped, n-type InP	4.35	3.49	1.25	1

TABLE IV. - CRITICAL NORMAL LOAD TO FRACTURE
INTERFACIAL ADHESIVE BONDS BETWEEN BN FILM
AND SILICON SUBSTRATE AND SHEAR STRENGTH
OF INTERFACIAL ADHESIVE BONDS

Coating condition[a]	Critical normal load, W_c, N	Interfacial adhesive strength, S, GPa	
		S obtained from eq. (1)	S obtained from eq. (2)
200 °C	11	1.1	1.0
350 °C	8	.91	.84

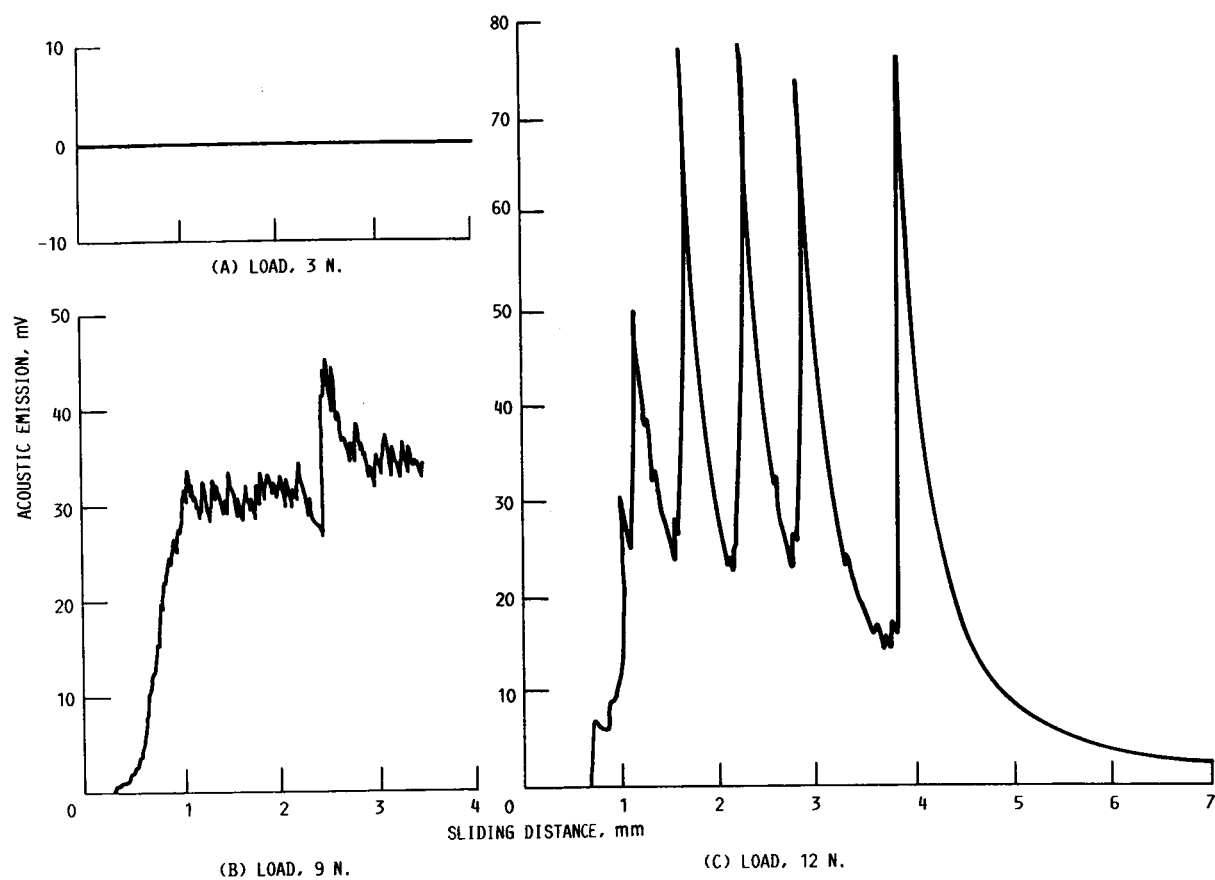[a]Nominal temperature of substrate during ion-beam deposition.

FIGURE 1. - TYPICAL ACOUSTIC EMISSION TRACES FOR A BN FILM ON A NONMETALLIC MATERIAL (SUBSTRATE, Si; SLIDING VELOCITY, 12 mm/min; LABORATORY AIR).
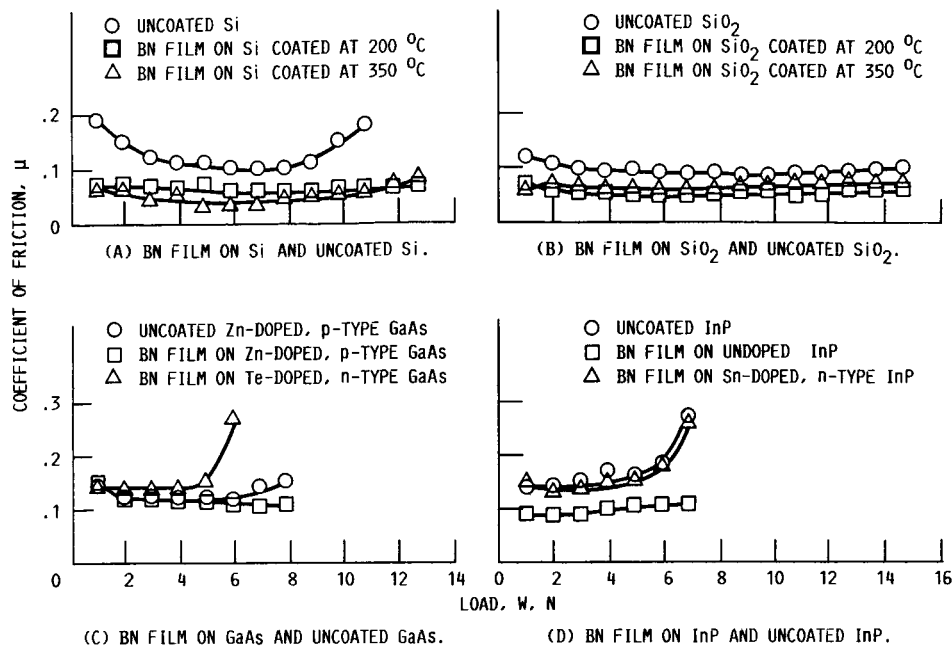
(A) BN FILM ON Si AND UNCOATED Si.

(B) BN FILM ON SiO_2 AND UNCOATED SiO_2.

(C) BN FILM ON GaAs AND UNCOATED GaAs.

(D) BN FILM ON InP AND UNCOATED InP.

FIGURE 2. - COEFFICIENT OF FRICTION AS A FUNCTION OF LOAD. SLIDING VELOCITY, 12 mm/min LABORATORY AIR.

$$A_s = \frac{\pi A^2}{2} = \frac{\pi D^2}{8}$$
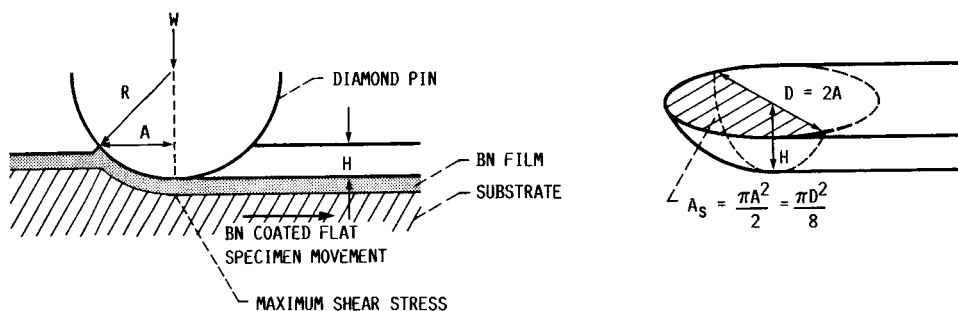
FIGURE 3. - DEFINITION OF MEAN CONTACT PRESSURE ($P = W/A_s$), OR SCRATCH HARDNESS.

O   UNCOATED Si
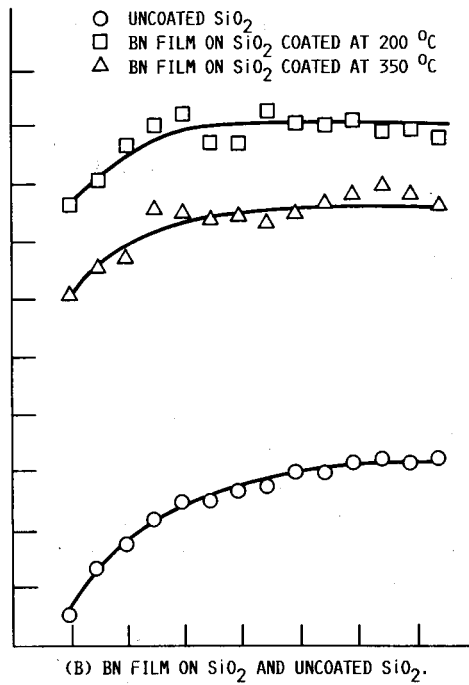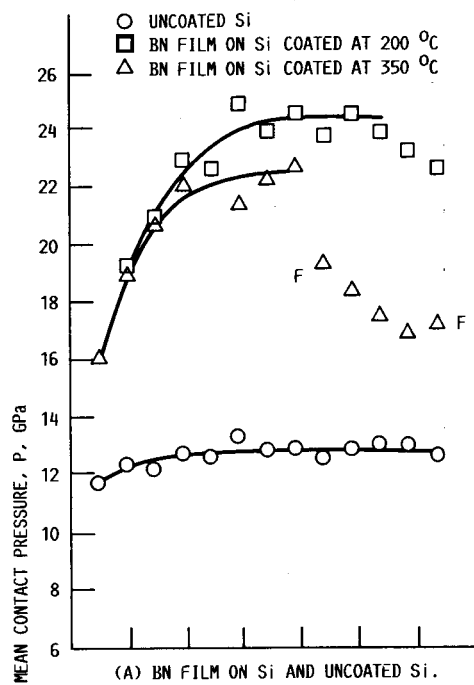□   BN FILM ON Si COATED AT 200 °C
△   BN FILM ON Si COATED AT 350 °C

(A) BN FILM ON Si AND UNCOATED Si.

O   UNCOATED SiO$_2$
□   BN FILM ON SiO$_2$ COATED AT 200 °C
△   BN FILM ON SiO$_2$ COATED AT 350 °C

(B) BN FILM ON SiO$_2$ AND UNCOATED SiO$_2$.

O   UNCOATED Zn-DOPED, p-TYPE GaAs
□   BN FILM ON Zn-DOPED, p-TYPE GaAs
△   BN FILM ON Te-DOPED, n-TYPE GaAs

(C) BN FILM ON GaAs AND UNCOATED GaAs.

O   UNCOATED InP
□   BN FILM ON UNDOPED InP
△   BN FILM ON Sn-DOPED, n-TYPE InP

(D) BN FILM ON InP AND UNCOATED InP.

MEAN CONTACT PRESSURE, P, GPa

LOAD, W, N

FIGURE 4. - MEAN CONTACT PRESSURE (YIELD PRESSURE) AS A FUNCTION OF LOAD. SLIDING VELOCITY, 12 mm/min; LABORATORY AIR.
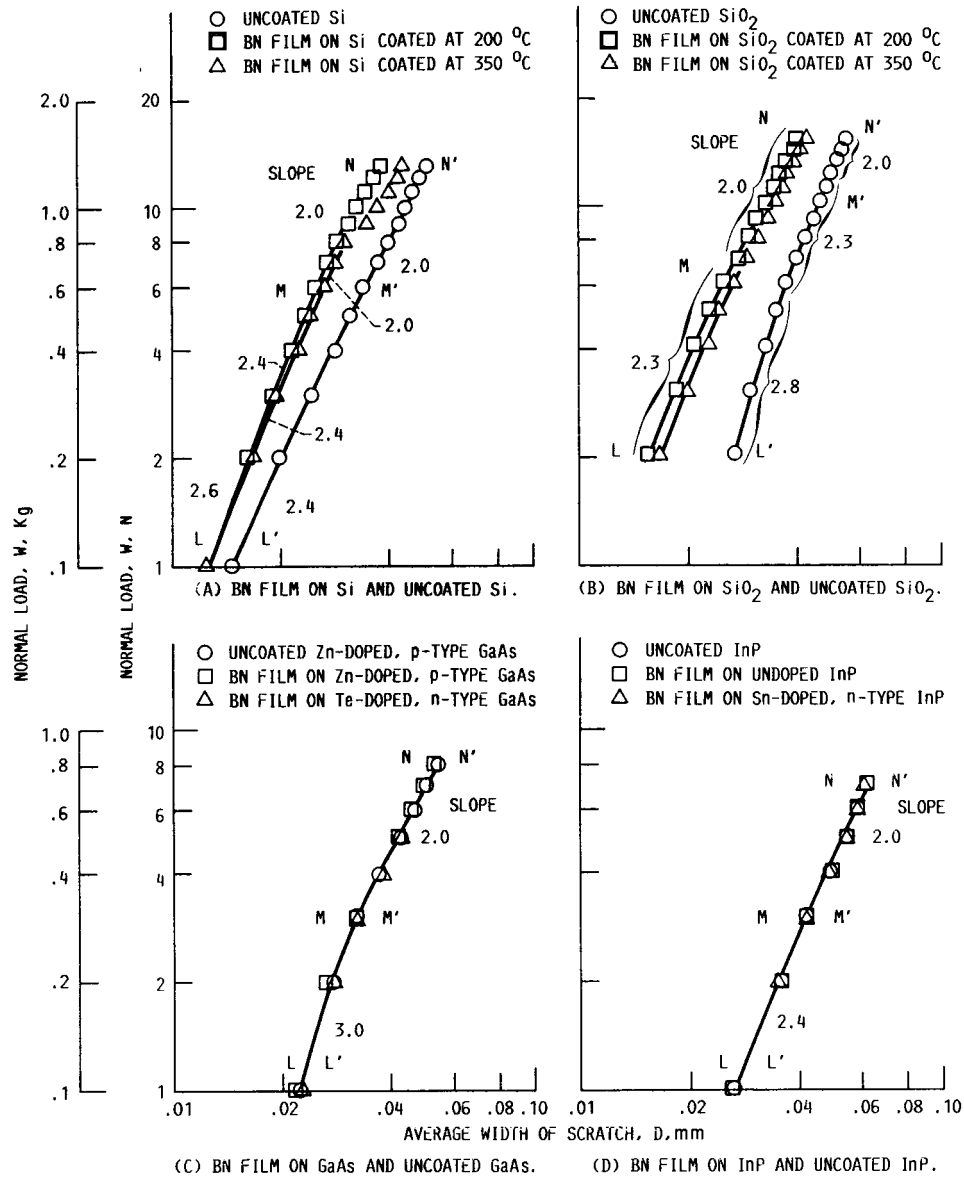
19

FIGURE 5. - SCRATCH WIDTH AS A FUNCTION OF LOAD. SLIDING VELOCITY, 12 mm/min; LABORATORY AIR.

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16. Abstract

An investigation was conducted to examine the mechanical strength and tribological properties of boron nitride (BN) films ion-beam-deposited on silicon (Si), fused silica (SiO_2), gallium arsenide (GaAs), and indium phosphide (InP) substrates in sliding contact with a diamond pin under a load. The results of the investigation indicate that BN films on nonmetallic substrates, like metal films on metallic substrates, deform elastically and plastically in the interfacial region when in contact with a diamond pin. However, unlike metal films and substrates, BN films on nonmetallic substrates can fracture when they are critically loaded. Not only does the yield pressure (hardness) of Si and SiO_2 substrates increase by a factor of 2 in the presence of a BN film, but the critical load needed to fracture increases as well. The presence of films on the brittle substrates can arrest crack formation. The BN film reduces adhesion and friction in the sliding contact. BN adheres to Si and SiO_2 and forms a good quality film, while it adheres poorly to GaAs and InP. The interfacial adhesive strengths were 1 GPa for a BN film on Si and appreciably higher than 1 GPa for a BN film on SiO_2.

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