PROCESSING AND EVALUATION OF PRE-PRODUCTION QUANTITIES OF COLUMBIUM ALLOY SHEET

August 20, 1963

Prepared Under U. S. Navy
Bureau of Naval Weapons
Contract N 600(19)-59546

Interim Report No. 2
24 April 1963 through 23 July 1963

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APPROVED: for F. L. Orrell
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Refractory Metal Products

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ABSTRACT

The 0.155-inch B-66 sheet processed in the preceding report period was further processed to final gage, 0.050 inch. The yield of 46.4% from extrusion billet compares favorably with yields for other columbium alloys which are generally less difficult to fabricate. Sheet flatness was excellent and was 3.1%, maximum. Gage uniformity was good, and AMS specifications were generally met; additional work is indicated to produce 1/2 AMS tolerances in the alloy. The FS-85 ingot, 6-3/8-inches in diameter and weighing 303 pounds, was readily forged to 2-5/8-inch thick slab and a high yield of material is expected from this operation.

I. INTRODUCTION

This, the second interim report under Contract N 600(19)-59546, describes the work conducted during the third, fourth, and fifth contract months. During the first reporting period a 6-inch diameter, 250-pound ingot of B-66 (Cb-5Mo-5V-1Zr) was processed to 0.155-inch thick sheet.

Under similar contracts, Westinghouse and Fansteel Metallurgical Corporation are to prepare two ingots of B-66 and two of FS-85 (Cb-27Ta-10W-1Zr), respectively. An ingot of each alloy is to be exchanged between the contractors for rolling to sheet (0.040 - 0.060 inch thick and 18 inches wide) for evaluations of mechanical properties and weldability. The dates of initiation of the two contracts were such that, at the end of the first report period, the only material being processed was the 250-pound B-66 ingot at Westinghouse.

II. PROGRESS DURING REPORT PERIOD

A. B-66

At the end of the first report period, heat DX-602 had been processed to 0.155-inch thick sheet and was in four pieces, each weighing 36 - 38 pounds. The total available weight at this point was 147 pounds, for a yield of 59% from extrusion billet.

Rolling from 0.500 inch down to 0.165 inch had been accomplished from 2150°F, and 0.010 inch was pickled from the sheet thickness to remove contaminated metal. The following data show that hydrogen was maintained at a low level and that oxygen contamination must have been limited to a very shallow surface layer.

Condition	O_2, ppm	H_2, ppm
As-rolled and sand blasted	103	5
0.004" pickled from gauge	108	---
0.010" pickled from gauge	104	---

The analyses also indicate that heating in a dynamic atmosphere of argon is an effective means of preventing contamination and, further, that B-66 will not be particularly contaminated by a short-time exposure to air at 2150°F.

Since all columbium alloys scale freely at the rolling temperatures used in this work, it was expected that loose particles of scale would produce an irregular surface pattern on the as-rolled sheet surfaces. Such surface conditions were actually obtained, and to produce uniform surfaces attempts were made to belt grind the sheets on a Hill-Acme pinch-roll machine. DX-602 B1 was ground using 80-grit silicon carbide cloth-backed belts under a full flow of a water-soluble coolant. Sixty-grit belts were evaluated and discarded since they produced areas on the sheet in which grind lines were objectionably deep. The life of the belts was extremely short; the silicon carbide grains were almost completely rounded off after four passes across the sheet. In addition, the removed metal clogged the belt, further contributing to poor belt efficiency. Approximately 10 mils per side were ground from DX-602 B1, and it was observed that the sheet edges were ground more rapidly than the central portions of the sheet. Consequently, edge-to-center crown was increased from 10 mils at 0.155-inch thick to 20 mils at 0.135-inch thick.

In view of the results of grinding it was decided to cold roll about 50% to 0.080-inch thick to planish the sheet surfaces. Any defects could safely be spot conditioned at this gauge and the final reduction from 0.080 inch to 0.050 inch was judged sufficiently large to remove any traces of spot conditioning. The ends of each sheet were, therefore, heated to 450°F and rolled down to 0.030 inch prior to attaching leaders. The sheets were then heated under quartz lamps to 450°F and rolled under tension on a 8" x 38" x 24" four-high mill. The pass schedules used and the results of this rolling are shown in Table 1.

The resultant surfaces were in very good condition and essentially all of the irregular pattern present in the hot rolled sheets was

TABLE I

First Cold Rolling of B-66

Sheet	Starting Gauge	Pass No.	Mill Reduction	Gauge
DX-602B	0.160	1	70*	-----
		2	15	0.150
		3	--	-----
		4	25	0.120
		5	25	-----
		6	10	0.095
		7	10	0.083
DX-602B1	0.135	1	90*	0.120
		2	15	0.105
		3	15	0.090
		4	10	0.080
DX-602T	0.150	1	85*	-----
		2	20	0.117
		3	20	0.105
		4	10	0.095
		5	10	0.082
DX-602T1	0.165	1	75*	-----
		2	20	0.140
		3	20	-----
		4	20	0.110

* Includes 0.040" for elastic deformation of mill components

Sheet	Remarks
DX-602B	Rolled well. Good surface
DX-602B1	One end cracked and was sheared off. End rolled down for new leader and rolling continued. Good surface.
DX-602T	Rolled well. Good surface
DX-602T1	Slight edge cracking necessitated stopping at 0.110". Good surface.

eliminated. Hand conditioning was limited to one or two small areas on each sheet and DX-602 T1 was trimmed slightly to remove the edge cracks. The sheets were then warmed to 400°F and rolled on the same mill as before. Data for this operation are in Table 2. Surface quality and shape were generally good except for DX-602 B1 which partially cobbled when one end of the sheet fractured. This sheet also contains numerous small mill marks. It is likely that the difficulties encountered during both rollings of DX-602 B1 resulted from the uneven profile developed during belt grinding.

The leaders were then removed from the sheets and the sheet ends sheared to remove the areas where leaders had been welded. Samples of as-rolled material were then wrapped in tantalum foil and vacuum annealed in a laboratory furnace at 100°F intervals. Metallographic observations and hardness tests yielded the following data:

Condition	Hardness, DPH*	Remarks
as rolled	333	
1700°F (1 hour)	283	
1800°F (1 hour)	260	Recrystallization beginning
1900°F (1 hour)	224	80% recrystallized
2000°F (1 hour)	249	90% recrystallized
2100°F (1 hour)	233	100% recrystallized
2200°F (1 hour)	230	
2300°F (1 hour)	221	
2400°F (1 hour)	215	
2500°F (1 hour	224	
2600°F (1 hour)	228	

* Average of five impressions

TABLE II

Second Cold Rolling of B-66

Sheet	Starting Gauge	Pass No.	Mill Reduction	Gauge
DX-602T1	0.110	1	0.060*	0.110
		2	0.020	0.105
		3	0.010	0.095
		4	0.010	0.083
		5	0.005	0.070
		6	0.005	0.065
		7	0.010	0.057
		8	0.005	0.052
DX-602T	0.080	1	0.100*	0.068
		2	0.013	0.061
		3	0.000	0.054
		4	0.000	0.052
		5	0.000	0.048
DX-602B	0.083	1	0.100*	0.075
		2	0.005	0.070
		3	0.000	0.063
		4	0.000	0.063
		5	0.005	0.054
		6	0.003	0.052
DX-602B1	0.080	1	0.100*	0.073
		2	0.005	0.065
		3	0.005	0.063
		4	0.000	0.057
		5	0.000	0.056
		6	0.000	0.053
		7	0.002	0.051

* Includes 0.030" for elastic deformation of mill components

Remarks

All sheets rolled well except DX-602B1 on which an end broke and sheet partially cobbled. DX-602B1 was sheared and new leader attached; resulting sheet is not as flat as others and contains mill marks.

The sheet had been rolled 90% below its recrystallization temperature, from 0.500 inch to 0.050 inch, which explains the relatively low recrystallization temperature of the final product. The sample annealed at 1900°F has low hardness and is recrystallized to an abnormally high degree. A malfunction of the laboratory furnace is suspected and a repeat sample has been scheduled.

Each sheet was degreased and vacuum annealed in a cold wall furnace at less than 0.1 micron pressure. Sheets T and B1 were stress relieved for one hour at 1800°F while sheets T1 and B were recrystallized at 2500°F.

The sheets were then roller-leveled, trimmed to the best size of usable material, inspected, and test cut according to a prearranged plan. Final yields were as follows:

Sheet	Total Length, in.	Weight, lbs.
T	120-1/4	32
T1	90-1/4	28
B1	84-1/4	27
B	91-1/2	29
	Total usable weight	116
	Yield from extrusion billet	46.4%

With the exception of sheet T, virtually all the material was 18 inches wide or wider (maximum width was 20 inches). The length of 18-inch or wider material for sheet T was 75 inches, and this was the expected consequence of a crack which developed during one of the forging operations. The final yield of 46.4% compares favorably with the anticipated yield of 49%.

Gauge variation and flatness are recorded in the table below.

Sheet	Flatness, %	Gage at Sides, in.	Gage at Crown, in.
T	3.1	0.047* - 0.051	0.048** - 0.051
T1	2.6	0.050 - 0.053	0.053 - 0.055
B1	3.0	0.050 - 0.053	0.052 - 0.058
B	2.6	0.049 - 0.051	0.052 - 0.055

* 0.046" at narrow (16-3/4") end

** 0.047" at narrow (16-3/4") end

Flatness was very good for all sheets and 3% flatness was almost universally met. Sheet B1, which partially cobbled during cold rolling, had a wavy shape after levelling, but no area had worse than 3% flatness. The AMS 2242 gage tolerance of ± 0.005 was met except for one corner on sheet B, the narrow end of sheet T, and in the crown of sheet B1 which had 0.020-inch crown after belt grinding.

Test coupons were cut from the sheets for the evaluation program outlined in the first quarterly report. The locations of the samples for each sheet are shown in Figures 1 - 3. The evaluations are in progress and the results will be presented and discussed in the final report under this contract. Test samples and procedures conform to those recommended in Materials Advisory Report MAB-192-M.

B. FS-85

A 303-pound, 6-3/8-inch diameter, 25-inch long, ingot was received from Fansteel Metallurgical Corporation. The ingot was reported to be ultrasonically sound and to have been turned to a surface finish of 500 rms. The chemical analysis reported by Fansteel is shown in Table III. The ingot chemistry is well within specification limits. Check analyses will be run on samples from the forged billets, in a manner similar to that reported for the extruded billets of B-66 in the first quarterly report.

The ingot was cut into two 150-pound forging billets which were subsequently heated for 25 minutes in argon to 2420°F (1325°C) and side forged on flat dies to the sizes indicated below. This was accomplished without reheating.

Bar	Forged Size, Thickness x Width x Length, in.
T	2-7/8 x 8 x 17-1/4
B	2-3/8 x 10 x 16-1/4

The B billet was forged first, and some edge checking occurred during the latter stages when the piece was being spread out to 10 inches. Consequently, it was decided to stop forging on the T billet at a greater thickness, and then condition and reforge both T and B to 1-1/2 inches thick prior to annealing. In general, the forging of FS-85 was easily accomplished; in fact it appears likely that lower forging temperatures can be used without impairing the fabricability of the alloy.

TABLE III

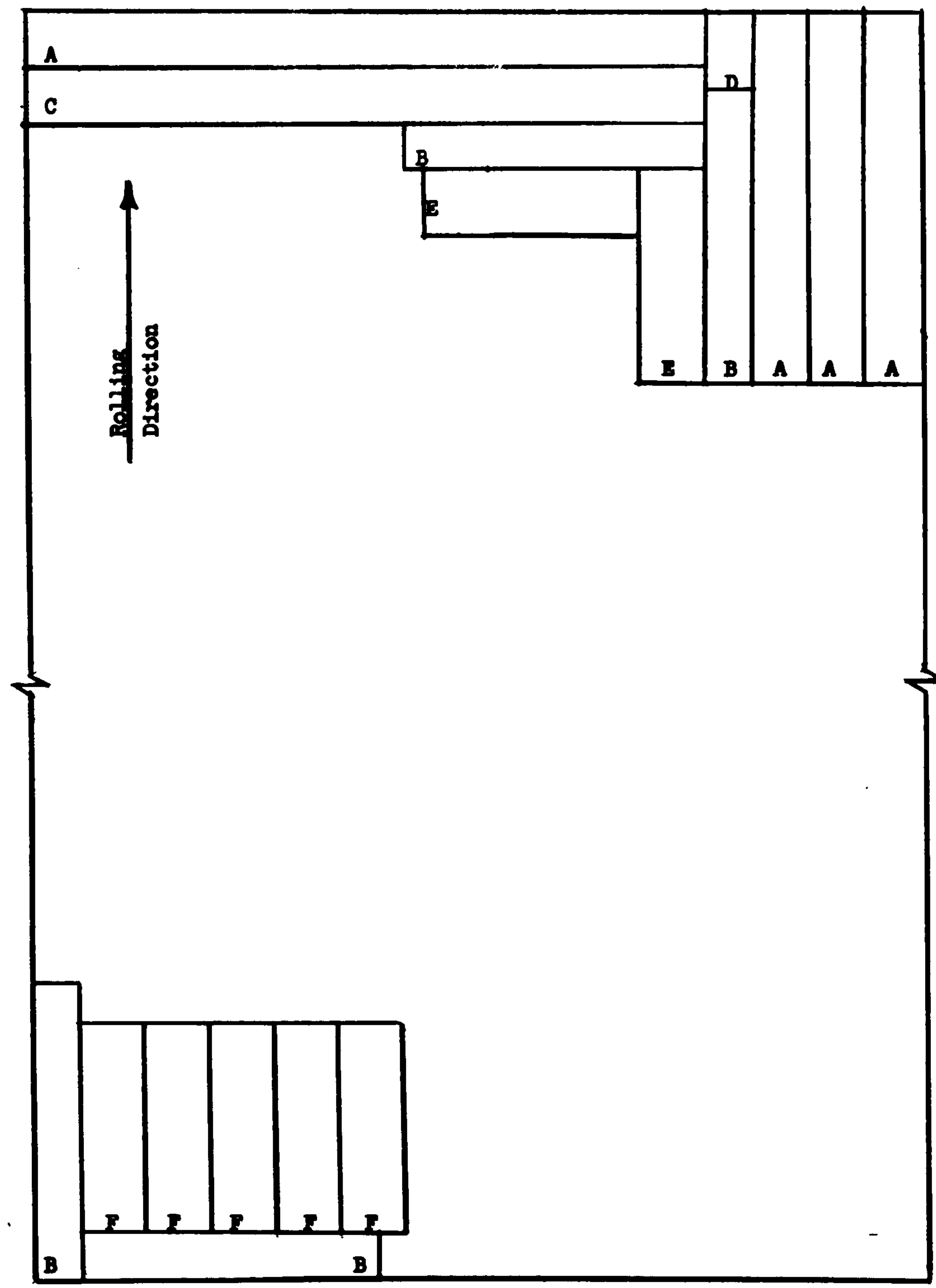
Chemical Analysis for Heat 85D738 (FS-85)

Location	Ta, %	W, %	Zr, %	C, ppm	O_2, ppm	N_2, ppm
Specification	26 - 29	10 - 12	0.6 - 1.1	100 max.	300 max.	150 max.
Top	28.20	10.37	0.91	70	20	46
Bottom	28.16	10.45	0.92	70	90	60

III. FUTURE WORK

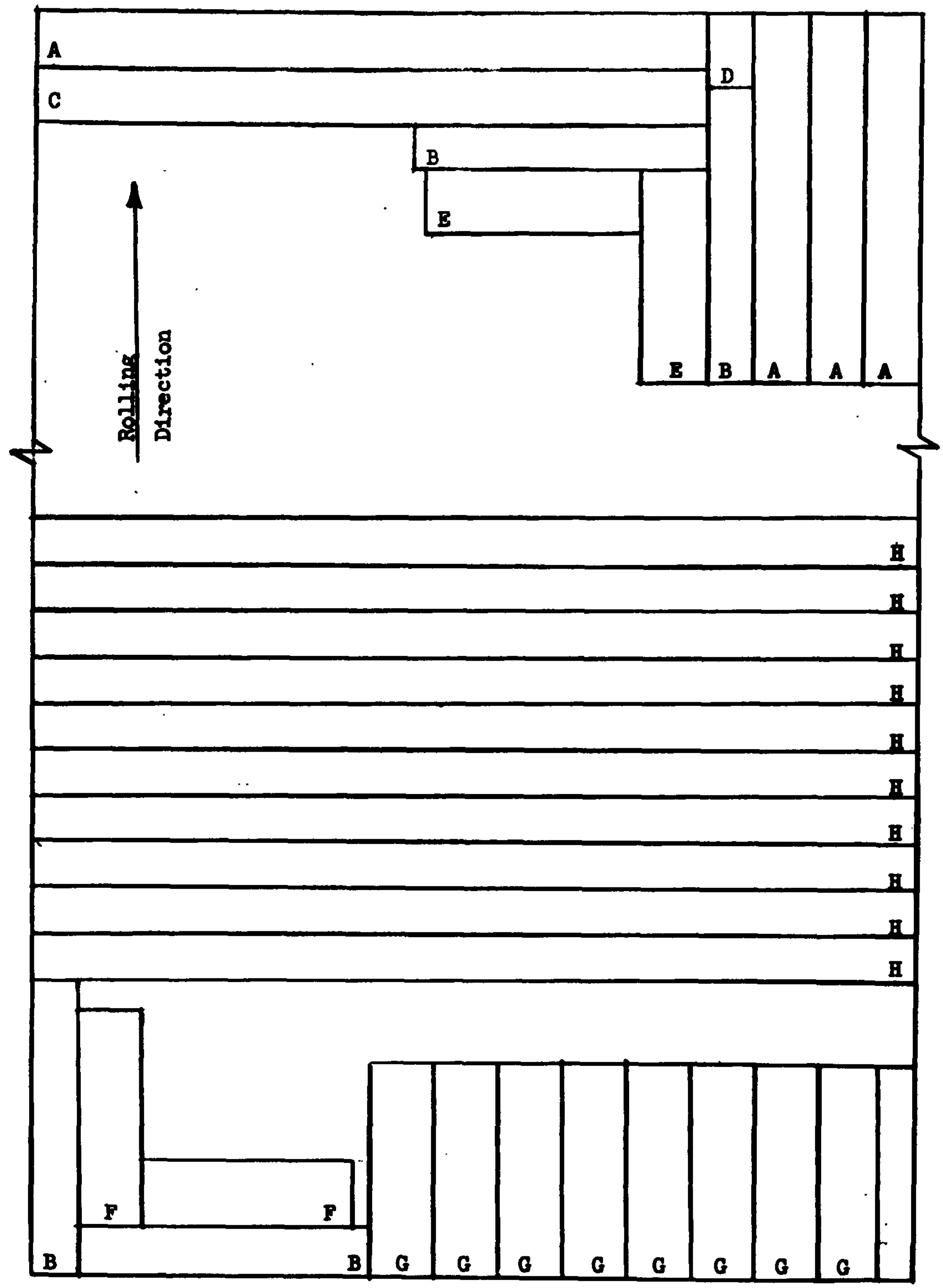
As processing for the B-66 alloy has been completed, the remaining work consists of the mechanical property and welding evaluations listed in the first quarterly report. This work is in progress and results should be available shortly.

The FS-85 material is being conditioned and will be reforged to 1-1/2 inches thick prior to recrystallization. Final processing and evaluation of the sheet material will follow the outline presented in the first quarterly report.



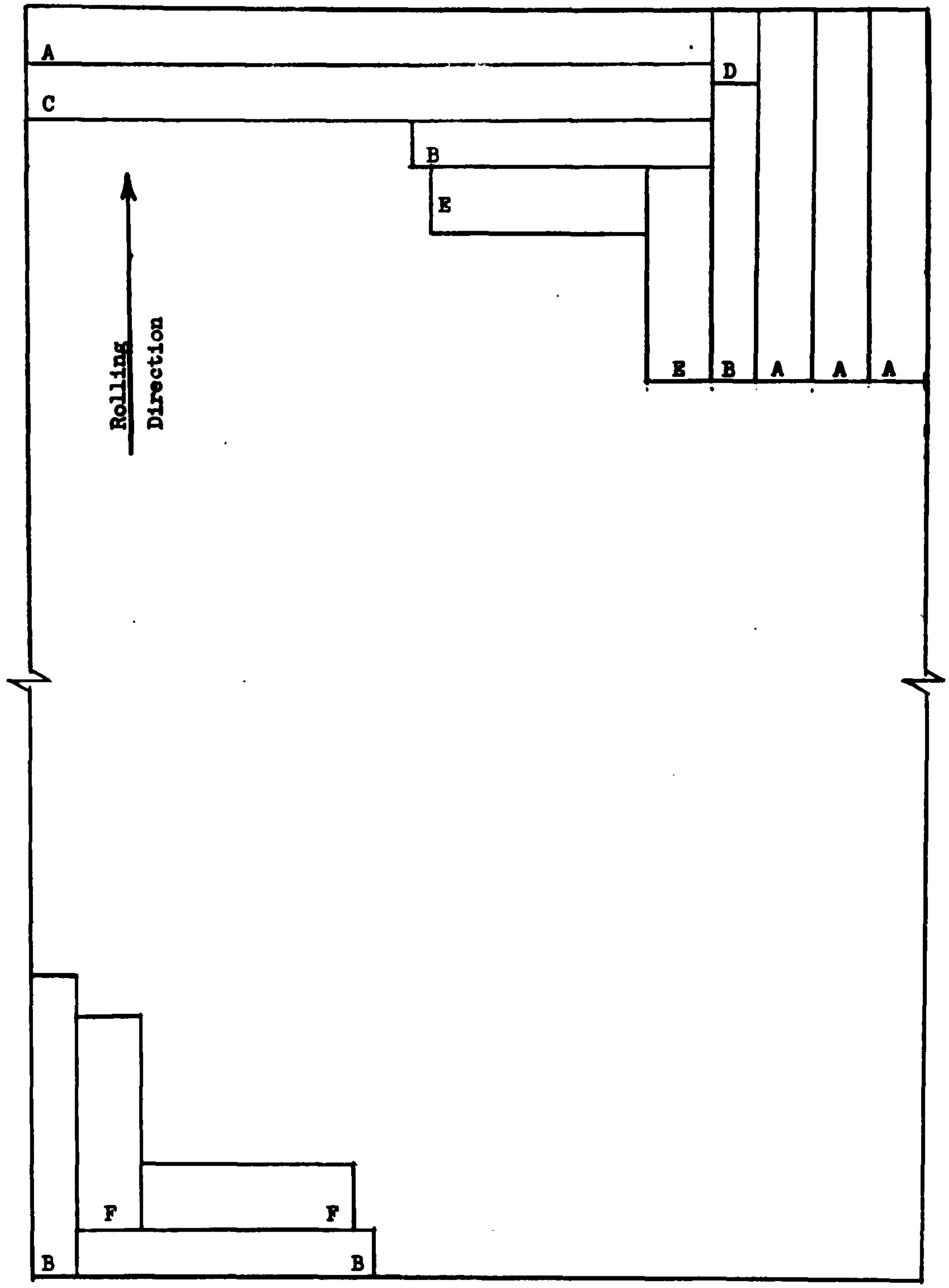
A - Bend
B - Room Temp. Tensile
C - Bend Transition
D - Chemistry
E - 2000°F Tensile
F - Stress Rupture

Figure 1 - Locating of Test Coupons for DX-602 T and DX-602 B1 (stress rupture sheets)



A - Bend
B - Room Temp. Tensile
C - Bend Transition
D - Chemistry
E - 2000°F Tensile
F - 2400°F Tensile
G - Stress Rupture
H - Welding

Figure 2 - Location of Test Coupons for DX-602 Tl (recrystallized)



A - Bend
B - Room Temp. Tensile
C - Bend Transition
D - Chemistry
E - 2000°F Tensile
F - 2400°F Tensile

Figure 3 - Location of Test Coupons for DX-602 B (recrystallized)

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