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Refractomet Division

UNIVERSAL-CYCLOPS STEEL CORPORATION

Technical Report

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Bridgeville, Pennsylvania

ASC TR 7-827 (VI)

ASC INTERIM REPORT 7-827 (VI)
February, 1963

TUNGSTEN SHEET ROLLING PROGRAM

Contract AF33(600)-41917

Sixth Interim Report

1 September 1962 - 31 January 1963

Prepared By
W. J. Schoenfeld

UNIVERSAL-CYCLOPS STEEL CORPORATION
REFRACTOMET DIVISION
BRIDGEVILLE, PENNSYLVANIA

The melting program was scaled up to provide 6-in. conditioned ingots for extrusion. Direct extrusion to sheet bar and extrusion to round for subsequent press forging to sheet bar was accomplished satisfactorily on four ingots. The round extrusions were forged to sheet bar and rolling was initiated.

BASIC INDUSTRY BRANCH
MANUFACTURING TECHNOLOGY LABORATORY

Directorate of Materials and Processes
Aeronautical Systems Division
United States Air Force
Wright-Patterson Air Force Base, Ohio

ABSTRACT - Summary ASC INTERIM REPORT 7-827 (VI)
Sixth Interim Technical Progress Report February, 1963

TUNGSTEN SHEET ROLLING PROGRAM

Refractomet Division
Universal-Cyclops Steel Corporation

During the period of this report, melting and extrusion was scaled up to meet the sheet size requirements of this phase. Specifically, four 8" ingots were melted, conditioned to 6" extrusion billets. Two were extruded to 3" round for subsequent forging to sheet bar and the remaining two were extruded direct to 1.75" x 4". sheet bar. All extrusions were evaluated and the 3" rounds press forged to 2" thick sheet bar.

The first rolling operation was accomplished successfully on both press forged and direct extruded rounds.

ASC TR 7-827 (VI)

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FOREWORD

This Interim Technical Progress Report covers the work performed under Contract AF33(600)-41917 from 1 September 1962 to 31 January 1963. It is published for technical information only and does not necessarily represent the recommendations, conclusions, or approval of the Air Force.

This contract with the Refractomet Division, Universal-Cyclops Steel Corporation, Bridgeville, Pennsylvania, was initiated under ASC Aeronautical System Division, Project 7-827, "Tungsten Sheet Rolling Program." It was administered under the direction of Mr. Hugh L. Black, Project Engineer, Basic Industry Branch, Manufacturing Technology Laboratory, Wright-Patterson Air Force Base, Ohio.

W. J. Schoenfeld of the Development Group, Refractomet Division, Universal-Cyclops Steel Corporation was the Engineer in charge.

Since the nature of this work is of interest to so many fields of endeavor, your comments are solicited as to the potential utilization of the material produced under this contract. In this manner, it is felt that a full realization of the resultant material produced will be accomplished.

****

PUBLICATION REVIEW

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I. Introduction

The continued interest and support of government agencies in refractory metal systems has in the past few years resulted in a revolutionary advance in their technology. The utilization of this technology is rapidly expanding our arsenal of intercontinental weapons and eliminating man's barrier to space exploration.

This program, supported through the Aeronautical Systems Division, USAF, is designed to further the existing state-of-the-art in producing tungsten sheet. The information derived should provide a significant advance in the missile and space vehicle programs. To accomplish the overall mission, the program has been divided into five separate phases as summarized below.

Phase I	State-of-the-Art Survey—Report Issued
Phase II	Ingot Development—Report Issued
Phase III	Development of Rolling Operations—Report Issued
Phase IV	Process Uniformity Verification and Post-Rolling Development—In Process
Phase V	Final Pilot Production

II. Phase IV - Program Objectives

The objectives of this phase are threefold:

A. Verify Process Uniformity

Using the sheet rolling parameters established in Phase III, several sheets of each gauge will be rolled. Physical and mechanical properties will be determined and compared to establish the degree of control which can be expected.

B. Scale-up to 36" x 36" Sheet

All efforts in this phase are toward processing 36" x 36" sheet. Many problems usually evolve when wider widths are attempted. The extent to which these problems will occur on this program can only be determined through rolling experience.

C. Post-Rolling Development

Goals which have been established for the physical quality of the final material are shown below. Although all are affected by the actual rolling operation, additional post-rolling practices will be utilized in an attempt to meet these goals.

1. Surface finish—Number 2 Matte
2. Gauge Control—± 5%
3. Flatness—4% per MAB 176-M
4. Gauge Tolerance—1/2 of AMS 2242

In initiating work on this phase, a scale-up of the melting and extrusion practices were required and most of the effort during this period was expended in this area.

III. Ingot Melting

The minimum ingot size requirement in this phase was conditioned 6" diameter in order to achieve an extrusion with a cross section compatible with the sheet size requirements. In Phase II an attempt was made to scale-up, however that resulted in complete failure principally due to melting furnace deficiencies. An additional problem at that time was starting material technology. The quality requirements of electrodes were not known and methods of assembling (joining) electrodes in the furnace had not been established.

Extensive modifications were made to the arc melting furnace. These included additional power and modified power input, modified cooling and a change in the electrode feed mechanism. Concurrently, an investigation of electrodes was completed and a quality specification written. Machining investigations were run and satisfactory threading procedures established. Connecting nipples were made by extruding small ingots to 1-5/8" diameter and subsequently threading these.

Four ingots were melted into an 8" ID mold. The melting conditions were essentially satisfactory, however, at intervals the melt became very erratic. This was attributed to the basic electrode as the conditions would initiate when proceeding from one bar to the next, and would stop when this bar was consumed and the melting of the next bar initiated. A typical as-cast ingot is shown in Figure 1. The melting history and billet yields for these ingots is shown in Table I.

TABLE I

Ingot Melting and Processing

	Heat Number			
	1147	1148	1167	1168
Mold Dia. (in.)	8	8	8	8
Electrode Dia. (in.)	3-1/4	3-1/2	3-7/8	3-7/8
Weight Melted (#)	518	523	571	453
Cond. Weight (#)	231	233	301	153
Yield %	44.7	44.6	52.8	33.8

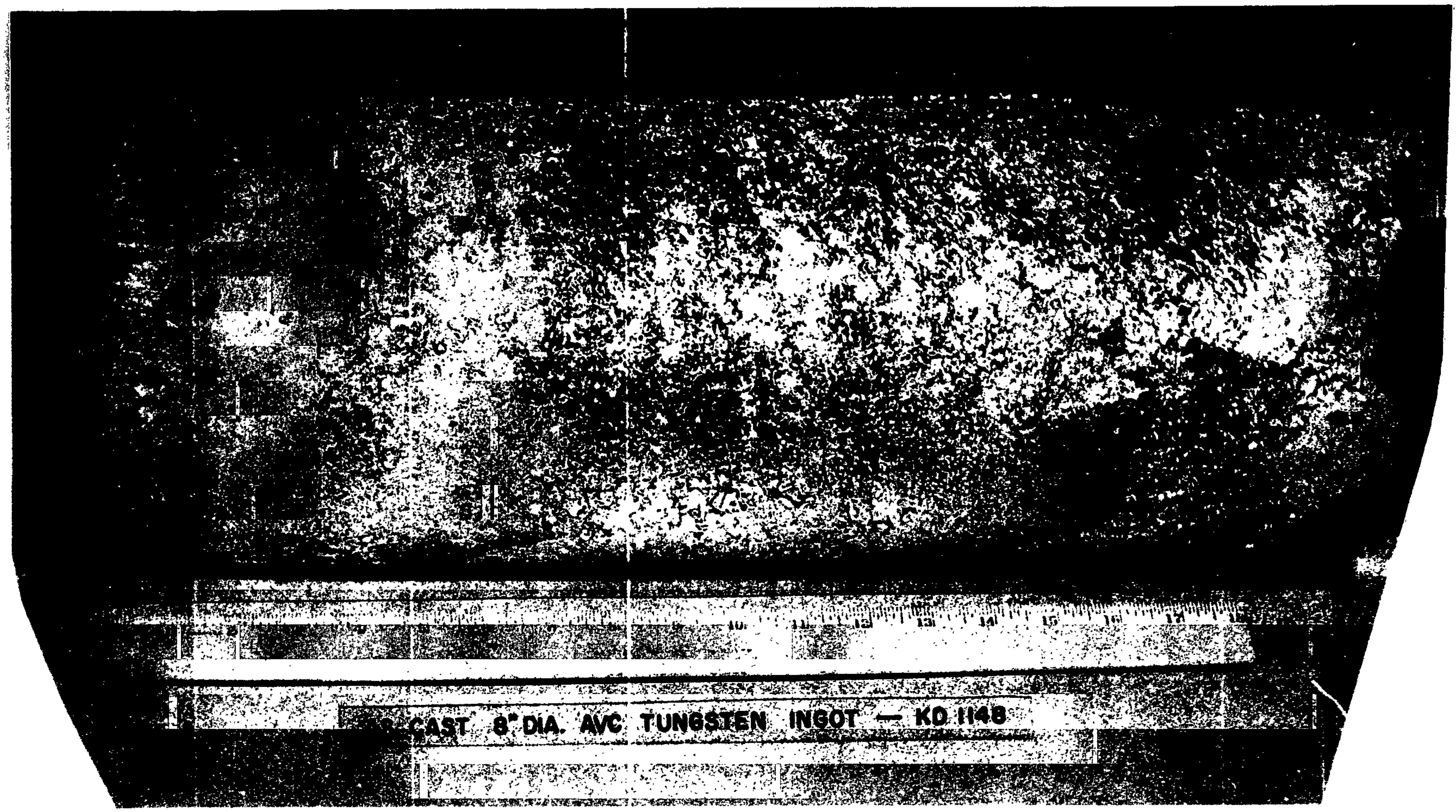


FIGURE 1 - AS-CAST 8" DIAMETER INGOT

It will be noted from the table that the yields were relatively low. This is due largely to the fact that, in order to insure a completely satisfactory ingot at the required 6" diameter, an 8" mold was used. In observing the machining of these ingots, sidewall porosity was eliminated in every case at 7" to 7-1/4", so that a much higher yield could have been realized if a 7" extrusion container were available. The other area of appreciable yield loss was on the hot top of three of the four ingots. On these three the average yield loss for hot top cropping only was 14.5%.

Chemical analysis of the starting electrode is listed in Table II. Table III lists the ingot chemistries. In comparing the two tables, two elements deserve discussion. In the second electrode powder lot the nickel content is relatively high and well above the 20 PPM maximum specification level. Rather than reject the material because of considerable time delay, the material was melted subject to rejection if the ingot chemistry were not satisfactory. As shown in the ingot chemistry for Heats KD1167 and 1168, the nickel content using these electrodes was below the 1 PPM detection limit. The molybdenum content in the two powder lots is shown to be 8 and 11 PPM respectively. In the ingot chemistries only one heat is below 100 PPM. This large deviation between electrode and ingot chemistry has been a continuing problem, yet unresolved. Heats 1147 and 1148 were both melted using the first powder lot, yet the molybdenum content is 100 and 500 PPM respectively. The remaining two heats were melted using the second powder lot and the ingot analyses are <10 and 190 PPM respectively.

It would appear from the work to date that the molybdenum in the powder lot is not uniform, however, analyses of the powder lot are consistently low.

TABLE II

Electrode Chemical Analyses

Element

Lot	As	Al	Ca	Cr	Cu	Fe	K	Mg	Mo	Na	Ni	Si
964	<3	0.6	6	2	0.1	4	<20	<2	8	7	6	3
996	<3	9	6	18	0.1	14	35	<2	11	20	45	3

TABLE III

Ingot Chemical Analyses

Element

Lot	Mn	Al	V	Cr	Cu	Fe	Co	Mg	Mo	Ti	Ni	Si	C	O	N	H
1147[1]	<10	<10	<10	<10	< 1	13	<5	< 1	100	< 1	< 1	<20	30	14	20	1.9
1148[1]	<10	<10	<10	<10	< 1	13	<5	< 1	500	< 1	< 1	<20	30	11	21	1.6
1167[2]	<10	<10	<10	<10	< 1	12	<5	< 1	<10	< 1	< 1	<20	10	9	2	< 1
1168[2]	<10	<10	<10	<10	< 1	5	<5	< 1	190	< 1	< 1	<20	44		14	

1 - Electrodes from powder lot 964

2 - Electrodes from powder lot 996

All analyses in PPM

(<) Indicates analysis was below detection limits

Based on the fact that these initial ingots were free of porosity at 7" to 7-1/4", it was concluded that a nominal as-cast 8-5/8" ingot should "clean-up" at approximately 7-7/8" which would be satisfactory for the 8" extrusion container. The existing 8" ID mold was machined to 8-5/8" ID and two additional ingots melted. No data are available on these two ingots as they have not been machined. It is doubtful if they will be free of porosity at 7-7/8", however, as the cooling shrinkage was much greater than anticipated. Both ingots as-cast were 8-1/8" to 8-1/4" diameter so that the shrinkage was a nominal 1/2" on the diameter. This compares with a nominal 1/4" shrinkage on the previous 8" ingots.

IV. Extrusion

Four billets were extruded on the du Pont 2750 ton press. Two were extruded to 3" diameter rounds for subsequent press forging to sheet bar and the other two were extruded directly to 1-3/4" x 4" cross section sheet bar. Table IV lists the extrusion parameters used and the resultant pressure requirements. As shown, all billets were extruded at a Shawmeter temperature of 3200°F. based on the successful results of 4" billets extruded at this temperature. The break-through pressure requirements were relatively consistent except for the first extrusion which was somewhat higher. It is shown by the running pressure that the sheet bar extrusions require slightly more pressure than the rounds. The average extrusion constant of the previous 4" billets was 81,600 psi. As shown, this is lower than the first billet but higher than the other three.

Figure 2 shows the two as-extruded rounds after sandblasting, with the end cropping requirements, determined by contact ultrasonic, indicated. The relatively large amount to be cropped off

TABLE IV

Extrusion Data for 6" Diameter Billets

Heat Number	Billet Weight	Temperature °F.		Pressure - psi		Speed	Extrusion
		Shaw	Optical	Breakthrough	Running	IPS	Constant (K)
1147	231	3200	2680	118,000	97,000	16.5	85,000
1148	233	3200	2665	108,000	104,000	16	78,000
1167	303	3200	2675	100,000	93,000	17	72,250
1168	153	3200	2705	108,000	105,000	18	78,000

Extrusion Constant $K = \dfrac{P}{A \ln \frac{A}{a}}$ where:

P = maximum pressure in pounds
K = extrusion constant in psi
A = cross sectional area of container
a = cross sectional area of extrusion

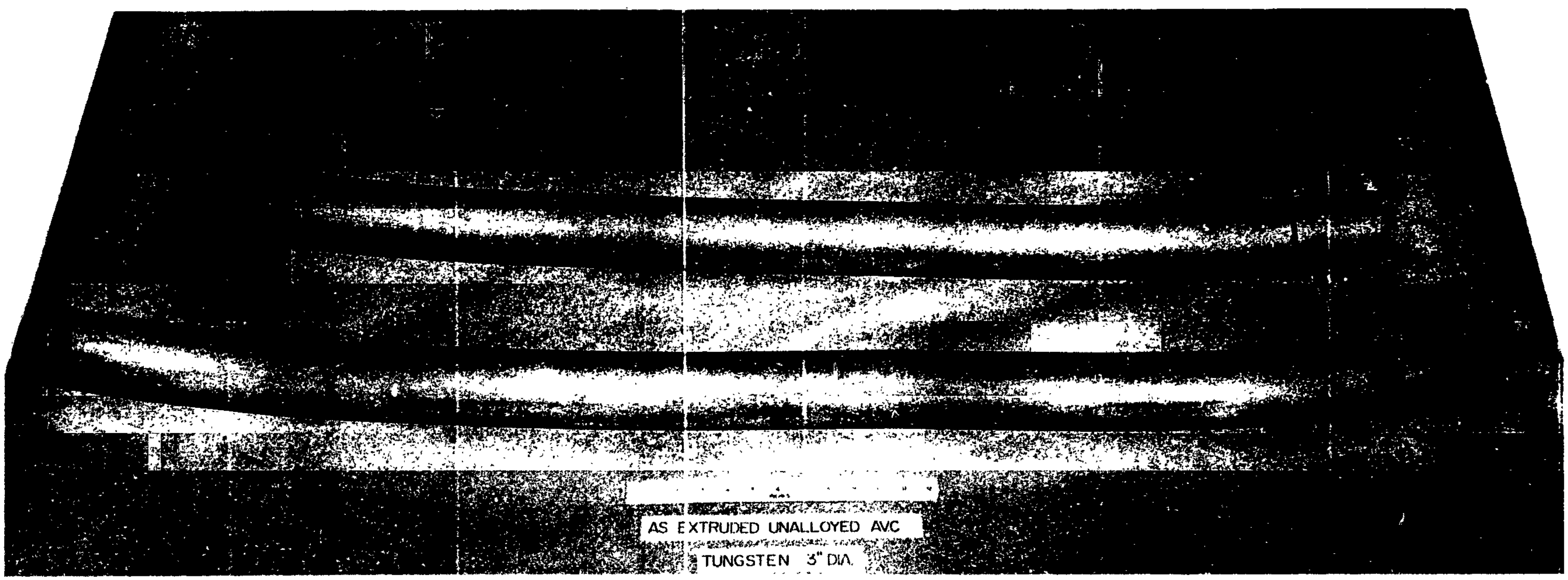


FIGURE 2 - AS-EXTRUDED 3" DIAMETER ROUNDS

the tail (nominal 4") is due to a deep tail pipe but actually does not represent a completely solid piece since this area is hollow. The picture shows that the general surface was excellent and it should also be noted that no die wash occurred. Actually, both billets were pushed through the same die which had not been accomplished previously. An as-extruded sheet bar is shown in Figure 3. This picture shows also that no die wash occurred which is remarkable considering the sharp corner angles required on the sheet bar die. The fact that no die wash occurred on these extrusions is further verified by the physical dimensions shown in Table V.

TABLE V

Physical Dimensions of Extrusions

	Heat Number			
Cross Section	1147	1148	1167	1168
Nose	3.034"Dia.	3.984"x 1.767"	3.043"Dia.	3.975"x 1.765"
Tail	3.018"Dia.	3.990"x 1.765"	3.030"Dia.	3.975"x 1.768"
As-Extruded Length	48	49	59	32

To more accurately evaluate the internal quality of the round extrusions, immersion ultrasonic evaluation was required. In order to accomplish this, they had to be straightened. They were, therefore, heated to 2300°F. in a hydrogen atmosphere furnace, straightened on a 1500 ton press and subsequently reheated to 2300°F., soaked for ten minutes and then buried in vermiculite.

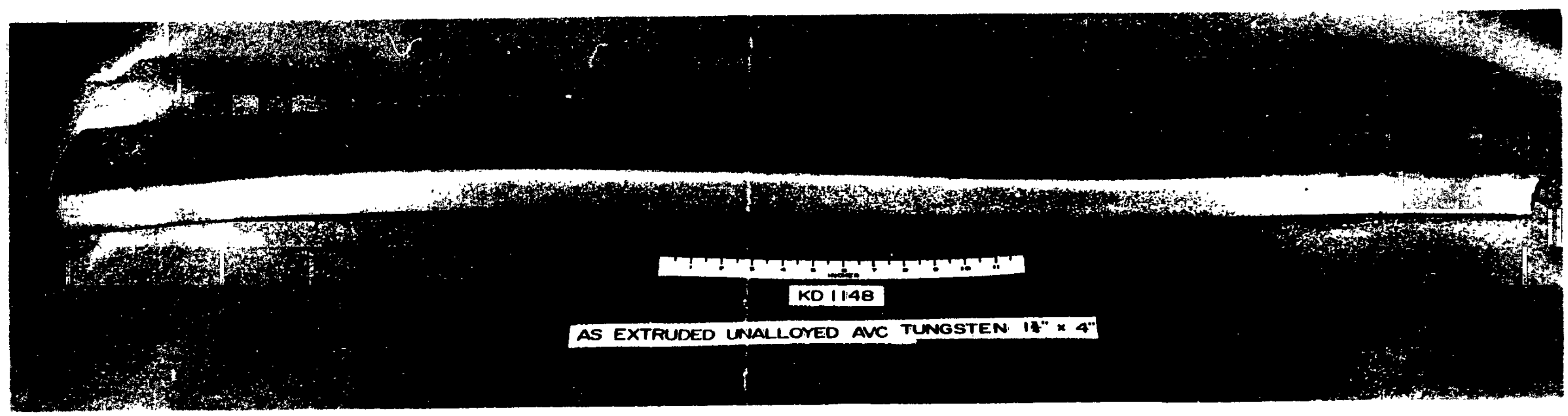


FIGURE 3 - AS-EXTRUDED 1-3/4" x 4" CROSS SECTION SHEET BAR

Immersion ultrasonic examination indicated that on both extrusions a longitudinal crack varying in depth up to 1/2" extended along the entire length, however, the results were not precise due to slight surface defects. In addition, 12" on the trailing end of 1167 appeared to be cracked from surface to center. As the surface defects were preventing an accurate evaluation, the extrusions were machined to 2.850" and then surface ground to 2.830". They were immersion ultrasonic examined again and the results are plotted in Figure 4. As shown in this figure, 1147 was free of defects except for 2.5" on the nose end. On 1167, 12" on the trailing was cracked tapering from the surface to the center. In addition, 11" was cracked to a maximum depth of 1/4" and 1" on the nose was cracked to a depth of 1/2". As shown on this diagram, two forging mults were cropped from each extrusion. The cracked areas on 1167 were ground out prior to forging.

After cropping the as-extruded sheet bar, minor surface conditioning was required to prepare it for subsequent rolling. This extrusion was also cropped into two mults for the rolling operation.

Macro discs were cropped from the ends of all extrusions. The macro structure of 1167 is shown in Figure 5. Note that the nose section has a much larger grain size than the tail. This can be attributed to the fact that the extreme nose end of a billet in proceeding through the extrusion die is not worked, particularly in the center, to the extent of material back several inches from the nose.

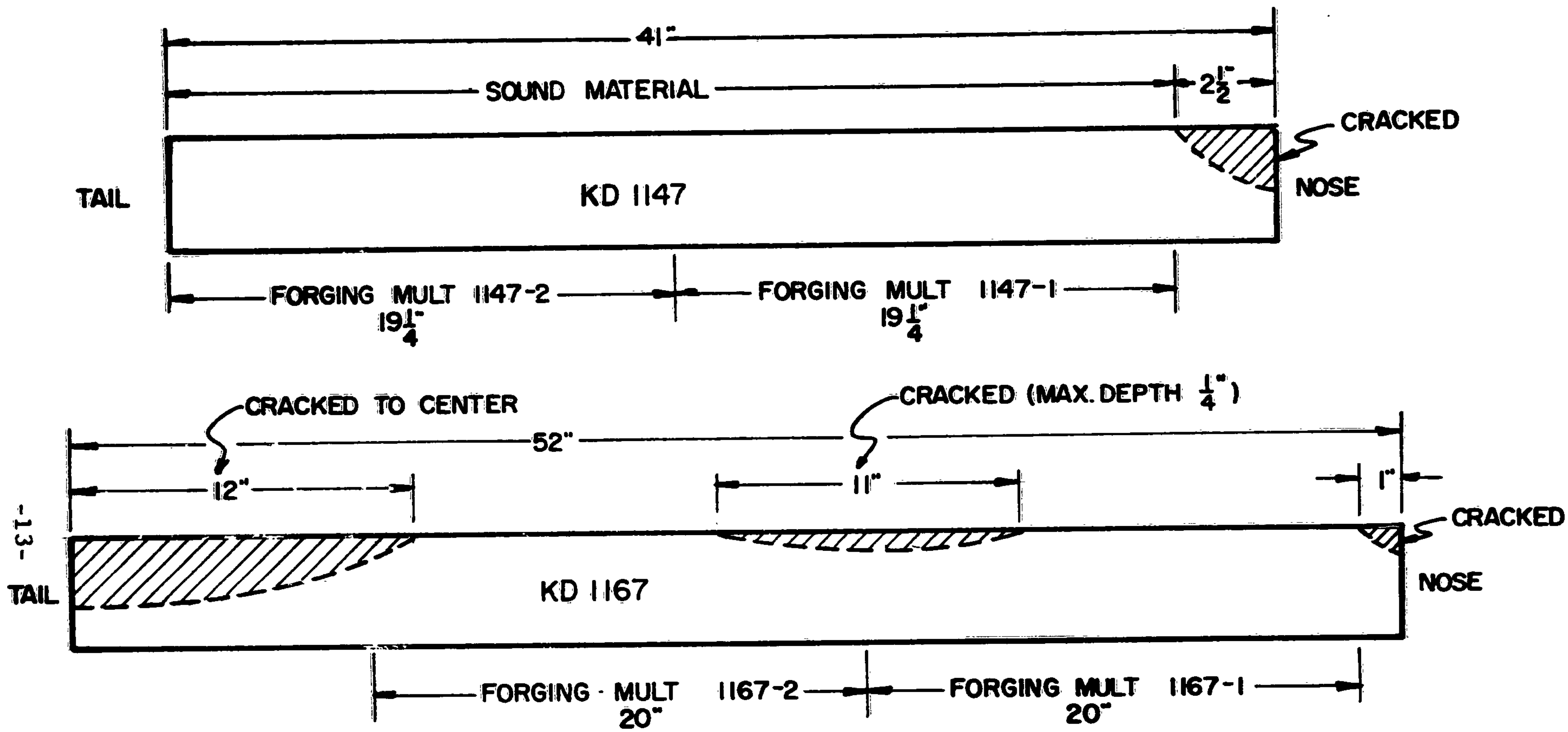


FIGURE 4
ULTRASONIC EVALUATION OF EXTRUDED ROUNDS

RTA-0294

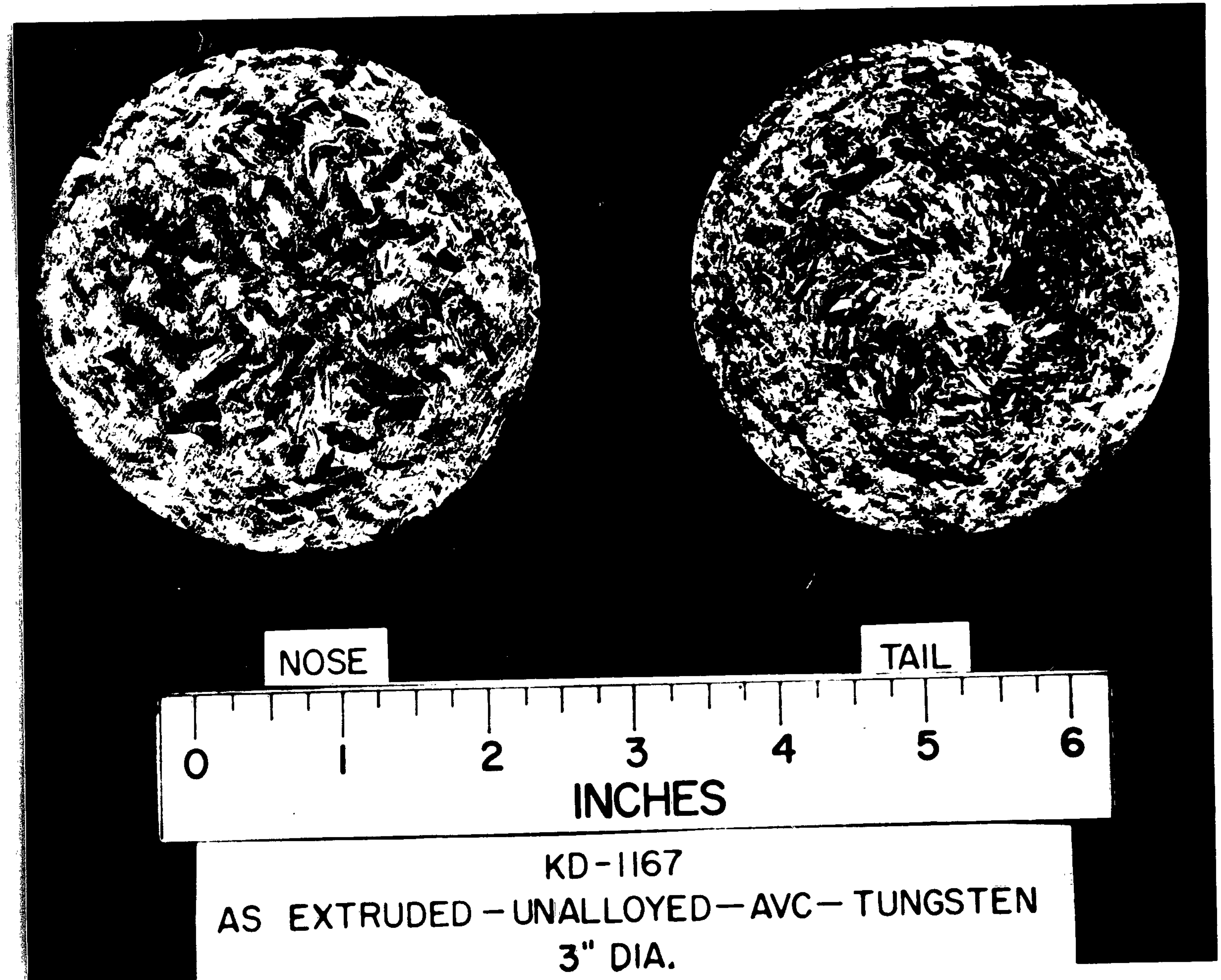


FIGURE 5 - MACROSTRUCTURE OF AS-EXTRUDED 3" DIAMETER ROUND

Figure 6 shows the macrostructure of the as-extruded sheet bar. The nose section shows a relatively equiaxed grain structure and the tail shows a wrought fiberous structure. The equiaxed structure in the nose is probably related to its close proximity to the area of initial deformation thus producing the same effect discussed for the extruded rounds.

A hardness survey was made on the nose and tail of each extrusion. This information is plotted in Figures 7,8,9 and 10. In comparing the two extruded rounds, it will be noted that the average hardness of the tails is within 1 DPH. The nose of 1147 is slightly harder than 1167 which can be attributed to the fact that 1167 hardness values were taken closer to the nose and therefore represent material with less work. As shown, the sheet bars are harder than the rounds. This can be attributed to a higher degree of work related to the sheet bar configuration.

V. Sheet Bar Forging

For the forging operation, a 1500 ton hydraulic press was used in conjunction with a hydrogen atmosphere furnace. As this press was relatively slow acting, the pieces were heated to 2600°F. in order to maintain a nominal 2000°F. forging temperature. The actual forging process used was as follows:

1. Charge forging mult into 2600°F. furnace;
2. Soak five (5) minutes after reaching temperature;
3. Transfer to press and forge 3/4" flats as shown in Figure 11;
4. Reheat to temperature and hold five minutes;
5. Transfer to press, rotate 90° to initial forging direction and forge to nominal 2" thick;
6. Reheat to temperature, hold 10 minutes, discharge and bury in vermiculite.

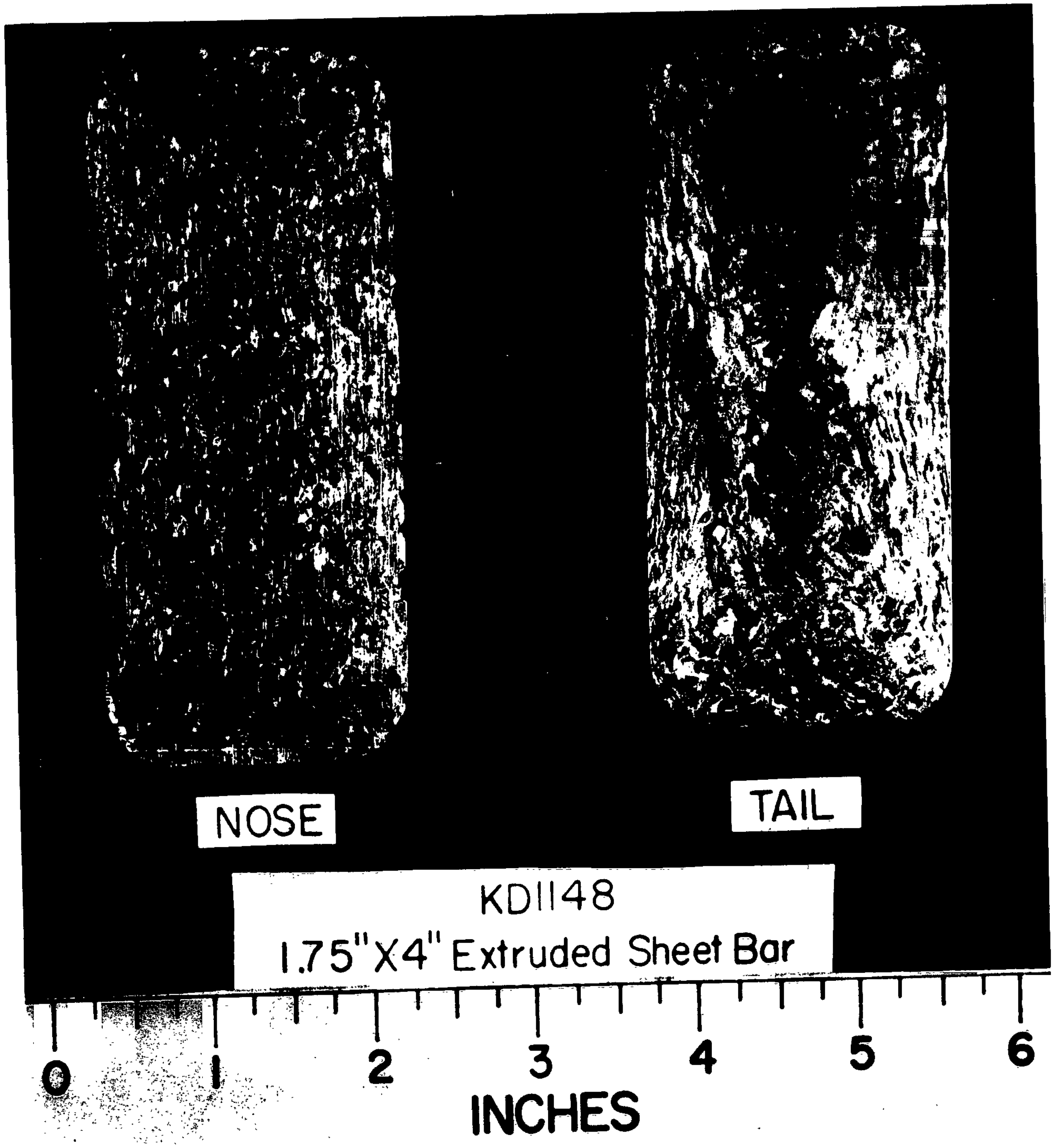


FIGURE 6 - MACROSTRUCTURE OF AS-EXTRUDED SHEET BAR

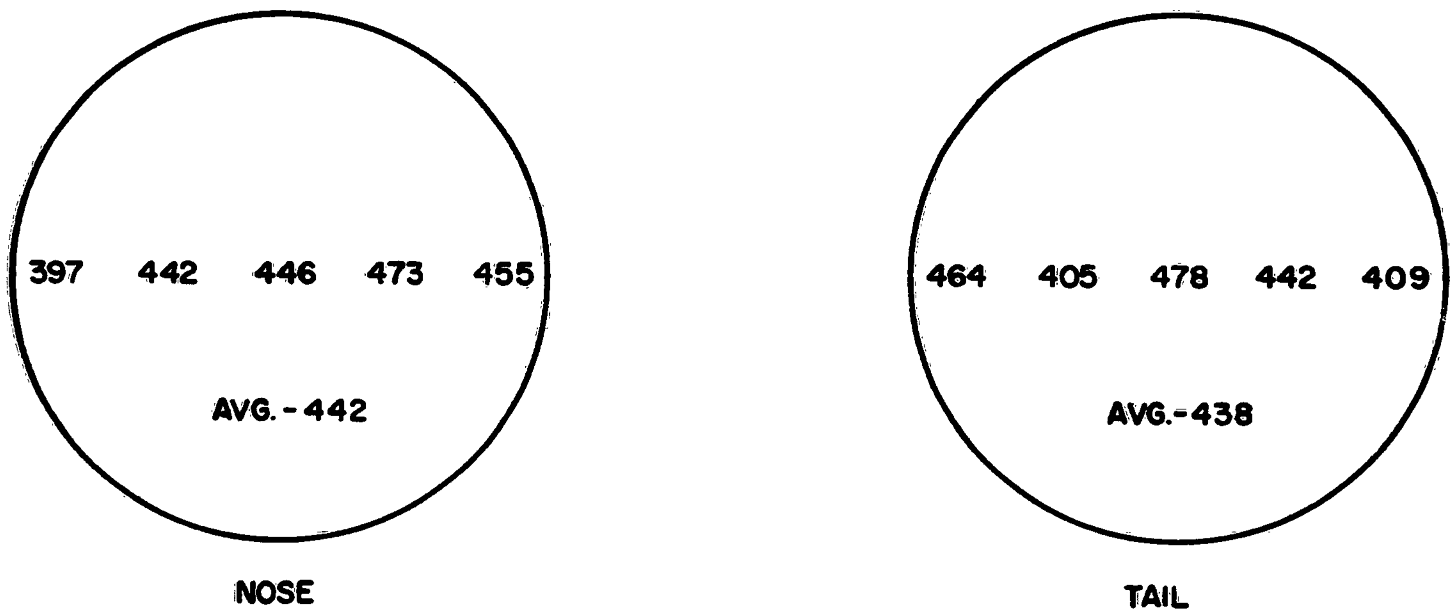


FIGURE 7
DPH HARDNESS ON KD 1147 AS-EXTRUDED
(10 KG LOAD)

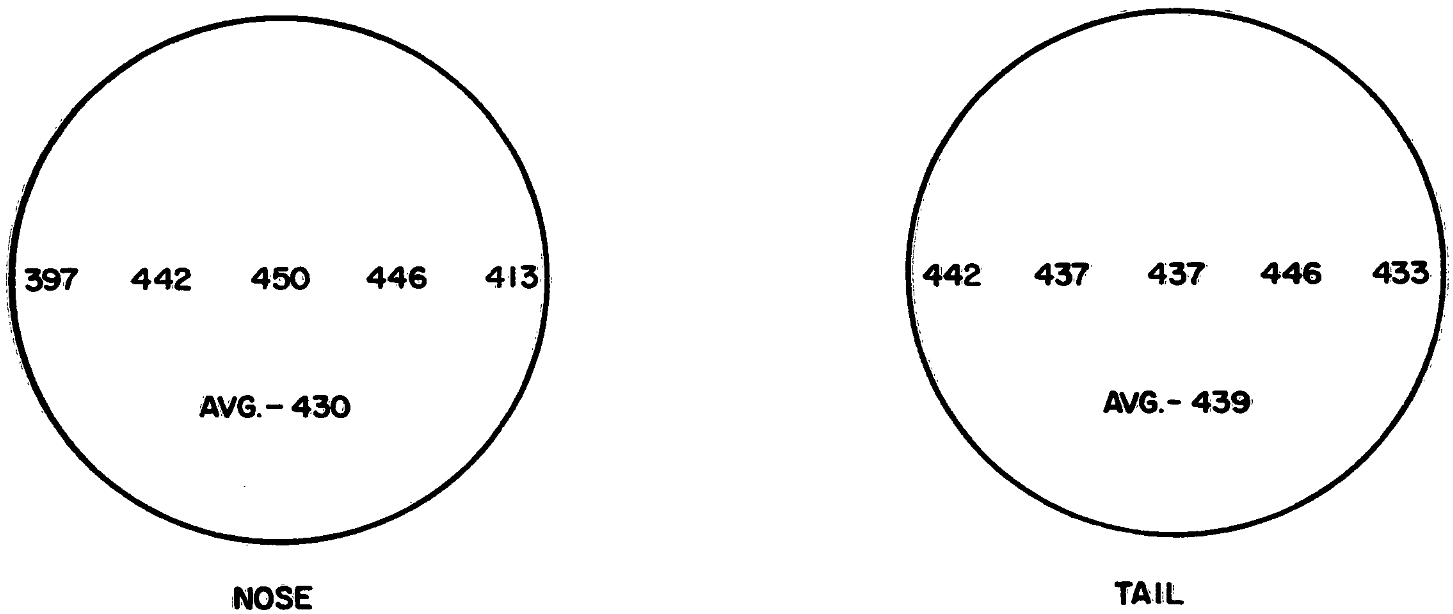


FIGURE 8
DPH HARDNESS ON KD 1167 AS-EXTRUDED
(10 KG LOAD)

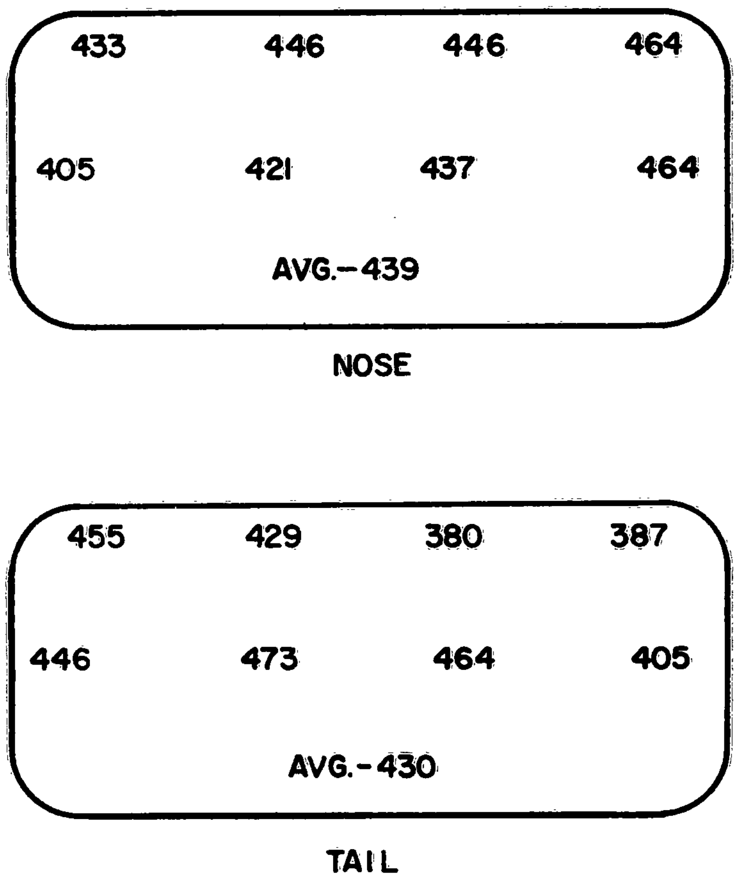


FIGURE 9

DPH HARDNESS ON KD 1148 AS-EXTRUDED
(10 KG LOAD)

RTA-0297

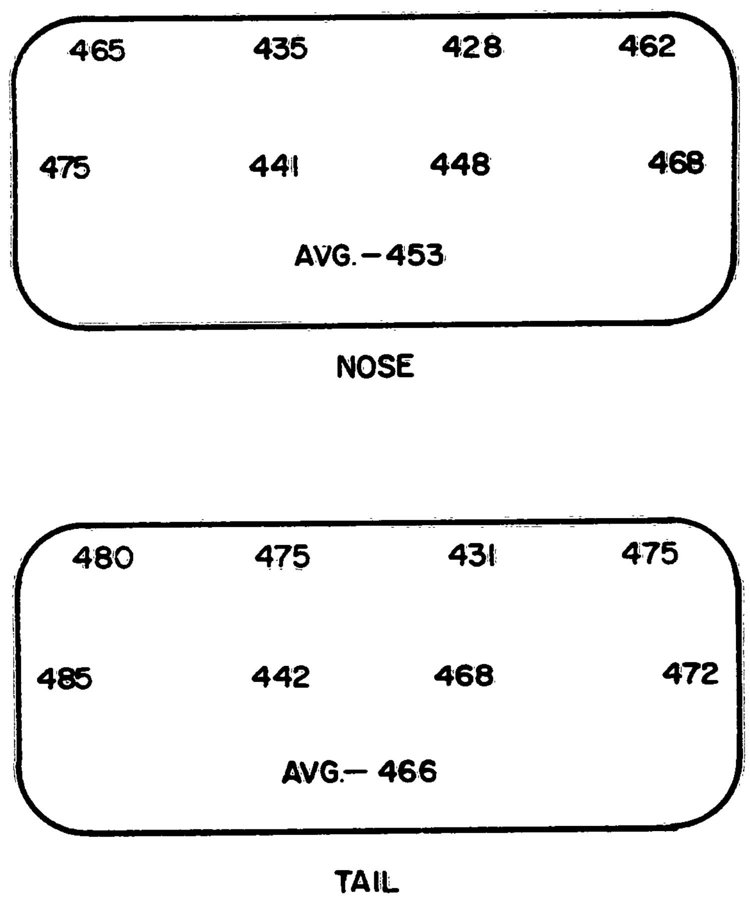
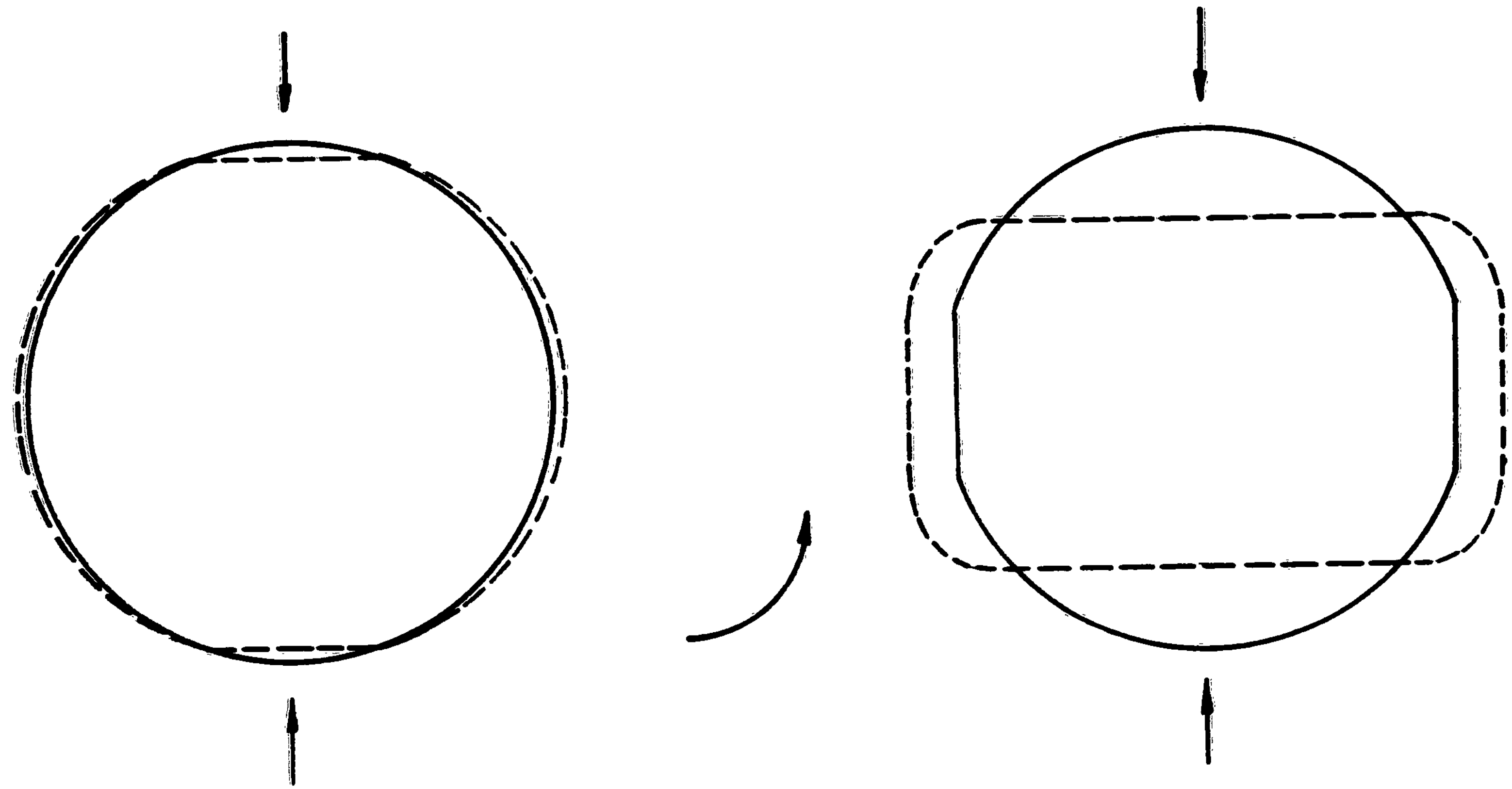


FIGURE 10
DPH HARDNESS ON KD 1168 AS-EXTRUDED
(10 KG LOAD)

RTA-0298



PRESS 1" FLATS — REHEAT — ROTATE 90° — FORGE TO 2" THICK

—— REPRESENTS INITIAL CROSS SECTION
----- REPRESENTS CROSS SECTION AFTER FORGING OPERATION

FIGURE 11
SHEET BAR FORGING SEQUENCE

RTA-0299

In Step 5 above, the press stalled out at a nominal 2-3/16" thick. The first mult was reheated to temperature and an attempt made to forge it down to 2", however, only 1/16" additional reduction was achieved. The remaining three pieces were only forged once in Step 5. After slow cooling, the pieces were sand blasted for inspection. The four mults are shown in Figure 12. Although no cracks are visible, closer inspection showed light surface ruptures on all of the pieces. Two of the pieces had one larger crack running parallel to the extrusion direction. These were probably related to the cracks initially indicated on the extrusions but which were supposedly removed by machining and grinding. It is suggested that these cracks were present in the conditioned extrusion but the depth was so minor that ultrasonic inspection did not pick them up. Although the pieces had also been dye penetrant inspected, flowed metal on the surface prevented detection by this method. These cracks in both mults were conditioned out at a depth of 1/8". The remaining surface of these two and the remaining two were ground lightly to remove the light surface ruptures. The yield losses on extrusion and forging are presented in Table VI.

TABLE VI

Yield Summary from Extrusion Billet to Sheet Bar

Heat No.	Extruded Weight	End Loss	Surface Loss	Forging Mult Weight	Cond. Sheet Bar	% Yield Extrusion to Sheet Bar
1147	231	23-1/4	33-1/2	168 2 pcs.	145 2 pcs.	62.8
1148*	233	25	12	--	192 2 pcs.	82.5
1167	301	72	35	180-1/2 2 pcs.	160-1/2 2 pcs.	53.3
1168*	153	27-3/4	Not Completed			

* Extruded directly to sheet bar

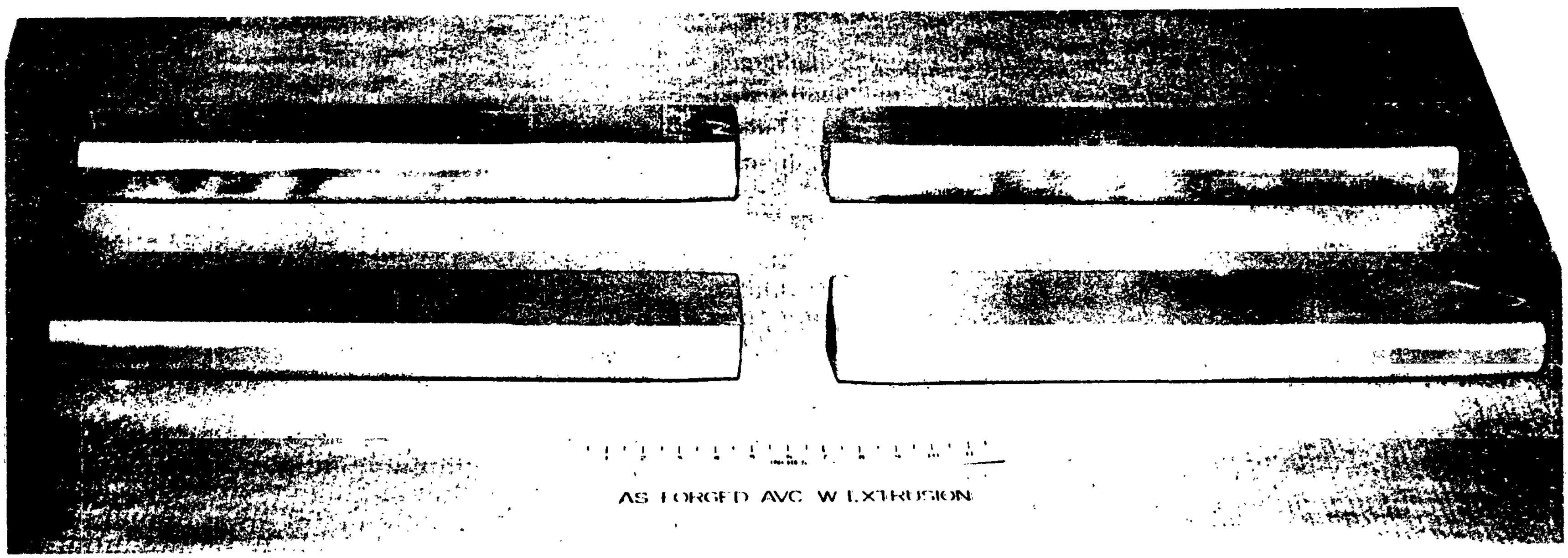


FIGURE 12 - PRESS FORGED SHEET BAR FROM EXTRUDED 3" DIAMETER ROUNDS

It will be observed immediately from the table that the yield in extruding directly to sheet bar is significantly higher and, in addition, eliminates the forging step. The low yield in 1167 is due in part to the cracked portions of the extrusion which in the table are included as end losses.

VI. Sheet Rolling

Two extruded sheet bar, mults 1148-1 and 1148-2, and two press forged sheet bar, 1167-1 and 1167-2, were rolled to an intermediate gauge of 1" using a 2300°F. furnace temperature. One pass per reheat was used for this initial rolling step with the reductions per pass shown in Table VII.

TABLE VII

Reduction Schedule for Initial Rolling

Mult Code	Gauge After Pass Indicated					
	Initial	First	Second	Third	Fourth	Fifth
1148-1	1.70	1.50	1.32	1.15	1.025	---
1148-2	1.70	1.47	1.30	1.15	1.015	---
1167-1	2.00	1.75	1.52	1.32	1.15	1.015
1167-2	2.00	1.75	1.52	1.32	1.15	1.010

No visible cracking occurred during rolling. No further work was accomplished to date.

VII. Conclusions

A. Arc cast tungsten ingots can be melted which will condition to 6" diameter extrusion billets.

B. The extrusion of 6" billets to 3" diameter and 1.75" x 4" sheet bar can be accomplished satisfactorily; however, minor cracking problems did occur in the rounds which will require some modification to the extrusion practice.

C. Sheet bar can be press forged from 3" diameter rounds in the temperature range of 2000°F.

D. Initial rolling of both press forged and extruded sheet bar can be accomplished in the temperature range of 2300°F.

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