

**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 Synthethetic 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. II

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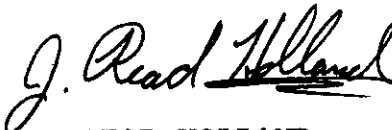
ABSTRACT

Single crystals of aluminum were pulled in tension in a solution of paraffin oil and stearic acid. The critical resolved shear stress did not change with the concentration of the stearic acid solution; however, the extent and slopes of Stages I and II were affected greatly. The observations lend evidence that the weakening effect of surface-active agents is controlled by the rate of desorption of the metal soap formed by the reaction of the surface-active agent and the metal surface.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:



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## I. INTRODUCTION

It has been shown by various investigators that surface-active agents markedly affect the mechanical properties of single crystals. (A surface-active agent, for the purpose of this report, is defined as a long chain organic molecule which contains one or more polar groups. Usually, however, investigations have considered only molecules having one polar group.) In general, when single crystals are deformed in some solutions containing surface-active agents, the creep rate is increased and the yield strength is decreased. The fatigue strength has been reported to be affected also.

Rehbinder first found an effect of surface-active substances in 1928 in connection with the pendulum hardness of minerals. Later Rehbinder (Ref. 1) and Rehbinder and Wenstrom (Ref. 2) observed that the flow of lead, tin and copper sheets under constant load was much faster in solutions containing small amounts of surfactants (cetyl alcohol, N-Valeric, N-Heptoic, stearic, oleic, palmitic and cerotic acids). The relationship (Refs. 2, 3 and 4) which was found is of particular interest. Figure 1 shows the effect on the creep behavior of tin single crystals and Table 1 gives the results on the yield strength. The data show that the yield strength first decreases with increasing concentration of the solution and then increases. The creep properties follow the same general behavior as a function of concentration. The maximum ( $C_m$ ) decreases with increasing concentration of the solution and then increases. The creep properties follow the same general behavior as a function of concentration. The maximum ( $C_m$ ) decreases with increasing molecular weight of the organic homologues by a factor of 1.3 to 1.4 for each  $-CH_2$  link. The nature of the solvent was also reported to be important.

Lichtman, et al. (Ref. 5) in his work on tin and lead single crystals, reported that both temperature and strain rate considerably influenced the activity of the surface-active agents on the flow characteristics. At 20° C, the maximum effect occurred at a strain rate of about 250% per minute. When the temperature was increased to 100° C, the curve was displaced and the maximum occurred at 1000% per minute. In these experiments, the decrease in rupture stress was taken as a measure of the adsorption effect. The type of surface-active agent used did not seem to change the deformation rate at which the maximum effect occurred. Oleic acid, palmitic acid and cetyl alcohol produced the same strain rate maximum for lead crystals.

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Manuscript released for publication March 1961 as a WADD Technical Report

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The above effects are believed by Rehbinder, et al., to show evidence that physical adsorption is important. It was stated that while oleic and palmitic acids react with lead to form chemical compounds (lead oleate and lead palmitate), cetyl alcohol does not. The decrease in the adsorption effect was explained by a change in the type of adsorption. It was supposed that at the higher temperatures, chemical adsorption took place and decreased the mobility of the adsorbed molecules so that fewer molecules were able to enter the microcracks which were postulated to exist on the surface.

Harper and Cottrell (Ref. 6), in their study of the effect of surface conditions on the deformation characteristics of zinc single crystals tested in a medium containing an electrolyte or a surface-active agent, inferred that the role of the polar molecule was to remove the strengthening effect of the oxide coating. Contrary to the results obtained by Rehbinder and his co-workers, they reported that the creep behavior of zinc crystals was affected by the addition of oleic acid to paraffin oil only when the specimens had an oxidized surface.

Klinkenberg, Lucke and Masing (Ref. 7) reported data which confirmed the results of Rehbinder, et al. Using zinc single crystals which had been cleaned by electrolytic polishing, a large increase in the creep rate was observed when the specimens were tested in a 1% oleic acid-paraffin oil solution. Similar results were found for cadmium crystals. They also demonstrated that the creep rate of gold specimens (composed of many single crystals) was increased when the specimens were tested in 0.2% oleic acid. Andrade and his co-workers (Refs. 8 and 9) also attempted to provide an explanation of the weakening effect. Cadmium single crystals were cleaned by heating in a vacuum (pressure  $\sim 10^{-4}$  mm Hg). When subjected to creep tests, there was no creep rate change of the "cleansed" specimens when cadmium sulphate was used as an electrolyte. Rozhanskiy and Rehbinder (Ref. 10) investigated both oxide-coated and clean tin specimens in a vacuum and in rarefied vapors of various polar and nonpolar sub-coated crystals (creep was up to 100 times faster in ethyl alcohol at the optimum pressure than in a vacuum). No great effect occurred on clean crystals unless they were very short. They suggested that stress concentrations greatly assist the effect and that these occur at the ends of a clean specimen or at the cracks in the oxide film. The explanations of Andrade, Harper and Cottrell, i.e., the effect of the surface-active substance nullifies the hardening effect of the oxide film, were discounted not only because of the existence of a small effect on clean crystals but also because the strength reduction by the surface-active agent is much greater than the strengthening produced by the oxide film.

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From these various investigations, several theories have been proposed to explain the weakening effect of surface-active agents. Rehbinder and his co-workers (Ref. 11) assume that the process is associated with the existence of microcracks on the surface. According to their theory, the surface-active agents are physically adsorbed and are able to migrate into the root of the crack. A large pressure is said to be created according to the equation developed for capillary pressure,

$$\Delta P = \frac{2 \gamma \cos \theta}{r} \quad (1)$$

where  $\gamma$  is the surface energy,  $\theta$  the contact angle between the medium and the surface of the metal and  $r$  the radius at the root of the microcrack. When the radius ( $r$ ) is small, the pressure may become very large. The validity of this explanation appears questionable upon consideration of certain facts. It has been shown (Ref. 12) that high purity liquids which have a low surface tension always wet ( $\theta = 0$ ) a high energy surface of a solid. The presence of an adsorbed layer will increase the contact angle,  $\theta$ . The surface tension will also be decreased by the presence of an adsorbed layer. Thus, in the above equation,  $\Delta P$  should be decreased by the presence of a surface-active agent and not increased. Further, the maximum  $\Delta P$  should occur when a pure solvent is employed (in the case,  $\cos \theta$  is equal to one).

An explanation (Ref. 6) of the weakening effect of surface-active agents has also been given in terms of the removal of an oxide layer which would, if present, tend to increase the strength of the crystal. However, this hypothesis seemingly may be discounted since investigations of gold (which does not have an oxide layer) have shown that surface-active agents decrease the creep strength. Masing (Refs. 7 and 13) attributed the weakening effect to a decrease in the surface energy.

When a metal is immersed in a medium containing organic molecules which have a polar group, the molecule first becomes attached to the metal through dipole-image forces, and any chemical combination with the surface atoms of adsorbing metal must develop later. Dubrissay (Ref. 14) and Prutton, et al., (Ref. 15), demonstrated that fatty acids react with many metals to form salts only when a metal oxide is present. Tingle (Ref. 16) has shown that traces of water also aid in the formation of the metal salt. However, Hilton-Smith and

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co-workers (Refs. 17, 18 and 19) have recently demonstrated that clean, dry, oxide-free metals can react with fatty acids at ordinary temperatures to form soap films when the metal has been freshly scraped or cut. This mechanical activation of the surface decreases rapidly with time and will not occur with an old surface. These investigators associated the reaction with the Kramer (Refs. 20 and 21) effect. This phenomenon involves a property of freshly exposed metal surfaces in which electrons with an energy somewhat below four electron volts are made available at room temperature at a work function considerably below that associated with an aged surface. This amount of energy is sufficient to cause copper and stearic acid to react to form copper stearate. Normally, this reaction would not occur if the surfaces were clean and undeformed.

It is the intent of this paper to show that the change in mechanical behavior of single crystals is associated with chemical adsorption and not with physical adsorption. Since previous investigations were primarily confined to creep statics or to tensile test in which the various deformation stages were not discernible, a study was made of the changes in the extent and slopes of Stages I and II.



## II. EXPERIMENTAL PROCEDURE

Aluminum single crystals were grown in a multiple-cavity mold using the Bridgman technique. Twenty-six specimens (1/8 inch x 1/8 inch x 4 inches) having the same crystallographic orientation were obtained (Fig. 2). The aluminum used in the preparation had a purity content of 99.997% and the orientations were determined by the back-reflection Laué technique. The specimens were mechanically polished in a fixture to prevent bending, electropolished and then vacuum annealed ( $\sim 10^{-5}$  mm Hg) for two hours at 600° C. After the vacuum-annealing treatment, the specimens were again electropolished just prior to testing.

The specimens were pulled in a hydraulic machine equipped with gimbaled attachment rods. The ends of the specimens were copper plated, then coated with a thin layer of solder and then placed in an aligning fixture which held teflon-coated steel specimens holders. They were fixed in place by filling the cavity between the specimen and specimen holder with a low-melting lead-bismuth alloy. The exposed surfaces of the seal were then coated with paraffin. The container which held the solution was constructed from teflon. Before the preparation of the solutions used in this investigation, the paraffin oil was passed through a column containing activated alumina and activated silica. The paraffin oil was tested for the presence of polar molecules by placing a drop of the oil on a clean surface of water adjusted to a pH of 2 and 11. The oil was considered free from polar molecules when the drop did not spread within 15 minutes.

During the course of the tensile test, the load was measured by a proving ring equipped with SR-4 strain gages and the motion of head was measured by a high frequency magnetic transducer. With this apparatus it was possible to measure elongations within  $3 \times 10^{-5}$  inch and loads of 0.1 pound. In all cases the tests were conducted at room temperature.

In addition to the tensile deformation tests, specimens were deformed approximately 1% and placed in various solutions containing surface-active agents. The solutions were allowed to stand at room temperature for 16 hours and were then analyzed for the presence of metal soaps.

## III. EXPERIMENTAL RESULTS

The data on the presence of metal soaps found in the solutions containing various surface-active agents, into which deformed specimens had been immersed, indicate that the solubility of the metal soaps is important in determining the weakening effect of such polar molecules (Table 2). In every case where a surface-active agent has been reported to affect the mechanical behavior of a metal, a metal soap is found in solution. Contrary to a previous statement (Ref. 5) that cetyl alcohol formed only a physical adsorbed layer, an analysis of such a solution showed the presence of lead cetylate. It is also seen from the data of Table 2 that the presence of metallic soaps in solution depends not only on the reacting molecule but also on the solvent. Soaps were not found in solutions when specimens were immersed in saturated solutions of the surface-active agent.

The data obtained from the stress-strain curves of aluminum crystals deformed in paraffin oil containing varying concentrations of stearic acid are summarized in Table 2 and in Figs. 3 and 4. Figure 5 shows the critical resolved shear stress obtained when the specimens were deformed in the stearic acid solutions. The critical resolved shear stress was defined to be the stress at which the stress-strain curve first became nonlinear. Also plotted in Figs. 3 and 4 are the results obtained from a specimen pulled in a paraffin oil bath containing 0.002 mol/liter of stearic acid and 0.00001 mol/liter of aluminum stearate.

As may be seen from Fig. 5, the critical resolved shear stress does not change with the concentration of stearic acid in paraffin oil; however, the extents and slopes of Stages I and II do change (Figs. 3 and 4). The strain,  $\epsilon_2$ , at the edge of Stage I at first increases with increasing concentration and then, at a concentration of 0.002 mol/liter of stearic acid, decreases. The strain,  $\epsilon_3$ , at the end of Stage II or the beginning of Stage III follows the same behavior as  $\epsilon_2$ ; however, the slope,  $\theta_1$ , of Stage I and the slope,  $\theta_2$ , of Stage II decrease until a concentration of 0.002 mol/liter (a minimum in the curve) is reached and, with increasing concentration, these slopes increase. An analysis was made of each solution after the tensile test in an attempt to determine the amount of aluminum stearate formed. Unfortunately, the method of analysis was not sensitive enough to obtain a quantitative result.

Figures 3 and 4 and Table 3 present the deformation behavior of specimens which were pulled in 0.002 mol/liter stearic acid solution containing 0.00001 mol/liter aluminum stearate. The presence of the aluminum stearate decreased the

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effect of the surface-active stearic acid. The values for  $\epsilon_2$  and  $\epsilon_3$  are decreased and  $\theta_1$  is increased as compared to the values for specimens tested in a solution containing 0.002 mol/liter of stearic acid. The Stage II deformation for Specimen 17 appeared only as a transition region for this test condition.

Specimen 18 (Table 3) was tested in a solution containing 0.002 mol/liter of stearic acid; however, prior to the test, the aluminum stearate content was increased by allowing the specimen to react in the solution for a period of 17 hours. The values for  $\epsilon_2$ ,  $\epsilon_3$  and  $\theta_1$  were about the same as those of Specimen 17. In this case also, a Stage II region did not appear.

## IV. DISCUSSION

The experimental data derived from tensile tests of aluminum single crystals in stearic acid solutions show that the critical resolved shear stress does not change with the stearic acid concentration. This implies that surface dislocation sources are not affected by the surface-active agent, since a change in the stress necessary to activate such a dislocation source would be expected to change the critical resolved shear stress but not the plastic flow characteristics thereafter.

The plastic flow parameters,  $\epsilon_2$ ,  $\epsilon_3$ ,  $\theta_1$  and  $\theta_2$ , were, however, strongly affected by the concentration of the stearic acid solution. For each of these parameters, the maximum effect was found to occur at the same concentration (0.002 mol/liter). The results are similar to those obtained by Reh binder and his co-workers (Ref. 10) for creep and tensile tests; however, from the present data, the effect of the surface-active agents on the changes in Stages I and II may be seen. Reh binder reported his data from tensile tests in terms of yield strength since the various stages were not discernible from the stress-strain curves.

The decrease in  $\epsilon_2$  and  $\epsilon_3$  and the increase in  $\theta_1$  which occurred when aluminum stearate was added to the stearic acid solution is strong evidence that the effect of surface-active agents is controlled by the rate of desorption of aluminum stearate molecules from the surface of the specimen. The rate of desorption of metal soaps may be expected to be affected by the concentration of metal soap already in solution. Smith and Fort (Ref. 18) showed that for the case of copper and monodecanoic acid, the rate of desorption of copper soap in a cyclohexane solution saturated with copper monodecanate was much less than the rate of desorption in pure cyclohexane. The behavior of Specimen 18, pulled in a solution containing the reaction products formed as a result of immersing an aluminum specimen in the stearic acid solution, lends additional support to the concept that the rate of desorption may control the effectiveness of a surface-active agent.

In another study (Ref. 22) in which the surface of aluminum specimens was removed in an electrochemical polishing bath while the specimens were being deformed, it was found that the extent of Stages I and II was increased and the corresponding slopes decreased as the rate of removal of the metal was increased. The critical resolved shear stress was not affected. It was also possible to effect a complete recovery in the work-hardening state by plating off about 0.041 inch from the transverse dimension of the specimen after it had been strained within the Stage I region. The results from the above experiments indicated quite strongly

# Conclusions

that the extent and slope of Stage I was determined primarily by surface barriers, and internal obstacles exert a more minor role. In Stage II, both surface and internal obstacles are important. The results obtained on the changes in Stages I and II as a function of the concentration of the surface-active agents appear to be related to those obtained by electrochemical removal of the surface. However, microscopic examination of specimens exposed to surface-active agents reveals that the chemical attack was localized and dislocation pits were formed, whereas the electrochemical attack was very general. Therefore, it is unlikely that the results of the two cases can be related directly to the total amount of metal removed since the important factor may be the rate of removal at dislocation sites.

The bell-shaped curve of Figs. 1, 3 and 4 may be explained in terms of rate of desorption of the metal soap which forms as a result of the reaction between the surface-active agent and the metal. At low concentrations, the rate at which the polar molecules reach the surface is low but the solution potential of the metal soap is high. The rate of removal of the metal, therefore, is correspondingly low. As the concentration is increased, the rate of adsorption of the polar molecules is increased and the number of metal soap molecules that pass into solution is increased. However, at a certain value of concentration, the rate of solution of the soap molecules will decrease because the solution becomes more saturated. This point will correspond to the maximum of the curve. In brief, on the rising portion of the curve, the effectiveness of the surface-active agent is limited by the rate of adsorption which will increase with increasing concentration. On the falling portion of the curve, the effect is limited by the rate of desorption of the metal soap molecules. The rate of desorption will decrease with increasing concentration of the polar molecule.

For a given solvent and a given metal, in general, the solubility of the metal salt will decrease with the chain length of the organic molecule. (The solubility is, of course, modified by the presence of double and triple bonds, side-branched groups, etc.) Thus it is expected, as shown in Fig. 1, that the maximum in the curve moves to lower concentrations as the chain length of the molecule increases.

As pointed out earlier, some experimental results seem to indicate that a surface-active agent does not affect the mechanical behavior of "clean" crystals. Gold seems to be the exception. Since the rate of desorption of the metal soap molecules appears to influence the effectiveness of the surface-active agent, two factors become important: (1) the energy of the system must be high enough to

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allow the soap reaction to occur, and (2) the energy of adsorption must not be so high that the soap molecules do not dissolve in the solvent. From the work of Dubrissay (Ref. 14) and Prutton (Ref. 15) it is shown that metal soap will not form unless an oxide is present. This may account for the observations that the mechanical behavior of clean crystals of zinc is not affected by an oleic acid-paraffin oil solution. However, since Hilton-Smith (Refs. 17, 18 and 19) has shown that wet working the surface of clean metals may promote the formation of metal soaps, it appears possible that under certain conditions, using the proper solvent and surface-active agent, an effect on the mechanical behavior of clean crystals may be observed, i. e. , gold.

V. CONCLUSIONS

The critical resolved shear stress for aluminum single crystals does not change as a function of the concentration of the solution. Changes in the extent of strain and slopes of Stages I and II are, however, found to occur and there is an optimum concentration at which the surface-active agent has the largest effect. The fact that the addition of aluminum stearate decreases the weakening effect of stearic acid lends strong evidence that the rate of desorption of the metal soap is the controlling factor governing the effectiveness of the surface-active agent. At low concentrations of the surface-active agent, the rate of desorption is limited by the relatively small number of stearic acid molecules that are adsorbed on the surface of the specimens. At high concentrations, the rate of desorption is limited by the solubility of the metal soap in the solution.

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TABLE 1

Effect of Additions of Oleic Acid to Vaseline Oil on the  
Variation of Yield Point of Tin Crystals  
(Ref. 11)

Tin Single Crystals

D = 0.76 - 96 mm

$41^\circ \leq X_o \leq 48^\circ$

V = 4.8%/min

Mechanical Properties

Oleic acid concentration in vaseline oil

	(% )				
Yield Strength (gm/mm <sup>2</sup> )	0	0.1	0.2	0.5	1.0
	250	210	145	180	200

TABLE 2

Formation of Metal Soaps by the Reaction of Single Crystals in Solutions  
of Various Surface-Active Agents

(Specimens compressed 1% and immersed in solution for 24 hours)

<u>Conc.</u> (mol/liter)	<u>Surface-Active</u> <u>Agent</u>	<u>Solvent</u>	<u>Results</u> (Presence of Soaps) (P=Soap Present, N=Soap Abs.)
<u>ZINC</u>			
0.25	Caprylic Acid	Hexadecane	P
0.10	Caprylic Acid	Benzene	N
0.10	Caprylic Acid	Paraffin Oil	P
0.50	Capryl Alcohol	Paraffin Oil	N
Sat'd	Stearic Acid	Benzene	N
0.62x10 <sup>-3</sup>	Stearic Acid	Hexadecane	N
0.01	Stearic Acid	Benzene	N
0.001	Stearic Acid	Paraffin Oil	N
0.005	Stearic Acid	Paraffin Oil	N
0.006	Oleic Acid	Paraffin Oil	P
<u>COPPER</u>			
0.10	Caprylic Acid	Benzene	P
0.05	Caprylic Acid	Hexadecane	P
0.10	Caprylic Acid	Paraffin Oil	N
Sat'd	Stearic Acid	Benzene	N
0.50	Capryl Alcohol	Paraffin Oil	N
0.10	Stearic Acid	Benzene	P
0.001	Stearic Acid	Paraffin Oil	N
0.005	Stearic Acid	Paraffin Oil	N
0.006	Oleic Acid	Paraffin Oil	P

TABLE 2 (continued)

<u>Conc.</u> <u>(mol/liter)</u>	<u>Surface-Active</u> <u>Agent</u>	<u>Solvent</u>	<u>Results</u> <u>(Presence of Soaps)</u> <u>(P=Soap Present, N=Soap Abs.)</u>
<u>ALUMINUM</u>			
0.50	Capryl Alcohol	Hexadecane	N
0.75	Capryl Alcohol	Hexadecane	P
1.00	Capryl Alcohol	Hexadecane	P
0.50	Capryl Alcohol	Paraffin Oil	P
0.10	Caprylic Acid	Paraffin Oil	N
Sat'd	Stearic Acid	Benzene	N
0.10	Caprylic Acid	Benzene	P
$0.62 \times 10^{-3}$	Stearic Acid	Hexadecane	N
0.01	Stearic Acid	Benzene	P
0.005	Stearic Acid	Paraffin Oil	P
0.006	Oleic Acid	Paraffin Oil	P
<u>LEAD</u>			
0.003	Cetyl Alcohol	Paraffin Oil	P

TABLE 3

The Effect of Stearic Acid-Paraffin Oil on the Various Stress-Strain  
Parameters of Aluminum Single Crystals

<u>Spec No.</u>	<u>Conc. of Stearic Acid (mol/liter)</u>	<u><math>\theta_1</math> (psi)</u>	<u><math>\theta_2</math> (psi)</u>	<u><math>\epsilon_2</math></u>	<u><math>\epsilon_3</math></u>	<u>CRSS (psi)</u>
10	0.0005	2560	5280	2.40	4.9	160
12	0.001	2410	3550	2.85	6.0	170
11	0.002	1620	3200	3.70	8.0	160
14	0.003	2060	4050	2.84	6.1	160
13	0.004	2540	4375	2.00	5.3	170
17	0.002 + 0.00001 mol/l aluminum stearate	1930	----	2.40	4.9	160
18	0.002 <sup>x</sup>	1960	----	2.40	4.9	165

CRSS = Critical Resolved Shear Stress

$\tau_1$  = Shear stress at end of Stage I

$\tau_2$  = Shear stress at end of Stage II

x = Specimen immersed in stearic acid solution for 17 hours to increase aluminum stearate concentration. Tested in same solution.

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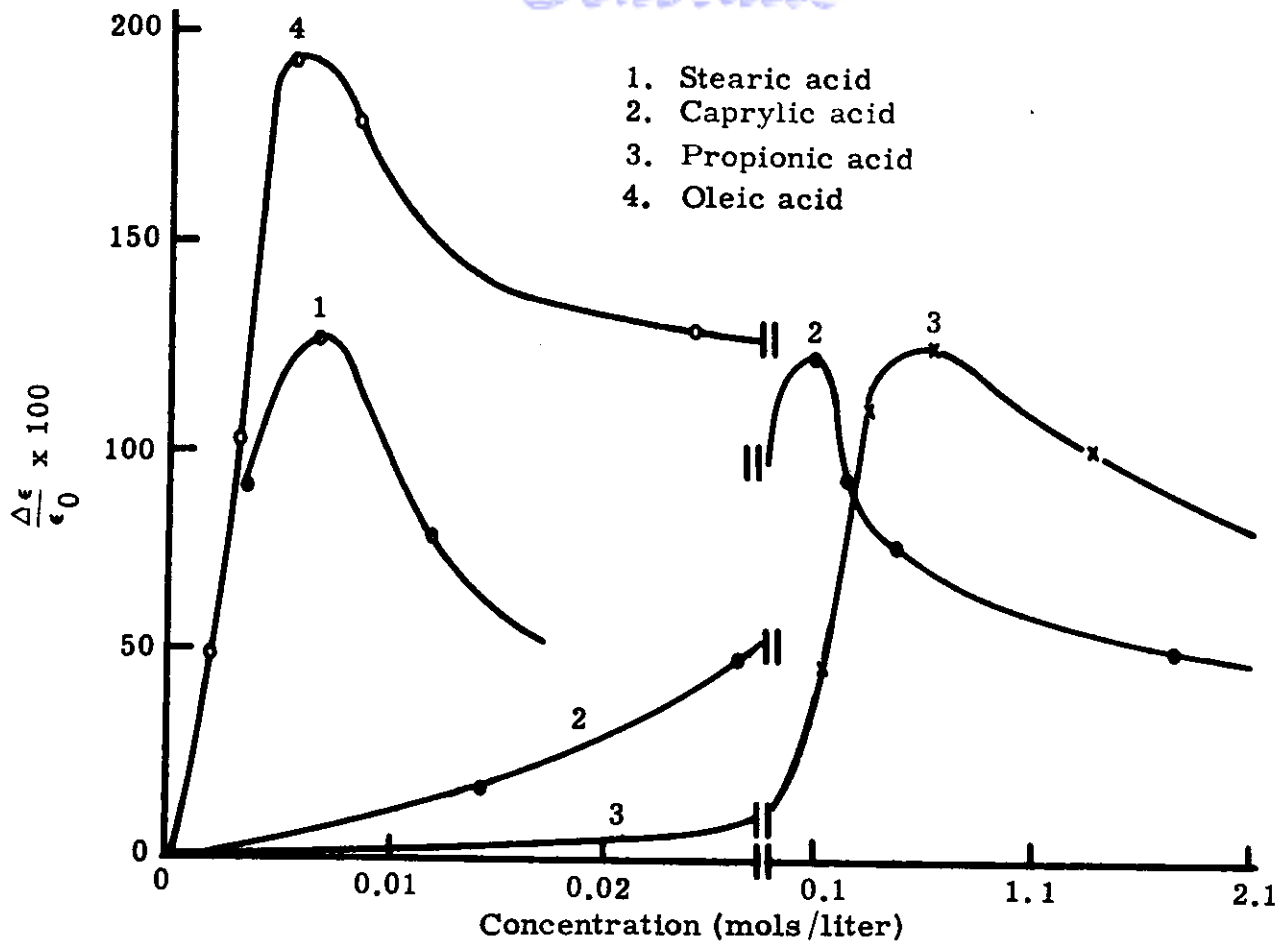


Fig. 1. Absorption Effect (Creep Acceleration) in Tin Single Crystals Against Concentration of Surface-Active Substance (Octane Used as Solvent) (Ref 11)

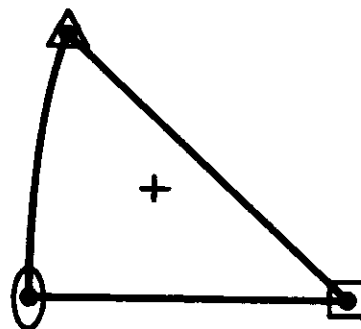


Fig. 2. Orientation of Aluminum Specimens 24

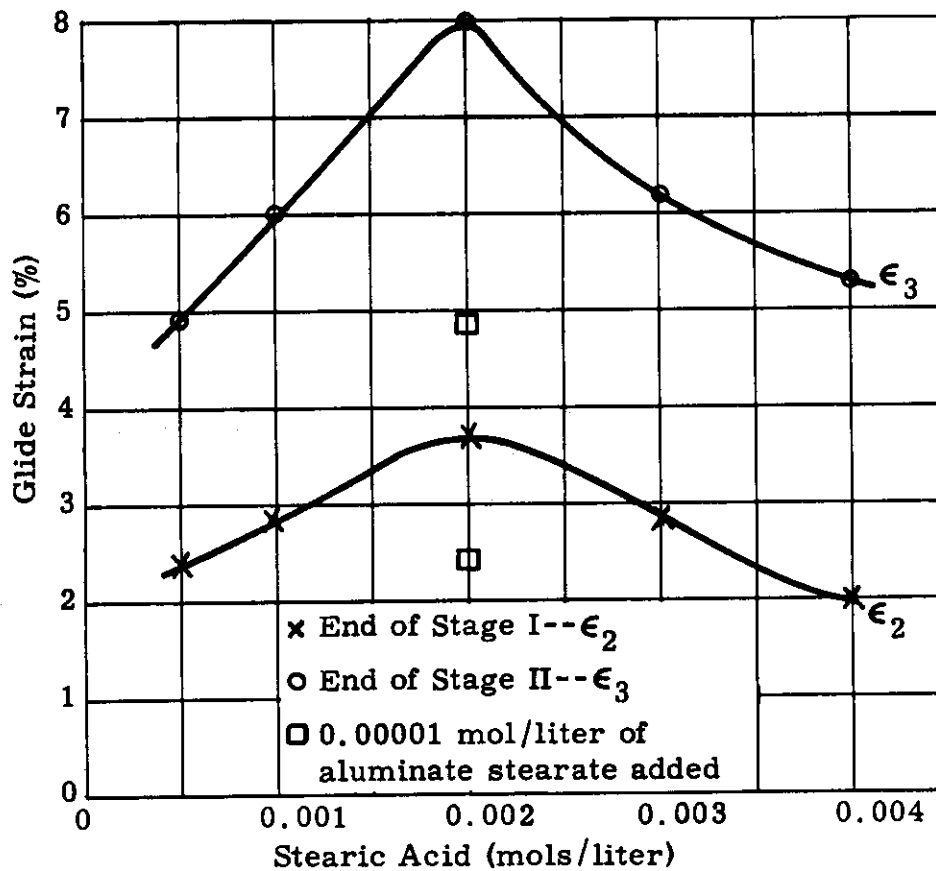


Fig. 3. Effect of Concentration of Stearic Acid in Paraffin Oil on Extent of Stages I and II of Aluminum Single Crystals 24

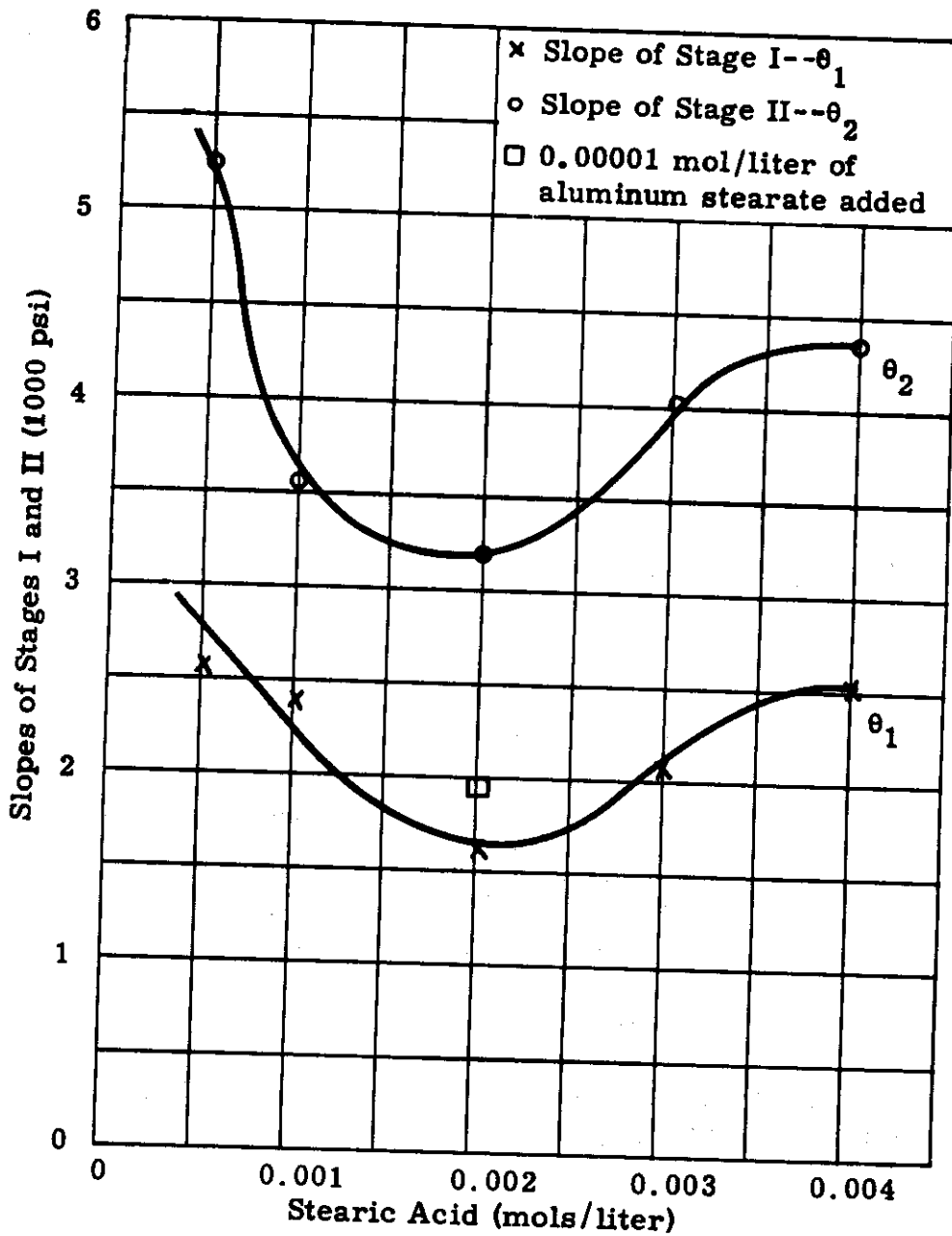


Fig. 4. Effect of Concentration of Stearic Acid on Slopes of Stages I and II of Aluminum Single Crystals 24

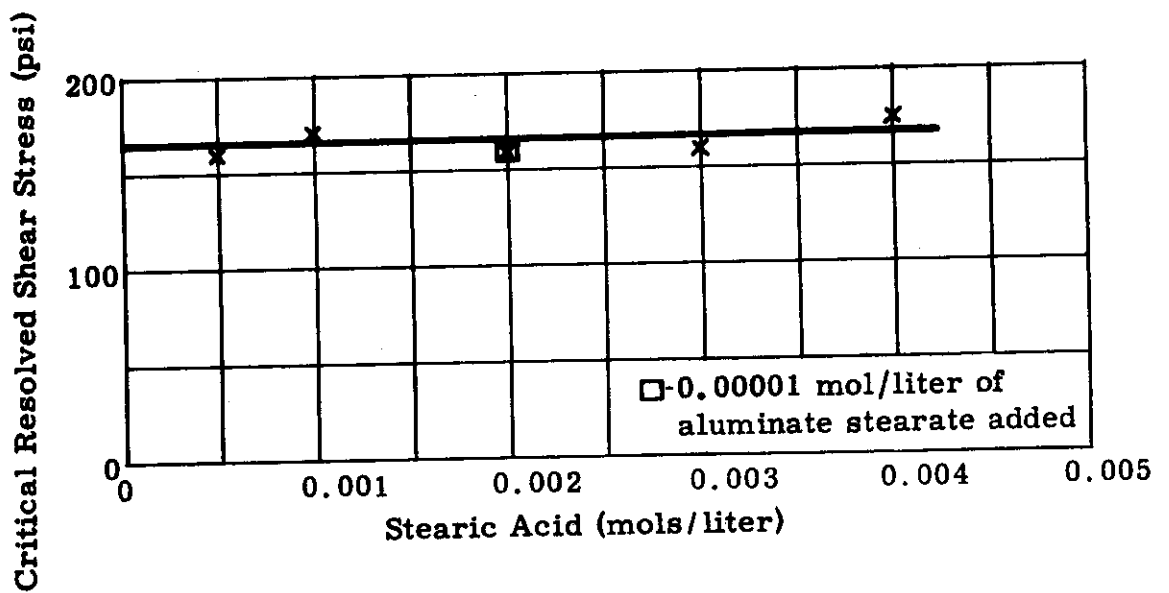


Fig. 5. Critical Resolved Shear Stress of Aluminum Single Crystals 24 in a Stearic Acid-Paraffin Oil Solution