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# THE EFFECT OF TEMPERATURE ON THE YIELD STRENGTH OF THE POLYCRYSTALLINE HEXAGONAL Ag-AI INTERMETALLIC PHASE

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December 18, 1961

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#### ABSTRACT

The effect of temperature on the yield strength of the polycrystalline hexagon al Ag-Al intermetallic phase was investigated over the temperature range 77 to 775 K. It was found that the curve for yield stress vs temperature for both polycrystalline Ag-33 at. % Al specimens that were heavily cold worked prior to deformation and those that were recrystallized prior to deformation was parallel to that for prismatic slip in single crystals.

Increase of the percent Al in the specimens resulted in an abrupt decrease in the ductility at a composition of about 37 at. % Al. This decrease in ductility was attributed to precipitates in the grain boundaries.

#### THE EFFECT OF TEMPERATURE ON THE YIELD STRENGTH OF THE POLYCRYSTALLINE HEXAGONAL Ag-Al INTERMETALLIC PHASE

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#### INTRODUCTION

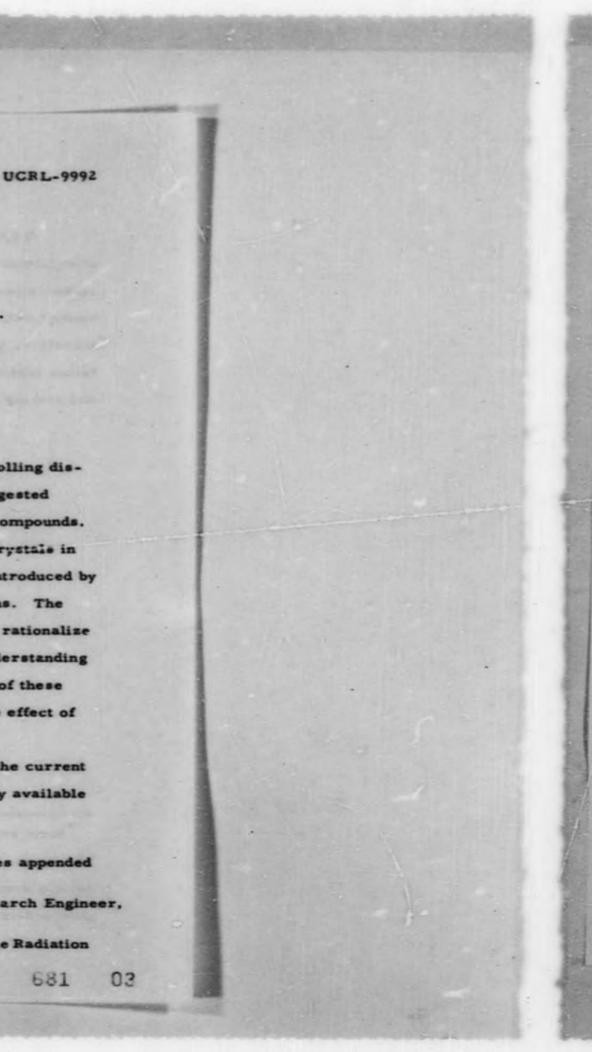
The current success in uncovering the strain-rate-controlling dislocation mechanisms for plastic deformation in pure metals suggested the extension of such investigations to alloys and intermetallic compounds. Many of the previous studies in this area were made on single crystals in order to simplify the analyzes by eliminating the complexities introduced by the presence of grain boundaries and by variously oriented grains. The ultimate goal of these investigations, however. is to completely rationalize the plastic behavior of polycrystalline aggregates through an understanding of the fundamental behavior of single crystals, the modification of these properties imposed by the presence of grain boundaries, and the effect of random orientation of the grains in the aggregate.

The H. C. P. Ag-Al intermediate phase was selected for the current investigation because of the extensive single-crystal data already available and continuing detailed studies of this system (1).

<sup>1</sup>The figures appearing in parentheses pertain to the references appended to this paper.

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It has been demonstrated that at least five independent mechanisms of deformation are necessary to satisfy the conditions imposed by the general strain tensor at the grain boundary (2). Most H. C. P. metals thus far examined exhibit only four independent slip mechanisms<sup>\*</sup> at low temperatures; therefore, what ductilities are found therein must arise from additional minor mechanisms of deformation such as twinning, grain boundary shearing, and kinking.

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Some recent evidence (3.4) indicates that certain H. C. P. metals deform by slip on  $\{11\overline{22}\}$   $\langle 11\overline{23} \rangle$ . This system has a Burger's vector which is not coplanar with other known slip systems, and therefore provides additional independent mechanisms of deformation.

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## EXPERIMENTAL PROCEDURE

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The alloy for this investigation was prepared by melting appropriate amounts of high-purity Al (99.995 at. %) and high-purity Ag(99.99 at. %) in an induction furnace sustaining an argon atmosphere, then chill casting the molten material into a copper mold to produce a 1700-g ingot of Ag-33 at. % Al. The ingot was hot forged to a 1  $\times$  1 in. square bar after being soaked at approximately 600 C for 45 min. Rectangular specimen blanks of about  $1/8 \times 1/4$  in. cross section were cut from the ingot by means of a slitting saw on a milling machine. The specimen blanks were cold rolled to reduce the thickness 15%, and finally a gauge section was milled in the central portion of the blanks.

One group of specimens prepared as above, and hereafter referred to as Type I specimens, was prestressed to 95,000 psi at room temperature prior to the execution of tension tests over the temperature range 77 to 775 K. Another group of specimens, hereafter described as Type II specimens, was recrystallized to produce an average grain size of 0.09 mm by annealing at 775 K for 30 min.; tension tests were performed over the same temperature range as for Type I specimens.

In addition, a few special experiments were conducted to determine deformation mechanisms and the effect of a change in composition on the mechanical behavior of the material. The details of the specimen preparation and experimental procedure for these experiments will be described later in this report.

#### **RESULTS AND DISCUSSION**

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#### Type I and Type II Specimens

The effect of temperature on the yield stress and strain to fracture of Type I and Type II specimens is shown in Fig. 1. Also, the yield stress for basal slip and prismatic<sup>\*</sup> slip of single crystals of the same composition is shown for comparison. For convenience of discussion the temperature effect will be described in terms of four regions documented in Fig. 1.

The stress-strain curves for Type I specimens are given in Fig. 2. those for Type II specimens in Fig. 3.

Regions I and II: Over Region I (see Fig. 1) the yield stress decreased with increasing temperature, whereas over Region II the yield stress was insensitive to temperature for both Type I and Type II specimens in a manner approximately parallel to that for prismatic slip in single crystals. (The terminal temperature for Region II was lowest for Type I specimens, intermediate for Type II specimens, and highest for single crystals oriented for prismatic slip. The reason for this will be discussed in the next section.) Therefore, the rate-controlling deformation mechanisms for polycrystalline Ag-33 at. % Al over Regions I and II is the same as that for prismatic slip in single crystals. Mote et al. (1) showed that prismatic slip in Region I might be ascribed to the Peierls' mechanism as proposed by Lothe and Hirth (5), whereas in Region II the results are in good agreement with Fisher's(6) short-range order mechanism. More detailed experiments by our group are now in progress to clarify the behavior in Region I.

<sup>\*</sup>The stress plotted for the single crystals in Fig. 1 is the stress that existed on the plane normal to the tensile axis when yielding occurred in single crystal oriented with the plane of deformation at 45<sup>°</sup> to the tensile axis.

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The yield stress for Type I specimens was about 1.8 times that for prismatic slip in single crystals, whereas the yield stress for Type II specimens was only about 1.07 times that for prismatic slip in single crystals over both Regions I and II. Thus, the grain boundaries (Type II specimens) in these coarse-grained specimens have only a minor effect on the yield stress, whereas severe cold work (Type I specimens) increases the flow stress by less than a factor of two. The stress-strain curves for Type I specimens exhibited no work hardening, as revealed in Fig. 2, while on the contrary the stress-strain curves for Type II specimens exhibited work hardening until necking or fracture, as revealed in Fig. 3. The stress at necking or fracture for Type II specimens was always less than the yield stress for Type I specimens. Microscopic examination revealed that severe twinning existed in Type I specimens, but only minor twinning could be detected near the corners of grain boundaries in Type II specimens. Grain boundaries are expected to increase the strength of a metal because of two main factors: first, the grain boundaries are barriers to slip, and second, the geometric requirement of continuity at the grain boundary imposes complex deformation modes within the grains (7). The first of these factors is expected to be important during the early stages of deformation, but not at large strains. There is little increase in the yield strength of the Type II specimens over that of single crystals, since both strengthening factors are insignificant in these large-grained specimens. The work hardening observed in the Type II specimens is undoubtedly due to pile up of dislocations at the grain boundaries. The yield strength of the Type I specimens may be attributed to three factors: (a) hardening due to the prior cold work, (b) hardening due to the smaller grain size, and (c) hardening due to the additional barriers introduced by the severe twinning in the interior of the grains. Since the

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Type I specimens have been severely cold worked, the grain boundaries are no longer effective barriers, due to the large stresses that exist at the head of the piled up dislocations; wherefore, there is no work hardening in these specimens.

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The strain to fracture or necking, as revealed in Fig. 1, was about constant for both Type I and Type II specimens over Region II, and was smaller over Region I, with Type II specimens showing the greater ductility over both Regions.

Region III: Over Region III (see Fig. 1) the yield stress decreased precipitously with increasing temperature. Mote et al. (1) ascribed this behavior in single crystals oriented for prismatic slip to the superposition of two effects. First, the degree of order decreases with increasing temperature; therefore, the yield stress will decrease in proportion. Second, since diffusion occurs reasonably rapidly in this region, fluctuations in the local degree of order (and composition) will allow dislocations arrested at these barriers to push through at lower stresses thandictated by the equilibrium degree of order (and composition). The fact that the initial temperature decreased with increasing yield stress may arise from two effects. Recovery may account for the observed behavior, or the apparent activation energy for deformation mechanism operative in this region may be a function of stress. Therefore, the higher yield stress of Type I and Type II specimens would induce deformation by this mechanism at a lower temperature.

The stress-strain curves for both Type I (Fig. 2) and Type II (Fig. 3) specimens exhibited strain softening in Region III. This undoubtedly arose from the disordering that accompanies slip.

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The strain to fracture or necking for Type I specimens remained at a very low value until near the middle of Region III, whereupon it increased markedly. The increased ductility was probably due to other deformation or recovery mechanisms which became operative at these temperatures. Somewhat surprisingly, the strain to fracture for Type II specimens decreased from the value of about 12% over Region II to a value of about 5% over the first half of Region III, then increased in much the same manner as the Type I specimens. The reason for this behavior was not clear.

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## Deformation Mechanisms Operative in Polycrystalline Ag-33 at. % Al

A special experiment to determine the operative deformation mechanisms in polycrystalline Ag-33 at. % Al at room temperature was performed as follows:

a. A specimen prepared in the usual way was recrystallized to produce a grain diameter of about 0.70 mm to facilitate Laue back-reflection photographs of selected grains.

b. The specimen was deformed about 6% and metallographic examination of 15 grains was conducted.

The results of this experiment are given in Table 1.

Basal slip, prismatic slip, and twinning was observed. There was no evidence of grain-boundary shearing. Although two of the grains exhibited three different mechanisms of deformation, the remaining grains exhibited only one or two mechanisms of deformation.

The resolved shear stress that existed on the various slip planes is also given in Table 1. In every case it was found that the resolved shear stress on the observed operative slip plane was about equal to or greater than that necessary to cause slip in single crystals of this material. In a few cases,

e.g., Grain No. 7, the resolved shear stress on a slip system was greater than that for slip in single crystals, yet no slip was observed. The reason for this was not clear; however, there may have been sufficient error in the orientation determination to account for this discrepancy, the stress field of the dislocation arrays on the other operative slip systems may have prevented slip from occurring on this system, or slip may have actually occurred but was not detected.

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#### Effect of Change in Composition on Yield Strength and Ductility

An ingot of Ag-40 at. % Al was prepared by the same procedure as for the previous samples; however, it was impossible to forge the ingot as it failed in a brittle manner. In order to study the effect of change in composition on the yield strength and ductility, specimen blanks were produced by chill casting  $1/4 \times 1/8$  in. rectangular bars and chemically milling gauge sections in these blanks. The yield strength and strain to fracture of specimens tested at room temperature are given in Fig. 4. Although there is some scatter in the data, it is clear that the yield strength is about constant with increasing composition, with a slight decrease in yield strength at 40 at. % Al; whereas there is an abrupt decrease in ductility in passing from 35 to 37 at. % Al. Microscopic examination of the specimens containing 40 at. % Al revealed heavy precipitation in the grain boundary, as shown in Fig. 5, but no precipitate could be detected in the specimens containing 37 at. % Al.

Single crystals of about 37 at. % Al were prepared and tested in tension at room temperature, the results of which are shown in Table 2. The results of experiments on Ag-33 at. % Al single crystals are given for comparison. These results indicate that the single crystals of Ag-37 at. % Al, though somewhat less ductile than the Ag-33 at. % Al single crystals, exhibit con-

siderable ductility. Although microscopic observation of the polycrystalline specimens containing 37 at. % Al revealed no precipitate in the grain boundary, the fracture was intergranular, as was that for specimens containing 40 at. % Al. The fracture of the specimens containing 33 and 35 at. % Al was transgranular. This strongly suggests that a very fine precipitate existed in the specimens containing 37 at. % Al. Therefore, the brittle behavior of the polycrystalline material containing 37 at. % or more of Al is probably due to the precipitate in the grain boundaries. Indeed, the reduction in ductility of the single crystals might be due to a very fine precipitate which could not be detected by the optical microscope.

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## CONCLUSIONS

 The curve of yield stress vs temperature for polycrystalline Ag-33 at. % Al parallels that for prismatic slip in single crystals of the same material.

2. Over the temperature range from 77 to about 450 K, the ratio of the yield stress for Type I specimens to the yield stress for prismatic slip in single crystals is 1.8, and the ratio of the yield stress for Type II specimens to the yield stress for prismatic slip in single crystals is 1.07.

 Over the temperature range from about 450 to 775 K, the yield stress for Type I and Type II specimens is approximately equal to that for prismatic slip in single crystals.

There is an abrupt decrease in ductility at a composition of about
37 at. % Al. This is probably due to a precipitate in the grain boundary.

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## ACKNOWLEDGMENTS

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Special thanks are extended to the Coulter Steel & Forge Company. Emeryville, California, who so kindly forged the ingots for these experiments. Gratitude is extended to C. Thompson for the many duties he performed during the course of this investigation. Finally, the authors thank Professor J. E. Dorn who suggested the problem, and who was a continuing source of inspiration throughout the inquiry.

This work was done under the auspices of the U.S. Atomic Energy Commission.

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1		Resolved shear stress, psi			
Grain no.	Twins at grain boundary	Basal	Prismatic J	Prismatic II	Prismatic II
1	x	2540	24,000**	18,100**	4900
2		8340	23,000**	16,700	2940
3		9800**	20,600**	20,100	7840
4		7350	22,500**	18,100**	4900
5		4900	21,100**	21,100**	0
6	x	1960	24,000**	18,100	6850
7		13,200	20,100**	19.600**	7350
8		4900	23,500**	13,600	6850
9		11,300**	22,600**	12,700	9800
10	x	12,700**	22,600**	12,700	10,800
11		1470	21,100**	18,600	4900
12		0	24,500**	13,200	10,800
13		0	23,000**	19.600	4300
14		12,250**	22,600**	15,200	7350
15		11,800**	24,000**	12,200	12,700

## Table 1. Deformation Mechanisms Observed in Polycrystalline Ag-33 at. % Al

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"The critical resolved shear stress for prismatic slip is about 21,000 psi at room temperature, and that for basal slip is about 10.000 psi at room temperature.

"Indicates observed operative slip system.

Slip system	Composition, at. %Al	Critical resolved shear stress, psi	Ductility, 4
Basel	37,3	11,000	0.80
Prismatic	36.8	22,600	0.63
Basal	33.0	10,000	2.20
Prismatic	33.0	21,000	0.72

## Table 2. Results of Tension Tests on Ag-37 at. % Al Single Crystals

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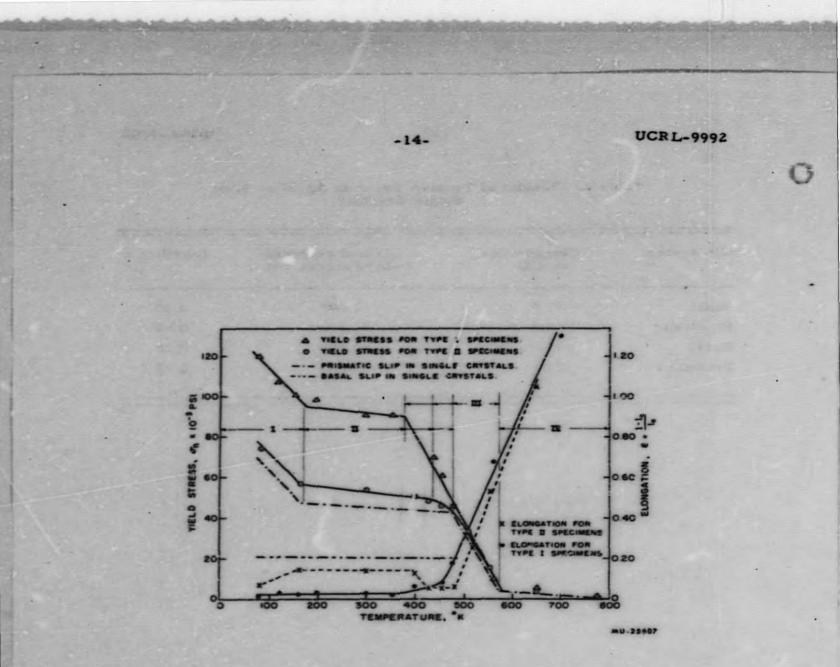


Fig. 1. Yield stress vs temperature for Type I and Type II specimens.

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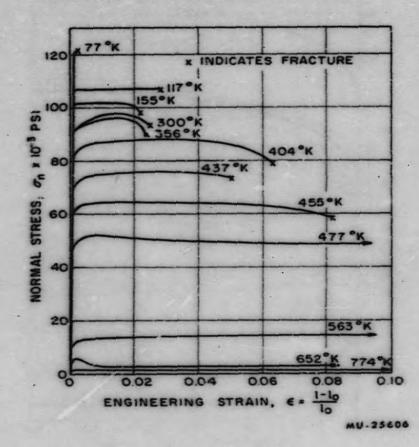


Fig. 2. Normal stress vs engineering strain for Type I specimens.

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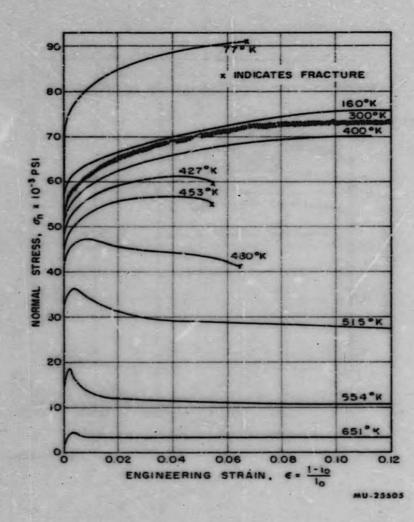
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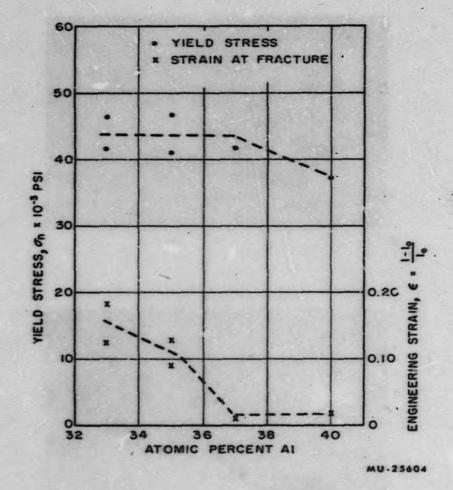
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Fig. 3. Normal stress vs engineering strain for Type II specimens.



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Fig. 4. Yield stress and breaking elongation vs concentration for polycrystalline specimens tested at room temperature.

