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AMERICAN ELECTRO METAL CORPORATION

YONKERS, NEW YORK

SUMMARY
PROGRESS REPORT

(Physical Properties)

Contract N6-ONR-256/1

Cemented Borides

May 1, 1953 - July 31, 1954

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Contract N6-ONR-256

CEMENTED BORIDES

(Physical Properties)

Progress Made from May 1, 1953 to July 31, 1954

Work Done By:

Frank W. Glaser

Michael J. Ford

David Moskowitz

William Ivanick

Norman Grossman

Philip Karp - Part Time

Kasimir Langrod - Part Time

William Styles - Part Time

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I. INTRODUCTION

During the period covered by this Summary Progress Report, the main emphasis of research work was put on the development of new high temperature materials having a chromium-boron base. In the previous Summary Report we briefly reported on some rather encouraging results in this connection and an attempt was made during the past report period to establish metallurgical characteristics, processing procedures and the physical properties for these new materials; the results reported below represent a summary of physical properties for the BOROLITE III (300 series) and BOROLITE IV (400 series) compositions investigated to date.

During the initial report period all samples were produced by hot pressing using direct conduction heating. However, it became quite obvious that control was rather poor and that from a commercial point of view greater reproducibility would be insured through the use of standard cold pressing and sintering techniques. Since the sintering techniques were yet to be established, a certain delay in research work as such occurred. It proved rather difficult to locate a furnace that would readily maintain sintering temperatures at 1500° and 1600° C and still have a pure and dry protective atmosphere. After these experimental difficulties had been overcome and new methods for part production developed, initial test samples were submitted to companies such as the General Electric Company and testing activities such as the Naval Research Laboratory in Washington, D. C. and the National Bureau of Standards in Washington, D. C. Results generally showed that BOROLITE III (300 series) had excellent stress to rupture properties.

During the past report period, a new alloy for use at high temperature was also worked on. The trade name "BOROLITE IV" (400 series) was

assigned to this alloy which is composed substantially of chromium, molybdenum and boron. Initial results on BOROLITE IV show excellent high temperature properties. In most instances, BOROLITE IV has shown superiority to BOROLITE III alloys which may have to be eliminated in the light of BOROLITE IV performance.

Previous Summary Reports also dealt with BOROLITE I Zirconium Boride. The task order for the period covering 1953 to 1954 called for elimination of BOROLITE I from the research program unless special test samples were requested by government agencies. In view of this, very little work was done in connection with BOROLITE I and all activities were limited to some part making and nozzle testing as carried out in connection with government requests.

A solid solution study, both of the diborides of the transition metals as well as of monoborides and certain other phases of the transition metal borides came to an end. A good understanding of the structural characteristics underlying solid solution formation of diborides was acquired. The Appendix includes a publication which appeared in Acta Metallurgica last January. The physical properties of pure transition metal diborides, being of extreme interest from a fundamental point of view, were also studied and the results of this study will be reported in a section of a forthcoming letter report.

In summarizing the work done during the last report period, it can be stated that a new high temperature material has been developed making use of the elements chromium, molybdenum and boron. This new high temperature material, designated as BOROLITE IV, appears to outperform in terms of heat shock resistance, resistance to oxidation and stress to rupture strength most

of the now known cermets commercially available. However, in terms of resistance to impact, BOROLITE IV still has to be improved and it will be the main effort of this forthcoming year to impart the necessary impact strength and a certain amount of ductility to BOROLITE IV to make it a very attractive and practical high temperature material. Considerable progress has already been made towards achievement of satisfactory impact strength through the use of new fabrication techniques and compositions. It can be expected that work along these lines now in progress can be completed during this project period.

II. DENSITY OF BOROLITE 300 AND 400 SERIES

The immersion densities of various fully-sintered BOROLITE compositions are given in Table I. For the 300 series the density ranges from 6.20 to 6.86 g/cc, whereas the density of the 400 series is somewhat greater, going from 6.77 to 7.31 g/cc.

TABLE I

DENSITY OF BOROLITE 300 AND 400 SERIES

300 Series		400 Series	
Composition	Density (g/cc)	Composition	Density (g/cc)
301	6.20	401	7.31
301 a	6.28	402	7.13
301 b	6.36	403	6.88
301 c	6.44	404	6.77
301 d	6.53		
302	6.60		
303	6.69		
304 a	6.78		
305 a	6.86		

III. MELTING POINTS OF VARIOUS BOROLITE COMPOSITIONS

Most of the BOROLITE compositions do not have true melting points. Rather, they liquefy over a range of temperature beginning with the first appearance of liquid (solidus) and ending with the temperature at which there is total liquid (liquidus).

Table II lists the approximate temperatures at which liquid first appears for some Borolite compositions. Most of them, with the exception of 301, melt in the region from 1500° to 1575° C.

TABLE II

APPROXIMATE MELTING POINTS OF BOROLITE 300 AND 400 SERIES COMPOSITIONS

300 Series		400 Series	
Composition	Melting Pt (°C)	Composition	Melting Pt (°C)
301	1760	401	1525
302	1560	402	1500
304 a	1500	403	1525
305 a	1520	404	1575

IV. RESISTIVITIES OF BOROLITE 300 AND 400 COMPOSITIONS

The electrical resistivity of BOROLITES 300 and 400 are shown in Table III. They are all good conductors, their resistivities extending from about 27 to 78 microhm-cm at room temperature. In addition, they all have positive temperature coefficients of resistivity.

TABLE III

ELECTRICAL RESISTIVITY OF VARIOUS BOROLITE COMPOSITIONS

300 Series		400 Series	
Composition	Resistivity (microhm-cm)	Composition	Resistivity (microhm-cm)
301	78	401	27
301 a	72	402	34
301 c	60	403	47
302	43	404	54
303	40		
304 a	33		
305 a	28		

V. THERMAL CONDUCTIVITY OF BOROLITE 300 SERIES

Measurements of thermal conductivity of four different BOROLITE 300 series compositions, made at 20° C and at 100° C, are given in Table IV. As the data show, all of them are good conductors of heat.

TABLE IV

THERMAL CONDUCTIVITY OF BOROLITE 300 SERIES

(cal/cm/sec/°C)

Composition	20° C	100° C
301 d	.034	.0375
302	.045	.049
303	.061	.0635
304 a	.074	.0805

VI. ROCKWELL "A" HARDNESS OF BOROLITES

Table V shows the hardness of BOROLITE 300 and 400 series compositions, as measured on the Rockwell "A" scale. These values range from 60 to 88 for the 300 series and from 78 to 88 for the 400 series.

TABLE V

ROCKWELL "A" HARDNESS OF BOROLITE COMPOSITIONS

300 Series		400 Series	
Composition	R_A	Composition	R_A
301	86	401	78
301 a	87	402	81
301 b	88	403	86
301 c	88	404	88
301 d	86		
302	82		
303	77		
304 a	68		
305 a	60		

VII. SPECIFIC HEAT OF BOROLITE 300 SERIES AT ELEVATED TEMPERATURES

The results of measurements of specific heat of various BOROLITE compositions at elevated temperatures are listed in Table VI. These were made in the usual manner, by measuring the temperature of a know weight of water contained in a calorimeter before and after immersing the heated specimens.

TABLE VI

SPECIFIC HEAT OF BOROLITES AT ELEVATED TEMPERATURES

300 Series			400 Series	
Composition	Cp 500°	Cp 1000°	Composition	Cp 1000°
301	--	.1701	401	.1287
301 a	--	.1734	402	.1380
301 d	.1515	.1671	403	.1418
302	--	.1434	404	.1507
303	.1438	.1538		
304 a	--	--		
305 a	--	.1426		

VIII. MODULUS OF ELASTICITY MEASUREMENTS AT ROOM TEMPERATURE AND AT 1000° C

Figure 1 shows a plot of the results of stress-strain tests on two different BOROLITE 300 compositions at room temperature. Young's modulus of elasticity has been calculated both from these measurements and from values obtained by the simple beam method. The latter values, which are given in Table VII, tend to be somewhat larger than those calculated from stress-strain data.

TABLE VII

ELASTIC MODULI OF BOROLITE 300 COMPOSITIONS AT ROOM TEMPERATURE AND 1000° C

(Simple Beam Method)

Composition	Modulus of Elasticity (psi) Room Temp.	Modulus of Elasticity (psi) 1000°C
304 a	43,100,000	31,400,000
302	38,200,000	27,400,000
301 d	38,300,000	27,500,000

Stress-Strain Curves Of Borolite 300 Series At Room Temperature

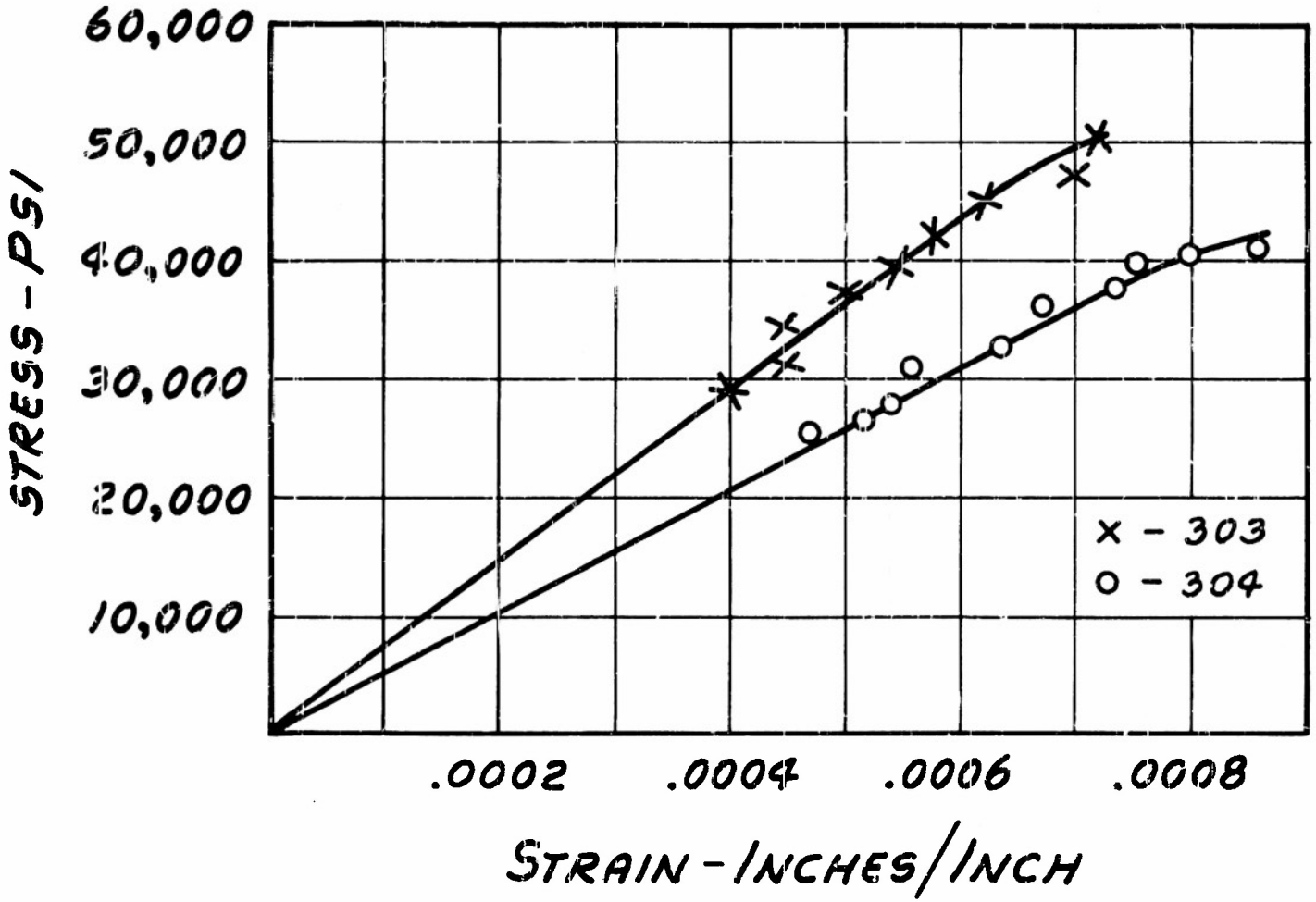

FIG. 1

IX. TRANSVERSE RUPTURE STRENGTH OF BOROLITE 300 AND 400 COMPOSITIONS AT 1800° F

Table VIII gives the transverse rupture strength of various of these materials as measured at 1800° F. They range from 80,000 to 125,000 psi for the 300 series and from 88,000 to 160,000 psi for the 400 series.

TABLE VIII

TRANSVERSE RUPTURE STRENGTH AT 1800°F OF BOROLITE 300 AND 400 SERIES

300 Series		400 Series	
Composition	T. R. Strength	Composition	T. R. Strength
301	80,000	401	160,000
301 a	82,000	402	111,000
301 b	85,000	403	88,000
301 c	85,000	404	108,000
301 d	88,000		
302	100,000		
303	125,000		
304 a	125,000		
305 a	105,000		

X. STRESS TO RUPTURE PROPERTIES OF BOROLITE 300 AND 400 SERIES

A composite curve of stress versus rupture life at 1800° F for a group of BOROLITE 300 compositions is shown in Figure 2. All of them, with the sole exception of BOROLITE 305 a, have 100-hour lives equal to or greater than 10,000 psi.

A similar plot for the BOROLITE 400 series is given in Figure 3. These compositions, with the exception of BOROLITE 401, have still greater stress-rupture strengths, being in excess of 10,000 psi for 1000-hour lives.

Table IX summarizes these stress-rupture values, as measured at 1800° F.

TABLE IX

STRESS-RUPTURE STRENGTH OF BOROLITE 300 AND 400 SERIES AT 1800°F

Composition	100 Hr. Life (psi)	1000 Hr. Life (psi)
301 a	18,500	13,000
301 c	12,000	—
301 d	15,000	11,000
302 s	18,000	13,000
302	13,500	—
303	12,500	—
304 a	10,000	—
305 a	7,600	—
401	14,000	7,700
401 s	24,000	19,000
402	16,000	11,500
403	17,500	12,500

STRESS TO RUPTURE STRENGTH AT 1800°F. OF BOROLITE 300 SERIES

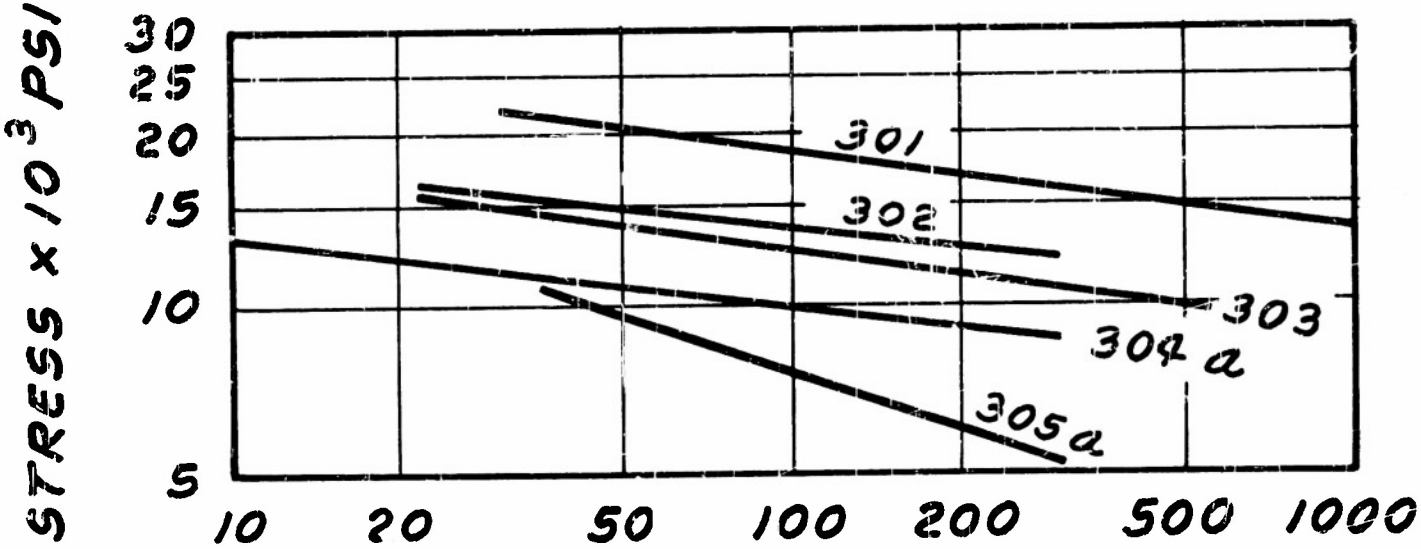

FIG. 2

STRESS-RUPTURE STRENGTH AT 1800°F. OF VARIOUS BOROLITE 400 SERIES

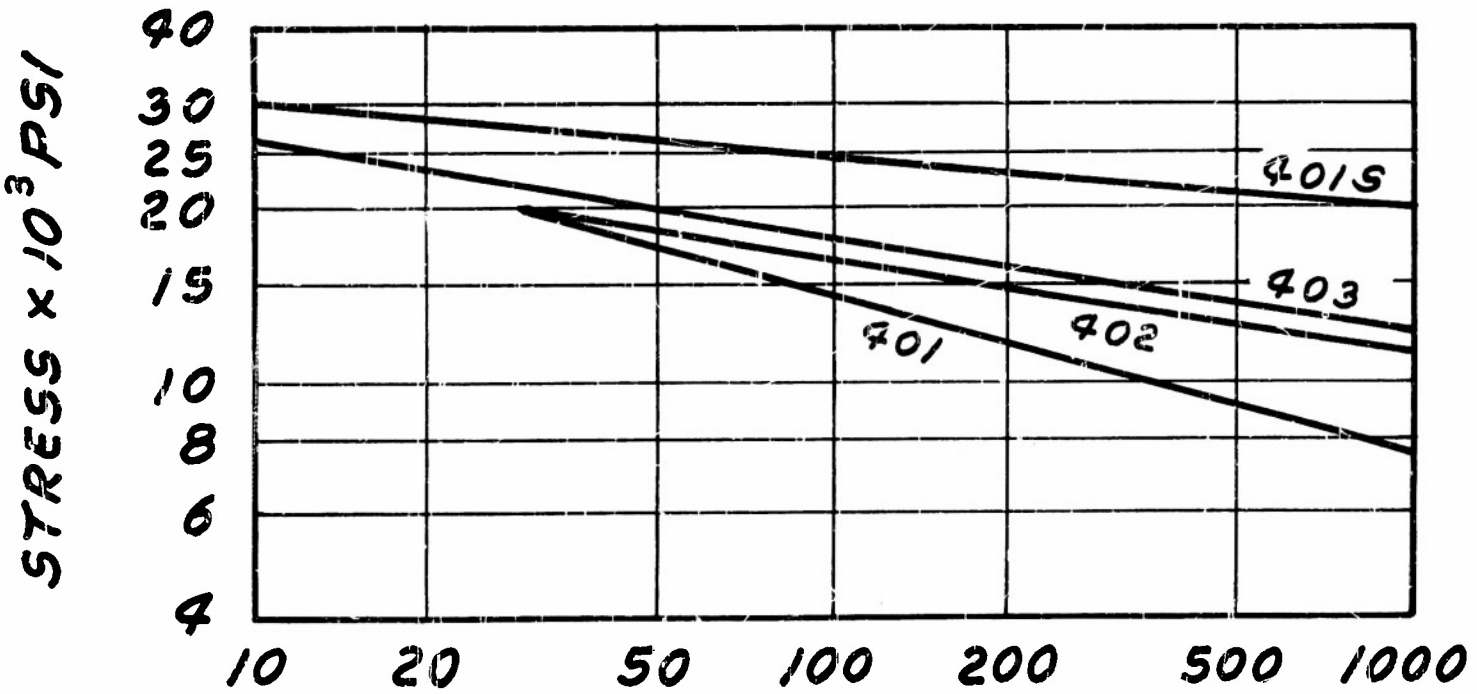

FIG. 3

Two BOROLITE compositions, 302 s and 401 s, have also been tested in stress-rupture at 2000° F. Figure 4 shows a plot of the results of these tests, comparing the curves obtained at 1800° F with the 2000° F curves. Table X compares the stress-rupture strengths of these compositions at 1800° F and 2000° F for 100-hour and 1000-hour lives.

TABLE X

COMPARISON OF STRESS-RUPTURE STRENGTHS OF TWO BOROLITE COMPOSITIONS AT 1800°F AND 2000°F

Composition	1800°F		2000°F	
	100 Hr. Life	1000 Hr. Life	100 Hr. Life	1000 Hr. Life
302 s	18,000 psi	13,000 psi	3,500 psi	2,500 psi
401 s	24,000 psi	19,000 psi	15,000 psi	12,000 psi

Comparison Of Stress-Rupture Strengths Of Two Borolite Compositions At 1800°F. & 2000°F.

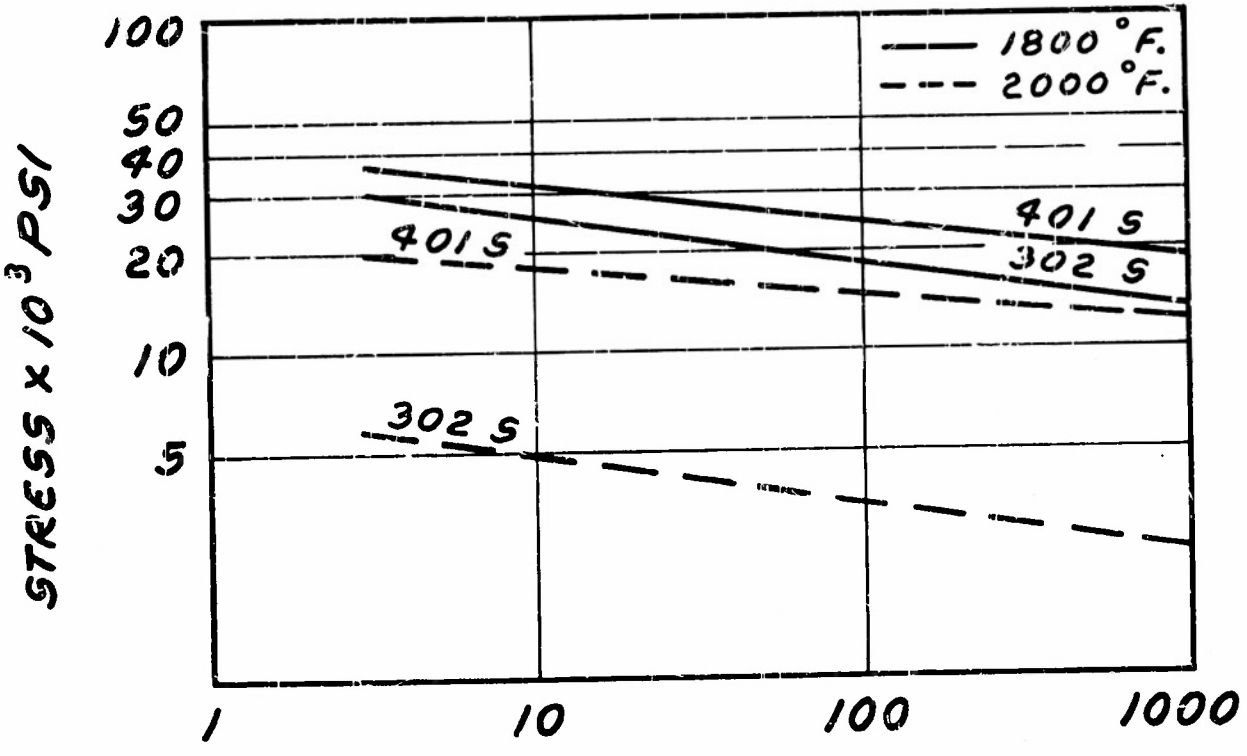

FIG. 4

XI. ADDITIONAL PHYSICAL PROPERTIES

The above materials have been tested for various other high temperature properties in addition to the ones listed above. Thermal expansion measurements in the range from room temperature to 1000° C give coefficients for both series of BOROLITES of between 9×10^{-6} and 10×10^{-6} in./in., a value somewhat smaller than that of most metals. Insofar as heat shock is concerned, the BOROLITE 400 series compositions meet the NACA specifications of 100 cycles in the standard NACA test. The 300 series compositions are not as successful, with thermal shock failures sometimes taking place prior to completion of the full 100 cycles.

The oxidation performance of these materials in stagnant air at 1000° C is very good. Most of the compositions have been able to successfully withstand oxidation tests for over 1000 hours at this temperature and even at 1100° C with little weight gain and slight growth in oxide coat.

The above BOROLITE compositions show poor performance in one property, namely impact resistance. Tests on BOROLITE 300 and 400 materials at room temperature yield impact resistances of 2 ft. lbs. or less in the Charpy test and less than one inch-lb. in the standard NACA test. Thus a research program designed to improve this particular property without harming other properties is now being followed.

XII. SUMMARY

The physical properties of some new materials for use at and above 1800° F have been described. Continuing research in connection with both the 300 and 400 series will probably tend to somewhat modify (increase) their strength characteristics as reported here; it is not expected that density, electrical resistivity, specific heat and modulus of elasticity will be materially affected.

Emphasis of future research activities will be placed on improvement of resistance to impact of the materials of the 400 series.

F. W. Glaser

D. Moskowitz

M. J. Ford

TRANSITION METAL DIBORIDES*

BENJAMIN POST,† FRANK W. GLASER‡ and DAVID MOSKOWITZ‡

Structural characteristics of eight transition metal diborides have been investigated. It was found that in these hexagonal compounds the length of the "a" axis is determined primarily by boron-boron contacts in the case of diborides of the smaller metal atoms, whereas in the diborides of the larger metal atoms, the metal atoms are the determining factc Enlarged metal atoms were postulated to explain variations in lattice dimensions and "c/a" ratios. An examination of the melting points of the diborides relative to those of their respective metals indicated that they reflect primarily the strength of the Me-B bonds in these structures. The extent of mutual solid solubility appeared to depend mainly upon size factor considerations. In cases of solid solution between two diborides where one of these was of a more highly ordered structure than the other, the more disordered phase was favored.

LES BIBORURES DES MÉTAUX DE TRANSITION

Les biborures de huit métaux de transition furent examinés afin d'étendre la connaissance de leurs caractéristiques structurales.

On a constaté que la longueur de l'axe "a" de ces composés hexagonaux est déterminée, soit par les contacts bore-bore, soit par les contacts entre les atomes métalliques; le premier cas s'applique aux biborures des métaux à petits atomes, et le deuxième cas aux biborures des métaux à grands atomes. Un accroissement des dimensions des atomes métalliques a été proposé pour expliquer les variations dans les dimensions des réseaux et dans les rapports "c/a". Un examen des points de fusion des biborures, en comparaison avec les points de fusion de leurs métaux respectifs, a permis de constater que ceux-là reflètent principalement l'intensité des liaisons Me-B dans ces structures. L'étendue de solubilité solide, mutuelle, dépend surtout du facteur de dimensions.

Dans les cas de solution solide entre deux biborures, dont un a une structure plus ordonnée que l'autre, la phase la plus désordonnée prédomine.

DIBORIDE DER ÜBERGANGSMETALLE

Strukturelle Eigenschaften der Diboride von acht Übergangsmetallen wurden untersucht. Es zeigte sich, dass in diesen hexagonalen Verbindungen die Länge der a-Achse in Diboriden mit kleineren Metallatomen in erster Linie von den Bor-Bor Kontakten bestimmt ist, während bei Diboriden mit grösseren Metallatomen die Metallatome die grösse—bestimmenden Faktoren sind. Es wurde eine Vergrösserung der Metallatome postuliert, die die Veränderungen in den Gitterkonstanten und im "c/a" Verhältnis erklären kann. Ein Vergleich der Schmelzpunkte der Diboride und der der entsprechenden Metalle deutet darauf hin, dass der Schmelzpunkt der Diboride in erster Linie die Stabilität der Me-B Bindung reflektiert. Das Ausmass der gegenseitigen festen Löslichkeit scheint hauptsächlich vom jeweiligen Raumbedarf abzuhängen. In festen Lösungen von zwei Diboriden, von denen eine Verbindung einen höheren Ordnungsgrad als die andere aufwies, wurde die Phase geringeren Ordnungsgrades bevorzugt.

Introduction

Isomorphous diborides of eight transition metals (Ti [1], Zr [2], Hf [3], V [4], Nb [5], Ta [6], Cr [7] and Mo [8a,b]), have been described in the extensive literature of metallic borides. Most of these, as well as the closely related Mo_2B_5 and W_2B_5 compounds, have been described by Kiessling in a comprehensive review article [9].

In this paper some structural and physical properties of these borides and their solid solutions will be discussed.

The crystal structures of the diborides are simple. They are of the C-32 type. The primitive hexagonal unit cell contains one formula weight of MeB_2. The space group is D'_{6h}–C 6/mmm, with the metal atom at 0,0,0, and boron atoms at ⅓, ⅔, ½ and ⅔, ⅓, ½. As shown in Figure 1, the metal and boron atoms lie in alternate planar layers. Each metal atom has six equidistant closest metal neighbors in its plane, and twelve equidistant boron neighbors, six in the layer above and six in the layer below the metal atom. Correspondingly, each boron atom has three closest boron neighbors in its plane, and also forms six boron-to-metal bonds.

Kiessling [10] has determined the crystal structures of the closely related Mo_2B_5 and W_2B_5. These resemble ordered modifications of the MeB_2 structure and are discussed below.

A diboride of uranium, apparently isomorphous with those listed above, has recently been prepared [11]; it is not included in this discussion, which is limited to diborides of transition metals of the first, second and third long periods.

1. Structural Considerations

1. *Lattice Dimensions*

Lattice constants of the isomorphous diborides are listed in Table I in order of increasing length of the "a" axis. Boron-to-boron distances are also listed.

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†Polytechnic Institute of Brooklyn, Brooklyn, New York.
‡American Electro Metal Corporation, Yonkers, New York.

Studies of a number of compounds in which boron-boron bonds exist indicate that the "normal" boron radius is 0.87 Å i.e., the "normal" boron-boron separation is 1.74 Å [9]. In the MeB_2 structure, the length of the "a" axis is $\sqrt{3}$ times the boron-boron separation. In the borides of the smaller metal atoms (e.g., Cr and V)the length of the "a" axis is determined primarily by boron-boron contacts. The length of the "a" axis increases with increasing size of the metal atoms. Thus, in ZrB_2, the boron-boron separation, which must equal $a/\sqrt{3}$, is 1.83 Å, or 0.09 Å in excess of the "normal" value. Evidently, in such borides, the metal atoms are in "contact" and determine the length of the "a" axis.

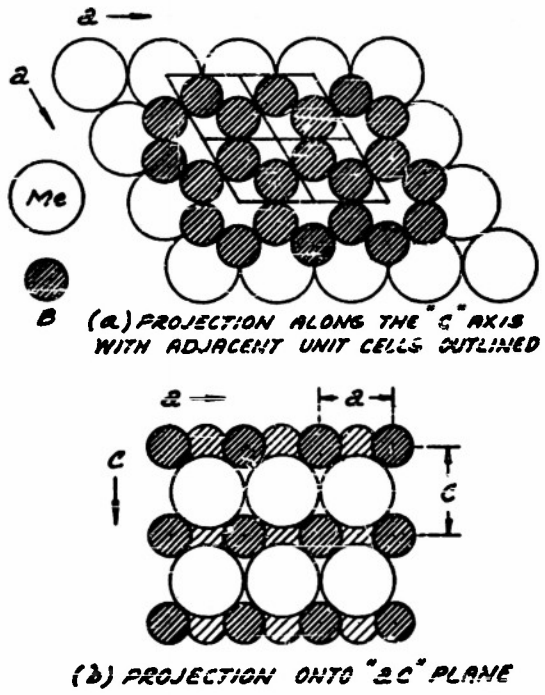

FIGURE 1. Arrangement of metal (Me) atoms and Boron (B) atoms in MeB_2.

These elementary considerations of atomic size indicate that, regardless of other factors, it is doubtful whether metal atoms much smaller than Cr, or much larger than Zr, can form diborides of the type discussed. The excessive separation of metal atoms in one case, and of boron atoms in the other, would undoubtedly lead to structural instability.

2. "c/a" Ratios and Me-B Bond Lengths

Values of the "c/a" ratios are listed in Table I. Norton *et al* [4] and Kiessling [9] have noted the fairly regular increase in this ratio which accompanies increasing size of the metal atom. It is of some interest to investigate this effect more closely.

Values of interatomic distances in the pure metals and in their diborides are listed in Table II. It will be noted that in all cases the "observed" Me-B bond length exceeds the corresponding calculated value by 0.07 ± .01 Å. This Me-B separation is related to the axial lengths in the following way:

$$d_{(Me-B)} = \left(\frac{a^2}{3} + \frac{c^2}{4}\right)^{\frac{1}{2}} \qquad (1)$$

TABLE I

UNIT CELL DIMENSIONS OF METAL DIBORIDES (IN Å)

	"a"	"c"	c/a	(B–B)
CrB_2 [7]	2.97	3.07	1.03	1.72
VB_2 [4]	3.00	3.06	1.02	1.73
TiB_2 [1]	3.03	3.23	1.07	1.75
MoB_2 [8a]*	3.05	3.08	1.01	1.75
TaB_2 [6]	3.08	3.27	1.06	1.78
NbB_2 [5]	3.09	3.30	1.07	1.78
HfB_2 [3]	3.14	3.47	1.10	1.81
ZrB_2 [2]	3.17	3.53	1.11	1.83

*Bertaut and Blum [8b] have reported a = 3.05 Å and c = 3.11 Å for MoB_2.

TABLE II

INTERATOMIC DISTANCES IN METALS AND METAL DIBORIDES (IN Å)

	(1)	(2)	(3)	(4)	(5)
Metal	r(Me) in Metal C.N. in brackets	r(Me) for C.N.12*	r(Me) (C.N.12) plus r(B) r(B) = 0.87 Å	r(Me–B) obs. in MeB_2	(4) minus (3)
Ti	1.45 (12)	1.45	2.32	2.38	0.06
Zr	1.59 (12)	1.59	2.46	2.54	0.08
Hf	1.57 (12)	1.57	2.44	2.51	0.07
V	1.32 (8)	1.37	2.2	2.31	0.07
Nb	1.43 (8)	1.48	2.35	2.43	0.08
Ta	1.43 (8)	1.48	2.35	2.41	0.06
βCr**	1.36 (12)	1.36	2.23	2.30	0.07
Mo	1.33 (8)	1.41	2.28	2.34	0.06
W	1.37 (8)	1.42	—	—	—

*Where values for C.N. 8 were the only ones available, the values for C.N. 12 were computed using Pauling's equation $R(12) - R(8) = .300 \log n$ [12].

**Values for β Cr have been used somewhat arbitrarily in this Table in place of the more common α Cr. The latter is so small (r = 1.25 Å) that it appears probable that in CrB_2 the Cr atom has a configuration similar to that in β Cr.

The calculated values are simply the sums of the "normal" boron radius and the radii of the metal atoms for twelve-fold coordination.

Evidently, the effective radii of either the metal or boron atoms, or both, are substantially greater than the radii used for these "calculated" values.

Considerable light is shed on this point by a consideration of the variation of the lattice constants of NbB_2 and TaB_2 with boron content [6; 13]. These compounds show relatively wide homogeneity ranges. Comparable data are not available for other diborides.

Kiessling [6] has reported that at the lower boron limit for TaB_2 (*ca.* 64 atomic per cent) "a" = 3.099Å and "c" = 3.224 Å. At the upper boron limit (*ca.* 72 atomic per cent) "a" = 3.057 Å and "c" = 3.291 Å. Similar results have been reported by Brewer *et al* [13] and have also been obtained in the course of this investigation.

In the case of NbB_2, Brewer *et al.* [13] found that, at the lower boron limit (the exact homogeneity range was not reported) a = 3.110 Å and c = 3.285 Å; at the upper boron limit a = 3.085 Å and c = 3.311 Å.

In both compounds, however, the Me-B distances remain substantially constant throughout the homogeneity ranges: these are 2.41 Å in TaB_2 and 2.43 Å in NbB_2. It is clear from equation (1) that, if $d_{(Me-B)}$ remains constant, an increase in "a" must be compensated for by a decrease in "c", and vice versa, as is observed.

In both cases, too, "a" decreases as the boron content of the phase increases, and increases as the boron content goes down. In the MeB_2 structure a boron content in excess of the stoichiometric amount indicates that the boron layers are filled while the metal layers are only partially full; the converse is true in cases of boron deficiencies.

In these borides the length of the "a" axis appears to be determined by the balance between two opposing forces: expansive forces due to "enlarged" metal atoms, which are opposed by strong cohesive forces within the boron network which resist any increases in the boron-boron separations. In these circumstances the "a" dimension decreases when the cohesive forces of a full boron layer are opposed by the weakened expansive forces of a partially filled metal layer. (It must be borne in mind that when the boron content rises to 72 atomic per cent, as in the case of TaB_2, the metal content is only 28 atomic per cent, and there are, therefore, 8 vacancies out of every 36 available metal positions.) Similar considerations explain the increase in "a" when the boron content is decreased. The observed variations in "c" simply compensate for the changes in "a" while $d_{(Me-B)}$ is maintained constant.

"Enlarged" metal atoms have been postulated to explain the variations in lattice dimensions. The magnitude of this "enlargement" appears to correspond closely to the values listed in column 5 of Table II (i.e. the radii for twelve-fold coordination appear to increase by these amounts in MeB_2). The length of the "a" axis will be close to twice this "enlarged" metal radius in cases where this increase does not involve a large increase in the boron-boron separation over the "normal" (1.74 Å) value. However, in cases like ZrB_2 and HfB_2, the effective radius of the metal atom in the "a"-direction is no greater than the metal radius for twelve-fold coordination. In both ZrB_2 and HfB_2 the strong cohesive forces in the "stretched" boron lattice (B-B = 1.83 Å in ZrB_2) prevent any expansion of the metal atoms in the "a"-direction. No such restraints are present in the "c" or the "Me-B" directions, and in these directions these metal radii increase by approximately 0.07 Å.

The variation in the c/a ratio can be explained on this basis. Where, as in CrB_2 and VB_2, even the expanded metal atoms are not in contact, the c/a ratio is small. It can readily be shown that, when the metal atoms are in "contact" and the boron-boron separation is "normal" or close to "normal," the c/a ratio will be about 1.08. In the case of the largest metal atoms the "normal" increase in "a" is prevented by factors mentioned above and the c/a ratio rises to 1.10 and 1.11.

3. *Melting Points of the Diborides*

A comparison of the melting points of the metals and their diborides (Table III) is of interest. The melting points of diborides that had not previously been reported were determined in the course of this investigation. The ratios of the melting points of the diborides to the melting points of their respective metals are also listed in Table III. These ratios (which reflect the thermal stabilities of the diborides relative to the pure metals) decrease regularly in going from Group IV to Group VI, and decrease also within each group in going from lower to higher atomic numbers. The melting points of the pure metals behave in the opposite fashion; they increase in going from Group IV to Group VI, as well as in going from lower to higher atomic numbers within a group.

It is evident that the bonds which determine the thermal stability of the diborides are not simply

Me-Me bonds. Nor do they appear to be B-B bonds. The melting points of diborides of large metal atoms have the highest melting points, although in these compounds the B-B separations are greatest, and the B-B bonds presumably weakest. It therefore appears probable that the melting points of the diborides reflect primarily the strength of the Me-B bonds.

The data in Table III indicate, too, that the diborides of Group VII would, if they existed,

TABLE III

MELTING POINTS OF METALS AND THEIR DIBORIDES

	MP°C. Metal	MP°C. MeB_2	Ratio MP (MeB_2) in °K / MP (Me)
Ti	1700	2920	1.62
Zr	1850	3050	1.57
Hf	2250	3240	1.40
V	1735	2400	1.33
Nb	2500	3050	1.20
Ta	2990	3200	1.06
Cr	1850	1900	1.02
Mo	2620	2190	.83
W	3410	2200*	.67

*Refers to melting point of W_2B_5; it is probable that W_2B_5, like Mo_2B_5, transforms to the MeB_2 form near the melting point.

probably exhibit low thermal stability relative to the pure metals. So far as is known diborides of these metals have never been prepared.

These results may be compared with recent findings of Hagg and Kiessling [14]. Their studies of ternary metal-boron systems indicate that, in transition metals of the first series, the strength of the Me-B bonds (in MeB and Me_2B) decreases with increasing atomic numbers.

II. Solid Solutions

It is evident that metal-to-metal replacement to form solid solutions should occur readily in the diborides. In this section we shall discuss the results obtained in the course of an investigation of these solid solutions.

1. *Preparation of Raw Material and Samples*

All the metal diborides were prepared by direct synthesis from the elements. The purity of the products was controlled by chemical and X-ray diffraction analyses.

To obtain solid solutions, two borides were mixed in the desired proportions and hot pressed into bars approximately ¼ × ½ × 1". Very high currents were then passed through these boride test samples. Samples were heated in this way very rapidly to their melting points in a helium atmosphere. Surfaces which had been in contact with carbon dies during hot pressing were carefully ground to remove all surface carbon. All samples were chemically analyzed; these latter values, rather than the proportions mixed, were used in determining composition.

Solid solutions of diborides of Zr, Ti and Cr with all the other diborides were studied. It was felt that results obtained with these three diboride systems would be fairly typical of all the diborides. Zr is the largest metal atom of the group studied; Cr is the smallest; and Ti is intermediate in size.

The extent of mutual solubility was estimated from X-ray diffraction measurements; a Norelco Geiger Counter spectrometer was used for all diffraction experiments.

A preliminary series of experiments conducted at 1500°C indicated complete solid solution in only two of a large number of trials. Even in these two cases (Cr-V and Nb-Ti) the similarities in lattice constants could easily have disguised incomplete solid solution. Much higher reaction temperatures were clearly needed. Sample bars were then heated until they melted. All temperatures were measured optically.

2. *Results*

The results of these experiments are outlined in Table IV. Table IV also shows the reaction temperatures reached during all these experiments. The ratios of the effective radii of the metal in the diborides, as computed in Part I above, are shown in column 2. In addition, mutual solubilities, as estimated from X-ray diffraction measurements, are shown in column 4.

3. *Discussion of Results*

It can be seen, from the data in Table IV, that in general the so-called "15 per cent rule" is obeyed for the systems studied. In addition, measurements of the lattice constants of the solid solutions indicated a practically linear variation of parameters with composition. These measurements are summarized in Table V. In a few cases, where X-ray diffraction measurements of 50–50 compositions of two diborides with favorable radius ratios showed clear evidence of only one solid solution phase, further measurements were considered unnecessary for the purposes of this investigation.

The deviations from Vegard's Law which occurred in a few cases were generally positive in the case of measurements of the "c" axis. This type of devia-

TABLE IV

MUTUAL SOLUBILITIES OF METAL DIBORIDES

(1) ZrB_2 in MeB_2	(2) Approx. Reaction Temp. (°C.)	(3) Radius Ratio % Diff.	(4) Solubility Mole %	(1) MeB_2 in ZrB_2	(3) Radius Ratio % Diff.	(4) Solubility Mole %
ZrB_2 in TiB_2	3000±100	11	100	TiB_2 in ZrB_2	10	100
" " HfB_2	3100±100	2	100	HfB_2 " "	2	100
" " VB_2	2500±200	16	0–5	VB_2 " "	14	10–15
" " NbB_2	3000±100	8	100	NbB_2 " "	7	100
" " TaB_2	3000±100	8	100	TaB_2 " "	8	100
" " CrB_2	2100±100	17	0–5	CrB_2 " "	14	10–15
" " MoB_2	2600±100	14	100	MoB_2 " "	12	100
" " W_2B_5	2700±100	13	*	WB_2 " "	12	*
TiB_2 in MeB_2				MeB_2 in TiB_2		
TiB_2 in ZrB_2	3000±100	10	100	ZrB_2 in TiB_2	11	100
" " HfB_2	3000±100	8	100	HfB_2 " "	9	100
" " VB_2	2700±100	5	100	VB_2 " "	5	100
" " NbB_2	3000±100	3	100	NbB_2 " "	3	100
" " TaB_2	3000±100	2	100	TaB_2 " "	2	100
" " CrB_2	2100±100	6	100	CrB_2 " "	5	100
" " MoB_2	2500±100	3	100	MoB_2 " "	3	100
" " W_2B_5	2700±100	2	*	WB_2 " "	2	*
CrB_2 in MeB_2				MeB_2 in CrB_2		
CrB_2 in TiB_2	2100±100	5	100	TiB_2 in CrB_2	6	100
" " ZrB_2	2100±100	14	10–15	ZrB_2 " "	17	0–5
" " HfB_2	2500±100	13	5	HfB_2 " "	15	0–5
" " VB_2	2000±100	—	100	VB_2 " "	—	100
" " NbB_2	2500±100	8	100	NbB_2 " "	8	100
" " TaB_2	2500±100	7	100	TaB_2 " "	8	100
" " MoB_2	2000±100	3	100	MoB_2 " "	3	100
" " W_2B_5	2100±100	3	*	WB_2 " "	3	*

*Exact limits not as yet determined.

TABLE V

CALCULATED AND OBSERVED LATTICE CONSTANTS OF METAL DIBORIDE SOLID SOLUTIONS

(50–50 Mole Per cent)

	"a"		"c"	
	Calc. $\left(\frac{a_1+a_2}{2}\right)$	Observed	Calc. $\left(\frac{c_1+c_2}{2}\right)$	Observed
Cr/Ti	3.00	2.99	3.147	3.14
V	2.984	2.99	3.061	3.045
Nb	3.029	3.03	3.185	3.20
Ta	3.023	3.025	3.165	3.21
Mo	3.005	3.01	3.065	3.12
Ti/Zr	3.098	3.10	3.379	3.392
Hf	3.085	3.085	3.35	3.368
V	3.013	3.01	3.142	3.15
Nb	3.054	3.06	3.266	3.264
Ta	3.053	3.05	3.248	3.246
Mo	3.035	3.035	3.147	3.206
Zr/Hf	3.155	3.155	3.50	3.497
Nb	3.129	3.128	3.426	3.42
Ta	3.124	3.12	3.397	3.40
Mo	3.105	3.085	3.30	3.40
Ti	3.098	3.098	3.38	3.39

tion is consistent with the considerations discussed above in Part I. In Table IV a number of reactions where only limited solubility could be observed are listed. The extent of solubility was estimated from X-ray diffraction measurements of lattice constants. It was assumed that, in the regions of interest, lattice constants varied linearly with composition. It was estimated that solubility limits could be approximated in this way to within 5 per cent by interpolation.

It will be noted that the behavior of the MoB_2 solid solutions showed anomalous variation of lattice constants with composition. Large and positive deviations of the "c" axis were observed in the solid solutions with ZrB_2, TiB_2 and CrB_2. The cause of these deviations is difficult to determine; it is, however, probably related to the variations which have been observed in the reported values of the lattice constants of pure MoB_2 (Table I).

Efforts were also made to prepare solid solutions of Mo_2B_5 and W_2B_5. As was mentioned above, the structures of these two compounds are very similar;

they differ only in the lengths of the "c" axes, i.e., in the extent of ordering in the "c" direction. In W_2B_5 the sequence of metal sheets in the "c" direction is $AABBAA$; in Mo_2B_5 it is $AABBCCAA$. Planar and puckered layers of boron atoms alternate between layers of metal atoms. In both W_2B_5 and Mo_2B_5 separations between layers of metal atoms are of two types; they are 3.07 Å and 3.85 Å in W_2B_5, and 3.13 Å and 3.82 Å in Mo_2B_5.

It was found that larger amounts of Mo_2B_5 can be accommodated in the W_2B_5 structure than vice versa. The W_2B_5 phase is the more disordered of the two and its structure is favored in solid solution formation.

Similar results were observed in the TiB_2–W_2B_5 system. In solid solutions containing 50 mole per cent of each compound, the phase present had the MeB_2 structure.

The result in this latter case is of particular interest since repeated efforts to prepare pure WB_2 were unsuccessful. It is possible that this compound, like MoB_2 [8a], forms at high temperatures; however, this phase (WB_2) was not observed at room temperature even after very rapid quenching of the high temperature reaction product.

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