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TECHNICAL NOTE 2802

BONDING OF MOLYBDENUM DISULFIDE TO VARIOUS MATERIALS TO FORM A SOLID LUBRICATING FILM

II - FRICTION AND ENDURANCE CHARACTERISTICS OF FILMS BONDED BY PRACTICAL METHODS

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SUMMARY

The use of molybdenum disulfide MoS_2 as a solid-film lubricant is being extended, particularly in applications where designs or higher temperatures preclude liquid lubricants, because of the good frictional and thermal characteristics of MoS_2. An investigation was conducted to determine (1) practical methods of bonding MoS_2 to materials to form solid-film lubricants and (2) friction and endurance characteristics of films so formed. The results indicated that satisfactory solid-film lubricants can be formed on a variety of materials by brushing on MoS_2 mixed with a resin-forming vehicle (1 part MoS_2, 2 parts vehicle, by weight) such as: thinned asphalt-base varnish, silicone-base varnish, or glycerol. The brushing is followed by air drying, infrared drying, and oven curing. The choice of the resin-forming liquid vehicle is governed by the types of application. In the use of asphalt-base varnish, the cleaning of the specimens was not critical because of mutual solubility of the varnish, thinner, and the usual surface contaminating greases. Scraping and rubbing tests showed that all the solid-film lubricants bonded equally well to as-cast, as-rolled, ground, and turned surfaces, indicating that surface finish did not influence tenacity and toughness of the film. Friction and endurance data obtained under severe conditions of high sliding velocities and high surface stress showed that solid-film lubricants (between 0.0002 and 0.0005 in. thick) of MoS_2, bonded with the various resins as well as corn-syrup resin, resulted in good lubricating effectiveness.

INTRODUCTION

The use of molybdenum disulfide MoS_2 as a solid-film lubricant is being extended, particularly in applications where designs or higher temperatures preclude liquid lubricants, because of the good frictional and thermal characteristics of MoS_2. The relatively low shear strength and high load-carrying capacity of MoS_2 (references 1 and 2) results in low coefficients of friction and minimization of surface damage, which are sustained only as long as the material remains between the sliding surfaces. The adherence of MoS_2 to one or both of the surfaces determines the lubricating life of the film. An investigation (reference 3) of the basic mechanism by which MoS_2 is bonded to various materials indicated that, when MoS_2 was applied to a surface as a mixture of MoS_2 powder and some liquid vehicle, the liquid decomposed and polymerized to a resin which bonded the particles of MoS_2 together and to the surface to be lubricated.

The liquid vehicle, corn syrup, used in the method of reference 4 bonded MoS_2 by means of a resin (reference 3) and the resultant solid-film lubricant has been shown to possess good frictional (reference 2) and fretting inhibition properties (reference 5). The high degree of cleanliness of the metal required for successful use of corn syrup as a vehicle, however, imposes severe practical limitations. The incompatibility of surface contaminating greases and water-soluble corn syrup often prevents uniform coverage of the surface by the final lubricating film. Furthermore, the method is impractical in requiring the preheating of the metal and in producing a voluminous charred mass and irritating smoke.

The use of other vehicles for simpler bonding methods is desired. Laboratory experiments have shown (reference 3) that a number of resin-forming viscous liquids will bond MoS_2 powder to a variety of materials. The investigation reported herein was conducted at the NACA Lewis laboratory in order to determine: (1) practical methods of bonding MoS_2 to materials with various resin-forming liquid vehicles and (2) the friction and endurance characteristics of films so formed. The vehicles used were chosen as examples of resin-forming liquids. Friction experiments were conducted using a sliding-friction apparatus that allowed friction of the films to be measured over a wide range of sliding velocities (75 to 8000 ft/min) with high surface loads (initial Hertz surface stress, 126,000 lb/sq in.). Endurance experiments were made using a kinetic-friction apparatus having a hemispherical, hardened-steel rider sliding on a rotating steel disk. The disks had films of MoS_2 bonded with various resins, including corn syrup, for comparison. Loads of 200 to 2000 grams were used at a constant sliding velocity of 445 feet per minute.

SPECIMENS AND SPECIMEN PREPARATION

The material used in the experiments on practical methods of bonding was primarily AISI 347 stainless steel. The extent of one practical method of application was determined, however, using a variety of steels, nonferrous alloys, heat-resistant alloys, and nonmetals. Prior to the application of the MoS_2 film, the metals were cleaned to pass the water-wet test (the ability of water to wet the surface was an indication that the surface was free of adsorbed greases). In the friction and endurance experiments, SAE 1020 cold-rolled steel was used for the disks and hardened (Rockwell C-60) SAE 1095 steel was used for the riders. The procedure for cleaning these specimens, described in detail in reference 6, consisted essentially of scrubbing consecutively with naphtha, acetone and benzene, and moist levigated alumina, then rinsing with water and alcohol.

The MoS_2 powder used in these experiments was the same as that of reference 3; that is, 99.9 percent pure MoS_2 with the following particle size analysis:

	Percent by weight
Over 200 mesh	1
Under 200 mesh and over 400 mesh	10
Under 400 mesh and over 22 microns	30
Under 22 microns and over 11 microns	27
Under 11 microns and over 5 microns	15
Under 5 microns	17

The following liquids were used as examples of resin-forming vehicles:

(a) Asphalt-base varnish, commercial wire-insulating type, GE 457

(b) Silicone-base varnish, commercial wire-insulating type, DC 996

(c) Glycerol

(d) Corn syrup, commercial brown

The following were used as thinners:

(a) Naphtha, boiling range 250° to 300° F

(b) Xylol

FRICTION AND ENDURANCE APPARATUS

Friction Apparatus

The sliding-friction apparatus used for these experiments is that described in reference 6. A diagrammatic sketch of the basic parts is shown in figure 1. The 1/4-inch diameter steel rider is elastically restrained by a dynamometer ring, is loaded vertically by dead weights, and slides in a spiral path on the 13-inch diameter rotating disk. Friction-force readings F were obtained from an indicating-type calibrated potentiometer connected to a strain-gage dynamometer-ring assembly. The coefficient of kinetic friction μ_k was computed from the equation

$$\mu_k = \frac{F}{P}$$

where F is the measured friction force and P is the applied normal load. Friction coefficients over the entire range of speeds were generally reproducible within ±0.02.

Endurance Apparatus

The apparatus used for determining film endurance was essentially the same as that described in reference 7. A diagrammatic sketch is shown in figure 2. The hemispherical end (3/16-in. rad.) of the cylindrical rider, which slides against the $2\frac{1}{2}$-inch rotating disk, is loaded by a dead weight and pulley system. Friction force was measured by a dynamometer-ring strain-gage assembly connected to a calibrated potentiometer. The disk rotated at 825 rpm, producing a linear sliding velocity between the specimens of 445 feet per minute. The load range covered in the endurance experiments was 200 to 2000 grams, producing initial Hertz surface stresses of 87,000 to 188,000 pounds per square inch.

PROCEDURE

Practical MoS_2 Bonding Methods

A large number of 1- by 3-inch panels were coated with mixtures of MoS_2 and thinned liquid vehicles varying regularly in concentration of each ingredient. All were partly dried under infrared bulbs and then cured, either by heating or by prolonged air-drying. The resultant specimens were examined to determine tenacity and toughness of the film. The films were subjected to five scraping passes with a knife edge loaded to approximately 15 pounds and to ten rubbing passes with a 1/2-inch steel ball loaded to approximately 15 pounds. Observations were made of the exposure of base metal (by cracking, flaking, or chipping), the degree of burnishing, the relative hardness, and the general completeness of the film. The mixtures for films exhibiting the best properties were used to coat the disks in the friction and endurance experiments. Films for the experiments of this report were abraded with fine steel wool to make them from 0.0002- to 0.0005-inch thick. Thickness of films was determined by measuring with a micrometer before and after the formation of the lubricant.

Results of experiments conducted to determine practical methods of bonding MoS_2 were resolved into four procedures using four different liquid vehicles: (1) asphalt-base varnish, (2) silicone-base varnish, (3) glycerol, and (4) corn syrup. Each vehicle was chosen for a particular advantage and its use would be governed by the facilities (particularly

Friction Experiments

The data showing the effect of sliding velocity on the friction of steel specimens coated with MoS_2 films are shown in figure 3. The disks were coated with MoS_2 according to the procedures described in the appendix. Except for the data on the corn-syrup resin bonded film, taken from reference 2, all the runs were made with film thicknesses from 0.0002 to 0.0005 inch. The data from reference 2 were obtained with somewhat thicker films. Other unpublished data have shown that when the thickness of a corn-syrup resin bonded MoS_2 film was decreased, friction values also decreased slightly.

At sliding velocities below 2000 feet per minute, the bonding material apparently has some effect on friction although it is only at the minimum sliding velocity that any marked effect is apparent. The one point at the lowest sliding velocity for the asphalt-varnish resin bonded film is appreciably higher than those obtained with other bonding materials.

Data obtained for sliding velocities above 2000 feet per minute do not show any marked effect of bonding media. The observed differences in friction with various bonding materials might be accounted for by different film thicknesses although the values of friction coefficients generally fall within the range (± 0.02) commonly obtained with the use of dry materials on this apparatus.

There was no evidence of film failure (welding or metal transfer) after any of the runs from which the data of figure 3 were obtained. In addition, no evidence of film failure was observed when continuous runs of 3-hour duration were made over the same circumferential wear track. The continuous runs constituted a reasonably severe test of the film since the sliding speed and stresses are higher than those generally experienced in practical operating mechanisms.

These data indicate that under severe conditions of high sliding velocities and high surface stress the frictional behavior of MoS_2 is affected very little by the resins of the liquid vehicles studied; in all cases the film provided excellent lubricating effectiveness. If a low coefficient of friction is desired, the use of the asphalt-varnish bonded film may not be completely satisfactory at low sliding speeds.

Endurance Experiments

The lives of the disks from the four sets of MoS_2 films bonded as described in the appendix and subjected to endurance tests are presented graphically in figure 4. Other similar films, prepared individually and at different times, showed greater endurance. Inasmuch as the reproducibility of these data were poorer, the data are not presented. With thicknesses as low as 0.0002 to 0.0005 inch the short lives magnified small differences in the films and resulted in poor reproducibility. The reproducibility should be proportionately better with thicker films of greater endurance. The lives of a cadmium plate (0.0005 in. thick), a silver plate (0.0005 in. thick), and a clean steel specimen are also shown for comparison. The results show that, under the conditions of this test, there were some differences in time to film failure among the MoS_2 bonded films. The corn-syrup resin film showed greatest endurance, asphalt-base resin was second, and silicone and glycerol were third with approximately equal endurance. The results show further that a MoS_2 bonded film is, for most of the tests, more durable than cadmium or silver plate. The friction forces measured during the experiments gave coefficients of friction in the range from 0.1 to 0.25 at all loads for all resins.

In the endurance tests the nature of the film failure was the same for all resins; that is, friction force at a given load would gradually decrease until failure suddenly occurred, resulting in severe metal pickup, scoring, and rapid rise in friction force. Silicone-resin bonded MoS_2 showed some peeling as failure occurred. Before failure, all the wear tracks appeared highly burnished and failure was apparently propagated from some point where the film was rubbed away so that metal to metal contact occurred and the resultant pickup quickly plowed the film in the remainder of the track. Generally, however, the films showed good endurance and relatively low coefficients of friction under the very severe conditions of the experiments.

SUMMARY OF RESULTS

An investigation of (1) practical methods of bonding molybdenum disulfide MoS_2 to materials to form solid-film lubricants and (2) friction and endurance characteristics of films so formed produced the following results:

1. Satisfactory solid-film lubricants can be formed on a variety of materials by brushing on MoS_2 mixed with a resin-forming vehicle (1 part MoS_2, 2 parts vehicle, by weight) such as: thinned asphalt-base varnish, silicone varnish, or glycerol. The brushing is followed by air drying, infrared drying, and oven curing. The choice of resin-forming liquid vehicle is governed by the application.

Cleaning of specimens was not critical in the use of the asphalt-base varnish as the resin-forming liquid. The scraping and rubbing tests showed that the films bonded equally well to as-cast, as-rolled, ground, and turned surfaces, indicating that the surface finish did not influence the tenacity of the bonded films.

metal cleaning) for bonding and the conditions (particularly temperature) under which the final film would be required to lubricate.

Generally, the use of thinned resin-forming liquids resulted in final MoS_2 films that were uniformly thin, hard, and tenacious. Brushing the mixture on the metal with a soft lacquer brush produced a more uniformly thick film than dipping. Also, predrying by infrared, or any method of heating such that the metal was not the source of heat, gave uniform films that were firm and did not flow when subjected to oven curing temperatures. The detailed procedure for each resin-forming liquid is given in the appendix.

Friction Experiments

The disks were measured, cleaned, and then coated with the MoS_2 solid film, which was between 0.0002 and 0.0005 inch thick, by one of the final practical methods. The disk was then assembled in the apparatus and rotated at a predetermined speed. The loaded rider was lowered onto the disk as the radial feed was started resulting in the rider traversing a spiral track on the disk. As the rider traversed the disk, friction force was indicated and recorded. The runs (of 3-sec duration) for obtaining each point on the friction curve were made with new locations on both disk and rider. A load of 269 grams was used, producing an initial Hertz stress of 126,000 pounds per square inch and sliding velocities were varied from 75 to 8000 feet per minute. In addition, continuous runs of 3-hour duration were made (over the same circumferential wear track) at a load of 269 grams and a sliding velocity of 5000 feet per minute.

Endurance Experiments

The disks were measured, cleaned, and then coated with the MoS_2 solid film, which was between 0.0002 and 0.0005 inch thick, by one of the final practical methods. For each practical method, a set of five disk specimens was prepared at the same time; these disks were then run consecutively to determine reproducibility. The disk and the rider were placed in the apparatus and subjected, successively, to the following load sequence: 200 grams for 2 minutes, 400 grams for 2 minutes, 600 grams for 2 minutes, 1000 grams for 2 minutes, 1600 grams for 2 minutes, and 2000 grams till film failure occurred. Each complete endurance run was made with a separate rider and disk; the rider moved in the same circumferential wear track of the disk. Film failure could be easily identified by high friction force, very rough sliding, and audible chattering. The time and number of cycles from starting to film failure were noted for each disk.

RESULTS AND DISCUSSION

Practical MoS_2 Bonding Methods

The detailed procedure for each resin-forming liquid is given in the appendix. Asphalt-base varnish (for example, GE 457) was chosen because it will air dry at room temperature if sufficient time for hardening is allowed, thereby eliminating the necessity of curing by heating and, consequently, extending the number of materials to which the film can be bonded. This mixture with air drying at room temperature was applied to a wide variety of metallic and nonmetallic materials including: (a) cold-rolled, spring, chrome, and stainless steels; (b) copper, brass, aluminum, monel, and zirconium; (c) heat-resistant alloys such as: Inconel, S-816, N-155, Timken alloy, and Stellite 21; and (d) glass, alundum, porcelain, and other miscellaneous materials such as wood, pressed composition wood, Bakelite, and Lucite. In each case, a satisfactory tenacious film was produced which readily burnished to produce a "slick" surface. This experiment suggests that there is little limitation to materials on which a solid MoS_2 film may be formed. Possibly the use of materials with a porous or interrupted surface to make a more tenacious bond and provide local sources of lubricant would be advantageous.

In the use of asphalt-base varnish as the resin-forming liquid, critical cleaning is unnecessary because the thinning agent, naphtha, the varnish, and the usual surface contaminating greases are mutually soluble.

Silicone-base varnish (for example, DC 996) was chosen as a resin-forming liquid vehicle for applications where greater heat resistance of the solid-film lubricant is required. The silicone used will not craze, if subjected to a temperature of 250° C (482° F) for 500 hours (reference 8). Inasmuch as the silicone thinned with xylol did not wet a surface contaminated with usual greases, steps to clean the surface were necessary.

The suitability of glycerol as a resin-forming viscous liquid was investigated in reference 3 in which it was shown that a good bond could be formed. Difficulties with excessive evaporation and boiling are experienced, however. The investigation reported herein has shown that some of the difficulties of bonding were eliminated when the glycerol was preboiled to approximately one-fifth its original volume. (Boiling should be done with provision for removal of irritating fumes). The boiling removes the chemically combined water and decomposes and polymerizes the glycerol to form a semiresinous material. The use of the preboiled glycerol permits MoS_2 to be bonded to the metal with less difficulty by minimizing boiling and evaporation on the surface during the curing of the MoS_2 film.

The results of the scraping and rubbing tests showed that all the solid-film lubricants bonded equally well to as-cast, as-rolled, ground, and turned surfaces, indicating that surface finish does not influence the tenacity or toughness of the film.

2. Friction and endurance data obtained under severe conditions of high sliding velocities and high surface stress showed that solid-film lubricants (from 0.0002 to 0.0005 in. thick) of MoS_2, bonded with the various resins including corn-syrup resin, resulted in good lubricating effectiveness.

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APPENDIX - FILM-PREPARATION PROCEDURE

MoS_2 applied with asphalt-base varnish. - The process of applying MoS_2 with an asphalt-base varnish follows:

(1) Clean material free of rust, dirt, and other contamination.

(2) Brush on thin film of continuously stirred mixture of 1 part (by weight) of MoS_2 and 2 parts (by weight) asphalt-base varnish thinned 60 percent (by volume) with naphtha.

(3) Air-dry at room temperature until tack-free. This film will become sufficiently hard (equivalent to hardness obtained when cured for 3 hours at 150° C (approximately 300° F)) if enough time at room temperature is allowed.

(4) If an acceleration of curing is desired, the specimen may be exposed to infrared until semicured then heated in a furnace or oven for 3 hours at 150° C (approximately 300° F).

(5) Rub down with steel wool and burnish with soft-clean cloth to form a solid-film lubricant less than 0.001 inch thick.

MoS_2 applied with silicone-base varnish. - The process of applying MoS_2 with silicone-base varnish is as follows:

(1) Clean material until virtually grease free (for example, blasting with sand-water-air mixture followed by rinses in fresh acetone or alcohol).

(2) Brush on thin film of continuously stirred mixture of 1 part (by weight) MoS_2 and 2 parts (by weight) silicone varnish thinned 20 percent (by volume) with xylol.

(3) Dry film under infrared until firm.

(4) Cure film by heating at 200° to 250° C (392° to 482° F) for at least 3 hours.

(5) Rub down with steel wool and burnish with soft-clean cloth to form a solid-film lubricant less than 0.001 inch thick.

MoS_2 applied with glycerol. - The process of applying MoS_2 with glycerol is as follows:

(1) Clean material until virtually grease free, for example, by blasting with sand-water-air mixture followed by several rinses in freshly distilled acetone or uncontaminated alcohol.

(2) Brush on thin film of a continuously stirred mixture of 1 part (by weight) MoS_2, and 2 parts (by weight) glycerin preboiled (to about one-fifth its original volume).

(3) Either (a) heat to 250° to 300° C (482° to 572° F), repeat application of mixture with rubbing until film covers surface completely and continue heating until film is dry, or (b) dry under infrared until film is firm then cure by heating for 3 hours at 250° C (482° F).

(4) Rub down with fine steel wool and burnish with clean soft cloth to form a solid-film lubricant less than 0.001 inch thick.

MoS_2 applied with corn syrup. - The method of applying MoS_2 with corn syrup is somewhat similar to that described in reference 4:

(1) Clean metal until grease free, for example, by scrubbing in uncontaminated solvent followed by rinses in alcohol.

(2) Preheat metal to 250° to 300° C (482° to 572° F) (sufficient to polymerize the vehicle).

(3) Apply mixture of MoS_2 and corn syrup (equal parts by weight) by rubbing and repeat application until film covers surface completely and then continue heating until film is dry.

(4) Scrape off excess and rub down with fine steel wool and burnish with clean soft cloth to form a solid-film lubricant less than 0.001 inch thick.

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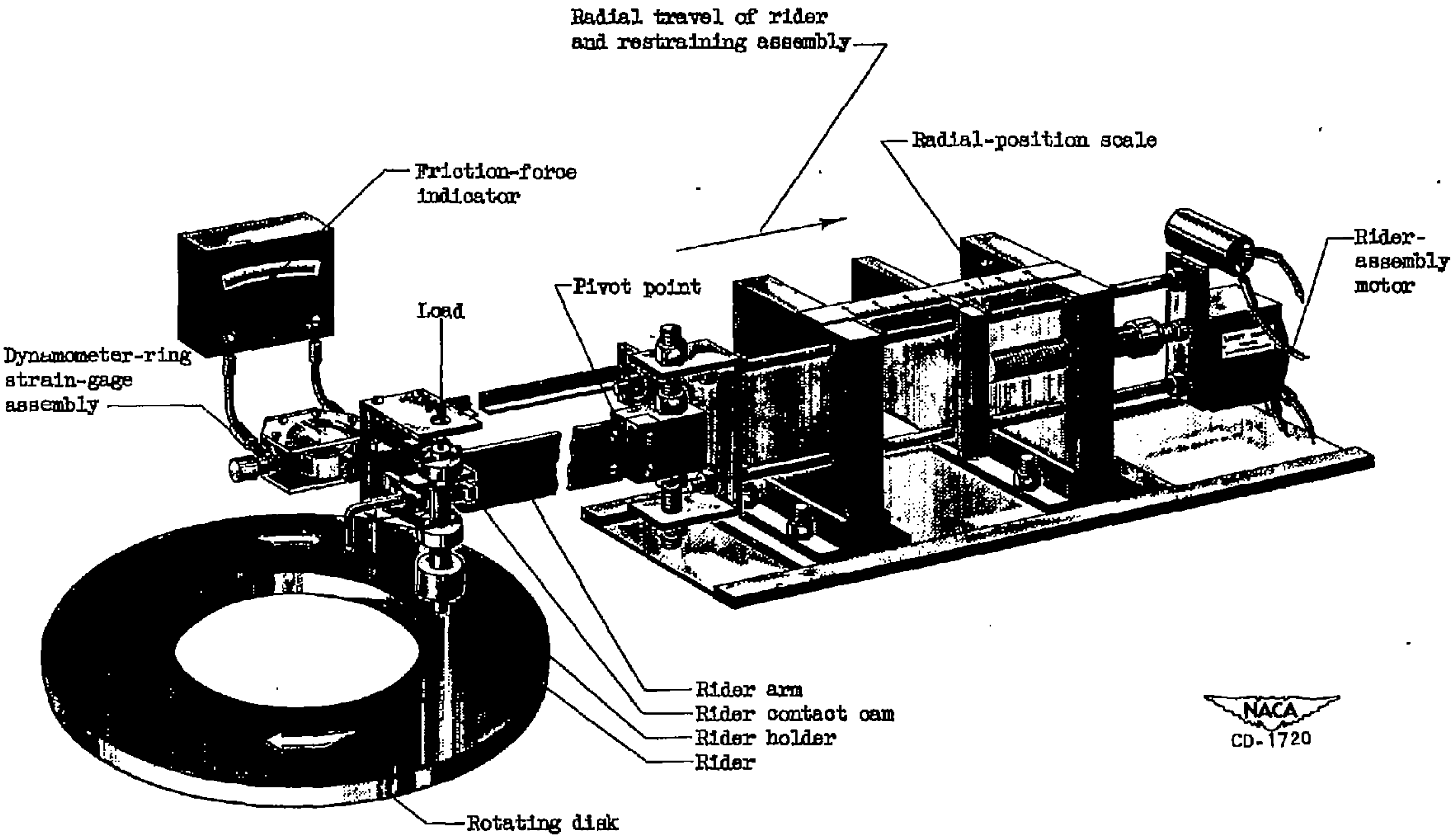

Figure 1. - Schematic diagram of sliding-friction apparatus.

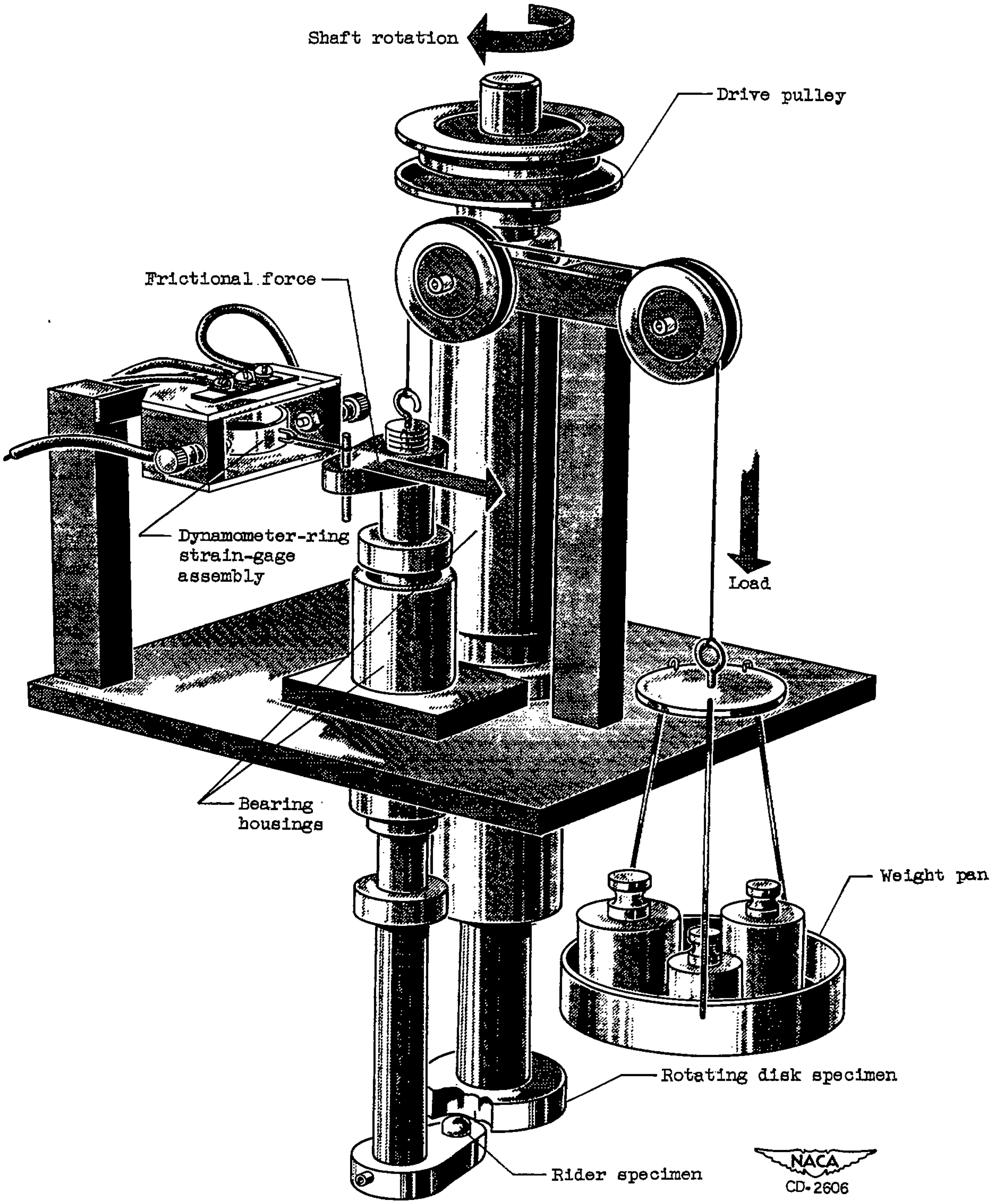

Figure 2. - Schematic diagram of endurance apparatus.

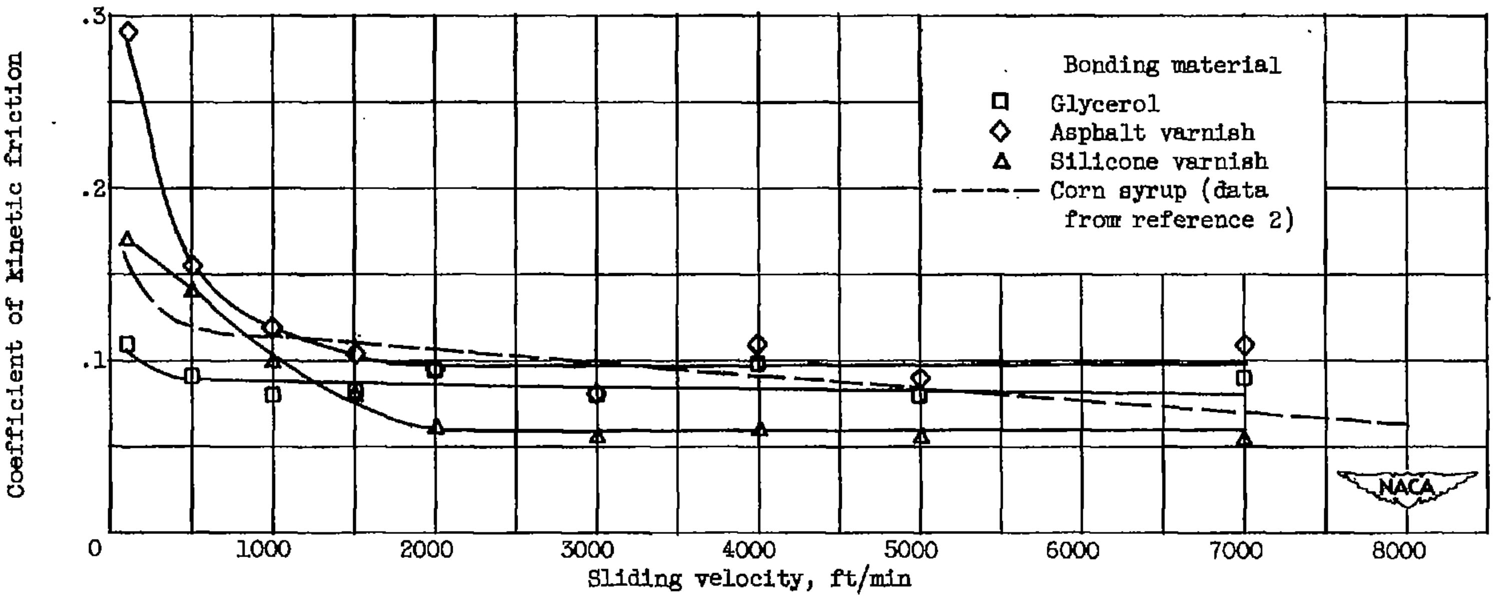

Figure 3. - Effect of sliding velocity on friction of steel specimens coated with solid films of molybdenum disulfide applied with various bonding materials. Load, 269 grams; approximate film thickness, 0.0002 to 0.0005 inch.

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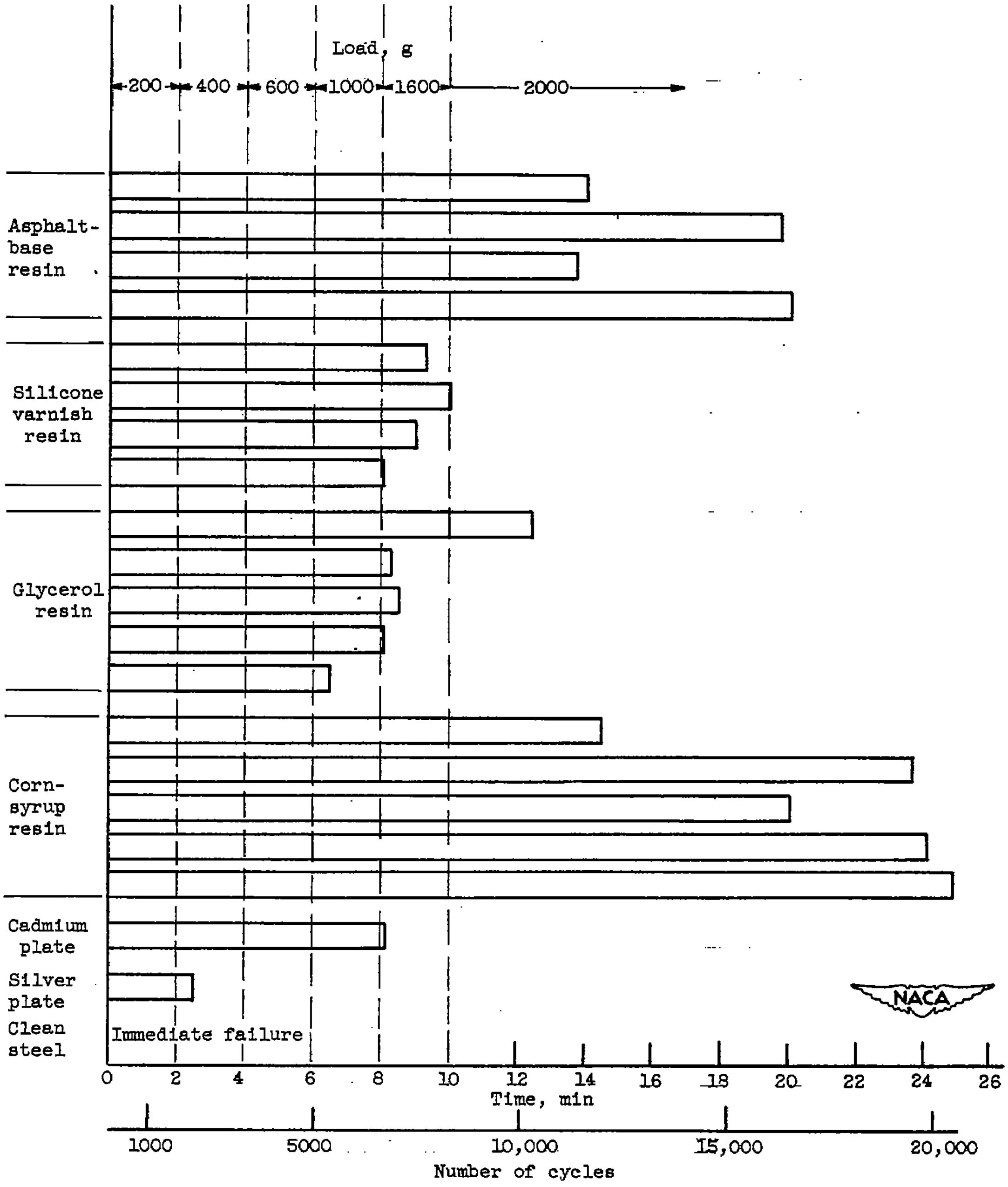

Figure 4. - Endurance tests, showing total number of cycles to failure of solid-film lubricants of MoS_2 using four different resin forming liquids. Sliding velocity, 425 feet per minute.