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The Effect of Different Natural Flake Graphite Additions on the High-Temperature Properties of a Dolomite-Carbon Refractory

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UNITED STATES DEPARTMENT OF THE INTERIOR

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UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

°F	degree Fahrenheit	mL	milliliter
g/cm^3	gram per cubic centimeter	mm	millimeter
h	hour	pct	percent
in	inch	psi	pound per square inch
L/min	liter per minute	wt pct	weight percent
lb	pound		

THE EFFECT OF DIFFERENT NATURAL FLAKE GRAPHITE ADDITIONS ON THE HIGH-TEMPERATURE PROPERTIES OF A DOLOMITE-CARBON REFRACTORY

By James P. Bennett[1]

ABSTRACT

The Bureau of Mines investigated the role of imported natural flake graphite in dolomite-carbon refractories used in steelmaking processes. Fundamental engineering data were obtained to enable the evaluation of substitute materials. Changes in oxidation resistance (2,000° F), hot strength (2,750° F), and deformation under load (2,750° F) caused by 10-wt-pct additions of five different kinds of natural flake graphite and increasing quantities (0 to 30 pct) of a 90-pct-carbon natural flake graphite were studied.

Results indicated that carbon purity of 10-wt-pct-graphite additions did not influence hot strength or deformation under load. When the quantity of 90-pct-carbon graphite addition varied between 0 and 30 wt pct in a dolomite-carbon brick, hot strength was highest and deformation under load lowest with 10-wt-pct additions. As hot strength test temperature was increased from 500° to 2,750° F, the strength difference observed between 0- and 30-wt-pct additions of a 90-pct-carbon graphite became smaller. Additions of a boron-treated graphite caused significantly higher strength and lower deformation under load at high temperatures, while an addition of ball clay significantly reduced strength and increased deformation.

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INTRODUCTION

Continuous casting of steel, combined with increased operating temperatures and demands for longer refractory life, has focused attention on carbon-containing refractories (1-2).[2] Carbon, in the form of natural flake graphite, imparts a high degree of oxidation resistance, reduces the level of wettability by slag, and increases the thermal conductivity of the refractory. Graphite-based refractories have become integrated into the basic oxygen furnace, the electric arc furnace, and transfer ladles, as well as pouring tubes and nozzles. The carbon-based refractories typically contain MgO, Al_2O_3, or dolomite bonded by pitch or resin with up to 30-wt-pct natural flake graphite. The natural flake graphite used in refractories is totally imported.

The need to develop a substitute material for natural flake graphite has been recognized (3), yet fundamental high-temperature engineering data such as is available on magnesia-carbon systems does not exist on dolomite-carbon refractories, making it difficult or impossible to predict or compare behavior. This report presents the results of a study on the variations in high-temperature physical properties caused by differences in graphite purity and in the quantity of natural flake graphite added to dolomite-carbon refractories. The information will be used by the Bureau to evaluate substitute materials for natural flake graphite.

ACKNOWLEDGMENTS

The fabrication of the dolomite-carbon test samples, room-temperature physical properties, and oxidation data were supplied under a memorandum of agreement with J. E. Baker Company of York, PA.

RAW MATERIALS AND SAMPLE DESCRIPTION

All dolomite-carbon refractories evaluated in this research were manufactured from dead-burned dolomite grain, resin, and natural flake graphite. Dolomite grain chemistry and density are listed in table 1. The particle size fraction of the dolomite grain was the same utilized in a commercial product. Five grades of natural flake graphite, ranging in carbon content from 85 to 100 pct, were chosen for this study. These graphites will be referred to as 85, 90, 95, or 100 grades. All were products from Madagascar and were similar in particle size distribution. The particle size distribution, ash chemistry, and ash pyrometric cone equivalent (PCE) test results of the graphite results are listed in table 2. The 100 grade was thermally purified from a lower purity graphite according to a process described by Hand (4). Refractory grade (RG) is a high-purity, high boron content material manufactured by

TABLE 1. - Fired dolomite grain and ball clay physical properties

	Fired dolomite	Ball clay
Chemical composition, wt pct:		
CaO	56.7	0.6
MgO	41.2	0.6
Al_2O_3	0.5	30.6
SiO_2	0.4	65.5
Fe_2O_3	1.2	2.7
Density, g/cm^3	3.25-3.28	NA
Loss on ignition, wt pct	0	10.1

NA Not analyzed.

[2] Underlined numbers in parentheses refer to items in the list of references at the end of this report.

TABLE 2. - Natural flake graphite properties

Property	Graphite grade				
	85	90	95	100	RG
Screen analysis, wt pct retained:					
-18	0	0	0	0.2	0.1
-18, +30	1.7	14.4	9.8	11.9	12.6
-30, +40	23	26.4	25.9	27.7	31.2
-40, +60	59.7	44.3	48.1	48.1	47.9
-60, +80	14.3	12.1	13.8	10.7	7.6
-80, +100	1.2	1.5	1.6	1.1	0.5
-100	0.2	1.3	0.9	0.3	0.1
Ash, wt pct	13.7	10.6	6	0	0.8
Ash chemistry, wt pct:					
SiO_2	55.9	45.2	43.8	NA	23.4
Fe_2O_3	11.8	23.7	26.1	NA	21.9
Al_2O_3	26.2	22.8	25.7	NA	0.9
MgO	1.1	6.8	4.3	NA	0.7
CaO	1	1.3	1.2	NA	2.2
B_2O_3	ND	ND	ND	NA	43.75
Ash, PCE	14-15	6-7	6	NA	NA

NA Not analyzed. ND Not detected.

TABLE 3. - Physical properties of dolomite-carbon brick with 10-wt-pct-graphite additions

Graphite grade	Green bulk density, g/cm^3	Cured bulk density, g/cm^3	Cured MOR, psi	Coked MOR, psi	Retained carbon, pct
85	2.94	2.91	3,030	350	9.2
90	2.95	2.92	2,310	420	8.9
95	2.95	2.91	2,290	370	9.9
95+1 pct ball clay	2.94	2.90	2,240	370	9.8
100	2.75	2.69	640	270	11.9
RG	2.94	2.91	2,360	430	10.0

Superior Graphite Company[3] and is treated with an oxidation inhibitor to improve high-temperature properties.

The following two sets of mixes were prepared: one with 10-wt-pct additions of different graphite grades (85, 90, 95, 100, and RG) added to dead-burned dolomite grain (table 3); the second with a 90-grade graphite added in increments of 0, 5, 10, 15, 20, and 30 wt pct (table 4). The mix listed in table 3 as 95+1-pct ball clay (95+1) contains 1-pct ball clay (chemistry listed in table 1) in addition to 10 wt pct of the 95-grade graphite. Four percent of a thermosetting resin was used as a binder in all samples. Compositions were mixed in 150-lb batches in a high-speed countercurrent mixer. Bricks of 6- by 9- by 3-1/2-in dimensions were pressed at 20,000 psi and cured on a commercial schedule.

[3]Reference to specific products does not imply endorsement by the Bureau of Mines.

TABLE 4. — Physical properties of dolomite-carbon brick with increasing additions of 90-grade graphite

Graphite addition, wt pct	Green bulk density, g/cm^3	Cured bulk density, g/cm^3	Cured MOR, psi	Coked MOR, psi	Retained carbon, wt pct
0..........................	2.98	2.96	4,230	590	2.3
5..........................	2.97	2.94	3,200	450	6.3
10.........................	2.95	2.92	2,310	420	8.9
15.........................	2.90	2.85	1,760	400	13.1
20.........................	2.87	2.80	1,360	300	14.3
30.........................	2.79	2.72	1,130	230	24.0

FIGURE 1.—Test equipment for hot MOR evaluation in N_2.

TEST EQUIPMENT AND PROCEDURE

The as-received bulk density was conducted on samples after pressing and curing of the resin. Coked physical properties were obtained by heating samples in a N_2 atmosphere according to ASTM schedule C-607 (5).

The room-temperature cured and coked modulus of rupture (MOR) strengths were measured following ASTM C-133 (6) on dolomite-samples containing 10-wt-pct additions of graphite (85, 90, 95, 100, RG) and on samples with a 90-grade graphite added in quantities of 0 to 30 pct.

Oxidation testing was conducted on 2-by 2- by 3-in dolomite-carbon samples with 10-wt-pct-graphite additions heated at 2,000° F. The loss in weight was measured periodically over 70 h.

Hot MOR strengths from 500° to 2,750° F were measured using ASTM C-583 (7). The testing was conducted in a sealed SiC element furnace (fig. 1) flushed with 2 L/min of dry N_2 gas. Samples 1-1/4 by 1-1/4 by 9 in were cut from the 9- by 6- by 3-1/2-in bricks, and strength was measured with the pressed surface (6 by 9 in) of the sample in contact with the MOR rod. Five samples were evaluated at each temperature. Lampblack was spread on and around the samples to prevent sample oxidation. Oxygen levels less than 0.01 pct were routinely measured in the furnace. Sample strengths were measured after a 1 h hold at test temperature.

Thermal expansion under load tests were conducted according to ASTM C-832 (8) at loads of 50, 100, and 150 psi on dolomite-carbon samples with 10-wt-pct additions of different graphite grades and with 0- to 30-wt-pct additions of a 90-grade graphite. Samples 1-1/2 by 1-1/2 by 4-1/2 in were cut from 9- by 6- by 4-1/2-in brick with the pressed 4-1/2-in height taken as the sample height. Samples were placed in a high-alumina crucible (fig. 2) surrounded by a loosely packed 50:50 mixture of lampblack and electrolytic graphite to prevent sample oxidation. A 0.005-in-thick flexible graphite sheet was placed between the alumina load transfer ram and the dolomite-carbon sample to prevent high-temperature reactions. The furnace could monitor six thermal expansion under load samples simultaneously (fig. 3). Samples were heated under load to 2,750° F, held at temperature for 2 h, and cooled to room temperature. The three variables investigated included: (1) thermal expansion versus temperature under load during heating to 2,750° F, (2) sample deformation versus time during the 2 h (creep) hold at 2,750° F, and (3) overall sample deformation after heating and cooling.

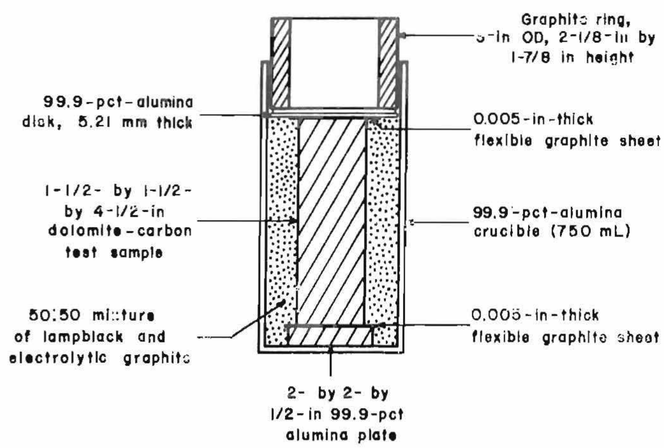

FIGURE 2.—Cross section of the sample holder used to monitor thermal expansion under load, while preventing sample oxidation.

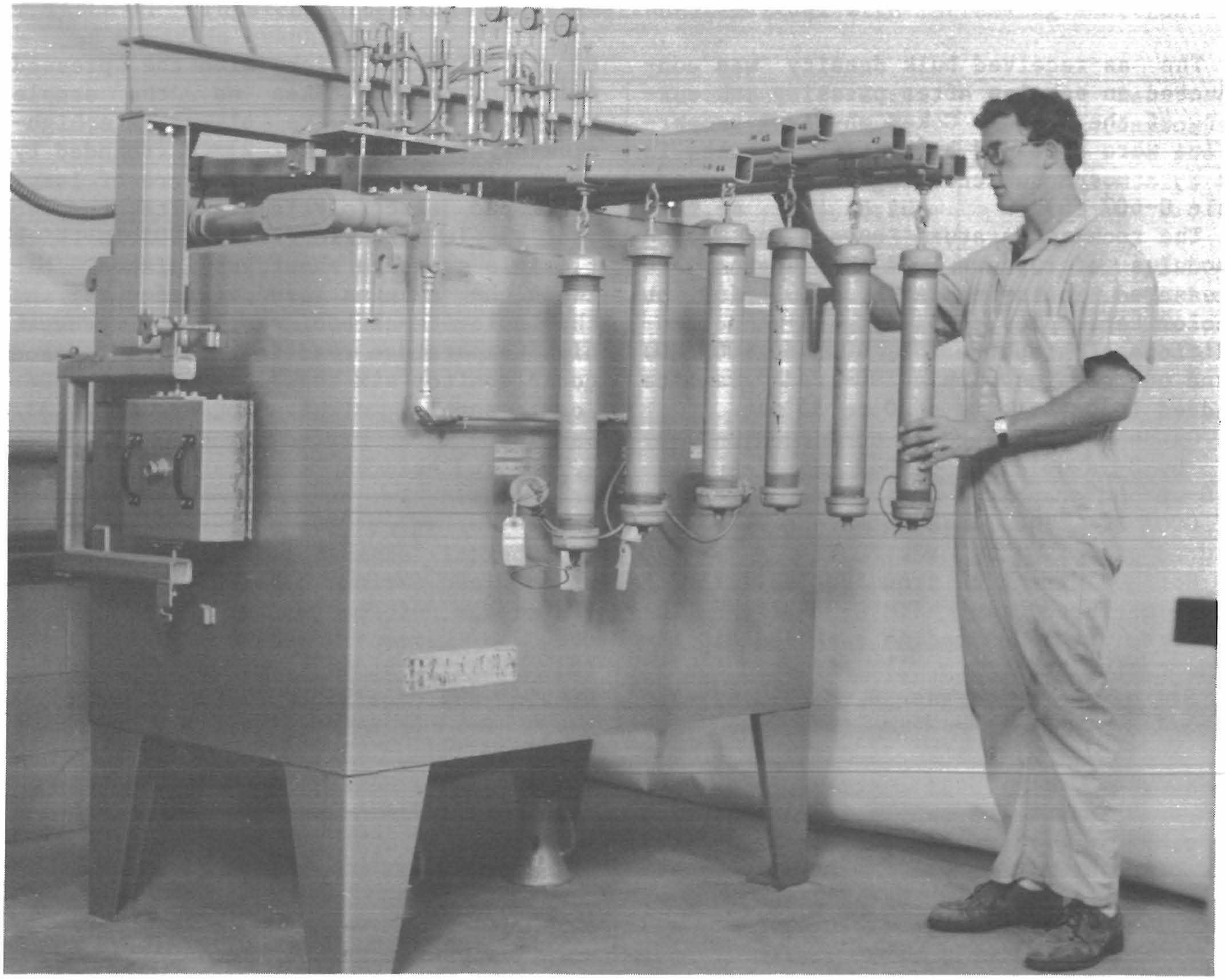

FIGURE 3.—Test equipment for thermal expansion under load testing.

RESULTS AND DISCUSSION

BULK DENSITY

The green and cured bulk density of the dolomite-carbon brick with 10-wt-pct additions of different graphite grades is listed in table 3. The green bulk density averaged about 2.95 g/cm^3 for all grades except the 100, which had a lower value of 2.75 g/cm^3. After curing, all samples decreased in density to about 2.91 g/cm^3, except for the 100 grade, which averaged 2.69 g/cm^3.

The bulk densities of samples containing increasing amounts (0 to 30 pct) of a 90-grade graphite are listed in table 4. As the quantity of natural flake graphite increased, the green and cured bulk densities decreased. With a 30-wt-pct-graphite addition, the cured bulk density dropped to a value of 2.72 g/cm^3, versus 2.96 g/cm^3 with no graphite addition.

CARBON CONTENT AND OXIDATION RESISTANCE

The percent carbon retained after coking the dolomite-carbon samples is listed in table 3 for 10-wt-pct additions of different graphite grades and in table 4 for increasing additions of a 90-grade graphite. When 10-wt-pct additions of different graphite grades are made to dolomite-carbon samples, as the carbon content of the graphite increases from 85 to 100 pct, the retained carbon increases from 9.2 to 11.9 pct, respectively. When the quantity of graphite is increased (table 4), the percent retained carbon increases from 2.3 (0-wt-pct graphite) to 24.0 (30-wt-pct graphite).

The loss of carbon by oxidation from 2- by 2- by 3-in samples with 10-wt-pct-graphite additions at 2,000° F is shown in figure 4. Ash content in the graphite did not cause large differences in the oxidation rates of the samples. Ash content in MgO-carbon brick, however, has been found by Ishibashi, Matsumura, Hosokawa, and Matsumoto (9) to promote oxidation. SiO_2 and B_2O_3 also promoted the reduction of MgO by carbon, causing a loosening of the brick texture. Oxidation of carbon was more rapid at higher test temperatures. There was no evidence of this effect in the dolomite-carbon refractories tested, however, the 2,000° F test was at the low-temperature range of the 1,650° to 3,000° F where carbon reduction is thought to occur (10).

MOR

Room-temperature cured and coked MOR data are listed in table 3 for dolomite-carbon samples containing 10-wt-pct-graphite grade additions. The room-temperature cured MOR strengths ranged between 2,200 and 3,100 psi for all the graphite grades (85, 90, 95, RG, and 95+1). The 100 grade, however, had a lower strength value of 640 psi. It also had the lowest coked strength, 270 psi, versus strengths from 350 to 430 psi for the other grades. The low-strength values for the 10-wt-pct additions of the 100-grade graphite were caused by the low bulk density obtained during pressing. Rebound (expansion of a brick after pressing) occurred, causing these low values. Because of the low bulk density and room-temperature strengths, further testing of the 100 grade was discontinued.

Hot MOR data for dolomite-carbon brick with 10-wt-pct additions of different graphite grades is listed in table 5 and plotted in figure 5. When the test temperature was increased from 500° to 2,750° F, brick strength decreased for all grades. The strength decrease between 500° and 1,500° F is due to the coking of the resin, when a carbon bond is formed between dolomite grains and graphite.

Statistically significant (Student's t-test, 11) strength changes at a 95-pct confidence level were determined. No significant strength differences occurred with 10-wt-pct additions of 85-, 90-, and 95-grade graphites. At 2,500° F and above, a 10-wt-pct addition of RG graphite resulted in a significant strength increase, while the 95+1 grade resulted in a strength decrease.

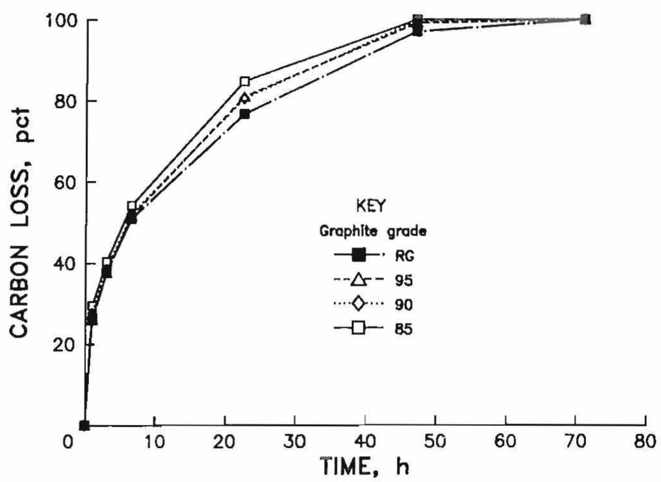

FIGURE 4.—Percent carbon weight loss versus time in dolomite-carbon brick containing 10-wt-pct-graphite additions at 2,000° F.

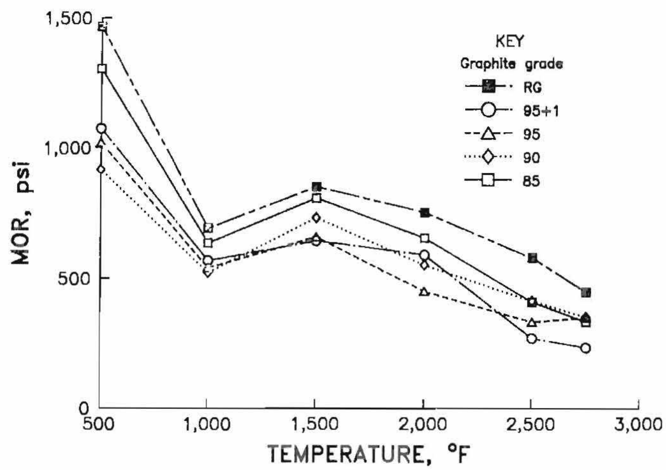

FIGURE 5.—Hot MOR strength versus temperature of dolomite-carbon brick containing 10-wt-pct-graphite additions.

TABLE 5. - Hot MOR strength of dolomite-carbon samples with additions of different grades and amounts of graphite, pound per square inch

Graphite	Temperature, °F					
	500	1,000	1,500	2,000	2,500	2,750
10-WT-PCT GRAPHITE ADDITIONS OF VARIOUS GRADES						
85	1,310±480	640±140	810±130	650±140	410± 80	330± 60
90	920±200	520±120	730± 60	550±110	410± 60	350± 50
95	1,020±160	540±100	660± 50	450±180	330± 70	350± 50
95+1	1,080±160	570± 70	640±120	590±140	270± 70	230± 50
RG	1,470±250	690±160	850±180	760±110	580± 80	450± 60
INCREASING ADDITIONS OF 90-GRADE GRAPHITE						
0	3,410±960	1,150±260	1,040± 70	670± 80	460± 80	350± 30
5	1,500±660	970±270	930± 80	710±180	270±110	280± 40
10	920±200	520±120	730± 60	550±110	410± 60	350± 50
15	740± 70	510± 80	590±140	530±140	250±100	220±100
20	430± 40	450±120	540± 90	370± 80	260± 30	200± 60
30	460±110	380± 30	370±150	320± 60	200± 10	170± 50

NOTE.--Plus-minus (±) values are at 95-pct confidence intervals.

At the elevated temperatures (above 2,900° F) encountered in a steel furnace in magnesia-carbon refractory systems, carbon can reduce compounds such as MgO, Fe_2O_3, or SiO_2, causing vapor transport of phases within the brick and a change in physical properties (12). High-purity graphite is thought necessary (9) because carbon reduces the graphite ash, resulting in a loose microstructure that allows the graphite to be easily oxidized (13) or results in the formation of a film along the graphite-magnesia grain boundaries (14). This ash-based film is composed of spinel, forsterite, or low-melting compounds in the CaO-SiO_2-Al_2O_3-MgO system and causes low MOR strength at elevated temperatures. Some mineral phases formed by graphite ash, such as monticellite ($CaO \cdot MgO \cdot SiO_2$, melting point 2,708° F), are thought to be more resistant to reduction by carbon than others, such as forsterite ($2MgO \cdot SiO_2$, melting point 3,450° F). Whether the ash in a dolomite-carbon system forms higher melting liquids, or a phase more resistant to oxidation, is not known.

The RG-graphite (high B_2O_3 ash content) addition had the highest hot strength at all temperatures evaluated. Boron may improve the bond strength by carbide formation causing strength increases, or it may make the graphite less reactive with other materials. The RG also had the highest coked MOR strength listed in table 3.

Room-temperature cured and coked MOR data for samples with increasing additions of a 90-grade graphite are listed in table 4 and hot MOR data in table 5. The room-temperature cured MOR strengths ranged from a high of 4,230 psi for no graphite additions, to a low of 1,130 psi for the 30-pct additions. As the amount of graphite added to a sample increased, both the cured and coked strength decreased. This trend was observed in hot MOR data through 2,000° F (fig. 6). At the test temperatures of 2,500° and 2,750° F, the strength values for the

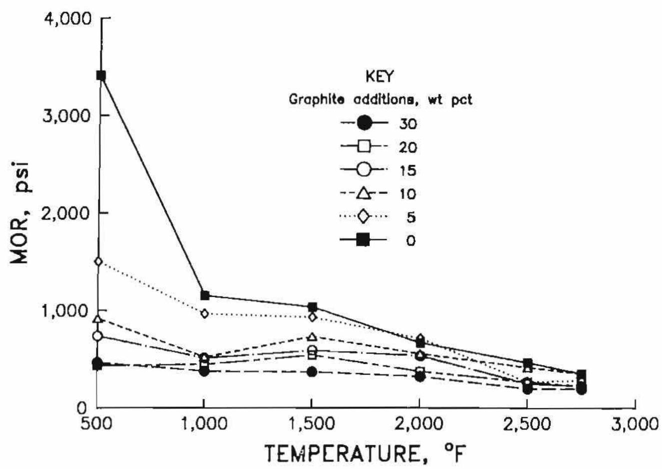

FIGURE 6.—Hot MOR strength versus temperature in dolomite-carbon brick with increasing additions of 90-grade graphite.

0- and 10-wt-pct additions were statistically higher than the other graphite additions.

The microstructure of a magnesia-carbon refractory has been shown (1) to have a continuous carbon matrix at graphite levels greater than 10 pct. At less than 10-pct-graphite additions, a significant number of periclase grain contacts occur. The optimum hot MOR strength at 10-wt-pct graphite may be a result of this matrix transition. The decrease in strength, at all test temperatures, for graphite additions above 10 wt pct may be due to the lower strength of the graphite or the poorer bond caused by the dilution of the resin-carbon bond.

Another effect of temperature on hot MOR strength is noted by comparing strength decreases in table 5 between 500° and 2,750° F as the quantity of a 90-grade graphite increases. At 500° F, the strength of the 0-pct-graphite sample was the highest (3,410 psi), with increasing graphite content leading to strength reductions (460 psi with 30-pct graphite). At 2,750° F, the hot strength of the 0- through 30-pct-graphite additions ranged between 350 and 170 psi, respectively. The strength advantage of the lower graphite content is largely eliminated at higher test temperatures.

THERMAL EXPANSION UNDER LOAD

The thermal expansion curves versus temperature under loads of 50, 100, and 150 psi for samples with 10-wt-pct additions of different graphite grades (85, 90, 95, 95+1, and RG) and for increasing additions of 90-grade graphite (0 to 30 pct) are similar to those shown in figure 7 for the 10-wt-pct addition of an 85-grade graphite. As the load increased on a sample, the amount of deformation increased. A slight brick expansion occurred between the temperatures of 500° and 1,200° F as the resin bond carbonized, and corresponded to the decrease in hot MOR observed. No trends were observed in the deflection point where expansion and deformation were equal in the samples (2,330° to 2,500° F).

A plot of deformation versus time (creep) at 2,750° F, 150-psi load, is shown in figure 8 for 10-wt-pct additions of different graphite grades and in figure 9 for increasing additions of a 90-grade graphite. With 10-wt-pct additions of different graphite grades, the largest creep occurred in the 85 grade, followed by the 90, 95, and RG. The addition of 1-pct ball clay caused increased creep in the 95 grade during the 2-h period. Similar creep behavior was noted at load applications of 50 and 100 psi in all samples, but at lower rates of deformation. Lower MOR strength also occurred with ball-clay additions in 95-grade samples at 2,500° and 2,750° F, as previously noted in the MOR discussion. The lower creep of the RG also agrees with the

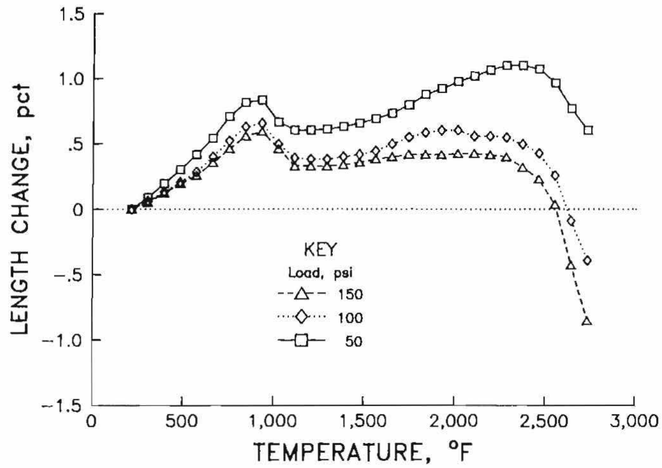

FIGURE 7.—Thermal expansion under load of a dolomite-carbon sample containing 10-wt-pct addition of 85-grade graphite.

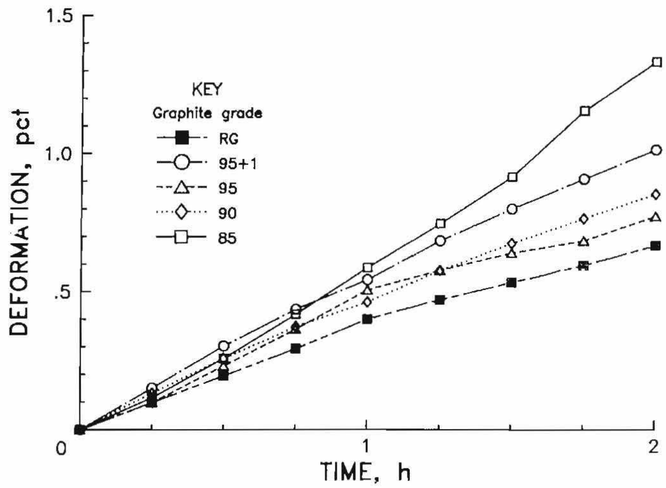

FIGURE 8.—Creep at 2,750° F for dolomite-carbon samples with 10-wt-pct-graphite additions.

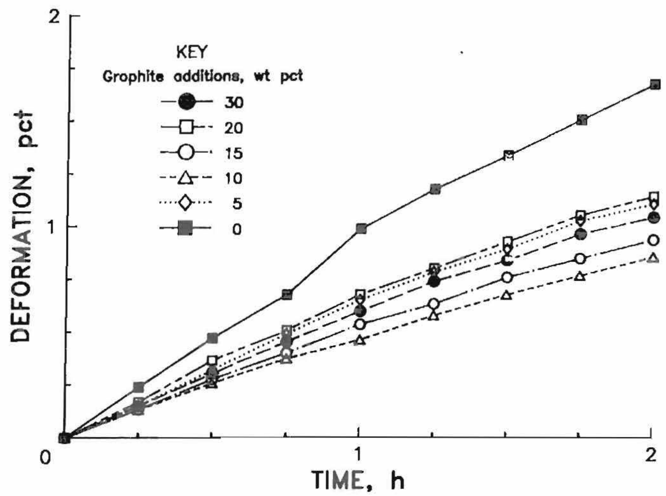

FIGURE 9.—Creep at 2,750° F for dolomite-carbon samples with increasing additions of 90-grade graphite.

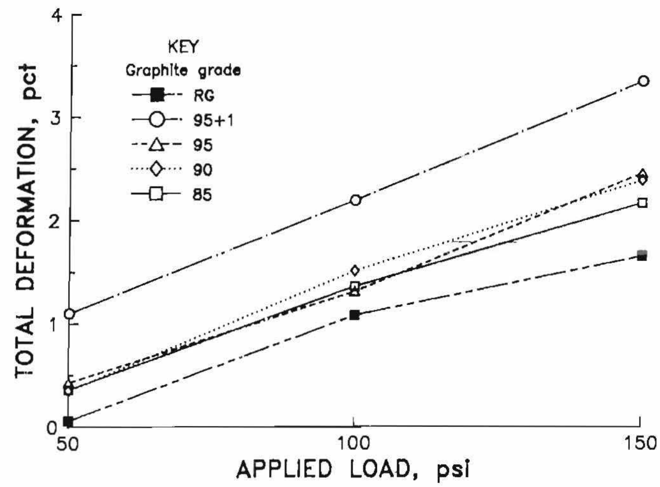

FIGURE 10.—Total deformation for dolomite-carbon samples with 10-wt-pct additions of natural flake graphite.

higher hot MOR strengths noted previously. Creep measurements were monitored over a 2-h time period and may not represent long-term load behavior.

Creep measurements of samples with increasing additions (0 to 30 pct) of a 90-grade graphite (fig. 9) were lowest for the 10-wt-pct addition; lower or higher graphite additions caused the creep to increase. Similar creep behavior was noted at load applications of 50 and 100 psi, but at lower rates of deformation. The 10-pct-graphite addition is the transition point to a continuous carbon matrix, as mentioned in the hot MOR data discussion. Creep-data variations agree with high-temperature hot MOR data in table 5, where strength peaked with 10-wt-pct-graphite additions at 2,500° and 2,750° F.

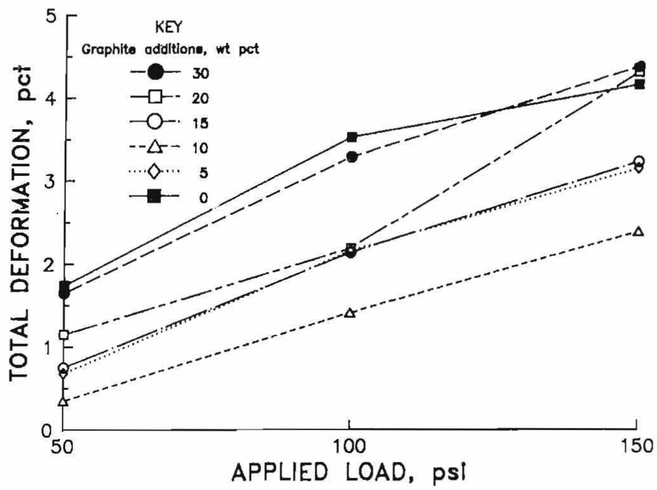

FIGURE 11.—Total deformation for dolomite-carbon samples with increasing additions of 90-grade graphite.

The total deformation occurring with applied loads of 50, 100, and 150 psi in dolomite-carbon samples (after being heated to 2,750° F, held for 2 h, and cooled) is listed in the following: table 6 and figure 10 for differences in graphite grades; table 6 and figure 11 for different additions of a 90-grade graphite. When 10-wt-pct additions of different graphite grades were made, the largest deformation always occurred in the 95+1 grade and the least in the RG, regardless of the applied load. The maximum deformation occurred with loads of 150 psi, and was 3.34 pct for the 95+1 grade and 1.65 pct for the RG. The deformation occurring in the 85, 90, and 95 grades of graphite were similar, regardless of the load, which was also the case for hot MOR strengths at 2,750° F.

The total deformation occurring in samples with increasing additions (0 to 30 pct) of a 90-grade graphite was minimum for the 10-wt-pct addition and increased with changes in graphite content from that level. A similar trend was observed in hot MOR data in table 5 at 2,500° and 2,700° F, where the strength value for the 10-wt-pct addition of 90-grade graphite was among the highest. As

TABLE 6. -Total deformation occurring in dolomite-carbon samples with different grades and amounts of graphite, percent

Graphite	Load application		
	50 psi	100 psi	150 psi
10-WT-PCT GRAPHITE ADDITIONS OF VARIOUS GRADES			
85	0.36	1.36	2.16
90	.35	1.51	2.38
95	.43	1.31	2.45
95+1	1.10	2.19	3.34
RG	.06	1.08	1.65
INCREASING ADDITIONS OF 90-GRADE GRAPHITE			
0	1.74	3.52	4.15
5	.68	2.15	3.14
10	.35	1.41	2.38
15	.75	2.13	3.22
20	1.15	2.18	4.30
30	1.65	3.28	4.37

the applied load increased, the deformation increased. At 150 psi-applied load, the deformation was a low of 2.38 pct for the 10-wt-pct addition and a high of 4.37 for the 30-wt-pct addition.

CONCLUSIONS

A study of the effects of different natural flake graphite purities and increasing graphite content on high-temperature properties of dolomite-carbon brick indicated the following:

1. Ten weight-percent additions of different purity graphites (80, 90, 95 grades) did not affect the oxidation rate, hot strength, or the deformation under load properties of dolomite-carbon samples.
2. Increasing graphite additions from 0 to 30 wt pct of a 90-grade graphite resulted in optimum hot strength and deformation under load properties at 10-wt-pct additions.
3. As hot-strength test temperature was increased from 500° to 2,750° F, the strength difference observed between 0- and 30-wt-pct additions of a 90-grade graphite became less.
4. Ten weight-percent additions of a boron-treated graphite (RG) caused higher hot stength and lower deformation under load than comparable graphite additions.
5. Ball-clay additions to a 95-grade graphite resulted in decreased hot strength and increased deformation under load properties.

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