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ADVANCES IN SPUTTERED AND ION PLATED SOLID FILM LUBRICATION

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SUMMARY

The glow discharge or ion assisted vacuum deposition techniques, primarily sputtering and ion plating, have rapidly emerged and offer great potential to deposit solid lubricants. The increased energizing of these deposition processes lead to improved adherence and coherence, favorable morphological growth, higher density, and reduced residual stresses in the film. These techniques are of invaluable importance where high precision machines tribo-components require very thin, uniform lubricating films (0.2 m), which do not interface with component tolerances. The performance of sputtered MoS_2 films and ion plated Au and Pb films are described in terms of film thickness, coefficient of friction, and wear lives.

INTRODUCTION

To prevent tribological failures of contacting tribo-element surfaces in sliding, rotating, rolling or oscillating motion, friction and wear has to be minimized by application and interposition of lubricating materials. Oils and greases have been found to be inadequate wear preventives under severe environmental conditions. To meet severe operational conditions such as high vacuum, high temperature, nuclear or spacial radiation, extreme loads, and chemically reactive or corrosive environments, it is of great importance to select the proper solid lubricant and a suitable deposition technique. The selected solid lubricant and its application technique will determine the tribological control in terms of friction, wear, and endurance life and is expected to perform over a broad spectrum of these harsh environments. Primarily, the deposition technique selected determines the mode and life of wear or the type of corrosion. In recent years, the glow discharge or ion assisted vacuum deposition techniques, primarily sputtering and ion plating have rapidly emerged and offer a great potential to deposit solid lubricants such as the lamellar solids (MoS_2, WS_2, $NbSe_2$, etc.); soft metals (Au, Ag, Pb); polymers (PTFE and polyimides); and wear resistant refractor compounds (nitrides, carbides, borides, oxides and silicides).

The sputtering and ion plating techniques are restricted to processes where particle condensation on the substrate is initiated by the ions which transfer energy, momentum, and charge to the substrate and the growing film, and which can be beneficially controlled to affect the nucleation and growth sequence. Consequently, the increased energizing of the deposition process leads to improved adherence and coherence, favorable morphological growth, higher density, and reduced residual stresses in the films. The coating thickness required is generally small, typically 0.2 to 0.6 m thick, and the

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highly polished and finished components are coated as the final processing step. All these foregoing coating properties beneficially affect the tribological, mechanical, and corrosive behavior of the contacting surfaces and are described and illustrated in this paper.

SPUTTERING TECHNOLOGY

When energetic ions strike a surface, a number of events can occur, as shown in Fig. 1. First, the incident ion can enter the surface and become trapped. This interaction is referred to as ion implantation. Second, the ion may be reflected from the surface, after being neutralized in the process. Third, as a result of momentum transfer, an atom of the surface can be ejected. This is the mechanism referred to as sputtering.

The sputtering technology offers a great versatility and flexibility in coating deposition, since the sputtered coatings can be tailored in any preferred chemical composition, and the coating morphologies can be modified. Numerous sputtering modes 9direct current-dc, radio frequency-rf, magnetron, reactive, etc.) have emerged and have been widely described in the literature (Refs. 1 to 3). A typical rf diode sputtering apparatus with dc bias capability is shown schematically and photographically in Fig. 2. This sputtering system has been extensively used for sputter-deposition of solid lubricants such as MoS_2, WS_2, PTFE, etc. In rf sputtering, the target (material to be deposited) is energized by the application of rf (13.56 MH_z) power. Before rf sputter deposition, the substrate surface is sputter cleaned and textured by ion bombardment, thus providing excellent adherence.

Performance of Sputtered MoS_2 Films

To achieve effective MoS_2 lubrication, two requirements have to be met: (1) strong film/substrate adherence, and (2) a low crystalline slip during shearing. Sputtered MoS_2 films generally display a strong adherence to most metallic surfaces, with the presently known exceptions being copper and silver and their alloys (Ref. 4). These two metals are highly reactive with the activated free sulphur during the sputtering process, thus forming sulphur compounds which have the tendency to flake off the surface.

Prior to sputter depositing MoS_2, the highly polished surface is cleaned by ion bombardment, thus providing an excellent adherence. Due to the strong adherence, extremely thin films (about 0.2 m) are more effective than thicker films applied by other techniques, as shown in Fig. 3. In thin solid film lubrication, the film thickness has a very pronounced effect on the coefficient of friction. Essentially, the coefficient of friction reaches a minimum value at an effective or critical film thickness, as shown in Fig. 4. The effective film thickness for sputtered MoS_2 films has been experimentally observed to be in the 0.2 m range (Ref. 5). Typical Scanning Electron Micrographs (SEM) in Fig. 5 show that a 1 m thick sputtered MoS_2 film after a single pass sliding has a tendency to break within the columnar region of the film, thus leaving a residual or effective film on the substrate. This effective lubricating film performs the actual lubrication and displays a low coefficient of friction (0.04) under vacuum conditions. The morphological growth zones for MoS_2 films are schematically illustrated after sliding in respect to film fracture in the columnar zone in Fig. 6. This film fracture behavior clearly indicates that the adhesive forces between the substrate and the MoS_2 film are stronger than the cohesive forces in the film itself. It should be remarked that the displaced excess film generates unnecessary wear debris, which has the tendency to increase the torque level and impair the lubrication cycle in precision bearings.

To strengthen the structural integrity of the MoS_2 film in the columnar zone and possibly increase the thickness of the remaining or effective film, gold dispersion was introduced into the sputtered MoS_2 film for densification and strengthening purposes. A compact Au-MoS_2 sputtering target prepared by powder metallurgy techniques with 5 to 8 wt % of gold was sputtered to achieve the gold distribution within the MoS_2 films. Similar co-deposited Ni-MoS_2 films have been also reported (Ref. 6). These co-sputtered Metallic-MoS_2 films displayed a lower and more stable coefficient of friction and even longer endurance lives than the pure MoS_2 films (Ref. 7).

To insure consistency in performance, precise sputtering quality control is necessary. Film morphology and chemical composition are sensitive to sputtering parameters. The integrity of the sputtered MoS_2 films can be identified by visual appearance prior to and after gentle rubbing or wiping across the surface. An effective lubricating film has a matte-black, sooty surface appearance, whereas an unacceptable film has a highly reflective mirror-type surface. When the matte-black, sooty surface is slightly wiped unidirectionally, the color changes from black to gray and is schematically represented in Fig. 7. The color change reflected from black to gray is due to the reorientation of the crystallites or platelets in the film (Ref. 8).

Sputtered MoS_2 films are particularly indispensable for applications where extremely thin films (0.2 m) are required for tribological control in high precision bearings, where tolerances are close, reliability requirements are high and the minimization of wear debris formation is critical. MoS_2 films are directly sputtered onto bearing components (races, cage, and balls) as shown in Fig. 8. These films have been successfully used in despin bearings for communications satellites, gimbal bearings of reactive motors, bearings for antenna and solar array pointing mechanisms, gears and bearings of spacecraft harmonic drive assemblies, and in numerous other aerospace and earth satellite applications. For instance, in Fig. 9 the adjusting screw which fits into a power nut, is sputter coated with a 0.3 m thick MoS_2 film (Ref. 9). Note, that the left hand bearing diameter is also sputtered to prevent fretting corrosion against the inner face of a ball bearing fit. This assembly is used in the Intelsat V Satellite to position the antennas. The operational requirements are from one to two cycles per day for 16 years.

ION PLATING TECHNOLOGY

The ion plating technique combines the high throwing power of electroplating, the high deposition rates of thermal evaporation, and the high energy impingement of ions and energetic atoms of sputtering and ion implantation processes. The basic difference between sputtering and ion plating is that the sputtered material is generated by impact evaporation and transfer occur by a momentum transfer process. In ion plating the evaporant is generated by thermal evaporation. The ion plating process is more energetic than sputtering, since the process uses a high specimen bias of several thousand volts to directly accelerate the positively ionized evaporant atoms into the substrate. The basic ion plating system consists of a dc-diode configuration, where the specimen is made the cathode of the high voltage dc circuit with an evaporation source as anode shown in Fig. 10. The ion planting technique and the process parameters are described in the literature (Refs. 10 to 13).

The interest in ion plating originates from its two unique features:

(1) The high energy flux of ions and energetic neutrals contribute to the excellent film/substrate adherence and the desirable morphological growth of the film.

(2) The high throwing power provides for three-dimensional coverage to coat complex, intricate components such as bearings cages and races without rotation.

These two features have generated new potentials in coating utilization. The soft metallic ion plated films such as Au, Ag, and Pb are primarily used for lubricating spaceborn tribo-element components and ion plated Al films for corrosion protection of aircraft engine parts and structural components.

Performance of Ion Plated Metallic Films

The excellent film adherence of ion plated films is attributed to the formation of a graded interface between the film and the substrate, even where the film/substrate materials are mutually incompatible. This graded interface not only provides excellent adherence, but also induces surface and subsurface strengthening effects which favorably affect the mechanical properties such as yield strength, tensile strength, and fatigue life, as shown in Figs. 11 and 12. Of the soft metallic films (Ag, Au, and Pb), gold and lead have found extensive uses in spaceborn bearings of satellite mechanisms such as solar array drives, despin assemblies, and gimbals. These ion plated lubricating thin films 0.2 m thick are very effective in increasing the endurance life, reducing the coefficient of friction, and eliminating instant or catastrophic failures. The favorable coating behavior is due to the superior adherence, graded interface, very small, uniform crystallite size which contributes to high density packing (pore free) and very continuous and uniform film with a high degree of lattice fit. Due to these favorable coating formation characteristics, extremely thin films in the 0.2 m range are far superior to thicker films in terms of the coefficient of friction, as shown in Fig. 13, for gold and lead (Ref. 14). For instance it has been shown (Ref. 14) that the Pb film's superior adhesion produced significantly lower torque noise and the torque behavior was stable over the first million revolutions.

The high throwing power and the excellent adherence is widely used to ion plate three-dimensional complex mechanical surfaces, such as internal and external surfaces of tubing, fasteners, ball bearings, and gear teeth, as

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shown in Fig. [illegible] hardware components ion plated [illegible] films. A highly loaded precision ball [illegible] plated with gold (0.12 to 0.2 m) as shown [illegible] is used in space where it transmits 136 joules of [illegible] film prevents cold welding of the gear pitch line.

In addition to [illegible] films for lubrication, in the aircraft [illegible] on a large-scale production basis to protect aircraft [illegible] from corrosion. Also, where required, it will be used on all [illegible] critical high strength aluminum and steel structures and on titanium and alloy steel fasteners (Ref. 16). A typical Al ion plated [illegible] gear cylinder for corrosion protection is shown [illegible] aluminum alloy bulkhead for corrosion and fatigue [illegible] Fig. 17.

[illegible]

The plasma or ion [illegible] techniques, such as sputtering and ion plating, offer great [illegible] solid film lubricants and corrosion protective [illegible] The excellent adherence and coherence reduces internal stresses and [illegible] morphological growth to form dense, cohesive, equiaxed grain [illegible] of which have a tendency to lower the coefficient of friction, [illegible] and increase surface corrosion protection. For consistent [illegible] it is essential that good quality control is maintained [illegible] plating process.

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1. Maissel, L.: Application of Sputtering to the Deposition of Films, Handbook of Thin Film Technology, Ch. 4, L.I. Maissel and R. Glang, eds., McGraw-Hill, New York, [illegible]
2. Holland, L.: A Review of Plasma Process Studies, Surf. Tech. 11, pp. 145-169, 1980.
3. Bunshah, R.F.: Deposition Technologies for Films and Coatings, Noyes Public, 1982.
4. Spalvins, T.: Influence of [illegible] Surface Pretreatments on Adherence of Sputtered Molybdenum Disulfide to Silver, Gold, Copper and Bronze, NASA TND-7169, 1973.
5. Spalvins, T.: Characteristic Morphological and Frictional Changes in Sputtered MoS_2 Films, ASLE Proceedings - 3rd International Conference on Solid Lubrication 1984. ASLE SP-14 (201-207) Denver Col., 1984.
6. Stupp, B.C.: Synergistic Effects of Metals Co-Sputtered with MoS_2, Thin Solid Films, 84, (257-[illegible]), 1981.
7. Spalvins, T.: Frictional and Morphological Properties of Au-MoS_2 Films Sputtered From a Compact Target, Thin Solid Films, 118, (375-384), 1984.
8. Spalvins, T.: Morphological and Frictional behavior of Sputtered MoS_2 Films, Thin Solid Films, 96 (17-24), 1982.
9. Stupp, B.: Practical Applications and Uses of Solid Lubricant Films, Tribology in the 80's, NASA CP-2300, vol. II. 681-702, 1984.
10. Mattox, D.M.: Fundamentals of Ion Plating, J. Vac. Sci, Technol., vol. 10, no. 1, (47-52), 1973.

11. Spalvins, T.: Survey of Ion Plating Sources, J. Vac. Sci. Technol., vol. 17, no. 1 (315-321), 1980.
12. Spalvins, T.: Ion Plating for the Future, Met. Finish., vol. 80, no. 5, (47-50), May 1982.
13. Mattox, D.M.: Ion Plating Technology, Deposition Technologies for Thin Films, Ch. 6, R.F. Bunshah ed., Noyes Public. 1982.
14. Spalvins, T.: Frictional and Morphological Properties of Ion Plated Soft Metallic Films, Thin Solid Films, 84 (267-272), 1981.
15. Todd, M.J. and Bentall, R.H.: Lead Film Lubrication in Vacuum, ASLE Conference on Solid Lubrication, Denver, ASLE SP-6, 1978.
16. Muehlberger, D.E. and Reilly, J.J.: Improved Equipment Productivity Increases Applications for Ion Vapor Deposition of Aluminum, SAE Reprint P-127, Proc. of the 19th Annual Airline Plating and Metal Finishing Forum, (McDonnel Douglas Corp. Report 830691), 1983.

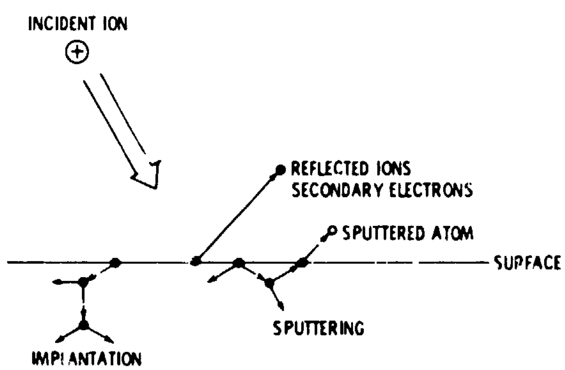

Figure 1. - Ion-surface interactions.

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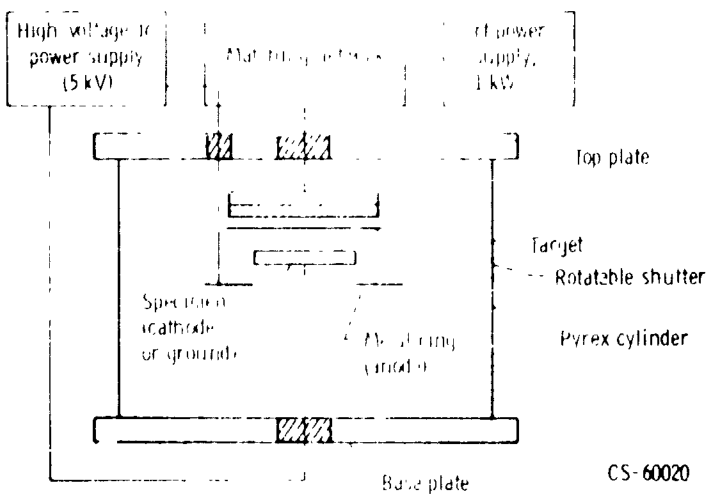
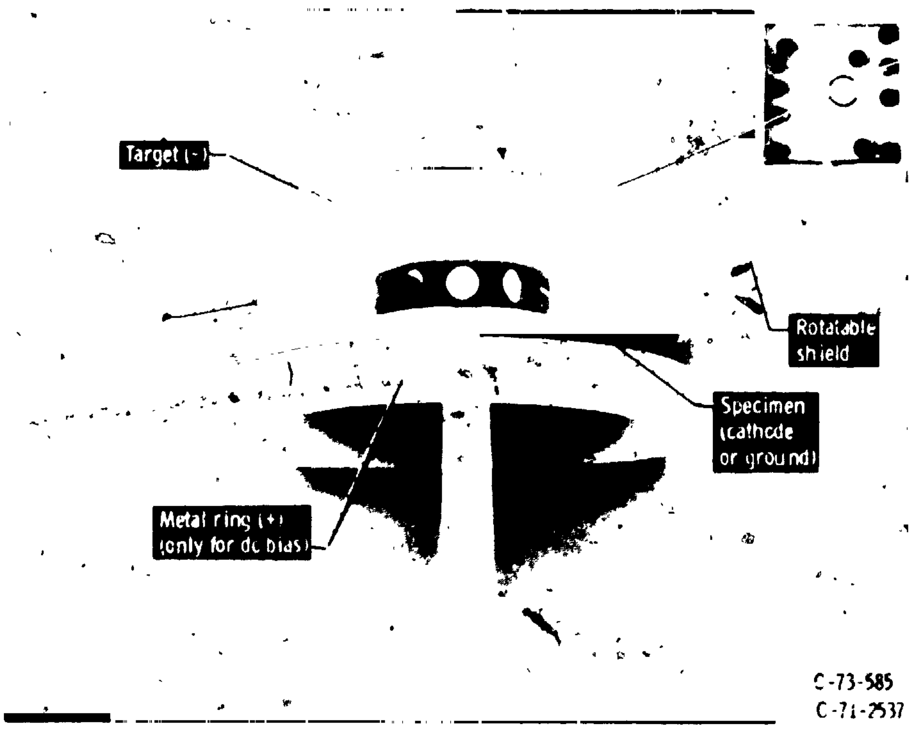


(a) Schematic diagram



(b) View of apparatus during sputter coating.

Figure 2. - Radiofrequency diode sputtering apparatus with direct-current bias.

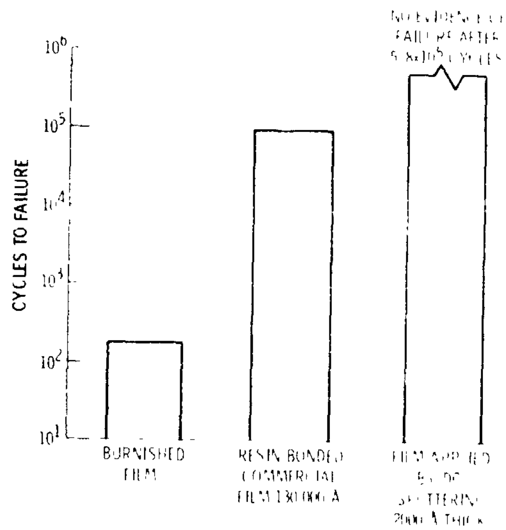


Figure 3. - Endurance lives of MoS_2 films applied by various techniques.

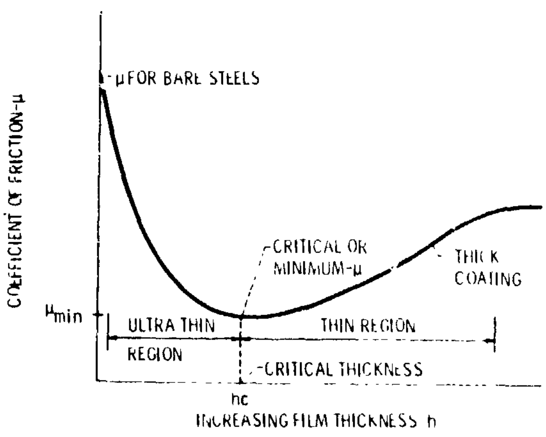


Figure 4. - Schematic representation of the effect of film thickness on the friction coefficient of low shear strength metallic films on steel.

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(a) On 440C stainless-steel substrate.

(b) On glass substrate.

Figure 5. - Sputtered MoS_2 film after a single-pass sliding.

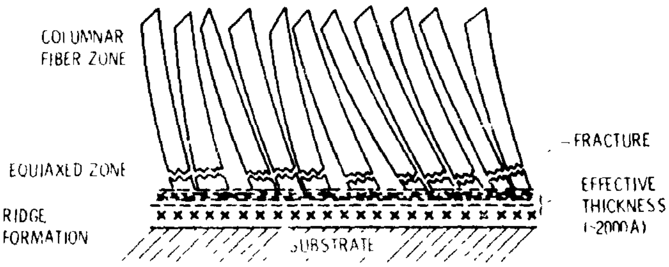

Figure 6. - Fracture during sliding of sputtered MoS_2 film in respect to morphological zones.

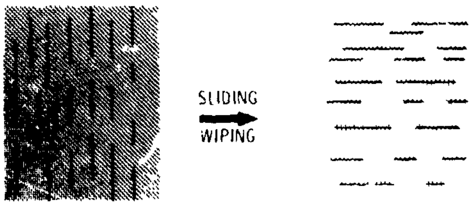

Figure 7. - Reorientation of sputtered MoS_2 platelets.

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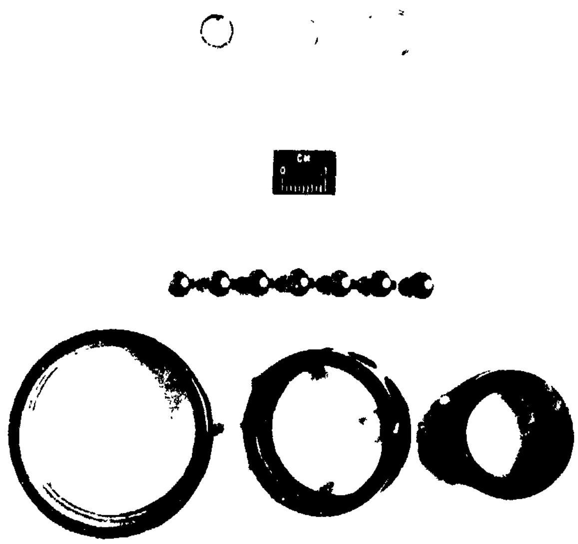

Figure 8. - Two sets of ball bearing assemblies sputter-coated with MoS_2 films.

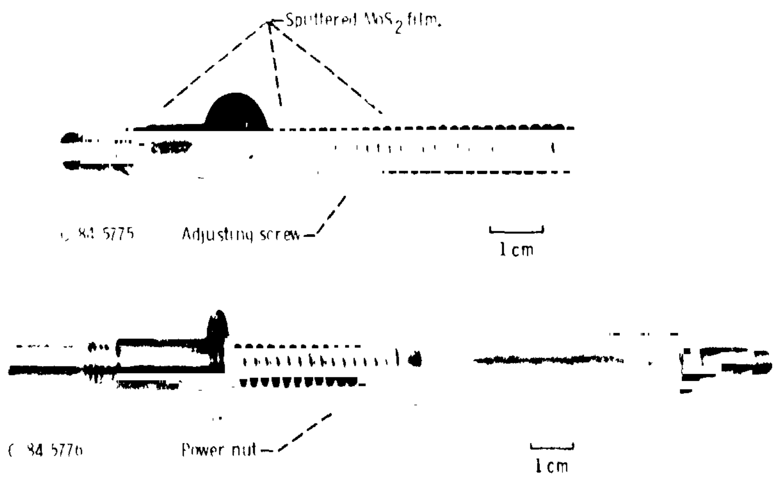

Figure 9. Power nut and adjusting screw assembly sputter coated with MoS_2.

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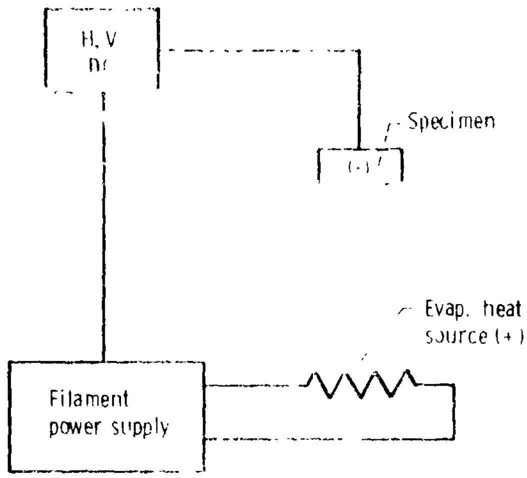
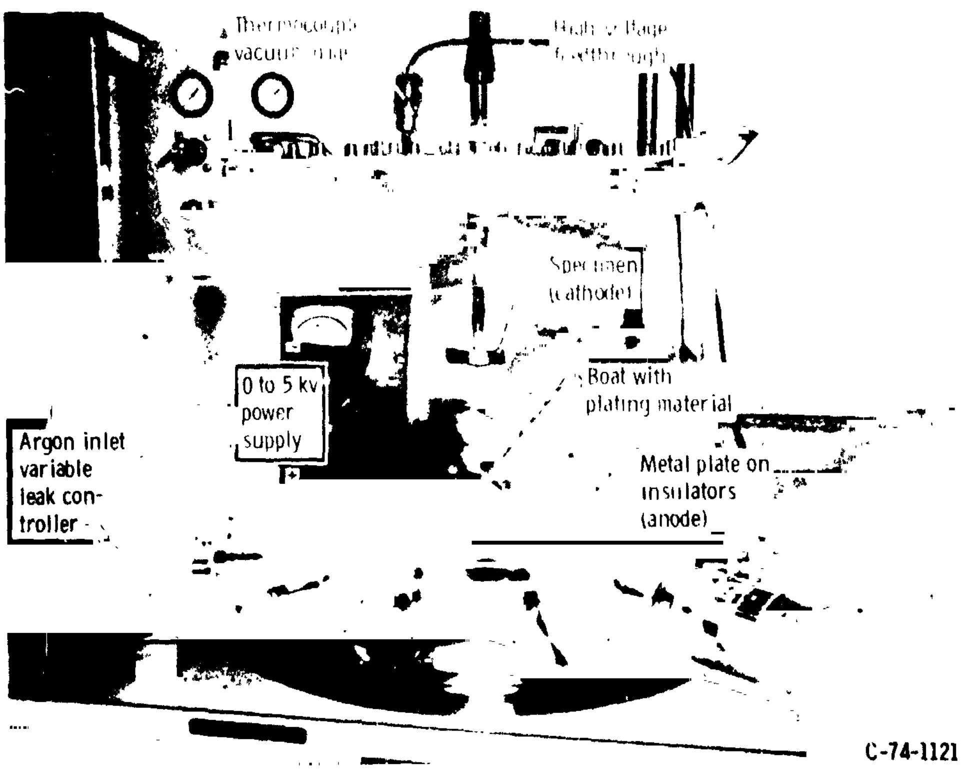


(a) Schematic.



(b) Ion plating chamber.

Figure 10. Ion plating system.

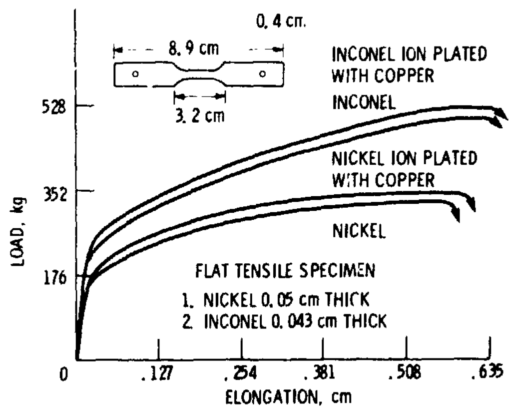


Figure 11. - Load elongation curves during tensile tests.

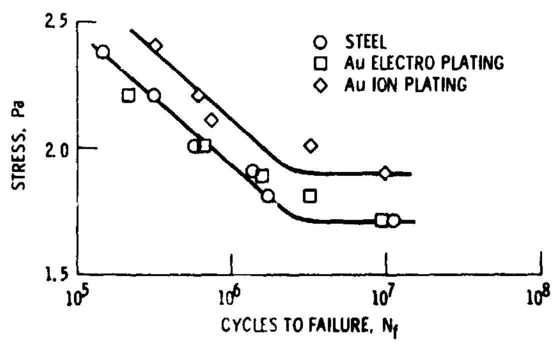


Figure 12. - Effect of ion plating on the fatigue property of low carbon steel.

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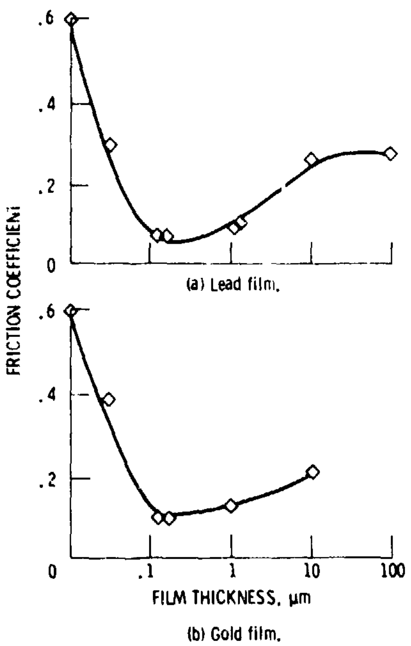

Figure 13. - The variation of friction coefficient with film thickness.

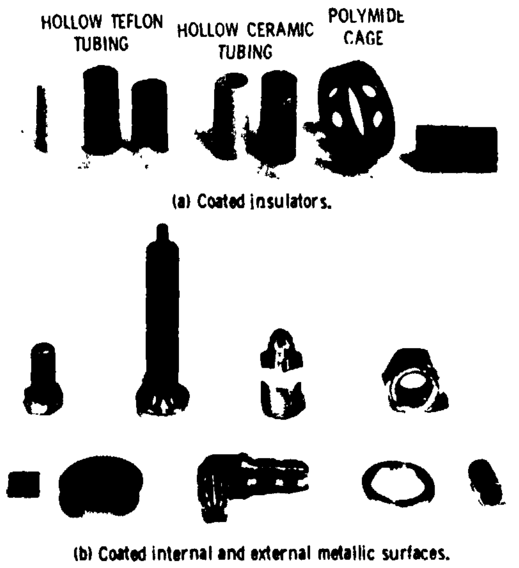

Figure 14. - Ion plated insulators and objects with metallic coating.

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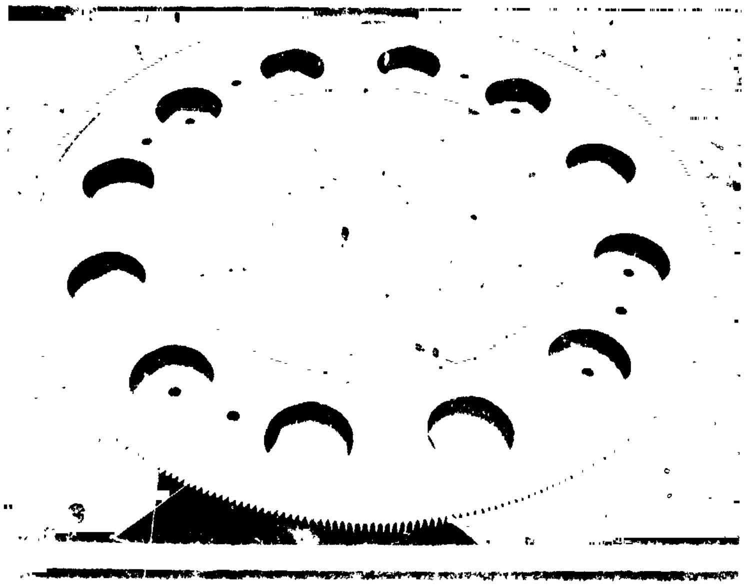

Figure 15. [illegible] C-84-5780

Figure 16. Main [illegible] with aluminum.

Figure 17. [illegible]