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AEC RESEARCH AND  
DEVELOPMENT REPORT

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**DEVELOPMENT OF  
HIGH STRENGTH COLUMBIUM  
AND TANTALUM ALLOY TUBING**

FIRST QUARTERLY PROGRESS REPORT  
DECEMBER 1, 1962 - FEBRUARY 28, 1963

APRIL 11, 1963

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UC-25, Metallurgy and Ceramics  
(TID-4500, 20th Edition)

FIRST QUARTERLY PROGRESS REPORT ON  
DEVELOPMENT OF HIGH STRENGTH  
COLUMBIUM AND TANTALUM ALLOY TUBING

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December 1, 1962 - February 28, 1963

R. W. Buckman, Jr.

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Development of High Strength Columbium  
and Tantalum Alloy Tubing

First Quarterly Progress Report

by

R. W. Buckman, Jr.

ABSTRACT

The production of tubing from B-66 (Cb-5V-5Mo-1Zr) and T-111 (Ta-8W-2Hf) requires the evaluation of the extrudability of these alloys at high reduction ratios (i. e., 7:1). Starting billets are being clad in mild steel and will be extruded within the 2000-2200°F temperature range. Material for the extrusion evaluation has been procured. The tube blank size of 2" O. D. x 1/4" thick wall will be extruded from a 4.22" O. D. x 1.30" thick wall starting billet.

B-66 tube blanks will be worked initially at 500-800°F and finish drawing operations will be done at room temperature. A portion of the B-66 billet material from the extrusion evaluation will be used for a warm drawing investigation. T-111 tube blanks will be processed at room temperature because of its excellent low temperature ductility.

During the next quarter, it is planned that the extrusion parameters will be defined and extrusion of tube blanks completed. In addition, the warm drawing evaluation of B-66 will be initiated.

## SUMMARY

This is the first quarterly progress report on AEC Contract AT(30-1)-3108, "Development of High Strength Columbium and Tantalum Alloy Tubing." The technical program for the production of tubing from B-66 and T-111, present status of progress, and the work outline for future activities is presented.

FIRST QUARTERLY PROGRESS REPORT ON  
DEVELOPMENT OF HIGH STRENGTH  
COLUMBIUM AND TANTALUM ALLOY TUBING

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## I. INTRODUCTION

This is the First Quarterly Progress Report on AEC contract AT(30-1)-3108, "Development of High Strength Columbium and Tantalum Alloy Tubing." The period covered is from December 1, 1962 to February 28, 1963.

The purpose of this program is to develop processing techniques for the production of tubing from the Westinghouse-developed alloys B-66<sup>Cb</sup> (Cb-5Mo-5V-1Zr) and T-111<sup>Ta</sup> (Ta-8W-2Hf). The tubing<sup>Cb, Ta</sup> produced under this contract will be evaluated for application in liquid metal containment at elevated temperature. The following tubing sizes will be produced on a best effort basis.

<u>O. D., inches</u>	<u>Wall Thickness, inches</u>	<u>Approximate Length, feet</u>
0.500	0.062	50
0.375	0.062	45
0.250	0.020	50

The production of the B-66 and T-111 tubing will require evaluation of three processing areas. They are:

- (1) Extrusion<sup>Cb, Ta</sup> of tube blanks
- (2) Tube blank reduction to redraw stock
- (3) Drawing<sup>Cb, Ta</sup> to finished sizes

The basic program outline for accomplishing the above is outlined in Figure 1. A detailed discussion of the technical aspects of the program is presented in Section IV.

## II. PROGRAM STATUS

During the initial quarterly period, a trip was made to Nuclear Metals, Inc., Concord, Massachusetts, to define technical details of the tube blank extrusion program.

The extrusion of B-66 and T-111 tube blanks will be conducted in two phases as shown

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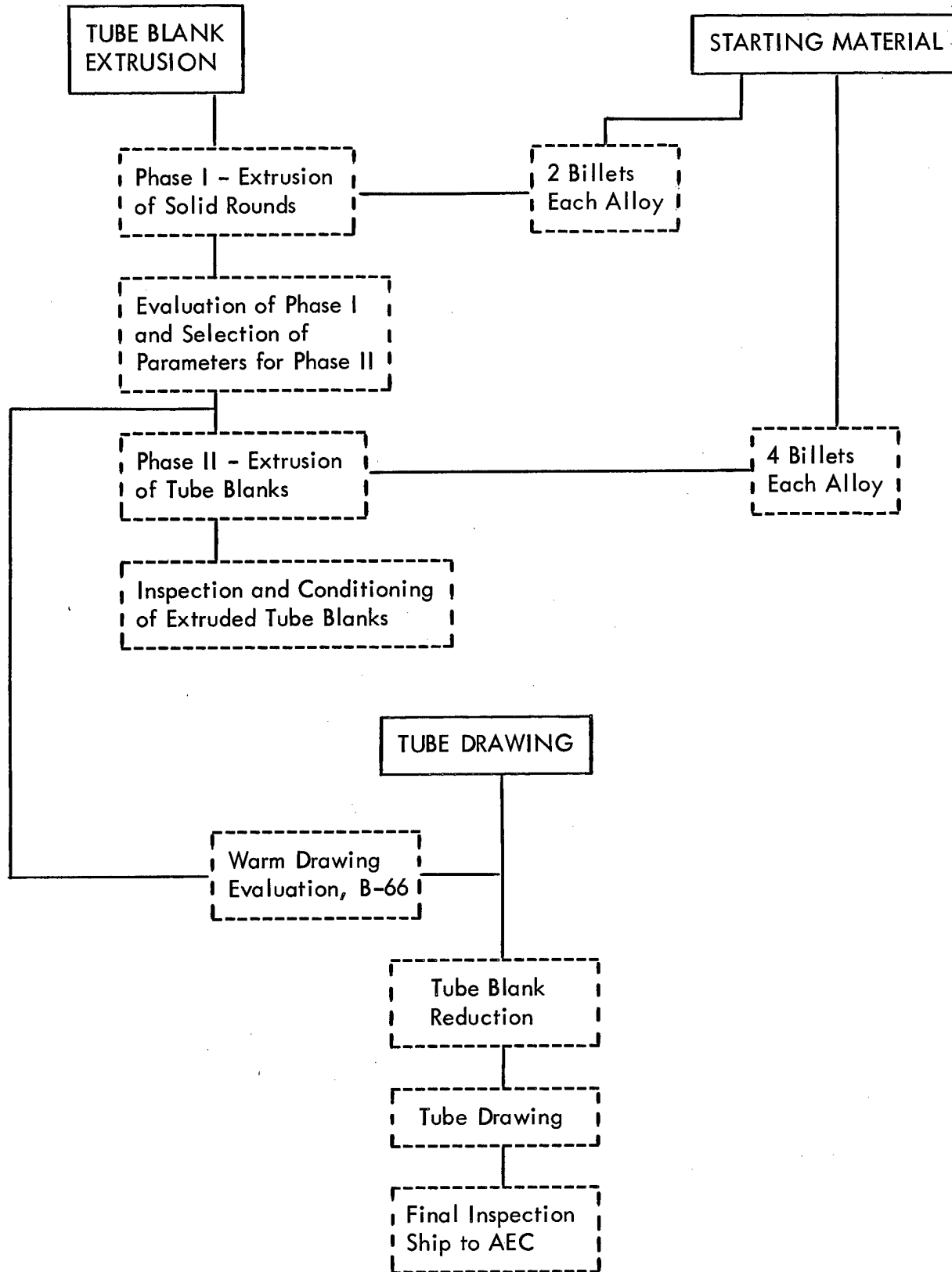


FIGURE 1. Program Outline for Producing B-66 and T-111 Tubing

in Figure 1. The basic parameters that have been defined are listed in Table I. Selection of the Phase II extrusion parameters are tentative pending the results obtained from the Phase I evaluation. A detailed discussion of the extrusion portion of the program is presented in Section IV.

*Type* →

TABLE I. Parameters for B-66 and T-111 Tube Blank Extrusions

	Phase I	Phase II
Billet Size	2.740" OD x 5-3/16" L	4.22" OD x 1.635" ID x 5-3/4" L
Billet plus Cladding	2.92" OD x 5-1/2" L	4.47" OD x 1.510" ID x 6" L
Cladding Material	Mild Steel	Mild Steel
Container Size	3"	4.5"
Reduction Ratio	7:1	7:1
Die Design	Cone Type-90° included Angle	Cone Type-90° included angle
Number of Billets	Two, each alloy	Four, each alloy

B-66 and T-111 starting material are being obtained from the Westinghouse Metals Plant pilot production stock in the form of wrought billets. The B-66 tube blank billets have been processed and will be shipped when proper authorization has been received. The solid B-66 extrusion billets have been processed and are being machined to the required configuration. The B-66 starting material has been processed from a single heat designated DX-601T. The ingot chemistry of DX-601T is given in Table II.

TABLE II. Ingot Analysis of B-66, Heat No. DX-601T

Ingot Position	Analysis, weight per cent						
	Mo	V	Zr	C	O	N	
Top	half-radius	5.0	4.8	0.94	.0023	.0012	.0067
	center	5.0	4.6	0.94	--	--	--
Bottom	half-radius	5.2	4.8	0.95	.0046	.0095	.0072
	center	5.2	5.0	1.00	--	--	--
Mid-Height	sidewall	5.0	5.2	1.09	--	--	--

The T-111 tube blank extrusion billets are scheduled for delivery in the latter part of April. The pilot plant production ingot from which this material will be obtained is being processed by the Westinghouse Metals Plant. The primary breakdown of this ingot will be by extrusion and is scheduled during the week of April 10, 1963. T-111 billets for the extrusion evaluation phase have been procured from material that was on hand at the Astronuclear Laboratory. The composition of this T-111, heat No. DX-571 is given in Table III. The use of multiple heats of T-111 will not introduce any significant undetermined variation. Sufficient T-111 has been melted to demonstrate reproducibility of chemistry and mechanical properties.

TABLE III. Ingot Analysis of T-111, Heat DX-571

Ingot Position	Analysis, weight per cent					
	W	Hf	C	N	O	
Top	center	8.15	2.03	--	--	--
	mid-radius	7.97	2.00	.001	.0016	.001
	edge	8.07	2.23	--	--	--
Bottom	center	8.20	1.93	--	--	--
	mid-radius	8.20	1.97	.001	.0027	.0011
	edge	8.15	1.93	--	--	--

Tube drawing of the extruded tube blanks will be done at the facilities of the Superior Tube Company, Norristown, Pennsylvania.

### III. REVIEW OF RELATED WORK

The feasibility of producing T-111 tubing has been demonstrated on a Westinghouse-supported program. Approximately, eight feet of 3/8" OD x 0.035" thick wall tubing has been processed. A starting tube blank was produced by extruding a 3" diameter, double vacuum melted arc cast ingot to a 1-7/8" diameter round. The extruded billet was machined to give a tube blank 1.5" diameter with a .220" thick wall. The surface finish of the ID was



32 rms. The initial tube reduction of 73 per cent was accomplished on the blank at room temperature. All processing was done at ambient temperatures with intermediate in process annealing being done at  $1400^{\circ}\text{C}$  ( $2550^{\circ}\text{F}$ ) at a maximum pressure of  $5 \times 10^{-5}$  torr.

The Air Force is sponsoring production of tubing from two columbium base alloys, B-66 and X-110, at the DuPont Metals Plant under Contract AF33(600)-40700. (1, 2)

The objectives of this program are essentially identical with the AEC contract effort in that the final tubing sizes are the same. However, the approach being used by DuPont is somewhat different than that being used at the Astronuclear Laboratory. DuPont is using high temperature glassing practice for tube blank extrusion of both B-66 and X-110.

The results reported indicate that the extruded B-66 tube blanks contain extrusion defects which cannot be removed except by excessive local conditioning. The defects have been observed on both ID and OD surfaces. The transverse ID defects observed on the B-66 extrusions were attributed to lubricity problems associated with the particular glassing practice used for the extrusion. The pertinent features of the processing of B-66 and X-110 tube blanks by DuPont are: (1)

- (1) Starting billets 3.90" OD x 1.225" ID are wrought, and have been produced from an 8" diameter ingot.
- (2) The extrusion ratio is approximately 8:1.
- (3) The billet is heated to  $3000-3200^{\circ}\text{F}$  for extrusion.

The effect of temperature on the extrusion constant of B-66, extruded from 8" diameter as cast ingots at reduction ratios of 2.8 to 3.6:1, is shown in Figure 2. The extrusion constants used for this plot were calculated using the breakthrough or upset pressure.

Extrusion constants for ingots extruded bare at  $2800-3100^{\circ}\text{F}$  using a high temperature glassing technique are equal to or greater than those observed for billets clad in mild steel and extruded at  $2000^{\circ}\text{F}$ . This anomalous behavior is apparently the result of a decrease in lubricity associated with the glassing practice used since the yield strength of B-66 decreases by a factor of four between  $2000^{\circ}\text{F}$  and  $3000^{\circ}\text{F}$ .

- REF 1, 2 EXTRUSION BILLET CANNED IN MILD STEEL
- REF 3 EXTRUSION BILLET GLASS COATED

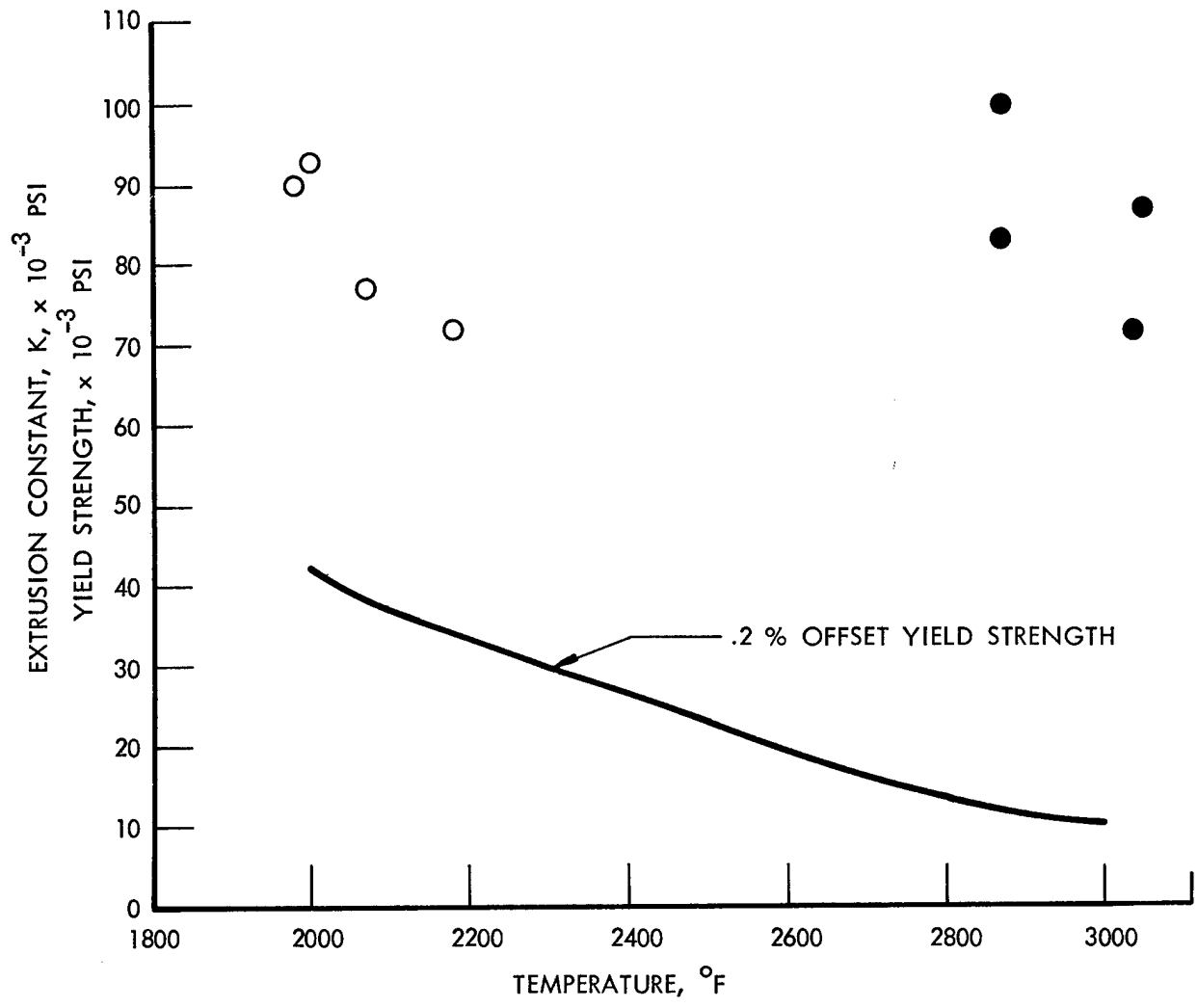


FIGURE 2. Effect of Temperature on the Extrusion Constant and Yield Strength of B-66

Subsequent attempts by DuPont to tube reduce two B-66 tube blanks at room temperature were unsuccessful. <sup>(1)</sup> Prior to tube reducing, the extruded tube blanks were annealed for one hour at 2550-2600°F which gave a recrystallized grain size of ASTM 3-7. <sup>(1)</sup>

Development of sheet processing techniques for B-66 at Westinghouse has indicated the following: <sup>(3)</sup>

- (1) Section sizes in excess of .1" but less than approximately .75" should be rolled in the temperature range of 500-800°F. Within this temperature range, a minimum is exhibited by the flow stress and the rate of work hardening also decreases.
- (2) Satisfactory rolling characteristics are exhibited by sheet bars which have been either recrystallized for one hour at approximately 1500°C (2730°F) or stress relieved at about 1150°C (2100°F).
- (3) A series of commercially available glasses designed to prevent contamination during high temperature working operations was found to be corrosive above 1200°C. <sup>(4)</sup>

Data from the sheet processing development were used as a guide for selecting extrusion and tube drawing schedules.

#### IV. PROGRAM DISCUSSION

##### (A) Starting Material

The surface quality of the as-extruded tube blank will be a function of the extrusion practice used and the grain size of the starting extrusion billet. The detrimental effects of coarse grain size on the surface quality of fabricated parts is well known, and applies equally to shapes formed by extrusion. <sup>(5)</sup> Thus, extrusion billets will be in the wrought and recrystallized conditions to ensure a fine starting grain size.

Starting billets are being produced from 8" diameter vacuum arc melted ingots by a combination of extrusion and swage forging. Final working will be done generally in the temperature range of 1000-1200°C (1800-2200°F). Prior to extrusion, the billets will be given the proper thermal treatment to produce as fine as possible grain size. The starting tube blank extrusion billets will have been reduced approximately 72 per cent from the cast ingot.

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[The solid billets for the extrusion evaluation will have been worked approximately 88 per cent from the ingot and will be heat treated to produce a microstructure similar to that expected in the tube blank extrusion billets.] Approximately 166 pounds of T-111 and 83 pounds of B-66 starting tube blank extrusion billets will be processed.

#### (B) Tube Blank Extrusion

B-66 and T-111 rounds and sheet bar shapes have been produced by extrusion. Reduction ratios have generally been less than 4:1, and the extrusion was for primary breakdown of as-cast ingots using high temperature glassing practice.

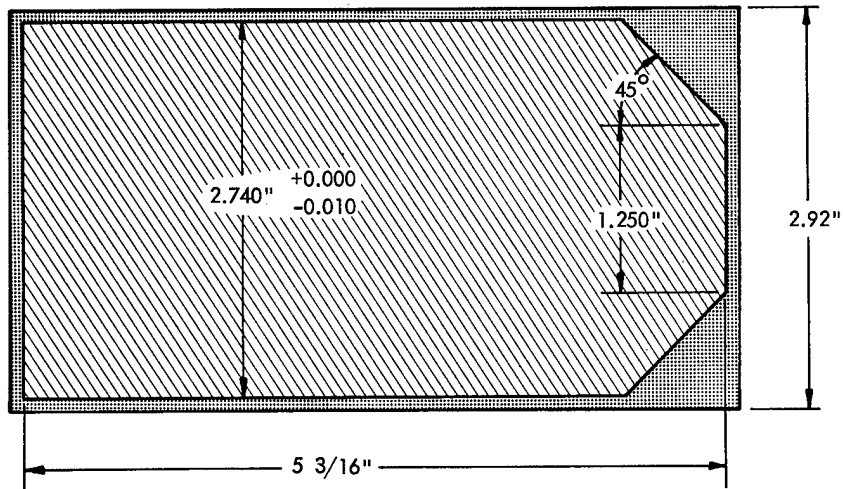
[Extrusion of B-66 and T-111 tube blanks] will require some development effort and thus [will be accomplished in two phases.] In the first phase, solid rounds will be extruded to establish basic extrusion data to permit selection of the parameters for tube blank extrusion. Phase I will provide data on the extrusion characteristics of B-66 and T-111 at relatively high reduction ratios (i. e., 7:1). [The information obtained from Phase I will include extrusion constants, as-extruded surface quality, depth of contamination, die wear, and metallurgical condition of the extruded billet. The results of Phase I will be used to select the parameters for the tube blank extrusions under Phase II.] The limited amount of starting material being processed necessitates that careful consideration be given to the selection of the most favorable conditions for extrusion to obtain maximum yields of usable material. [The billet configurations to be used are shown in Figure 3.]<sup>(5)</sup> The starting Phase II billet configuration is dictated by the finished tube blank size of 2" OD x 1/4" wall and the preset reduction ratio of 7:1. The extrusion billet will be clad in an evacuated mild steel can. The cladding cross-section is approximately 13 per cent of the total cross-sectional area of the billet. The reasons for avoiding the use of high temperature glassing practice have been discussed previously under Section III. The extrusion portion of this program will be carried out on a subcontract at the facilities of Nuclear Metals, Inc., on a 1400-ton high speed extrusion press.

#### (C) Tube Drawing

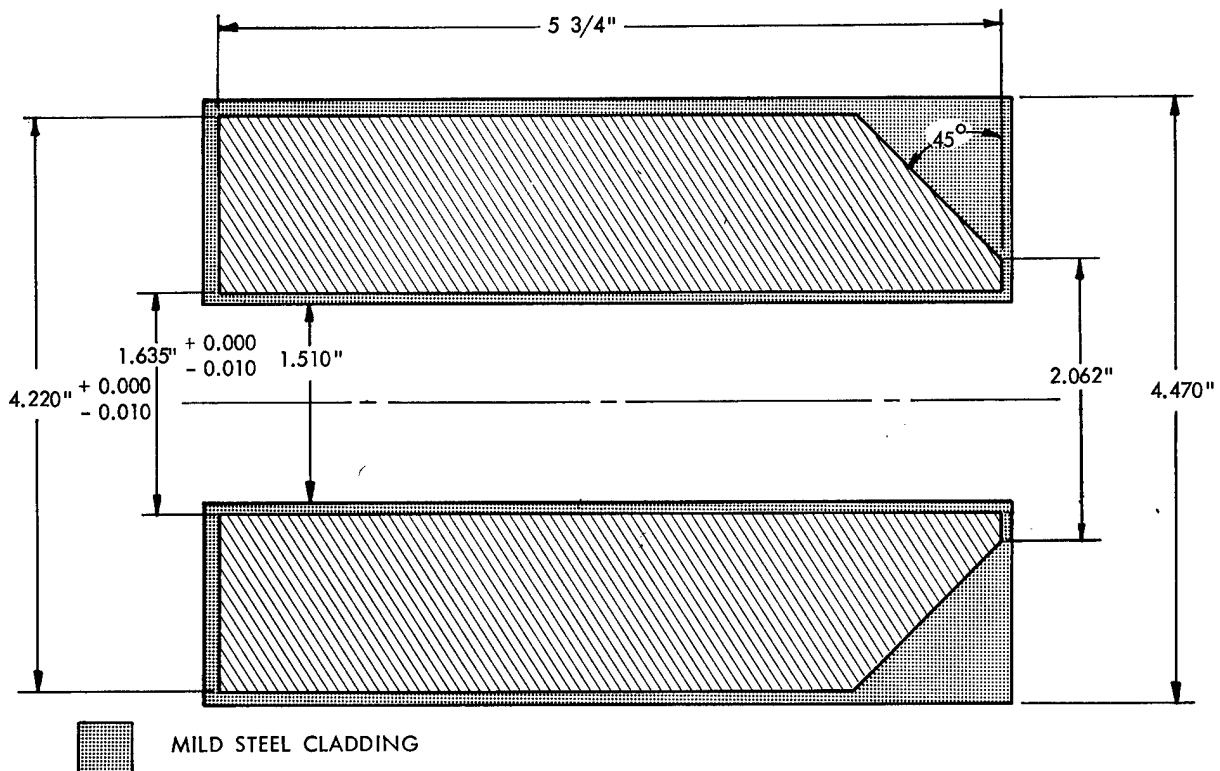
[Processing of the B-66 and T-111 tube blanks to the finished tubing sizes will be done at the facilities of the Superior Tube Company, utilizing essentially conventional]

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PHASE I



PHASE II



561221

FIGURE 3. Extrusion Billet Designs

tubing manufacturing methods. The T-111 will be processed at ambient temperatures. However, B-66 will require warm working during the initial stages of tube reduction. The temperature range for this initial working will be 500-800°F. Final drawing operations will be done at ambient temperatures. The starting tube blanks will be in the optimum metallurgical condition prior to the initial tube reduction.

A warm drawing evaluation of B-66 will be performed using a portion of the Phase I extruded rod. The extruded rod will be conditioned, heat treated, and gun-drilled to give a starting shape of approximately 1-1/8" in diameter x 1/8" wall x 12" long. This rod will be drawn to 1/2" OD x .062" thick wall.

Intermediate in-process thermal treatments will be performed as required. High temperature annealing treatments will be performed in a vacuum annealing furnace located at Westinghouse. This furnace has a temperature capability of 3000°F and a working zone of 2" in diameter and 60 inches long. The operating pressure is less than  $5 \times 10^{-5}$  torr.

#### (D) Inspection

The finished tubing will be 100 per cent inspected using dye penetrant and ultrasonic techniques. Notched standards for internal defects will be tentatively established at 5 and 10 per cent of the wall thickness. Supplementing the non-destructive tests on the finished tubing will be chemical analysis, metallographic examination and room temperature mechanical property measurements.

The inspection schedule outlined in Figure 4 will permit surveillance of the material at all stages of processing.

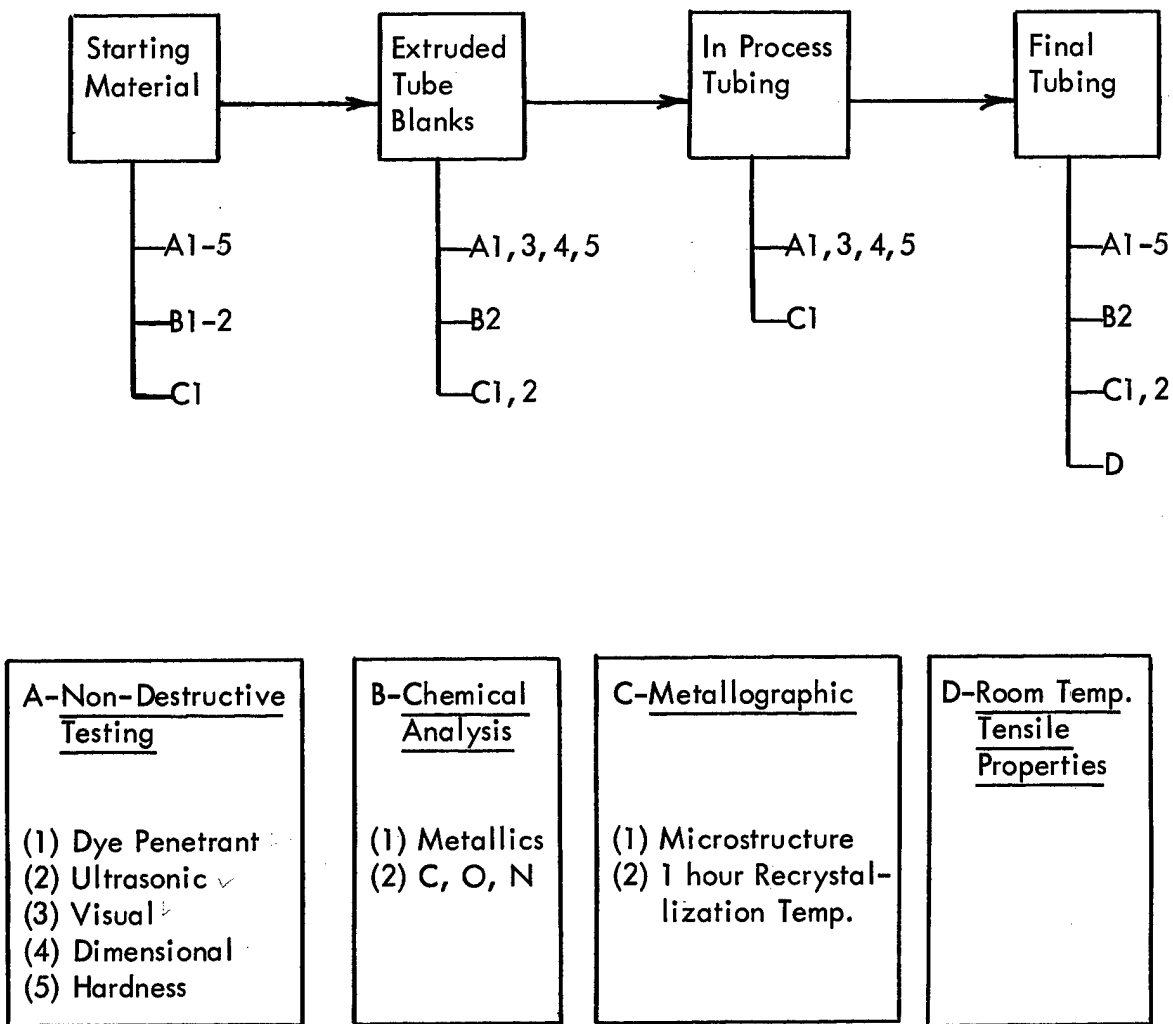


FIGURE 4. Inspection and Testing Schedule

## V. FUTURE ACTIVITIES

During the next quarter, it is planned that the extrusion parameters will be defined and tube blank extrusion will be accomplished. In addition, the hot drawing evaluation of B-66 will be started using a portion of the billet material from the Phase I extrusion evaluation.

## VI. REFERENCES

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