312.1

Buffalo Tube Works

GENERAL ELECTRIC COMPANY
SCHENECTADY, N. Y., U. S. A.

DATA FOLDER No. 86906

Title Boron Analysis

By

Tube Engineering Div.

Information prepared for Electronic Tube Engineering Div.

Tests made by

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Date July 15, 1946

DF4

Boron Analysis

I Summary:-

Variations from lot to lot of our Laboratory Boron as analyzed by chlorination methods, were small. Comparisons with boron from other companies showed some anticipated differences.

While boron is not the only possible source of variation in ignitor point manufacture, it was felt that differences in its chemical composition would be significant. The chemical tests show more uniformity in the boron than we expected from a study of the results of production lots of the p points made with the Laboratory Boron.

Microscopic examination of the incoming boron should be made - but chemical tests at this time could be used only to accumulate data which would take a long time to correlate into a form for a useful specification. Microscopic examination for these indicate boron contains more glass now than it should. Microphotos of boron and of aluminum borides are included.

II Procedure:-

Fourteen samples of boron were tested by Miss Shirley Betar under the guidance of Dr. E. H. Winslow in Dr. H. A. Liebhafsky's laboratory of our Research Laboratories. The method was that of the "Rapid Chlorination" given in "Crude Boron, Analysis and Composition" by Earl H. Winslow and Herman A. Liebhafsky (JACS 64, 2725 (1942)).

III Results:-

Sample	% Residue	% Sublinated Fe	% Sublinated Al	%Boron
1. Batch Lab. Boron	29.7	2.02	--	69
2. Macky Boron	92	.4	--	7.6
3. Lot 17 Lab. Boron	29.7	1.04	--	70
4. Berk Boron	36.7	.35	--	63
	36.8	--	.7	62.5
5. Lab. Boron 12-16-44	20.9	1.84	--	77.3
6. Lab. Boron 2A	26.4	1.52	--	72.1
7. Lab. Boron 2B	28.5	1.	--	70.5
8. Lab. Boron 2C	26.8	1.3	--	72
9. Westinghouse #1	7.4	1.48	--	91.2
10. Westinghouse #2	9	1.56	--	89.5
	9.3	--	9.4	81.3
11. Westinghouse #3	11.2	1.2	--	87.6
12. Lab. Boron 2D	30.9	1.56	--	67.7
	29.5	--	7.6	62.9
13. Lab. Boron 2F	34.6	2.08		63.4

Samples (con't.)	% Residue	% Sublinated Fe	% Sublinated Al	% Boron
	47.9	2.44	--	50.7
14. Lab. Boron 2E	29	2.3	--	6

The spectrographic analysis of the residues (Works Lab. #66472) reported by Mrs. O'Hara are as follows:

	1	2	3	4	5	6	7	8	9	10
Cu	low	low	nil	tr	low	low	pres	pres	low	low
Fe										
Se	pres	tr	pres	pres	pres	pres	pres	pres	pres	pres
Na	tr	nil	tr	nil	tr	nil	nil	tr	nil	nil
Min	low	tr	low	pres	pres	pres	pres	pres	pres	tr
Ca	pres	tr	pres	tr	low	pres	low	pres	tr	nil
Sr	tr	nil	tr	nil	tr	tr	tr	tr	nil	nil
Hg	tr									
Ni	tr	tr	tr	tr	low	low	low	low	low	nil

	11	12	13	14
Cu	tr	pres	low	pres
Fe	tr	tr	tr	low
Se	pres			
Na	nil	nil	nil	low
Min	tr	low	low	low
Ca	tr	tr	pres	pres
Sr	nil	nil	tr	tr
Hg	tr			
Ni	nil	low	low	pres

IV Discussion -:

Large experimental error was probably present in some individual samples. However, it can be stated that the average samples of Laboratory Boron now received are about 60 to 65% free pure boron. It should further be stated that the lots 2A through 2F of Lab. Boron were all of the same melt (made by S-12536-9) with the letters indicating the chronological order of leaching and drying the lots of boron (by S-12546-10).

In a paper "The Preparation of Pure Boron Metal" (April 9, 1942) Mr. A. C. Cooley of our Research Laboratory states in review of chemical analysis of material prepared by Mr. Nickle:

"The results indicate that the total boron content is somewhere between 70 and 80%."

Later, in the same report, Mr. Cooley mentions that some material made by Mr. Nickle and found satisfactory in ignitors was found to be 72% boron.

These statements indicate that the boron content is lower now than it was then in our Laboratory Boron.

In addition to the chemical analyses indicated above we have made some microscopic examination of boron received from different lots. Examination was made at 100 X to 210 X using transmitted polarized light to detect the glass left in the boron after washing (S-12536-10). These microscopic examinations have shown that the minute particles of glass which may sometimes be noted by a gritty feeling that they impart to the boron, and sometimes by microscopic examination, are more numerous in later lots of our Lab. Boron. Apparently this condition has become progressively worse over the past two years.

Our lots of Laboratory Boron suffer by microscopic examination comparisons with those of Westinghouse just about as badly as by the chemical analyses. This is true despite the fact that Westinghouse made its boron according to instructions furnished by us -- and now has discontinued even using that -- having changed to Berk Boron, which also uses glass for production.

Some "hyperpure" boron has been received from the DuPont Company Research Department. The amount of this boron was too small to conduct conclusive tests in ignitor points. However, some attempts at sintering points were made; and the indication is that the resultant ignitors are very brittle. This boron is reported to be better than 99% pure by DuPont, while a letter from Westinghouse giving results of a test they made on a sample of this boron shows:

Soluble Boron (in HNO_3)	92.7%
Insoluble Boron (probably BN)	1.13%
Silica	2.06%
Al_2O_3 and Fe_2O_3	1.02 (very little Fe_2O_3)
Copper	Trace
Magnesium	None

The lot 3A, made from the melt following the "2" series has almost 30% glass by apparent volumes. This lot has not been tested by chlorination. Microscopic revelation of this excess glass indicates it may be bad.

A list of mixes made with some of the various lots of boron tested as follows. Tests of points made from these mixes have been accumulated by Mr. E. W. Scott and results are available from him in the Production Dept. of Electronic Tube Division.

Boron	Mixes
Laboratory Batch #1	111, 112, 114, 115, 115-1
Laboratory Lot #17	EWS 9/30/42, EWS 10/1/42, EWS 10/15/42 EWS 12/13/42
Laboratory 2A	116, 116M, 117, 117-1, 117-2, 117-6
Laboratory 2B	127, 125, 120, 122, 123, 124, 117-4 117-5
Laboratory 2C	126, 117-7, 117-8, 117-9
Westinghouse #2	166, 166A
Laboratory 2D	117-10, 117-11, 128, 129, 130, 131
Laboratory 2F	135B, 139, 163B
Laboratory 2E	163A, 163, 161, 147A, 163C

Unfortunately samples of older lots of boron were not available for test and comparison.

Pictures of G. E. Laboratory 2E, Westinghouse #1, and DuPont Hyperpure taken by the Metallurgical Section of Research Laboratories show the glass (in the first two) visible by transmitted polorized light.

Pictures of aluminum borides, materials with about the same solubility as boron, are included. These materials appear in small amounts in various mixes. They are laminar plates with an orange-red color by transmitted light and a metallic appearance by reflected light.

Attempts to separate the last of the glass from the boron by careful selective settling and attempts to remove it by using the Bldg. 269 antoclave (work by J. Easthan) for the leaching were unsuccessful.

Conclusions:

The chemical tests indicate the boron used in ignitor point manufacture is not of as high a purity as formerly. It might be possible to increase its purity by refinements of the present method of manufacture by a few percent, but this change would probably be insignificant.

However, to ascertain the advantages of using pure boron in ignitor manufacture, the following steps are being taken:

1. To obtain tests of pure boron in ignitor points, arrangements are being made to obtain small quantities of the material from Research Laboratory. This boron is manufactured by an entirely different process.

2. More easily available boron hydride will also be tried, with the intentions of removing the chemically bound hydrogen of the compound in the sintering of the ignitor.

The results of these tests will be available from Mr. E. W. Scott when he has completed them, after receipt of the materials. These tests should influence future ignitor manufacture.

Raymond E. Roth

R. E. Roth
7-12-46

Countersigned by:
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July 15, 1946

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Section Leaders
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CC Kirchenbauer - #269 Fact.

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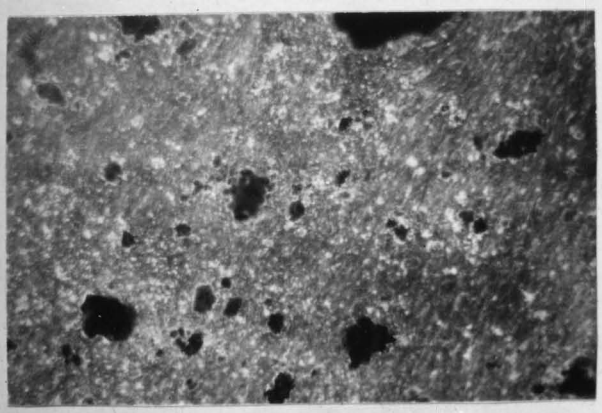
SCHENECTADY, N. Y.

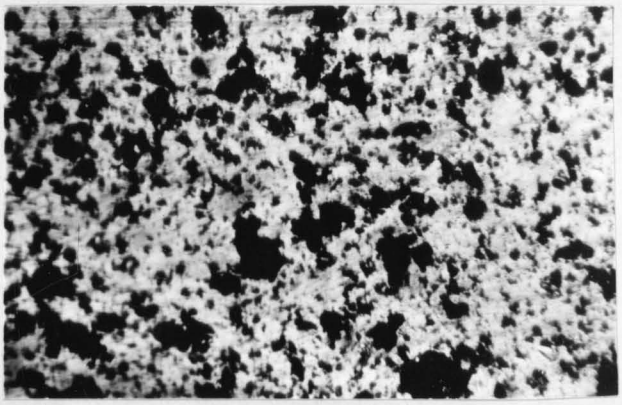
Metallographic Section

GLYCERIN SUSPENSIONS OF BORON POWDERS

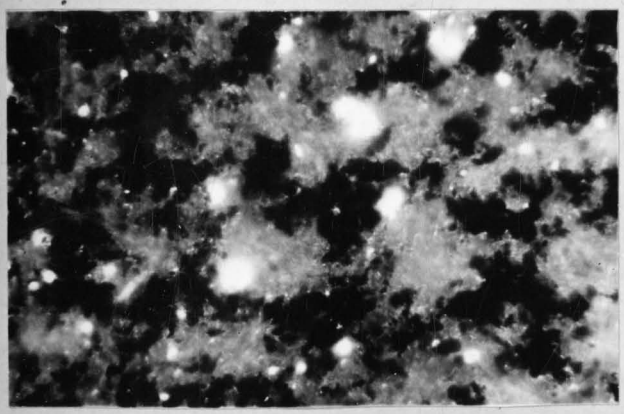
x115



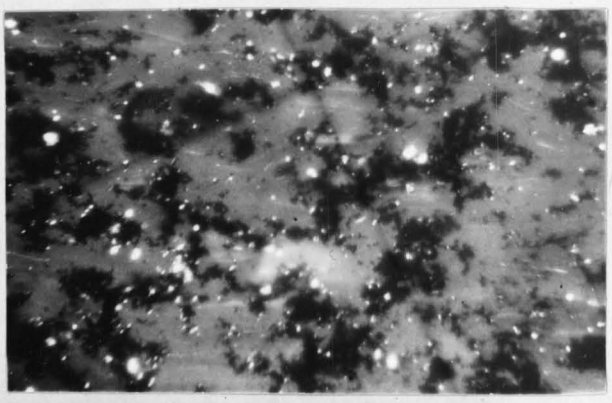
DuPont



Westinghouse



General Electric



Berk Co.

Plate 2341

RESEARCH LABORATORY
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SCHENECTADY, N. Y.

Metallographic Section

ALUMINUM BORIDE

x90

Plate 2798

RESEARCH LABORATORY
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Metallographic Section

ALUMINUM BORIDE

x15

Plate 2398