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PROCEDURES FOR QUANTITATIVE CHARACTERIZATION OF WORKPIECE AND DIE COATINGS  
FOR ISOTHERMAL FORGING OF TITANIUM ALLOYS

WESTINGHOUSE ELECTRIC CORPORATION  
ADVANCED ENERGY SYSTEMS DIVISION  
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AIR FORCE MATERIALS LABORATORY (LLM)  
WRIGHT-PATTERSON AIR FORCE BASE, OHIO 45433

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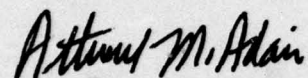
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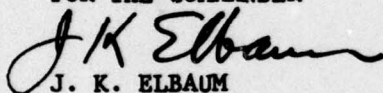
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The end result for use of these techniques will allow the formulation of a scientific basis for these types of forging operations.

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# FOREWORD

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## SECTION I

### INTRODUCTION

A recent Air Force-Industry Conference on cost reduction of aircraft structural components<sup>(1)</sup> revealed that the final component volume is typically only 15 percent of the purchased material volume. This low percentage of material utilization is primarily owing to the large envelope required to obtain the good metal flow needed to insure complete die-fill when forging the more difficult-to-work aerospace alloys and to allow non-destructive inspection which cannot currently verify near-surface regions. The machining operations required for the removal of this excess material were found to be the major cause for the high cost of these components. Of additional significance, however, is that a considerable portion of the raw material for these advanced structural applications is from foreign sources and in the case of titanium alloys, the scrap material is of almost no value because of contamination which results during the machining process. The increasing costs of foreign supplied raw materials and the increased world energy shortages give greater emphasis to the requirement that lower costs in aerospace components production must be associated with a program to reduce machining and to increase material utilization.

One of the methods proposed for achieving increased material utilization and lower cost production of aircraft components is through isothermal forging operations. This process entails forming on dies which are heated to the same temperature as the workpiece. Surface chilling of the piece is eliminated by this process and good die-fill is enhanced through uniform low flow stress achieved by use of low deformation rates. The greatly enhanced metal flow condition allows the production of components to near-net and, in some cases, net-shape configuration. This shape capability eliminates or significantly reduces the amount of machining required and consequently allows greater improved material utilization to be achieved. As a result, an economic gain can be realized in the production of certain components even though the isothermal forging process itself is significantly more costly than conventional forging since it necessitates a die heating system, it requires dies produced from costly and difficult-to-fabricate material and the process itself has low production rates.

A major factor influencing the success of isothermal forging operations is the behavior of the interface between the workpiece and the die. Consideration of this interface is frequently limited to a workpiece applied coating, but it may also include a die coating and the characteristics of the die material and the die geometry itself. The behavior or performance characteristics of the interface is a function of two factors; the coating properties and the process conditions. Proper analysis of the effects of the interface condition must allow a differentiation of these two factors by a numerical rating system. Such a rating system would allow the establishment of a quantitative basis for the determination of the performance characteristics with the coating properties during actual processing conditions.

Results obtainable from this type of analysis would establish a more economical and more sound engineering basis for the selection and control of coatings than that which is obtained by the present trial and error method. These results also provide the basis for quantitative control of the process and for development of coatings for improved performance. Methods for quantitative measurement of coating performance in isothermal forging of titanium alloys and the correlation with coating characteristics have been developed at the Air Force Materials Laboratory and are described in this report.

## SECTION II

### BACKGROUND

#### 2.1 Role of Coatings in Forging

In both conventional and isothermal forging, coatings are applied to the workpiece to achieve two main effects:

- (1) Separation of the workpiece from the die.
- (2) Formation of a low friction interface between the workpiece and the die during the deformation process.

The coating also serves other useful purposes:

- (3) Protection of the workpiece from reaction with the heating furnace atmosphere without excessive chemical attack between the coating and workpiece.
- (4) Thermal insulation during transfer of the workpiece from the furnace to the die and during the time that the workpiece is in contact with the tooling prior to deformation.

In isothermal forging, thermal insulation properties of the coatings are not essential since any temperature loss occurring during the workpiece transfer is regained on contact with the heated dies. However, because of the higher die temperatures and longer contact times in isothermal forging operations, it is essential that the coatings in these operations also provide:

- (5) Chemical compatibility with the die material.

After completion of the forging process the coatings must provide other properties:

- (6) Low adhesion interfaces to allow the forged component to be removed from the die without distortion of the component or damage to the die.
- (7) Limited build-up on the die and limited accumulation in the die cavities to allow the production of the maximum number of forgings capable of meeting the final component dimensional requirements.

#### 2.2 Coating Selection

Examination of the role of coatings in forging operations reveals that the role is dictated by process conditions such as:

- (1) Workpiece and die materials



- (2) Workpiece and die geometry
- (3) Processing temperature and atmosphere
- (4) Equipment kinetics and kinematics

The characteristics of coating selection from the coating viewpoint requires several other considerations.

- (1) Ease of application to and removal from the tooling and the die
- (2) Ability to form and maintain a continuous film during the billet heating and during the forging operation
- (3) Chemical composition giving minimum reaction with both the workpiece and die materials under processing conditions
- (4) Low shear strength in the interface between the workpiece and the die
- (5) Low adhesion strength of the interface film between the workpiece and the die after forging
- (6) Controlled build-up and accumulation of the coating substances on the die surfaces after removal of the forged part from the die
- (7) Compatibility with health and environmental standards

The inference that can be deduced from the above lists is that coating performance under actual operations is not only a function of the coating chemistry, but is also dependent upon the process condition as well. The determination of the optimum coating for specific applications, therefore, requires consideration of two fundamental questions.

- (1) What performance criteria do the process conditions demand that the coating provide?
- (2) What characteristics of the coating allows these performance criteria to be met?

The ability to answer these two questions determines the degree to which an optimum coating can be selected for a specific process.

### 2.3 Barriers to the Advancement of Coating Technology

One of the prime limitations to obtaining answers to the fundamental questions in coating selection is the limited ability to distinguish between the various aspects of the isothermal processing technology. In most cases, isothermal forging operations are conducted on load controlled presses.

As such, the actual forging rate is dependent upon the resistance offered by the internal stresses of the workpiece material and the friction stresses of the coating. Since both the material flow stress and the coating friction stress are rate dependent, it becomes somewhat questionable as to which effect dominates. Thus, one of the first limitations to assessment of coating characteristics is the ability to distinguish between equipment characteristics, workpiece characteristics and coating characteristics. Coating evaluations performed on speed controlled equipment where the material flow stress can be determined allows this limitation to be removed.

A second limitation to assessment of desired coating characteristics is the controlled measurement of processing effects. Reliable and reproducible measurements of temperatures and loads are required. But, of even more importance is a system of measurements which are free of contamination from earlier evaluation tests. This latter factor requires interchangeability of forging dies so that controlled surfaces can be assured.

Perhaps the most serious limitation to the assessment of coating characteristics is the proprietary nature of commercial coating formulations. Since every quantitative aspect of the coating is directly related to its chemical composition, the lack of information on chemical composition and resultant viscosity-temperature relationship of commercially available coatings necessitates that evaluation of these coatings be limited to a "good" or "bad" rating. The limited information available from these types of evaluations hinders the ability to establish the direction of composition modification needed to obtain improvement. In order to circumvent these proprietary aspects, basic desired characteristics must be established through the use of known standard glass compositions using programmed additions and modifications.



### SECTION III

#### EQUIPMENT FOR COATING EVALUATION

Conventional industry practice in isothermal forging is to utilize load controlled press operation. As such, the actual forging rate is dependent upon the resistance offered by the changing deformation volume of the workpiece material and the changing interface area where friction stresses occur. It is evident then that the resistance offered to the deformation process involves deformation rate effects in both the interface coating and the material shear stress. In order to determine the optimum lubrication substances for use and also to optimize the workpiece temperature conditions, it would be necessary to distinguish between these two interacting effects. To accomplish this, it is necessary that the material flow stress-strain rate relationship be determined for the temperature of interest and separated from the friction stress exerted by the interface substances. Such a separation of effects can be accomplished concurrently with the ring compression test (to be described later) using equipment which allows controlled deformation at ram speeds typical of those expected for isothermal forging operations.

#### 3.1 Ram Speed Control

Equipment modifications to allow controlled slow-speed ram motion have been adopted on the  $4.4 \times 10^4 \text{ N}$  (500 Ton) Hydraulic Press located at the Air Force Materials Laboratory. The equipment modifications entail an auxiliary hydraulic system which can be easily activated through high pressure valves after the main hydraulic system of the press has been isolated. Controlled motion of the ram is achieved through use of a closed loop electrical control system which consists of a rotary velocity transducer, a variable potentiometer servo-control system and a high pressure hydraulic servo-valve. Use of this auxiliary hydraulic system allows controlled ram speeds from  $1.26 \times 10^{-5} \text{ ms}^{-1}$  (0.030 ipm) to  $2.1 \times 10^{-3} \text{ ms}^{-1}$  (5.0 ipm). The conventional or standard ram control system allows the achievement of ram speeds of about  $1.26 \times 10^{-2} \text{ ms}^{-1}$  (300 ipm).

#### 3.2 Die Temperature Control

In order for evaluation data to be reliable, a system for close control of die temperature must be assured. Several possible heating techniques are available for achieving this control. Among these are: 1) induction, 2) gas flame, 3) imbedded cartridge heaters, 4) quartz radiation heaters and 5) silicon carbide resistance heaters. Each of these heating systems has certain advantages and certain limitations in regard to uniformity of temperature achieved, maximum temperature achievable, durability under forging operations and adaptability to specific geometry forging dies. A discussion of these factors is given in other publications<sup>(2,3)</sup>.

For isothermal forging of titanium alloys, temperatures of  $1227^\circ \text{K}$  ( $1750^\circ \text{F}$ ) and lower are generally used. These temperatures can be achieved through use of imbedded cartridge heaters. This type of heating system is

employed on the AFML isothermal forging system. The basic system has already been described elsewhere<sup>(4)</sup> but has since been somewhat modified to utilize a solid heater block for both the upper and lower dies as shown in Fig. 1. Supplemental heat for the AFML system is obtained from Kanthal resistance coil heaters imbedded in ceramic plates which are located on two opposing interior surfaces of the die inclosure chamber. These plates are positioned as near as possible to the movable die system to insure temperature uniformity over the surface of the dies.

Temperature control for each region of supplied heat is achieved through use of SCR type controllers. During the placement and removal of the workpiece, temperatures drops of 31.7°K (25°F) to 37.2°K (35°F) are measured at a position just below the die surface. These temperature drops are recovered within one-minute after the die inclosure chamber is closed.

### 3.3 Die Contamination Control

One of the principal difficulties in evaluating workpiece coatings for isothermal forging is to insure that each evaluation test is free of contamination from earlier tests. The occurrence of any such contamination would make the test results very difficult to interpret and would essentially negate any hope of simulating actual production conditions.

In order to insure that each coating evaluated in this test program met the contamination free requirement, a die and die holder system was developed which allowed the forging dies to be interchanged rapidly. This system is shown in Fig. 1 for flat upset forging dies. The dies are beveled on three edges with a handling tab welded on the fourth edge. The die holder is fabricated with a dove-tailed slot which allows easy placement and removal of the dies while each are at the operating temperature. The use of a sufficient supply of dies and an independent heating furnace at the desired operating temperature allows a large number of different workpiece coatings to be evaluated rapidly. After the dies have cooled to room temperature, the surfaces can easily be reconditioned and thereby allowing the next series of tests to be conducted on contamination free dies.

The ability to interchange the dies rapidly offers some other advantages. It allows the surface of the dies to be examined after a controlled number of forgings to detect the extent of die wear and coating build-up. It also allows the effects of different die materials to be determined in the same test sequence with coatings to optimize die materials. The ease with which these dies can be coated by conventional or flame spraying techniques allows die coatings to be evaluated either alone or in combination with workpiece coatings.

## SECTION IV

### ASSESSMENT OF INTERFACE CHARACTERISTICS

The many variables associated with the isothermal forging operations seem to allow considerable flexibility of choice for optimization of the process. After a more careful examination, however, this flexibility appears so restricted that often the least detrimental choice is utilized for optimization. Since the workpiece selection is dictated by design considerations and service properties and since the die material is either already available or dictated by strength considerations, only the coating lubricant allows any degree of freedom of selection to develop optimum forging quality and production. Even then the many requirements that the coating lubricant must fulfill impose severe restrictions on the selection and development of the optimum coating lubricant. These considerations are illustrated in Fig. 2 and are discussed in more detail in the following sections.

#### 4.1 Film Continuity

Economic considerations for isothermal forging of titanium alloys suggests that these processes require minimum modification of existing equipment and facilities. Use of normal atmosphere billet heating furnaces and only slightly modified processing equipment best allows the achievement of economic advantages by the isothermal forging process. Indeed, the consideration of larger production runs of large plan area forging significantly limits consideration of inert atmosphere heating and handling operations.

The desire for eliminating inert atmosphere control in isothermal forging of titanium in turn stipulates the first requirement of a workpiece coating for these processes. The coating must provide environmental protection to the titanium workpiece. This requirement stems from the high reactivity of titanium with oxygen and from the tendency for titanium to form low melting point eutectics when in contact with nickel and iron base alloys in the temperature range between 1144°K (1600°F) and 1311°K (1900°F).

In order to achieve this required environmental protection, the coating lubricant must completely wet the titanium. Any beading of the coating or reaction of the coating with the titanium which produces gas pockets will cause bare areas on the workpiece and thus cause failure of the coating. Visual examination of the coating after a thermal exposure cycle typical of that to be expected in the billet heating cycle provides a suitable technique for evaluation of this lubricant characteristic. Examples of acceptable and nonacceptable coating are shown in Fig. 3.

Of additional importance is the requirement that the coating maintain its continuous film characteristics during the deformation process. If film breakdown occurs during forging, then galling between the titanium and the die can occur as shown in Fig. 4. When these galled regions are exposed to temperatures above 1200°K (1700°F), very rapid and very severe die wear results.



#### 4.2 Compatibility Test For Chemical Reactivity

A coating quality of equal importance to film continuity is chemical compatibility with the workpiece and tooling. The condition of chemical compatibility must consider the total time-temperature history of contact between the coating and workpiece or die. For the workpiece, this would entail the condition for application of the coating, the time required during heating to the desired forging temperature, and the hold time at the forging temperature. If secondary processing conditions are to be performed without removal of the coating, then this additional time-temperature exposure condition must also be considered.

The compatibility of the coating lubricant with the die material is perhaps even more important than the compatibility with the workpiece. The period of high temperature contact between the lubricant and the die often extends for several hours and in some cases several days. Die materials for isothermal forging of titanium alloys are generally of nickel base superalloys and therefore are particularly susceptible to intergranular attack by sulfur, phosphorus, bismuth and to some extent by other elements.

The determination of chemical compatibility is best assessed by applying a layer of coating lubricant to a surface of the workpiece and die materials and subjecting these coated pieces to various time-temperature exposures simulating those experienced in actual forging operations. A specimen design for determining these effects is shown in Fig. 5. A reaction zone typical of that from high temperature exposure with the workpiece is shown in Fig. 6. Compatibility of the lubricant with the die can be determined by the same procedure. However, since corrosive attack is almost always heavier in dynamic systems than in static systems this condition must be simulated by removal and resupply of the coating substance. The reaction zones resultant from such exposure with a die material are shown in Fig. 7.

Quantitative assessment of the extent of chemical reaction can be made by controlled time-temperature exposure between the coating substance and the material of interest. The extent of the reaction zone can be measured by metallographic techniques and identification of the chemical constituents causing the reaction can be made by microprobe analysis.

#### 4.3 Anti-Friction Characteristics

The achievement of favorable metal flow at high workpiece temperatures and low deformation rates, requires very low anti-friction characteristics from the coating lubricant. A technique for determining these anti-friction characteristics is through the ring compression test<sup>(5)</sup>. The basic principal of the ring compression test is that interface friction acting at the tool-workpiece interface during plastic deformation affects the geometric shape change of the workpiece. For ring forging, this shape change is illustrated in Fig. 8 which shows an undeformed ring and two rings deformed

with different interface friction conditions, but in an otherwise identical manner. The amount of interfacial resistance is measured by an interface shear factor denoted by the letter "m". This factor is the ratio of the shear stress of the lubricant to the shear stress of the material undergoing deformation. The range of values of the interface friction factor are,  $m = 0$ , for zero friction (an unobtainable condition) to a value of  $m = 1$  for maximum friction. At high values of  $m$ , a large amount of the metal of the ring flows inward and results in a considerable reduction of the internal diameter of the ring. Lower values of  $m$  reduce the inward flow of the metal and can cause the internal diameter to increase.

Experimental and mathematical calibrations of this geometric change have been performed and a typical calibration curve, based on the assumption of constant interface friction is shown in Fig. 9. A mathematical analysis of the ring forge test which does not require the assumption of constant friction is also available<sup>(6)</sup>. Results from a non-constant friction analysis allow the breakdown condition of the lubricant film during forging to be determined. Typical results for this condition are shown in Fig. 10.

#### 4.4 Adhesion Characteristics

The adhesive bond strength that the workpiece coating forms between the workpiece and the die during forging is of significant importance in the production of controlled geometry product. As stated earlier, the bond strength of some of the workpiece coatings currently used for isothermal forging operations is of such magnitude that severe workpiece distortion occurs upon ejection of thin components and in some cases the "knock-out" pins have almost punched through the forged piece. In order for the economical potential of the isothermal forging process to be realized, a technique for measuring the adhesion loads must be verified so that the chemical composition which allows low bond strengths can be formulated.

A technique for verifying these measurements has been developed. The equipment for these measurements is shown in Figs. 1 and 11. The technique involves producing dove-tailed slots across the bottom die plate and using a disk specimen incorporating a rectangular tab as shown in Fig. 1. The width of the tab is slightly less than the narrowest portion of the dove-tailed slot, but the height of the tab is greater than the depth of the dove-tailed slot. The placement of the workpiece in the die prior to forging is such that the tab is positioned into the dove-tailed slot. The initial forging of the test specimen causes the tab to upset and almost fill the dove-tailed slot in the die. This process essentially locks the workpiece into the bottom die. After a selected amount of forging reduction, the ram is reversed and separation is forced to occur at the selected interface.

During die separation the adhesion load is transmitted through the dove-tailed locked tab to the insert die and then to the die hold-down, Fig. 1. The die hold-down is positioned onto the heated block through five superalloy bolts which are capped but not snugged; approximately one-eighth inch of separation is allowed between the hold-down and the heated block.



The adhesion load is thereby allowed to be transmitted by way of the hold-down tabs to strain gaged instrumented strips located outside of the hot zone of the system, Fig. 11. This load is monitored through use of a Honeywell 906B Visicorder.

Adhesion test data are obtained in triplicate. It is convenient to utilize an average value of these tests to make comparisons between the various coating lubricants. However, in actual application the maximum loads are of most significance. The importance of this controlled separation is that it prohibits separation on the weaker interface and thereby allows the determination of the maximum value of the adhesion loads that might be experienced in actual forging operations.

#### 4.5 Coating Accumulation on the Die

Closely related to the problem of the high strength adhesion bond which the coating lubricant forms between the workpiece and the die is the problem of lubricant "build-up" on the die surface and in the die cavities. The "build-up" is the accumulation of coating resulting from successive forging in the same die system. The pattern of "build-up" is shown in Fig. 12, where an initial steady state or limit value is approached beyond which no further accumulation occurs\*. This condition can be described mathematically and the amount of accumulation after any select number of forgings can be predicted†. Comparison of the amount of "build-up" with the limiting acceptable loss of part detail allows the acceptability of a coating to be established.

The quantitative determination of the amount and rate of "build-up" can be made by at least two techniques. One technique is to use repeated forgings of the adhesion specimen on the same set of dies. The change in the thickness of the central portion of the die measured after each forging gives the rate of "build-up". Only a few forgings are required to establish a fit to mathematical equations (Eq. 5 and Eq. 6 in Section V). The technique for making these measurements is shown in Fig. 13.

A second technique is to use a roof truss shaped forge specimen as proposed and used by IITRI<sup>(7)</sup>. A modification of this type of specimen is shown in Fig. 14. Repeated forging of these types of specimens in the same die would result in a "build-up" of the coating lubricant in the peak section of the roof truss shape. Measurement of the reduction in the height of the peak with each successive forging should thereby give a direct measurement of the "build-up" and allow a fit to mathematical equations to be obtained (Eq. 5 and Eq. 6 of Section V). It should be noted that the actual values

\*It is not likely that a linear "build-up" of the coating will occur on the die because this would imply that an equal amount of the coating is transferred from the workpiece to the die by each successive forging.

†The mathematical equation form will be discussed in the next section.

of developed mathematical equations determined by different techniques might be slightly different, but the trends from lubricant-to-lubricant should be the same.

#### 4.6 Application and Removal

The industrial approach to application of coating lubricants must consider several different aspects.

1. Application of a coating of uniform thickness.
2. Application to non-uniform geometry shaped preforms.
3. Application to allow high production rates.
4. Be able to touch-up while the workpiece is cold before heating and while the piece is hot prior to forging.
5. Consideration for dipping, brushing or spraying.

Arguments have been presented<sup>(2)</sup> which consider the importance of application of the coating to the workpiece. The overall applicability of these arguments still remain to be verified and should be examined in more detail.

Consideration must be given to the ease of removal of the coating lubricants. For the workpiece, it would be desirable if the coating would crumble upon cooling to room temperature or could be removed easily by being soluble in water or some other low-cost readily available media. Grit blasting will easily remove the glossy coating from the workpiece, but the process requires that each individual piece be handled and also affects the surface finish of the forging.

#### 4.7 OSHA and EPA Requirements

The safety of operating personnel and ecological disposal of waste products are of prime importance in any coating lubricant development program for use in industry. Details of these requirements are covered in existing regulations by the OSHA and EPA organizations and are subject to change by them as requirements change. An example is a recent OSHA ruling on benzene<sup>(8)</sup> which can be found in commercial xylene. This ruling might affect the use of xylene as a carrier for powdered glasses in these coatings.

#### 4.8 Cost and Availability

The importance of cost and availability are obvious considerations for the selection and development of coating lubricants. Shelf life, particulate settling characteristics and component deterioration must be considered as an aspect of cost and availability. These factors are a function of the particular type of coating lubricant and must be considered with the overall cost of the specific forging operation.

## SECTION V

### DISCUSSION

The coating evaluation techniques outlined in the previous section allow service performance characteristics of isothermal forging interface coatings to be determined quantitatively. This capability allows the adhesion, friction, accumulation and chemical reaction characteristics of each coating to be determined under service conditions. As such, a considerable advancement can be made in the determination of optimum coating properties. The most ideal condition, however, is the ability to formulate interface coatings so that the desired service performance characteristics can be obtained for a particular processing operation. This ideal condition would require that the desired performance characteristics be related to fundamental chemical properties of the coating itself, such as viscosity or surface energy. The preliminary requirement toward the achievement of this ideal condition is to be able to relate the measured service performance characteristics to these fundamental chemical properties. A schematic arrangement of this condition has been shown previously in Fig. 2.

To a limited extent, a basis for this approach can be established. The first aspect is the consideration that the viscosity of a glass is directly related to the chemical composition. The viscosity of a glass has been shown<sup>(9)</sup> to follow an empirical inverse relation with temperature such as:

$$\log \eta = -A + \frac{B}{T - T_0} \quad (1)$$

where  $\eta$  is the viscosity,  $T$  is the absolute temperature and  $A$ ,  $B$ , and  $T_0$  are constants known as Fulcher constants.

The relation of these glass properties to desired performance characteristics during processing can be made through use of standard formulations. The interface shear factor or "m" factor has been shown<sup>(10)</sup> to follow actual metal deformations closely. This factor is formulated as:

$$m = \frac{\tau}{\sigma/\sqrt{3}} \quad (2)$$

where  $m$  is the interface shear factor or friction factor,  $\tau$  is the shear stress of the coating and  $\sigma/\sqrt{3}$  is the shear stress of the workpiece. All of the factors of Eq. 2 can be determined from the ring compression test.

For glass coatings, it is generally accepted that the shear stress can be approximated in general terms of a Newtonian fluid formulation as:

$$\tau = \eta \frac{\Delta v}{\Delta t} \quad (3)$$

where  $\eta$  is the viscosity

$v$  is the velocity of flow

$t$  is the film thickness

The combination of Eq. 2 and Eq. 3 gives the interface friction during a forging operation in terms of the characteristics of the material, the equipment, and the coating.



As mentioned earlier, the adhesion bond strength between the workpiece and die is directly related to the logarithm of the viscosity. This finding allows a relation for adhesion strength to be developed as:

$$P = \alpha \left( -A + \frac{B}{T - T_0} \right) \quad (4)$$

where the value  $\alpha$  is a function of the glass thickness and the rate of separation.

The build-up on the die also seems to be related through an empirical relation such as:

$$\log y = C_1 + \frac{C_2}{T} \quad (5)$$

Both Eq. 4 and Eq. 5 are similar in the inverse relation to temperature and therefore, the relation of build-up to viscosity seems appropriate.

Of particular interest, however, is the longer time effect of the coating build-up such as would occur during a production run. This type of build-up would be expected to obey a logarithmic relationship expressed as:

$$y = b (1 - e^{-ax}) \quad (6)$$

where  $y$  is the amount of build-up in inches (or weight) and  $x$  is the number of forgings. The constant " $b$ " represents the limit or maximum acceptable amount of build-up and " $a$ " represents the rate of approach to the maximum value. Since the build-up after three tests has been shown to be closely related to the viscosity of the coating it then seems likely that the constant " $b$ " would also be relatable to the viscosity.

The depth of the chemical reaction zone is governed by diffusion conditions and therefore can also be related by simple mathematics if diffusivity values were known. Of more importance, however, is the fact that the prime diffusion specie can be determined by electron microprobe analysis and then neutralized or replaced.

The quantitative evaluation techniques discussed in this report are presently being utilized in the evaluation of commercial and experimental lubricants<sup>(11,12,13)</sup> to be used in isothermal forging of titanium alloys. To date, the use of the evaluation techniques and mathematics developed from them are yielding favorable results and thus show promise of allowing mathematical or computer techniques to be utilized in the selection of particular processing operations.

SECTION VI  
SUMMARY OF IMPORTANT CONSIDERATIONS

Standardized procedures for evaluations of lubricants for isothermal forging have not been adopted. However, several important aspects must be considered and techniques for obtaining quantitative evaluation of these aspects are currently available. A summary of the important considerations for evaluating coating lubricants may be listed as:

1. Controlled ram speeds or strain rates are necessary to differentiate between equipment characteristics, material characteristics and coating characteristics when evaluating coating lubricants.
2. Controlled temperature die systems are essential to distinguish between material characteristics and interface characteristics.
3. Controlled die surfaces are necessary to distinguish between die roughness effects. Clean die surfaces are essential to avoid cross-contamination of the coating being evaluated with those coatings previously evaluated.
4. The use of the ring compression test can allow simultaneous determination of anti-friction effects of coatings and material flow stress effects during testing.
5. The use of a coating lubricant adhesion test allows the adhesion bond strength of the lubricant to the die to be determined. This property will allow die "knock-out" pins to be designed as needed.
6. The use of repeated adhesion tests or similar type of repeated tests can allow the "build-up" of lubricant to be determined.
7. Chemical reactivity of the coating lubricant with the workpiece and with the dies can be assessed quantitatively by metallographic or microprobe techniques.
8. Film continuity of the coating lubricant can be determined by visual examination of the coating after time-temperature exposures typical of those expected in actual operation.
9. Ease of application and removal of the coating lubricants cannot yet be rated quantitatively but must be considered in overall lubricant evaluation.
10. Health factors and cost must also be considered in evaluation of suitable coating lubricants.
11. Chemical compositions, viscosity temperature relationships, wettability measurements and surface energy properties of lubricants are necessary to allow lubricants to be selected on the basis of fundamental properties.



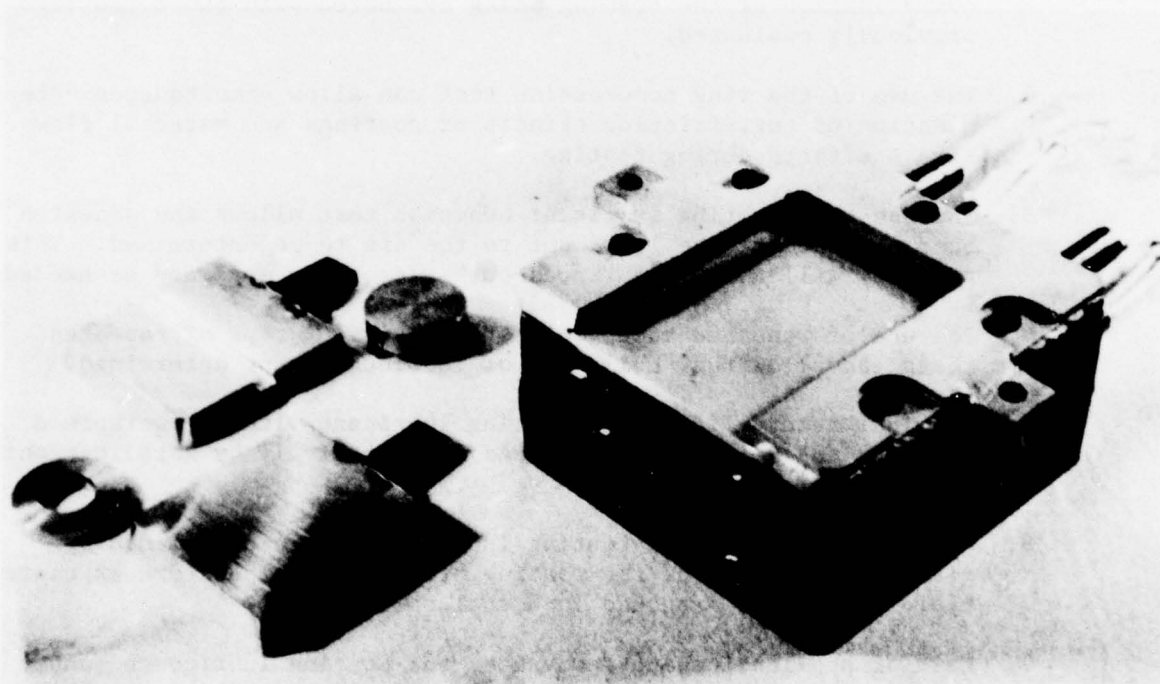


Figure 1. Heated die assembly showing specimens and interchangeable dies for friction and adhesion evaluation.

PROCESS CONDITIONS	COATING CHARACTERISTICS	PERFORMANCE CHARACTERISTICS
<ol style="list-style-type: none"> <li>1. Metallurgy of the Die and Workpiece</li> <li>2. Equipment Characteristics</li> <li>3. Die and Part Geometry</li> <li>4. Temperature</li> <li>5. Environment</li> </ol>	<ol style="list-style-type: none"> <li>1. Chemistry</li> <li>2. Application Technique</li> <li>3. Thickness</li> <li>4. Mechanical Properties</li> </ol>	<ol style="list-style-type: none"> <li>1. Interface Shear</li> <li>2. Part-Die Adhesion</li> <li>3. Die Accumulation</li> <li>4. Reaction Zone Depth (on die and part)</li> </ol>

Figure 2. Schematic illustration of coating characteristics selected so that service performance characteristics can be achieved from controlled processing conditions.

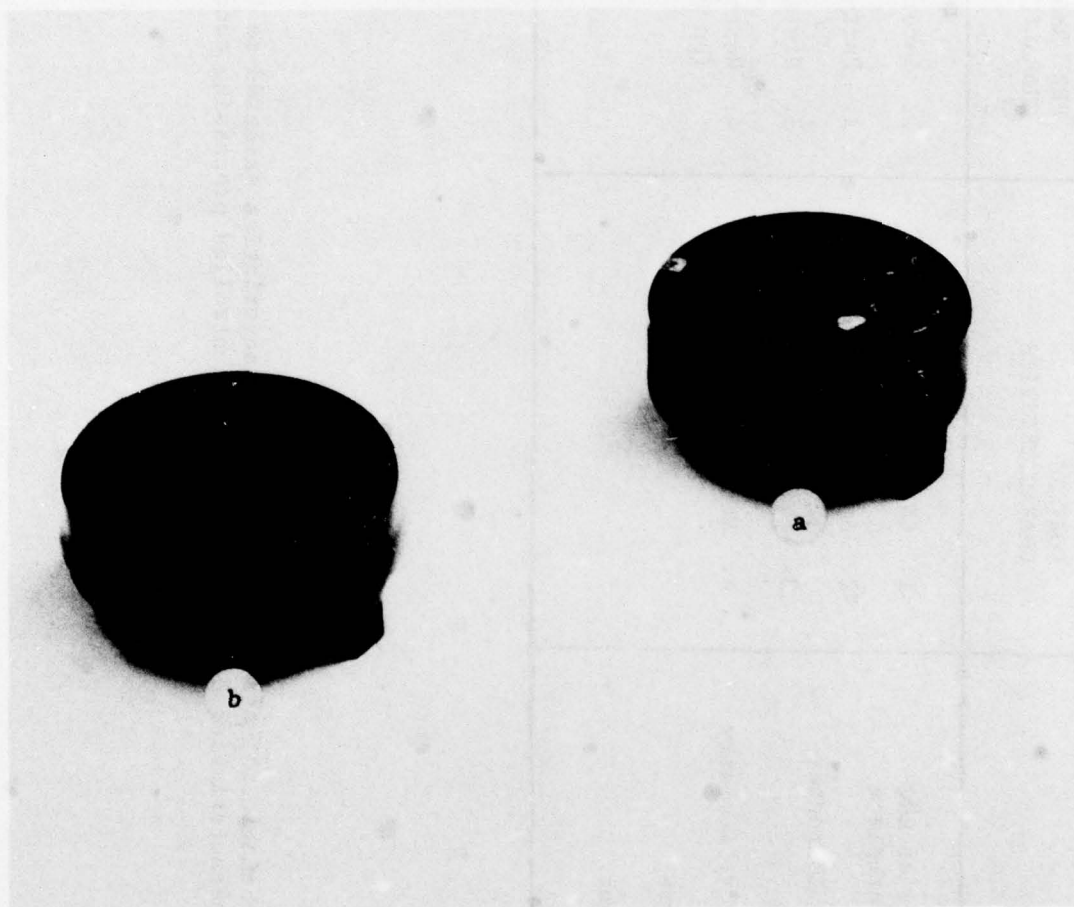


Figure 3. Example of a bad film coating, (a) where ruptured gas bubbles allow the metal to be exposed and a good film coating (b) where a continuous film results.





Figure 4. Titanium galling on nickel superalloy die.

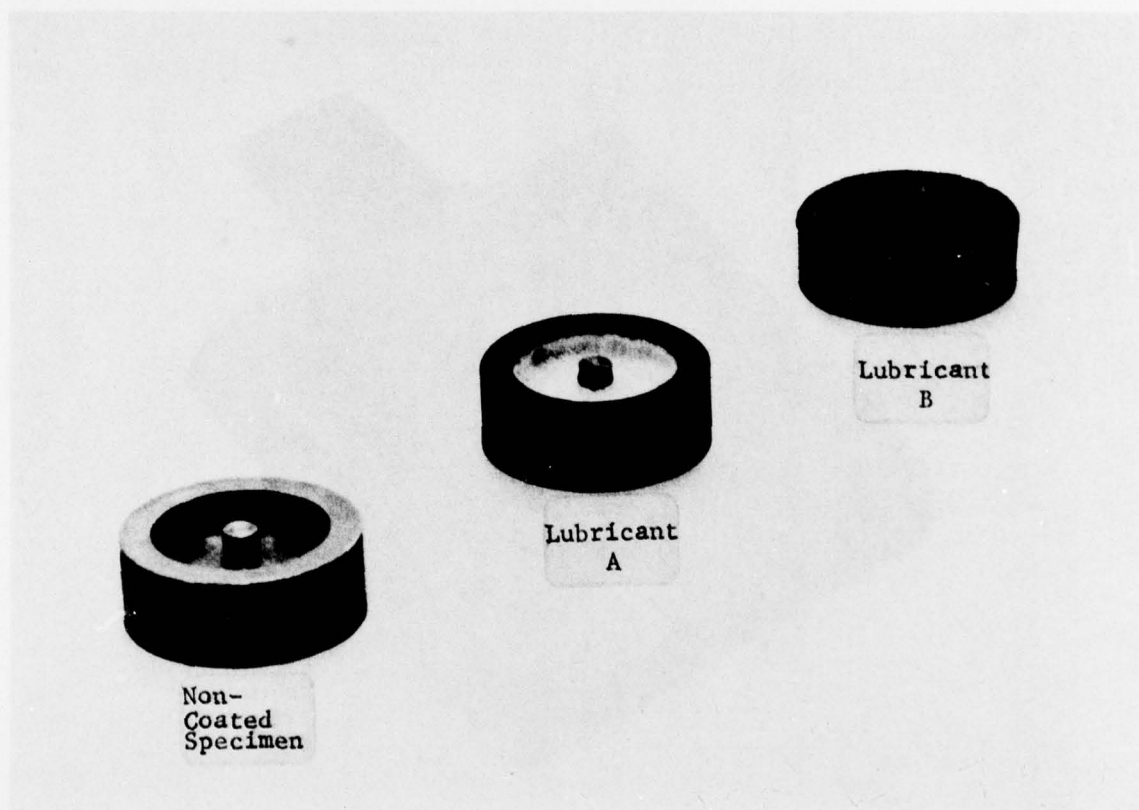


Figure 5. Coating compatibility specimens.

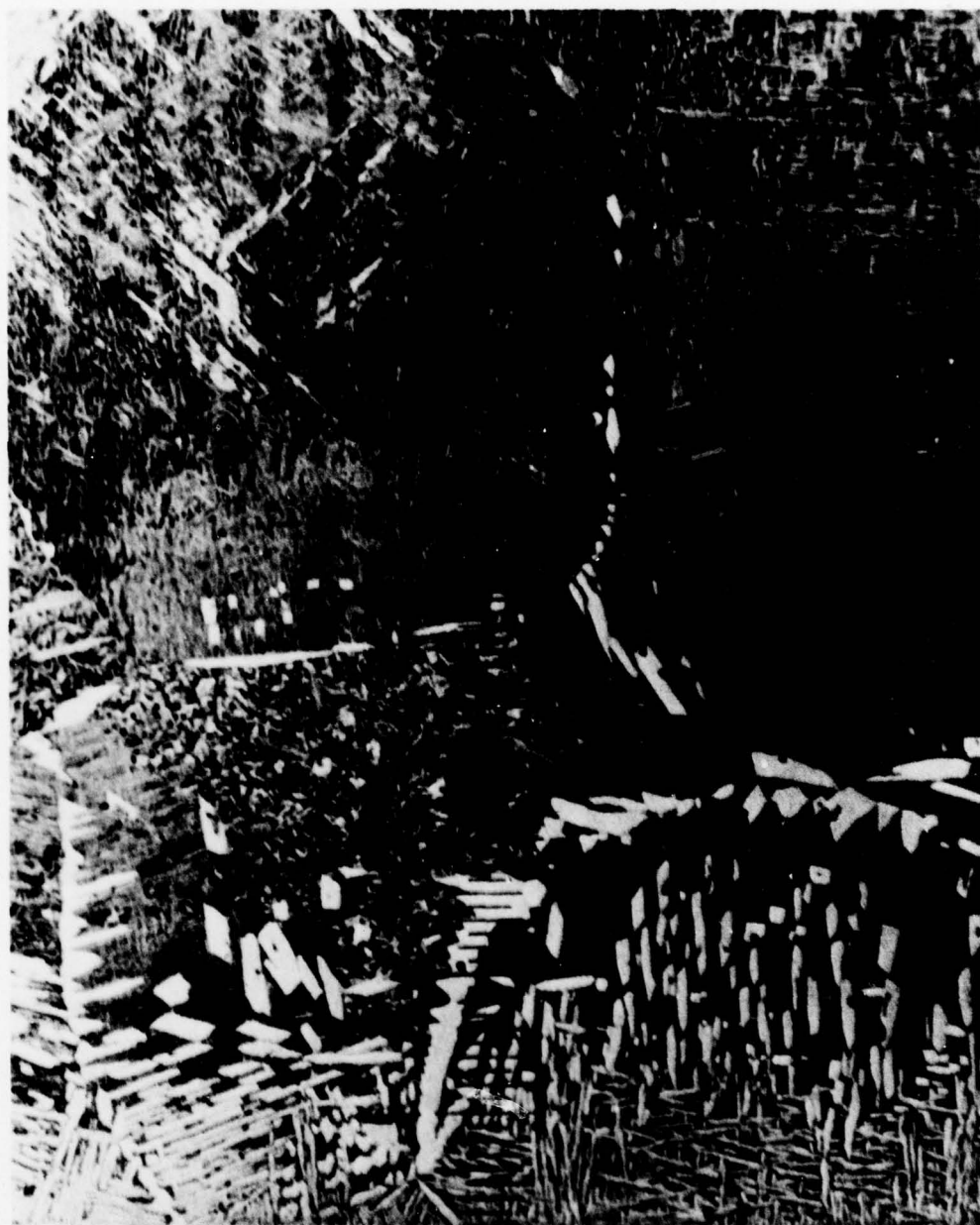


Figure 6. Extended chemical reaction zone resultant from 5 hrs exposure at  $1800^{\circ}\text{F}$  between the coating and beta pre-treated Ti-6Al-4V. Uniform reaction zone occurs to a depth of 0.015 in. and grain boundary alpha occurs to a depth of 0.040 in. Scale graduations are 0.0005 in.



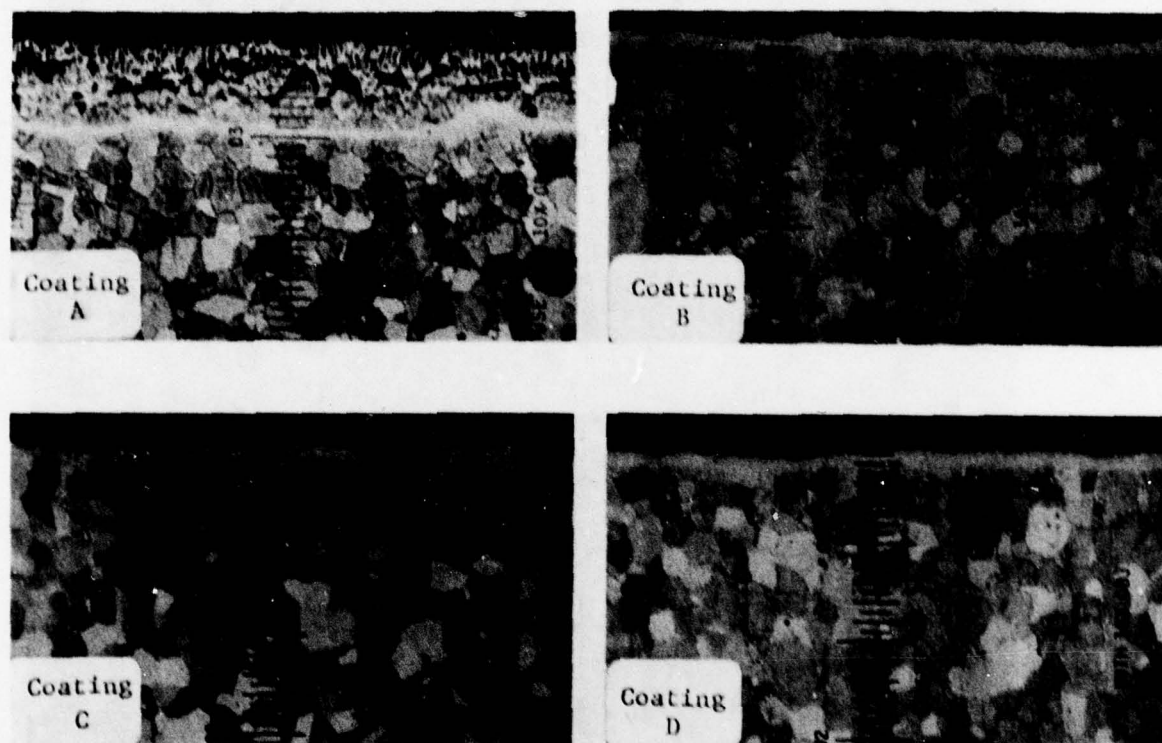


Figure 7. Chemical reaction between various coatings and nickel base 713C die material after 24 hours at 1800°F. Wavy surfaces resultant from Coatings, B, C and D indicate loss of die material through reaction with the glasses, but without the occurrence of porous scale evident with lubricant Coating A. Scale graduations are 0.0005 in.

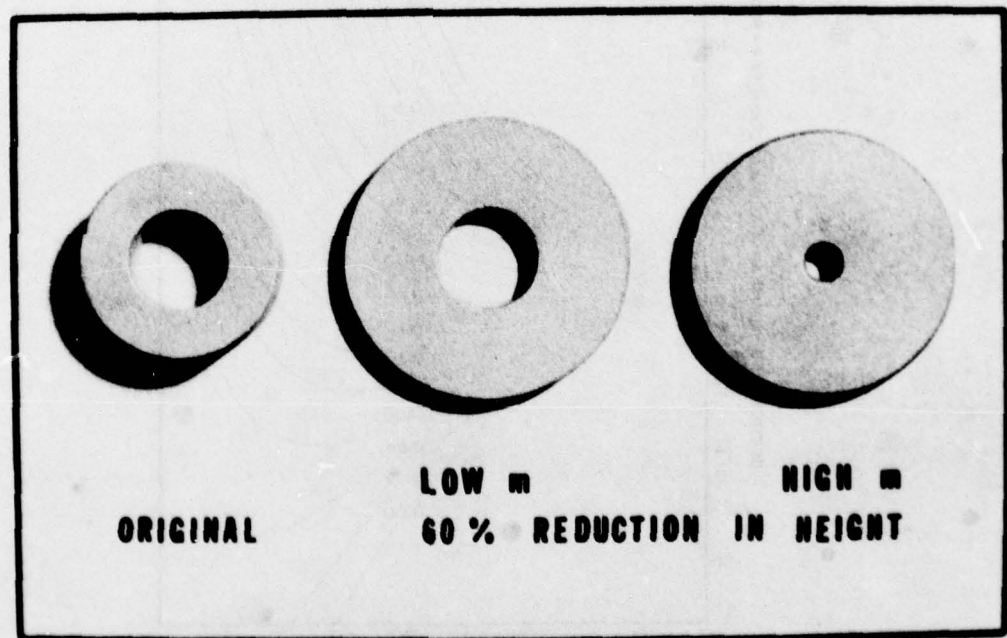


Figure 8. Friction effects on metal flow during ring forging. The parameter " $m$ " is the ratio between the shear strength of the coating and the shear strength of the material.

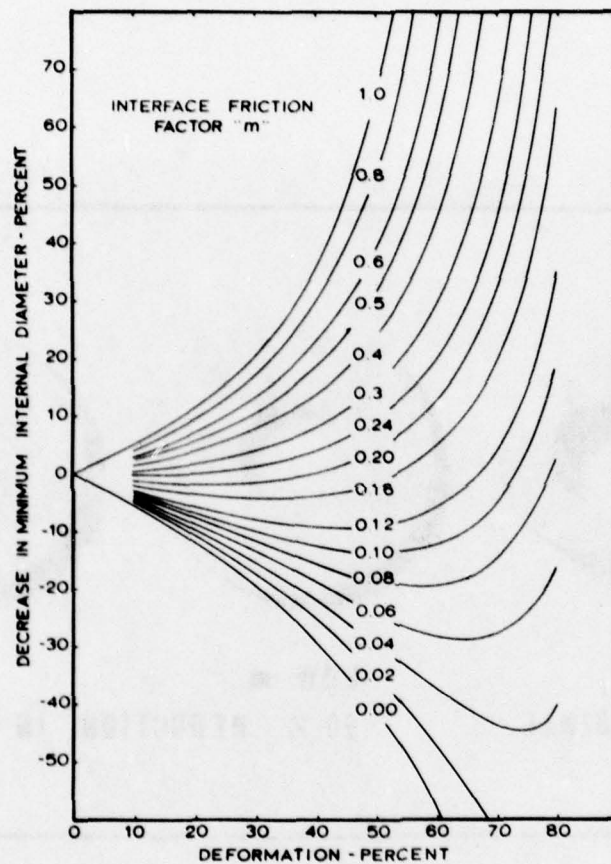


Figure 9. Theoretical calibration curve for interface friction from the ring forging test. Based on 6.3:2 ratio rings with non-varying interface conditions but with consideration for bulge formation<sup>(4)</sup>.



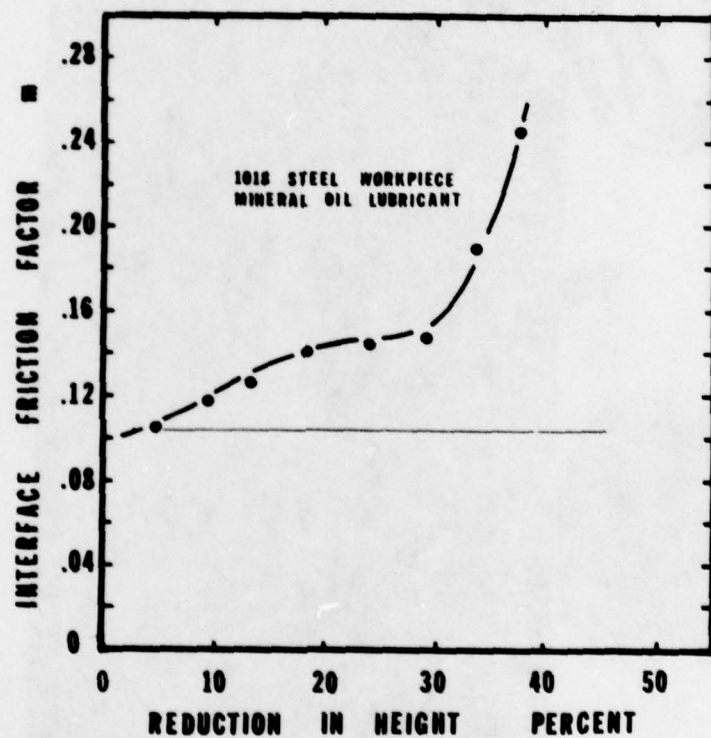


Figure 10. Friction variations showing film break-down at approximately 30% height reduction in forging. Based on analysis of the ring forge test with varying friction conditions<sup>(5)</sup>.

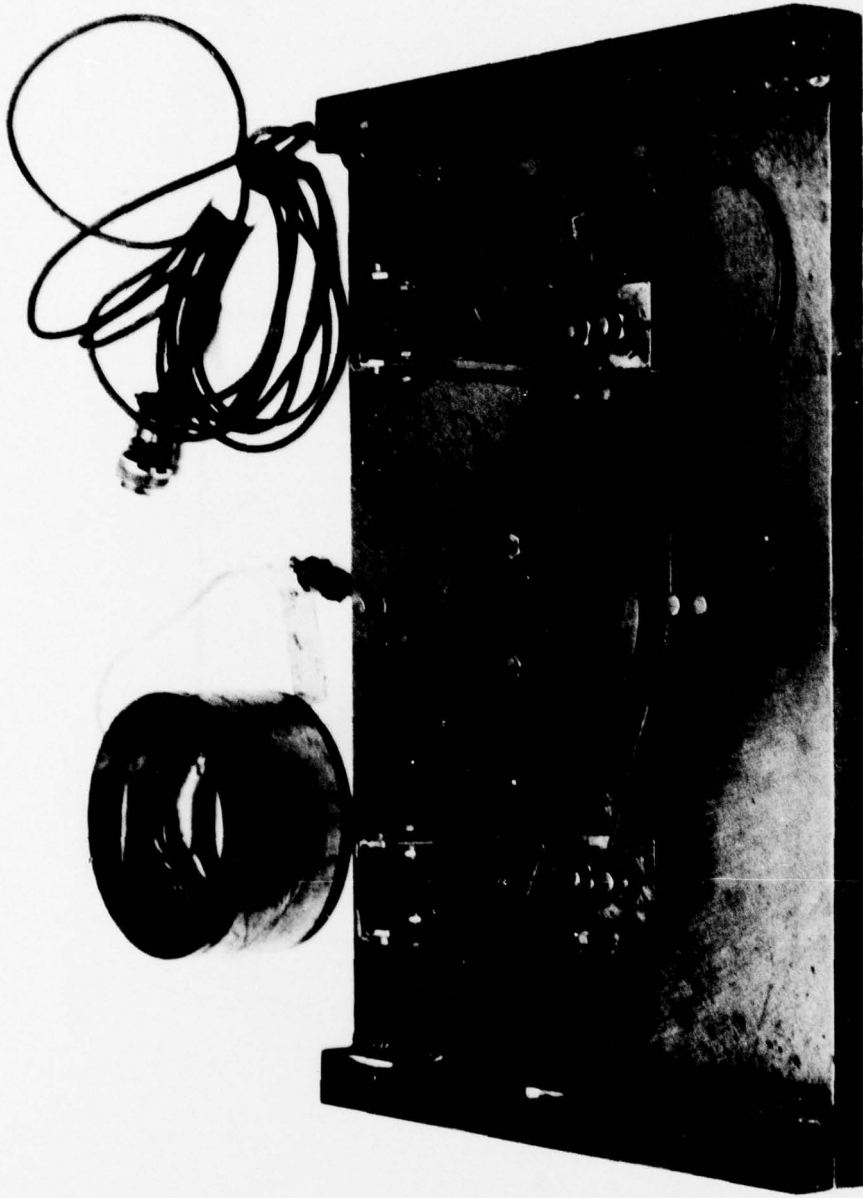


Figure 11. Cylindrical strain gaged load cell for determining deformation load and linear strain gaged strip for determining adhesion loads.

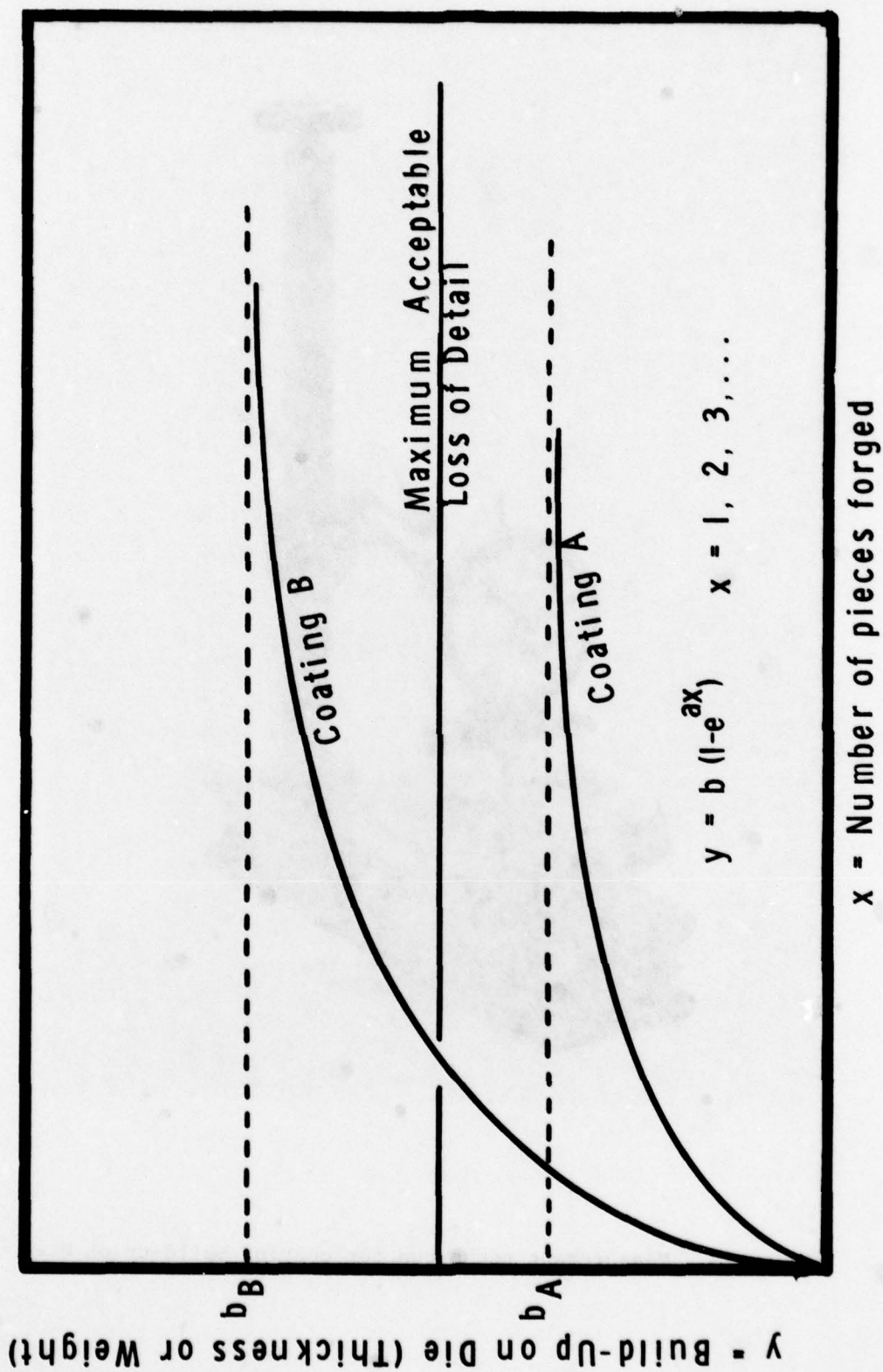


Figure 12. Technique for predicting maximum coating accumulation during a production run.



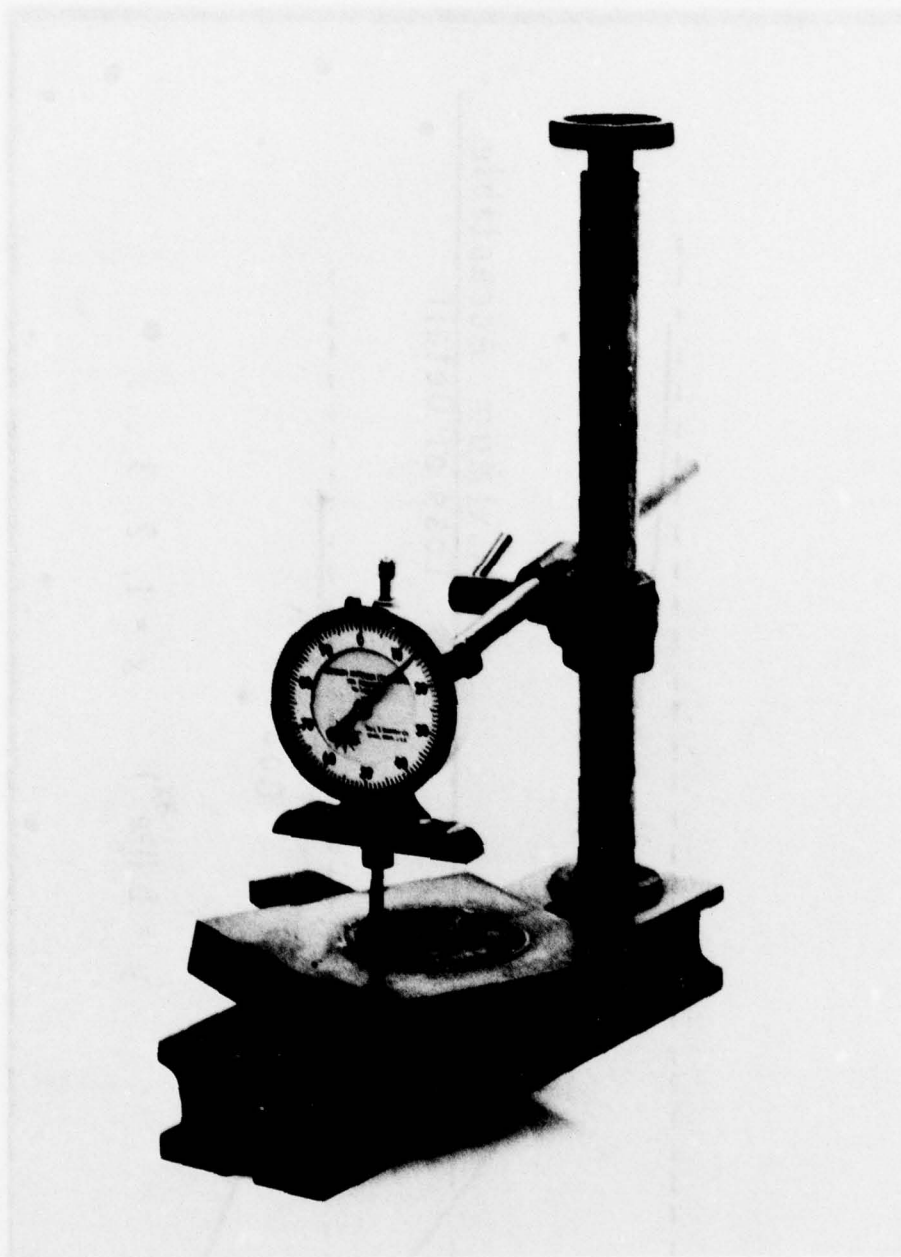


Figure 13. Measurement technique for coating build-up on die.

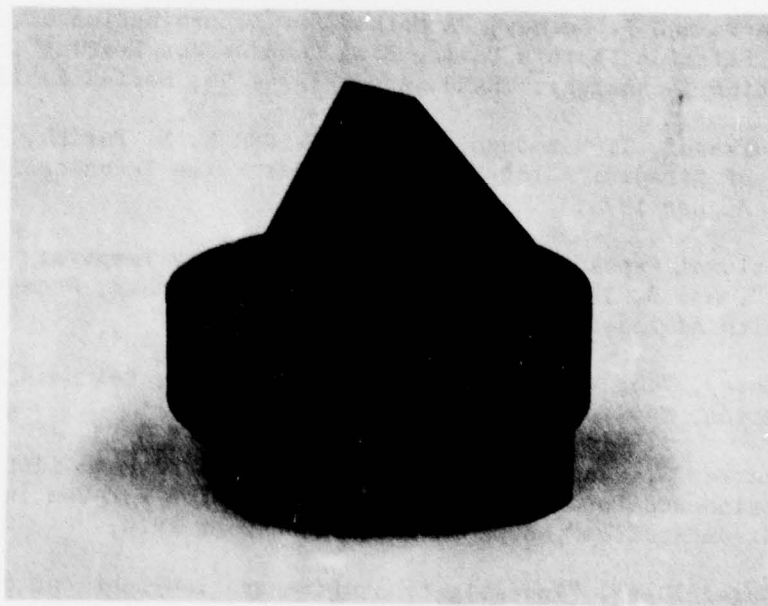


Figure 14. Variation of IITRI<sup>(7)</sup> Roof Truss type lubricant build-up specimen.

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