WADC TECHNICAL REPORT 55-372

THE EFFECT OF SURFACE PREPARATION AND CONDITION ON MICROHARDNESS

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OCTOBER 1955

Materials Laboratory
Contract No. AF 33(616)-2803
Project No. 7360
Task No. 73604

Wright Air Development Center
Air Research and Development Command
United States Air Force
Wright-Patterson Air Force Base, Ohio

FOREWORD

This report was prepared by the Department of Mechanics and Materials of the University of Minnesota under USAF Contract No. AF 33(616)-2803 and covers work performed during the period May to September 1955. The contract was initiated under Project No. 7360, "Materials Analysis and Evaluation Techniques", Task No. 73604 (formerly RDO No. 614-16), "Fatigue Properties of Structural Materials", and was administered under the direction of the Materials Laboratory, Directorate of Research, Wright Air Development Center, with Mr. W. J. Trapp acting as project engineer.

The advice and criticism of Professor B. J. Lazan, who is Head of the Department is greatly appreciated. Acknowledgment is due to Mr. J. Klumpp for proofreading and editorial assistance. Drafting work and manuscript preparation were by B. Gulbrandson, D. Schultz, H. Thomas, and J. Whittier.

ABSTRACT

Observations on the load dependence of the pyramid microhardness are discussed and various explanations of it are briefly reviewed. Experimental results are presented which indicate the effect of the polishing procedure on the hardness-load relationship. A simplified mathematical analysis is presented which shows the relationship between the hardness-load behavior and the stress-strain curve under uniaxial deformation. The various hardness-load trends are explained in terms of strain hardening, effect of the free surface, and cold working introduced by the polishing procedure.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

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SECTION 1. INTRODUCTION

The determination of reliable and meaningful fatigue and other strength properties requires careful attention to specimen preparation details. Particularly critical in the case of fatigue specimens, especially of notched type, is the surface condition. While investigating the effect of surface conditions in fatigue test results, a study of certain aspects of microhardness measurement was undertaken.

Since the very beginning of hardness testing there have been attempts to develop a hardness testing method which is independent of load magnitude. The methods which produce indentations of similar shape, such as cone (1) or pyramid (2), fulfill this requirement for testing loads above approximately 10 kilograms. However, using a diamond pyramid as indenter in the range up to 1 kilogram, the so-called microhardness is very sensitive to the load magnitude. The general trend of the pyramid hardness value as a function of load is indicated in Fig. 1. With increasing test load the hardness increases at first, then decreases, and finally becomes independent of the load. The hardness peak appears, in general, at test loads of 10 to 100 grams. However, the curve shown in Fig. 1 is very sensitive to the type of specimen preparation. Work-hardening due to polishing moves the peak of the curve towards small loads, which in many cases are below the testing range of the instrument. This appears to be the reason why a majority of investigations are concerned with the decrease of hardness with increasing test load 1/. There is a widely held opinion that the load dependence of the microhardness is a deficiency of the method and a number of explanations of it have been suggested which are based primarily on the behavior or deficiencies of the test instrument or the testing procedure. The following analysis indicates that the load-dependence of the microhardness can be a valuable tool for studying surface conditions and the plastic behavior of the test material near the surface.

The various explanations for the load dependence of microhardness can be classified into three categories:

(a) effects due to the testing machine and testing procedure,
(b) effects caused by specimen preparation, and
(c) effects due to the specific mechanism of deformation caused by the indentation.

1.1 Effect of Test Instrument and Test Procedure on the Microhardness Readings

The increase of hardness number with increasing load could be explained by deviations of the indenter from the specified geometry near the tip. Studies by Campbell, Henderson, and Donleavy (3) with three different indenters seem to support this explanation. However, previous investigations with conical indenters (4) indicate that the faults in the geometry of the indenter must be relatively large and definitely visible in order to cause the sometimes relatively large effects. Furthermore, the faults, such as blunt edges, blunt tip or roof shaped tip, can only explain the increase in hardness number and not the decrease with increasing test load.

The same trend of hardness versus test load caused by faults of the indenter can be caused by vibrations (5). Vibrations affect all microhardness testers, to a greater or lesser degree according to their design. The main effect of the vibrations is to cause an uncontrolled overload (22) which is more effective for small loads than for larger ones; thus causing, in most cases, a decrease of hardness with decreasing test load.

1/ In general, hardness values in the constant range at high loads is taken as reference. From this viewpoint there occurs an increase in hardness with decreasing load.

Indentation speed can effect the hardness-load curve in various ways. In certain cases a change in testing speed can reverse the trend entirely (6). The effect of indentation speed can be attributed to a dynamic effect of the machine, causing overload, and the strain rate effect on the yield strength and strain hardening of the test material. The dynamic effect and the relative overload increase with increasing testing speed, whereas a higher indentation speed would cause a relatively smaller indentation due to the strain rate effects in the material (7). The concurrence of these two effects explain why Bergsman(6) observed an increase of the hardness number with increasing test load at high loading speed and the reversed behavior at low loading speed.

Bueckle (8) drew attention to a possible error in the measurement of the indentation due to faults in the aperture of the microscope. According to Tarasov and Tibault (9) many objectives behave as though they had a higher numerical aperture than theoretically determined; thereby causing smaller diagonal readings. The errors can amount to 1 micron and could cause an apparent decrease of hardness with increasing test load.

Friction between indenter and test specimen could be a cause of the load dependence of microhardness. Bischöf and Wenderott (10) tried to reduce the effect of friction by repeated loading and unloading into the same indentation, and found a considerable decrease of hardness at small loads. Another way of reducing friction is to use a lubricant. This usually results in a decrease of hardness. Investigations by the authors, discussed later, show that the use of a lubricant decreases the hardness, but does not eliminate the load dependence of microhardness.

1.2 Effect of Specimen Preparation Procedure on Microhardness

There is evidence that the polishing methods commonly used for the preparation of test specimens introduced cold working and hardening of the material in the vicinity of the surface to a depth of one hundred microns or more. The zone affected by the hardness indentation is approximately of the same order of magnitude as the diagonal of the indentation. The cold worked zone introduced by polishing causes an increase of hardness with decreasing test load. The polishing effect was studied by Bueckle (8) who also lists a great number of references on this subject. Figure 2 shows a diagram, published by Bueckle, which illustrates the effect of various polishing procedures on microhardness. It is interesting to note that the hardness on cleavage planes (7) or of grown surfaces (11) shows no such cold working effect and that the hardness on such surfaces increases at first and then approaches a constant value. A similar behavior was found for hardened and drawn steel by Campbell et al. (3) by etching the test specimen between each polishing step.

1.3 Load Dependence of Microhardness As Related to the Mechanism of Deformation

Schulz and Hanemann (12) correlated their test results by an exponential function of the form

$$P = ad^n \tag{1}$$

where P is the test load, d is the diagonal of the indentation, and a and n are constants. This relationship, Eq. (1), is the same as that established for the Brinell hardness test by E. Meyer (13). An exponent n of 2 indicates load independent hardness, an exponent n smaller than 2 expresses a decreasing hardness with increasing test load, an exponent n larger than 2 means that the hardness increases with increasing test load. Some investigators thought that n is a material constant and should be listed together with the hardness number. However, a more intensive study indicates that the exponent n is a function of the magnitude of the test load (7), (8), (12) and is primarily affected by the preparation of the surface (8, 14, 15). In Figs. 3 through 8, some of the test data of several investigations are replotted and shown together with the test results. The diagrams indicate that undisturbed surfaces give an increase of hardness with increasing test load up to a certain magnitude of the indentation after which the hardness remains constant. According to this behavior the exponent n is greater than two at small loads and decreases asymptotically to a value of 2 with increasing test load. Cold working of the surface introduces a maximum in the hardness-load curve and a range of decreasing hardness. Accordingly the exponent n decreases from a value greater than 2 to a value smaller than 2 and then increases asymptotically to 2. In extreme cases of cold working, the initial stage of increasing hardness is entirely missing and the exponent n is smaller than two. An interesting trend of the exponent n can be observed in Fig. 3 for aluminum and magnesium. There the n-P curve has a maximum at intermediate loads.

One meets the general opinion that due to similar indentations the pyramid hardness should be independent of the load magnitude. This statement is based on Kick's law of similarity (16) which states that the work to cause equal degrees of deformation of geometrically similar and materially identical bodies is proportional to the volume of the weights of the bodies. This means that the work of deformation should be proportional to any linear dimension of the deformed body. In the case of pyramid hardness testing, the work of deformation is assumed to be proportional to the third power of the diagonal of the indentation. Since microhardness is not independent of the load, it has been concluded that Kick's law of similarity does not hold for hardness indentations. However, the application of Kick's law as described above implies the assumption that the volume of deformation is proportional to the penetrated volume, a relationship which has not been proved. On the other hand, if we assume Kick's law to be valid, then we conclude that the volume taking part in the deformation is not proportional to the volume of indentation (17). The assumption of load independent hardness for indenters which cause similar indentations holds only for continuous and homogeneous media. The surface as a discontinuity and the structure of the material in the microrange can be considered as inhomogeneities. Therefore, hardness becomes load independent only when the affected volume becomes large enough to have the material behave as a quasicontinuous and homogeneous medium.

One of the explanations for the load dependence of microhardness is based on the elastic recovery of the indentation after unloading (9, 18, 19). It is assumed that the elastic recovery is independent of the magnitude of the indentation and therefore has a larger effect for smaller indentations.
Equation (1) then must be rewritten

$$P = a(d+c)^2$$

where c is the constant elastic recovery.
This mechanism can explain only decreasing hardness with increasing load. Various attempts to apply Eq. (2) indicate that c must be a variable in order to fit the data for load independent hardness. Several attempts have been made to measure the elastic recovery. The results of various investigations, however, seem to indicate that elastic recovery is very small, or at least too small to explain the observed load dependence (7) (8).

In an analysis of the mechanism of deformation during the indentation Bernhardt (7) assumes that the total energy of deformation consists of the energy of translation and the energy for the formation of the new surface. The translation energy is assumed to be proportional to the volume of the indentation ($P = ad^2$); whereas the energy for the formation of the new surface should follow Rittinger's law, which was originally established for crushing, and be proportional to the formed surface $(P = bd)$. The combination of these two expressions gives the following relationship between the testing load and the diagonal of the indentation

$$P = ad^2 + bd \tag{3}$$

Braun (20) combines elastic recovery with strain-hardening, arriving at an equation of the form

$$P = ad^m (d + c)^{2-m} \tag{4}$$

The parameter m is determined from the strain hardening characteristics of the material. Equation (4) is the most flexible one. However, in order to fit a curve as that shown in Fig. 1, m must be assumed to be a function of the load P and not a constant.

Bueckle (8) used interferometric methods for studying the formation of the bulge around the indentation. He proved that the deviation of the shape of the indentation from the geometric square form is caused by the edges cutting into the bulge, and not by elastic recovery. Bueckle found that the height of the bulge above the original surface is not a linear function of the diagonal of the indentation. The height of the bulge increases slowly at first and then more rapidly with increasing size of the indentation. Bueckle connects this change in the height of the bulge with the load dependence of microhardness. He arrives at the conclusion that the formation of a bulge is the primary cause of the load dependence and shows that if the area of the indentation is computed from the original surface (by subtraction of the additional area due to the bulge) the hardness values are independent of the load. However, it is difficult to understand why the bulge does not contribute in carrying the test load, especially since the bulge of the extruded material is very flat. On the other hand it seems plausible that any deviation of the shape of the indentation from the prescribed form due to the special shape of the bulge can affect the microhardness readings.

SECTION II. EXPERIMENTS ON LOAD DEPENDENCE OF MICROHARDNESS

For the practical application of microhardness testing the load dependence of hardness data is of significant importance. It is possible to find hardness differences of one hundred percent occurring, and as the literature survey indicates, the reason for it is not well understood at present. Many contradictory results have been reported. In most cases a decrease of hardness with increasing test load has been observed. Several investigators (3), (7), (9), (11), (17), (21), (23), however, found an increase of hardness at very small load and then a decrease in hardness with increasing test load, the hardness approaching a constant value for high loads. Such a trend is shown in Fig. 1. This variation in hardness with load makes it difficult to compare results obtained by different investigators or in different laboratories (24). For this reason it seems necessary to study in each separate case the load dependence of microhardness for the specific hardness testing instrument available and the specimen preparation procedure applied. Only then can conclusions be drawn from the test data with any real confidence in their validity.

For the present investigation a Kentron Microhardness tester, manufactured by the Torsion Balance Company was used. This instrument has a wide loading range, from 1 to 10,000 grams, of which only the range from 1 to 1000 grams was used in this investigation. It is of the weight balance type similar to the Tukon hardness tester which was also used in the test program for comparison purposes. For measurements of the indentations, a Bausch and Lomb turret microscope with lens magnifications of 125 x, 250 x, and 562 x is mounted on the instrument. This permits selection of the proper magnification; an item which is especially important for the readings of small indentations at small loads. In order to make sure that the observed load dependence of the hardness readings are not caused by the instrument, the load calibration and the calibration of the micrometer eyepiece with the particular light source used were accurately checked. Furthermore, two indenters, each of slightly different geometry, have been used. Both, however, gave coincident results. In order to reduce the effects of vibrations, the instrument was mounted on a shock absorbent base and readings were taken at times of the day when vibrations or shocks were at the minimum. The loading speed on the hardness readings was studied within a range of 0.5 to 7mm per minute. The results obtained were similar to those observed by Bernhard (7), e.g. a slight decrease of hardness with decreasing testing speed, which was more pronounced at smaller loads. No reversal of the trend of load dependence due to change of loading speed, such as observed by Bergsmann (6), could be detected.

The test materials used in this investigation were: aluminum single crystal, copper, mild steel with approximately 0.1 percent carbon content, Rockwell-hardness test block, coarse grained zinc, cadmium, and tin. Table I lists the annealing treatment for the test materials and the polishing procedures for each material. All test specimens were polished on emery paper under intermediate etching in order to avoid as much cold working as possible. For the final polishing step three different procedures were applied: polishing with AB Alpha Alumina, polishing with Elgin Dymo diamond compound grade 1 - medium, and electrolytic polishing. All specimens were tested in the etched condition. The etchants applied are listed in Table I. The test loads applied were generally 1, 5, 10, 25, 50, 100, and 1000 grams. In certain cases tests were performed with loads at every whole number of grams in the range from 1 to 10 grams. The data plotted in Figs. 9 through 13 are arithmetic means of 10 to 20 indentations with the same load.

In several cases a solution of 2 percent oleic acid in vaseline oil, as used by Rehbinder (25) for studying surface effects in machining and fatigue, was applied. In his investigations, Rehbinder detected a marked effect of this solution (surface active substance) on the type and magnitude of deformation introduced by machining and other deformation processes. He found that oleic acid increases the rate of plastic flow five to ten times. The original thought in using this solution for the hardness test was that probably the "Rehbinder-effect" and not the lubrication causes the decrease in hardness. However, creep tests performed by the authors using coarse grained copper specimens did not show any effect of Rehbinder's solution on the creep rate. On the other hand, an application of the solution caused a marked decrease of microhardness. Because of the negative result of the creep test, the reduction of hardness by oleic acid is thought to be caused by a reduction in the friction between specimen and the indenter. Gogoberidze, Kopatsii, and Sakhov (26) arrived at a similar conclusion.

The test results diagramed in Figs. 9 through 13 indicate the same trend as shown in Fig. 1, an increase of hardness with increasing test load and either an asymptotic approach of hardness to a constant value or a maximum of hardness with a slight decrease following. The polishing procedure has a marked effect on the hardness readings; it can be related to the magnitude of cold working introduced. According to these results electrolytic polishing gives the smallest hardness readings, diamond polishing caused somewhat higher hardness at small loads, and polishing with aluminum oxide caused the highest hardness. The difference of hardness caused by the various polishing methods decreases with increasing test loads and is practically negligible at very high loads. It is interesting to note that aluminum oxide polishing not only produces the maximum in some cases, but also moves the maximum, when already present with the other procedures (Fig. 4) to smaller loads. This trend is more pronounced in the exponent n load diagram, which will be discussed more fully in the following sections.

The most important factor in the preparation of specimens seems to be that all the cold-worked, or affected, material must be removed by the following polishing steps in order to obtain a sound surface. In many cases this requirement is not met even by the metallographic polishing procedures where polishing is continued only until the scratches of the previous step have been removed. This criterion may not be sufficient for the removal of the cold-worked material introduced by the first step for the preparation of the surface.

SECTION III. EFFECT OF STRAIN HARDENING ON THE HARDNESS LOAD RELATIONSHIP

A possible method of analysis of the hardness-load relationship was shown by Sachs (27), on which the following derivations are based. According to Ludwik (28) it is possible to relate shearing stress, τ, with compressive strain. In a first approximation the normal strain due to a shear stress can be taken from the compressive stress-strain diagram for $\sigma = 2\tau$. Nadai (29) showed that the maximum shear stress in the elastic range, due to a uniformly distributed compressive stress p on the surface, as a function of the distance from the surface, is as shown in Fig. 14.

$$\tau_{max} = \frac{p}{\pi} \sin \frac{\alpha}{2} \tag{5}$$

The depth of indentation v of a punch is according to the relationship indicated by Figs. 14 and 15.

$$v = \int_{r=0}^{r=\infty} \epsilon \, dr \tag{6}$$

or with

$$r = (b/2) \ \text{ctg} \ \frac{\alpha}{2} \tag{7}$$

$$v = \frac{b}{4} \int_{\pi}^{0} \epsilon \, (\sin^2 \alpha/2) \, d\alpha \tag{8}$$

Sachs evaluated equation (8) by applying Simpson's rule and found good agreement with experimental results, as shown in Fig. 16. This indicates that the changes of stress distribution due to plastic deformation occur relatively slowly.

In order to obtain an expression for the depth of indentation v as a function of stress (or load p) in closed form, the compressive stress-strain curve can be approximated by

$$\epsilon = C_1 \sigma^m \tag{9}$$

where C_1 and m are material constants.
Substituting Eq. (9) into Eq. (8) gives

$$v = \frac{b}{4} C_2 \left(\frac{p}{\pi}\right)^m \int_{\pi}^{0} \sin^{m-2} \frac{\alpha}{2} \, d\alpha \tag{10}$$

with $C_2 = C_1 2^m$

The evaluation of the integral gives

$$v = C_4 p^m \tag{11}$$

with $C = (b C_1 2^m)/(4\pi^{m-1})$ for even m,

or $C_4 = (bC_1 2^m)/(2\pi^m)$ for odd m.

The exponent m varies between 1 and 5 according to the structure of the material. For cubic face centered polycrystalline metals m is close to two and for cubic body centered metals m is four or five. The test results published by Sachs can be satisfactorily represented by Eq. (11) using $m = 2$ and $C_1 = 9.7 \times 10^{-4}$ as shown by Fig. 16.

The analysis presented above is only a first approximation. However, the good agreement between Eq. (11) and the test data justifies the simplified procedure. Fig. 17 shows a diagram derived from Eqs. (5) and (9), of the strain as a function of the distance from the surface for various values of p. Eq. (11) can now be applied to determine the relationship between load P and the magnitude of the indentation of the Vickers-pyramid.

With

$$P = \int_{0}^{A_0} p \, dA \tag{12}$$

and Eq. (11) we obtain for the particular geometry of the pyramidal indenter

$$P = C_5 a^{2+\frac{1}{m}} \tag{13}$$

where

$$C_5 = (2.47 C_4)^{-\frac{1}{m}} \left(\frac{m}{1+m} - \frac{m}{1+2m}\right) \Big/ \left(2^{2+\frac{1}{m}}\right)$$

Eq. (13) is a special form of the empirical exponential relationship, Eq. (1), with $n = 2 + \frac{1}{m}$. This shows that the relationship Eq. (1), as proposed by Haneman and Meyer respectively, also has a physical meaning for the pyramid indentation hardness (8).

According to the value for the strain hardening coefficient m, as listed above, the exponent n should vary between the values 3 and 2.2, indicating that the hardness increases with increasing test load. Constant hardness, independent of load, requires an exponent $m = \infty$ which expresses the stress-strain curve of an ideal plastic material. In order to obtain an exponent n smaller than 2 the exponent m must be negative, which causes a hyperbolic stress-strain relationship indicating "strain softening" instead of strain hardening.

The diagrams Figs. 9 through 13 show that the exponent n depends on the test load and is not constant as assumed in the above derivation for a homogeneous material. This behavior is due to the inhomogeneity of the material in the vicinity of the surface, as discussed in the next section, and cold working effects caused by the specimen preparation. However, it is interesting to note that the exponent n for the electrolytically polished cubic face centered aluminum and copper starts from a value of approximately 2.5, resulting in a strain hardening exponent m of 2; whereas the cubic body centered ferritic steel gives $n = 2.2$ with $m = 5$; and the hexagonal zinc gives $n = 3$ with $m = 1$, which corresponds with the strain hardening coefficient of the single crystal. The n values have been determined independently of the above derivation by one of the authors.

The diagrams show, furthermore, that cold working due to polishing not only increases the n values for small loads, but also can considerably affect the trend as exhibited by the cadmium specimen, Fig. 11.

The analysis presented above can be used to study the effect of geometrical faults of the indenter, such as a missing tip, on the hardness readings. It is assumed that the indenter has the shape of a frustum of a pyramid, and the area of the flat square top is the fraction ψ of the area of indentation:

$$A_k = \psi a^2 \tag{14}$$

combining Eqs. (14) and (13) results in an expression for the ratio of hardness obtained with the faulty indenter H_f to that obtained with a geometrically perfect indenter H_o:

$$\frac{H_f}{H_o} = \frac{2\left[\frac{m}{1+m}\left(1-\sqrt{\psi}\right)^{\frac{1+m}{m}} - \frac{m}{1+2m}\left(1-\sqrt{\psi}\right)^{\frac{1+2m}{m}}\right] + \psi\left(1-\sqrt{\psi}\right)^{\frac{1}{m}}}{2\left[\frac{m}{1+m} - \frac{m}{1+2m}\right]} \tag{15}$$

A graphical representation of Eq. (15) is shown in Fig. 18 for various exponents m. The diagram indicates that the effect of geometric faults on hardness decreases with increasing strain hardening coefficient m and is zero in case of an ideal plastic material with $m = \infty$. An evaluation of the test results by Eq. (14), presented in Figs. 9 through 13, indicates that more than 90 percent of the tip of the pyramid must be missing in order to explain the hardness increase with increasing test load. Obviously such large deviations in the geometry have never been observed.

SECTION IV. LOAD DEPENDENCE OF HARDNESS AS RELATED TO THE STRUCTURE OF THE MATERIAL NEAR THE SURFACE AND THE MECHANISM OF DEFORMATION CAUSED BY INDENTATION.

In order to study the mechanism of deformation during small indentations, it is necessary to consider the free surface as a discontinuity which has a basic effect on the structure and plastic behavior of the material near the surface. It is not only the cold working or the chemical changes introduced by polishing of the surface that cause these changes in material behavior. A grain at the surface behaves plastically in a different way than a grain in the interior which is surrounded by other grains of different orientation.

Due to extremely localized application of the load and the resulting stress gradient, even a single grain behaves inhomogeneously with respect to plastic properties. The mechanism of deformation is affected by the free surface insofar as the parts near the surface can freely extrude out of the surface,whereas parts further inside are elastically supported thereby displaying a higher yield strength. From this viewpoint the local yield strength is a function of the distance from the free surface, increasing rapidly at first and asymptotically approaching a limiting value with increasing distance. This effect would tend to cause a more rapid increase of hardness with increasing test load than that computed from the stress-strain diagram obtained under uniform load application. Such an effect of the free surface in glass was reported by Klemm and Smekal (30).

A similar effect of free surface in polycrystalline material results from the difference in crystallographic orientation between the grains. In metal grains plastic deformation occurs due to slip along definite crystallographic planes. In polycrystalline materials the deformation of each crystallite is hindered by the influence of its neighboring crystallites. The greater the deviation in crystallographic orientation between two grains, the greater is their mutual resistance to flow (31). This hindering, or restraining effect decreases exponentially with increasing distance from the grain boundary into the grain; thus causing a smaller effect on the core of the grain. The magnitude of this effect is a function of the grain size (32), (33), and (34). The smaller the grain, the higher its average strength (35). The total hindering effect of the environment surrounding any crystal upon its deformation can thus be estimated by the summation of the effects of a finite number of successive surrounding spherical layers (36). For crystallites near a free surface, a part of this spherical field of influence is missing, and crystallites at the surface are affected only by a hemispherical field. Since these grains are less hindered to deform because they have fewer neighbors, they respond plastically under stress which is locally smaller than that required to deform the grains in the interior of the body.

The magnitude of the restraining effect described above depends primarily on the crystal system, i. e. the number of glide possibilities, and is therefore greatest in hexagonal, smaller in cubic body centered, and least in cubic face centered metals. This restraining effect explains the difference of the mean stress-strain behavior of the single crystal and the polycrystal as shown in Fig. 19.

This effect of the free surface explains both the initial increase in hardness approaching a maximum and the following decrease which is introduced by cold working due to polishing, as shown by the foregoing experimental results. Figure 20 illustrates the effect of cold working introduced by polishing procedure on the local plastic yield strength. The local yield strength of the undeformed material increases rapidly at first with increasing distance from the surface and then approaches a constant mean value. Due to the increasing restraint also strain hardening increases with increasing distance from the surface. Superimposed on these variables is the nonuniform deformation caused by machining or polishing. The superposition of these three variables can produce a maximum of yield strength at a certain depth as illustrated by Fig. 20. The surface effect on the strain hardening behavior also explains why microhardness at small loads and scratch hardness are relatively insensitive to strain hardening of the specimen caused by unidirectional deformation.

In the diagrams of Figs. 9 and 13, the loads P are indicated at which the diagonal of the indentation is equal to the mean grain size. The diagrams show that the initial increase of hardness with increasing test load and the maximum occurs for indentations smaller than the grain size, thus indicating the effect of grain size on the load dependence of microhardness.

The general trend of the n value as a function of the test load can be explained by the mutual restraining effect of the grains which acts far into the enveloped grain, and the effect of the free surface. In sound specimens, such as electrolytically polished ones, the exponent n corresponds to the theoretically determined one as long as the indentations are small compared with the grain size. With increasing magnitude of indentation the interaction of surrounding grains becomes effective and the exponent n increases according to the transition of the strength or hardness of the weaker surface crystal (partly restrained single crystal) to that of the polycrystal. When the indentation exceeds the size of a single grain, the n value decreases to its original value, and finally the further deformation occurs according to Kick's law and the hardness approaches a constant value.

In case of single crystals the restraining effect of surrounding grains is missing and the n value decreases continuously with increasing load.

SECTION V. SUMMARY AND CONCLUSIONS

The pyramid indentation hardness, the so-called Vickers Hardness, is independent of the test load above loads of approximately five to ten kilograms. Microhardness, however, in the load range up to 1000 grams, is a function of the magnitude of the test load. In most cases a continuous decrease of microhardness with increasing load has been reported. Several other investigators, however, have observed an initial increase of microhardness with increasing load which is followed by a range in which the hardness becomes either independent of load or decreases continuously to a constant value. Various explanations of the above have been offered; such as deficiencies of the testing machine or the testing procedure (faults in the geometry of the indenter, vibrations, faults in the aperture of the microscope, friction between indenter and specimen), effects caused by specimen preparation (cold working, chemical effects), and effects due to the specific mechanism of the indentation (elastic recovery, formation of a bulge, formation of a new surface). The load dependence of microhardness is difficult to reproduce and makes it difficult to compare the data of different investigators.

In order to study the load dependence, hardness tests with loads of 1, 5, 10, 25, 50, 100, and 1000 grams on specimens of copper, aluminum single crystal, mild steel, high-alloy steel, zinc, cadmium and tin were performed by the authors. The specimens were prepared by three different methods: electrolytic polishing, mechanical polishing using diamond compound, and polishing with alumina. The tests indicate an initial increase of hardness with increasing test load and then a slight decrease after reaching a maximum value for polycrystals. In electrolytically polished single crystals (aluminum) an asymptotic increase to a constant value was observed.

Haneman proposed a relationship between load and diagonal of the indentation similar to that of Meyer for the Brinell hardness, which is of the form $P = ad^n$. An analysis of the relationship of uniaxial deformation and hardness indentation presented in this paper gives physical meaning to the above empirical equation if the exponent $n = 2 + \frac{1}{m}$, where m is the exponent in the parabolic approximation of the stress-strain curve $\epsilon = C\sigma^m$. This relationship explains the increase of hardness with increasing load due to strain hardening.

Another factor to be considered is the effect of the free surface on the local yield properties of the material in the vicinity of the surface. In a polycrystalline material the slip planes of the grains do not coincide. Therefore, the grains hinder each other in regard to plastic deformation and this is the general explanation for the higher strength of a polycrystal with respect to that of a single crystal. Crystals on the free surface of a specimen are surrounded by a smaller number of grains than those inside the material. Therefore, surface grains are weaker and deform at average stresses which are below the nominal yield strength of the material. These effects contribute to the initial increase of microhardness with increasing depth of indentation, or testing load, and are explained by the transition from a single crystal action to the polycrystal action.

Internal stress may also affect the load dependence of microhardness. The effect of internal stress on microhardness is currently under study.

The results of this investigation may be summarized as follows:

1. In electrolytically polished single crystals or on cleavage planes of a single crystal the microhardness increases asymptotically to a constant value with increasing test load.

2. In electrolytically polished polycrystals hardness increases at first and after reaching a maximum decreases slightly to a constant value. The test load at which the maximum occurs is a function of the grain size.

3. Cold working introduced by manual polishing moves the hardness maximum to smaller loads and considerably reduces the initial hardness increase.

4. In preparation of a test specimen it is important to remove the entire cold worked or otherwise affected zone introduced by previous preparation or polishing steps. The removal of scratches is not a sufficient criterion.

5. The initial increase of hardness with increasing load can be attributed to strain hardening and the effect of the free surface on the local yield strength.

6. The exponent n in the relationship $P = ad^n$ starts from the theoretical value derived from the stress-strain relationship under uniaxial load, then increases for polycrystalline materials according to the transition from the yield strength of the single crystal to that of the polycrystal, then decreases again below a value of two and finally approaches asymptotically a value of two. For a single crystal the exponent n decreases from the theoretical value asymptotically to a value of two.

7. Application of a solution of oleic acid in vaseline oil reduces the hardness at small loads considerably. This reduction is primarily due to the reduction of friction between indenter and test specimen.

8. Load dependence of microhardness and its sensitivity to the specimen preparation procedure is not considered to be a deficiency of the testing method. It can be a valuable tool in many engineering applications for studying the condition of the material in the vicinity of the surface.

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TABLE I. TEST MATERIAL AND TREATMENT

Material	Annealing Temperature And Time	Polishing Procedure	Etchant	Grain Size
Copper	500° F for One Hour	a) Electrolytic	Ferric Chloride Sol. $FeCl_3$ 5 g HCl 10 ml H_2O 100 ml	ASTM No. 5
Aluminum	None	Electrolytic	Flick's Reagent	Single Crystal
Mild Steel	932° F for One Hour	a) Electrolytic b) Dymo 1-Medium Diamond Compound c) AB Alpha Alumina	1% Nital	ASTM No. 9.5
"Rockwell" Test Block	None	a) Dymo 1-Medium Diamond Compound b) AB Polishing Alumina No. 1	1% Nital	None
Tin	212°F for One Hour	a) Electrolytic	1% Nital	ASTM No.-2.5
Cadmium	212°F for One Hour	a) Dymo 1-Medium Diamond Compound b) AB Alpha Alumina	Ferric Chloride Solution Same as for Copper	ASTM No.-1
Zinc	400° F for One Hour	a) Dymo 1-Medium Diamond Compound		ASTM No.-1

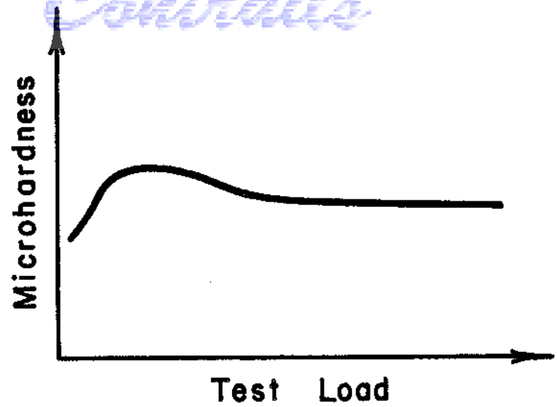

Fig. I Schematic Diagram of the Microhardness-Load Relationship.

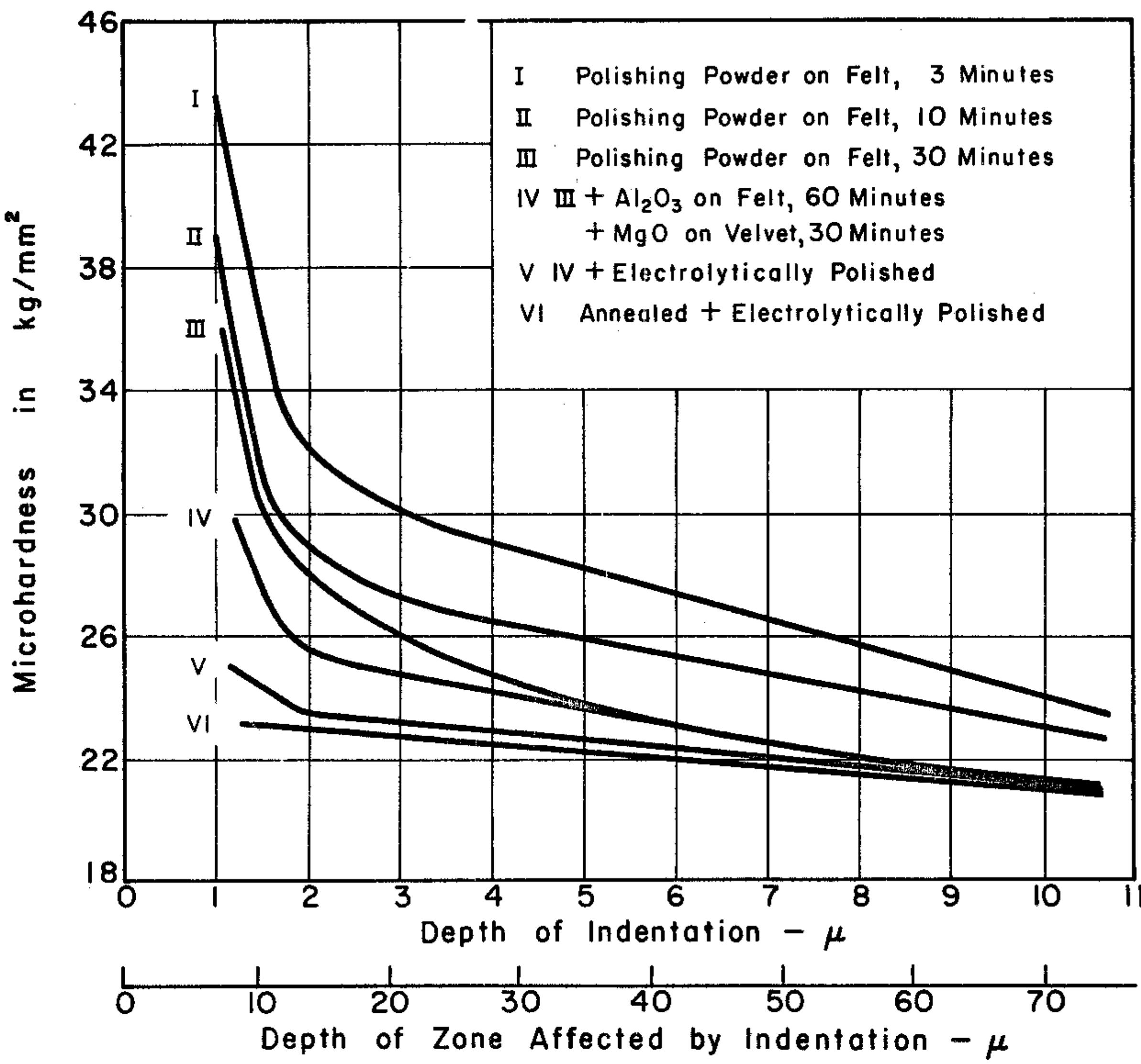

Fig. 2 Effect of Polishing Procedure on the Load Dependence of Microhardness. Bückle.

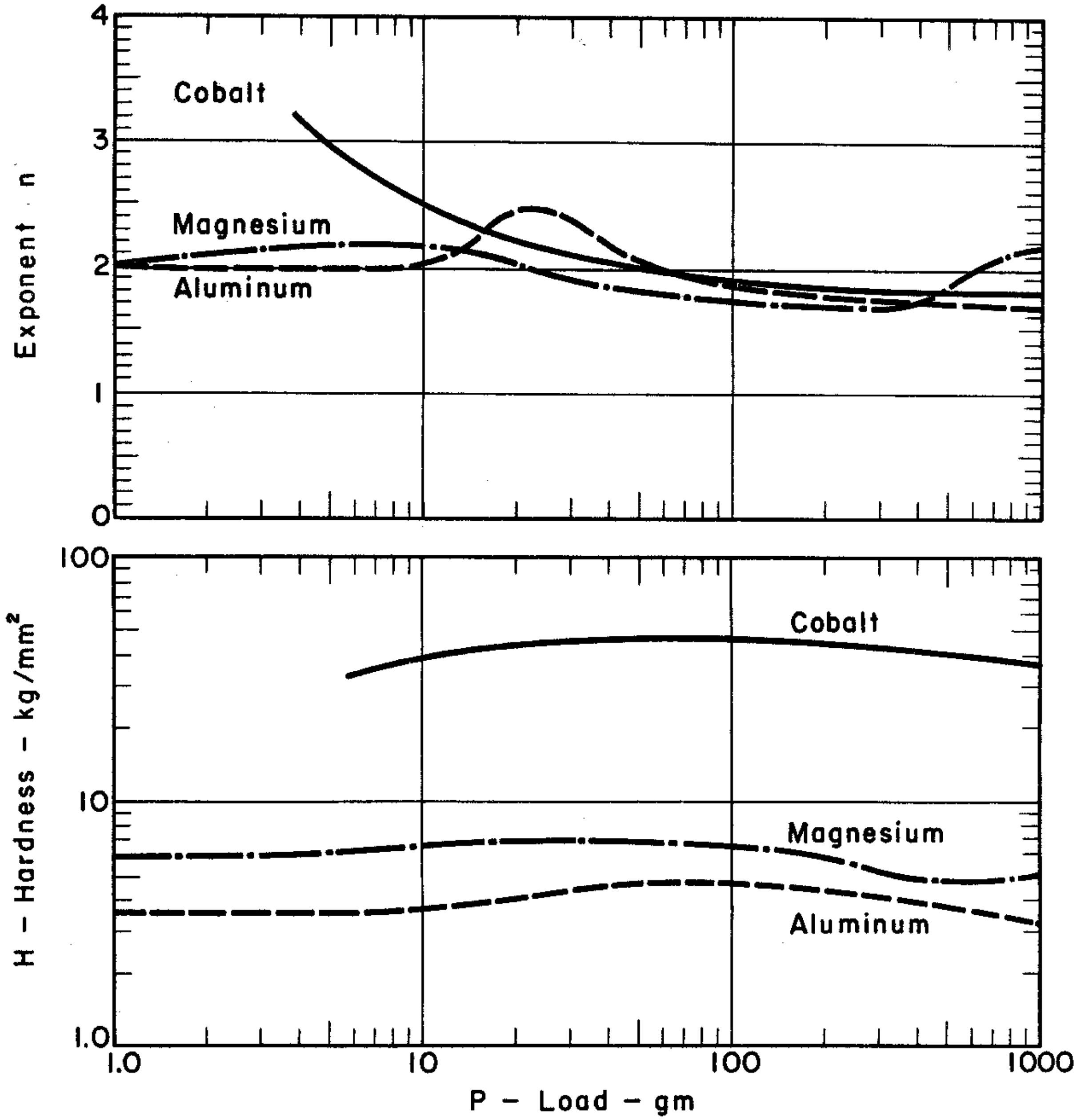

Fig. 3 Hardness and Exponent n Versus Load for Cobalt, Magnesium, and Aluminum. Onitsch-Modl.

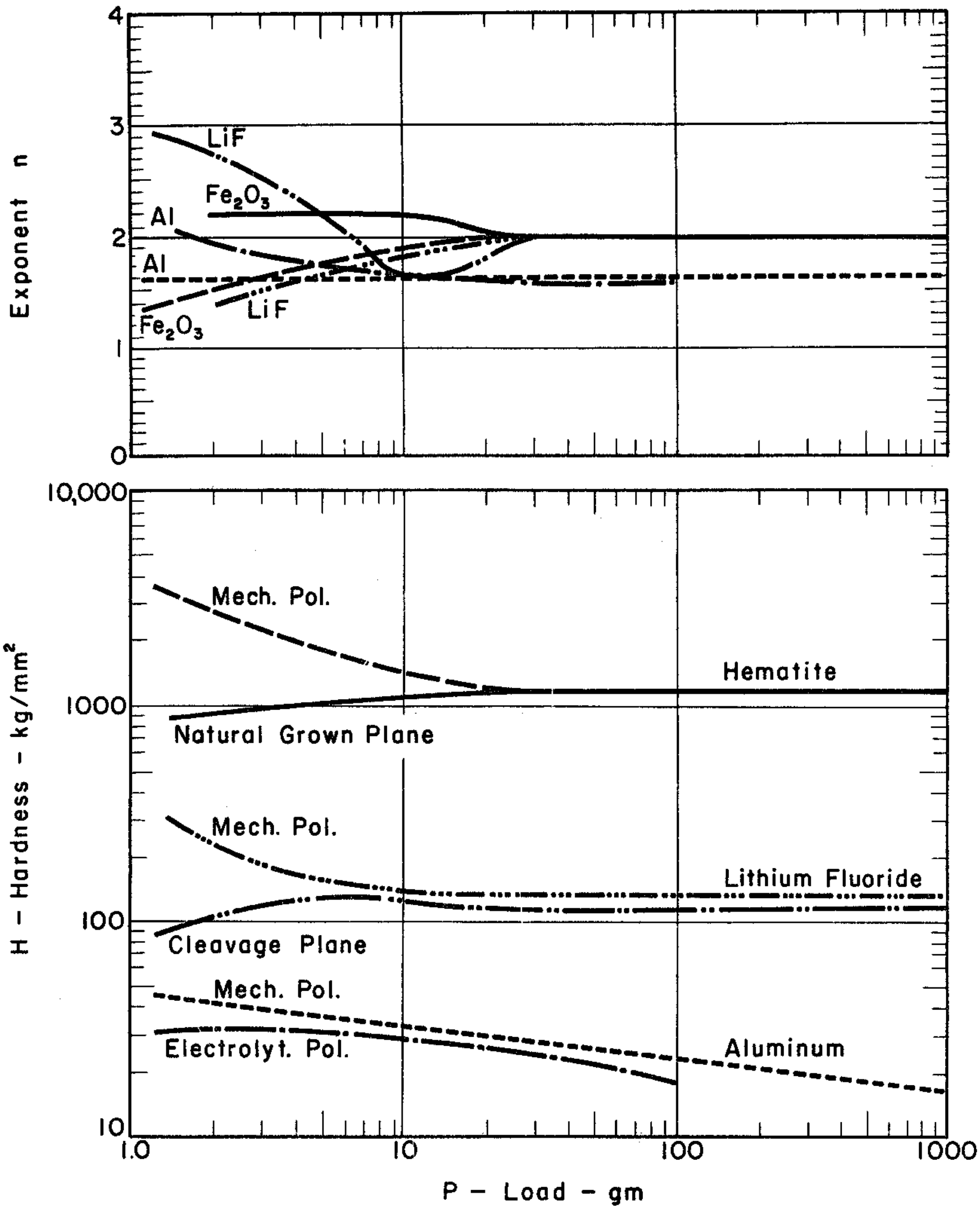

Fig. 4 Effect of Specimen Preparation on the Hardness-Load Relationship for Various Materials. Bernhardt.

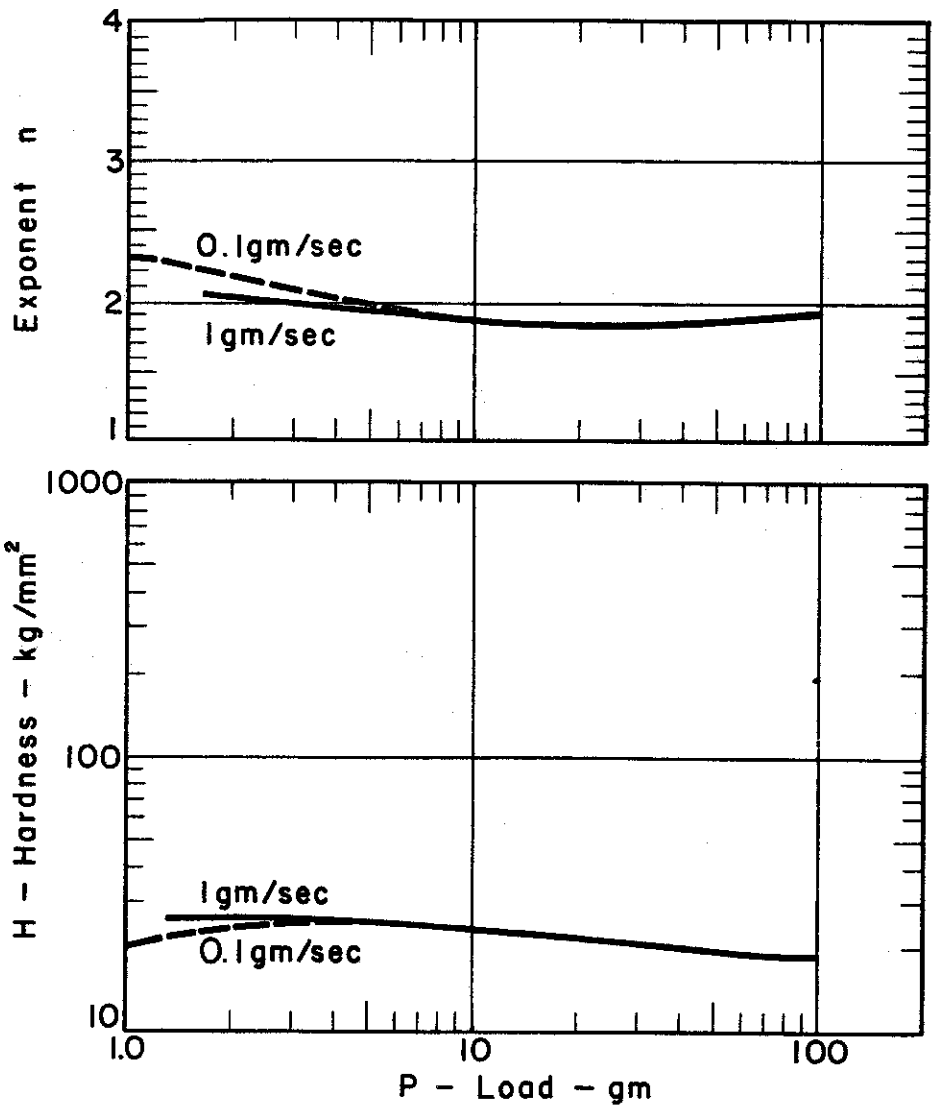

Fig.5 Effect of Loading Speed on the Hardness-Load Relationship for Electrolytically Polished Aluminum. Bernhardt.

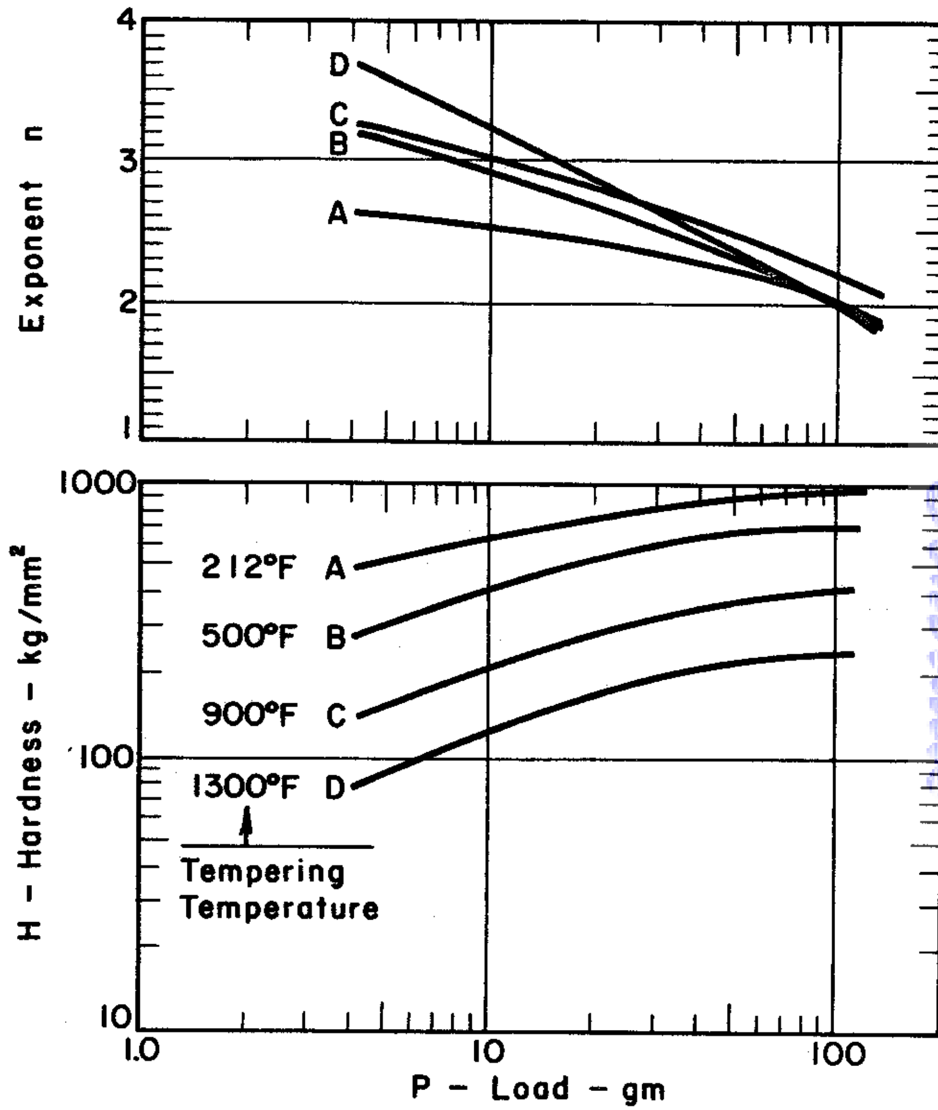

Fig.6 Effect of Tempering Temperature on the Hardness-Load Relationship for Quenched Carbon Steel. Campbell et al.

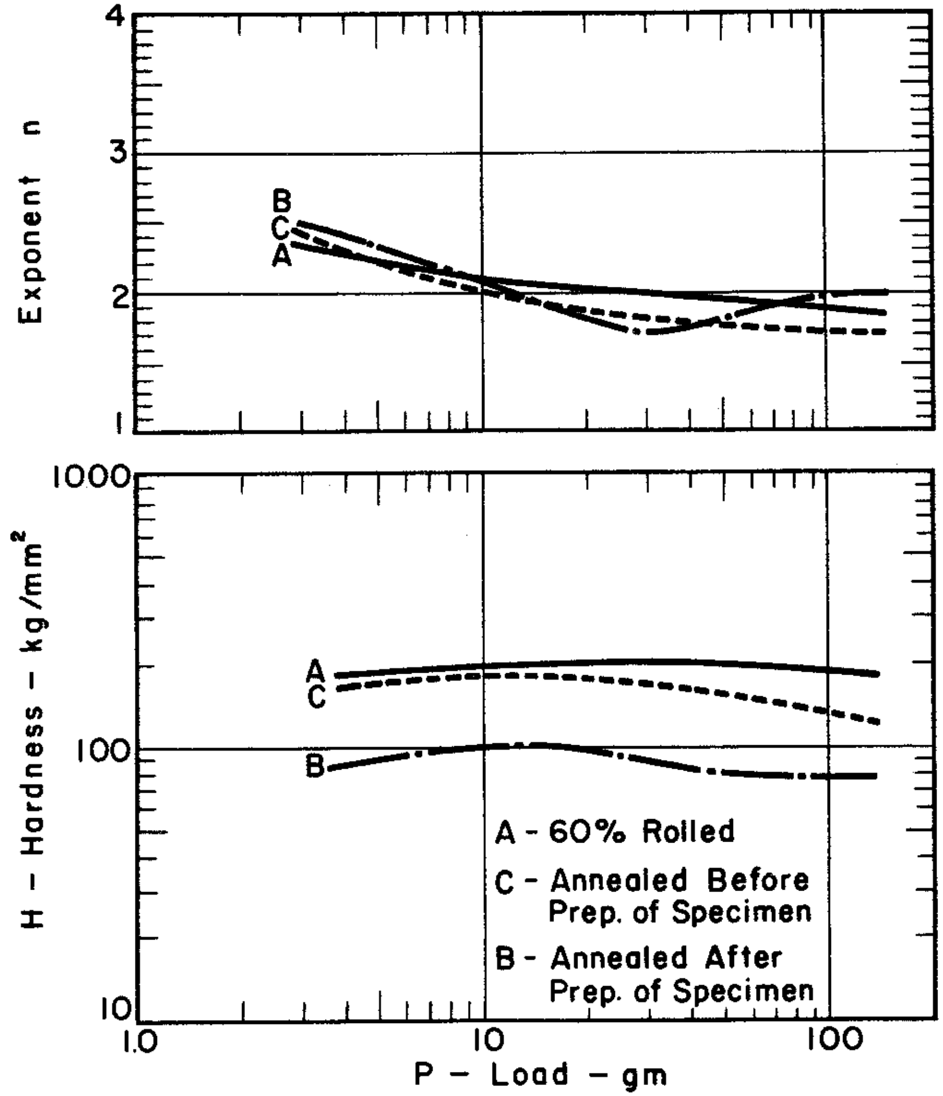

Fig.7 Effect of Specimen Preparation on the Hardness-Load Relationship for Nickel. Svetsova and Lebedeva.

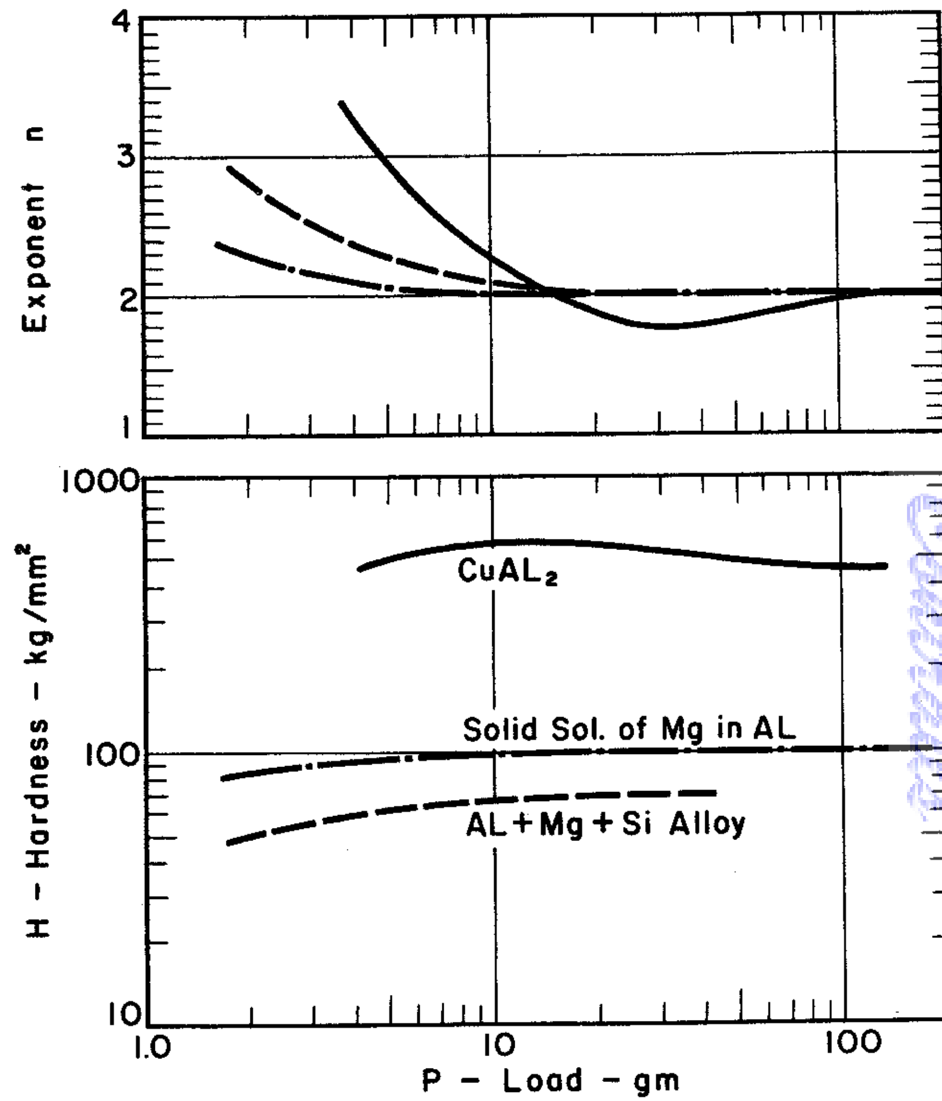

Fig.8 Hardness and Exponent n Versus Load for Various Aluminum Alloys. Svetsova and Lebedeva.

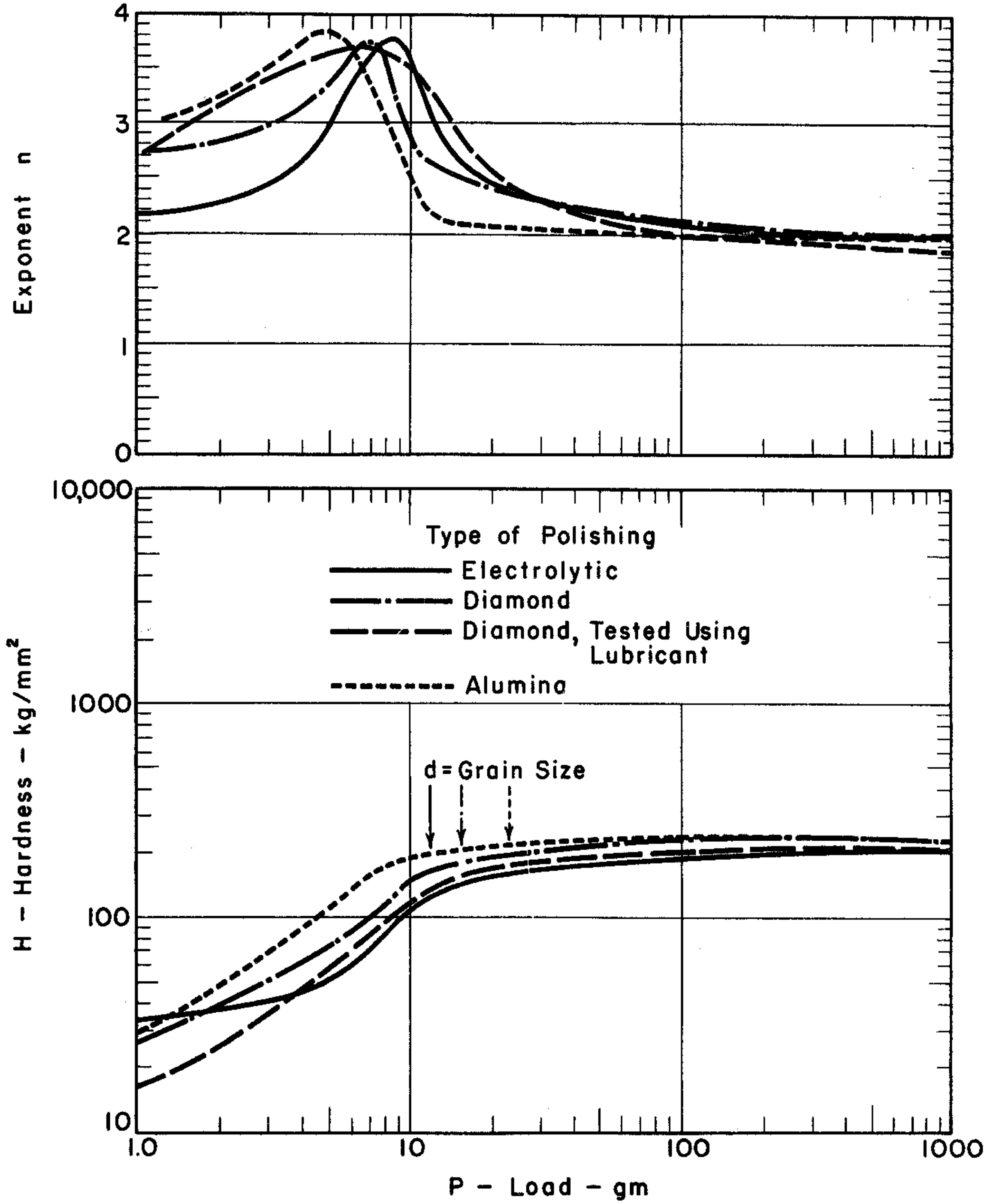

Fig. 9 Hardness and Exponent n Versus Load for Mild Steel Polished in Different Ways.

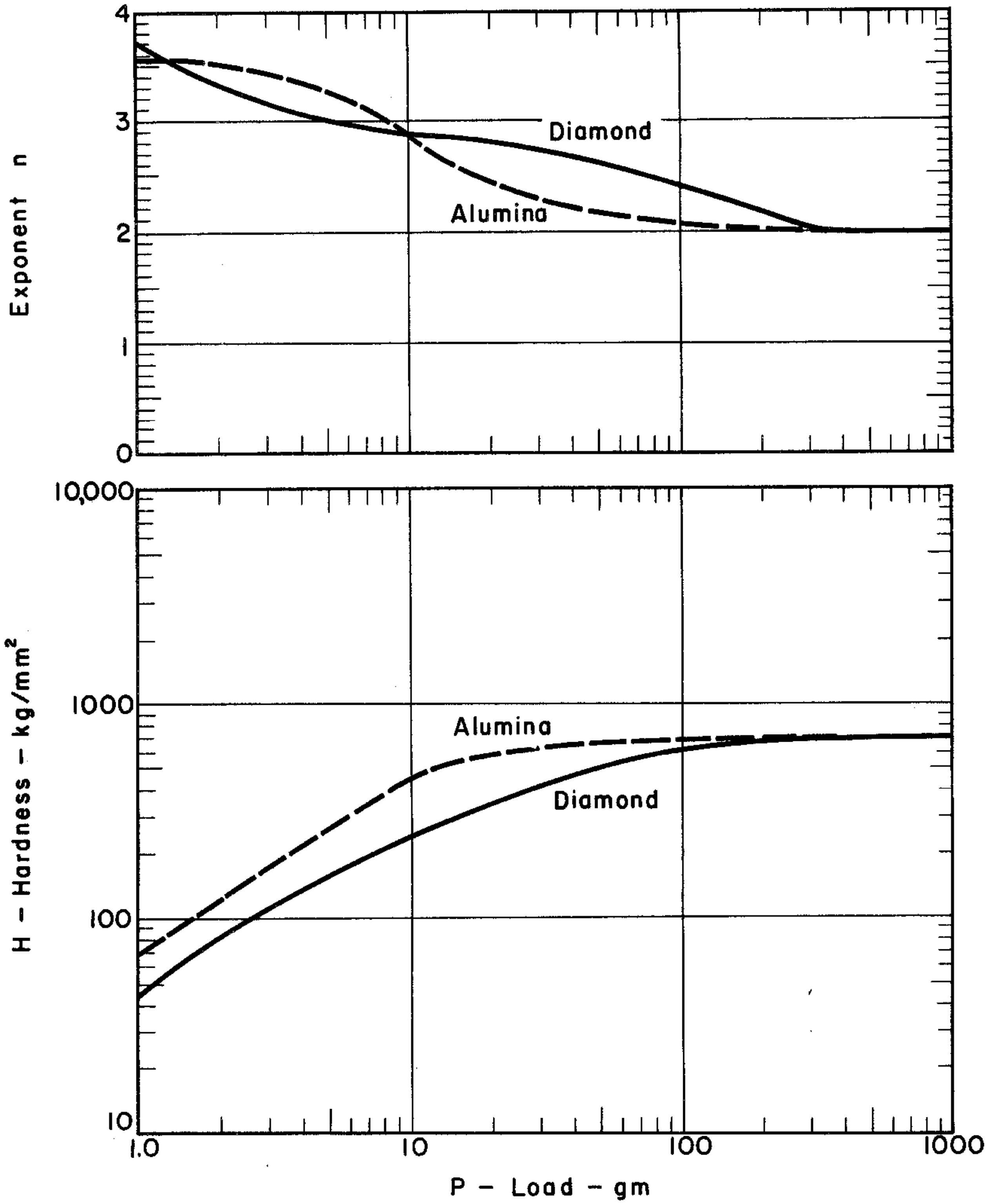

Fig.10 Hardness and Exponent n Versus Load for Alloy Steel (Hardness Testblock RC57.1) Polished by Diamond Compound and Alumina.

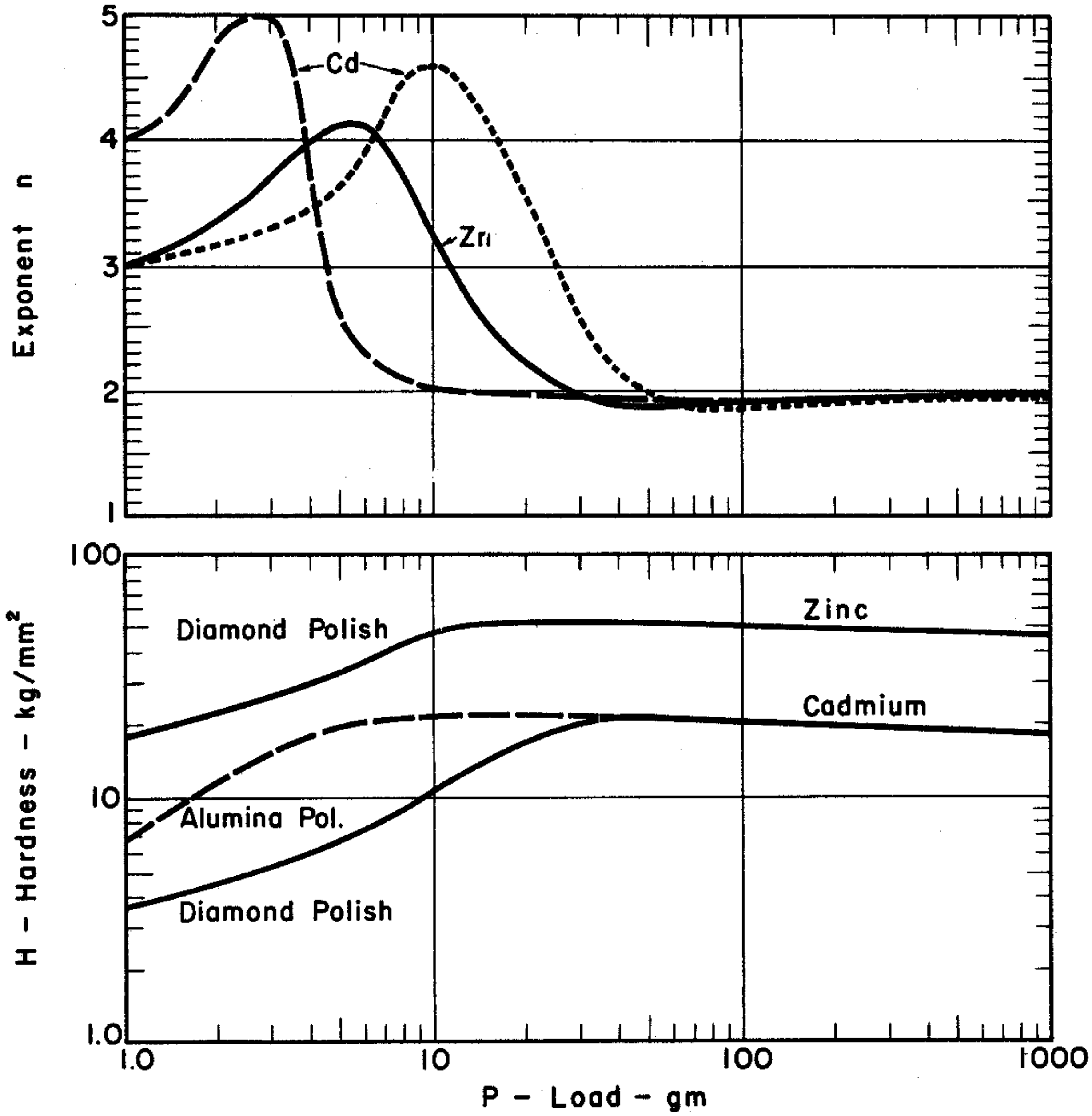

Fig. 11 Hardness and Exponent n Versus Load for Cadmium and Zinc.

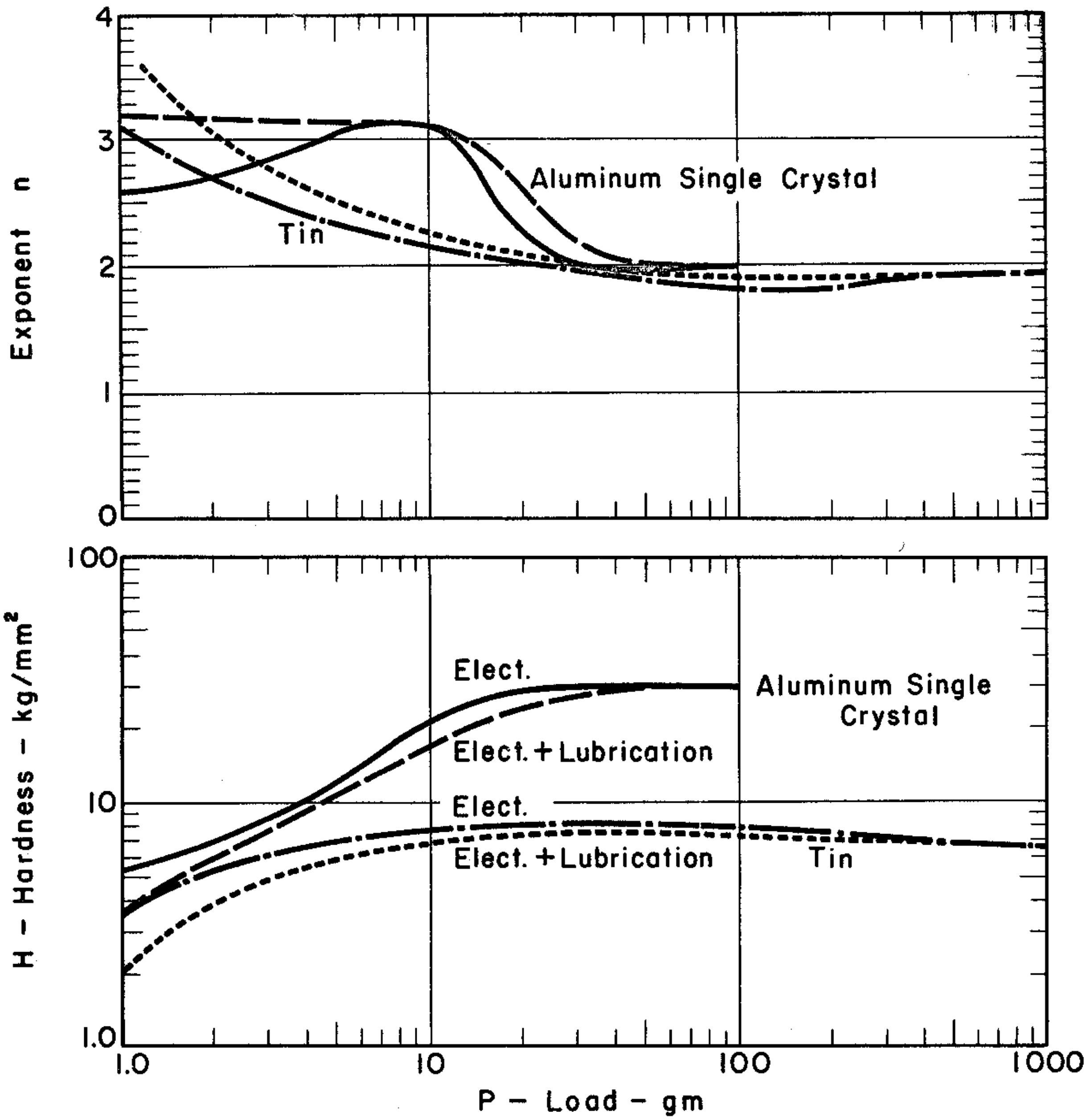

Fig. 12 Effect of Lubrication on the Hardness-Load Relationship of Electrolytically Polished Aluminum and Tin.

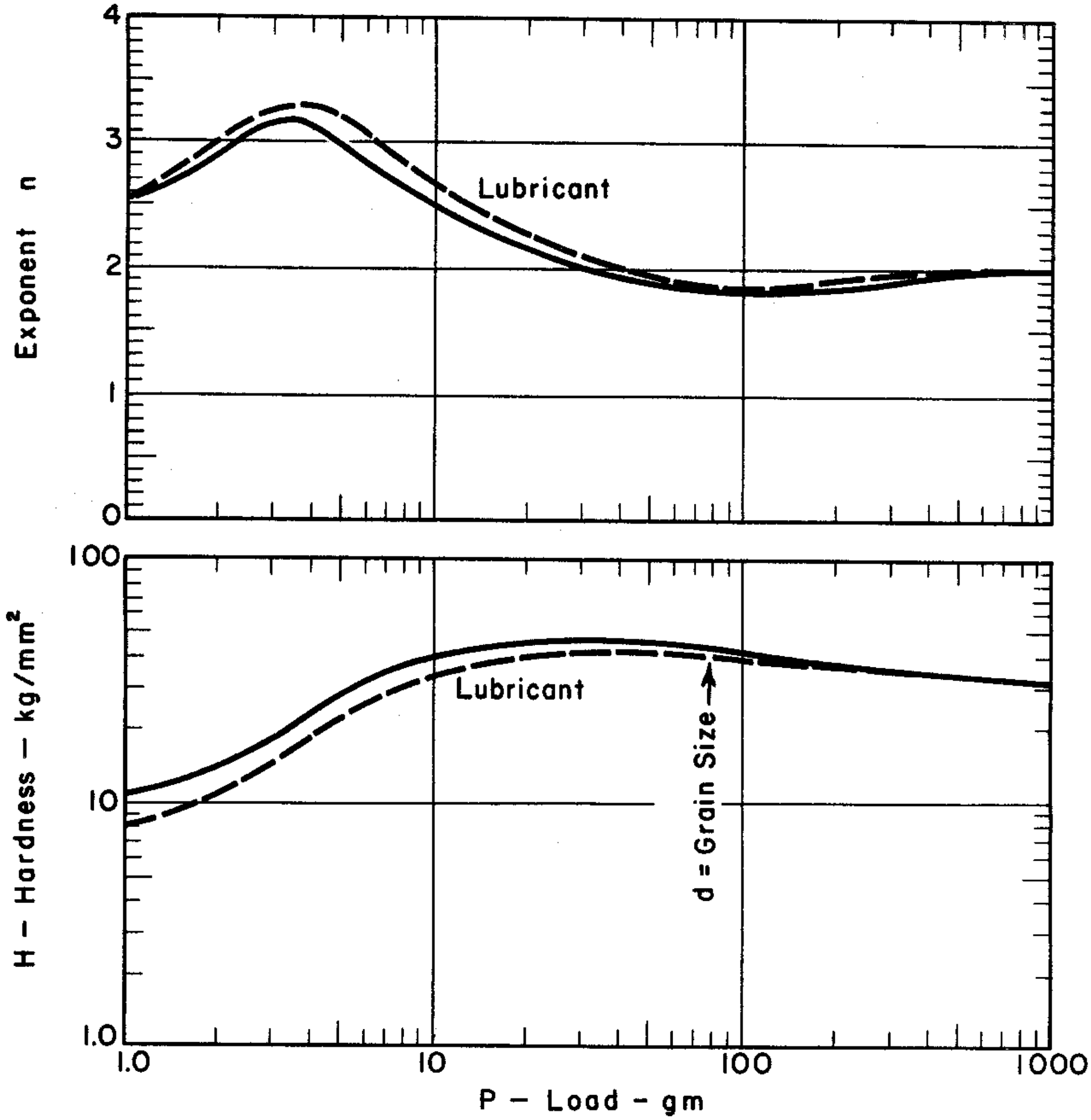

Fig. 13 Effect of Lubrication on the Hardness-Load Relationship of Electrolytically Polished Copper.

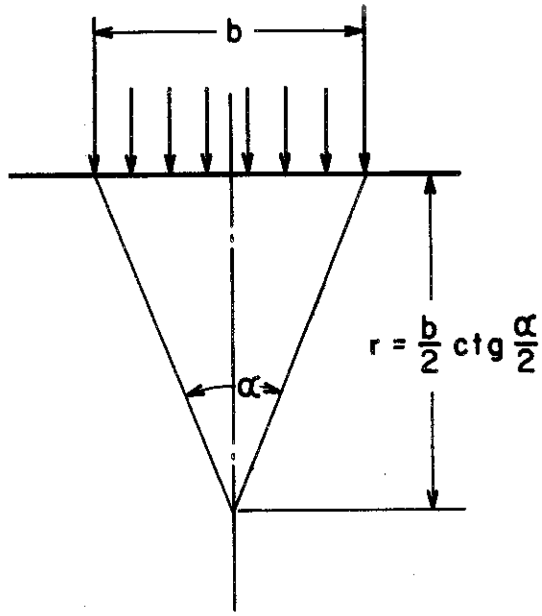

Fig. 14 Locus of Shear Stress.

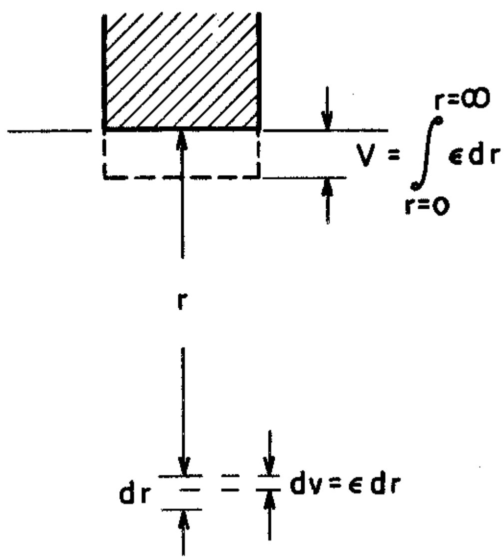

Fig. 15 For the Determination of the Relationship Between Stress and Depth of Indentation.

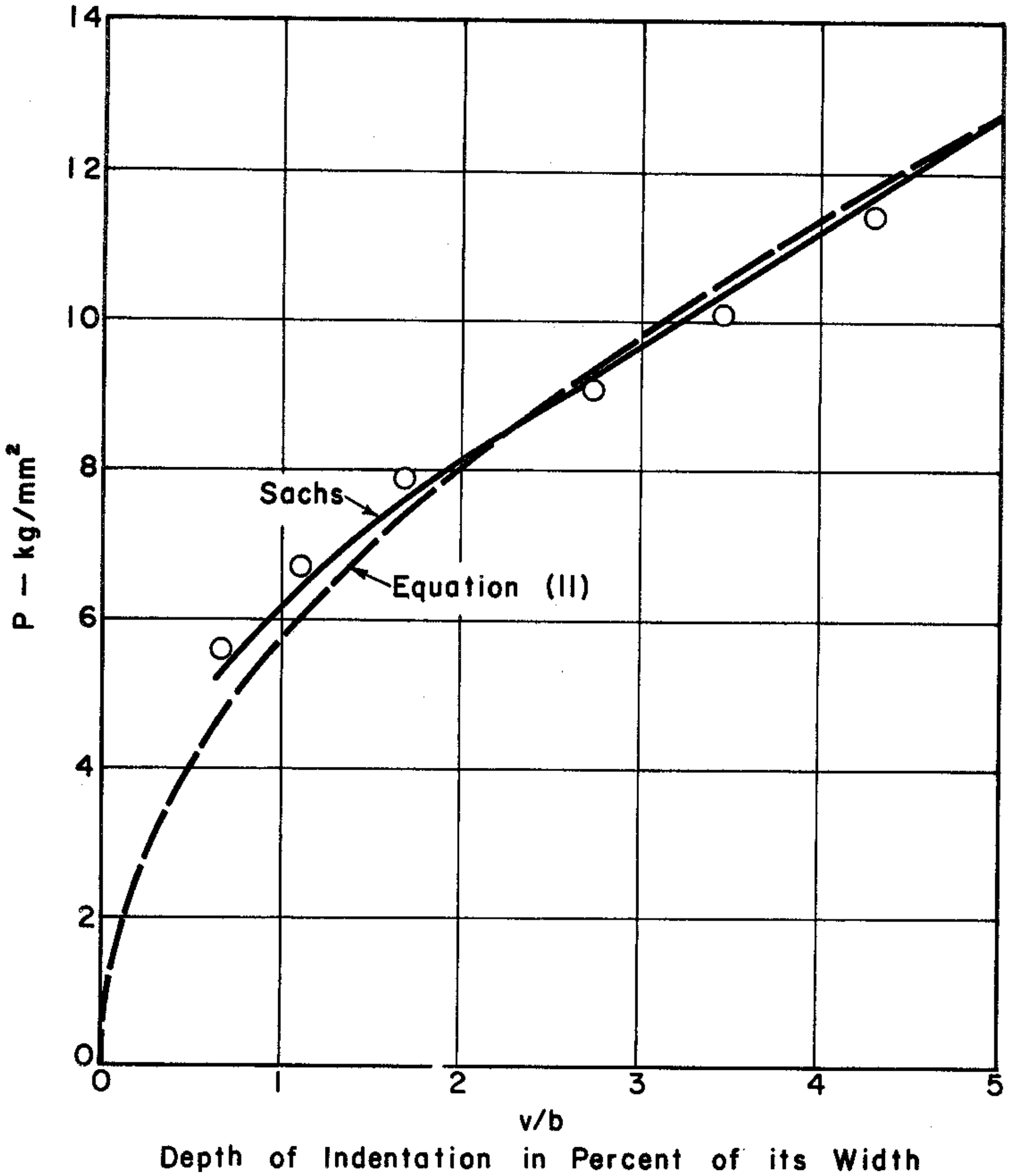

Fig. 16 Load Versus Depth of Indentation of a Punch. From Data Reported by Sachs.

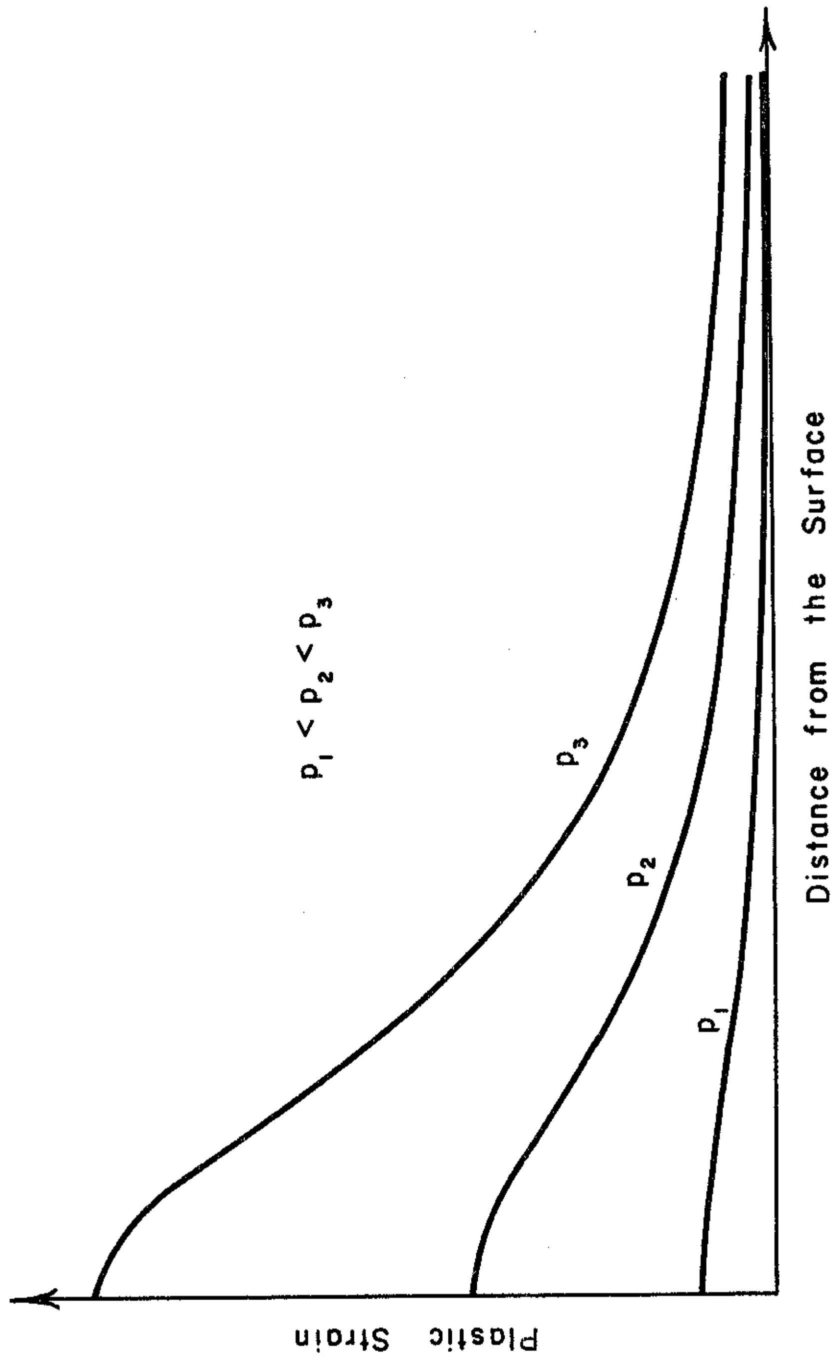

Fig. 17 Degree of Deformation as a Function of the Distance from the Surface for Various Load Magnitudes.

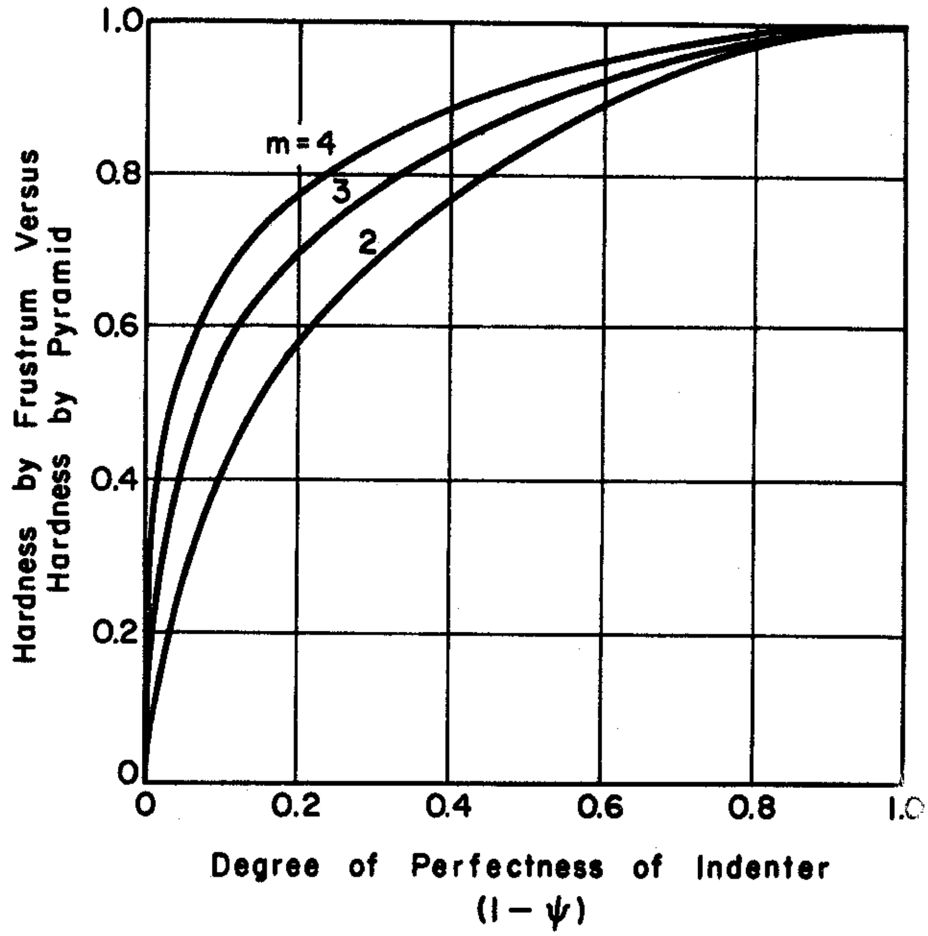

Fig. 18 Ratio of the Hardness of an Imperfect Indenter Versus that of a Perfect one as a Function of the Degree of Perfectness of the Indenter for Materials with Different Strain Hardening Behavior.

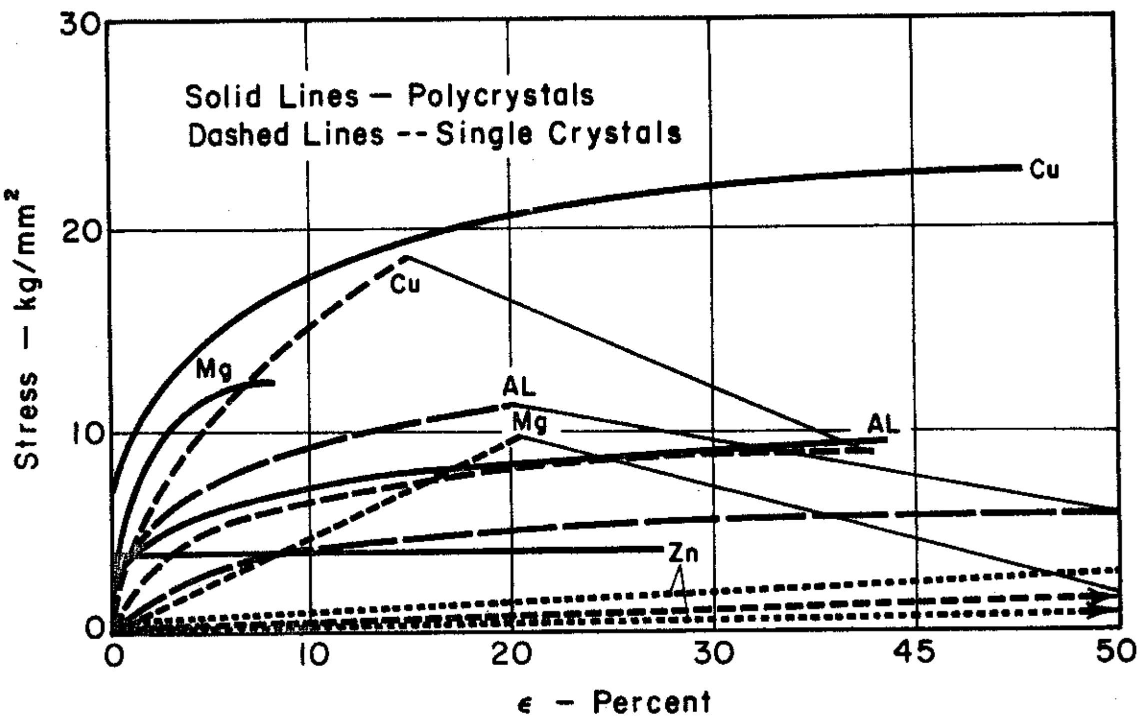

Fig. 19 Stress-Strain Curves of Single Crystals and Polycrystals. Kochendoerfer.

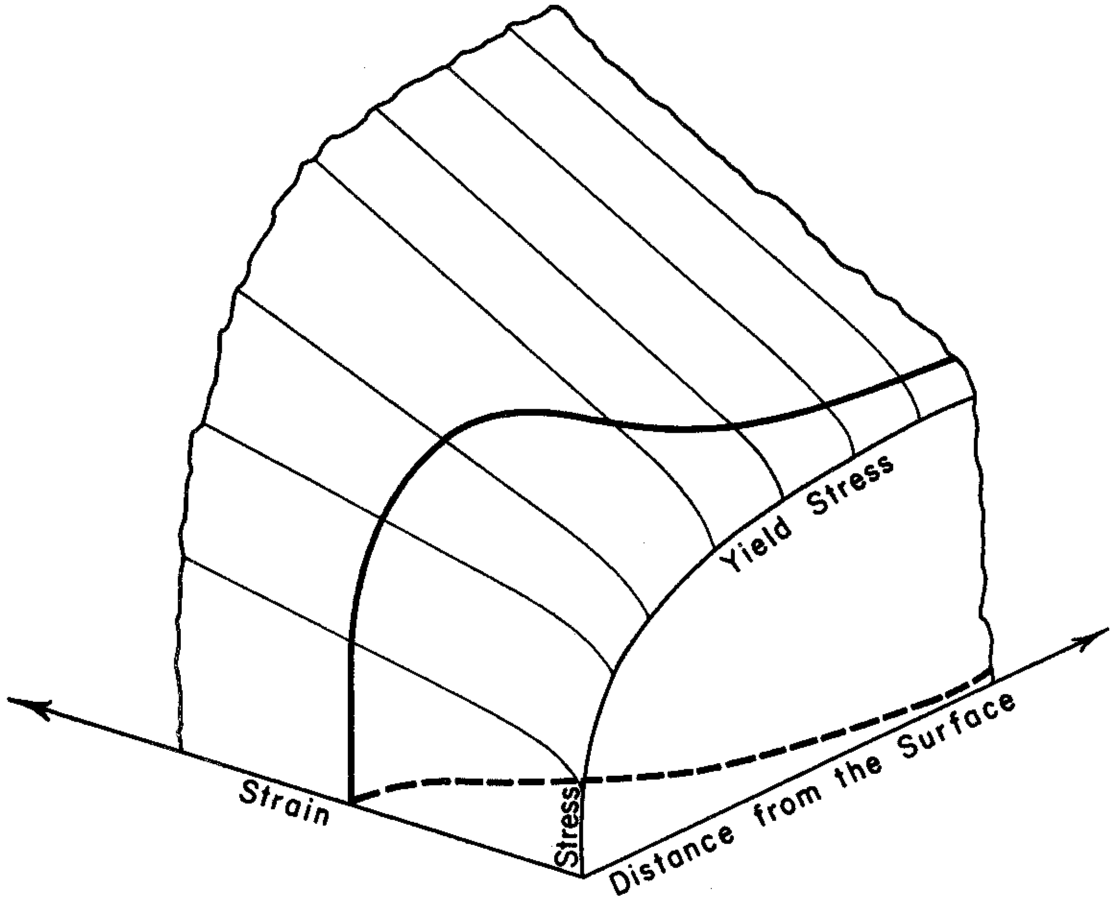

Fig. 20 Surface Effect on Strain Hardening due to Polishing.