

FOREWORD

This report was prepared by the Martin Company under USAF Contract No. AF 33(616)-6220. This contract was initiated under Project No. 7023, "Research on New Chemical Systems and Methods of Synthesis", Task No. 73667, "New Synthetic Methods for Inorganic Materials." The work was administered under the direction of the Materials Central, Directorate of Advanced Systems Technology, Wright Air Development Division, with Mr. H. L. Gegel acting as project engineer.

This report covers work conducted from January 1960 to January 1961.

WADD TR 61-58 Pt. I

Contrails

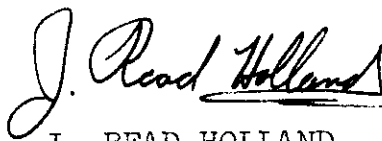
ABSTRACT

Aluminum single crystals were pulled in an electrolytic cell and the surface of the crystal was removed during the deformation. The extent of Stages I and II was increased and their slopes decreased as the rate of metal removed from the surface was increased. Increasing the strain rate caused a decrease in the extent of Stages I and II and increased the slopes. The experimental data indicate that the work-hardening coefficient of Stage I is determined primarily by the conditions which exist on the surface of the crystal. In Stages II and III, both surface effects and internal barriers are important.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:



J. READ HOLLAND
Actg. Chief, Advanced Metallurgical
Studies Branch
Metals and Ceramics Laboratory
Materials Central

TABLE OF CONTENTS

	PAGE
I. Introduction	1
II. Experimental Procedure	6
III. Experimental Results	8
IV. Discussion	12
V. Conclusions	15
VI. References	16

LIST OF FIGURES

Figure		Page
1	Orientation of Aluminum Crystals	18
2	Comparison of Calculated and Measured Cross-Sectional Area of Aluminum Specimens Pulled in Electrolytic Polishing Bath (rate of metal removal was 25×10^{-5} in./min)	19
3	The Effect of Rate of Removal on the Stress-Strain Curves of Al-37 (temperature 3° C and strain rate 10^{-5} sec $^{-1}$)	20
4	The Effect of Rate of Removal on the Extent (ϵ_2) and Slope (θ_1) of Stage I of Al-37	21
5	The Effect of Rate of Removal on the Extent (ϵ_3) and Slope (θ_2) of Stage II of Al-37.	22
6	The Effect of Rate of Removal on the Increase of ϵ_2 and ϵ_3 . . .	23
7	The Effect of Rate of Removal on the Decrease in Slopes θ_1 and θ_2	24
8	The Effect of Rate of Removal on the Critical Resolved Shear Stress, the Stress of the End of Stage I, τ_1 , and the Stress at the End of Stage II, τ_3	25
9	The Effect of Strain Rate on the Increase in ϵ_2 and Decrease in θ_1	26
10	The Effect of a Change in the Rate of Removal on the Slope in Stage I (at A, the rate of removal was changed from 12.5×10^{-5} to 25×10^{-5} in./min)	27
11	The Effect of Change in the Rate of Removal on the Slopes in Stage III	28
12	The Effect of Removing 0.041 In. from the Transverse Dimen- sions of the Specimen on the Recovery of Work-Hardening. . . .	29

Contrails

I. INTRODUCTION

Although numerous investigations have been conducted on the plastic flow characteristic of metals in an attempt to explain the mechanism of work hardening, relatively few studies have taken into account the influence of the surface. In all current theories of work hardening, it is assumed that the impediments for the movement of dislocations are within the crystal. The barriers due to the surface and the existence of solid and liquid films have been neglected even though it has been shown that the surface exerts a large effect.

Roscoe (Ref. 1), using cadmium wire single crystals, found that the conditions of the surface exercised a surprisingly large effect on the critical resolved shear stress of the crystals. He reported (Ref. 2) that an oxide film less than 20 atoms thick caused an increase of approximately 50%; an increase in the thickness of the oxide film to approximately 1200 atoms increased the initial stress for slip by a factor of nearly 100%. He suggested that added strength had its origin in the sealing of submicroscopic cracks by the film analogous to the behavior of rock salt, as shown by Joffe (Ref. 3), in its behavior under water. Cottrell and Gibbons (Ref. 4) reported that a thin oxide film on cadmium crystals free from nitrogen increased the critical shear stress from 12 to 30 gm/mm². Harper and Cottrell (Ref. 5), from their study of zinc single crystals, concluded that the greater strength of specimens with an oxide coating was a dynamic effect connected with the amount and rate of plastic flow rather than with the stress required to start flow. Takamura (Ref. 6) reported an increase in the critical resolved shear stress from 76 to 174 gm/mm² for aluminum crystals when the oxide coating was increased from 100Å to 500Å. From his experimental observations, Takamura concluded that the yield point could be interpreted as the stress at which the surface film could no longer withstand the pressure of piled-up dislocations.

Andrade and Henderson (Ref. 7) noted that the surface contamination of silver single crystals, which takes place by exposure to air at room temperature, raises the critical shear stress a small degree but has more effect on the hardening after considerably greater strain has occurred. They also found that the length of Stage I of the stress-strain curve was reduced.

Manuscript released for publication February 1961 as a WADD Technical Report

Gilman and Read (Ref. 8) observed that the difference between the shear stress of cleaned and copper-plated zinc crystals was usually less at the critical shear stress than after 10% elongation. Takamura (Ref. 6) also noted changes in the character of the stress-strain curves for aluminum as a result of the presence of an oxide film. The specimens with a thicker anodic film did not show the Stage I region as clearly as did a companion specimen with a thinner surface film. Garstone, Honeycombe and Greetham (Ref. 9) found that the plated crystals of copper resulted in the elimination of Stage I.

Shapiro and Read (Ref. 10) made internal friction measurements on cadmium single crystals with and without oxide films formed by immersion in cadmium nitrate solutions. The results obtained indicated that the oxide film caused a much more rapid rise in internal friction with increase in vibration amplitude. Upon removal of the film and recovery of the crystal, the internal friction behavior returned to that observed prior to oxidation. It was pointed out that this result made it possible to ascribe the effect of oxide films on plastic properties to an inhibiting effect on dislocation formation at the specimen surface.

Barrett (Ref. 11) demonstrated an abnormal twisting when the oxide film was removed from both single crystals and polycrystalline wires of iron and zinc. When an etchant was suddenly applied during the test, a transient condition occurred and the specimen twisted instead of untwisting. Similar results were obtained for polycrystalline decarburized mild steel wires. Barrett (Ref. 12) also showed an abnormal twisting on polycrystalline cadmium wires when the film was removed by etching.

The transient reversal of the twisting was explained by Barrett (Ref. 11) on the basis that adherent oxide films on metals act as barriers to the movement of dislocation. Upon twisting a wire, dislocations pile up beneath the oxide-metal interface and when it is removed by the acid, the dislocations escape through the surface.

The plastic flow characteristics of single crystals in aqueous solutions containing electrolytes seem to be limited mainly to studies of the creep behavior. Rehbinder and Wenstrom (Ref. 13) first reported an effect on both hardness and tensile properties of metal in various electrolytes when an electrical potential

was applied to the specimen. They found that the strength increased and then decreased when the specimen became more cathodic. The effect of an applied potential on the creep behavior of lead and tin single crystals followed the general behavior as found in the stress-strain curve, i.e., the creep rate increased as the cathode potential was increased. Later, Pfitzenreuter and Masing (Ref. 14) found similar effects on gold and platinum and concluded that since the experiments were conducted under conditions of prolonged cathodic polarization (which excludes the possibility of surface oxide films) and on gold and platinum (which normally do not have surface oxides), the increase in the creep rate could not have been due to a removal of surface oxides.

A number of investigators have studied the creep behavior of metal single crystals when immersed in a solution containing electrolytes. Andrade and Randall (Refs. 15 and 16) first noticed that immersion of cadmium crystals in cadmium nitrate gave an initial increase in the rate of creep which quickly stopped. However, it was also found that the creep rate increased by a factor of twenty when the cadmium crystals were immersed in a plating solution. Phillips and Thompson (Ref. 17) also found that addition of an aqueous solution of cadmium nitrate decreased the creep rate in cadmium single crystals. The removal of the hydroxide layer by the addition of sulphuric acid caused the creep rate to increase immediately. Andrade and Randall (Ref. 18) were able to show that an electrolyte alone, without an applied potential, had a marked effect on the creep behavior of cadmium and zinc crystals. In some cases, the creep rate increased by a factor of nine.

From the foregoing, it becomes quite obvious that solid films on the surface of single crystals markedly affect their mechanical behavior. In general, the presence of a solid film tends to increase the yield stress and increase the work-hardening rate. Often, on single crystals, Stage I and, at times, Stage II regions are completely suppressed.

Various mechanisms have been offered for the effects of oxide and metal films as well as the influence of electrolytes. Of these, concepts concerned with the locking of surface dislocation sources and the blocking of dislocations at the surface resulting in pileups, appear to be actively considered at present. Barrett (Ref. 11), Takamura (Ref. 6), Gilman (Ref. 19), Lipsett and King (Ref. 20),

Shapiro and Read (Ref. 10) and Weiner and Gensamer (Ref. 21) are among those who have interpreted their results in terms of piled-up dislocations at the surface, while Adams (Ref. 22) and Chalmers and Davis (Ref. 23) have explained their experimental observations in terms of locking of surface dislocation sources. In general, the change in plastic flow properties due to electrolytes has been explained in terms of the unblocking or unlocking of dislocations by the removal of the oxide films. In considering the two proposed mechanisms, it appears that the locking of sources of surface dislocations by a solid film should exert a primary influence only on the critical resolved shear stress for flow and not on the slopes of Stages I and II. However, the blocking at the surface of dislocations from internal sources may also affect the critical resolved stress and exert an influence throughout the whole plastic range. In certain cases, it does not seem feasible to explain the results of experimental observations in terms of locking of surface dislocation sources. The abnormal after effects found by Barrett (Refs. 11 and 12) by removing the oxide by an acid treatment is excellent evidence of the blocking of dislocations at the surface. Additional evidence in favor of a blocking due to a pileup of dislocations at the surface may be found from the observations that the critical resolved shear strength continues to increase with the thickness of the oxide layer until very heavy oxide layers are formed. If the locking of surface dislocation sources were the dominant factor, the critical resolved shear stress would not be expected to increase after all of the surface sources were locked by the formation of the oxide. This may be expected to happen after a few atomic layers of the oxide are formed.

In spite of the above evidence on the strong influence of the surface on the plastic flow characteristic, this has been ignored in current theories of work hardening. Seeger (Refs. 24 and 25) suggested that most of the dislocations may slip out of the crystal only when the specimen axis was within certain areas of the orientation triangle. In other areas, the resolved shear stress in other glide systems is large enough to generate dislocations which can form Lomer-Cottrell locks, thereby decreasing the average slip distance in some directions and causing a larger hardening rate. Characteristic of the tensile behavior of face-centered cubic metal crystals is the stage of rapid work-hardening which follows Stage I. Friedel (Ref. 26) assumed that at the beginning of Stage II, a large number of Lomer-Cottrell dislocations are formed by a catastrophic process which uses up all the Frank-Read sources on the secondary slip planes. In this manner, a

fixed number of Lomer-Cottrell locks are formed which act as barriers against which the dislocations can pile up. Seeger (Ref. 24) and Diehl, Mader and Seeger (Ref. 25) proposed that Lomer-Cottrell barriers are circumvented by the cross slip of extended screw dislocations. Cottrell and Stokes (Ref. 27), Friedel (Ref. 26), Cottrell (Ref. 28) and Stroh (Ref. 29) suggest that the Lomer-Cottrell dislocations collapse under the stress field of the dislocation pileup. However, Seeger's (Ref. 24) calculations show that the activation energy for cross slip of extended screw dislocations is always smaller than that for the collapse of Lomer-Cottrell barriers.

It is the purpose of this paper to report the changes in Stages I, II and III of the deformation process in aluminum single crystals pulled in tension while the surface was being continuously removed. It is intended that through these studies, a clearer insight may be obtained on the effects of surface conditions on the passage of dislocations out of the crystal surface and the associated effects on the work-hardening characteristics. Some effects of the surface on the irreversibility of plastic flow and the recovery of critical resolved shear stress after plastic deformation will also be given.

II. EXPERIMENTAL PROCEDURE

Throughout this investigation, the aluminum single crystals used were prepared by a modified Bridgman technique using a multiple-cavity graphite mold which was capable of yielding 35 crystals of the same orientation. The aluminum was 99.997% pure and contained as impurities magnesium, silicon, zinc, copper and iron. From this material, five sets of crystals 4 inches x 1/8 inch x 1/8 inch were prepared.

The orientation of the crystals as determined by back-reflection Laué patterns is shown in Fig. 1. Prior to testing, the specimens were prepared by a mechanical and electrolytic polishing treatment after which they were annealed in vacuum at 630° C for two hours; then the furnace was allowed to cool. Just before testing, the specimens were again electrolytically polished. In order to remove the surface continuously during the testing procedure, a methyl alcohol-nitric acid polishing solution was employed. The specimens were electrically insulated from the tensile machine by the use of phenolic resin specimen holders containing an electrical lead which allowed the current to pass through the 3-inch gage length of the crystals. The movement of the head of the tensile machine was used as a measure of the strain.

The specimen holders were approximately 1-1/2 inches deep and 5/16 of an inch in diameter. The ends of the crystals, for a distance of 1/2 inch, were copper plated and coated with a thin layer of soft solder. The specimens were placed in a special fixture which allowed the specimens to be accurately aligned axially and with respect to the distance between the specimen holders. A low melting point lead-bismuth alloy was poured between the crystal and its holder to keep it firmly in place. The exposed portion of the lead-bismuth alloy was coated with paraffin to prevent it from reacting with the electrolyte. The temperature was maintained constant within $\pm 0.1^\circ$ C by means of a water bath. In all cases, unless otherwise specified, the tensile tests were conducted at 3° C.

A stiff tensile machine equipped with an automatic recorder was used to measure the loads and deformations. It was possible to measure elongations to within 3×10^{-5} inch and loads of 0.01 pound.

Contrails

Since it was necessary to know the area of the specimen while the surfaces were being removed in the polishing bath, a series of calibration curves were obtained to determine the amount of metal removed as a function of the current density. These values were used for the construction of the stress-strain curves. The average rate of metal removed as a function of the cell current is given in Table 1 for the aluminum crystals specimens of Group 37. The rate of metal removed as defined here refers to the rate of change in the transverse dimensions. A typical curve showing a comparison between the measured and calculated values of the area for various periods of time during the extension of the specimen is given in Fig. 2.

III. EXPERIMENTAL RESULTS

A set of stress-strain curves typical of those obtained from aluminum single crystals pulled at a strain rate of 10^{-5} sec^{-1} in an electrolytic polishing bath are shown in Fig. 3.

In this paper, the ϵ_2 and ϵ_3 values were taken at the end of the linear portion of Stages I and II, respectively. The critical resolved shear stress was defined to be the stress at which the stress-strain curve first became nonlinear. The terms τ_1 and τ_3 are the shear stresses which occur at the strains ϵ_2 and ϵ_3 . From the curves of Fig. 3 it is seen that ϵ_2 and ϵ_3 , the extent of Stage I and Stage II, respectively, increased as the rate of metal removal, R , was increased. The slopes θ_1 and θ_2 for the two stages correspondingly decrease. Figures 4 and 5, which contain the data obtained from crystals 37, show that ϵ_2 and ϵ_3 increase in a linear fashion with the rate of metal removal. ϵ_2 increased from a value of 0.8% when R was zero to 1.65% and when R was 50×10^{-5} inch per minute. ϵ_3 changed from 2.75% to 4.25%. The slopes of Stages I and II decrease continuously, but not linearly. There seems to be a tendency for θ_1 to approach some constant value when R becomes sufficiently high. This tendency was not noticed for θ_2 in the ranges of R used in these studies. From the data reported in Figs. 6 and 7, it is seen that for a given R , the change in ϵ_2 is not as large as that of ϵ_3 ; however, the change in θ_1 is larger than that of θ_2 . The slope, θ_1 , decreased from a value of 7400 to 2900 psi, a difference of 4500, while that of θ_2 decreased from 13,000 to 10,000 psi. Throughout this study, as well as in an associated study using surface-active agents, it was noticed that the critical resolved shear stress and τ_1 did not change with the rate of metal removal (Fig. 8). The ratio of these two stresses is equal to 1.9 Rosi (Ref. 30), and Garstone and Honeycombe (Ref. 31) found that for copper, copper-gold and copper-silver alloys, the ratio was 2. The stress τ_3 , at which Stage III begins, decreased linearly as the ratio of removal of the metal increased (Fig. 8). When the specimens were pulled at $R = 0$, $\tau_3 = 500$ psi; however, at $R = 50 \times 10^{-5}$, $\tau_3 = 440$ psi.

In another series of experiments, the effect of strain rate on the behavior of the various plastic flow parameters was determined, at a constant rate of metal removal of 60×10^{-5} inch per minute. For these experiments, specimens of crystals 42 and 43 were pulled in a water bath and in an electrolytic polishing bath.

The difference in θ_1 and ϵ_2 obtained under the two conditions as a function of strain rate is shown in Fig. 9. As may have been expected, the extent of Stage I increased but the slope decreased as the strain rate decreased. At a strain rate of 10^{-4} sec^{-1} , the increase in ϵ_2 and decrease in θ_1 due to the removal of the surface during the tensile deformation is small; however, at a strain rate of 10^{-5} sec^{-1} , ϵ_2 increased from 1.10% to 2.20% while θ_1 decreased from 7500 psi to 2750 psi. Sufficient data were not obtained to establish a reliable relationship between strain rate and ϵ_3 and θ_2 since, in the case of specimens of aluminum 43 tested at a strain rate of $2 \times 10^{-5} \text{ sec}^{-1}$, Stage II was not fully developed. From the data obtained from specimens of aluminum 42, there were definite trends which showed that ϵ_3 increased and θ_2 decreased as the strain rate decreased.

A series of specimens of crystals 35 and 40 were pulled in a chemical polishing bath at 78° C to determine whether a Stage I region would be present at this temperature when the rate of metal removal was rapid. Specimens tested in water did not show a Stage I region at strain rates of 10^{-4} or 10^{-5} sec^{-1} . However, the stress-strain curve of those specimens pulled at these strain rates in a polishing bath, where the rate of metal removal was 2×10^{-3} inch per minute, had a Stage I region which extended to 1% and a slope of 1700 psi.

The back-reflection Laue patterns were obtained after pulling specimens of crystal 35 to various amounts of strain in the chemical polishing bath at 78° C . When the deformation was confined to Stage I, the Laue spots did not show asterism or streaking; however, when the strain was increased to the start of Stage II, there was definite evidence of the formation of streaks. Thus, confirming other observations, it appears that the start of Stage II is associated with lattice bending. It was also found that, for specimens strained within the Stage I region, the Laue spots were broken up into several discrete smaller units roughly 1 degree apart. Laue spots obtained from specimens pulled in water did not show this breakup. Subgrain boundaries were apparently formed in the first case and not in the second.

Since it was shown that the slopes of Stages I and II could be influenced by the rate at which the metal was removed from the surface of the specimen, it was of interest to determine the degree to which the work-hardening coefficient was

reversible. A series of specimens of aluminum 37 was pulled in an electrolytic polishing bath and the rate of metal removed was changed at various positions along the stress-strain curve. Figure 10 is typical of the changes which occurred in the slope of Stage I when the rate of removal of the metal was varied. In this case, the initial removal rate, R , was 12.5×10^{-5} inch per minute. At the point A, R was changed to 25×10^{-5} inch per minute and the slope θ_1 changed from 6300 to 4900 psi. In other experiments done in the same manner, R was changed from 25×10^{-5} to 12.5×10^{-5} inch per minute. In these cases the slope changed from 4900 to 6300 psi. In a similar manner, the same reversible changes in slope occurred in Stage II. However, in no case did the slope of Stage II become as low as that of Stage I. Figure 11 shows an example of the effect of a series of changes of R on the work-hardening coefficient of Stage III. The Stage III region is not linear but the work-hardening coefficient may be approximated for comparative purposes by determining the average slope between the regions where the changes occur. In the portion of the Stage III region shown in Fig. 11, R_1 was 25×10^{-5} inch per minute and the average slope was 8600 psi. When R_2 was 12.5×10^{-5} inch per minute, the slope increased to 12,500 psi and an increase to R_3 to 50×10^{-5} inch per minute decreased the slope to 5350 psi. Upon returning to R_2 , the slope again assumed the value of 12,500 psi. Throughout the course of this investigation at least 25 measurements were made wherein the rate of metal removal was changed from one value to another. In all cases the slope of the stress-strain curve changed reversibly.

It will be noticed in Figs. 10 and 11 (points A) that whenever the rate of metal removal was changed from a low value to a high value, a large drop in load occurred. This drop in load is due to an elongation of the specimen and could be reproduced at will during any of the three stages of deformation. In no case was there a large increase in load when R was changed from a high to a low value. The drop in load is very rapid and starts immediately when R is increased. A series of investigations showed that the large drop in load was not due to magnetic or electrical interactions with the strain gages of the load cell. Since an increase in temperature of the specimen could possibly cause such a change, an extensive program was undertaken to determine whether this factor was important. Thermocouples were placed along the surface and in the center portion of the specimens. For the time period during which the drop in load occurred, no change in temperature could be noted. A change in temperature could not have

been responsible, for the drop in load is evident from the rapidity of the change of load and the fact that no change of load occurred when the current through the cell was changed from a high to a low value. The entire drop in load took place in less than 0.1 second. In Fig. 11 at point A, the current through the cell was changed from 1/2 to 2 amperes while at point B the change was from 2 to 1/2 amperes. Changing the specimen from anodic to cathodic did not produce a sudden decrease in the load when the current was varied.

Having shown that the work-hardening coefficient within all three stages was reversible, it was of interest to determine whether the original work-hardening state could be recovered by polishing off a given amount of the surface. For this portion of the study, crystals of aluminum 37 were strained within the Stage I region and then polished, usually after the load had been removed, for various periods of time. In the initial experiment only 0.001 and 0.004 of an inch were removed. No effect on the critical resolved shear stress was found. There was, however, an increase in the extent of Stage I for a specimen from which 0.004 of an inch had been removed. Normally, ϵ_2 for this specimen would have been 1%; however, upon retesting, ϵ_2 was 1.5%. The effect of removing 0.041 inch from the surface on the critical resolved shear stress is shown in Fig. 12. This specimen was deformed in the electrolytic polishing bath and zero voltage maintained until a strain of 0.5% at a shear stress of 192 psi was reached. The critical resolved shear stress was 120 psi. The load was then removed and the specimen polished, after which the width was measured with a traveling microscope. Upon reloading, again at zero voltage, the critical resolved shear stress became 120 psi and the slope θ_1 was the same as that before the metal was removed. The end of Stage I in this case occurred at a total strain of 1.25% as compared to 0.75 to 0.80% obtained on specimens pulled without removal of the surface. The extent of Stage I obtained after the removal of the surface layers was 0.75% and compares favorably with that obtained on virgin specimens. A recovery in the critical resolved shear stress, by removal of the surface layers, was also found in KCL crystals by T. Suzuki (Ref. 32).

The experimental data show that the extent and slopes of Stages I and II as well as the stress at which Stage III begins are markedly affected by the removal of the surface during tensile deformation. In general, the extent of Stages I and II is increased and the slopes decreased as the rate of metal removal is increased. For a constant rate of metal removal, increase in the strain rate decreases the extent of Stages I and II and increases the slopes. The critical resolved shear stress does not seem to be affected by the rate of metal removal; however, the stress at which Stage III begins is decreased. The stress at which Stage I ends appears to be constant. Within the range of the rates of metal removal, used in this investigation, the slopes of all three stages of deformation are reversible; however, it has not been possible, thus far, to change the slope of Stage II to that of Stage I. By polishing off the surface of the specimen, there appears to be a complete recovery of the work-hardening stage of crystals deformed in the Stage I region.

From the foregoing experimental observations that the slope continues to decrease with increasing polishing rate, it appears that in Stage I the major portion of the work-hardening coefficient of face-centered cubic metals is due to dislocation barriers at the surface. Internal obstacles such as Lomer-Cottrell barriers apparently exert a minor influence. The observations that the original value of the critical resolved shear stress can be recovered after deformation by removing a given amount of the surface also show that the dislocations blocked by the surface do not extend throughout the cross-section of the specimen.

In the Stage II region, both internal obstacles and the surface appear to contribute to the work-hardening coefficient. It was possible to decrease the slope of Stage II about 25% when the rate of metal removed was 50×10^{-5} inch per minute and presumably it may have been decreased somewhat more with faster polishing rates. Since it has not been possible to produce a marked decrease in the work-hardening coefficient by removing the surface layers of the specimen, the internal obstacles apparently thread the entire cross-section of the crystal.

Stage III is also affected to a large degree by surface barriers. In the example given it was shown that 60% change in the slope of Stage III occurred when

the rate of removal was changed from 12.5×10^{-5} to 50×10^{-5} inch per minute. In some cases, it was also possible to change the slope of Stage III from a negative to a positive value by decreasing the rate of removal of the metal. Apparently, if in Stage III the work-hardening is associated with the cross slip of screw dislocations as suggested by Seeger (Ref. 24), then a large percentage of the obstacles are at the surfaces as well as in the interior of the crystal.

The rapid decrease in load which occurred whenever the rate of removal of the metal from the surface was changed from a low to a high value seems to be associated with a dislocation "pop out" phenomenon similar to that found by Barrett (Refs. 11, 12) on the untwisting of wires. Apparently, during the deformation process, only a certain number of dislocations leave the crystal and a number of dislocations remain in a piled-up array at the surface. When the current density is increased, the surface energy is decreased, especially at the piled-up sites, and the dislocations run out in an avalanche which causes a sudden elongation in the specimen.

From the observations of this investigation, it is clear that the surface plays a very important role in the plastic deformation process of single crystals. Apparently during the deformation process in Stage I, dislocations may pile up or form tangles at the surface. In Stage II, internal obstacles which may form first at the surface and then later in the interior appear to be formed only after the piled-up array of dislocations sources on secondary systems. This latter effect is indicated by the fact that the end of Stage I occurs at the same stress regardless of the slope θ_1 . Once internal obstacles are formed, the slope θ_2 is governed primarily by these barriers. However, as indicated by the drop in τ_3 , the effectiveness of the barriers is governed to an appreciable extent by the surface.

It is not entirely clear how the surface blocks the egress of dislocations and forms an effective barrier. Under usual test conditions there is present on the surface of the specimens an oxide film which can retard the passage of dislocations out of the crystal. The effectiveness of the oxide film in retarding dislocation escape depends upon the nature and thickness of the film. It also appears possible that a clean surface may impede the egress of dislocations from the crystal. Experiments (Ref. 14) on gold and platinum, metals which do

Contrails

not have oxide films, have shown that the creep behavior could be increased by testing in solutions containing surface-active agents. There is further evidence from the observation that the extent of Stage I for gold crystals is far less than that which would be expected on the basis of applied shear stress on the secondary slip system. In the present experiments on aluminum crystals pulled in an electrolytic polishing bath, it is not known whether the observed effects are associated with the oxide or hydrate film on the surface. These effects may be due to a weakening of these barriers with increasing current density or directly to the removal of the aluminum surface. The latter effect also appears possible since it is known the aluminum ions can migrate easily through the anodic film. A series of experiments using gold crystals is planned to determine whether the plastic flow characteristics are affected by surface-active agents and by plating off the surface during deformation.

It was found that the extent of Stages I and II was increased and their slopes decreased as the rate of metal removed from the surface was increased. Increasing the strain rate caused a decrease in the extent of Stages I and II and increased the slopes. The critical resolved shear stress and the stress, τ_1 , at which Stage I ended appeared to be constant regardless of the surface treatment; however, the stress, τ_3 , at which Stage III began was lowered as the rate of metal removed was increased. Within all three stages of deformation, the work-hardening coefficients were reversible. It was possible to produce a complete recovery of the work-hardening state, if the deformation was confined to the Stage I region. A phenomenon which may possibly be due to a "pop out" of dislocations was observed whenever the rate of metal removed was changed from a low to a high value.

From the experimental observations it appears that the slope and extent of Stage I is determined primarily by the conditions which exist on the surface of the crystal. In Stages II and III both the surface effects and internal barriers determine the plastic flow characteristics. Apparently, the starting point for the formation of internal barriers is at the surface; however, once the internal barriers are formed they thread the entire cross-section of the specimen.

In this paper it has been shown that the surface effects are important to the mechanical deformation of single crystals pulled in tension. It is believed that these surface effects are important to theories of creep and fatigue.

1. R. Roscoe, Nature 133 (1934) 912
2. R. Roscoe, Phil. May 21 ser. 7 (1936) 399
3. A. F. Joffe, Physics of Crystals (1929) M-L
4. A. H. Cottrell and D. F. Gibbons, Nature 162 (1948) 488
5. S. Harper and A. H. Cottrell, Proc. Phy. Soc. B. 63 (1950) 331
6. J. Takamura, Mem. Fac. Eng. Kyoto Univ. 18 (3) (1956) 255
7. E. N. da C Andrade and D. Henderson, Phil. Trans. Roy. Soc., A244 (1951) 177
8. J. J. Gilman and T. A. Read, Trans. AIME 194 (1952) 875
9. J. Garstone, R. W. K. Honeycombe and G. Greetham, Acta Met. 5 (1956) 485
10. S. Shapiro and T. A. Read, Phy. Rev. 82 (2) (1951) 341
11. C. S. Barrett, Acta Met. 1 (1953) 2
12. C. S. Barrett, Trans AIME 197 (1953) 1652
13. P. A. Rehbinder and E. K. Wenstrom, Z. Fich (1) 19 (1945 Acta Physiochim) URSS 11 (144) 417 Dokl ANSSSR (2) 68 (1949)
14. A. Pfutzenreuter and G. Masing, Z. Metallkunder 42 (1951) 361
15. E. N. da C Andrade and R. F. Y. Randall, Nature 162 (1948) 890
16. E. N. da C Andrade, Nature 164 (1949) 538
17. D. J. Phillips and N. Thompson, Proc. Phy. Soc. (B) (1950) 839
18. E. N. da C Andrade and R. F. G. Randall, Proc. Phy. Soc. (B) 65 (1952) 445
19. J. J. Gilman, ASTM STP No. 171, Phila., Pa. (1955) 3
20. F. R. Lipsett and R. King, Proc. Phy. Soc. (B) 70 (6) (1957) 608
21. L. C. Weiner and M. Gensamer, Jrnl. Inst. Metals 85 (1956-57) 441
22. M. A. Adams, Acta Met. 6 (1958) 327
23. B. Chalmers and R. S. Davis, Conf. Dislocations Mech. Properties of Crystals, 1956 Lake Placid, John Wiley and Sons, Inc. (1957) 232
24. A. Seeger, "Dislocations and Mechanical Properties of Crystals," John Wiley and Sons, Inc. (1957) 243
25. S. Diehl, S. Mader and A. Seeger, Phil. Mag. 2 (1957) 323
26. J. Friedel, Phila. Mag. 46 (1955) 1169
27. A. H. Cottrell and R. S. Stokes, Proc. Roy Soc. (London) A, 233 (1955) 17
28. A. H. Cottrell, "Deformation and Plastic Flow of Solids," Springer-Verlag, Berlin (1956) 33

29. A. N. Stroh, Phil Mag. (1956) 1, 489
30. F. D. Rosi, Trans. AIME 200 (1954) 1009
31. J. Garstone and R. W. K. Honeycombe, "Dislocations and Mechanical Properties of Crystals," John Wiley and Sons, Inc., (1957) 391
32. T. Suzuki, "Dislocations and Mechanical Properties of Crystals," John Wiley and Sons, Inc., (1957) 215

TABLE 1
Relationship Between Current Density and Rate of
Removal of Aluminum from the Surface

Current (amp)	Current Density (amp/sq in.)	Rate of Metal Removal (in./min x 10 ⁵)
0.5	0.417	12.5
1.0	0.835	25.0
2.0	1.67	50.0

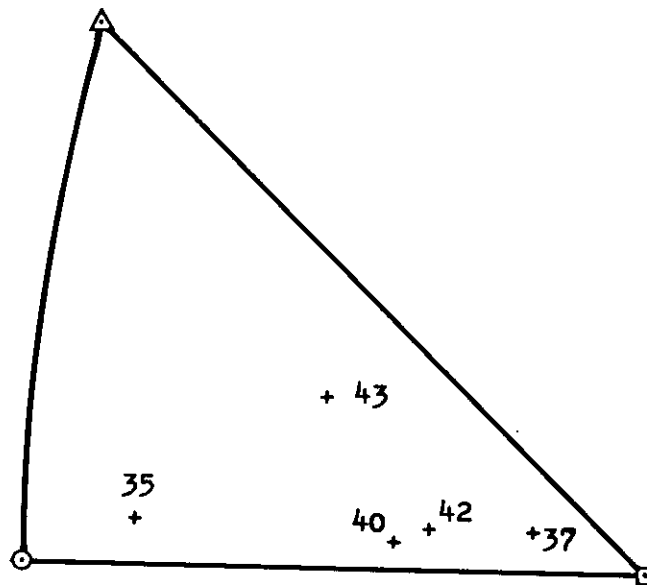


Fig. 1. Orientation of Aluminum Crystals

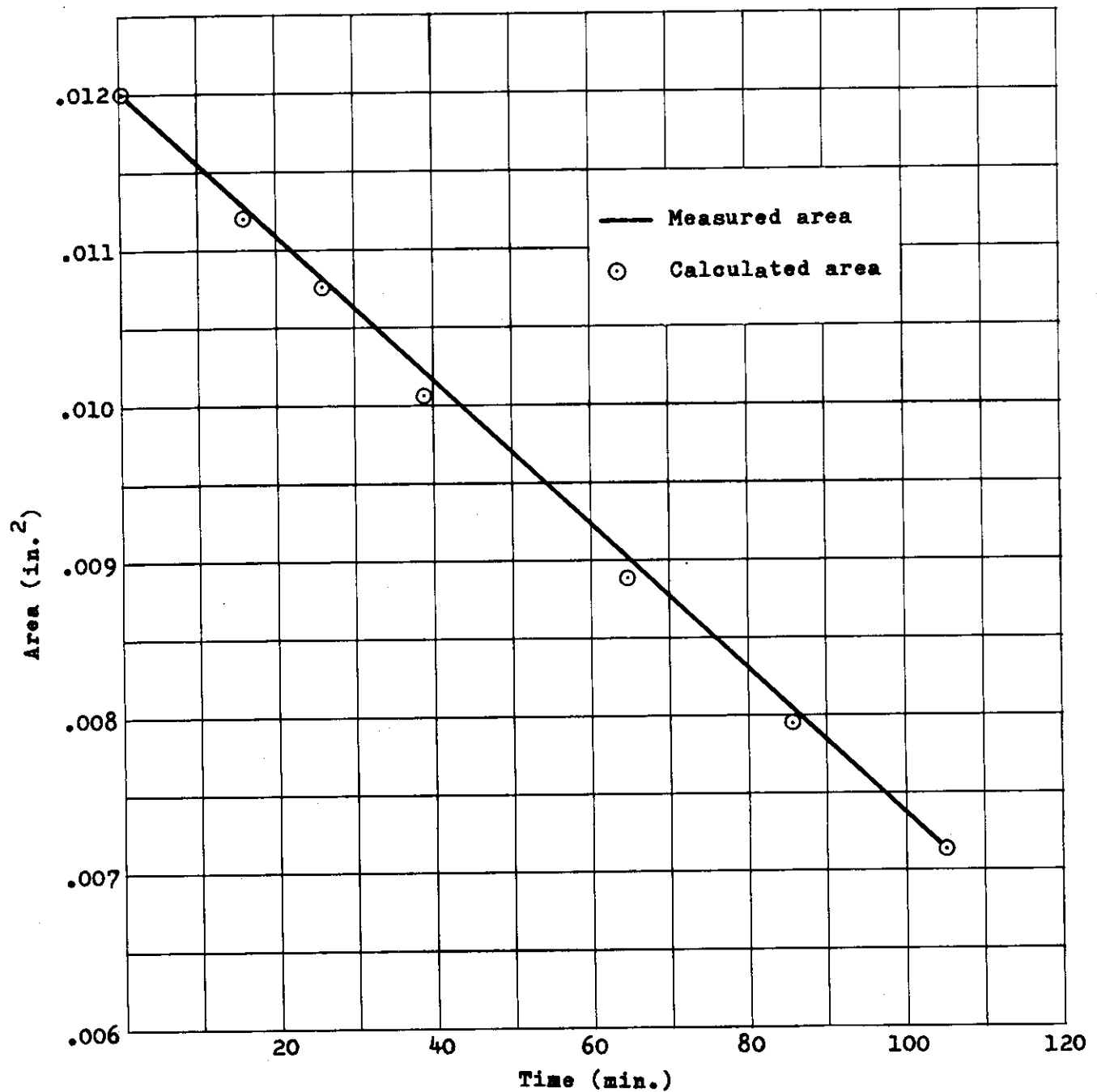


Fig. 2. Comparison of Calculated and Measured Cross-Sectional Area of Aluminum Specimens Pulled in Electrolytic Polishing Bath (rate of metal removal was 25×10^{-5} in./min.).

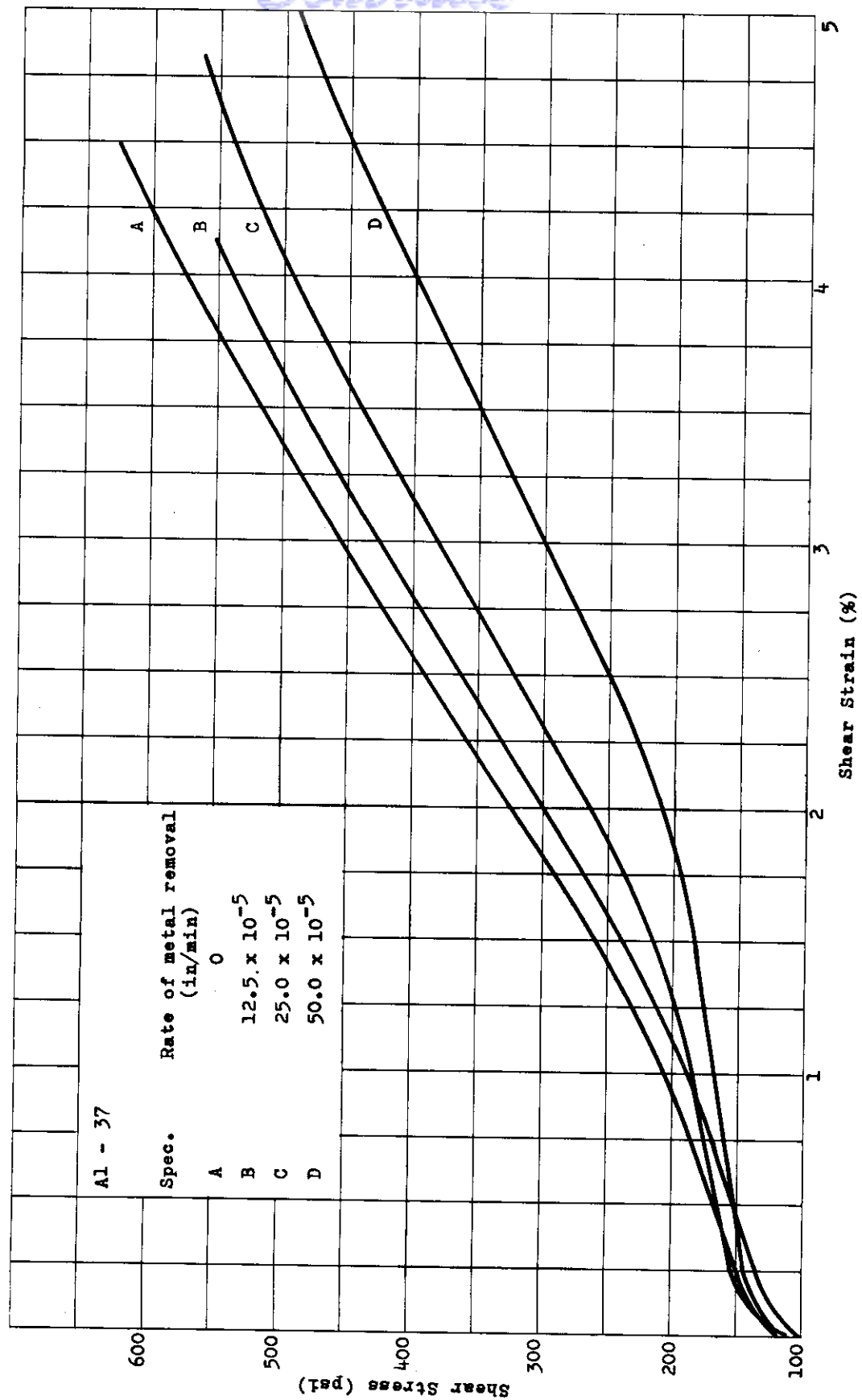


Fig. 3. The Effect of Rate of Removal on the Stress-Strain Curves of AL-37 (temperature 3°C and strain rate 10^{-5} sec $^{-1}$).

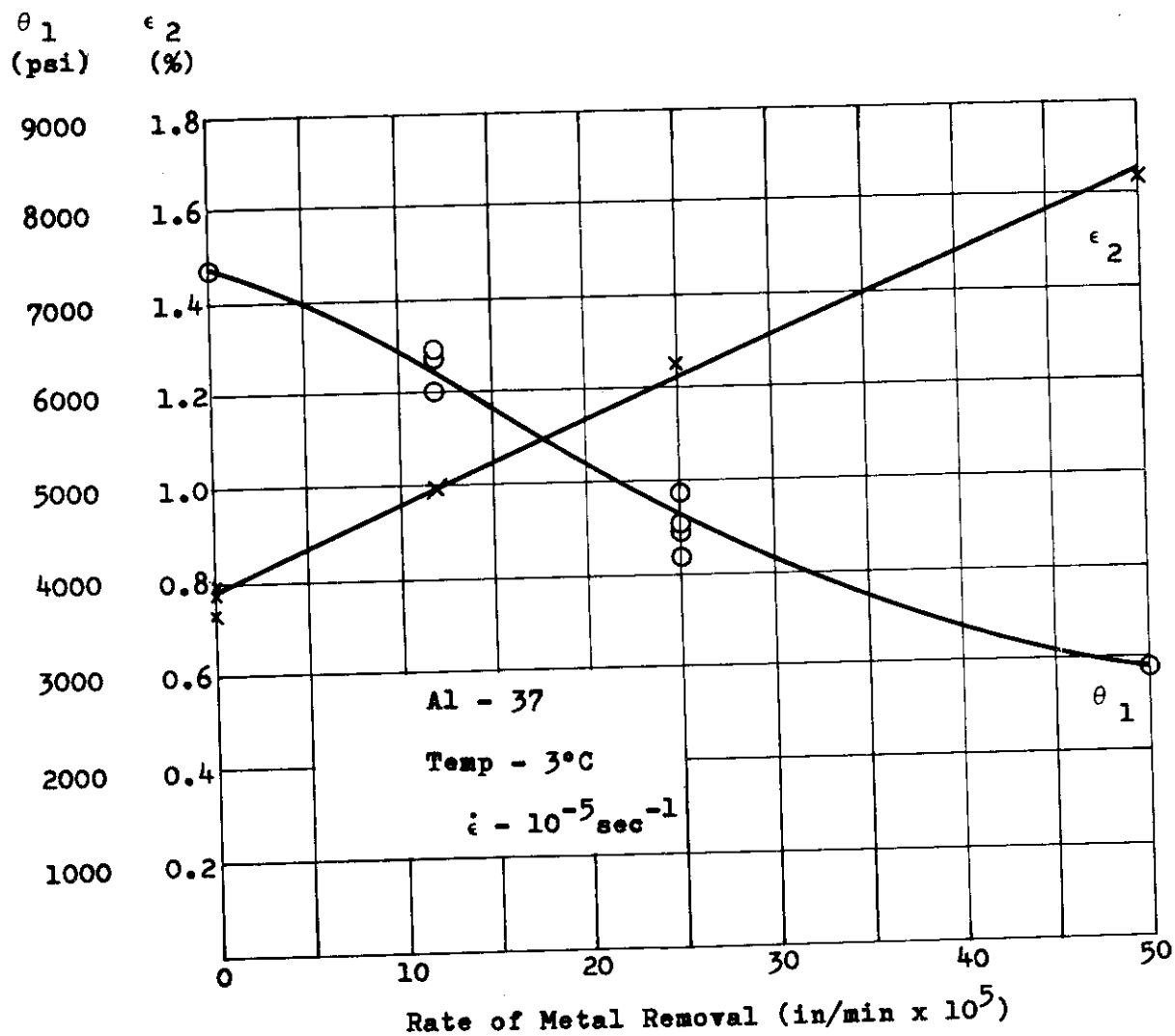


Fig. 4. The Effect of Rate of Removal on the Extent (ϵ_2) and Slope (θ_1) of Stage I of Al-37.

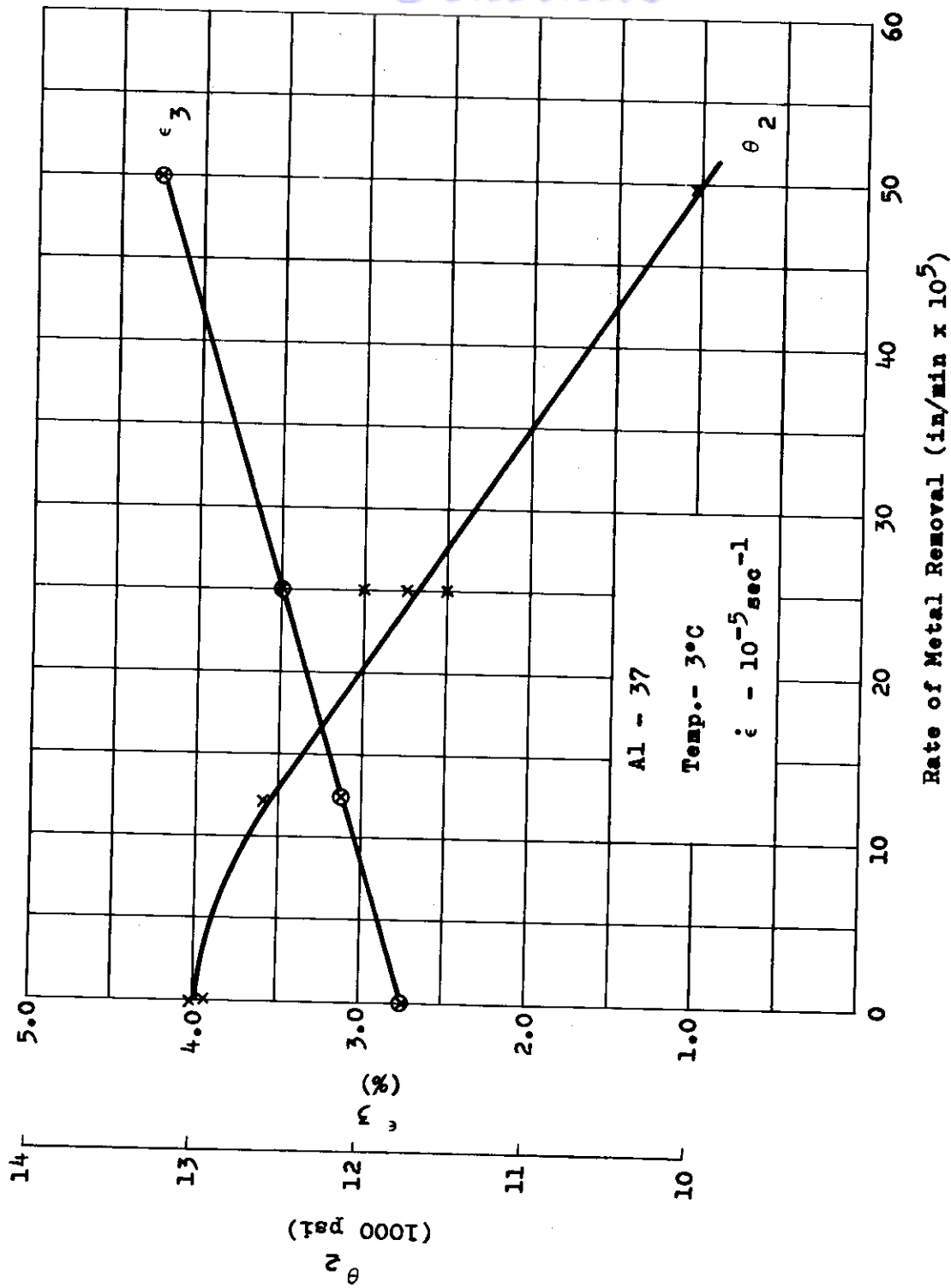


Fig. 5. The Effect of Rate of Removal on the Extent (ϵ_3) and Slope (θ_2) of Stage II of Al-37.

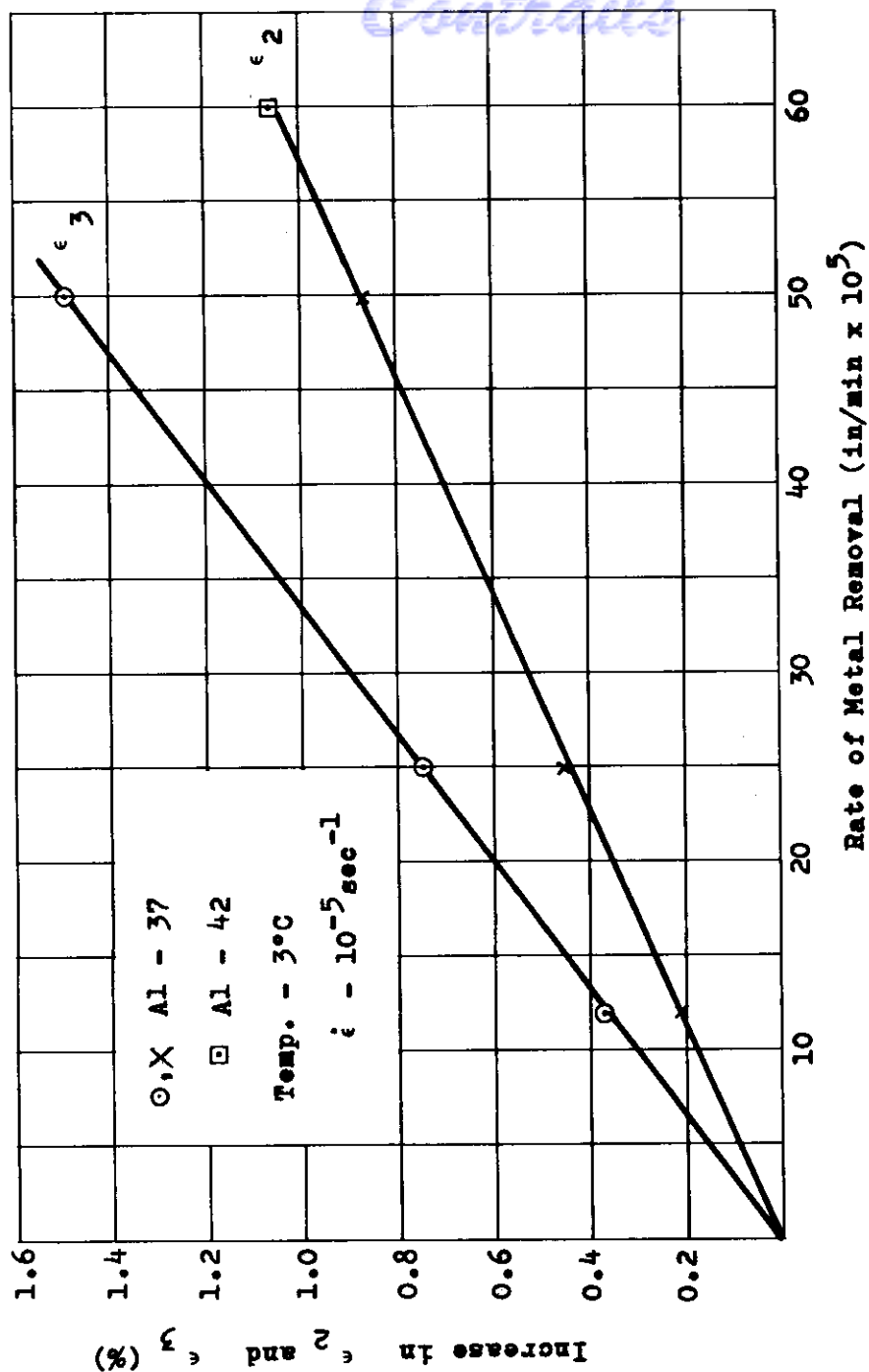


Fig. 6. The Effect of Rate of Removal on the Increase of ϵ_2 and ϵ_3 .

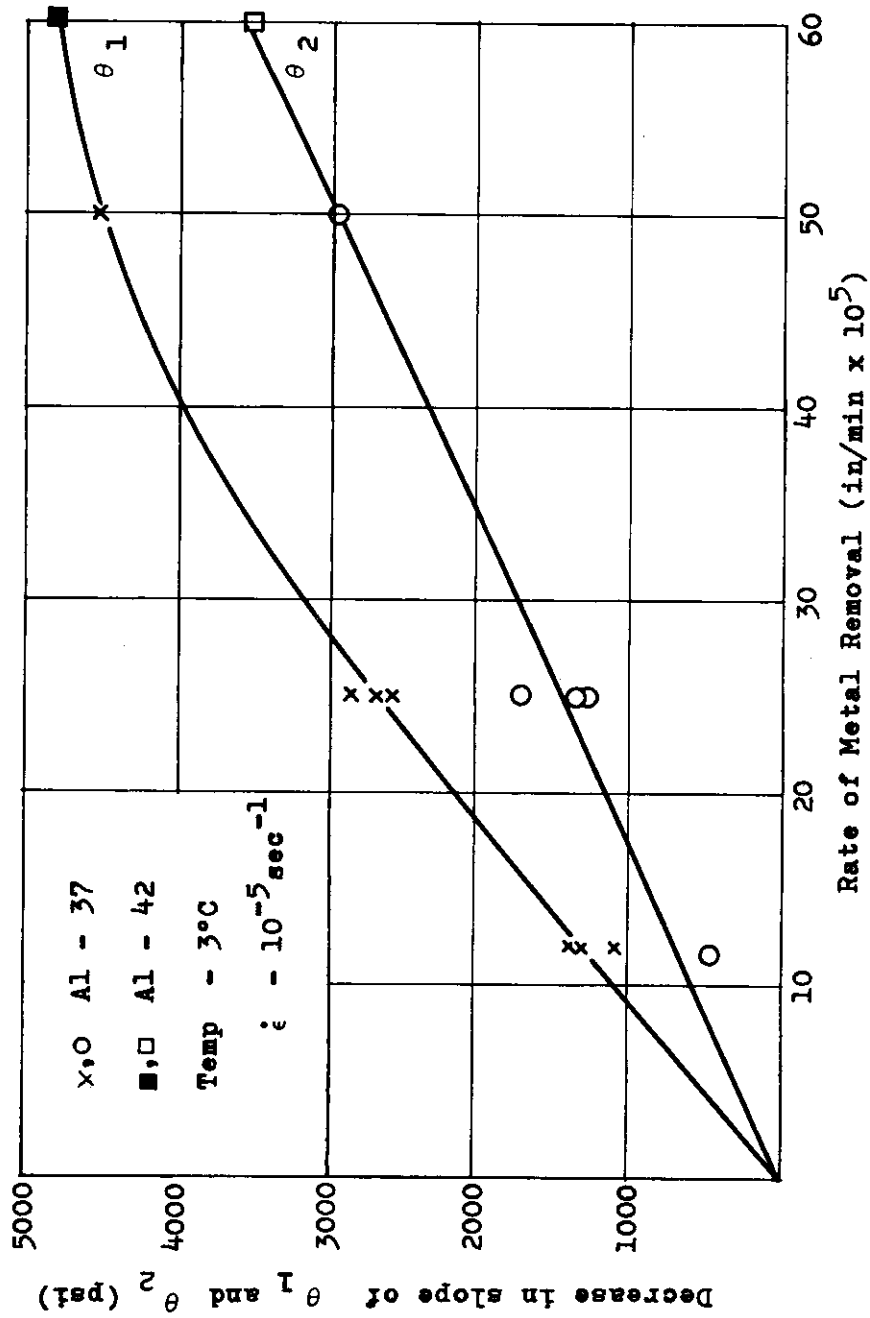


Fig. 7. The Effect of Rate of Removal on the Decrease in Slopes θ_1 and θ_2 .

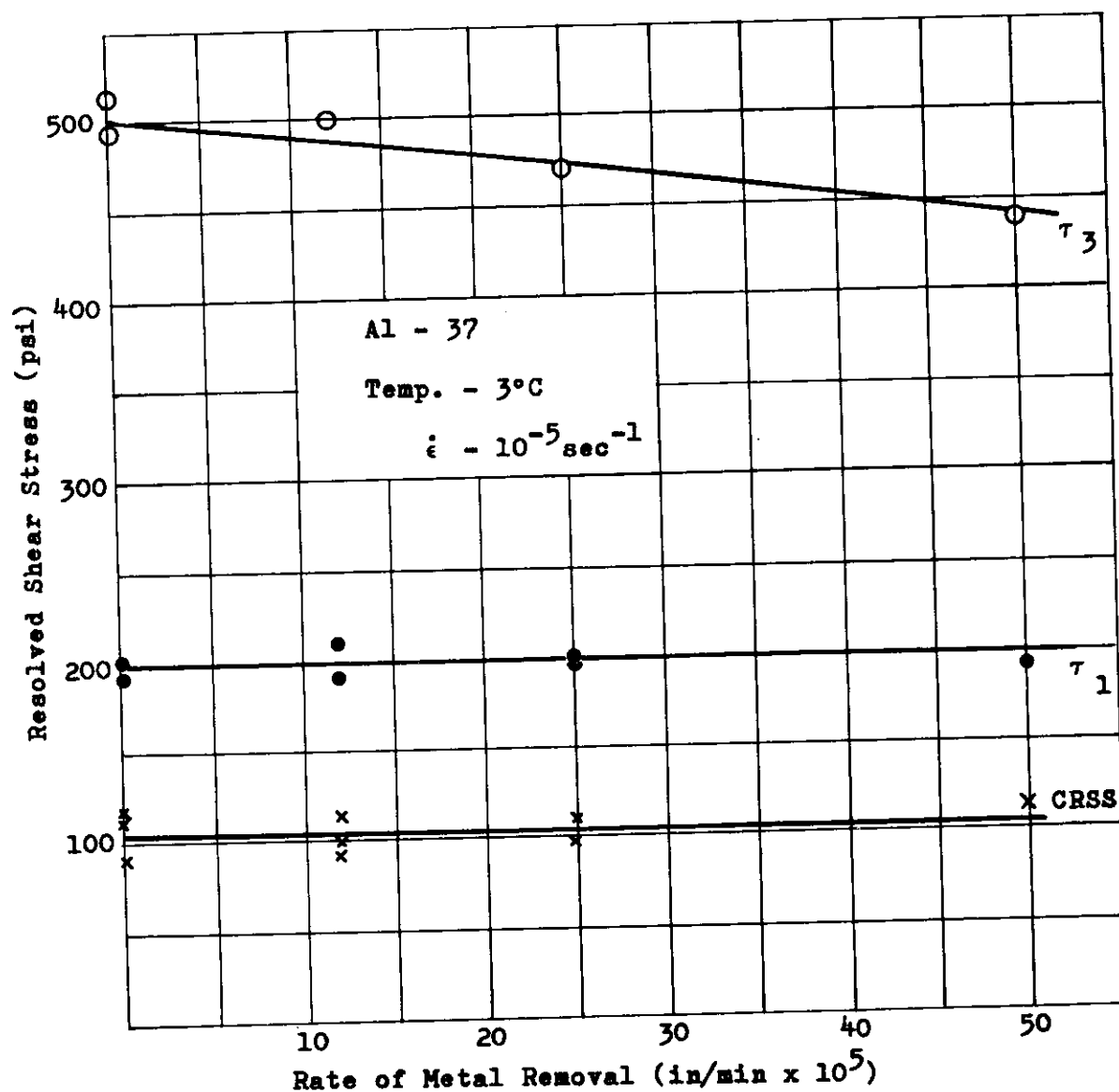


Fig. 8. The Effect of Rate of Removal on the Critical Resolved Shear Stress, the Stress at the End of Stage I, τ_1 , and the Stress at the End of Stage II, τ_3 .

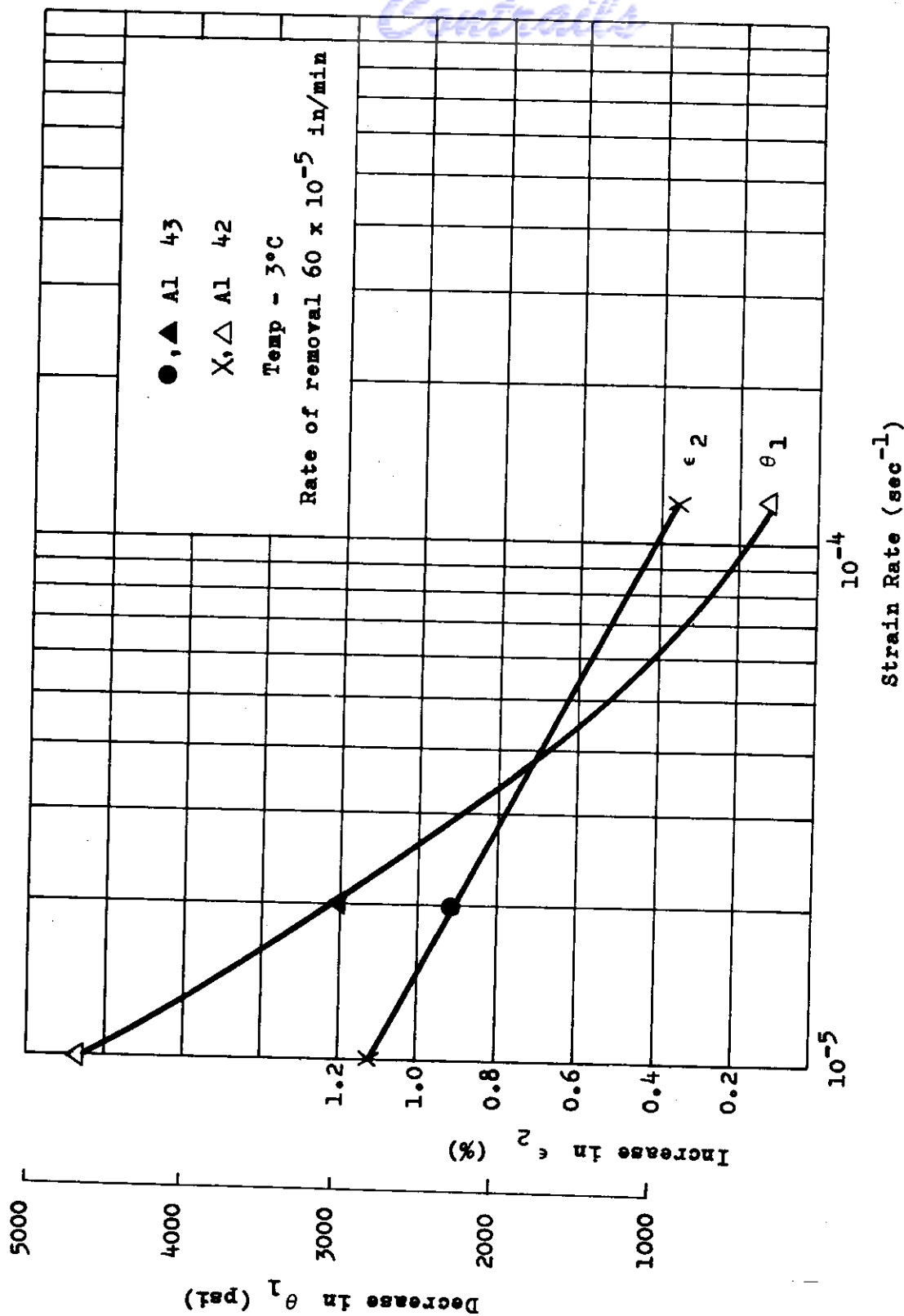


Fig. 9. Effect of Strain Rate on the Increase in ϵ_2 and Decrease in θ_1 .

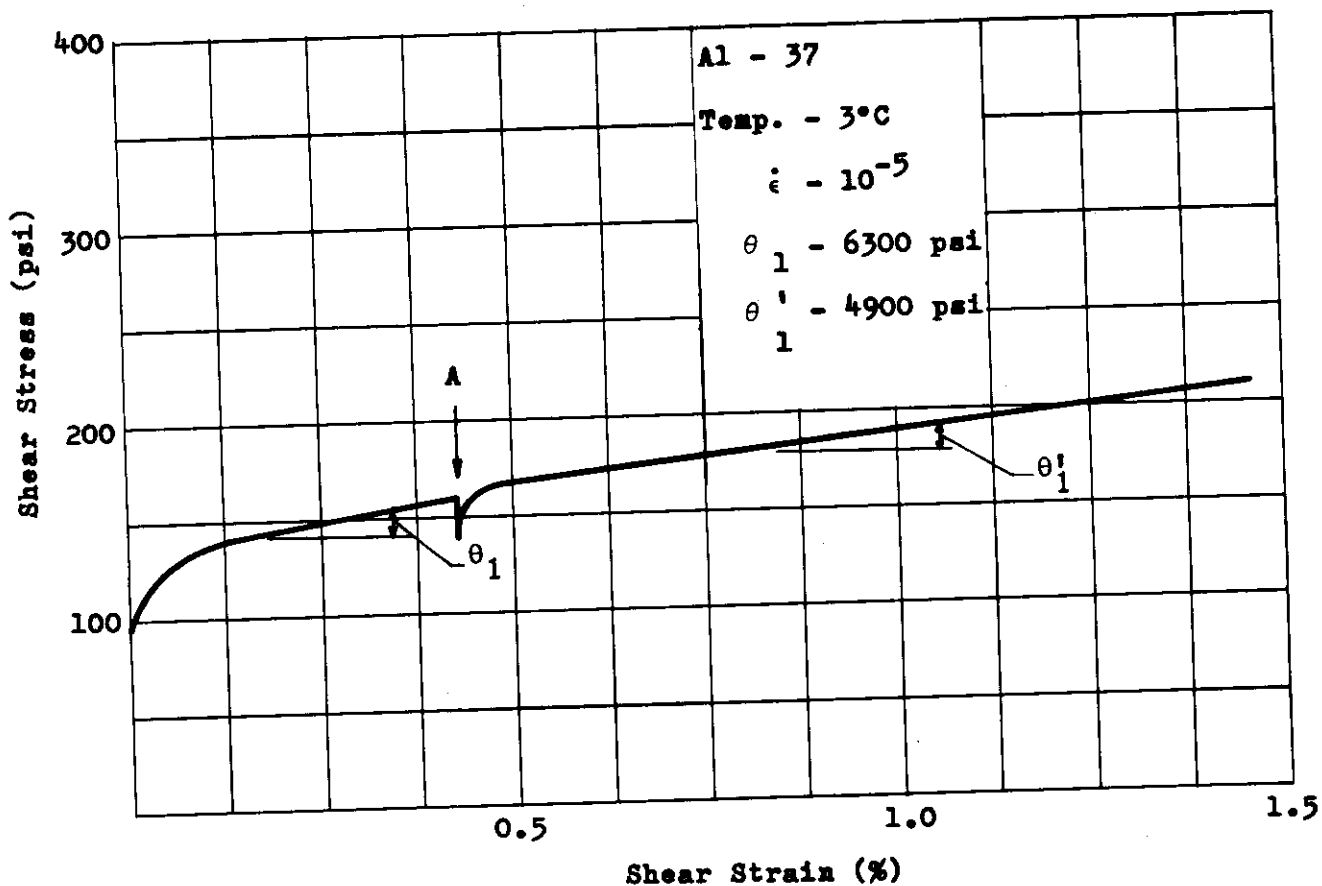


Fig. 10. The Effect of a Change in the Rate of Removal on the Slope in Stage I (at A, the rate of removal was changed from 12.5×10^{-5} to 25×10^{-5} in./min.).

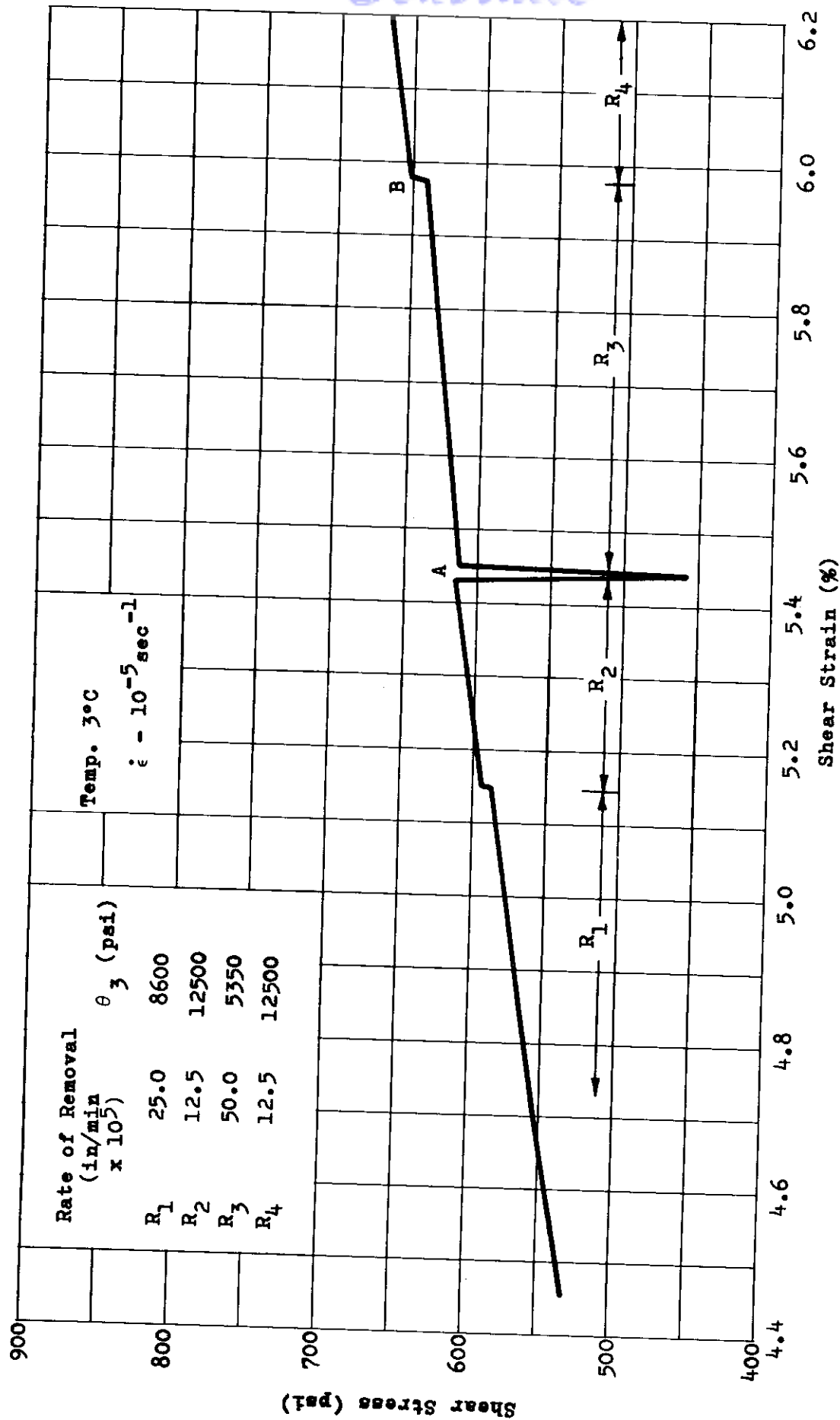


Fig. 11. The Effect of Change in the Rate of Removal on the Slopes in Stage III.

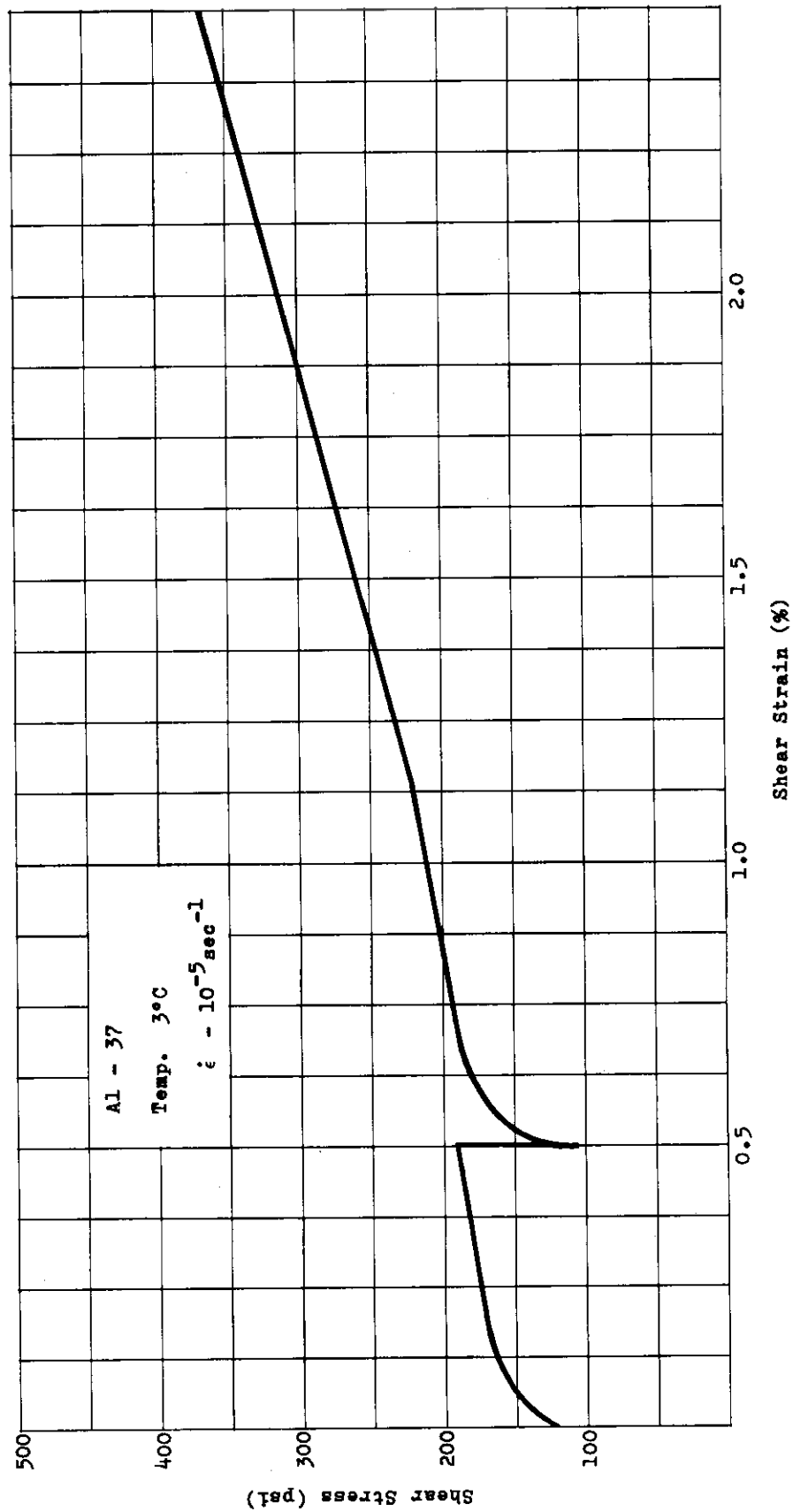


Fig. 12. The Effect of Removing 0.041 In. from the Transverse Dimensions of the Specimen on the Recovery of Work-Hardening.

Contrails