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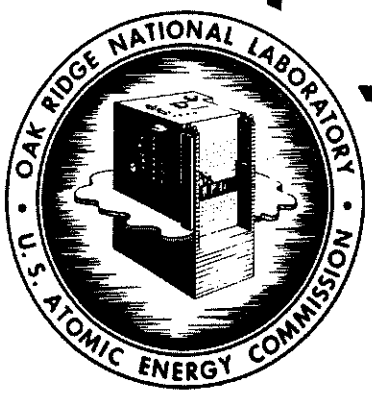
BORON-ALUMINUM AND BORON-URANIUM-ALUMINUM ALLOYS FOR REACTOR APPLICATION

W. C. Thurber
J. A. Milko
R. J. Beaver



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Printed in USA. Price $1.00 cents. Available from the

Office of Technical Services
U. S. Department of Commerce
Washington 25, D. C.

ORNL-2149
Reactors—General
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Contract No. W-7405-eng-26

METALLURGY DIVISION

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DATE ISSUED

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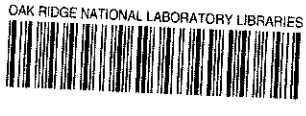

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BORON-ALUMINUM AND BORON-URANIUM-ALUMINUM ALLOYS FOR REACTOR APPLICATION

W. C. Thurber J. A. Milko R. J. Beaver

SUMMARY

Additions of small quantities of the burnable poison, boron, in aluminum reactor fuel elements offer the possibility of reducing undesirable neutron-flux perturbations, especially those caused by fuel burnup. This report describes techniques for induction melting of boron-aluminum and boron-uranium-aluminum alloys for producing materials with maximum homogeneity. Of the several boron additions investigated, a nominal 12 wt % B–Ni master alloy was the most satisfactory for preparing boron-uranium-aluminum castings, while both 1.5 to 5 wt % B–Al and 12 wt % B–Ni master alloys were suitable for preparing boron-aluminum castings. Data are presented which indicate that boron improves the strength of aluminum. The corrosion resistance of a nominal 0.1 wt % B–0.8 wt % Ni–Al alloy is comparable with type 1100 aluminum in distilled and aerated water at both 60 and 100°C. Without the nickel, this alloy appears to be marginal in these environments.

INTRODUCTION

The primary purpose of this investigation was to establish procedures for preparing boron-aluminum and boron-uranium-aluminum alloys for utilization in plate-type aluminum fuel elements.

Although the boron isotope, B^{10}, which is 18.8% abundant in natural boron, has a relatively high neutron-absorption cross section of 4020 barns (*1*), it burns out at a rate which nearly compensates for the burnup of U^{235}. In aluminum reactors this characteristic of boron can be used to reduce undesirable flux perturbations in two ways: (1) by disposition of boron-bearing fuel elements in selected locations in the reactor where the flux is distorted, and (2) by additions of boron to all fuel elements in order to reduce reactivity variations caused by fuel burnout, thus minimizing flux perturbations due to control rods and control-rod movement. For example, in designing the 175-Mw Engineering Test Reactor, it was calculated that by incorporating small quantities of boron in the fuel elements, the change in reactivity due to burnup of the U^{235} atoms could be reduced from 15% to 7% (*2*).

Because of the small quantity of boron required per fuel element, this investigation has been limited to aluminum-boron and aluminum-uranium-boron alloys containing 0.02 to 0.4 wt % B. Studies were made to evaluate the effect of various casting variables, including mold temperature and type of boron additions, on the homogeneity of these alloys. A method was devised for rating the alloys on the basis of boron recovery and degree of homogeneity.

Conventional tensile and hardness measurements were made to determine the effect of boron on the strength of aluminum; the microstructure was examined by means of metallographic and x-ray diffraction techniques.

The corrosion resistance of the boron-bearing alloys was compared with that of type 1100 aluminum by testing for 1000 hr in distilled and aerated static water at 60°C, and for 1000 and 2000 hr in distilled and aerated boiling water at 100°C.

CONCLUSIONS

The conclusions drawn as a result of the work described in this report are listed below.

1. Conventional air induction melting techniques, using graphite crucibles and slab shaped molds, can be successfully employed in the preparation of boron-aluminum and boron-uranium-aluminum alloys containing 0.02 to 0.4 wt % B.

2. Chill casting in graphite slab molds and in 3-in.-dia water-cooled copper molds does not appear to improve homogeneity in binary or ternary alloys.

3. Extrusion of 3-in.-dia cylindrical billets into ¼-in.-thick plates does not result in a significant improvement in homogeneity of either boron-aluminum or boron-uranium-aluminum alloys.

4. Aluminum-boron master alloys, containing 1.5 to 5 wt % B, and nickel-boron master alloys, containing 11 to 13 wt % B, are the most suitable agents for preparing boron-aluminum alloys containing 0.020 to 0.200 wt % B.

5. Of the master additions investigated, a nickel-boron alloy containing 11 to 13 wt % B is the most suitable agent for incorporating boron homogeneously in a 17 wt % U-Al alloy.

6. It is more difficult to prepare homogeneous boron-uranium-aluminum alloys than boron-aluminum alloys.

7. Boron exhibits a tendency to segregate at the bottom and top of the castings. If extremely homogeneous material is required, it is likely that cropping of the bottom and top of the casting will be necessary.

8. AlB_{12}, wrapped in aluminum foil, is not a suitable master agent for preparation of boron-uranium-aluminum alloys.

9. Boron addition improves the strength properties of aluminum.

10. In static, distilled and aerated water at 60°C and in boiling, distilled and aerated water at 100°C, the corrosion resistance of nominal 0.1 wt % B–0.8 wt % Ni–Al alloys is more favorable than the corrosion resistance of nominal 0.1 wt % B–Al alloys, and is comparable to the corrosion resistance of type 1100 aluminum under similar conditions.

EXPERIMENTAL WORK

Casting Practice

During this development program, 53 castings were prepared by use of a wide variety of casting conditions and several boron-containing additions. The boron-containing additions investigated included finely divided AlB_{12} wrapped in aluminum foil, pressed and sintered compacts of both AlB_{12} and amorphous boron with aluminum, and small chunks of aluminum-boron and nickel-boron master alloys. Twenty-six billets of 17 wt % U–Al alloy with boron compositions ranging from 0.2 to 0.4 wt % B, and 27 billets with boron compositions ranging from 0.02 to 0.3 wt % B–Al were cast during this investigation. The ternary compositions were representative of a typical fuel-element core alloy, while the binary alloys were representative of a potential cladding material containing an equivalent amount of boron. The casting variables involved in preparing ternary alloys are compiled in Table 1, while similar data for the binary alloys are presented in Table 2.

This wide diversity of effort was necessitated in order to achieve satisfactory solutions to the two serious casting problems encountered, namely,

1. erratic and often low recovery of boron in the cast billet,
2. concentration of boron in the upper portion of the casting.

With the exception of billets Nos. 27 and 28, which were melted under vacuum and argon, respectively, all melting was done in open-air induction furnaces. Crucibles were generally graphite, although refractory oxides were used occasionally. Molds were of two types:

1. The slab-type mold was 3 in. wide, 10½ in. high, and varied in thickness from ½ in. to 1¼ in. A tapered cross-section slab mold was used extensively though not exclusively.

2. The cylinder-type mold was 3 in. in diameter and 10 in. high; it was made of copper and was water-cooled.

The basic melting stocks were high-purity (99.99%) aluminum and depleted uranium. The spectrographic analysis of the uranium used is included in Appendix A.

Casting practice entailed the melting down of 1-lb aluminum ingots and superheating the melt to

Table 1. Casting Variables in Preparation of Boron-Uranium-Aluminum Alloys

Graphite used as both crucible and mold material except as noted

Billet No.	Intended Boron Content (wt %)	Analyzed Boron Content (Spectrographic) (wt %)	Analyzed Boron Content (Potentiometric) (wt %)	Boron-Boride Addition Temperature (°C)	Pouring Temperature (°C)	Mold Temperature (°C)	Mold Size (in.)	Form of Boron Addition
1	0.208	0.093		950	900	375	3 × 10½ × ¾ to 1 tapered cross section	−40 mesh arc-melted AlB_{12} (77.4% B)
2	0.208	0.071		950	900	275	3 × 10½ × ¾ to 1 tapered cross section	20–30 mesh arc-melted AlB_{12} (77.4% B)
3	0.205	0.161	0.140	950	900	300	3 × 10½ × ¾ to 1 tapered cross section	40–200 mesh Cooper AlB_{12} (81.4% B) wrapped in Al foil
4	0.205	0.037	0.054	950	900	300	3 × 10½ × ¾ to 1 tapered cross section	40–200 mesh Cooper AlB_{12} (81.4% B) wrapped in Al foil
5	0.205	0.117	0.092	950	900	270	3 × 10½ × ¾ to 1 tapered cross section	40–200 mesh Cooper AlB_{12} (81.4% B) wrapped in Al foil
6[a]	0.205	0.212	0.176	950	900	40	3 ID × 10 high	40–200 mesh Cooper AlB_{12} (81.4% B) wrapped in Al foil
7[a]	0.205	0.234	0.190	950	900	32	3 ID × 10 high	40–200 mesh Cooper AlB_{12} (81.4% B) wrapped in Al foil
8[b]	0.205	0.020	0.065	950	900	300	3 × 10½ × ¾ to 1 tapered cross section	40–200 mesh Cooper AlB_{12} (81.4% B) wrapped in Al foil
9	0.205	0.097	0.110	950	900	100	3 × 10½ × ¾ to 1 tapered cross section	40–200 mesh Cooper AlB_{12} (81.4% B) wrapped in Al foil
10	0.244	0.139	0.121	950	900	340	3 × 10½ × ¾ to 1 tapered cross section	40–200 mesh Cooper AlB_{12} (81.4% B) wrapped in Al foil
11	0.244	0.051	0.043	950	900	340	3 × 10½ × ¾ to 1 tapered cross section	40–200 mesh Cooper AlB_{12} (81.4% B) wrapped in Al foil
12	0.263		0.111	Room temp	900	75	3 × 10½ × ¾ to 1 tapered cross section	Blended, compacted, and sintered Al + AlB_{12} powders
13	0.263		0.142	Room temp	900	75	3 × 10½ × ¾ to 1 tapered cross section	Blended, compacted, and sintered Al + AlB_{12} powders
14	0.360		0.135	Room temp	900	100	3 × 10½ × ¾ to 1 tapered cross section	Blended, compacted, and sintered Al + AlB_{12} powders
15	0.360		0.095	Room temp	900	100	3 × 10½ × ¾ to 1 tapered cross section	Blended, compacted, and sintered Al + AlB_{12} powders
16	0.360		0.124	Room temp	900	100	3 × 10½ × ¾ to 1 tapered cross section	Blended, compacted, and sintered Al + AlB_{12} powders
17	0.219	0.121		950	850		3 × 10½ × ¾ to 1 tapered cross section	Small chunks of 19.93% boron–aluminum master alloy
18	0.220	0.158		900	900		3 × 10½ × ¾ to 1 tapered cross section	Small chunks of 43.7% boron–aluminum master alloy
19	0.220	0.131			900		3 × 10½ × ¾ to 1 tapered cross section	Scrap from billets 17 + 18
20	0.301	0.347		1000	800		3 × 10½ × ¾ to 1 tapered cross section	Small chunks of 43.7% boron–aluminum master alloy
21	0.349		0.258	Room temp	900	325	3 × 10½ × ¾ to 1 tapered cross section	Small chunks of Cooper 4.67% boron–aluminum master alloy
22	0.233		0.212	Room temp	900	350	3 × 10½ × ¾ to 1 tapered cross section	Small chunks of Ni-B (~12% B) master alloy
23	0.220		0.183	Room temp	900	325	3 × 10½ × ¾ to 1 tapered cross section	Small chunks of Ni-B (~12% B) master alloy
24	0.220		0.200	Room temp	900	305	3 × 10½ × ¾ to 1 tapered cross section	Small chunks of Ni-B (~12% B) master alloy
25	0.220		0.198	Room temp	900	290	3 × 10½ × ¾ to 1 tapered cross section	Small chunks of Ni-B (~12% B) master alloy
26	0.220		0.134	Room temp	900	315	3 × 10 × ½ (flat)	Small chunks of Ni-B (~12% B) master alloy

[a] Copper mold used.
[b] MgO crucible used.

Table 2. Casting Variables in Preparation of Boron-Aluminum Alloys

Graphite used as both crucible and mold material except as noted

Billet No.	Intended Boron Content (wt %)	Analyzed Boron Content (Spectrographic) (wt %)	Analyzed Boron Content (Potentiometric) (wt %)	Boron-Boride Addition Temperature (°C)	Pouring Temperature (°C)	Mold Temperature (°C)	Mold Size (in.)	Form of Boron Addition
27[a]	0.243	0.268	0.206	Room temp	890	Room temp	$4 \times 4 \times \frac{1}{2}$	Blended, compacted, and sintered Al + AlB_{12} powders
28[b]	0.243	0.270	0.183	Room temp	925	Room temp	$4 \times 4 \times \frac{1}{2}$	Blended, compacted, and sintered Al + AlB_{12} powders
29[c]	0.200	0.179	0.172	Room temp	900	50	3 ID × 10	Blended, compacted, and sintered Al + B powders
30[c]	0.174	0.240	0.201	Room temp	900		3 ID × 10	Blended, compacted, and sintered Al + AlB_{12} powders
31	0.200	0.166	0.140	Room temp	900	250	$3 \times 10\frac{1}{2} \times \frac{3}{4}$ to 1 tapered cross section	Blended, compacted, and sintered Al + B powders
32	0.174	0.171	0.146	Room temp	900	175	$3 \times 10\frac{1}{2} \times \frac{3}{4}$ to 1 tapered cross section	Blended, compacted, and sintered Al + AlB_{12} powders
33	0.194		0.182	Room temp	900	310	$3 \times 10\frac{1}{2} \times \frac{3}{4}$ to 1 tapered cross section	Small chunks of Ni-B (~12% B) master alloy
34	0.099		0.091	Room temp	900	350	$3 \times 10\frac{1}{2} \times \frac{3}{4}$ to 1 tapered cross section	Small chunks of Ni-B (~12% B) master alloy
35	0.099		0.084	Room temp	900	330	$3 \times 10 \times \frac{1}{2}$(flat)	Small chunks of Ni-B (~12% B) master alloy
36	0.129		0.130	Room temp	900	340	$3 \times 10\frac{1}{2} \times \frac{3}{4}$ to 1 tapered cross section	Small chunks of Alcoa Al-B (0.75% B) master alloy
37	0.100	0.064	0.074	Room temp	900		$3 \times 10\frac{1}{2} \times \frac{3}{4}$ to 1 tapered cross section	Small chunks of Cooper Al-B (4.67% B) master alloy
38	0.101		0.083	Room temp	900	~75	$3 \times 10\frac{1}{2} \times 1$ to $1\frac{1}{4}$ tapered cross section	Small chunks of Cooper Al-B (4.67% B) master alloy
39	0.101		0.099	Room temp	900	~75	$3 \times 10\frac{1}{2} \times 1$ to $1\frac{1}{4}$ tapered cross section	Small chunks of Cooper Al-B (4.67% B) master alloy
40	0.100		0.119	Room temp	900	~75	$3 \times 10\frac{1}{2} \times 1$ to $1\frac{1}{4}$ tapered cross section	Small chunks of Cooper Al-B (2.60% B) master alloy
41	0.100		0.099	Room temp	900	~75	$3 \times 10\frac{1}{2} \times 1$ to $1\frac{1}{4}$ tapered cross section	Small chunks of Cooper Al-B (2.60% B) master alloy
42	0.100		0.109	Room temp	900	~75	$3 \times 10\frac{1}{2} \times 1$ to $1\frac{1}{4}$ tapered cross section	Small chunks of Cooper Al-B (2.60% B) master alloy
43	0.100		0.115	Room temp	900	~75	$3 \times 10\frac{1}{2} \times 1$ to $1\frac{1}{4}$ tapered cross section	Small chunks of Cooper Al-B (2.60% B) master alloy
44	0.100		0.120	Room temp	900	~75	$3 \times 10\frac{1}{2} \times 1$ to $1\frac{1}{4}$ tapered cross section	Small chunks of Cooper Al-B (2.60% B) master alloy
45	0.100		0.111	Room temp	900	~75	$3 \times 10\frac{1}{2} \times 1$ to $1\frac{1}{4}$ tapered cross section	Small chunks of Cooper Al-B (2.60% B) master alloy
46	0.100		0.106	Room temp	900	~75	$3 \times 10\frac{1}{2} \times 1$ to $1\frac{1}{4}$ tapered cross section	Small chunks of Cooper Al-B (2.60% B) master alloy
47	0.100		0.115	Room temp	900	~75	$3 \times 10\frac{1}{2} \times 1$ to $1\frac{1}{4}$ tapered cross section	Small chunks of Cooper Al-B (2.60% B) master alloy
48	0.100		0.121	Room temp	900	~75	$3 \times 10\frac{1}{2} \times 1$ to $1\frac{1}{4}$ tapered cross section	Small chunks of Cooper Al-B (2.60% B) master alloy
49	0.100		0.103	Room temp	900	~75	$3 \times 10\frac{1}{2} \times 1$ to $1\frac{1}{4}$ tapered cross section	Small chunks of Cooper Al-B (2.60% B) master alloy
50	0.100		0.100	Room temp	900	~75	$3 \times 10\frac{1}{2} \times 1$ to $1\frac{1}{4}$ tapered cross section	Small chunks of Cooper Al-B (2.60% B) master alloy
51	0.100		0.115	Room temp	900	~75	$3 \times 10\frac{1}{2} \times 1$ to $1\frac{1}{4}$ tapered cross section	Small chunks of Cooper Al-B (2.60% B) master alloy
52	0.100		0.125	Room temp	900	~75	$3 \times 10\frac{1}{2} \times 1$ to $1\frac{1}{4}$ tapered cross section	Small chunks of Cooper Al-B (2.60% B) master alloy
53	0.022		0.029	Room temp	900	~75	$3 \times 10\frac{1}{2} \times 1$ to $1\frac{1}{4}$ tapered cross section	Small chunks of Al + B^{10} (1.55% B) master alloy

[a] ZrO_2 crucible and stainless steel mold used.
[b] ZrO_2 crucible used.
[c] Copper mold used.

900°C. If uranium was to be added, the addition was made at this time, using 1-in. squares of $\frac{1}{8}$-in.-thick sheet. The melt was further superheated to 1075°C to ensure complete dissolution of both the uranium, if added, and the boron-containing material; it was then cooled to 900°C and poured into the mold, which had a wall temperature that varied from ambient to 375°C.

In a majority of the melts the boron addition was included with the initial aluminum charge, while in the few remaining cases the addition was made to the melt at 950 to 1000°C. When the aluminum-wrapped AlB_{12} was used, the material was held under the surface of the melt with a graphite stirring rod in order to facilitate solution of the boride.

Sampling Procedure

Alloy castings containing uranium and boron were generally hot-rolled to a thickness of 0.255 in., and then 2.3 × 2.0 in. cores were punched from the fabricated plate. Samples for boron analyses were taken from the material remaining after the cores had been removed. Figure 1 shows a punched and sampled ternary alloy billet. Six to ten samples were taken along the length of each of these rolled billets for the determination of boron content at representative locations.

The binary alloy castings were rolled to 0.090 in. in thickness before sampling. Although some of the binary material was extensively sampled, slabs Nos. 38 through 52 were sampled only at the top, middle, and bottom after cropping of the segregated ends, as shown in Fig. 2.

Ternary alloy billet No. 7 and binary alloy billets Nos. 29 and 30 were extruded through a flat-face die in 0.250-in.-thick strips and sampled by drilling along the length of the extruded strip. A typical extruded billet after sampling is shown in Fig. 3.

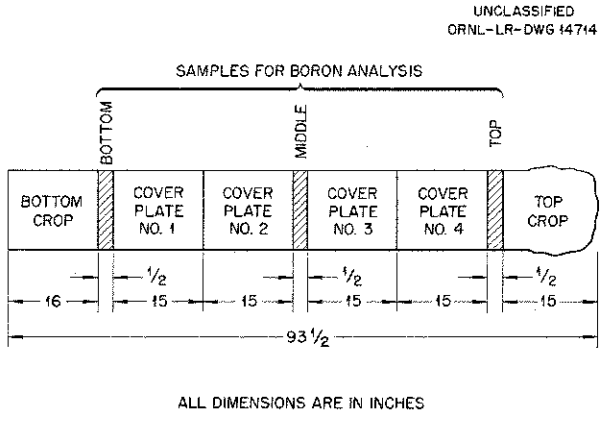

Fig. 2. Diagram Showing Cropping and Sampling of Al + 0.1% B Alloy.

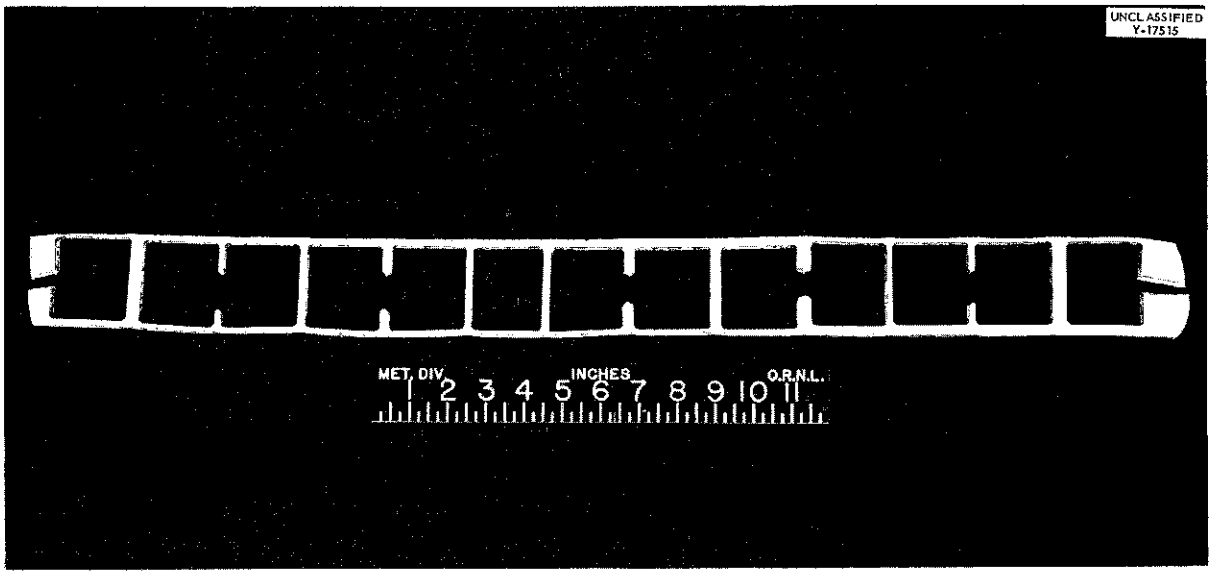

Fig. 1. Skeleton of Hot-rolled 0.2 wt % B–17 wt % U–Al Slab After Punching of Cores and Removal of Samples for Boron Analyses.

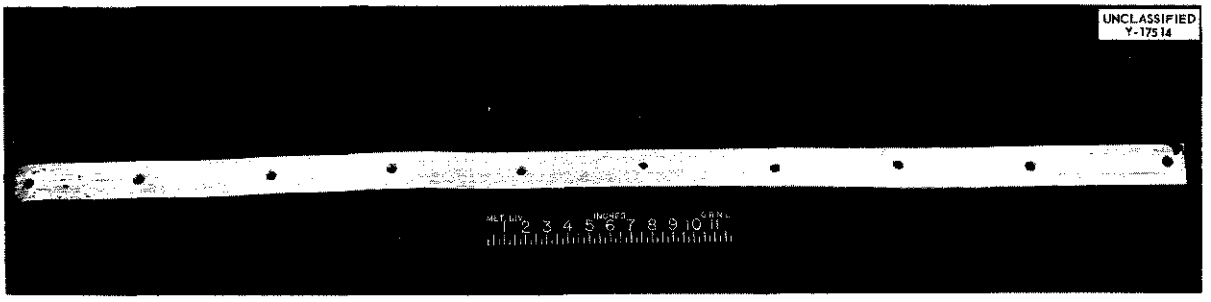

Fig. 3. Extruded 0.2 wt % B–Al Alloy Strip Showing Location of Samples for Boron Analyses.

Analytical methods used in this investigation are summarized in Appendix D.

Boron Addition Agents

AlB_{12} Wrapped in Aluminum Foil. – Boron has no known solubility in solid aluminum, and its solubility in liquid aluminum at 950°C is only 0.35 wt % (*1*). In addition, two high-melting intermetallic compounds, hexagonal AlB_2 and tetragonal-monoclinic AlB_{12}, are reported to be in the system (*2*). Difficulties anticipated from the inherent characteristics of this binary system led to the selection of finely divided AlB_{12} as the boron addition agent for initial evaluation. The reasons for the selection of this material were as follows:

1. It was postulated that increased dissolution rate could be obtained with the finely divided compound.
2. If dissolution of AlB_{12} did not occur readily, a mechanical dispersion of AlB_{12} in an aluminum matrix could be attained with greater facility.
3. Since the boron-to-aluminum ratio is higher in AlB_{12} than in AlB_2, the amount of addition required would be reduced.

A 100-g ingot of AlB_{12} was prepared by arc melting of blended and cold-compacted aluminum and boron powders. The ingot was crushed and the resulting material was sized; only the –20-mesh fraction was retained for use. Chemical analysis of this material revealed a boron content of 77.4 wt %, although AlB_{12} is stoichiometrically 82.74 wt % B. As listed in Table 1, ternary alloy slabs Nos. 1 and 2, weighing about 1750 g each, were prepared with this material.

Subsequently, a commercially available AlB_{12} which contained 81.4 wt % B was obtained from Cooper Metallurgical Associates, Cleveland, Ohio, and substituted for the ORNL arc-melted compound. A detailed vendor's analysis of this material, which was in the –40 +200 mesh range, is included in Appendix B. Several experimental ternary alloy castings (slabs Nos. 3 through 11), listed in Table 1, were prepared by using this material wrapped in aluminum foil as the boron addition. These slabs were subjected to various casting modifications in an effort to minimize segregation and increase boron recovery. Slabs Nos. 10 and 11 contained enriched uranium and were used in fabricating a fuel element for irradiation testing of a boron-bearing fuel alloy in the Materials Testing Reactor (*3*).

Compacts of AlB_{12} in Aluminum. – Five ternary castings (Nos. 12 through 16), listed in Table 1 and four binary castings (Nos. 27, 28, 30, and 32), listed in Table 2, were prepared by using compacted additions of AlB_{12} in aluminum. These compacts were prepared by mixing, for 1 hr in an oblique blender, the charge of component powders in the weight ratio of 5 parts aluminum to 1 part AlB_{12}, cold compacting in an 0.8-in.-dia die at a pressure of 20 tons, and sintering in H_2 at 600°C for $1\frac{1}{4}$ hr. The five ternary castings prepared with this type of addition also contained enriched uranium and were used in manufacturing fuel assemblies in order to obtain additional irradiation testing data on the ternary alloy (*3*).

Compacts of Amorphous Boron in Aluminum. – Two boron-aluminum binary alloy castings (Nos. 29 and 31), listed in Table 2, were prepared by using compacts of amorphous boron in aluminum as melt additions. These compacts were manufactured in a manner analagous to that used in making the aluminum and AlB_{12} compacts described previously.

Aluminum-Boron Master Alloys. – Aluminum-boron master alloys were also examined as melt additions. Some of these master alloys were prepared at ORNL by arc melting mixtures of elemental powders, while others were obtained from commercial sources. Master alloys containing 0.75, 2.60, 4.67, 19.93, and 43.7 wt % B were used in preparing five ternary alloy castings (Nos. 17 through 21), listed in Table 1, and 17 binary alloy castings (Nos. 36 through 52), listed in Table 2. The alloy additions were in the form of small chunks $\frac{1}{4}$ to $\frac{1}{2}$ in. in size.

Slab No. 52, listed in Table 2, with an intended composition of 0.022 wt % B, was also cast by using a 1.55 wt % B-Al arc-melted master alloy. The boron had an enrichment of 90% in the B^{10} isotope.

Nickel-Boron Master Alloys. – A nickel-boron eutectic alloy with a nominal composition of 13 wt % B was also included as a boron addition agent, principally because of its low melting temperature of 900°C (*4*). It was postulated that the use of an addition containing boron combined with an element other than aluminum might alter the mode of boron dissolution or dispersion in the melt with a resultant improvement in homogeneity and recovery.

Nickel-boron master alloys with an intended composition of 13 wt % B were prepared by arc melting cold-pressed and sintered compacts of the elemental powders. The resultant alloys, which were shown by chemical analysis to contain about 12 wt % B, were extremely brittle and could be readily broken into small chunks for use as melt additions. These master alloys were used to prepare the five boron-nickel-uranium-aluminum alloys (Nos. 22 through 26), listed in Table 1, and the three boron-nickel-aluminum alloys (Nos. 33 through 35), listed in Table 2.

DISCUSSION OF RESULTS

Evaluation of Castings

On the basis of the analyzed boron content at the various locations sampled, each billet was rated according to recovery of boron and homogeneity. By use of a semiempirical evaluation scheme, each billet was assigned an over-all rating. Ratings for the ternary alloy and binary alloy castings are listed in Tables 3, 4, and 5, and the rating system is explained in detail in Appendix C.

Alloys Prepared with AlB_{12} Wrapped in Aluminum Foil. – The utility of this master alloy was investigated extensively in an effort to prepare homogeneous ternary aluminum alloys containing approximately 17 wt % U and 0.2 to 0.4 wt % B. As indicated in Table 3, the highest rating for any one of the 16 castings investigated was 76%, which is not considered to be satisfactory. In the vast majority of these alloys, the recovery of boron and the index of homogeneity were highly erratic. Boron exhibited a marked tendency to segregate in the top of the casting, as exemplified by the data presented in Fig. 4. The technique of chill casting 3-in.-dia billets (Nos. 6 and 7) in water-cooled copper molds yielded patterns, illustrated in Fig. 5, which indicate that this modification failed to eliminate segregation in the top portion of the casting. A further effort to chill-cast slab No. 9, by immersing a graphite mold in liquid nitrogen immediately after pouring, again resulted in a casting with poor boron recovery and mediocre homogeneity. The melting of casting No. 8 in a magnesia crucible resulted in excessive dross because of the absence of the semiprotective $CO\text{-}CO_2$ atmosphere present during melting in graphite crucibles. Analytical chemistry results revealed that the dross was an aluminum alloy of 0.6 to 1.0 wt % B, compared with an average boron content of only 0.02 wt % in the cast billet. The intended alloy composition was 0.205 wt % B.

These data indicate that the boron is entrapped in the dross during melting and that it segregates to the top portion of the casting during solidification. It may be possible that the difference in densities between the 17 wt % U–Al alloy melt (3.13 g/cc) and the AlB_{12} (2.58 g/cc), coupled with deleterious surface-tension effects, is responsible for the loss of boride to the dross. A possible explanation of the marked tendency of the boron to segregate to the top portion of the casting may be that the rejection of the first solidifying intermetallic compound to the upper portion of the casting by the advancing solid front occurs during freezing of the alloy.

Alloys Prepared with Compacted Additions of AlB_{12} in Aluminum. – Binary alloy castings Nos. 27, 28, 30, and 32 (Table 4), which were prepared by using compacted additions of AlB_{12} in aluminum as the master agent, exhibited satisfactory boron recoveries varying from 75 to 100%. Except for No. 30, which was cast in a water-cooled copper mold and subsequently extruded, these castings

Table 3. Rating of Boron-Uranium-Aluminum Alloy Castings

Casting No.	Intended Boron Content (wt %)	Recovery* (%)	Index of Homogeneity	Rating	Type of Boron Addition
1	0.208	44	76	65	AlB_{12}, arc-melted at ORNL
2	0.208	34	70	58	AlB_{12}, arc-melted at ORNL
3	0.205	68	78	75	Commercial AlB_{12}
4	0.205	26	70	55	Commercial AlB_{12}
5	0.205	45	58	54	Commercial AlB_{12}
6	0.205	86	20	42	Commercial AlB_{12}
7	0.205	93	68	76	Commercial AlB_{12}
8	0.205	32	78	63	Commercial AlB_{12}
9	0.205	54	75	68	Commercial AlB_{12}
10	0.244	50	88	75	Commercial AlB_{12}
11	0.244	18	86	63	Commercial AlB_{12}
12	0.263	42	22	29	Commercial AlB_{12}
13	0.263	54	25	35	Commercial AlB_{12}
14	0.360	38	33	35	Commercial AlB_{12}
15	0.360	26	20	22	Commercial AlB_{12}
16	0.360	34	29	31	Commercial AlB_{12}
17	0.219	55	85	75	Arc-melted Al-B master alloy (19.9 wt % B)
18	0.220	72	77	75	Arc-melted Al-B master alloy (43.7 wt % B)
19	0.220	60	78	72	Scrap from billets 17 and 18
20	0.301	100	85	90	Arc-melted Al-B master alloy (43.7 wt % B)
21	0.349	74	76	75	Commercial Al-B master alloy (4.67 wt % B)
22	0.233	91	88	89	Ni-B master alloy (11.8 wt % B)
23	0.220	83	78	80	Ni-B master alloy (11.7 wt % B)
24	0.220	91	92	92	Ni-B master alloy (12.0 wt % B)
25	0.220	90	92	91	Ni-B master alloy (11.8 wt % B)
26	0.220	61**	50	54	Ni-B master alloy (11.8 wt % B)

*Recovery based on potentiometric boron analyses except for billets Nos. 1, 2, and 17 through 20, which are calculated from spectrographic boron analyses.

**Flat cast slab.

Table 4. Rating of Boron-Aluminum Alloy Castings Based on 100% Casting-to-Plate Yield

Casting No.	Intended Boron Content (wt %)	Recovery (%)	Index of Homogeneity	Rating	Type of Boron Addition
27	0.243	85	98	94	Compact of AlB_{12} in Al
28	0.243	75	97	88	Compact of AlB_{12} in Al
29	0.200	86	33	51	Compact of B in Al
30	0.174	100	28	47	Compact of AlB_{12} in Al
31	0.200	70	73	72	Compact of B in Al
32	0.174	84	83	83	Compact of AlB_{12} in Al
33	0.194	94	93	93	11.4 wt % B–Ni alloy
34	0.099	92	74	80	11.7 wt % B–Ni alloy
35	0.099	85	96	92	12.0 wt % B–Ni alloy
36	0.129	100	66	77	Commercial 0.75 wt % B–Al alloy
37	0.100	74	90	85	Commercial 4.67 wt % B–Al alloy
53	0.022	100	92	95	1.55 wt % B–Al alloy arc-melted at ORNL

Table 5. Rating of Boron-Aluminum Alloy Castings, Containing 0.10 wt % Boron, Prepared with B-Al Master Alloys Based on 66% Casting-to-Plate Yield

Casting No.	Recovery (%)	Index of Homogeneity	Rating	Boron in Commercial Master Alloy (wt %)
38	82	99	93	4.67
39	100	87	91	4.67
40	100	87	91	4.67
41	99	93	95	4.67
42	100	94	96	4.67
43	100	96	97	2.60
44	100	92	95	2.60
45	100	94	96	2.60
46	100	98	99	2.60
47	100	99	99	2.60
48	100	96	97	2.60
49	100	94	96	2.60
50	100	94	96	2.60
51	100	95	97	2.60
52	100	84	89	2.60

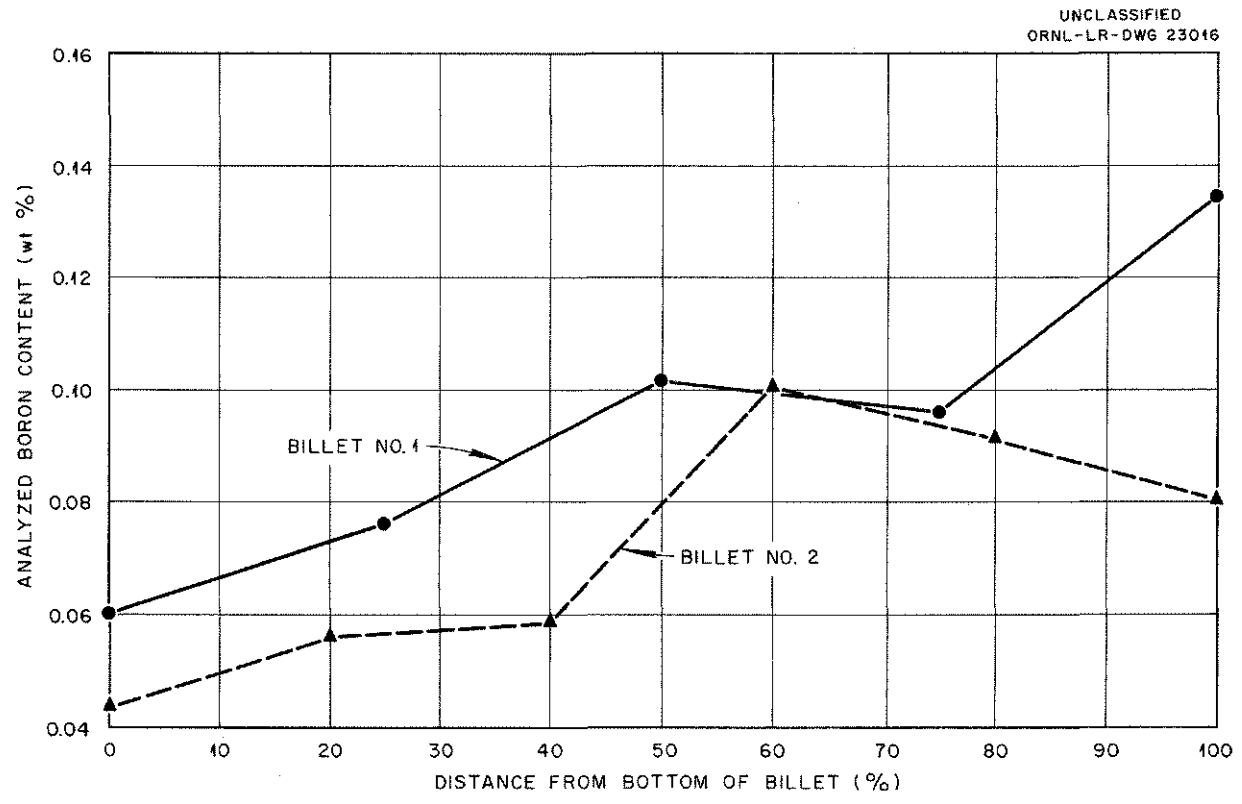

Fig. 4. Boron Distribution in B-U-Al Alloys Prepared with AlB_{12} and Cast in Heated Graphite Molds.

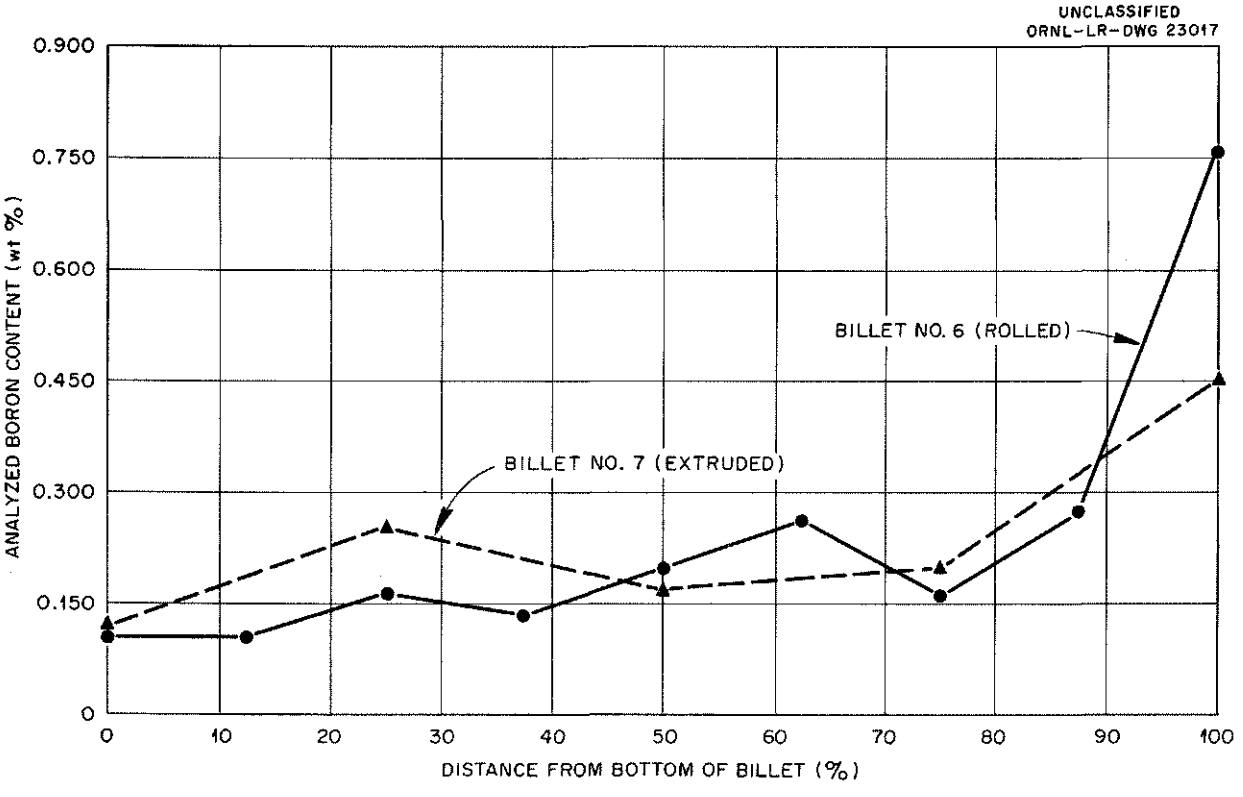

Fig. 5. Boron Distribution in Rolled and Extruded B-U-Al Alloys Prepared with AlB_{12} and Cast in Water-cooled Copper Molds.

also had high indices of homogeneity, ranging from 83 to 98. Apparently, the use of a compacted master addition, in which the boride is intimately associated with the aluminum, overcomes adverse surface-tension effects during melting and casting of the binary alloys. This allows the AlB_{12} to be wet by the melt and thus minimizes loss to the dross.

Figure 6 illustrates the needlelike borides in the microstructure of a typical cladding alloy which contains 0.16 wt % B prepared with a compacted addition of AlB_{12}. Figure 7 shows, in the same alloy, a boride particle which appears to have been sectioned parallel to the basal plane of a hexagonal crystal. On the basis of crystal geometry, this particle was assumed to be the intermetallic compound AlB_2.

Attempts to extend the use of this compacted type of boron addition to ternary castings were not considered to be successful. Ternary castings Nos. 12 through 16, prepared with Al + AlB_{12} compacts, had very poor boron recovery and were badly segregated. Boron distribution curves for slabs Nos. 12 and 13, shown in Fig. 8, typify the top segregation of boron existing in these ternary castings.

The reason for the poor results is not clear but again appears to be associated with the greater density of the boron-uranium-aluminum alloy melts, as compared with the boron-aluminum melts. It was observed that the boron-containing compacts readily submerged in aluminum melts, while in the uranium-aluminum alloy melts they tended to float on the surface, increasing the probability of boron loss by entrapment in the dross.

Alloys Prepared with Compacted Additions of Amorphous Boron in Aluminum. – Binary castings Nos. 29 and 31 were prepared with compacted additions in which amorphous boron was substituted for the AlB_{12}. Casting and fabrication procedures for these two billets were made identical with those for billets Nos. 30 and 32 for the purpose

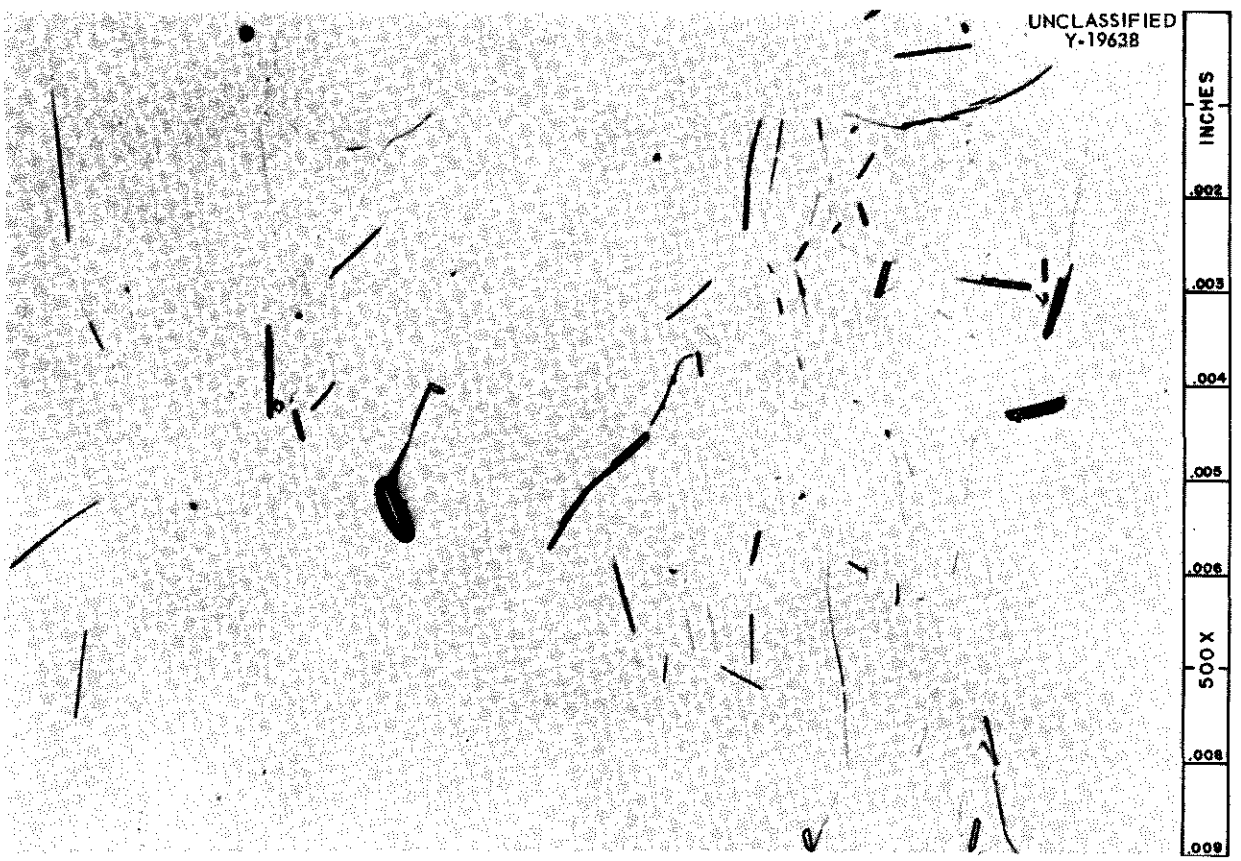

Fig. 6. An Al + 0.16% B Cladding Alloy Prepared with Compacted Addition of Al + AlB_{12}. As polished. 500X.

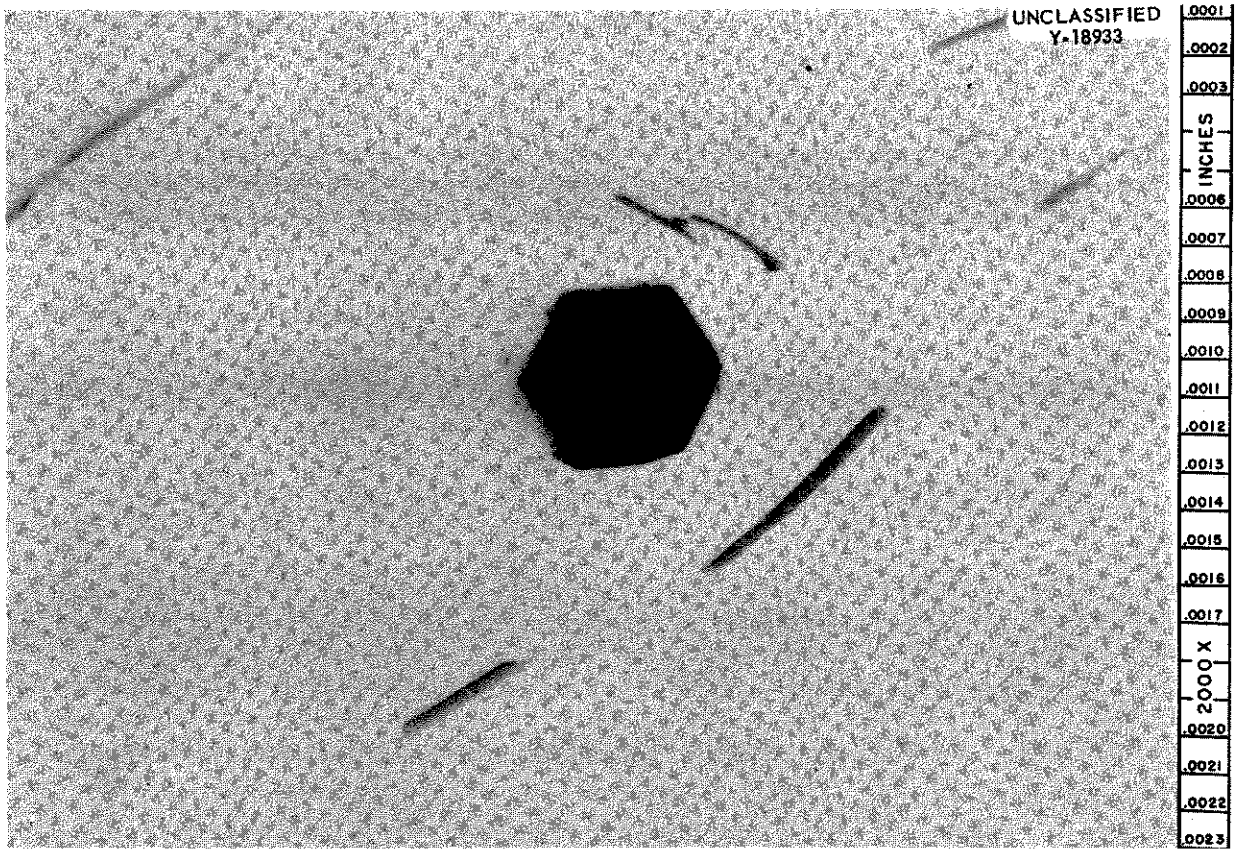

Fig. 7. Same Material as Indicated in Fig. 6 Showing AlB_2 Particles Preserved in Aluminum Matrix. As polished. 2000X.

of comparison. As shown in Fig. 9, the distribution curves for the two types of material (AlB_{12} and B) are quite similar. The alloy cast in the water-cooled copper mold also exhibited, in the upper portion of the casting, the marked segregation which was previously observed in billet No. 30.

Preliminary information on changing the deformation process from rolling to extrusion for breakdown of the casting into plate did not reveal any significant improvement in the subsequent homogeneity of the alloy. Billets Nos. 6, 7, 29, and 30 were cast in 3-in.-dia water-cooled copper molds. Billet No. 6 was rolled at 600°C; the others were extruded at 600°C. Figures 5 and 9 illustrate that all these billets exhibited segregation in the upper portions. As mentioned previously, it appears that this segregation occurred during casting and was not significantly changed by the severe deformation of an extrusion process.

Alloys Prepared with Aluminum-Boron Master Alloys. – Five ternary castings (Nos. 17 through 21, listed in Table 3), prepared with aluminum-boron master alloys, had reasonably reproducible boron distribution patterns with indices of homogeneity varying from 76 to 85%. Boron recovery, however, was erratic and generally low. Dissolution of the 19.9 and 43.7 wt % B–Al master alloys proved to be difficult; in fact, a sizable inclusion of undissolved master alloy was observed in slab No. 17. A subsequent alloy (billet No. 21) was prepared with a 4.67 wt % B–Al master addition, but again the recovery and homogeneity were not considered to be satisfactory. The boron distribution curves for these alloys are presented in Fig. 10.

The results for billet No. 37 (given in Table 4), in which a 0.100 wt % B–Al binary alloy was prepared with a 4.67 wt % B–Al master alloy,

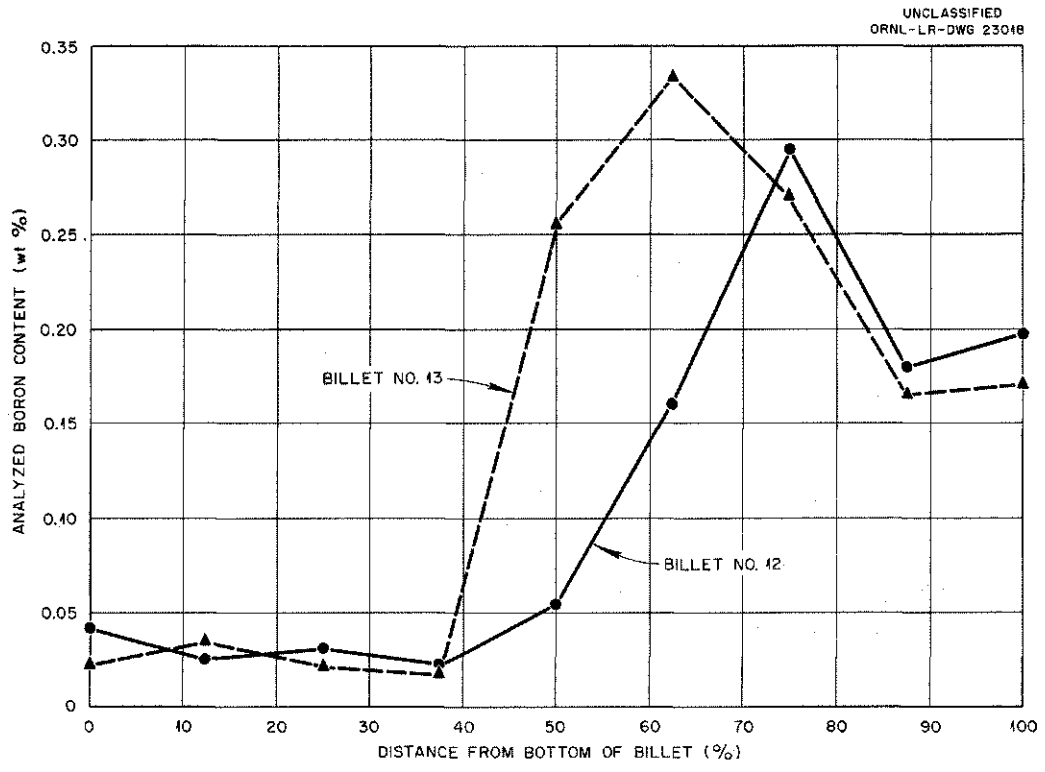

Fig. 8. Boron Distribution in B-U-Al Alloys Prepared with Compacted Additions of Al + AlB_{12}.

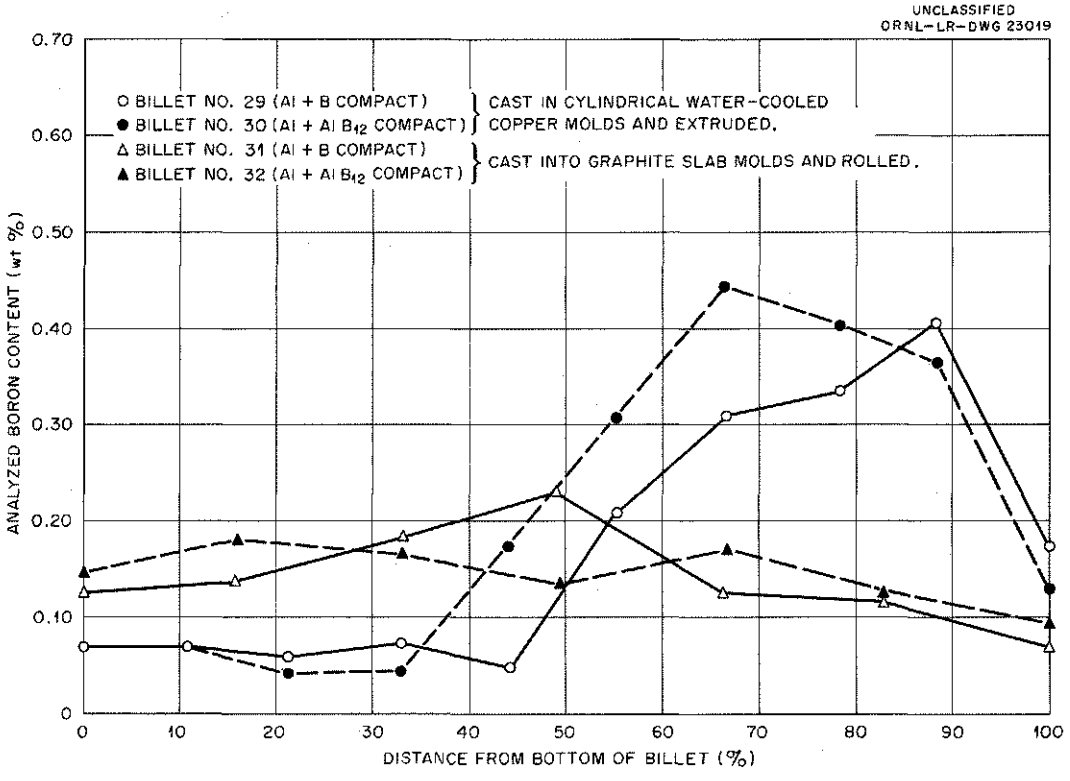

Fig. 9. Boron Distribution in Rolled and Extruded B-Al Alloys Prepared with Compacted Additions of Al + AlB_{12} and Al + B.

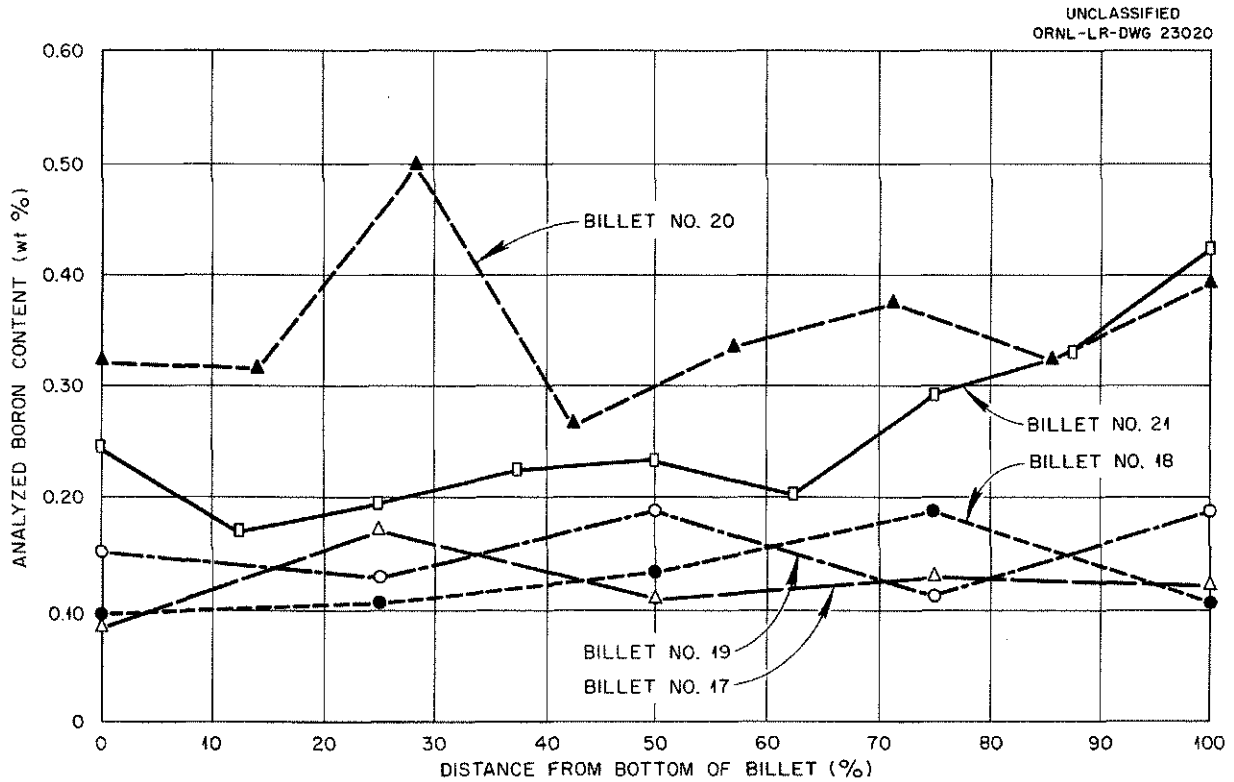

Fig. 10. Boron Distribution in B-U-Al Alloys Prepared with Al-B Master Alloys.

appeared to be promising. Although the recovery was not satisfactory, the homogeneity was considered to be good. It was observed that the index of homogeneity could be improved by cropping the top 17% and the bottom 17% of the rolled casting. This procedure reduced the casting-to-plate yield to 66%. The results of an appreciable number of plates, based on the 66% yield and the minimum sampling previously illustrated in Fig. 2, are listed in Table 5 and are represented graphically in Figs. 11 and 12. The ratings of the majority of castings are well above 90%. The 100% recovery of boron in these alloys is misleading because, in the majority of cases, the recovery based on chemical analysis was greater than the recovery calculated on the basis of intended boron content. This anomaly probably results from segregation in the master alloy and from inaccuracies in the analytical chemistry procedures.

One experiment, involving casting No. 53, which is listed in Table 4, was conducted to determine homogeneity in a boron-aluminum binary alloy containing 0.022 wt % B^{10}. The master alloy used contained 1.55 wt % B, as 90% enriched B^{10}. The results revealed complete recovery of the boron and very good homogeneity. These data suggest the possibility of utilizing boron, highly enriched in the B^{10} isotope, to reduce the total boron required.

Alloys Prepared with Nickel-Boron Master Alloy Containing Approximately 12 wt % B. – The results obtained by adding boron to aluminum with this master alloy in castings Nos. 33, 34, and 35 are listed in Table 4, and boron distribution curves are presented in Fig. 13. The ratings for slabs Nos. 33 and 35 compare quite favorably with the ratings of the binary alloys prepared with the 2.6 and 4.7 wt % B–Al master alloys.

As shown in the ratings listed in Table 3 for slabs Nos. 22 through 25, and in distribution curves illustrated in Fig. 14, boron-uranium-aluminum alloys prepared with the nickel-boron master alloy were consistently superior to ternary alloys prepared by other master additions. It may also be observed that billet No. 26 was cast in a radically different manner from the others, and it is presumed that the results are independent of the master-alloy addition.

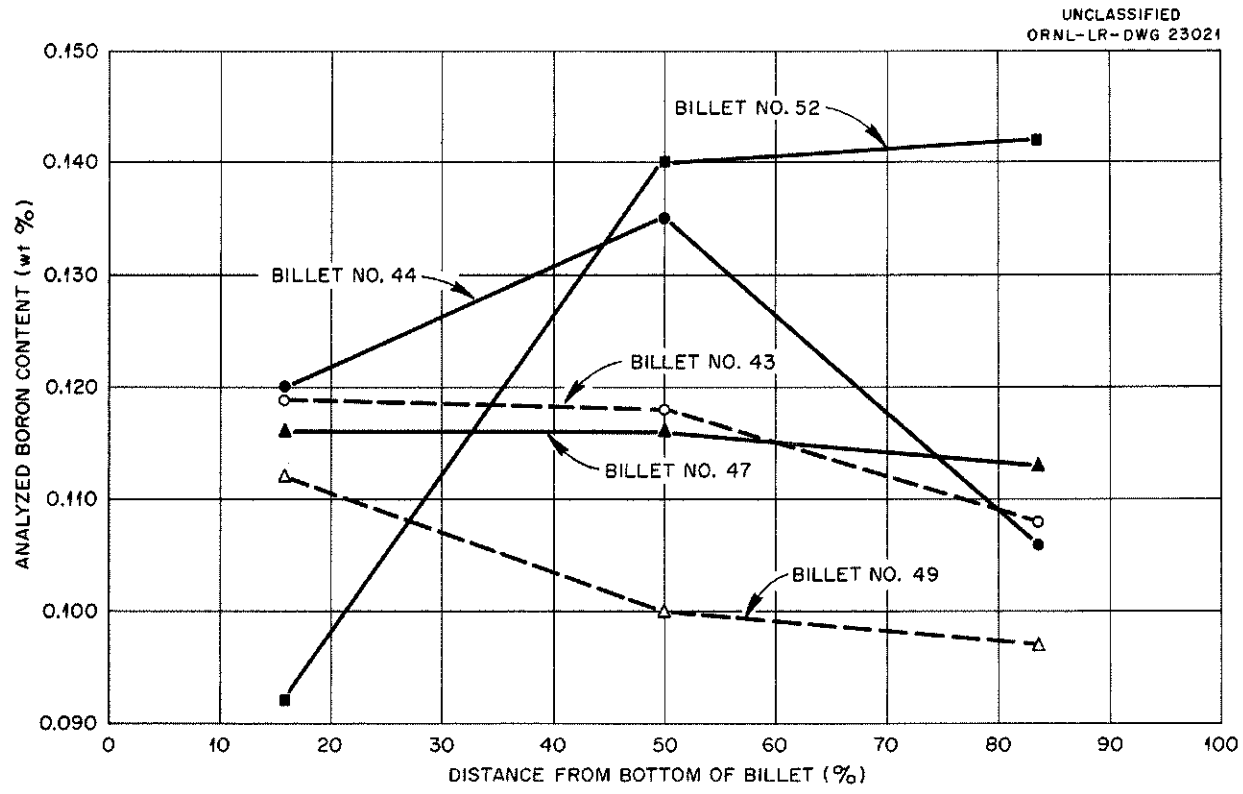

Fig. 11. Boron Distribution in B-Al Alloys Prepared with Al + 2.6% B Master Alloy.

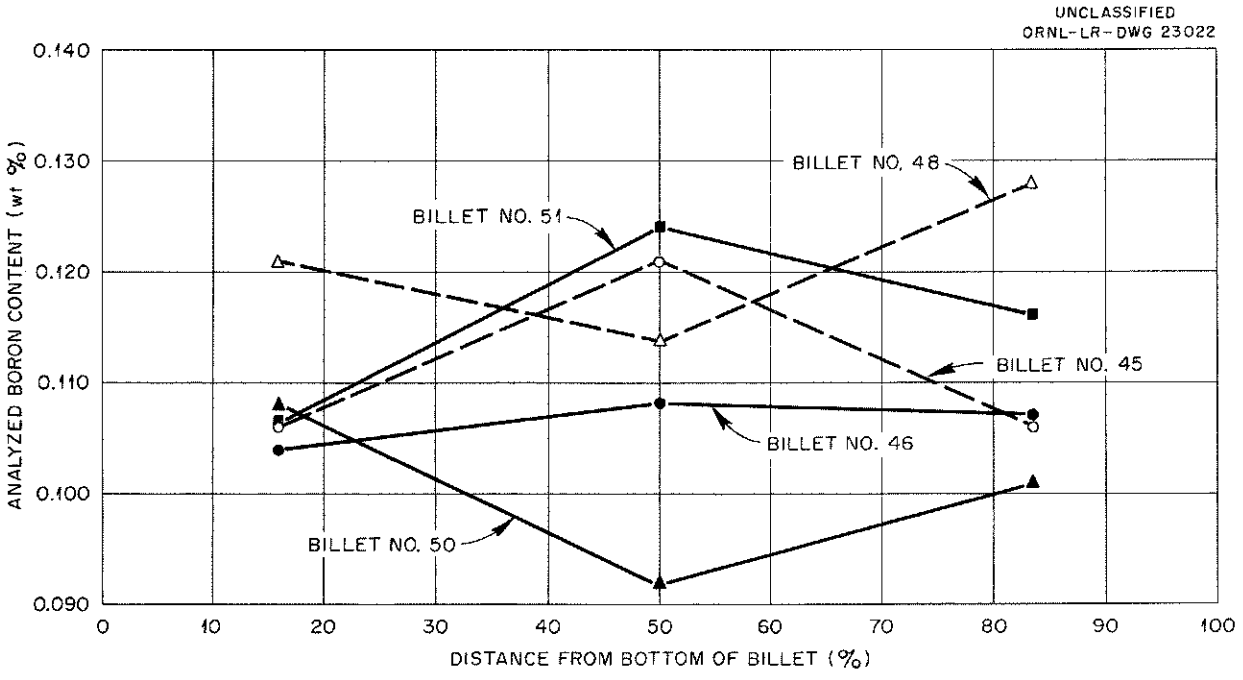

Fig. 12. Boron Distribution in B-Al Alloys Prepared with Al + 2.6% B Master Alloy.

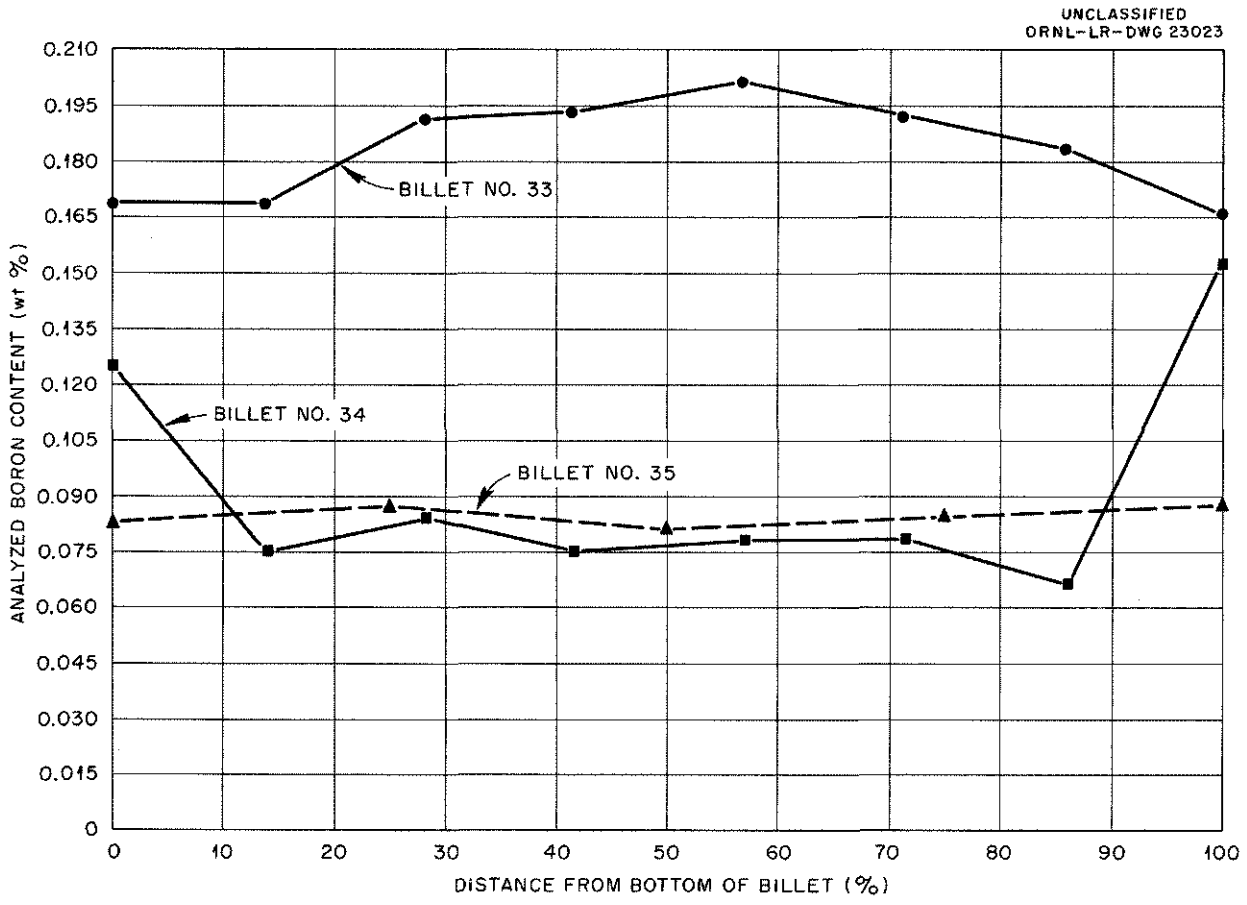

Fig. 13. Boron Distribution in B-Ni-Al Alloys Prepared with Ni-B Master Alloy.

Figure 15 illustrates the microstructure of an alloy with a nominal composition of 0.18 wt % B–1.5 wt % Ni–Al, showing the acicular borides distributed in a matrix of primary aluminum and eutectic (Al + Al_3Ni). These borides have not been identified but are assumed to be a complex aluminum-nickel boride.

Properties of Aluminum-Boron Alloys

Mechanical Properties. – The mechanical properties of a series of alloys ranging from 0.18 to 2.11 wt % B–bal Al were evaluated during the course of this investigation. Standard sheet-metal tensile specimens were obtained from arc-melted compacts of elemental powders which had been cold-rolled 90% to a thickness of 0.055 in. and annealed for 1.25 hr at 300°C.

Table 6 lists the analysis of these alloys and the extent to which each was cold-rolled before initiation of edge cracking. Binary alloys, containing up to 0.72 wt % B, exhibited no edge cracking.

Results of tensile tests on these materials as well as hardness measurements are included in Table 7, and the same data are illustrated graphically in Fig. 16. The strength properties increase with boron concentration, but at a decreasing rate. Apparently a dispersoid strengthening mechanism, involving a finely disseminated AlB_2 phase in a soft aluminum matrix, produces this observed trend.

The cross-sectional microstructures of 0.18 wt % B–Al and 0.55 wt % B–Al alloys, after 90% cold reduction and annealing, are shown in Figs. 17 and 18, respectively. The small rectangular particles distributed throughout the aluminum matrix have been tentatively identified by x-ray techniques as

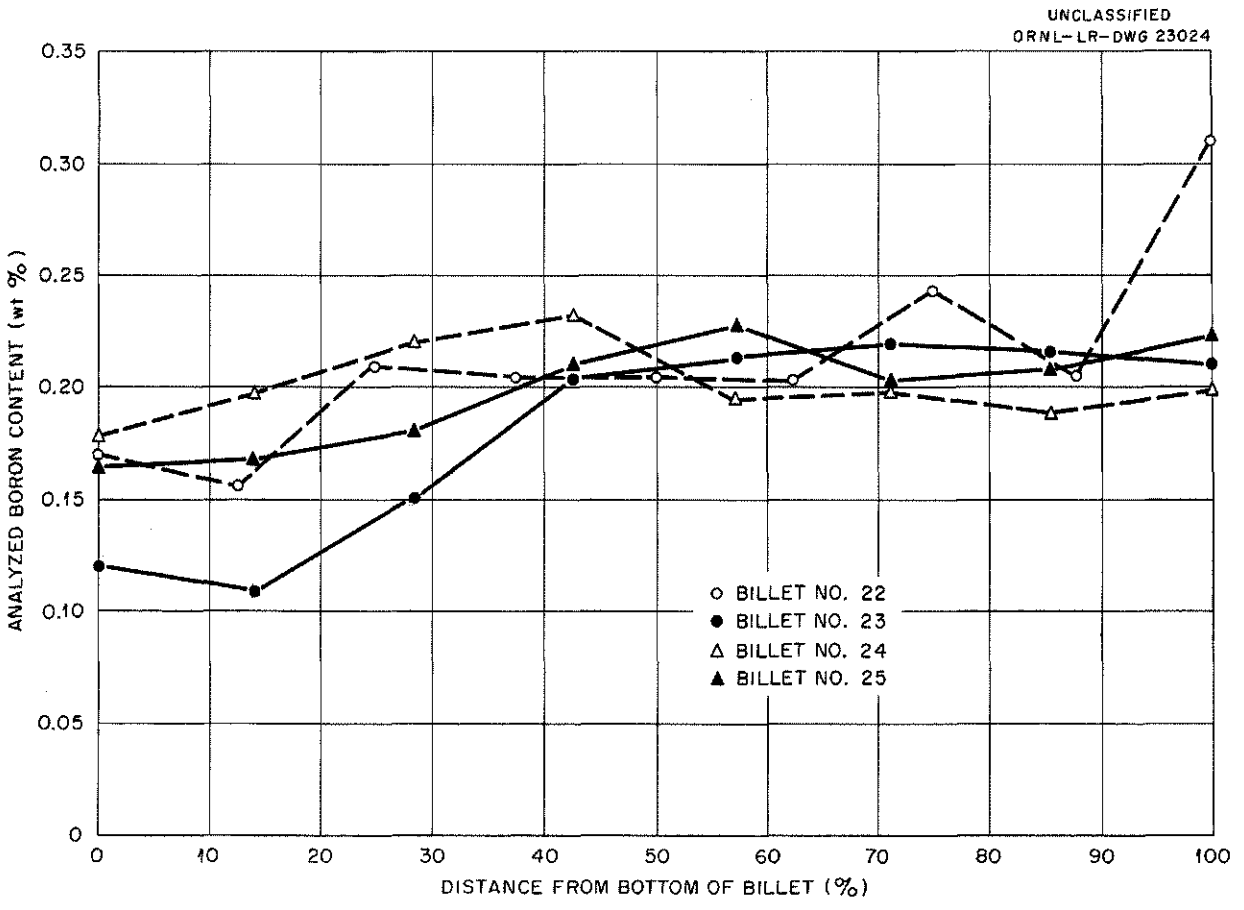

Fig. 14. Boron Distribution in B-U-Ni-Al Alloys Prepared with Ni-B Master Alloy.

Table 6. Composition and Cold Workability of Arc-Melted Boron-Aluminum Alloys

Alloy No.	Intended Boron Content (wt %)	Actual Boron Content (wt %) at			Average Boron Content (wt %)	Cold Reduction at First Edge Cracking (%)
		End 1	Center	End 2		
Al-1	0				0	
B-1	0.10	0.219	0.167	0.156	0.181	
B-2	0.20	0.418	0.319	0.279	0.339	
B-3	0.40	0.555	0.523	0.578	0.552	
B-4	0.80	0.890	0.656	0.630	0.725	
B-5	1.20	0.877	0.857	1.188	0.974	84
B-6	1.60	1.398	1.203	1.975	1.525	78
B-7	2.00	1.950	2.385	1.984	2.106	75

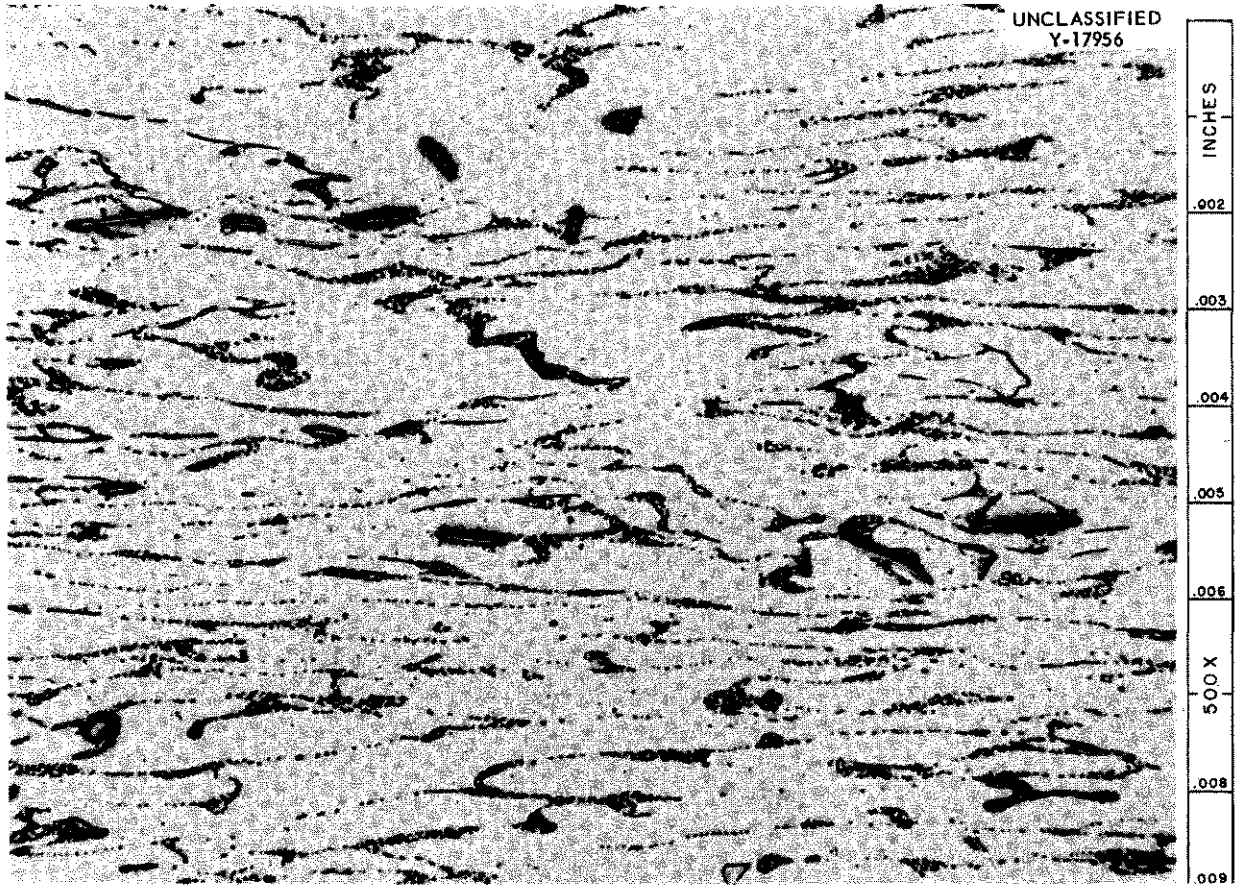

Fig. 15. Sample of Al + 1.5 wt % Ni + 0.18 wt % B Showing Borides Distributed in a Matrix of Primary Aluminum and Eutectic (Al + Al_3Ni). Etchant: 5% HF solution. 500X.

Table 7. Mechanical Properties of Boron-Aluminum Alloys

Alloy No.	Average Boron Content[a] (wt %)	Tensile Strength[a] (psi)	Yield Strength[a] (psi)	Elongation,[a] 2-in. Gage Length (%)	Hardness After 90% Cold Reduction[b] (VHN)	Hardness After Annealing[c] (VHN)
Al-1	0	7,470	2100	47.5	33.6	18.4
B-1	0.181	9,210	2870	37.4	39.0	21.0
B-2	0.339	9,660	3470	35.6	41.2	21.3
B-3	0.552	10,060	3380	32.0	36.8	22.3
B-4	0.725	11,160	3810	27.5	43.5	24.6
B-5	0.974	11,410	4040	22.2	44.1	24.4
B-6	1.525	12,050	4650	30.8	44.0	26.4
B-7	2.106	13,020	5160	19.4	48.7	30.3

[a]Average of two tests.
[b]Average of 20 readings.
[c]Average of ten readings.

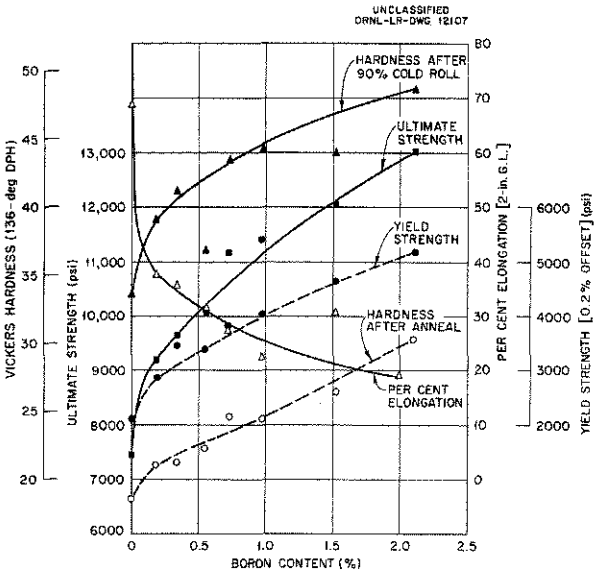

Fig. 16. Mechanical Properties of Al-B Alloys as a Function of Boron Content.

the intermetallic compound AlB_2. In Fig. 18, microscopic inhomogeneity of the boride is observed even after extensive remelting and rolling. Chemical analyses of the boron in the arc-melted alloys, presented in Table 5, further emphasize the marked segregation of the boron.

Corrosion Properties. – The corrosion resistnace of the various boron-bearing aluminum alloys was determined by testing them for 1000 hr in distilled and aerated static water at 60°C, and for 1000 and 2000 hr in distilled and aerated boiling water at 100°C. One group of specimens received approximately 75% cold reduction prior to corrosion testing, while the other group of alloys received a standard fuel-plate heat treatment (5), including flux, stress-relief, and blister anneals, as well as a simulated braze. The results of these tests are presented in Table 8. From these results, the following effects may be summarized:

1. The corrosion rate of the alloys was of the same order of magnitude as that of the type 1100 controls.

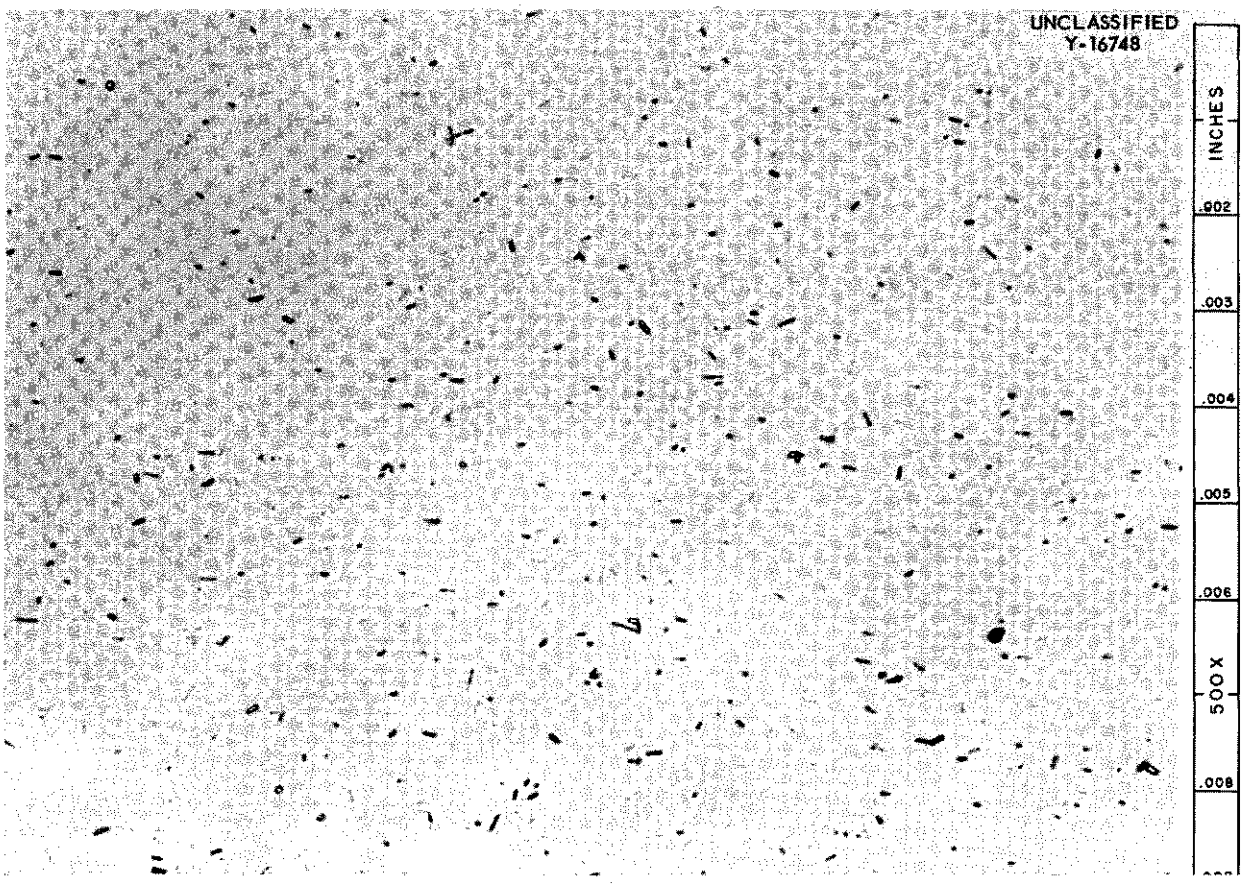

Fig. 17. An Al + 0.18% B Arc-melted Alloy Showing Random Dispersion of AlB_2 in Aluminum Matrix. As polished. 500X.

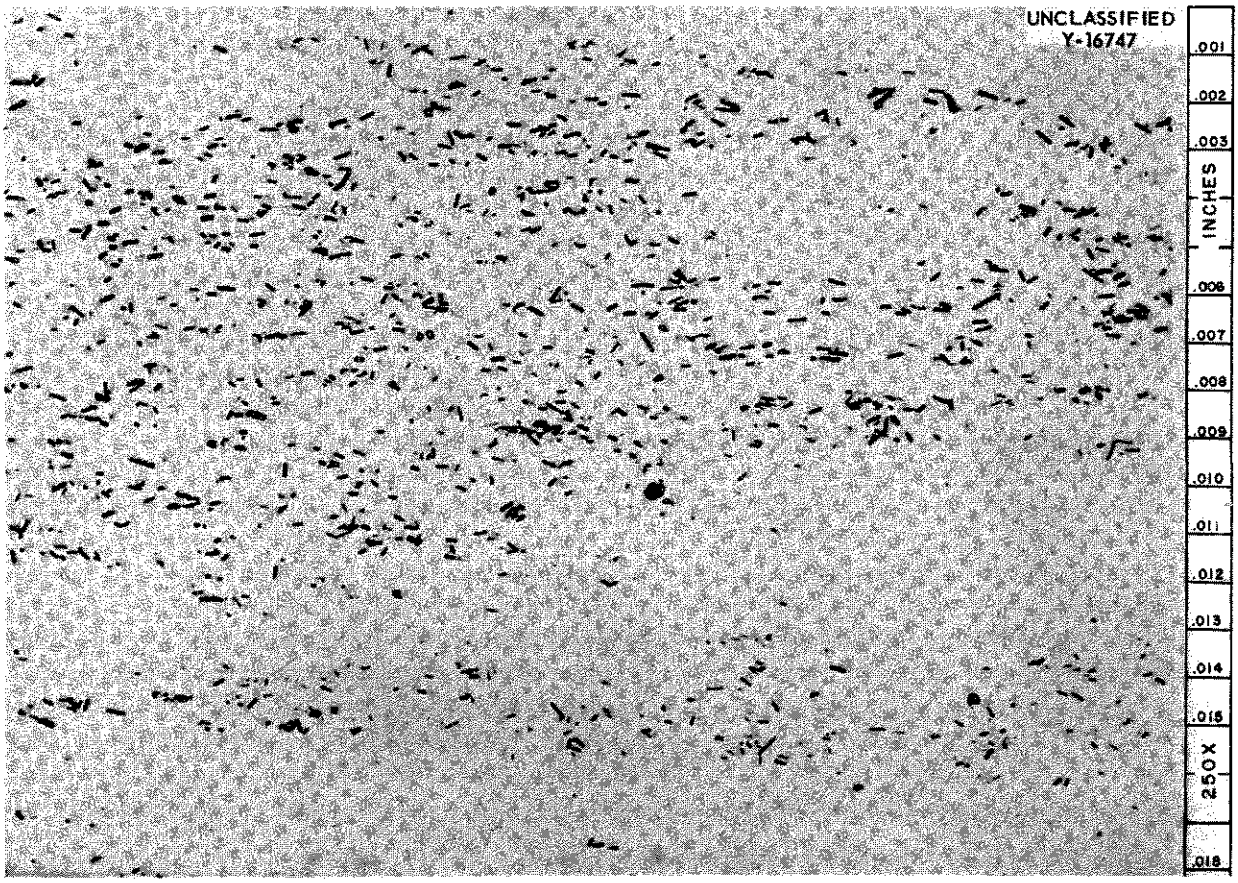

Fig. 18. An Al + 0.55% B Arc-melted Alloy Showing Nonhomogeneous Distribution and AlB_2 in Aluminum Matrix. As polished. 250X.

2. The corrosion rate of the flux-annealed alloy specimens appeared to be slightly greater than that of the cast and cold-rolled specimens.

3. The pitting phenomenon of all boron alloys in 60°C water was comparable with the pitting observed in the type 1100 aluminum control.

4. In 100°C boiling water, the nickel-bearing alloys, whether flux-annealed or not, had pitting which was no more serious than the pitting observed in the type 1100 aluminum control.

5. In 100°C boiling water, the nickel-free boron alloys which had not been flux-annealed exhibited pits of the same order of magnitude as those of the type 1100 aluminum control.

6. In 100°C boiling water, the nickel-free boron alloys which had been flux-annealed exhibited severe pitting, compared with the type 1100 aluminum control.

7. Nickel-free boron alloys, flux-annealed or not, blistered in boiling 100°C water.

8. Nickel-containing boron alloys which had been flux-annealed did not blister in boiling 100°C water. The same alloy without the flux-annealing treatment did blister.

9. Nickel-free and nickel-containing boron alloys which had been flux-annealed did not blister in 60°C water.

Although it is difficult from these data to quantitatively compare the boron alloys with type 1100 aluminum under reactor conditions of irradiation, water purity, temperature, etc., the data indicate that the boron-aluminum nickel-free binary alloys are very marginal for reactor application in which the water may at times be at 100°C and boiling. Blistering of this alloy was pronounced after the corrosion tests at 100°C, even though the material had been previously flux-annealed. In addition, the flux-annealing treatment appears to increase the depth of pitting of such material in boiling 100°C water. On the other hand, the alloys containing nickel appeared to be comparable with type 1100 aluminum in both 60°C and boiling 100°C water.

Table 8. Corrosion Testing of Boron-Containing Alloys in Distilled and Aerated Water

Alloy (wt %)	History	Time of Test (hr)	Corrosion Rate (g/cm^2)	Pitting	Blisters
		Corrosion Testing in Boiling 100°C Water			
0.075% B–bal Al	Cast and cold-rolled	2000	-0.2×10^{-4}	Random, 1–3 mils	Random
0.075% B–bal Al	Cast and cold-rolled	2000	-0.8×10^{-4}	Random, 1–3 mils	Random
0.075% B–0.6% Ni–bal Al	Cast and cold-rolled	2000	-0.2×10^{-4}	Random, 1–3 mils	Random
0.075% B–0.6% Ni–bal Al	Cast and cold-rolled	2000	-0.3×10^{-4}	Random, 1–3 mils	Random
0.078% B–bal Al	Flux-annealed	1000	-3.2×10^{-4}	Significant, 2–20 mils	Random
0.078% B–bal Al	Flux-annealed	1000	-2.8×10^{-4}	Significant, 2–20 mils	Random
0.091% B–0.80% Ni–bal Al	Flux-annealed	1000	-1.4×10^{-4}	Negligible, 1–2 mils	None
0.091% B–0.80% Ni–bal Al	Flux-annealed	1000	-1.4×10^{-4}	Negligible, 1–2 mils	None
0.116% B–bal Al	Cast and cold-rolled	2000	-0.4×10^{-4}	Random, 1–3 mils	Random
0.116% B–bal Al	Cast and cold-rolled	2000	-0.7×10^{-4}	Random, 1–3 mils	Random
0.156% B–0.75% Ni–bal Al	Cast and cold-rolled	2000	-0.9×10^{-4}	Random, 1–3 mils	Random
0.156% B–0.75% Ni–bal Al	Cast and cold-rolled	2000	-0.5×10^{-4}	Random, 1–3 mils	Random
Type 1100 Al control	As-received cold-rolled	2000	$<0.02 \times 10^{-4}$	Negligible, 1–2 mils	None
		Corrosion Testing in 60°C Water			
0.078% B–bal Al	Flux-annealed	1000	-7.1×10^{-4}	Significant, 4–10 mils	None
0.078% B–bal Al	Flux-annealed	1000	-8.0×10^{-4}	Significant, 4–10 mils	None
0.091% B–0.80% Ni–bal Al	Flux-annealed	1000	-5.0×10^{-4}	Significant, 3–9 mils	None
0.091% B–0.80% Ni–bal Al	Flux-annealed	1000	-12.2×10^{-4}	Significant, 3–9 mils	None
Type 1100 Al control	As-received cold-rolled	1000	-1.8×10^{-4}	Significant, 2–7 mils	None
Type 1100 Al control	As-received cold-rolled	1000	-1.5×10^{-4}	Significant, 2–7 mils	None

APPENDIX

Spectrographic Analysis of Uranium Melting Stock

Al	1.3×10^{-3} wt %
B	3.8×10^{-4} wt %
Ca	2.7×10^{-4} wt %
Cr	5.3×10^{-4} wt %
Cu	3.7×10^{-3} wt %
Fe	7.3×10^{-3} wt %
Mg	4.6×10^{-3} wt %
Ni	6.3×10^{-3} wt %
Si	1.4×10^{-4} wt %

Vendor's Analyses of AlB_{12} (−40 +200 Mesh)

B	79.10 wt %*
Al	17.28 wt %
C	0.14 wt %
Fe	0.56 wt %
Al_2O_3	2.83 wt %

*ORNL analysis – 81.4 wt % B

Explanation of Tables 3, 4, and 5

In an effort to evaluate each casting semiquantitatively, a measure of boron segregation, boron recovery, and an over-all casting rating have been established. The recovery is defined by the following ratio:

$$\text{recovery} = \frac{\text{mean analyzed boron content}}{\text{intended boron content}} \times 100 .$$

The index of homogeneity is another arbitrary value defined as

$$I \text{ of } H = 100 - \frac{\text{average absolute deviation of each determination from mean boron content}}{\text{mean analyzed boron content}} .$$

An unsegregated material would thus have an index of homogeneity of 100. The over-all rating, similarly, is an arbitrary number, combining the recovery and index of homogeneity on the merits of the relative importance of each. It is defined as

$$\text{over-all rating} = \frac{\text{recovery} + 2(I \text{ of } H)}{3} .$$

Any consideration of the resultant ratings should be tempered by the following limitations:

1. Recovery is not important in terms of absolutes, but only as a measure of the consistency of the casting procedure. Thus if a reproducible boron recovery, for example, 70%, could be obtained, recovery would no longer be important in evaluating any given billet.
2. Segregation in the casting may lead to misleading recovery figures.
3. The index of homogeneity reveals no segregation trends. Therefore a casting having marked *bottom-to-top boron segregation might have the* same index of homogeneity as one having only random segregation.

Even in view of these limitations, this rating system does give a concrete scale of comparison for the multitude of castings prepared during this investigation.

Analysis of Boron in Aluminum Alloys

The limiting feature in the evaluation of all castings produced was the difficulty encountered in analyzing for boron in aluminum and uranium-aluminum alloys. In this present work, both spectrographic analyses and potentiometric titrations were employed. The majority of the analyses were performed by the potentiometric method, which involved titrating the end point of the reaction between a boron-manitol complex and NaOH. This analytical procedure will be described in the *ORNL Master Analytical Manual* as method 100701 and method 1211220. Analyses of control samples revealed that this method was accurate to within ±7% of the true boron content, while analytical precision between duplicate determinations was ±1%.

ACKNOWLEDGMENT

The authors wish to thank J. B. Flynn and G. E. Angel, who prepared the alloys; J. H. Erwin and H. J. Wallace, who handled fabrication; J. H. Edgerton, who performed chemical analyses; C. K. H. DuBose, who performed metallography; J. L. English, who performed corrosion tests; and J. Woods, who determined mechanical properties of the alloys.

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