

UNCLASSIFIED

AD 404 727

DEFENSE DOCUMENTATION CENTER

FOR

SCIENTIFIC AND TECHNICAL INFORMATION


CAMERON STATION, ALEXANDRIA, VIRGINIA



UNCLASSIFIED

NOTICE: When government or other drawings, specifications or other data are used for any purpose other than in connection with a definitely related government procurement operation, the U. S. Government thereby incurs no responsibility, nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use or sell any patented invention that may in any way be related thereto.

63-3-5

AD NO. 404727
ASTIA  COPY

CATALOGED BY ASTIA
AS AD NO. _____

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

May 3, 1963

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

Interim Report No. 1
24 January 1963 through 23 April 1963

Westinghouse Electric Corporation
Materials Manufacturing Division

404727

DDC
RECEIVED
MAY 27 1963
ASTIA D

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

May 3, 1963

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

Interim Report No. 1
24 January 1963 through 23 April 1963

Westinghouse Electric Corporation
Materials Manufacturing Division
Blairsville, Pennsylvania

APPROVED:


F. L. Orrell
Manager
Refractory Metals

ABSTRACT

A 250-pound, 6-inch diameter, B-66 ingot (Cb-5Mo-5V-1Zr) was processed to 0.165-inch thick sheet by means of extrusion, forging, and rolling. Data for these and intermediate operations are given and plans are outlined for future work on the B-66 and FS-85 (Cb-27Ta-10W-1Zr) alloys.

TABLE OF CONTENTS

	<u>Page No.</u>
I. INTRODUCTION	1
II. PROGRESS DURING REPORT PERIOD	
A. Material	2
B. Extrusion	2
C. Forging	11
D. Warm rolling	13
III. FUTURE WORK	15
IV. REFERENCE	18

I. INTRODUCTION

The purpose of this project is to produce and evaluate 0.040 to 0.060-inch thick, 18-inch wide sheet of B-66 (Cb-5Mo-5V-1Zr) and FS-85 (Cb-27Ta-10W-1Zr) from single 6-inch diameter, 300-pound ingots. Under similar contracts, Westinghouse is preparing two B-66 ingots and Fansteel Metallurgical Corporation is preparing two ingots of FS-85. An ingot of each composition is to be exchanged between the two contractors for processing and comparative evaluations of mechanical properties and weldability. The over-all objective of the contracts is to provide data which will permit the selection of an alloy for future development of 36-inch by 96-inch sheet and production of significant production quantities of this sheet. It should be emphasized that process development is not specifically involved in the current contracts and that the contractors are producing sheet by the best practices known at this time.

II. PROGRESS DURING REPORT PERIOD

A. Material

A nominal 300-pound, 6-inch diameter ingot of B-66 was prepared by double electron-beam melting a columbium-molybdenum-zirconium alloy and subsequent double vacuum-arc melting to incorporate the vanadium (which would vaporize in electron-beam melting) and to improve homogeneity. At the time the ingot was melted, development work at Westinghouse was directed toward improving the forgeability of as-cast B-66, which is somewhat marginal. While the results of this work were promising, the forging process has not been developed to the stage where acceptable yields are obtained. Therefore, it was decided to machine the ingots to 5-7/8 inches in diameter and extrude to sheet bar in two pushes. The total extrusion billet weight was, therefore, 251 pounds. No ultrasonic or penetrant indications were observed in the conditioned ingot.

Analyses of the 5-7/8-inch diameter ingot and the extrusions are presented in Table I. With the exception of one slightly low value of zirconium, all analyses for metallics and interstitials were within specification limits. The differences in oxygen and nitrogen reported for the ingot and extrusions can be traced to differences in analytical techniques. For the ingot, oxygen was determined conductometrically and nitrogen was determined by the Kjeldahl method, while vacuum fusion techniques were used to determine both elements in the extrusions.

B. Extrusion

The ingot was spot conditioned and cut into two billets which are shown in Figure 1. Both billets were extruded on the 2750-ton horizontal

TABLE I

Chemical Analysis for Heat DX-602

<u>Location</u>	<u>Mo, %</u>	<u>V, %</u>	<u>Zr, %</u>	<u>C, ppm</u>	<u>O₂, ppm</u>	<u>N₂, ppm</u>
Specification	4.5-5.5	4.5-5.5	0.85-1.3	200 max.	300 max.	200 max.
Top, mid-radius	4.6	5.4	0.86	28	<10	90
Top, mid-radius*	4.60	4.50	0.90	70	128	68
Top, center	4.7	5.3	0.86	--	---	--
Middle, sidewall	4.7	4.6	0.91	--	---	--
Middle, mid-radius*	4.60	5.36	0.82	20	114	74
Bottom, mid-radius	4.6	4.5	0.95	28	48	95
Bottom, mid-radius*	4.50	4.96	0.85	20	137	60
Bottom, center	4.7	4.5	0.96	--	---	--

* Analyses on extrusions; all others on as-cast ingot

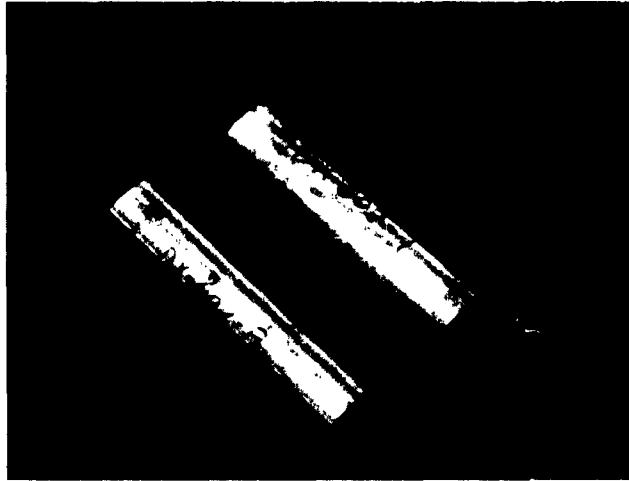


FIGURE 1 - B-66 Extrusion Billets

press at the DuPont Metals Center, Baltimore, Maryland. The equipment and extrusion techniques have been adequately described.⁽¹⁾ In this work, steel nose blocks with a 45° chamfer were used ahead of the billets to reduce the amount of nose burst in the extrusions. The extrusion data are given in Table II. As shown in Figure 2, the extrusion representing the top half of the ingot (IX-602T) developed deep defects near the leading end. Since earlier work had shown that the best surfaces were obtained at low extrusion rates, the temperature for the bottom billet was lowered and the press throttle valve was closed as shown in Table II. By so doing, the ram speed was lowered from 14 to 7 inches per second and the severity of the defects in the second extrusion was reduced.

The extrusions were sand blasted, conditioned by grinding, and pickled. The nose and tail ends were cropped and both extrusions were cut into two bars which were identified as shown in Figure 3. The four sheet bars were vacuum annealed at 2910°F (1600°C) for one hour under a pressure of 0.1 micron. Samples from the cropped ends were annealed along with the slabs, and representative macrostructures and microstructures are shown in Figures 4 and 5, respectively. The nature of the second phase evident in the recrystallized microstructures is not known at this time.

After annealing, the bars were inspected ultrasonically and by fluorescent penetrant techniques. Several additional small surface defects were detected and removed by grinding. There were ultrasonic indications of a small internal defect four inches from one end of the Ti bar, but since this had not been observed previously and there were large variations in penetration of the ultrasonic signal, the "defect" was not removed. No difficulties in later

TABLE II

Extrusion Data for IX-602

	<u>Top Billet</u>	<u>Bottom Billet</u>
Billet size	5-7/8" dia. x 15-1/4"	5-7/8" dia. x 15-1/2"
Die opening	2.3" x 4.6"	2.3" x 4.2"
Reduction ratio	2.56:1	2.81:1
Nose block	Steel @ 2200°F	Steel @ 2200°F
Follower blocks	2 carbon @ 2200°F + 1 steel (cold)	2 carbon @ 2200°F + 1 steel (cold)
Glass	Proprietary	Proprietary
Transfer time	31 sec.	30 sec.
Throttle valve	35% of full open position	30% of full open position
Ram speed	14 in./sec.	7 in./sec.
Break-through pressure	98,000 psi	98,000 psi
Avg. running pressure	84,000 psi	91,000 psi
Extrusion constant, K	85,100 psi	84,800 psi
Temperature *		
Top	2810°F	2750°F
Middle	2725°F	2640°F
Bottom	2700°F	2640°F
Die	Shear, zirconia-coated	Shear, zirconia-coated
Heating atmosphere	Argon	Argon
Length of usable extruded metal	35-1/4"	40"

* Optically read and corrected for emissivity



FIGURE 2 - B-66 extrusions from the top and bottom halves of DX-602. Both the top and bottom surfaces are shown for each extrusion, and the leading (or nose) ends of the extrusions are at the left.

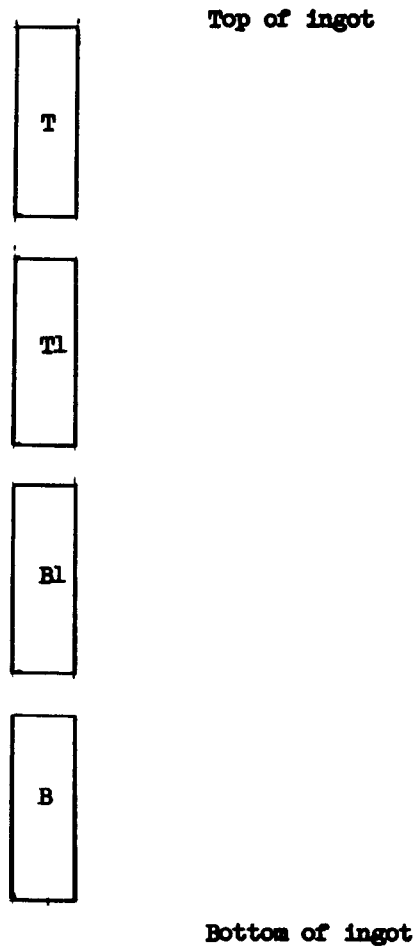
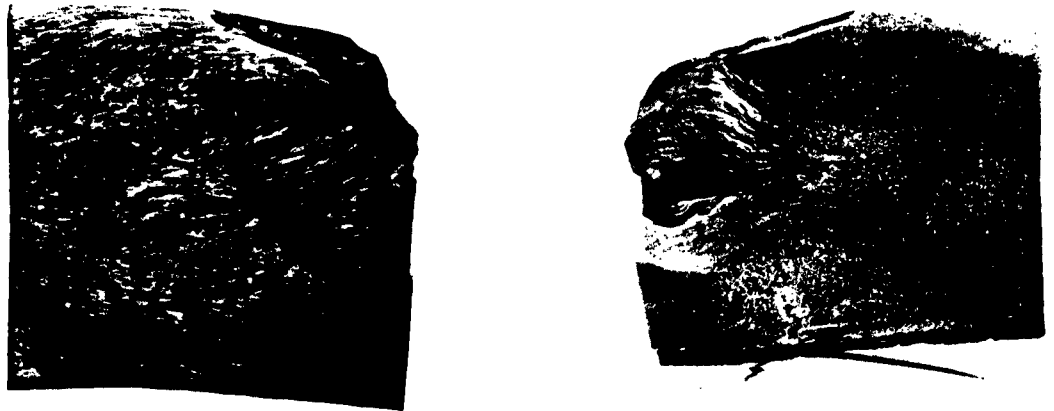


Figure 3 - Identification of DX-602 bars



Nose

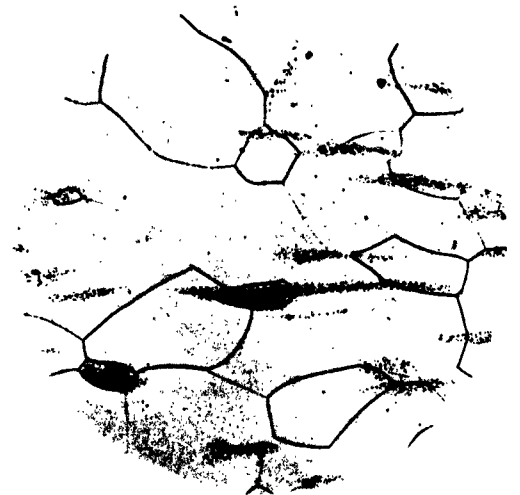


Tail

FIGURE 4 - Macrostructures of B-66 extrusions before (left) and after recrystallization at 2910°F (1600°C). 1X



Nose



Tail

FIGURE 5 - Microstructure of B-66 extrusions before (left) and after recrystallization at 2910°F (1600°C). 100X

processing could be attributed to this ultrasonic indication and re-inspection of the piece, after forging to one-inch thick, failed to reveal this "defect".

The yields after extrusion and conditioning were as follows:

<u>Bar</u>	<u>Conditioned Thickness x Width x Length, in.</u>	<u>Conditioned Weight, lbs.</u>
T	2-1/8 x 4-5/8 x 17	51
TI	2-1/8 x 4-1/2 x 17-7/8	50
BI	2-1/16 x 4-1/8 x 19-1/4	51
B	2-1/16 x 4-1/8 x 19-7/8	<u>48</u>
	Total conditioned weight	200
	Yield from conditioned ingot	80%

C. Forging

The bars were heated to 2350°F (1285°C) under a positive pressure of argon and cross-forged (without appreciable lengthening) to about one-inch thick on an 18,000-pound-capacity hammer. Heating time was 30 minutes and forging was accomplished in one heating except for the B bar, which required one reheat. Two small cracks developed directly opposite each other in the ends of the T bar. All bars were conditioned and vacuum annealed at 2730°F (1500°C). During the anneal, stress-relief of the material delineated a minor edge crack in the BI bar. Ultrasonic inspection showed that the defects in the BI and T bars did not extend deeply; consequently, these areas were not cut from the bars at this time. Yields after forging and conditioning were as follows:

<u>Bar</u>	<u>Conditioned Weight, lbs.</u>
T	46
TL	46
BL	47
B	<u>44</u>
Total conditioned weight	183
Yield from conditioned ingot	73%

All pieces were heated in argon to 2280°F (1250°C) and re-forged in the same direction to approximately 0.800 inch on the same hammer used for initial forging. Heating time was 20 minutes and the TL and B bars required one reheating. The crack on one end of the T bar propagated slightly necessitating the cutting of a small equilateral triangle from one corner of the piece; therefore, portions of the resultant sheet from this bar will be slightly under 18 inches wide. Following are the yield after the second forging and conditioning operations.

<u>Bar</u>	<u>Conditioned Thickness x Width x Length, in.</u>	<u>Conditioned Weight, lbs.</u>
T	0.800 x 8-3/4 - 10-1/2 x 18-1/4	43
TL	0.790 x 7-3/4 - 9-7/8 x 20-1/8	43
BL	0.825 x 7 - 9-1/8 x 20-1/2	44
B	0.760 x 7 - 9-1/4 x 21-3/4	<u>42</u>
Total conditioned weight		172
Yield from conditioned ingot		68.5%

D. Warm Rolling

Mixed success was obtained in early rolling of forged B-66 on Westinghouse's 15" x 24" mill when the slabs were heated to 500-600^oF. Most recently, it has been determined that success, at least with the given rolling mill, depends upon starting with a slab of uniform thickness; if the starting piece is non-uniform, complex stresses are imposed upon the material which apparently cannot adjust to these stresses at this stage of its fabrication.

As a result of the early experiences, rolling of the contract material was performed at more elevated temperatures to eliminate the cracking which sometimes occurred at lower temperatures. This procedure has several disadvantages. First, the depth of contamination is greater at the higher temperature and, secondly, there is a tendency to roll the loose scale (which forms only at the higher rolling temperatures) into the surface of the sheet, thus resulting in a non-uniform sheet thickness. Nevertheless, the rolling performed under this project at 2100-2200^oF can be classified as cold working since it was done below the recrystallization temperature of the B-66 alloy.

Rolling was conducted in two stages on the 15" x 24" mill, which is backed up by two 32-inch diameter rolls. In the first operation, the pieces were heated for 15 minutes to 2200^oF (1200^oC) under a positive pressure of argon and rolled in 8 - 10 passes to about 0.500-inch thick. The pieces were rolled transverse to the original ingot and extrusion directions, and lengths after this rolling ranged from 13-3/4 to 17 inches. Yields after conditioning and pickling were as follows:

<u>Plate</u>	<u>Conditioned Weight, lbs.</u>
T	39
TL	40
BL	41
B	<u>39</u>
Total conditioned weight	159
Yield from conditioned ingot	63.5%

At this stage, the pieces had received 50% cold work, i.e., they were reduced from one inch to one-half inch thick after the second anneal. Consequently, the plates were given a third vacuum anneal at 2500°F for one hour. This will be the last in-process anneal for the B-66 alloy, which will have about 90% cold work at final gauge (0.050").

For the second rolling, the plates were heated in argon for 10 minutes to 2150°F (1175°C) and rolled (in the same direction as previously) to 0.165 inch thick in eight passes. The rolled plates contain surface defects from rolled-in scale, and these will be removed prior to and/or during the cold rolling sequence of operations. Each piece was sand blasted and pickled to remove 0.010" from the thickness of each piece.

Yields at this stage are as follows:

<u>Plate</u>	<u>Thickness x Length, in.</u>	<u>Length of Full 18" Width, in.</u>	<u>Wt., lbs.</u>	<u>Anticipated Length of 18" Wide Sheet at .050", in.</u>
T	.150 x 46-3/8	25	36	75
TL	.155 x 40	33-1/2	37	100
BL	.153 x 42	34	38	102
B	.155 x 35-1/2	32	<u>36</u>	96
Total available weight			147	
Yield from conditioned ingot			59%	

III. FUTURE WORK

The 0.150-inch thick B-66 plates will be cold rolled to 0.050-inch thick strip on the 24-inch wide, 4-inch high cold Bliss mill. All rolling will be done under tension after the attachment of leader material to the ends of each piece. Half of the material will be stress relieved under vacuum at 2000^oF and the balance will be recrystallized at 2500^oF. The sheets will then be roller-leveled, inspected, trimmed, and samples taken for the following test program:

- A. Room temperature tensile tests: longitudinal and transverse tests on each sheet.
- B. Room temperature bend tests: determine minimum bend radius for longitudinal and transverse samples from each end of each sheet.
- C. Elevated temperature tensile tests: duplicate longitudinal and transverse tests for each heat-treated condition at 2000 and 2400^oF.
- D. Welding evaluations: duplicate tensile and bend tests both parallel and transverse to the direction of welding in the as-welded, stress-relieved and recrystallized conditions.
- E. Transition temperature: determine ⁴T transition temperature for base metal (transverse to rolling direction) and as-welded metal (transverse to welding direction).
- F. Recrystallization temperature: determine 1-hour recrystallization temperature for as-rolled sheet.
- G. Stress rupture tests: determine 1- and 10-hour life at 2000 and 2400^oF for both heat-treated conditions.
- H. Chemical analysis: determine carbon, oxygen and nitrogen for each sheet.

Test procedures will conform to those recommended in Materials Advisory Board Report MAB-176-M.

Representatives of Westinghouse and Fansteel Metallurgical Corporation met at the latter's facilities to discuss processing and evaluation of the B-66 and FS-85 alloys. On the basis of Fansteel recommendations, the following procedure will be used for the 6-inch diameter FS-85 ingot.

1. Side forge at 2300-2400°F (1250-1315°C) to about 1-1/2-inch thick.
2. Inspect ultrasonically.
3. Condition, pickle, crop ends, and cut into 4 equal lengths (about 17-1/2 inches).
4. Vacuum anneal 1 hour at 2550°F (1400°C).
5. Inspect with fluorescent penetrant.
6. Cross-forged (spread width) and reduce thickness to 3/4 inch. Bars will lengthen to about 18-1/2 inches.
7. Inspect ultrasonically.
8. Condition and pickle.
9. Vacuum anneal 1 hour at 2550°F (1400°C).
10. Inspect with fluorescent penetrant.
11. Heat to 500-600°F and roll to 1/2 inch thick.
12. Condition and pickle.
13. Heat to 500-600°F and roll to 0.100 inch thick.
14. Vacuum anneal 1 hour at 2550°F.
15. Cold roll to 0.050 inch.
16. Stress relieve half of material at 2000°F, recrystallize balance at 2550°F.

17. Roller-level, inspect, trim and cut test coupons.
18. Evaluate as for B-66, paragraphs A - H, above.

The process recommended by Westinghouse for B-66 was as follows:

1. Cut into 2 billets, 5-7/8-inches in diameter.
2. Extrude from 2700-2800°F at a ratio of 2.5 - 3:1 using Sejournet lubrication practices.
3. Condition, pickle, crop ends and cut each extrusion into two sheet bars.
4. Vacuum anneal 1 hour at 2910°F (1600°C).
5. Inspect ultrasonically and with fluorescent penetrant.
6. Heat in argon to 2350°F (1285°C) and cross forge to 1-inch thick.
7. Condition, pickle and inspect with fluorescent penetrant.
8. Vacuum anneal 1 hour at 2730°F (1500°C).
9. Heat in argon to 2280°F (1250°C) and cross forge in same direction as previously to about 0.800 inch thick.
10. Condition, pickle and inspect with fluorescent penetrant.
11. Warm roll at 500°F (250-300°C) or, preferably, at 1800-2000°F (1000-1100°C) to 0.500-inch thick plate.
12. Condition and pickle
13. Vacuum anneal 1 hour at 2500°F (1370°C).
14. Warm roll at 500°F or 1800-2000°F to 0.150-inch thick.
15. Condition and pickle.
16. Warm to 500°F and roll on cold mill to about 0.100-inch thick.
17. Condition surface as required.
18. Warm to 300°F and roll to final gauge.
19. For recrystallized material, vacuum anneal 1 hour at 2500°F; for stress-relieved material, vacuum anneal 1 hour at 2000°F.

20. Roller level, inspect, trim and cut test coupons.

21. Evaluate.

Fansteel personnel indicated that they would follow Westinghouse recommendations for the B-66 alloy with the following exceptions:

1. Final gauge would be 0.060 inch instead of 0.050. This should not result in markedly different properties between Fansteel- and Westinghouse-processed sheet.
2. Ingot breakdown would be accomplished by side forging to 1-inch thick. Fansteel was cautioned that this operation was critical and that gross cracking could occur if reduction between anneals exceeded 30-35%.
3. The forged sheet bars would probably be warm rolled at 500^oF and Fansteel was cautioned that higher temperatures may be required, at least for initial rolling operations, to prevent cracking.

The second 6-inch diameter ingot of B-66 for the Fansteel contract has been requisitioned and is expected to arrive at Fansteel's plant by about May 10. Fansteel has indicated that an FS-85 ingot should be delivered to Westinghouse on about May 20; if delivery is achieved by this date, the work on this contract will be extended from the original termination date (June 24) to about October 1, 1963.

IV. REFERENCE

1. L. J. Klinger, J. R. Norton and D. C. Evans, "Research for Consolidation of Refractory Metal Ingots", Interim Engineering Progress Report No. 2 under U. S. Air Force, Aeronautical Systems Division Contract AF 33(657)-8792, covering period of 15 October 1962 - 15 January 1963.

DISTRIBUTION LIST

1. Defense Metals Information Center, Battelle Memorial Institute, 505 King Avenue, Columbus 1, Ohio - Attn: H. R. Ogden
2. Dr. Robert I. Jaffee, Department of Metallurgy, Battelle Memorial Institute, 505 King Avenue, Columbus 1, Ohio
3. Mr. Louis P. Jahnke, Manager, Metallurgical Engineering, Applied Research Operation, Flight Propulsion Laboratory Department, General Electric Company, Cincinnati 15, Ohio
4. Mr. Alan V. Levy, Department Head, Materials Research & Development Dept. (4610), Solid Rocket Plant, Aerojet General Corporation, Sacramento, California
5. Mr. Roger A. Perkins, Research Scientist, Metallurgy and Ceramics (53-36), Lockheed Aircraft Corporation, Missiles and Space Div., Sunnyvale, California
6. Mr. L. M. Raring, Chief, Metallurgical & Chemical Laboratories, Pratt & Whitney Aircraft, Connecticut Aircraft Nuclear Engine Lab., P.O. Box 611, Middletown, Connecticut
7. Dr. William Rostoker, Metals Research Department, Illinois Institute of Technology Research Foundation, Technology Center, 10 West 35 St., Chicago 16, Illinois
8. Dr. L. L. Seigle, Manager, Metallurgical Laboratory, General Telephone and Electronics Labs., Inc., P.O. Box 59, Bayside, New York
9. Mr. John T. Stacy, Senior Group Engineer, Boeing Airplane Company, Aero-Space Division, P.O. Box 3707, Seattle 24, Washington
10. Mr. G. Mervin Ault, Assistant Chief, Materials & Structure Division, Lewis Research Center, National Aeronautics & Space Administration, 21000 Brookpark Road, Cleveland 35, Ohio
11. Mr. George Glenn, Manufacturing Technology Lab., Aeronautical Systems Division, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio
12. Mr. S. V. Arnold, Associate Director, Army Materials Research Agency, Watertown, Massachusetts
13. Mr. J. Simmons, Division of Reactor Development, Atomic Energy Commission, Washington 25, D. C.
14. Mr. I. Perlmutter (ASRCMP-2), Metals & Ceramics Lab., Aeronautical Systems Division, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio

15. Dr. J. R. Lane, Materials Advisory Board, National Academy of Sciences, 2101 Constitution Avenue, N.W., Washington 25, D. C.
16. Dr. A. B. Michael, Fansteel Metallurgical Corp., 2200 Sheridan Road, North Chicago, Illinois
17. Wah Chang Corp., P.O. Box 366, Albany, Oregon
18. Dr. E. M. Mahla, Technical Assistant to the Director of Metal Products, Pigments Dept., E. I. DuPont de Nemours & Co., Wilmington 98, Delaware
19. Union Carbide Stellite Co., Kokomo, Indiana
20. Stauffer Metals Division, Stauffer Chemical Co., 1201 S. 47th Street, Richmond 4, California
21. Climax Molybdenum Co. of Michigan, 14410 Woodrow Wilson, Detroit 38, Michigan
22. Thompson Ramo Wooldridge, Inc., 23555 Euclid Avenue, Cleveland, Ohio
23. Oak Ridge National Laboratory, P.O. Box X, Oak Ridge, Tennessee
24. The Pfaudler Company, Rochester 3, New York
25. Naval Research Laboratory, Washington 25, D. C., Attn: Dr. B. F. Brown
26. McDonnell Aircraft Corp., Box 516, St. Louis 66, Missouri, Attn: Mr. R. Hepper
27. E. I. DuPont de Nemours & Co., Metals Plant, Curtis Bay, Baltimore, Md.
28. Dyna-Soar Project Office, Aeronautical Systems Division, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio
- 29-31. National Aeronautics & Space Administration, 1520 H. Street, Washington 25, D. C. (3 copies)
- 32-43. Armed Services Technical Information Agency, Arlington Hall Station, Arlington, Virginia (for distribution without restriction) (12 copies)
- 44-50. Bureau of Naval Weapons, Department of the Navy, Washington 25, D. C. Attn: DLI-3 (1 copy), RMSP-23 (1 copy), RRMA-23 (remaining copies)