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TiN COATINGS ON M2 STEEL PRODUCED BY PLASMA ASSISTED CHEMICAL VAPOR DEPOSITION

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M.R. Hilton, G.J. Vandentop,
M. Salmeron, and G.A. Somorjai

September 1987

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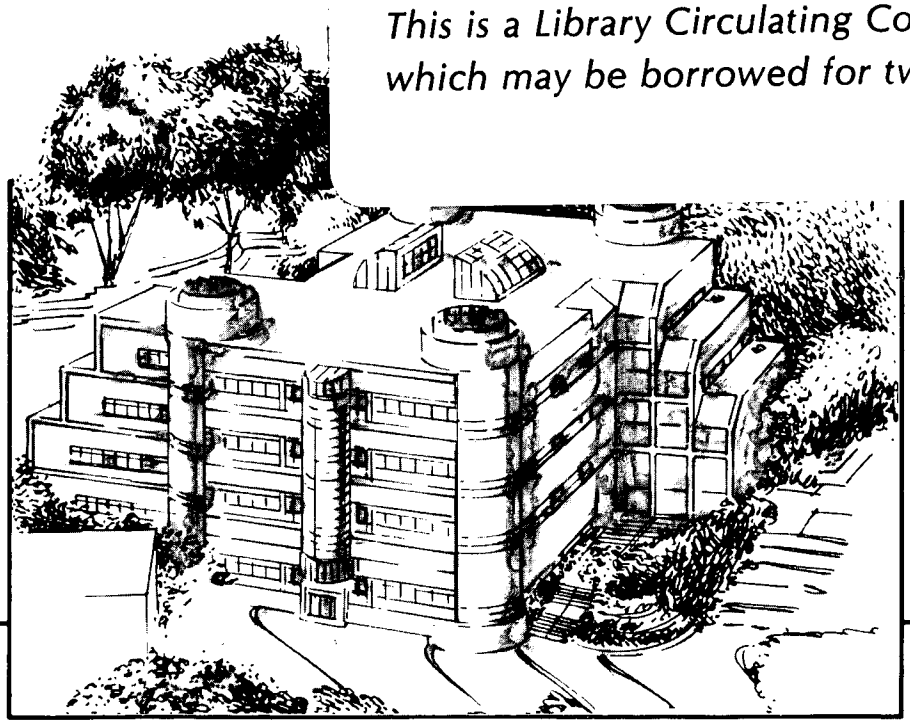

Materials and Chemical Sciences Division

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TiN Coatings on M2 Steel Produced by Plasma Assisted Chemical Vapor Deposition

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September 3, 1987

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Presented at the 14th International
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Abstract

TiN films were deposited onto M2 steel using plasma assisted chemical vapor deposition. $TiCl_4$, N_2 and H_2 were the reactant gases used at 1 Torr. A plasma was essential for TiN formation at 500°C and below. At 500°C, a highly crystalline stoichiometric TiN coating was formed with a (200) surface orientation and having a columnar Zone 2 grain structure. At 400 °C, a Zone T structure with an additional amorphous component was observed . Both types of films had good adherence with scratch adhesion critical loads comparable to values for sputtered TiN. The Zone 2 films failed by cracking and occasional chipping within the coating. The Zone T films failed by cracking in the coating and by chipping, within the coating and by the interface. AES depth profiling of thin films indicates that the interface was sharp and no accumulation of Cl is observed. AES and SEM suggest that distinct TiN islands or nuclei are present in the early stages of growth. The use of an organometallic source, titanium tetrakis (dimethylamide), resulted in the formation of carbonitride powders.

1 INTRODUCTION

The number of studies of the formation of titanium nitride (TiN) by low temperature (<600°C) chemical vapor deposition (CVD) methods has increased within the last few years. Low deposition temperatures have been obtained by utilizing very reactive feed gases ($TiCl_4$ with NH_3[1], or organometallic compounds[2]) at atmospheric pressure or by excited state chemical reactants created in a low pressure plasma.[3–8] A major goal of these efforts is to develop a hybrid process which combines the advantages of CVD (uniform coverage, relatively minor substrate preparation) and that of physical vapor deposition (PVD) processes (low substrate temperature). The applicability of these approaches for tool applications is still under investigation. Adhesion is often a problem, particularly on iron-based substrates. There are additional difficulties for the specific approaches. For example, the use of titanium dialkylamides to form pure, dense TiN has not been adequately reproduced[1,9], whilst films formed by CVD or plasma-assisted CVD (PACVD) using $TiCl_4$ and NH_3 contain large amounts of chlorine which degrade mechanical properties when the deposition temperature falls below 350°C.[1,8] This chlorine retention occurs because the thermodynamically favored chlorine by-product of the $TiCl_4$-NH_3 reaction shifts from gaseous HCl above 350°C, to solid NH_4Cl at lower temperatures. Film adhesion to stainless steel[1] or M2 tool steel[8] was poor, relative to reported

scratch adhesion test (SAT) critical loads (L_C) for sputtered TiN on M2 tool steel.[10] We have previously shown that the interface between TiN, formed with a $TiCl_4$-NH_3 plasma at 400°C, and M2 steel contains a sharp gradient in chlorine, presumed to be detrimental to adhesion.[11]

TiN films of apparently better mechanical quality have been reported for $TiCl_4$-H_2-N_2 plasmas.[4,6,7] However, systematic study of both the physical properties (composition, crystallinity, morphology) and mechanical properties (Vicker microhardness and, particularly, SAT critical load, as a function of thickness) of these films has been incomplete. These properties in turn, are dependent upon deposition conditions. In this paper we report the physical and mechanical properties of TiN films formed by PACVD from $TiCl_4$, N_2 and H_2 on M2 steel, as a function of deposition parameters including temperature, gas composition, power, and bias. The use of titanium tetrakis (dimethylamide) to produce TiN is also discussed.

2 EXPERIMENTAL PROCEDURES

2.1 Plasma Deposition using $TiCl_4$, N_2, and H_2.

The PACVD TiN films produced on M2 steel were prepared with an r.f. apparatus with a planar electrode configuration described previously[8], except that a more powerful r.f. amplifier (frequency 13.6 MHz) was used. The substrate hardness was 62-63 Rockwell C. In this study, the substrate temperature, H_2/N_2 gas ratio, r.f. power, and substrate bias were varied. The $TiCl_4$ flow rate was held constant at 0.21 mmol min^{-1} during the experiment. Total pressure was 1 Torr. Substrate temperature ranged from 350°C to 500°C, H_2/N_2 ratio varied from 0.7 to 4.5, power ranged from zero to 200 W, and applied bias from -250 V to 0 V (both grounded and floating conditions were tried). Two classes of films were prepared: 1) thick coatings (2-10μm), used to correlate bulk physical and mechanical properties, 2) thin films (deposition times: 5, 15, 30, 60, 120, 300, and 600 seconds), used to elucidate interface composition and film nucleation. In almost all cases, the M2 substrates, previously cleaned in acetone and ethanol ultrasonic baths, were placed on the lower electrode and

heated to the desired temperature. N_2 gas was allowed to flow for five minutes, followed by H_2 (bringing P_{TOT} to 1 torr) for five more minutes. An r.f. plasma was then ignited and $TiCl_4$ was added after five minutes. In some cases, the plasma pretreatment was extended to 15 minutes. Alternatively, the entire pretreatment was shortened to less than two minutes. It was not possible to start the gas flow (in a stable condition) and ignite the plasma instantly to avoid this pretreatment.

2.2 Characterization of PACVD films

2.2.1 Physical Properties

The morphologies of the M2 substrates and TiN coating were investigated by optical microscopy and scanning electron microscopy (SEM). The coating cross-section was observed by examining the region around a Rockwell C hardness indentation on a tilted stage in an SEM. Fracture at the indentation edge would generally reveal film cross sectional morphology and thickness. Film crystallinity was investigated with X-ray Diffraction (XRD). Chemical composition was determined by Auger electron spectroscopy (AES) using a system described previously.[8] Argon ion sputter depth profiling was used to remove surface contaminants on thick films and to reveal the interface of thin films. The sputtering rate was 3 $Åmin^{-1}$.[11] In addition, the scratch removal technique (SRT)[11] was attempted on thick films to expose the interface for AES analysis.

2.2.2 Mechanical Properties

The coating-substrate interfacial strength was evaluated by SAT using a device built in our laboratory, described previously.[8] The diamond tip radius was 0.2 mm, like the commercial testers built by Centre Suisse D'Electronic et de Microtechnique (CSEM).[12–13] The Rockwell C hardness test was used to determine if the M2 hardness had changed during deposition because of sample heating. Vickers microhardness testing was performed on coated and uncoated

regions of the samples. Indentation loads of 100 and 200 grams were used, with ten measurments at each condition.

2.3 Organometallic CVD

Experiments were performed to determine the suitability of titanium tetrakis (dimethylamide) [hereafter Ti amide] as a precursor for TiN formation by PACVD. Early studies utilized the plasma apparatus (with no plasma) as a deposition chamber. Later studies utilized a glass chemical vapor transport (CVT) apparatus patterned after the design used by Sugiyama et. al. who reported success in making golden TiN films at temperatures as low as 200°C.[2] A stainless steel version, evacuated by a turbomolecular pump, was later used. A variety of conditions were explored: deposition temperature, 200 to 800°C; Ti amide bath temperature, 40°C to 80°C; carrier gases, Ar, He, and N_2; chamber pressure, 0.1 to 760 torr.

3 RESULTS

3.1 Thick Films - Bulk Properties

3.1.1 Film structure and composition

The properties of TiN coatings deposited on M2 steel were extensively investigated at substrate temperatures of 500°C and 400°C without an applied bias. SEM indicated that the films grown at 500°C grew in a columnar Zone 2 structure (well-defined columnar morphology with uniform grain diameters and flat tops, identical to the structure shown in Ref. 8) while the films deposited at 400°C grew in a fibrous Zone T structure (large 'grains' with partially curved tops having a poorly defined, fibrous internal morphology). In contrast to the poorly adherent NH_3-based TiN coatings reported previously[11], film delamination did not occur around the Rockwell indentation in the Zone 2 films, though some film delamination occurred with the Zone T films. The average grain diameter of the Zone 2 structure was 2500 Å. The diameter of the Zone T

structures was 2 to 3 μm while the internal fiber diameters were 600 to 3000 Å. In addition, XRD showed that the films had strong (200) preferred orientation. For Zone 2 films only the (200) and (400) peaks were observed, these peaks were sharp and in contrast to the broader (200) peaks and small amorphous background peak observed in the Zone T films.

AES revealed that the $(Ti+N)_{387eV}$:Ti_{418eV} ratio of the films following sputtering ranged between 2.1 and 2.3 under all unbiased deposition conditions at 400°C and 500°C. Comparison of the experimental ratio values with those found in the literature suggests that these films are stoichiometric or nearly stoichiometric. The Zone 2 films contained less than 2% Cl, while the Zone T films generally had 3 to 5% Cl.

3.1.2 Mechanical Properties

Figure 1 shows the SAT critical load (L_C) values as a function of thickness for TiN films prepared at 400°C and 500°C with no applied substrate bias. Several different deposition conditions are plotted together. Also plotted, for comparison, is a linear summary of values obtained by Sproul[10] for sputtered TiN on M2 steel and the L_C values found from an earlier study of PACVD-produced TiN formed from NH_3 and $TiCl_4$.[8] The data indicates that PACVD-produced TiN from $TiCl_4$, N_2 and H_2 can be made as adherent as sputtered TiN. Critical load values were repeatable within 200 gm for a given sample. There was a significant amount of scatter as a function of thickness, which could not be correlated with deposition conditions or by changing the duration of plasma pretreatment. Failure in the Zone 2 films took the form of cracks emanating adjacent to the stylus track or by occasional chipping within the coating (Fig. 2a, 2b). The Zone T films failed by similar cracking or by periodic chipping, both within the coating and at the interface (Fig. 2c, 2d). The orientation of the fracture plane with respect to the direction of stylus motion was observed to differ between the two morphologies. Vector lines constructed by the intersection of the plane of the fracture and the coating surface (pointing away from the track) were inclined towards the direction of stylus motion (the angle between the intersecting vectors was less than 90°)

in the Zone 2 films and were inclined away (angle between intersecting vectors was greater than 90°; see Figure 2) from the motion direction in the Zone T films. These failure modes were unsuitable for interface composition analysis by SRT[11] (The poor resolution of our SAM system prevented the study of the chip failure mode). These failure modes indicate better substrate-film adhesion than the continuous delamination observed in NH_3-based films.[8]

Rockwell C hardness tests indicated that the heating of the substrate during deposition did not degrade the M2 microstructure. The highest intended operating temperature was 500°C. Since the minimum temperature for softening of M2 steel is around 560°C to 590°C[14], the results indicate that the r.f. plasma did not impart substantial thermal energy to the substrate. Vickers microhardness (Hv) increased with film thickness. For a 10μm film, the Hv_{200} was 1520 kg/mm^2. This is lower than the bulk value (2000 kg/mm^2) and lower than that obtained for this thickness (2300 kg/mm^2) by Sproul. However, the grain size of the Zone 2 films in this study (2500 Å) is larger than those of Sproul (500 Å) which may account for the differences in hardness reported here.

3.1.3 Effect of Deposition Parameters

As in a previous study, film deposition temperature influenced chlorine incorporation into the films, with chlorine content increasing as temperature decreased. At 400°C and above, a constant $(Ti+N)_{387eV}$:Ti_{418eV} ratio was observed. Plasma conditions mainly influenced growth rate. In general, the growth rate using H_2 and N_2 was 10 to 25% of that using NH_3 under equivalent conditions. However, unlike NH_3, a plasma was essential for TiN formation at 500°C and below. Without hydrogen, a titanium-chlorine compound was obtained. Without nitrogen, a thin titanium-oxygen-chlorine compound was obtained. Power would generally show an optimal behavior for growth (growth rate would increase with power up to some value and then decline), as would H_2/N_2 ratio. A negative bias inhibited growth: grounded, -100V, and -250V had growth rates of 4 Åsec^{-1}, 0.3 Åsec^{-1}, and 0.1 Åsec^{-1} respectively. The growth rates of a floating or grounded substrate did not differ, except at 200W,

where the floating condition had a growth rate twice that of the grounded. The effect of substrate bias on the physical and mechanical properities of the films is the subject of current research.

3.2 Thin Films–Interfacial Properties and Nucleation

The films prepared in this study had an average growth rate (based on measurements of thick films prepared with the same deposition conditions) of 3-4 Åsec^{-1}. All samples had surface contamination from transport in air in the form of carbon and excess oxygen which could be removed by 60 to 90 sec of sputtering. Figure 3 shows two representative spectra of samples with deposition times of (a) 30 seconds and (b) 300 seconds. Both spectra indicate that the TiN-M2 interface is fairly sharp (<60 Åwide); chlorine accumulation was not seen. The constant presence of iron in the initial stages of sputtering of the 30 second sample suggests that TiN islands or nuclei on M2 steel are present in the early stages of growth. Investigation of the films with SEM, prior to sputtering, supports this idea. Distinct island-like structures are observed at short deposition times (Figure 4a, 30 sec), while these nuclei have coalesced at longer deposition times (Figure 4b, 300 sec). These nuclei have a center to center distance on the order of the grain size of the Zone 2 films.

3.3 Organometallic Studies

The material produced from titanium dimethylamide decomposition was one of two forms: dense oxides or carbonitride powders. The oxides were produced in studies with the plasma deposition chamber under pressure conditions at which $TiCl_4$ and NH_3 produced TiN. Oxides were also formed in the CVT chambers if either air leaks or the residual water were present. If the CVT chambers were properly baked, an agglomerated powder (or porous nodular coating) was formed indicating that gas phase nucleation had occurred. These results highlight the general difficulty with titanium organometallics: 1) They

are extremely sensitive to water and oxygen; 2) The compound vapor pressures are low which necessitates heating of the feed line; 3) The compounds are very reactive and can easily decompose during transport.

4 DISCUSSION

The experimental evidence indicates that surface temperature is an important parameter in determining the structural and thus the mechanical properties of TiN films. The change in film morphology (from a partly amorphous Zone T to a highly crystalline Zone 2 structure) occurs because increasing substrate temperature improves surface adatom mobility, as reported in the literature for other vapor deposition technologies.[15] As in a previous study with NH_3-based plasmas[8], surface temperature influences the expulsion of chlorine from the growing films. However, unlike the NH_3 case where the effect of the plasma was primarily to increase growth rates, TiN formation from $TiCl_4$, N_2, and H_2 requires a plasma at 500°C and below. The plasma creates excited species which allows film formation to occur. The slower growth rates of the N_2-H_2 plasma may be due to the relative difficulty in breaking the dinitrogen bond in N_2 versus the N-H bond in NH_3. The reduction in growth rate with negative bias is almost certainly a resputtering effect which has been seen in other physical vapor deposition (PVD) technologies.[16] Power and gas composition affect the growth rates of PACVD films probably by changing the population of excited species in the plasma.

The SAT critical load values of N_2-H_2-based PACVD TiN are comparable to values reported for sputtered TiN on M2 steel.[10] The SAT behavior of N_2-H_2-based TiN is much better than earlier NH_3-based coatings. One important factor which is crucial to the adhesion of titanium nitride coatings is substrate cleaning and Helmersson et. al.[17] have shown that sputter cleaning of substrates (using a negative bias of a kilovolt or more on the sample) promotes good adhesion of sputtered TiN films on high speed steels. In the present work, the samples were grounded and the sheath potential was of the order of tens of volts which means that these bombardment cleaning effects were minimized.

However it is believed that the excited state hydrogen from the plasma facilitates sample cleaning. Activated hydrogen may also alleviate the problem of chlorine accumilation at the TiN-M2 interface, which also occurs in NH_3-based plasmas[11] and which is extremely detrimental to coating adhesion. The scatter in the SAT critical load data did not systematically correlate with deposition temperature, r.f. power, N_2/H_2 gas ratio, plasma pretreatment exposure time, bulk film chlorine content (when less than 5%), or morphology (though fracture mode did change with morphology). This scatter may correlate with residual interface contamination. Further study is necessary to clarify this important point. The change in SAT fracture mode with morphology may actually stem from the difference in bulk chlorine contents and not the morphology itself. The fact that the Zone 2 films had better resistance to delamination during Rockwell (or Hertzian) impact than the Zone T films suggests that the former morphology would be better for tool applications. The value of microhardness are quite low even for the large grain sizes established for the films. The residual bulk chlorine content may be the cause. Also we do not know the composition of the grain boundaries of the TiN films. Chlorine or other impurity content may be higher there.

The AES and SEM studies of thin films shows that heterogeneous nucleation on the surface is occuring. Further investigation is required on the exact nature of the nucleation site. The effect of sample preparation (especially doping) on nucleation density and film adhesion also requires further study. While good critical loads have been obtained, further research of coating adhesion is justified because the correlation between SAT behavior and actual field performance is not universal, for example in forming tool applications.

5 CONCLUSIONS

1. The deposition temperature is a key parameter in determining the nature of PACVD TiN films formed from $TiCl_4$, N_2, and H_2. At 500°C, Zone 2 columnar grains are formed with a (200) orientation. At 400°C, Zone T fibrous grains with a (200) orientation and an amorphous component are formed. Chlorine

content increases as deposition temperature decreases.

2. At these temperatures, a plasma is essential for TiN formation from $TiCl_4$, N_2, and H_2. Growth rates are 10 to 25 % of those for $TiCl_4$-NH_3-based PACVD TiN at similiar operating conditions. Power and H_2/N_2 ratio primarily affect growth rate. Negative substrate bias inhibits growth. The influence of bias on mechanical properties is under investigation.

3. SAT critical loads of $TiCl_4$-N_2-H_2-based PACVD TiN on M2 steel are comparable to values reported for sputtered TiN. Zone 2 films failed primarily by cracking in the film with occasional film chipping. Zone T films failed by cracking and by more extensive chipping, both within the film and at the interface. The Vickers microhardness (Hv_{200}) of the deposited TiN ($10\mu m$ thick) is 75% of the value of bulk TiN.

4. AES sputter profiles of thin films indicate that the interfaces are sharp (<60Å). There is no sign of chlorine accumulation. The AES data indicates that early deposition proceeds by nucleation and subsequent growth of TiN islands. SEM investigation indicates island-like features which fuse at increased deposition time. The density of these features is of the order of the TiN grain size.

5. Titanium dimethylamide decomposition produced carbonitride powders. These formed because of the fact that the heating necessary for vapor transport induced gas phase nucleation.

Note Added in Proof:

The authors have recently become aware of P.J. Burnett and D.S. Rickerby's article now in press in *Wear*, entitled 'The relationship between hardness and scratch adhesion'. We wish to direct attention to their SAT failure mode classification system (their Figure 8). We believe that the cracking pattern observed in our Zone 2 films suggests a tensile failure (Fig. 8e) while our Zone T films exhibit a spallation/buckling failure (Fig. 8b,c).

ACKNOWLEDGEMENTS

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List of Figures

1. Scratch Adhesion Test Critical Loads (L_C) of TiN films on M2 steel as a function of film thickness produced by PACVD from $TiCl_4$, N_2, and H_2. A linear representation of data for sputtered TiN[10] and the value for NH_3-based PACVD TiN are also plotted for comparison. The data shows that PACVD-produced TiN can be as adherent as sputtered TiN.

2. SEM Micrographs of SAT Failure modes: a) Zone 2 cracking, b) Zone 2 chipping within the coating, c) Zone T cracking, d) Zone T chipping within the coating and at the interface.

3. Typical AES sputter depth profiles for TiN films formed with deposition times of: a) 30 seconds and b) 300 seconds. The sputter rate was 3 $Åmin^{-1}$. The initial and constant presence of iron in the early stages of sputtering of the 30 second sample suggests that film deposition starts by the nucleation of TiN islands on the steel surface.

4. SEM Micrographs of the surface of TiN films on M2 steel formed with deposition times of: a) 30 seconds and b) 300 seconds. Distinct island-like features are observed at the early stages of growth which fuse as deposition time progresses.

Critical Load vs. Thickness

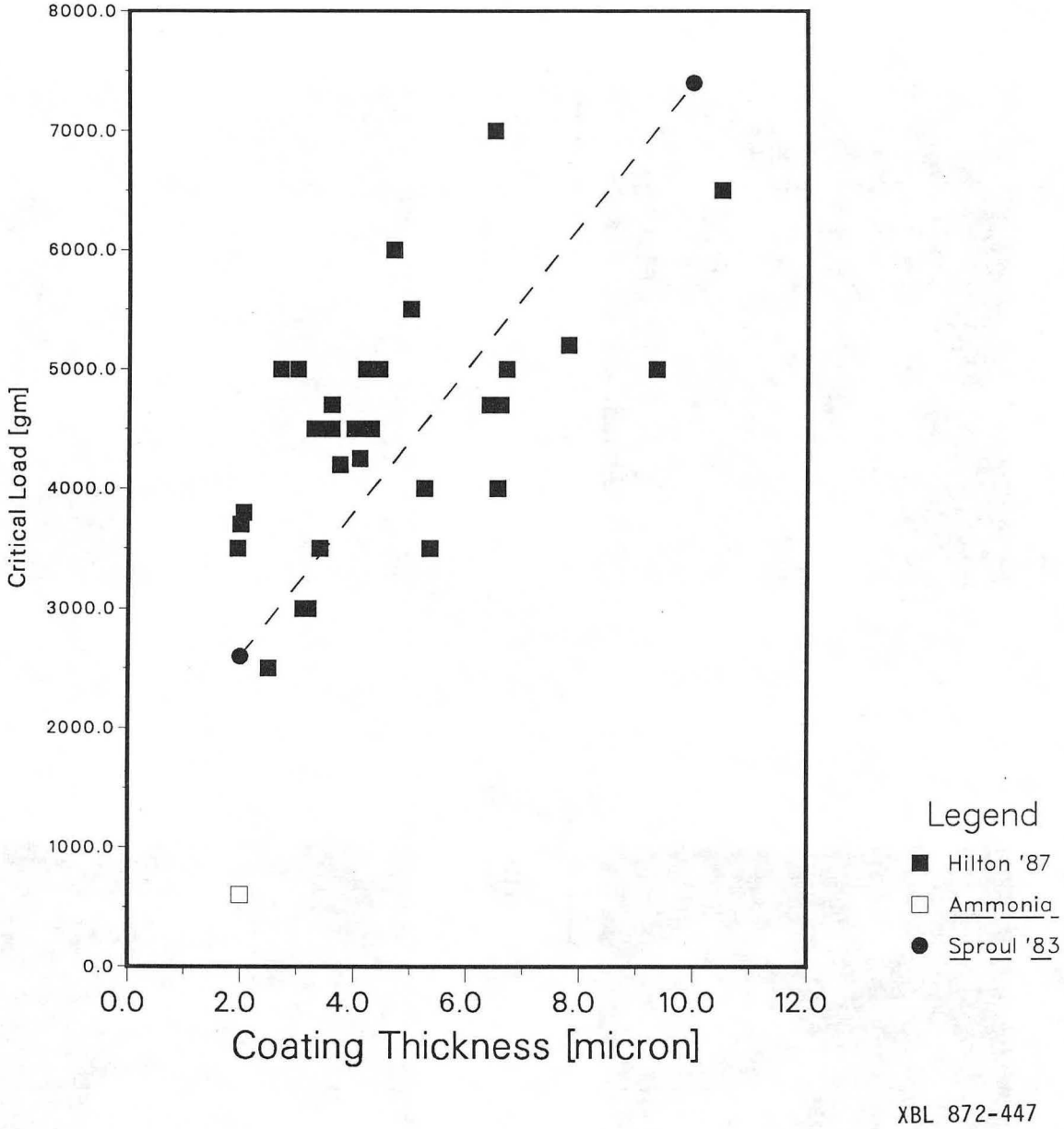
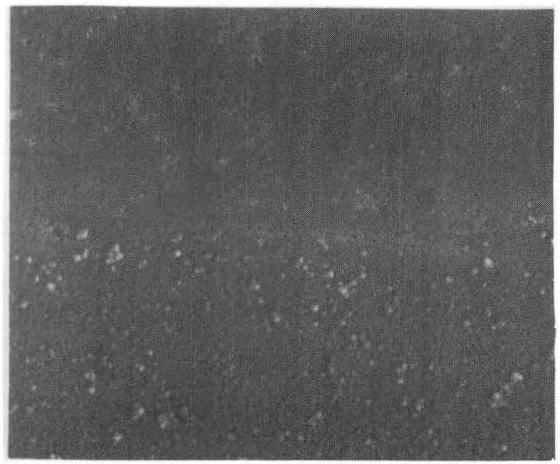


Fig. 1

Stylus Motion

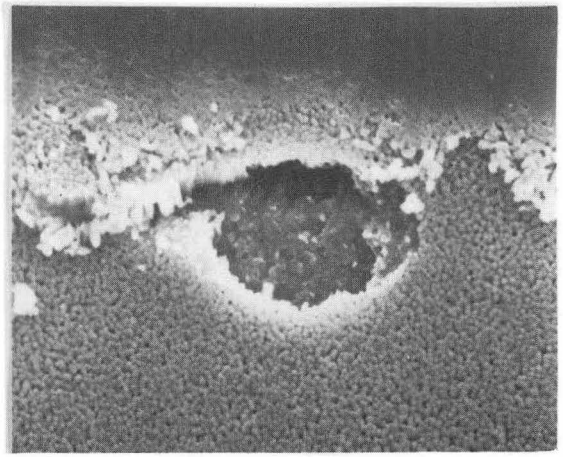
a.



10 μm.

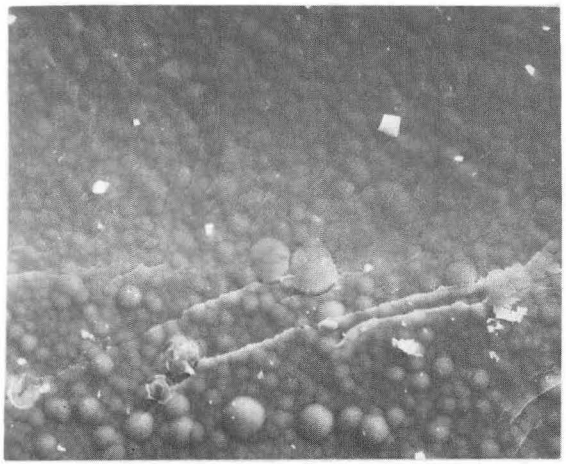
500 ^{o}C

b.



5 μm.

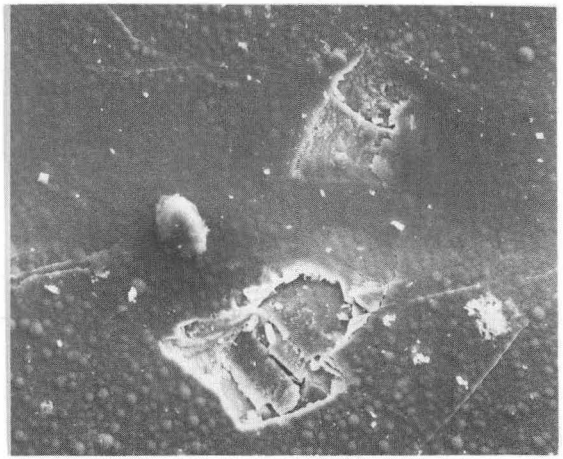
c.



20 μm.

400 ^{o}C

d.



40 μm.

Fig. 2

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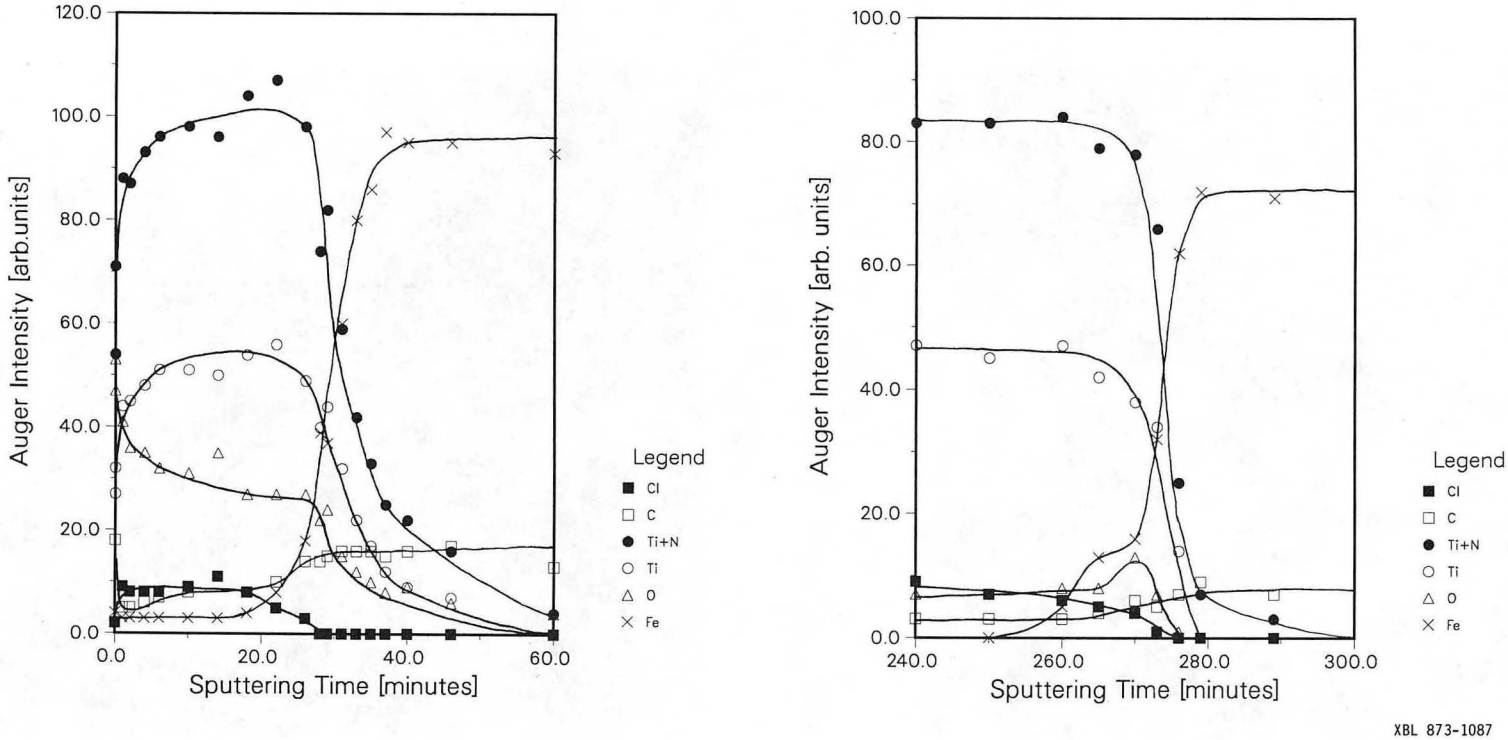
Auger Intensity [arb.units]
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60.0
40.0
20.0
0.0
0.0
20.0
40.0
60.0
Sputtering Time [minutes]
Legend
Cl
C
Ti+N
Ti
O
Fe
Auger Intensity [arb. units]
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80.0
60.0
40.0
20.0
0.0
240.0
260.0
280.0
300.0
Sputtering Time [minutes]
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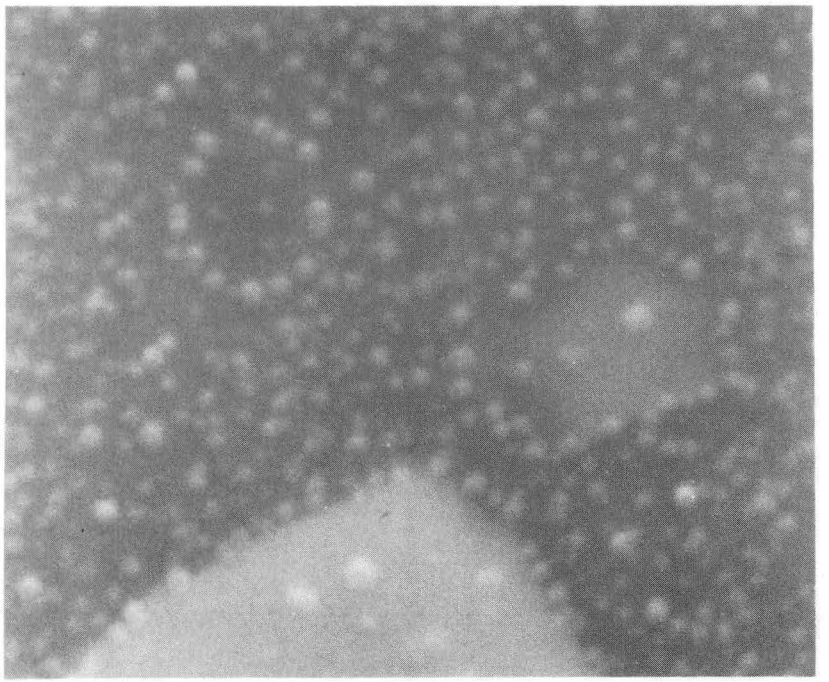
C
Ti+N
Ti
O
Fe
XBL 873-1087

Fig. 3

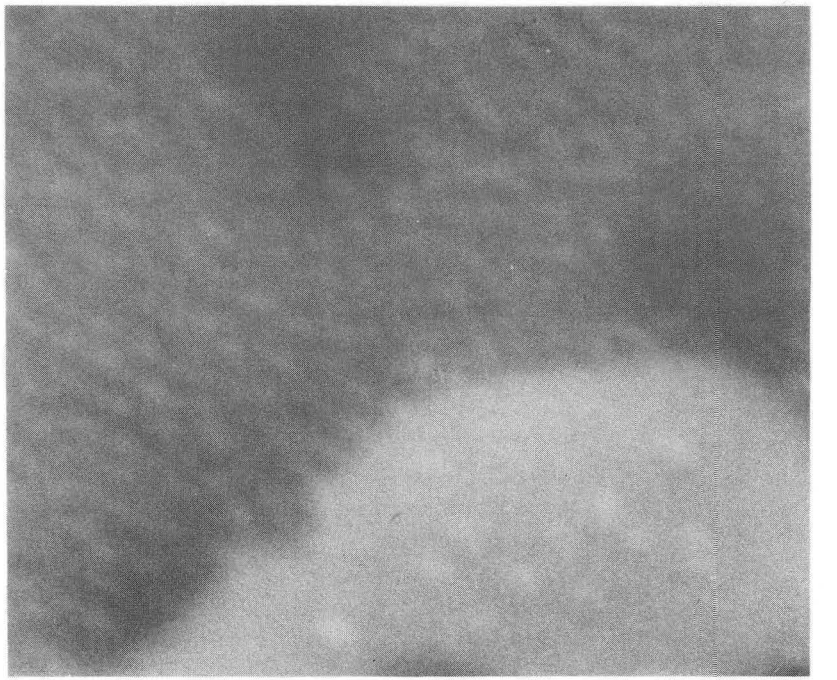
Deposition Time:

30 Seconds



50 μm.

300 Seconds



50 μm.

Fig. 4

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