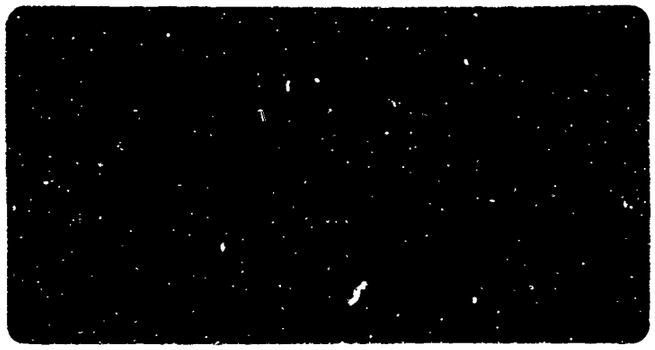

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Second Quarterly Progress Report

Fabrication and Properties of

Tungsten Single Crystals

"Growth of Single Crystal Tungsten

To Be Used for Rolling Into

Single Crystal Sheet"

Work Done and Reported By:

F. R. Charvat
G. W. Edwards

For Period June 1 to August 31, 1964

Prepared Under Contract NOw 64-0055-c

For the Materials Branch,
Bureau of Naval Weapons
Department of the Navy

SUMMARY:

This is the second quarterly report covering the work done for the Materials Branch, Bureau of Naval Weapons under Contract Number NOw 64-0055-c and covers the period from June 1 to August 31, 1964.

Twenty-four tungsten crystals were grown by the Arc-Verneuil method from which 7 were selected for machining into rectangular billets on the basis of minimum crystal misorientation and deviation of crystal orientation from cylindrical axis. Chemical analysis of the finished billets for interstitial and metallic impurities showed satisfactory low levels.

The continued effort for the third quarter will be the production of high quality oriented single crystal tungsten billets suitable for rolling.

I. INTRODUCTION:

The tungsten crystal billets (see Table I) produced for the program were examined intensively to determine the crystal imperfections in the billet before commencing the rolling of the material into sheet. As was stated in the previous program, this was done with the intent of not only removing unsatisfactory billets but also for forming a basis for explaining various phenomena and anomalies which occur during rolling of the billet into the finished product.

The chemical impurities and structural imperfections were determined for all billets and are presented below. The finished billets were sent to Philco Research Laboratories as soon as all the pertinent data mentioned above was obtained so that our subcontractors could proceed with the rolling program and subsequent evaluation.

II. CRYSTAL GROWTH:

The tungsten crystals produced for this evaluation were grown via the Arc-Verneuil technique using commercially available high purity tungsten powder. The tungsten seeds described in the first quarterly report were used to grow fifteen [110] and nine [001] crystals, with the designated crystallographic directions being parallel to the cylindrical axis of the crystal. The average size of the as-grown crystals was 7/16-inch in diameter and 8-inches long. The as-grown crystals were identified by data book number and page and by the crystal growth direction.

III. BILLET PREPARATION:

The billet preparation procedures described previously[1] were used to prepare the selected crystals into billets suitable for rolling. After determining the desired rolling plane direction by Laue Pattern, the crystals were mounted on machining blocks using DeKhotinsky cement and surface ground into billets approximately 0.230" x 0.375" x 6-1/2" with the rolling plane normal to the smallest dimension. A 1/2" sample was cut from the cap end of the billet, electrolytically polished in a 5% NaOH solution to remove work hardened material and analyzed by Schulz-Wei [2] and Laue Patterns to determine crystal misorientation and deviation from crystallographic and rolling direction. The results of the X-ray examination of the crystal orientation and billet structure imperfections are shown in Table II. The low-angle grain boundaries observed in the billets are shown by two representative Schulz-Wei photographs, Figures 1 and 2.

The machined billets were chemically etched in a 70% HF-30% HNO_3 solution to dissolve 0.005-0.007 inches from the face of each billet to remove the distorted surface layers caused by machining. These surfaces, if not removed, would interfere in the recrystallization and rolling-annealing studies.

IV. CHEMICAL ANALYSIS:

The 1-1/2 inch length cut from the cap end of the crystal was carefully cleaved and pieces obtained from the interior portion of the crystal were used for chemical analysis. The larger pieces were used for carbon and oxygen analysis using a Leco conductometric analyzer. The smaller pieces, crushed between platinum sheet, were used to determine metallic impurities spectrometrically using a 1.5 meter A.R.L. spectrograph and the Harvey-Semi quantitative method[3]. Nitrogen or hydrogen was not analyzed since past results have shown the content of each to be less than 1 ppm. Results are shown in Table III.

V. FUTURE WORK:

The above group of billets has been sent to Philco Research Laboratories for rolling studies this quarter and during the third quarter. Future work will be the continued production of single crystal tungsten billets and the production of tungsten-0.6 niobium single crystal billets so that a comparison can be made of the effect metallic impurities have on recrystallization temperatures of single crystal tungsten.

G. W. Edwards

G. W. Edwards:nm

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Figure 2. Schulz-Wei Photo of 1690-38 (110)[001] Cross-Section

TABLE I

CRYSTALS SELECTED FOR ROLLING

Crystal Number	Rolling Plane	Rolling Direction
1690-22	(110)	[001]
1690-24	(110)	[001]
1690-27	(001)	[110]
1690-34	(110)	[001]
1690-38	(110)	[001]
1690-39	(110)	[001]
1690-47	(001)	[110]
1690-50	(100)	[001]
1690-52	(100)	[001]
1690-53	(110)	[001]

TABLE II

X-RAY ANALYSIS OF TUNGSTEN CRYSTAL BILLETS

Crystal Number	Deviation of Crystal Axis from Rolling Direction	Deviation of Rolling Plane Normal To Ground Flat	Maximum Low-Angle Grain Boundary
1690-22	1° ← 6°↓	7°	5°
1690-24	3° → 0°	3°	50'
1690-27	5° → 7°↓	2°	4°
1690-34	0° 2-1/2°↓	2°	50'
1690-38	3° ← 3°↓	5°	~ 30'
1690-39	1° → 4°↓	4°	2°
1690-47	5° → 7°↑	3°	15'
1690-50	1° → 1°↓	1°	1-1/2°
1690-52	1° ← 1-1/2°↓	1-1/2°	1-1/2°
1690-53	0° 3°↓	4°	1°

TABLE III

CHEMICAL ANALYSIS OF TUNGSTEN CRYSTAL BILLETS

Crystal Number	C PPM	O PPM	Weight Percent Al	Co	Cr	Cu	Fe	Mg	Mn	Mo	Ni	Si	Sn
1690-22	9	10	← Not Detected →										
1690-24	7	12	← Not Detected →										
1690-27	3	9	← Not Detected →										
1690-34	9	9	N.D. →					10^{-3}	N.D. →				
1690-38	3	12	← Not Detected →										
1690-39	3	10	N.D.	N.D.	N.D.	N.D.	10^{-3}	10^{-3}	N.D.	N.D.	N.D.	N.D.	10^{-2}
1690-47	Not Completed												
1690-50	"	"											
1690-52	"	"											
1690-53	"	"											
Nominal Lower Limits of Detectability			10^{-4}	10^{-3}	10^{-3}	10^{-4}	10^{-3}	10^{-4}	10^{-3}	10^{-2}	10^{-3}	10^{-3}	10^{-3}

Note: Metallic analysis semi-quantitative accuracy: 10^{-2} = 0.01 - 0.1%
10^{-3} = 0.001 - 0.01%
10^{-4} = 0.0001 - 0.001%

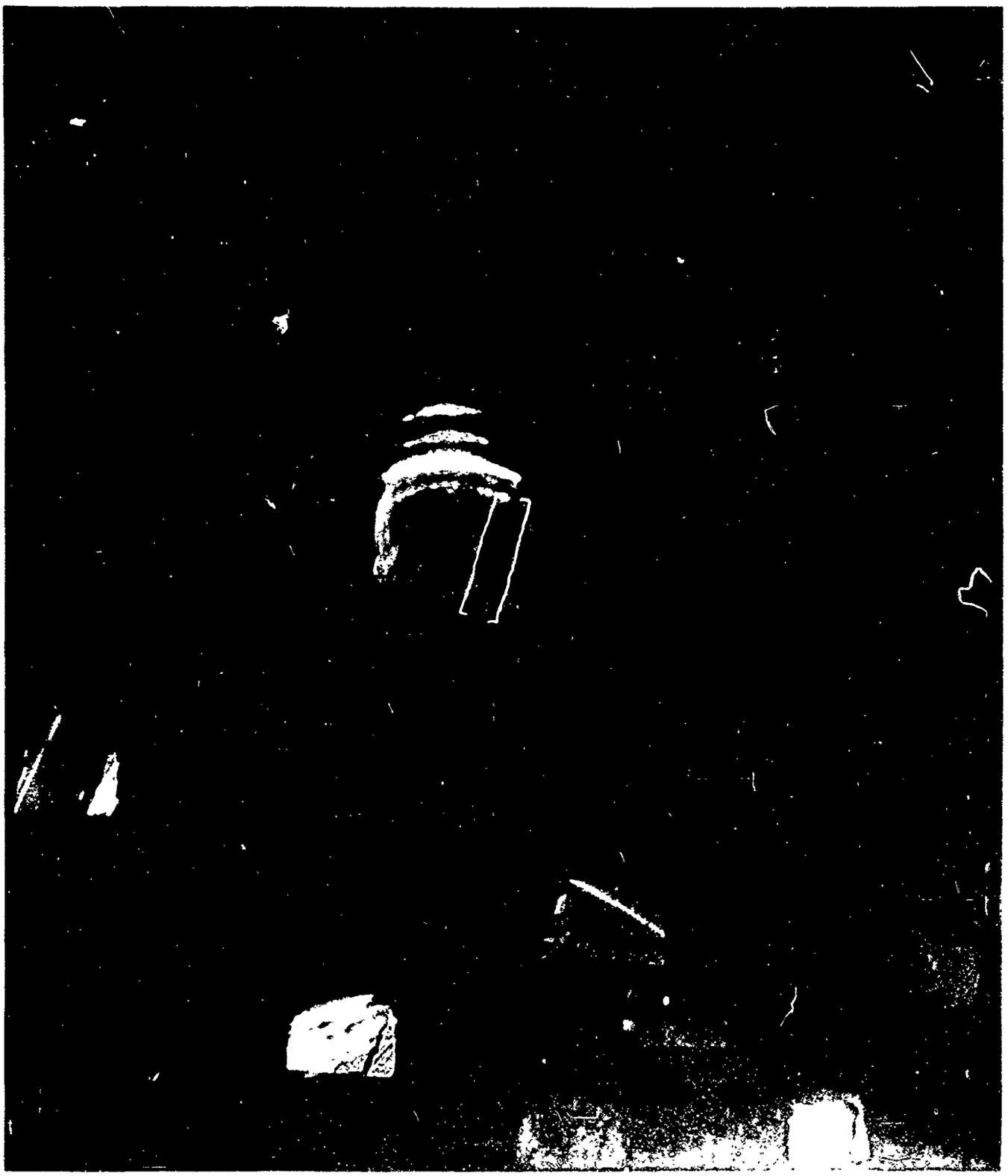

Figure 1. Schulz-Wei pattern of 1690-34 (110)[001] cross-section. [001] is perpendicular to page, (110) normal lies along vertical in page. Bracket shows misorientation of $\sim 50'$. White lines on image are characteristic radiation from tungsten target.

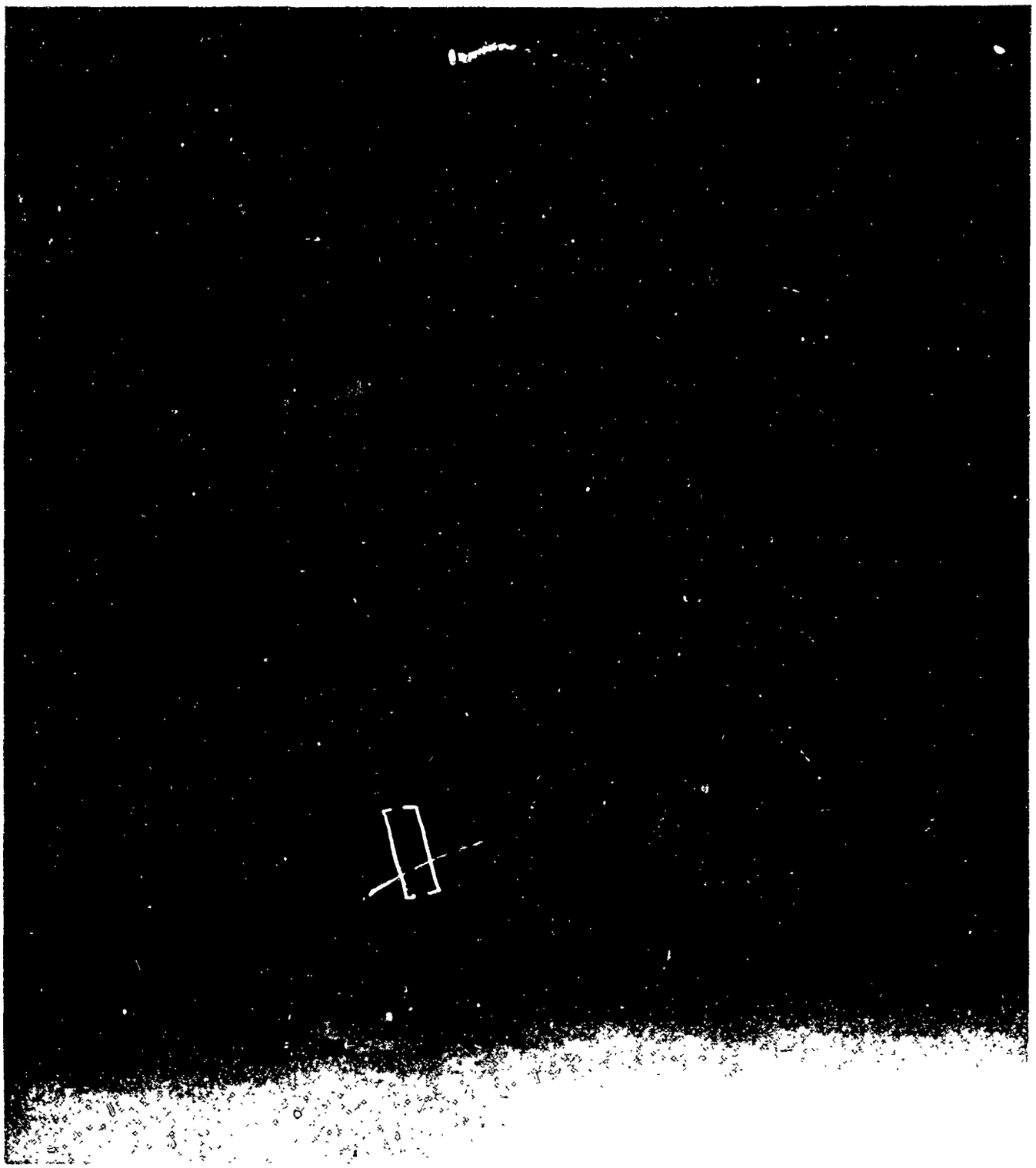

Figure 2. Schulz-Wei photo of 1690-38 (110)[001] cross-section. [001] is perpendicular to page, (110) normal lies along vertical in page. Bracket shows misorientation $< 1/2°$. There is larger misorientation of $\sim 1°$ at corner of billet. White lines on image are characteristic radiation from tungsten target.

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TECHNICAL REPORT

SINGLE CRYSTAL TUNGSTEN SHEET

Prepared for: Linde Company
A Division of Union Carbide Co.
Indianapolis, Indiana

Under Contract: Subcontract No. 1 to
Prime Contract NOw 64-0055-C

Prepared by: L. Raymond
I. Neumann

Approved by: Henry Kayfield for S.W.W
S. W. Weller, Director
Materials Laboratory

Reporting Period 1 May - 31 August 1964

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INTRODUCTION

The kinetics of recrystallization of a rolled tungsten single crystal have been shown[1] to be strongly dependent on the orientation of the crystal. For small amounts of deformation the (001) [110] crystal had the highest recrystallization temperature. Another possibility was the (110) [001] crystal. The (110) [$\bar{1}$10] crystal had the lowest recrystallization temperature with the other orientations, such as the (100) [001], having intermediate values.

Taking advantage of this orientation dependence, a roll-anneal cycle was developed that provided a means of deforming a single crystal by rolling and still retaining monocrystallinity at temperatures and deformations at which recrystallization was normally observed. The immediate value of such a process is the advantageous inherent ductility and competitive strength levels that a single crystal sheet should possess when compared to polycrystalline tungsten, to say nothing of the much lower working temperatures of the original billet.

The results of the previous program illustrated a principle that, from a practical point of view, was limited because of the small amounts of deformation involved - 3% per pass. The purpose of the present program is to show that advantage can be taken of prior recovery anneals to allow the use of larger amounts of deformation per pass and thus make the entire operation more practical.

QUARTERLY WORK STATEMENT

The slip systems activated during the deformation of tungsten single crystals by rolling at 10% per pass and 1000°C were investigated by means of (A) macroscopic dimensional changes of the billets, (B) Laue back-reflection photographs, (C) Vickers hardness measurements, and (D) dislocation etch-pit techniques.

The orientation of the single crystal billets chosen for the investigation, in terms of their rolling plane and rolling direction, were the two most favorable orientations, the (001) [110] and the (110) [001]. For comparative reasons, the most unfavorable orientation was also selected, the (110) $[\bar{1}10]$.

Deformation was carried to a nominal value of 50% of the original thickness. Sub-size round tensile bars were machined from the sheet to measure the influence of warm-working on the room temperature mechanical properties. For comparison, other billets of the same orientation were deformed 50% in one pass. In this way, an extreme condition can be used to study the influence of the contact angle during rolling relative to (1) the number of induced, operative slip systems, (2) the resulting crystallographic changes, and (3) the corresponding room-temperature tensile properties.

SUMMARY OF RESULTS

1. For a thickness reduction of 10% per pass, each favorable orientations, the (001) [110] and the (110 [001], deformed by duplex slip. This did not change the original orientation of the crystals but only caused 10° bending of the lattice about the transverse axis.

2. The deformation behavior of all three orientations can be explained on the basis of one set of slip systems, the $\{112\} < 11\bar{1} >$.

3. Both favorable orientations have only <u>two</u> available slip systems. In each case, the rolling plane is a symmetry plane.

   (a) In the (001) [110], each of the two slip directions is inclined at 35° to the rolling direction. This angular relationship favors duplex slip by the self-alligning nature of each slip system into the position of maximum applied resolved shear stress.

(b) In the (110) [001], each of the two slip directions is inclined at 55° to the rolling direction. This angular relationship favors slip on only one plane as soon as the billet is slightly misoriented relative to the rolls. When this occurs, the crystal begins to curve and one slip system continues to operate. Only by adjusting the rolling procedure can the second available slip system again become operative and therefore induce duplex slip which would physically be observed by a straightening effect on the curved crystal. The behavior is different than in the (001) [110] crystal, where rotation of the lattice by the active slip system will automatically cause the inactive system to become more favorable.

4. For the least favorable (110) $[\bar{1}10]$ crystal, two $\{112\} < 11\bar{1} >$ slip systems with maximum resolved shear stress, lie in the transverse direction. On the initial pass, it seems that only these systems were active since only broadening but no elongation was observed. But in subsequent passes, multiple slip became apparent as observed from the Laue photographs. In this case, even the other available $\{110\} < \bar{1}11 >$ set of slip systems became operative.

5. The 10° bending of the (001) [110] lattice occurred in the center of the crystal. Near the edge in the direction of the rolling plane, only a 5° bending of the lattice occurs. This angular relationship near the rolling surface is equal to the angle of contact for a 10% reduction in thickness of a 0.25 inch thick specimen when using 6 inch diameter rolls.

6. For 50% reduction in one pass, the contact angle is 12° but the ideal rolling conditions are changed so much that all three crystals deform by multiple slip. For the (110) [001] crystal under ideal rolling conditions, no broadening of the crystal is observed after 53% reduction in thickness. For the same amount of deformation in one pass, 48% broadening of the crystal was recorded.

7. The final proof of the existence of different modes of deformation which depend on crystal orientation and on the contact angle or amount of deformation per pass, is reflected in the room temperature tensile properties.

After a total of about 50% reduction in the original thickness, all crystals deformed by multiple-pass techniques had greater ductility than the same crystal orientation deformed in a single pass. The two crystals deformed by duplex slip had about 14% elongation at room temperature. For the (110) [001] crystal, the reduction in area was uniform until failure occurred by cleavage on the (001). For the (001) [110] crystal, localized necking occurred with the cross-section of the fractured surface being elliptical in shape.

8. Vickers hardness readings, although anisotropic, showed a rapid increase with small amounts of deformation but very small subsequent changes as the deformation was increased to large values.

EXPERIMENTAL TECHNIQUES

Specimen Preparation

The Linde Company supplied single crystals billets with three orientations:

Crystal Number	Orientation
1690-38	(110) [001]
1690-27	(001) [110]
1616-57	(110) [$\bar{1}$10]

The billets, which had a thickness of approximately 1/4 inch and a width of 3/8 inch, were cut with a diamond wheel into pieces of about 2 1/2 inch length and electrolytically polished in an aqueous solution of 20g NaOH per liter in order to remove a surface layer of .001 inch. The crystals were then annealed in vacuum (10^{-4} Torr) at 2400°C for 30 minutes.

After each rolling operation a test piece of about 1/4 inch long was cut off from the aft end. For the cutting operation the crystals were mounted on ceramic tiles with DeKhotinsky cement. The transverse section was then used for x-ray, hardness, and etch pit studies. On an annealed crystal, the damage introduced by cutting with a water cooled diamond wheel and subsequently grinding on 600 grit paper did not go deeper than .001 inch. A surface layer of .002 inch was therefore removed from all 1/4 inch samples by electrolytically polishing in the 2% NaOH-solution in order to eliminate all surface damage.

Rolling Operation

The diameter of the rolls of a Loma Model 600 mill is 6 inches; the peripheral velocity was 500 inch/min.; the surface temperature of the rolls was about 400°-450°C. The crystals were heated before each pass for 1/2 hour in argon at 1000°C. During rolling, oxidation of the crystals in air resulted in thin layer of WO_3. This greenish-yellow oxide was removed by immersing the crystals for a few minutes in a boiling NaOH solution.

All crystals were rolled in the same direction i.e., the same end was always introduced into the rolls. The rolling program for each orientation was conducted in two ways:

1. A reduction of 10% per pass in several passes to a total reduction in original thickness of 50%.

2. A reduction of 50% in one pass.

The rolling was not carried beyond a final thickness of 100 mils in order to obtain round tensile bars identical to those used by Rose, Ferris and Wulff.[2]

Laue Photographs

Initial orientation of the crystals and crystallographic changes during the rolling were observed by means of the standard Laue-Back reflection technique (30 mm distance). The pictures were generally taken in the center sections of the transverse plane with the rolling direction parallel to the incident beam. Since the effective x-ray penetration depth is less than 0.0005 inch (assuming a linear absorption coefficient of 2000 cm^{-1}), complete removal of damaged surface layers was again essential.

Hardness Measurements

For the hardness measurements on the transverse plane, the Vickers diamond pyramid was chosen since it is less sensitive to the orientation of the crystal than the Knoop hardness. But, the Vickers hardness still remained anisotropic. This orientation dependence was used to some extent to obtain information concerning the active slip system at room temperature. Measurements were again carried out on electrolytically polished surfaces.

Etch Pit Investigations

The etch pit technique was found to be a very valuable tool in determining the mode of deformation through the thickness of the ingots, particularly at low deformations. Reduction of the voltage from 10 for polishing to 3 for etching in a 2% NaOH solution revealed subgrain boundaries and individual dislocations. The etch pits on the {100} faces have a square shape, with the sides of the squares parallel to the < 100 > directions. At room temperature, the etching time was about 5 to 10 seconds.

Tensile Specimens

Because of the extreme difficulty experienced in machining sub-size sheet tensile specimens and the further difficulty in gripping the specimens during testing, the mechanical properties where measured on round tensile bars ground from the rolled sheet. The size was chosen to be exactly the same as that used by Rose, Ferris and Wulff[2] in studying annealed single crystal properties, the results of which will be used for comparison.

Testing was done on an Instron at a strain rate of 0.025 in./in./min. The gage length was about 0.8 inch with a 0.050 $\pm$ .002 inch diameter. At least .004 inch was previously removed from the diameter by polishing in order to remove all of the surface damage introduced by grinding the specimens to their final shape. All specimens were successfully ground and tested with no premature breakage during either operation.

QUARTERLY RESULTS

Macroscopic Dimensional Changes

The orientation of the active slip systems during rolling, especially if only one or two are active, determines the macroscopic dimensional changes of the crystal during deformation. From a measurement of the thickness, the width after each pass, and assuming constant volume, the change in length can be calculated from the relationship:

$$1 = (1 + \epsilon_r)\ \ (1 + \epsilon_t\ )\ \ (1 + \epsilon_n)$$

where, ϵ_r = strain in the rolling direction or length change.

ϵ_t = strain in the transverse direction or width change.

ϵ_n = strain in the normal direction or thickness change.

Table 1 gives the measured values of ϵ_t and ϵ_n, and the corresponding calculated values of ϵ_r. The (001) [110] and the (110) [001] crystals did not show any widening for multipass rolling although the latter orientation developed a curvature which increased with every pass about the transverse direction. After 27%, the specimen was turned over without changing the lead-end in order to straighten the billet. The cross-section of the (110) $[\bar{1}10]$ crystal was barrel shaped, indicating larger deformation in the center than on the surface. The same dimensional effect was observed for all crystals deformed a nominal 50% in one pass.

TABLE I

DIMENSIONAL CHANGES OF BILLETS DURING ROLLING

(110) [001]:

Reduction in Thickness (%)	12	22	27	35	44	53	53-1 pass
ϵ_t (%)	0	0	0	0	0	0	48
ϵ_r (%)	14	28	37	54	80	108	44

(110) [$\bar{1}$10]:

Thickness Reduction (%)	7	20	28	37	49	57-1 pass
ϵ_t (%)	7	19	24	31	31	75
ϵ_r (%)	0	5	12	21	50	33

(001) [110]:

Thickness Reduction (%)	11	22	30	37	44	55	53-1pass
ϵ_t (%)	0	0	1	1	2	2	35
ϵ_r (%)	12	28	41	57	75	118	58

X-Ray Photographs

Before rolling, the original orientation of the crystals was checked. The (110) [001] crystal was very close to the ideal orientation, but the (001) [110] crystal had its [110] pole, 4^{o} North and 6^{o} West, off the rolling direction. The (110) $[\bar{1}10]$ was also several degrees off.[3]

The most significant result of the Laue back-reflection photographs was that the original orientation of the crystals was maintained up to 50% reduction in the case of the (110) [001] and (001) [110] crystals for a 10% reduction per pass. Only a symmetrical streaking of 5^{o} towards both sides of the Laue spots occurred. These streaks appeared more washed out at higher deformations but did not substantially increase in length (See Fig. 1). The results indicate that only a simple bending of the crystals about the transverse axis had occurred. Heating the test specimens to 1000^{o}C for 1/2 hour prior to the subsequent pass, did not result in any substructural changes as verified by no detectable changes in the Laue photographs after the annealing treatment.

Photographs taken of rolled (110) [001] crystals on the transverse plane, close to the top and bottom surfaces, showed that at the top surface the Laue streaks extended only 5^{o} downward, while at the bottom surface they extended 5^{o} upward. In the center section of the transverse plane, a combined 10^{o} streak about the rolling plane was observed. This indicates that at the surfaces only one slip system was operating, while in the center section both were activated.

In contrast, the (110) $[\bar{1}10]$ crystal had Debye rings from the characteristic radiation already appearing at 20% reduction, indicating rotation of the crystal about several axes. Figure 2 shows the similarity in Laues after either multipass or single pass deformation in contrast to the significant difference in each illustrated by Figure 1. The Debye rings appeared in all three crystals deformed 50% in one pass. No attempt was made to identify the slip systems under these conditions of multiple slip.

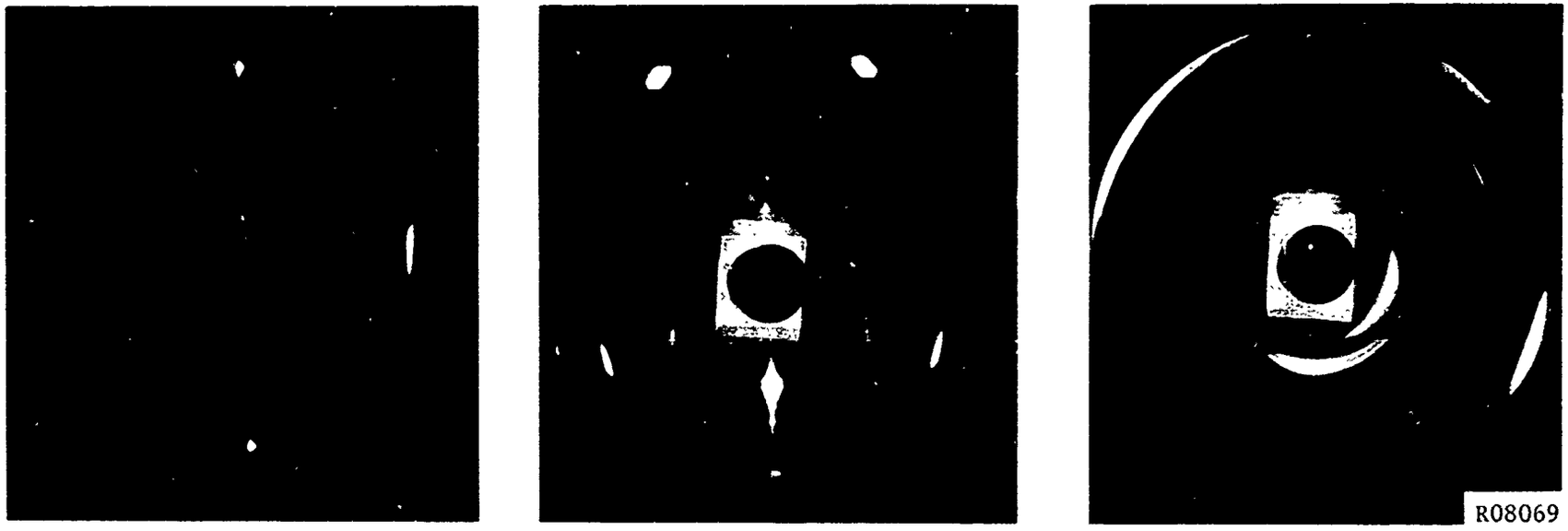
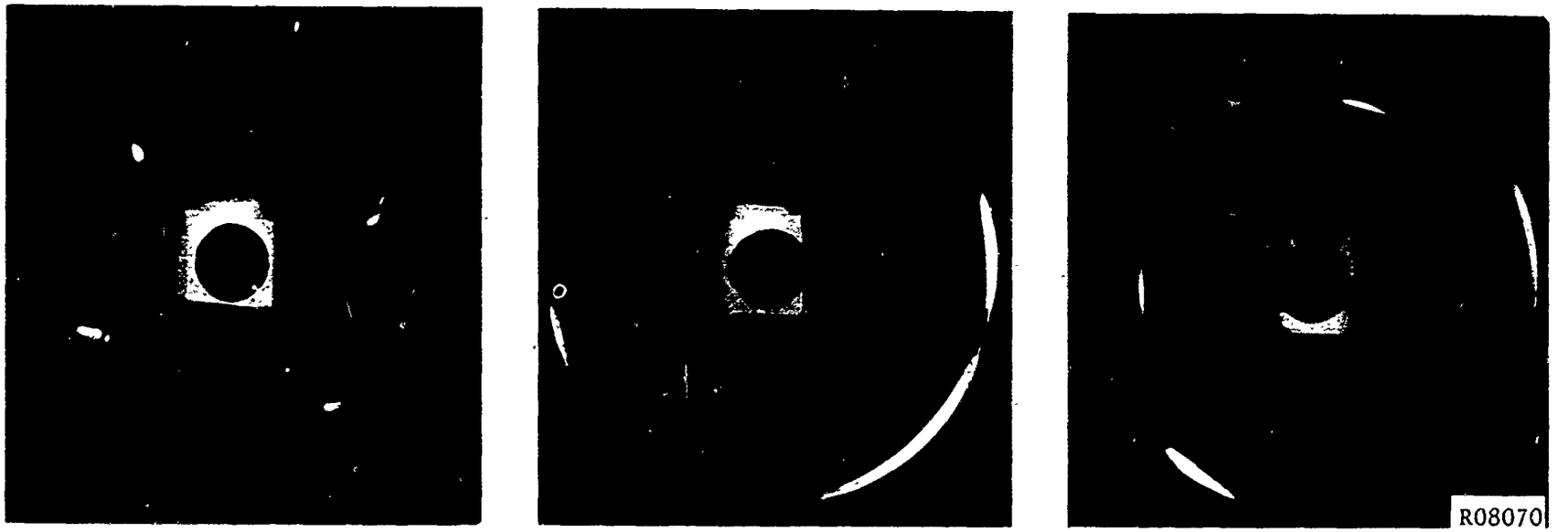


FIGURE 1. INFLUENCE OF REDUCTION PER PASS ON THE CRYSTALLOGRAPHIC CHANGES FOR (110) [0̄01] CRYSTAL



10% SINGLE PASS

50% FIVE PASSES

50% SINGLE PASS

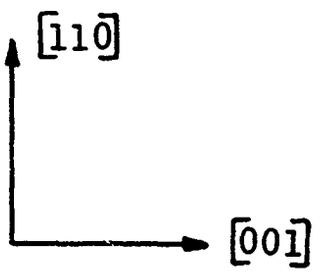


FIGURE 2. INFLUENCE OF REDUCTION PER PASS ON THE CRYSTALLOGRAPHIC CHANGES FOR (110) [$\bar{1}$10] CRYSTAL.

Etch Pit Studies

Etch pit studies were carried out on the transverse plane for the (110) [001] crystal. The etch pit density within the subgrains of an annealed crystal was about $10^5/cm^2$. After the first rolling pass the density went up sharply, increasing slowly thereafter. As previously reported[5], no residual as-grown subgrain boundaries were apparent after the first 10% pass.

In the center section, the etch pits formed lines parallel to the rolling plane (Fig. 3). This alignment could be observed up to 50% (multiple passes). Towards the surfaces, the arrangement was less regular with the parallel lines becoming less evident. A cut on a {100} plane, parallel to the rolling direction, shows a crisscross pattern of etch pits in the center section (Fig. 3), indicating two intersecting slip systems. Towards the top surface only one direction appeared, while at the bottom surface the complimentary direction predominated.

Hardness

The hardness was measured on the transverse plane, across the thickness of the specimens. The sides of the Vickers indenter were parallel and perpendicular to the rolling plane. Figure 4 demonstrates the orientation dependence of the hardness. Two indentations, taken on the same (100) plane of an annealed crystal, only rotated 45° against each other, have entirely different shapes. The concavities in the diamond shape are normally observed in annealed materials. The barreling effect of the diamond shape is generally associated with a work hardened material. In the tungsten single crystal both shapes appear, depending on the orientation of the indenter. Anisotropy accounted for 6% deviation.

The Vickers hardness was used since it is less sensitive to anisotropy than the Knoop hardness.[6] Measurements were identified with respect to the plane and direction of the indenter axes. Variations across the thickness of the specimens were not significant enough to draw any conclusions as to the amount of local work hardening. The values plotted in Figure 5 represent the average of all measurement. In comparing Vickers with Knoop hardnesses, it must be remembered that although the length of the long axis of the Knoop indentation is measured, it represents the hardness in the direction of the short axis.

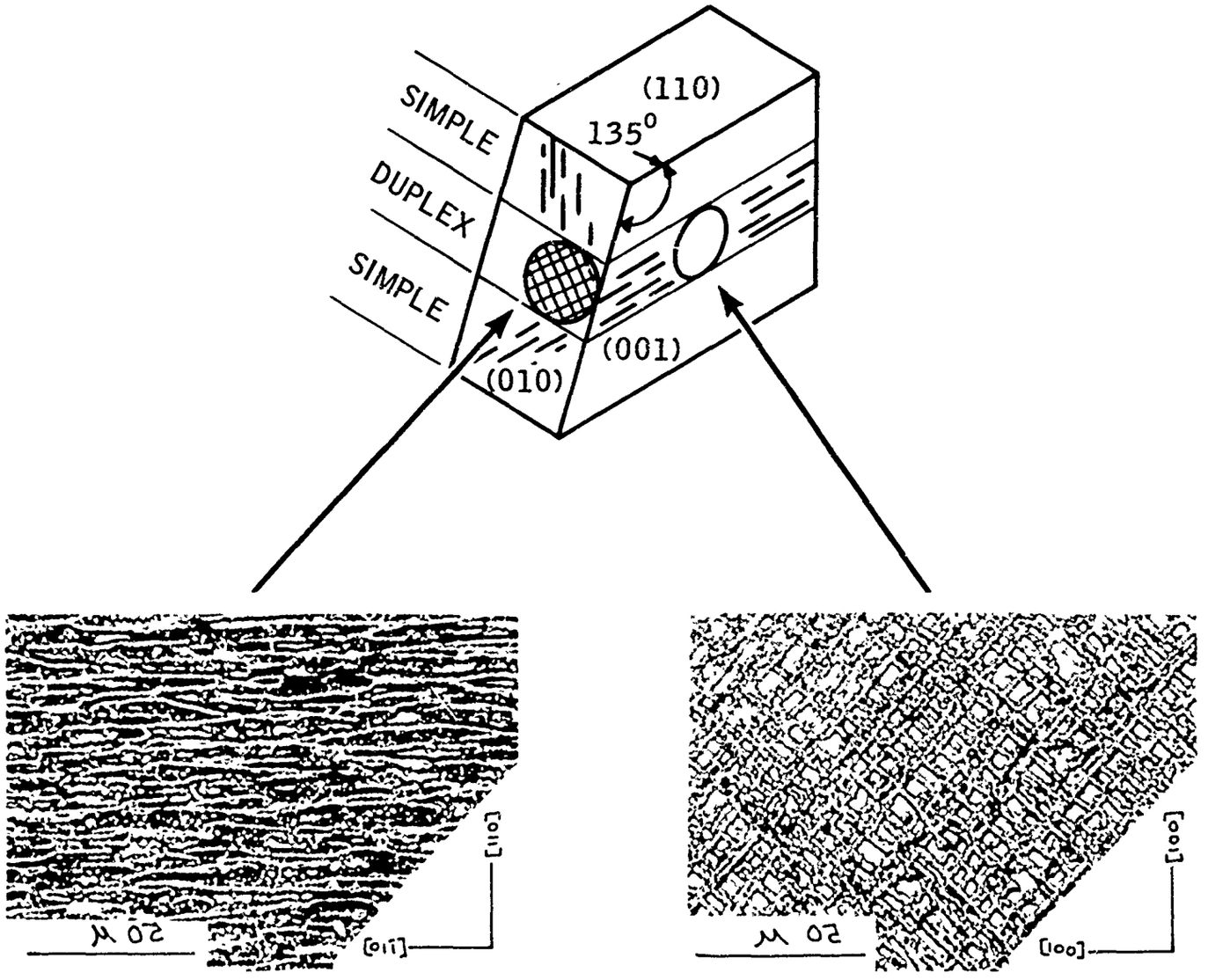


FIGURE 3. ETCH PIT PATTERNS IN THE CENTER OF TRANSVERSE AND LONGITUDINAL PLANES OF THE (110) [001] CRYSTAL

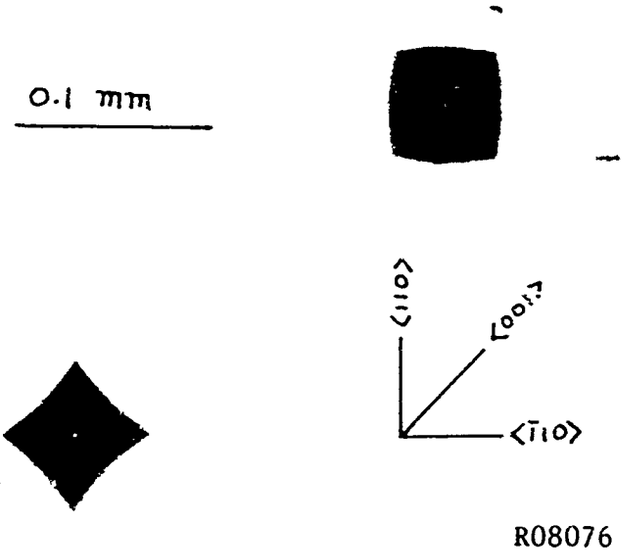


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FIGURE 4. ANISOTROPY OF VICKERS HARDNESS ON THE (100) IN AN ANNEALED CRYSTAL

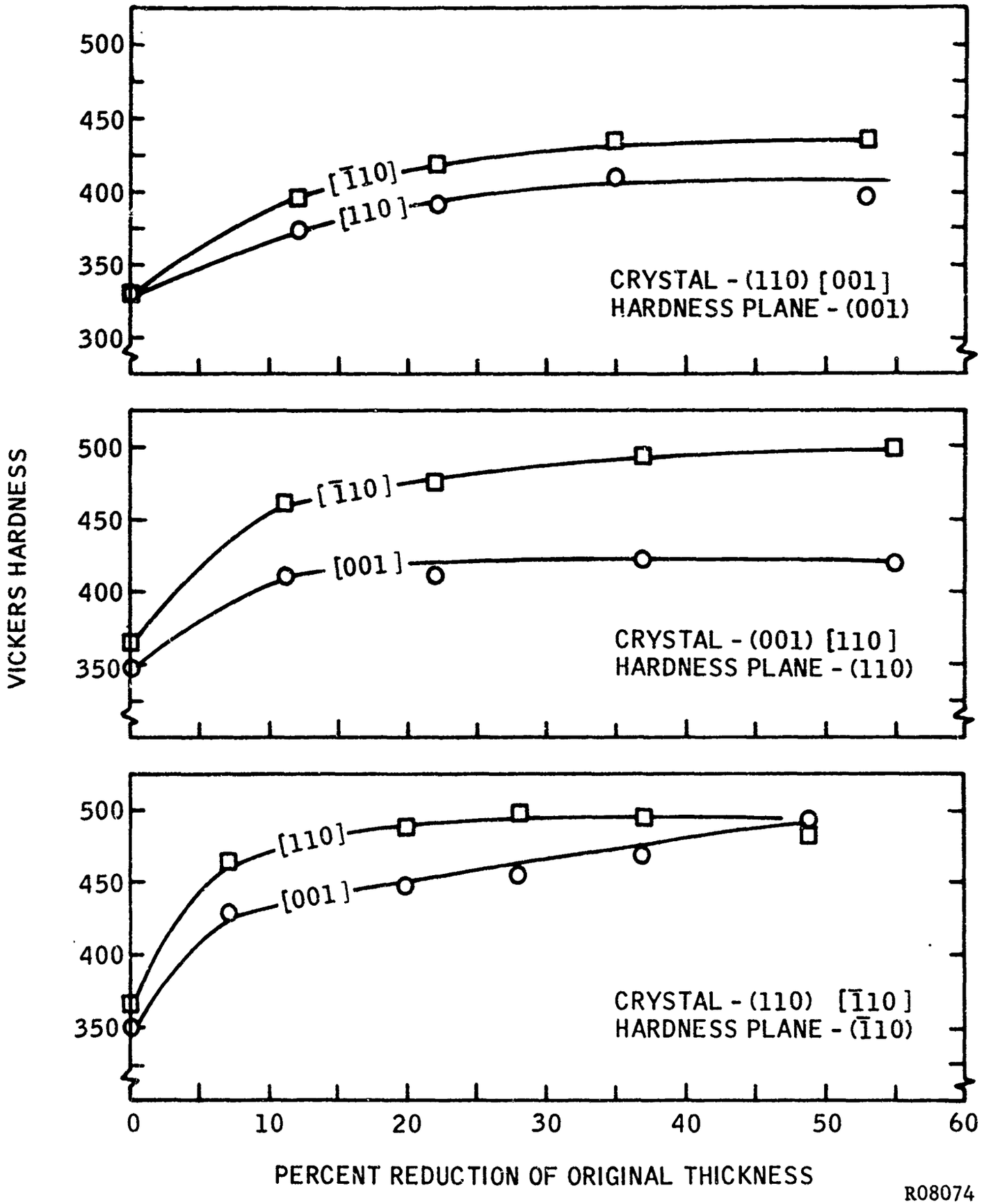


FIGURE 5. VICKERS HARDNESS AS A FUNCTION OF ORIENTATION AND DEFORMATION

Tensile Tests

The axis of the round tensile bars, machined from the deformed crystals, was the same as the rolling direction. Four tensile specimens had a common axis, the < 110 >. For the same amount of warm-working, extremes in ductility were obtained from 2.5% to 14.2%. The only variables were (1) the rolling plane and (2) the number of deformation passes. Both variables influence the mode of deformation and the resulting crystallographic changes. It becomes immediately apparent, that the substructural changes accompanying deformation by multiple slip are detrimental to the ductility. Maximum ductility occurs when deformation is by duplex slip. The orientation with the highest proportional limit and tensile strength is the most work hardened (110) [$\bar{1}$10] crystal. Likewise at decrease in elongation to 2.5% is observed for the corresponding maximum 213,000 psi tensile strength condition. Annealed crystals of this orientation have a proportional limit of 95,000 psi and a tensile strength of 128,000 psi.[2] The resulting stress-strain curves are plotted in Figure 6.

Also plotted are annealed [100] tensile specimens which have a significant rate of work hardening. The p.l. was reported as 31,000 psi and the stress at 5% strain equal to 138,000 psi.[2] The <100 > tensile bars from the (110) [001] warm worked crystals has a p.l. of 95,500 psi and the stress at 5% strain equal to 148,500 psi with 13.3% elongation to failure which finally occurred by cleavage on the (100). See Figure 7.

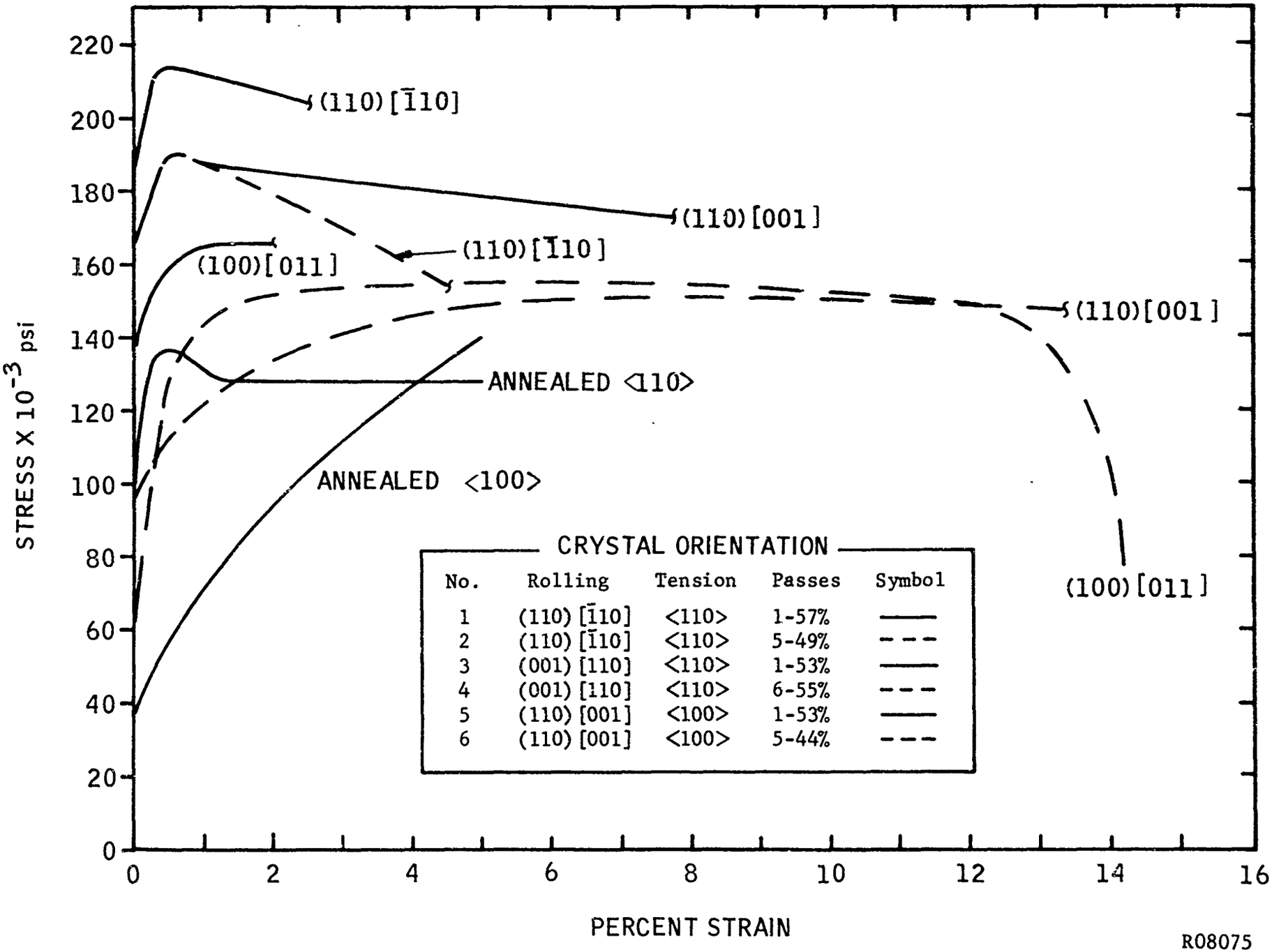


FIGURE 6. TENSILE STRESS-STRAIN CURVES OF ROUND BARS MACHINED FROM SHEET

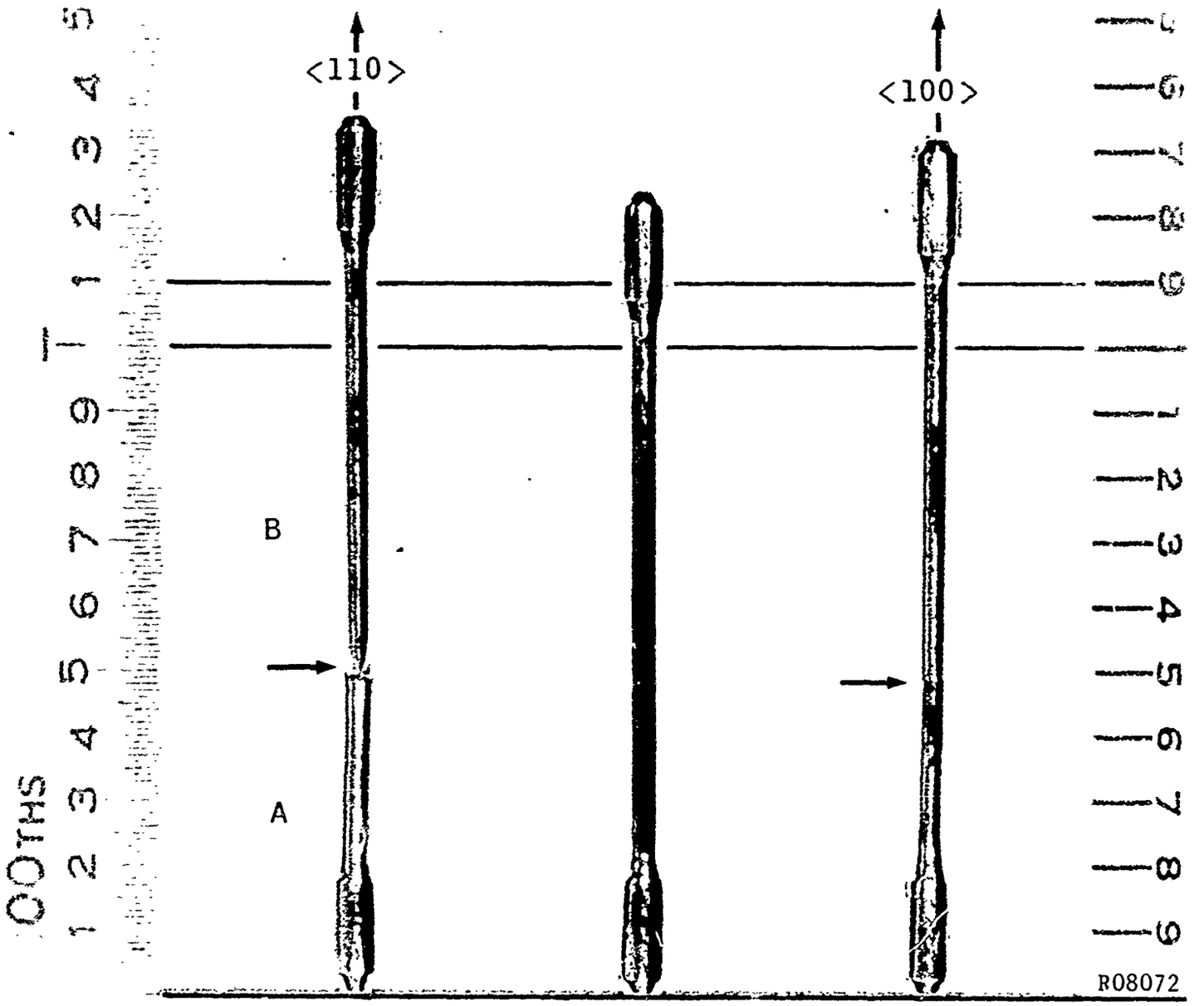
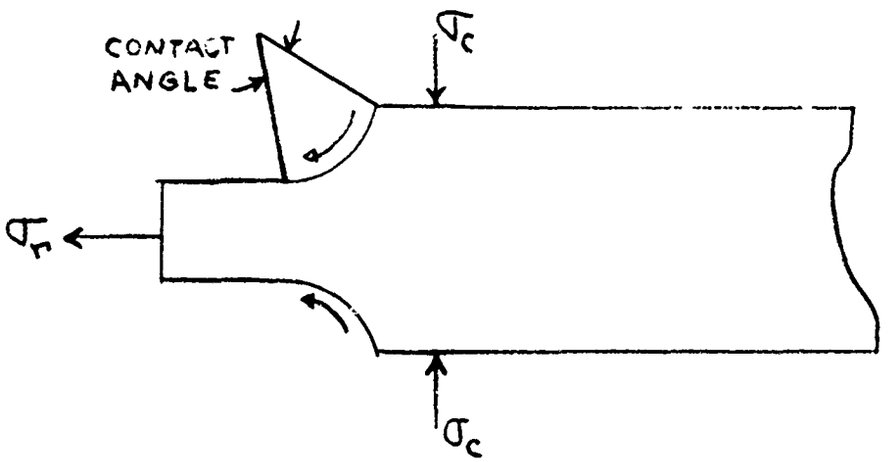


FIGURE 7. TENSILE BARS FROM W SINGLE CRYSTALS ROLLED 50 PERCENT AT 1000°C IN 5 PASSES ILLUSTRATING ORIENTATION DEPENDENCE OF FRACTURE AT ROOM TEMPERATURE. A ROTATED 90° FROM B.

DISCUSSION OF RESULTS

The results of the (A) macroscopic dimensional changes, (B) Laue back-reflection photographs, (C) Vickers hardness measurements and (D) dislocation etch-pit patterns can all be correlated by considering only one set of slip systems as being operative during rolling at 10% per pass at 1000°C. These are the $\{112\} < 11\bar{1} >$. Room temperature tensile properties are used to further substantiate the correlations.

If it is assumed that the rolling stresses at 1000°C can be resolved into tensile and compressive stresses as indicated schematically below,



the shear stress (τ) acting on a particular slip plane and in a particular slip direction can be calculated from the relationship:

$$\tau = \sigma_r \cos \Phi_r \cos \lambda_r + \sigma_c \cos \Phi_c \cos \lambda_c ,$$

where

σ_r = resolved tensile component of the rolling stress,

σ_c = resolved compressive component of the rolling stress,

Φ = angle between the applied stress direction and normal to the slip plane,

λ = angle between the applied stress direction and slip direction.

The orientation of the three crystals rolled at 1000°F is shown in Figure 8 where the $\{112\} < 11\bar{1} >$ slip systems, which have the highest resolved shear stress, are also indicated on the stereographic projections. The rolling and transverse directions are also labelled. Each crystal will be discussed separately.

First, the (001) [110] crystal has two slip systems in the rolling direction (Figure 8) with $\tau_r = 0.47\ (\sigma_r + \sigma_c)$, and two in the transverse direction with $\tau_t = 0.47\ \sigma_c$. Since $\tau_r > \tau_c$, the two slip systems in the rolling direction should be operative and ideally result in duplex slip. For this crystal, the slip direction is inclined at an angle of 35° to the rolling direction. If by a slight misorientation, either by crystal growth or rolling, one of the two slip systems has a slightly higher resolved shear stress, only this system will be active and start rotating towards the rolling direction. In so doing, the rotation will automatically cause the inactive system to become more favorable i.e., the maximum value of the resolved shear stress to shifts to the inactive system. The (001) [110] crystal is therefore self-stabilizing. Under these conditions, very little widening of the billet occurred (2%) and the Laue streaks only extended about the transverse axis in one direction (Figure 1).

Second, the (110) [001] crystal has two slip systems, both lying in the rolling direction (Figure 8), with a maximum resolved shear stress of $\tau_r = 0.47\ (\sigma_r + \sigma_c)$. Again the two slip systems are symmetrical about the rolling plane with the direction of the rolling stress, direction of slip, and normal to the slip plane all coplanar. For perfect crystal orientation relative to the rolls, duplex slip should again ideally result with no change of the original crystal orientation and elongation only in the rolling direction. This observation is born out by the sharpness and direction of Laue streaks (Figure 1), the absence of billet widening (Table I), and the orientation of the etch pits (Figure 3).

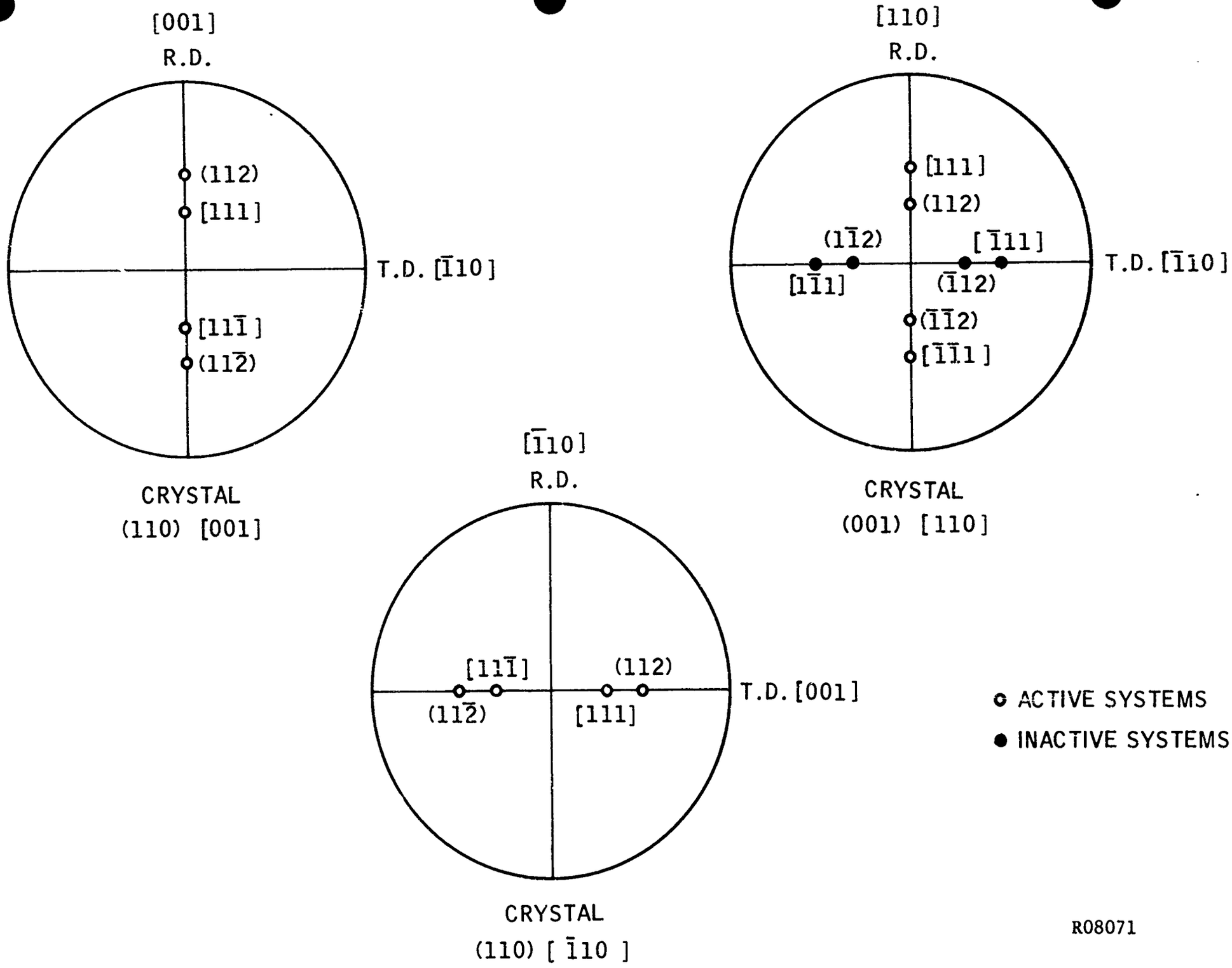


FIGURE 8. SLIP SYSTEMS WITH HIGHEST RESOLVED SHEAR STRESS FOR THREE CRYSTAL ORIENTATIONS

While the behavior and results of the (001) [110] and the (110) [001] crystals seem to be the same, there is however one essential difference: in the (100) [001] crystal the slip directions are inclined at an angle of 55° to the rolling direction. If by slight misorientation one of the two slip systems has a slightly higher resolved shear stress and only one system initially becomes active, the resolved shear stress for the active system will continuously increase with rotation of the lattice, in contrast to the self-stabilizing behavior of the (001) [110] crystal. The imperfect duplex slip of the (110) [001] crystal is physically observed by a rolling curvature in the crystal and can only be corrected by modifying the rolling procedure.

An interesting observation was the length and direction of streaks of the same Laue spot taken in the center of the crystal (10°) as compared to the surface (5°). The angle of contact between the rolls and crystal for a 10% reduction in thickness of a 0.25 inch thick specimen using 6 inch diameter rolls is 5°. This 5° contact angle would cause the slip plane in the top portion of the crystal to rotate downward; whereas the contact angle at the bottom rolls would cause the second slip plane to rotate 5° upward, which is in complete agreement with the observed direction of the streaks from the Laue spots. Although not substantiated, it is here suggested that the bending of the lattice is related to the contact angle. To substantiate the last observation, the thickness of the 0.25 inch billet was reduced 50% in one pass, which corresponds to a contact angle of 12°. Under these conditions, the ideal rolling conditions were changed such that after the first pass, multiple slip had been observed in all crystals. (compare Figure 1 to Figure 2). Therefore, the results with the 12° contact angle would not be compared to those with 5°.

Third, an entirely different behavior is observed for the (110) [$\bar{1}$10] crystal, which has its two most favorable $\{112\} < 11\bar{1} >$ slip systems lying in the transverse direction (Figure 8) with a maximum resolved shear stress of $\tau_t = 0.47\ \sigma_c$. This crystal is only different from the (110) [001] in that the rolling and transverse directions are exchanged. Since broadening but no elongation was observed on the first pass (Table I), it seems that only the above mentioned two slip systems were active; but on subsequent passes, several other slip systems start operating as shown by the Laue photographs (compare Figure 1 to Figure 2) and multiple slip predominated.

During the first pass for both the (110) [001] and the (110) [$\bar{1}$10] crystals, the same slip system couple was active. If one assumes that the plastic strain is proportional to the applied stress, an estimate can be made of the ratio of the rolling stresses, σ_r and σ_c. From Table I, $\epsilon_t = 0$ and $\epsilon_r = 14\%$ for the (110) [001] crystal where $\tau = 0.47\ (\sigma_r + \sigma_c)$. Likewise $\epsilon_t = 7\%$ and $\epsilon_r = 0$ for the (110) [$\bar{1}$10] crystal where $\tau = 0.47\ \sigma_c$. It follows that $\sigma_r = \sigma_c$.

The difference in room temperature tensile properties is final proof of the existence of different substructural configuration resulting from 50% reduction by duplex slip in contrast to multiple slip. Deformation by duplex slip possessed superior ductility. The two specimens which showed about 14% elongation at room temperature were the (110) [001] and (001) [110] crystals deformed in several passes. Deformation by multiple slip showed about 4.5% elongation at room temperature for the (110) [110] crystal deformed in several passes. In all cases, multipass rolling possessed better ductility than the corresponding crystal orientation deformed the same amount in a single pass.

The deformation characteristics of the room temperature tensile bars (Figure 7) suggests that multiple slip has occurred in the < 100 > tensile coupon and duplex slip in the < 110 > tensile coupon. The shank of the < 100 > tensile bar is reduced uniformly to 10% RA until fracture occurs by cleavage on the {100}. The stereographic projection (Figure 8) shows four equally favorable {112} < 11$\bar{1}$ > slip systems which would be operative and cause excessive work hardening until the {100} cleavage stress is exceeded. For the < 110 > tensile bar, only two slip systems are available and duplex slip occurs which results in very little work hardening. In this case necking was observed. The fractured surface resulted in an elliptical cross-section with the major axis retaining its original dimensions. In this connection it is interesting to look at the tungsten single crystals experiments by Rose, Ferris and Wulff.[2] They found that in uniaxial tension, along the < 110 > direction, no work hardening occurred, while a strong work hardening was observed in the < 100 > direction. They explained these results by the formation of the < 100 > type sessile dislocations. In the light of the present results, it seems the essential difference is that for the < 110 > tensile direction only duplex slip occurs, while for < 100 > direction four slip systems belonging to two different zones, are intersecting.

For the Vickers hardness, the most typical observation is that the direction perpendicular to the rolling plane for the (110) [001] and the (001) [110] crystals is lower than in the transverse direction. For example, (Figure 8), the $[\bar{1}10]$ is greater than the [110] for the (110) [001] crystal and the $[\bar{1}10]$ is greater than the [001] for the (001) [110] crystal. This is in agreement with the etch pit observations plus the fact that only two slip systems have been active during rolling in each case.

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4. Ibid, p. 48

5. Ibid Ref. 1, p. 20.

6. D. L. Douglass, "Hardness Anisotropy of Columbium," ASM Transactions V 54, (1961) p. 322.