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PREPARATION AND EXAMINATION OF BERYLLIUM CARBIDE

by

M. W. Mallett*, E. A. Durbin*, M. C. Udy*,
D. A. Vaughan*, and E. J. Center*

ABSTRACT

The properties of beryllium carbide were studied to determine its suitability as a high-temperature refractory. Various methods of preparing it were tried and a number of the physical and chemical properties of the resulting products were determined. Lots containing 85 wt % of useful product were prepared by the beryllium metal-carbon reaction. Because the material was to be fabricated into refractory bodies, particular attention was paid to the chemical analysis for unreacted BeO, beryllium metal, and free carbon; X-ray diffraction identification of the various phases present; and microscopical examination for mineral composition, crystal size, and crystal habit.

As a refractory, beryllium carbide has several disadvantages. It tends to hydrolyze in atmospheric moisture and must be protected against both oxygen and nitrogen, when heated.

*Battelle Memorial Institute, Columbus, Ohio.

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INTRODUCTION

Because of the recent interest in the use of unconventional refractories such as carbides, nitrides, and sulfides, an investigation was made of the suitability of beryllium carbide as a high-temperature refractory.

The earliest work on beryllium carbide appears to be that of Lebeau⁽¹⁾ in 1895. He prepared the carbide by two methods: (a) by heating a mixture of beryllium and sugar charcoal in an electric furnace,

(b) by heating beryllium oxide with sugar charcoal in an electric furnace (40 Kw) for about 10 minutes,

Lebeau gave it the formula Be_4C_3 but, after Henry⁽²⁾ pointed out an error in the atomic weight and the valency of beryllium had been proven, he adopted the formula Be_2C . No other carbides seem to exist. This compound was described⁽³⁾ as consisting of yellowish-brown transparent crystals with properties quite similar to those of aluminum carbide. It is so hard it scratches quartz easily.

The crystal structure of Be_2C was studied by Stackelberg and Quatram⁽⁴⁾. X-ray investigation showed that Be_2C has an antiferite (CaF_2) lattice like Mg_2Si . The following constants were determined: $a_0 = 4.33 \text{ \AA}$; X-ray density, $d = 2.44$; Be - C distance = $1.87 \text{ \AA}$; and C - C distance = $3.06 \text{ \AA}$.

The work described in the present paper was completed in 1948. Subsequently, Geller⁽⁵⁾ summarized the previously known data on the physical and chemical properties of Be_2C and Coobs and Koshuba⁽⁶⁾ discussed the fabrication of this compound. Procedures for the determination of the principal constituents and contaminants of Be_2C were recently described by Gallagher⁽⁷⁾.

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PREPARATION

The Be_2C prepared for this investigation was made by the two basic methods used by Lebeau, that is, by heating an intimate mixture of BeO and carbon and by reacting beryllium metal and carbon*.

Apparatus

A typical furnace setup is shown in Figure 1. A semipermanent graphite heater crucible was packed in the induction coil with Norblack powder insulation. Vent holes were formed by pushing twelve 1/8-inch-diameter wires into the Norblack packing. The top of the packing was then sealed with Alundum cement before the wires were removed. Care was taken not to form the vent holes after applying the Alundum cement, because any oxide carried into the packing forms gas when heated and forces the Norblack out -- sometimes explosively. A second graphite crucible containing the charge was slipped into the heater crucible. Holes were provided near the top of the inner crucible, into which a heavy iron wire bail could be placed for lifting the hot crucible from the assembly upon completion of a heat. Another crucible and charge could then be placed in the hot furnace and reacted without delay. A solid graphite cover rested on the charge leaving about 1-1/2 inches of space between this cover and a clay-graphite cover which rested on the top edge of the crucible. Into this space was flowed a continuous stream of argon through a fused-silica tube. This provided a blanket of inert gas over the charge during heating. A second hole in the outer lid was used for making optical pyrometer readings on the inner lid. Occasional setups contained a graphite-tube sight-well (3/4 in. I.D. x 1 in. O.D.) centrally located in the charge and extending downward through both covers to within one inch of the bottom of the charge. Lid temperatures were compared with

*A proposed method, which appears feasible but has not been tried, is a vapor-deposition method in which a mixture of a beryllium halide and a hydrocarbon gas, carried in a stream of hydrogen, would be reacted on contact with a hot filament depositing Be_2C on the filament.

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"true" temperatures throughout the range of temperatures used. Lid temperatures varied from 300 C low at 1700 C to about 600 C low at 2200 C.

Experimental Procedure

In a small exploratory heat, a mixture of stoichiometric quantities of BeO (Drush Beryllium Company, H. F. grade, -200 mesh) and graphite powder (-60 mesh) was placed in a graphite crucible and heated in a vacuum furnace ($<10^{-4}$ mm Hg). As the temperature was raised, the first indication of reaction occurred at 1500 C, when rapid gas evolution began. The reaction had not reached completion after two hours' heating at 1650 C. X-ray diffraction analysis indicated that considerable BeO and unreacted carbon still remained. Subsequent larger heats (600-gram charge) were made using a similar mixture except that Clifton Products, Inc., fluorescent-grade BeO was substituted for the more refractory H. F.-grade BeO. The oxide and graphite were intimately mixed by ball milling together. This mixture was heated in an argon atmosphere to a minimum temperature of 1900 C. Even though a temperature of about 2800 C was reached in the hottest part of the charge, some unreacted BeO and carbon remained after four hours' heating. About 60 per cent yield of material containing about 90 per cent Be₂C, with the balance BeO and free carbon, was obtained.

Several lots of Be₂C were prepared by heating a mixture of beryllium metal flake (Clifton Products, Inc.) and minus 60-mesh graphite powder. In some cases, carbon black (Norblack) was used in place of graphite. It was impossible to get uniform mixing because of the nature of the flakes which were about one inch in diameter and foil-like in thickness. However, this was inconsequential because the beryllium apparently vaporized during heating so that the end product consisted of a cylindrical mass, approximately 3-1/2 inches in diameter by 8 inches in length, of loosely sintered Be₂C grains of about 30-mesh grain size. The product ranged from red, through tan, bronze, brown, or black according to the free-carbon content.

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Later, a rather large supply of finely divided beryllium metal in the form of saw chips and turnings was available so this material was substituted for beryllium flake. Similar results were obtained. Some advantage was gained in using the saw chips because about 650 grams of charge could be packed in a crucible which would hold only 500 grams using the bulkier flake.

Twenty to thirty minutes at 1700 C was found sufficient to complete the metal-carbon reaction. This was much more rapid than the BeO-carbon reaction and a greater percentage (85 wt %) of useful product was obtained. Therefore, this method was used to the exclusion of other methods and about 40 lots of beryllium carbide were prepared.

An Alternative Method

In addition to the above methods of producing Be₂C, several other methods were tried. Experience gained during the pouring of beryllium into hot graphite molds showed that beryllium reacts exothermically with graphite to form Be₂C. The reaction seemed to start about 50 C above the melting point of beryllium. Similar reactions were noted when beryllium melts in graphite crucibles were superheated. The reaction could not always be brought about, apparently, because in some cases, a thin adherent layer of carbide formed on the inner surface of the crucible preventing further reaction.

When the reaction did occur, the melt solidified in spite of increasing temperature. This thickening or freezing of the melt was caused by the formation of many individual grains of Be₂C in the metal matrix. In many cases, 80 to 90 per cent of the volume was comprised of these carbide crystals. See Figure 2. The average particle size ran from about 0.5 micron to about 100 microns, depending, among other things, on the temperature of formation and time at temperature. Although the grain size varied greatly from heat to heat, the grains of an individual heat were all of

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the same approximate order of magnitude. Upon exposure of a polished section of this material to air for several days, the carbide particles took on a pink color. The air etch was the result of hydrolysis of the carbide by the moisture of the atmosphere. Highlights in the large grains (Figure 2A) are reflections from within the translucent Be_2C grains. The Be_2C grains were separated by dissolving the beryllium metal matrix in dilute hydrochloric acid. Although smaller grains were greatly hydrolyzed, the large well-formed crystals showed little attack. This method appears well suited for producing relatively pure Be_2C , although it has been used only on a small scale to date.

Sloman⁽⁸⁾ found similar grains of Be_2C formed by the action of graphite on the metal during electrolysis. Remelting allowed the carbide particles to segregate in the bottom of the beryllium ingot. Upon solution of the metal with very dilute HCl , pink crystals were obtained which were identified as Be_2C by chemical and X-ray analysis. Sloman states that, in metal containing segregated carbide, the carbides appear greyish-brown under ordinary vertical illumination. Using a wide-angle lens, an intense pink coloration was noted. He thought this pink color to be caused by a trace of iron carbide. This is unlikely and, in view of observations made on many Be_2C materials, the pink color in reflected light is evidently due to the hydrolysis product on the surface of the carbide.

ANALYSIS AND PROPERTIES OF Be_2C

The various lots of Be_2C were examined and analyzed as they were made or processed. In addition to visual examination with the unaided eye, the principal analyses made included (a) chemical analysis for total and free carbon, water, and Be_2C ; (b) X-ray diffraction identification of phases; and (c) microscopic observations which included measurement of crystal size, identification of crystal types, identification of phases and estimation of percentages by area measurement, and

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adjusted mineral composition after correction for relative apparent densities of the constituents. In addition, observations were made in regard to hardness, density, and melting point of Be_2C . Also, a technique was worked out for recording in natural color the appearance of the carbide under the microscope.

Types of Beryllium Carbide

Casual examination of the product of the usual oxide-carbon or metal-carbon reaction with the unaided eye has led to varied descriptions of the carbide. It has been described as pink, red, yellow, orange, tan, bronze, brown, black, metallic, or gray. These designations taken alone are quite deceptive. As seen under the microscope, in either transmitted or reflected light, Be_2C appears as translucent amber grains. The masking power of black free-carbon particles is great. As little as 10 per cent by area, as measured under the microscope, causes the product to look black to the naked eye.

The types of Be_2C observed in the investigation may be described as:

(1) coarsely crystalline, (2) microcrystalline (nodular), and (3) vermiculate (defined below). The coarsely crystalline Be_2C was obtained in several ways. One product containing 100 per cent of this type of crystal was extracted from beryllium metal (see Figure 2A) which had reacted with a graphite crucible. This was the purest carbide produced, containing only traces of opaque material (free carbon or metal) and a small amount of hydrolysis product resulting from the acid-extraction process. In lots of Be_2C made by the metal-carbon reaction at about 2000 C for 1 hour, 95 to 99 per cent of the crystals were coarse and well formed. (See Table 1.) In addition, this material usually contained 5 to 10 per cent unreacted carbon. However, the Be_2C grains contained no internal free carbon (graphite flakes).

Microcrystalline Be_2C of the nodular or "grape cluster" type was produced by reacting beryllium metal and carbon at lower temperatures (1700 C) or for short

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times (20 to 30 minutes) at 2000 C. A few lots were made in which 98 to 99 per cent of the Be_2C phase was microcrystalline (see Table 1). This material, when crushed and pressed, showed better sintering characteristics than the more coarsely crystalline material. Modular Be_2C is readily made by the metal-carbon reaction; whereas, in the oxide-carbon method, where a long time at temperature is required to complete the reaction, only coarse crystals are produced.

The term "vermiculate" applies primarily to the mass of wormlike tubes often produced when beryllium carbide heats are taken above 2200 C. (See Figure 3.) These tubes appear to be formed from drippings of a viscous fluid. Although they intermingle, they do not adhere to each other. The interior of the hollow structure is an aggregate of angular reddish crystals while the exterior is smooth and metallic in appearance. The metallic color is caused by a thin layer of highly oriented carbon crystals. Because of the similarity of the individual crystals of this and clinker-like porous material which exhibits the vermiform structure to a lesser degree, all high-temperature products with the metallic-appearing surface have been termed "vermiculate".

Vermiculate Fe_2C is evidently a decomposition product, formed by loss of beryllium through vaporization. This leaves an excess of carbon which precipitates as graphite flakes in a Be_2C matrix. (See Figure 4.) In one case, graphite flakes were formed by heating "pure" Be_2C (no free carbon) to 2200 C. The vermiculate structure appears to have been formed by pressure from within. The fact that the tubes distort and conform to the contour of the crucible wall is evidence that incipient melting has occurred. However, this is not a useful melting point as the material cannot be cast at atmospheric pressure.

Samples of Be_2C powder contained in graphite capsules and heated in argon for 30 minutes at 2400 C formed a clinker-like product. Incipient fusion appeared to have taken place. At 2300 C, some sintering occurred but no fusion. When similar

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samples were heated in closed graphite capsules in a vacuum at $>2100^{\circ}\text{C}$, Be_2C sublimed and condensed as red crystals on the lid (2100°C) of the capsule which was slightly cooler than the bottom of the capsule. This sublimation seems to take place through dissociation of the carbide to its elements and recombination at a favorable temperature. A similar experiment at slightly higher temperature (2120°C on the lid) yielded metallic-appearing crystals. The metallic appearance was caused by a thin layer of vaporized carbon on the surface of the carbide crystals. It is apparent that, if Be_2C is to be melted, it must be done at high pressures in an inert atmosphere.

Chemical Analysis

The various lots of Be_2C were analyzed for total and free carbon and soluble beryllium. The percentage of Be_2C was calculated from these figures. A list of the typical chemical analyses appears in Table 2. Usually, a mineral balance (free carbon plus Be_2C) of about 98 per cent was obtained. The rest may be accounted for as BeO , as evidenced by the X-ray diffraction analysis and $\text{Be}(\text{OH})_2$, small amounts of which form by hydrolysis of Be_2C with atmospheric moisture. Total carbon was determined by combustion of a sample of Be_2C in O_2 at 2500°F for 15 minutes.

Ingot iron was added as a flux for the BeO , combustion product, which otherwise tends to protect the carbide from complete oxidation. The carbon was collected as CO_2 and weighed. The total carbon figure was not always reliable because, even with the precautions taken, the analyses appear low and often did not agree with the calculated Be_2C .

Soluble beryllium was determined by hydrolyzing Be_2C in 2.5 N HCl near the boiling point until hydrocarbon evolution, as evidenced by bubble formation, ceased. The free carbon was filtered off and determined by combustion as above.

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The free-carbon analyses appear reliable. Microscopic estimates of carbon are sometimes low. This is probably caused in part by the presence of colloidal carbon within the Be_2C crystals. This is measured by chemical means but not by the microscope. For the soluble-beryllium analysis, it was assumed that the BeO , which was highly fired during production of the Be_2C , was insoluble. The presence of unreacted beryllium was ruled out because of the high vapor pressure (about 0.4 cm Hg) at the minimum reaction temperature, 1700 C. The only other soluble beryllium compound likely to be present was $\text{Be}(\text{OH})_2$. In our early work, no analysis was made for H_2O and all soluble beryllium was assumed to be Be_2C . Later it was found that about 1 per cent H_2O was picked up during handling even though the material was stored in tightly sealed containers immediately after being produced. Assuming that 1 per cent H_2O is present as $\text{BeO} \cdot \text{H}_2\text{O}$ (2.39 per cent), then the Be_2C value is 0.833 per cent high and the total assay 1.556 per cent low. Thus, the BeO shown to be present by X-ray diffraction and the small amount of $\text{Be}(\text{OH})_2$ which is probably present in all samples account for the difference between the total of the free carbon and Be_2C and 100 per cent.

As indicated above, Be_2C has a strong tendency to hydrolyze. This reaction takes place according to the following reaction:

Hydrolysis has been particularly noticeable in connection with vermiculate (high temperature) carbide. Hard clinker-like lumps of this material, which were very difficult to break when first made, crumbled into small grains after exposure to air for one or two weeks. Samples of the clinker mounted in Bakelite hydrolyzed after several days in air and swelled to such an extent that the plastic was cracked into several pieces. Typical lots of -30 mesh Be_2C , as made and stored in covered fruit jars for several days, analyzed 1.0 wt % H_2O . A sample of Be_2C crystals

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(Figure 2A), when extracted from Fe metal with dilute HCl, washed with H_2O , and dried with ether, analyzed 6.5 per cent H_2O . Hydrolysis appears unavoidable in this process. A sample of vermiculate material, which had hydrolyzed in air, was analyzed in the hydrolyzed condition and after drying at various temperatures. Before drying, it contained 4.2 per cent H_2O ; drying at 100 C for one hour reduced this but slightly to 3.7 per cent. Even drying at 400 C for one hour left appreciable H_2O , 0.71 per cent. When the 400 C treatment was followed by an hour at 650 C, the H_2O fell to 0.20 per cent. An additional hour at 800 C practically completed the drying, leaving only 0.09 per cent H_2O . The H_2O analysis was made by collecting the H_2O evolved when Ba_2C was heated at 1000 C for 15 minutes in a slow stream of dry oxygen.

It appears that certain porosity of beryllium castings, made in previously used graphite molds, may be attributed to hydrogen from hydrolysis products of Ba_2C . Ba_2C formed on the surface of the mold by previous castings may hydrolyze and, in spite of preheating to 500 F (316 C), may retain sufficient moisture to cause porosity. Preheating to at least 1000 C is recommended. After drying, the mold may, of course, be cooled to a lower temperature for use.

X-Ray Diffraction Analysis

In general, the X-ray diffraction analyses of the major phases agreed with chemical analyses and with observations made using the microscope. Constituents present in quantities less than 5 to 10 per cent are often not detectable by X-ray methods. However, in this series of analyses, BaO as low as 1 per cent was detected. $Ba(OH)_2$ was found in nearly all the samples examined by X-ray diffraction. This is probably, in part, the result of hydrolysis of the Ba_2C during preparation of samples for X-ray analysis. The density of Ba_2C determined from lattice parameter measurements agrees with the value $d = 2.44$, as reported by Stackelberg⁽⁴⁾.

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MICROSCOPY OF BERYLLIUM CARBIDE

Examination of beryllium carbide under the petrographic microscope gave much useful information regarding the nature of this compound. The various phases present in the usual "as made" material were identified and percentage estimates made by area measurement. An attempt was made to convert area measurements to weight percentages by use of conversion factors based on the apparent density of the constituents. The minimum, maximum, and average size of the carbide crystals were determined, and the physical nature of the crystals of typical lots were assigned on the basis of crystal-size measurement. The results of microscopical examination, chemical analyses, and X-ray diffraction analysis are summarized in Table 2. The agreement of the results by the three methods is self-evident.

Identification of Phases

As one would expect in consideration of its strong tendency to hydrolyze, beryllium carbide does not occur in nature. No optical data on beryllium carbide were found in the literature. It was found to crystallize in the isometric system and to exhibit an amber color in either reflected or transmitted light. The color in reflected light tends to be somewhat more red; whereas, in transmitted light, the color is slightly more brown. Examined in plane polarized, transmitted light, the crystals are translucent. They are isotropic and so appear dark in polarized light between crossed nicols. A typical lot of coarsely crystalline Be_2C , produced from the metal-carbon reaction, is readily crumbled to minus 30-mesh grain size in the hand. The individual particles thus produced are, in certain cases to a large extent, single crystals.

Because Be_2C crystallizes in the isometric system, it has only one index of refraction value. Through use of fused solid melts containing mixtures of selenium and sulfur, the average index of refraction of crystalline Be_2C was found

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to be $n = 2.64_{L1} \pm 0.01_{L1}$. White light was employed for making the index of refraction measurements because monochromatic yellow, sodium vapor light was absorbed within the dark-red embedding medium. Therefore, the refractive index measurements were made by means of the red wave band portion of the transmitted white light.

In addition to the carbide phase, the usual lot of "as made" beryllium carbide may contain free carbon, free beryllium metal, BeO, and Be(OH)₂. It was not found possible to distinguish between beryllium metal and the black, opaque carbon particles. It may be possible that this could be done by some technique, such as mounting the material in clear plastic and examining polished sections under reflected light. However, if free metal occurred in the usual product, it must have been very minor compared to free carbon, as evidenced by X-ray examination data.

Area measurements of free carbon, including graphite flakes in the Be₂C matrix, were made. In order to convert these measurements to weight percentages for comparison purposes, a conversion factor for carbon was first determined. Comparison of microscopical area measurements and chemical free-carbon analyses of 30 samples of the product of the metal-carbon reaction indicated that, if the percentage of carbon by area measurement were divided by 1.6 to convert area percentage to weight percentage, satisfactory agreement between microscopical and chemical methods would be obtained. Having assigned a weight percentage value to carbon by this means, the area measurements of Be₂C and Be(OH)₂ were converted to weight percentage on the basis of their relative densities. Carbon could not be corrected on the basis of density alone because it is evidently present as porous, bulky particles of low apparent density. The carbon of materials from the high-temperature BeO-carbon reaction was even bulkier than that of the metal-carbon reaction and required use of a conversion factor of 3.0. A possible explanation of the difference is that the

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carbon of the metal-carbon reaction product is unreacted and may be considered as rough spheres comparable to the shape of the Be_2C particles, while the carbon in the BeO-carbon reaction product, which is present principally in the form of thin graphite flakes within the Be_2C matrix, retains some of its flaky nature even when ground to <10 microns and so covers a greater area than an equal weight of spherical particles.

BeO was occasionally present as prismatic crystals that were birefringent in polarized light between crossed nicols. It apparently formed in the cooler regions of the charge because of incomplete shielding by the argon atmosphere.

$\text{Be}(\text{OH})_2$ was formed through hydrolysis of Be_2C by the moisture in the air. Some carbide products appeared more subject to hydrolysis than others. In the typical hydrolyzed material, $\text{Be}(\text{OH})_2$ completely enveloped the Be_2C crystals. The hydroxide appeared to possess a cryptocrystalline, fibrous structure that was birefringent in polarized light between crossed nicols.

Crystallography of Beryllium Carbide

The crystallographic properties of Be_2C appear to resemble closely those of fluorite (CaF_2). Euhedral (Be_2C) occurs as octahedrons, dodecahedrons, tetrahedrons or as truncated octahedrons containing the usual 8 octahedral faces plus 6 "cubic" pinacoid faces, adding up to 14 faces in the completed form. Twinning of the truncated octahedrons can occur parallel to the "cubic" pinacoid face to form "accordion-like" structures. The crystalline phase of Be_2C exhibited perfect octahedral cleavage along four directions. Twinning of Be_2C occurred only in high-temperature, coarsely crystalline products. Threelings were the highest order of twinning observed to date, although fourlings are possible if all conditions for stable crystal twinning symmetry were satisfied. The threelings were joined together through two 45-degree-angle bends. Simple, contact-twin crystals of the

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Carlsbad or Siamese twin type were observed, as well as twolings composed of two crystals joined together at a 45-degree angle. The crystals also tended to form an open link-chain type structure composed of from 5 to 25 euhedral or subhedral crystals that adhere together in a continuous, irregular chain. Also, a closed-chain type of structure occasionally occurred in which the individual crystals formed densely packed, parallel rows, having the appearance of a beaded fabric. Both porous and very dense nonporous chain-type structures were observed.

Other crystal structures include the individual euhedral and subhedral crystals mentioned previously and coarsely crystalline intergranular aggregates of these crystals. In addition, there is the microcrystalline "grape cluster" or nodular type of crystal aggregate that predominates in beryllium carbide produced by the metal-carbon reaction at relatively low reaction temperatures (1700 C). The "grape clusters" are composed of anhedral and subhedral crystals which form dense and non-porous aggregates.

Colored Photographs of Beryllium Carbide

Because of the difficulty of accurately describing in words the colors of beryllium carbide particles, a technique was worked out by which the appearance of several representative lots of Be_2C were recorded in natural color on Iltochrome transparencies. A sample of the material was suspended in a cilanthien red Y dye solution in nitrobenzene on a glass slide. Limited transmitted light produced a light-brown background. Peripheral lighting from small fluorescent tubes illuminated the translucent Be_2C grains. The exposures were made with a Bausch & Lomb research metallograph, each exposure requiring approximately 45 minutes. In the colored transparencies, one can distinguish between translucent, amber Be_2C , the black opaque unreacted carbon, and precipitated flakes of graphite occurring within the Be_2C

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crystals. Occasional grains of Be_2C exhibited an olive-green color, apparently caused by the presence of precipitated colloidal carbon. In other Be_2C grains, the greenish color may be caused by the thinness of section.

HARDNESS MEASUREMENTS

Coarse crystals of Be_2C were mounted in Lucite and polished. Knoop hardness numbers were then taken by means of a Tukon hardness tester. When a 100-gram load was used, the crystals shattered or chipped away at the impression. Satisfactory readings were taken with lighter loads. Readings of 2678, 2520, and 2613 were obtained with a 50-gram load, and 2740 with a 25-gram load. It appears that the hardness of Be_2C is greater than that of silicon carbide and approaches the hardness of boron carbide.

CONCLUSIONS

Be_2C may be made by several methods, principal of which are the BeO -carbon and the Be -metal-carbon reactions. The metal-carbon reaction appears better when controlled crystal size is desired. The true translucent amber color of Be_2C is revealed only under the microscope. As a refractory, it has several disadvantages. It tends to hydrolyze even in atmospheric moisture. In heating, Be_2C must be protected against both oxygen and nitrogen. It decomposes at about 2150 C with vaporization of the beryllium which reunites with carbon in cooler regions or with oxygen or nitrogen if present. Be_2C has no useful melting point at atmospheric pressure.

ACKNOWLEDGMENT

This work was performed at Battelle Memorial Institute under the sponsorship of the NEFA Division of the Fairchild Engine and Airplane Corporation.

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May 20, 1953

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TABLE 1. THE PREPARATION AND CRYSTAL PROPERTIES
OF BERYLLIUM CARBIDE

Lot* Number	Preparation of Material		Crystal Sizes (Microns)			Crystal Habit	
	Time, Minutes	Temperature, C	Variation		Average Size	Per Cent of "Grape-Cluster" Finely Crystalline Nodules in Calcined Powder	Per Cent of Individual Crystal Grains Plus Crystals in Calcined Powder
			Min.	Max.			
1	30	1700	0.5	32	5	98-99	<2
2	30	2200	3.0	95	42	5	95
3	30	2020	0.5	50	13	90-95	5-10
4	30	2020	0.5	33	9	98-99	<2
5	30	2020	0.5	47	12	93-95	5-7
6	50	2020	1.0	249	82	5	95
7	15	2020	-	-	-	-	-
	+ 30	2200	4.	230	83	Traces Only	98-99
8	15	2020	-	-	-	-	-
	+ 30	2200	1.	154	58	2-5	95-98

*These products were made by the metal-carbon reaction using the stoichiometric ratio for Be₂C.

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TABLE 2. ANALYTICAL DATA ON BERYLLIUM CARBIDE

Petrographic Analysis*					Chemical Analysis				X-Ray Diffraction Analysis		
Adjusted Weight, % Mineral Composition After Applying Ratio Factors					Total Carbon (C), %	Free, Uncom- bined Carbon (C), %	Soluble Beryllium Compound (Be), %	Calculated Beryllium Carbide (Be ₂ C), %			
Lot Number	Beryllium Carbide (Be ₂ C)	Carbon (C)	Beryllium Oxide (BeO)	Beryllium Hydroxide (Be(OH) ₂)							
1	91.5	7.5	Trace**	None	44.8	9.6	53.1	88.5	S	VVF	MF
2	92.4	5.6	None	Trace**	45.3	10.2	53.6	89.4	VS	VVF	H
3	—	14.1	—	—	41.0	11.5	51.7	86.1	S	VF	MS
4	73.4	21.9	1.2	3.8	50.4	23.2	43.8	72.7	S	VF	VS
5	88.0	9.7	0.6	1.8	37.5	5.0	56.0	93.4	VVS	MF	S
6	—	9.1	—	—	39.2	9.0	52.8	87.9	VS	—	H
7	—	7.8	—	—	39.0	11.6	51.5	85.8	H	—	H
8	—	4.9	—	—	39.6	7.5	55.1	91.8	VS	MF	H

* Two lots analyzed for beryllium silicate (2 BeO-SiO₂) showed none in one case and a trace to <1% in the other instance.

** Less than 1%, but visible.

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FIGURE 1. FURNACE SETUP FOR METAL-CARBON REACTION

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500X

53705

A.

500X

53581

B.

500X

55585

C.

FIGURE 2. Be₂C CRYSTALS IN A BERYLLIUM METAL MATRIX, ILLUSTRATING DIFFERENCES IN PARTICLE SIZE

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A. Core of high-temperature heat

54994

B. Individual tubes showing hollowness

54995

FIGURE 3. VERMICULATE Be_2C

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250X

55657

A. Unetched

250X

56015

B. Air etched

FIGURE 4. GRAPHITE FLAKES IN A Be_2C MATRIX

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END