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**Characterization of Commercial
Carbide Powders**

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by

R. E. Riley

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CHARACTERIZATION OF COMMERCIAL

CARBIDE POWDERS

by

R. E. Riley

ABSTRACT

The problems associated with the development of a procurement specification for commercial carbide powders through powder characterization are reported. The morphological types of structures observed in commercial NbC, TaC, and ZrC powders are discussed. Also given are the results obtained for the various parameters determined to characterize powders. A technique for using these parameters in developing a procurement specification for a carbide powder to meet a particular requirement is proposed.

INTRODUCTION

A development program on powder metallurgical processing of carbide-carbon composites was undertaken by the Los Alamos Scientific Laboratory (LASL). The aim of the program was to determine the physical properties of this new composite structure. Preliminary results indicated wide scatter bands and anomalies, and, at this point, a review of powders used to process the test samples was initiated. This evolved into an extensive powder evaluation program which is the subject of this report.

The characteristics of powders influence their behavior in powder metallurgy processing. Numerous mixing theories and size and shape factor formulations have been developed, but, unfortunately, they have been of little or no value to the investigator in his development of practical and reproducible applications of commercial powders. The characterization of refractory carbide powders obtained from vendors has proven extremely difficult. The lack of a meaningful procurement specification coupled with the fact that a variety of morphological types

are obtainable from a single vendor, as well as from one vendor to the next, has complicated the work still further. After discussing these factors with vendors who expressed an interest in the problem of powder characterization, it now is possible to explain some of the reasons for the existence of some of the various morphological types. The report shows that one can, indeed, develop a meaningful procurement specification for a particular powder requirement if one has this information and analyzes characterization data on many lots of commercially available powders.

RAW MATERIALS

The powder analyzed for this investigation represented 42 lots of NbC, 14 lots of TaC, and 6 lots of ZrC which were obtained from various vendors. The lot numbers and chemical analyses of these powders are summarized in Table I and the vendors are coded by a letter designation in Table II. (Both tables are at the end of this report.)

POWDER CHARACTERIZATION

The need for full characterization of powders was recognized early in a related powder metallurgical program on refractory carbide powders, and considerably more work in the area of powder characterization is an absolute must. Different sampling techniques were employed, depending on the size of the powder lot to be evaluated, e.g., a 5-lb bottle as compared to a 50- to 100-lb container. The sample assumed to be representative of the powder lot was blended for 1/2 h, making sure that the sample bottle was not over three-fourths full. Sufficient material was then extracted from the blend for the various evaluation tests. The characterization data obtained for the various powder lots are summarized in Table II.

Fisher Sub-Sieve Size

The limitations of the Fisher analysis have long been known to the powder metallurgist, but its rapid determination of the approximate average particle size of a powder has advantages when the results are related to other parameters such as bulk, tap density, or both. The as-received powder was evaluated per ASTM-B-330-65, after which it was ball-milled for 30 min and re-evaluated according to ASTM-B-430-65-T.

The Fisher sub-sieve sizer (Fig. 1) is a commercial form of permeability apparatus developed by Gooden and Smith.¹ The basic principle of this equipment is the measurement of the resistance of a bed of packed powder to the flow of a gas. The quantity actually being determined is the total surface area of the powder sample which is converted to "average particle size" through the use of a chart furnished with the equipment. No information about particle size distribution is obtained from this test.

Bulk (Apparent) Density

This test is to determine the apparent density by means of a Scott volumeter in accordance with the procedure described in ASTM-B-329-61. Specifically, the powder is poured into a baffle box where it falls into a funnel which directs the flow into a cup whose volume is $1.0 \pm 0.002 \text{ in.}^3$. The powder is leveled with a spatula, the side of the cup is tapped gently, and the full cup is weighed to the

Fig. 1. Fisher sub-sieve sizer.

nearest 0.1 g.

Tap Density

The tap density is a measure of the packing characteristics of a loose powder. A 10-cc graduate is weighed to the nearest 0.01 g, and approximately 10 cc of powder are carefully poured with a minimum of vibration. The volume is measured and noted, after which the total weight is determined and the net weight of the powder recorded. The graduate is slowly rotated while being tapped with a plastic rod, reducing the apparent volume until further tapping causes no further decrease. The volume is then recorded, and the tap density determined by

dividing the weight of the powder by the final volume after tapping.

Particle Size Distribution

In powder metallurgy, the physical characteristics of powders are important. Of primary interest are particle size distribution and the related property, specific surface area, which will be discussed later.

Sedimentation methods, in general, determine particle size distribution by the application of Stokes Law to the settling of particles in a viscous medium. The sedimentation balance used in this work was made by Sartorius-Werke (Fig. 2) and consists of a cylindrical vessel with a balance pan at the bottom. The pan is connected to an automatic recording balance. A homogeneous dispersion of sample and settling medium is introduced into the cylinder, and the sample is allowed to settle on the pan. The balance records a continuous, settled weight of material as a function of time. Using the Stokes Law of settling, the time scale can be converted to an equivalent particle diameter. Differentiation of the weight-time curve gives the particle size distribution curve as cumulative weight percent vs d , where d is assumed to be the length of a cube edge rather than the diameter of a sphere.

Metallography

Metallography is one of the most useful evaluation tools in the characterization of powders. It was used to examine powders for particle size and shape, and soundness and cleanness of structure. The procedure for preparation of powder samples for metallographic examination is detailed in Reference 2. Briefly, the powder sample is poured into the cavity of a machined epoxy disc which is inserted into a silicone rubber mold. A heated pre-mixed solution of Epon 828, an epoxy resin manufactured by Shell Chemical Company, and DEA, a diethanolamine manufactured by J. T. Baker Chemical Company, is poured into the mold and over the powder. The powder and the epoxy solution are stirred to ensure bonding of the powder and epoxy during hardening. The hardened mount is prepared for examination by standard metallographic techniques.

Fig. 2. Sedimentation balance.

Surface Area

Specific surface area was determined by two techniques. The Fisher readings may be converted to specific surface by the formula:

$$S_w = \frac{6}{\rho d_m}$$

where: ρ = powder density in g/cc

and d_m = Fisher average particle diameter in μ .

This formula gives S_w in m^2/g .

Specific surface measurements are also being made using a NUMEC Model AFA-2 unit (Fig. 3) which utilizes low-temperature gas adsorption methods and was designed to be used either with the single-point Innes method³ or the multiple-point Brunauer, Emmet, Teller (BET) method.⁴

The principle of operation for either method is the measurement of the volume of nitrogen gas required to form a monomolecular layer when adsorbed on the surface of the sample at liquid nitrogen temperature. From a knowledge of the area occupied by each molecule, the total surface area of the sample may be calculated.

The original BET analytical method is an extension of the Langmuir theory which assumed that molecules were adsorbed only in a monolayer. The BET method considers that multimolecular layers are possible. When a plot is made of quantity of gas adsorbed vs relative pressure at constant temperature, the adsorption isotherm is obtained. Over a

Fig. 3. Numec Model AFA-2 specific surface unit.

certain range of equilibrium pressures, this plot is essentially linear, and its slope and intercept are used to determine the volume of a monomolecular layer.

The Innes method is an approximation to the BET method designed to reduce the time required for a determination. In this method a slow, constant flow of gas is maintained so that equilibrium pressure is approximated, and the amount of gas adsorbed is proportional to time. The surface area is calculated from the time required to reach a predetermined relative pressure. The Innes method is considerably quicker; however, it was found that the BET method, while more time consuming, was considerably more accurate. Therefore, the BET method was used in all determinations made in this investigation.

The calculations were programmed on the LASL MANIAC computer to save time. In contrast to other methods of specific surface area determinations,⁵ the adsorption method actually measured the total exposed area, including all cracks and fissures that are open to the surface.

In the past, for a powder having a particular Fisher number, there appeared to be no correlation between specific surface and Fisher average particle size whether the specific surface was determined by the BET or the Fisher technique. The current data which are discussed later show that a correlation is indicated.

DISCUSSION

H. H. Hausner⁶ has very descriptively summarized the characteristics which influence the behavior of a metal powder.

Particle shape, size, and specific surface of a powdered material greatly influence its behavior for reasons which are not generally understood by the applications engineer. This influence on behavior is not recognized primarily because such parameters as size, shape, and specific surface of the powders in question are not clearly defined. These factors, along with the complications imposed by the effects of particle size distribution, compound the problems encountered by the fabricator.

Most theories on the behavior of powders do not take these factors into serious consideration, but apply simple parameters such as those for spherical or square particle shapes of a fully dense material, all of the exact same size. These factors sometimes average out, as in the case of the Lewis-Goldman⁷ mixing theory, providing that the powders being combined are dense and of a normal distribution. However, as will be shown, particle density can vary considerably, and, depending on the manufacturing process employed (which differs among vendors), a variety of morphological types of structures are possible in powders received from the various vendors and, in some cases, from the same vendor. The author feels that if the characterization data for the various morphological types are separated on the basis of morphology, they will better fit the hypotheses presented by the theoreticians.

H. E. Rose⁸ has shown that it is not possible to define particle shape by a single parameter. In practice, however, most granular materials have a restricted range of forms and it is possible to define their shape with an accuracy sufficient for many purposes. However, this still does not explain many of the anomalies encountered by the applications engineer trying to employ these factors in making a product.

The morphology of a powder may be influenced by the various manufacturing steps employed. The starting raw material may be a controlling factor; the chemical reaction itself may be significant and, in turn, may be complicated by the impurities in the raw materials or the system; the batch size and the type of equipment used may be important; and the method of comminution may affect the size, shape, and distribution of the particles as well as the types and levels of impurities in the powders.

A metallographic study of powders by T. I. Jones² included a measure of the size, shape, and particle distribution of many powders and, more specifically, the presence of extraneous phases. This report is not concerned with those factors of morphology involving extraneous phases, but attempts to identify some of the morphological types encountered in commercial powders and, where possible, to identify the manufacturing technique employed.

At the beginning of the program on carbide-graphite composites at IASL, a single lot of powder (TaC-13A) was used. Weighed quantities of the carbide were blended with various quantities of graphite, loaded into graphite dies, and hot pressed at temperatures of about 3000°C and pressures ranging from 2000 to 5000 psi. This powder had a Fisher average particle size of 2.0 μ , and approximately 50% of its particles were less than 2.0 μ in diameter. It was not determined until later that the large percentage of fines had a tendency to agglomerate during blending and hot pressing, producing a non-uniform distribution of very large carbide platelets. This resulted in highly directional properties in the as-pressed product and presented serious machining problems.

A second lot of powder, NbC-9 (Fig. 4), was selected for the work that immediately followed and

Fig. 4. NbC-9 control lot. 250X.

was subsequently used as a control lot. As the composite program expanded, it became necessary to procure more powder. Sample lots (NbC-10A through -10E) were received from five different vendors in accordance with the following procurement specification: NbC powder, -325 mesh, highest purity available, nominal Fisher sub-sieve size of 2.0 to 2.5 μ . Information upon which to base a more detailed specification was not available at that time.

The distribution curves of these vendor evaluation lots presented in Fig. 5 vary significantly from that of lot NbC-9. This difference is also reflected in the metallographic structures of these samples shown in Fig. 6.

Based on our very loose specification at the time of purchase of these evaluation lots and careful consideration of price quotations, delivery times, and metallographic and other powder characterization results, it was decided to evaluate NbC powders from Vendor "E." The sample powder lot supplied by Vendor "E" most nearly met our specification. Figure 7 shows the differences among six additional lots of powder from Vendor "E." There were differences not only in the physical characteristics but also in the chemistry (See Table I), even though all of the powder lots were represented as being similar by the vendor.

Until this time, the powder characterization data had little meaning, and it became apparent that the morphology of these powders might be the key to understanding the data. Therefore, all ZrC, NbC, and TaC powder lots on hand were sampled and examined metallographically. Comparison of morphological types from several lots of powders showed that 250X magnification was best suited for this work.

Historically, the internal characteristics (morphology) of the powders have not been considered in the numerous blending and mixing hypotheses extended by the theoreticians. However, it is the opinion of the author that if morphology were incorporated into the theoretical calculations, they would become more useful to the applications engineer. The morphology of the powder is just as important as the particle size, shape, and distribution in producing a product which is uniform and reproducible and which meets specifications.

The powder characterization data (which represented approximately one-third of the powder lots

presented in Tables I and II) enabled a revised specification to be written based on the observation that powders of a particular Fisher average particle size and having a minimum bulk and tap density would have the same morphological structure. This revised specification for NbC powder is as follows:

-325 mesh NbC powder - highest purity available, nominal Fisher sub-sieve size of 3.5 to 4.0 μ . Material must have a minimum bulk density of 2.7 g/cc and a minimum tap density of 4.25 g/cc.

Additional powder lots were procured, and the morphology and powder characterization study continued. The Fisher average particle size specification was increased in hopes of having fewer fines (particles less than 2 μ) in the as-received powders. In the past the percentage of particles less than 2 μ in size varied from less than 5% to more than 20%.

The particle size distribution curves for the new NbC powders are too numerous to include in this report; however, the increase in Fisher average particle size did not significantly decrease the quantity of fines from lot to lot of as-received powder. (See the typical distribution curves in Fig. 8.)

The various types of structures resulting from the different techniques used to manufacture commercial carbides may have a profound effect on the mechanical and physical properties of a fabricated part. The factors evaluated for the various types of structures are: 1) Density - particles are either solid or porous; 2) Structure - a discrete particle or an agglomerate of fines; and 3) Size and shape of the particles.

The approach at LASL was to try to provide a basis for identification of the morphological types observed in commercial as-received carbide powders. This investigation involves only those morphological types of structures and shapes associated with the manufacturing techniques used, and does not involve the morphology of second phases, oxide films, and vestiges of segregate.

With these factors in mind, one is ready to identify the basic types of structures observed at LASL. The first morphological type, "Type A" (Fig. 9), is a porous particle which is apparently the result of the manufacturing technique employed. The menstrum process, in which the particle is formed in a molten metal bath, was used in making this powder.

Fig. 5. Particle size distribution curves of selective NbC evaluation powder lots.

NbC-10A

NbC-10B

NbC-10C

NbC-10D

NbC-10E

Fig. 6. Microstructure of NbC powders from various vendor evaluation lots. 250X.

NbC-6

NbC-7

NbC-11

NbC-12

NbC-13

NbC-17

Fig. 7. Microstructure of various lots of NbC from a single vendor. 250X.

Fig. 8. Particle size distribution of selective NbC powder lots.

This powder (Lot NbC-14) had a Fisher average particle size of 12.7μ which was much coarser than most of the powders investigated. It is not known whether size had any bearing on the porosity of this powder, but another lot of powder of much lower average particle size but manufactured by the same technique yielded a very dense particle of quite uniform size.

"Type B" (Fig. 10) may be described as a dense particle of either rounded or angular shape.

"Type C", shown in Fig. 11, is similar to "Type A" in that both are porous. But while "Type A" is a discrete particle resulting from the manufacturing technique employed, "Type C" is an aggregate particle made up of very many fines.

A fourth type of structure was observed and was designated "Type D" (Fig. 12). Discussions with the technical representatives of two vendors supplying such a product indicated that the process of carbothermic reduction of the oxide, depending on the temperature and time involved, yielded a variety of structures. However, the as-reacted product tends to be of "Type D" morphology.

In most cases the vendors can alter these structures by changes in their manufacturing processes. Depending on the process, a variety of combinations can exist. Types "B" and "D", for example, were made by the same process, but the "Type D" structure is the result of a single-step process and represents the as-reacted structure after crushing. The "Type B" structure, on the other hand, can be obtained from the "Type D" product by an additional one or two high temperature sintering treatments. Both types are available from most vendors. One vendor gets the "Type B" structure from a single carbothermic reaction step, while another has to sinter once or twice. The reason for the difference between the two vendors' products is not known since they both use the thermite process.

Identification of the morphological types noted appeared to be the answer to some of the problems encountered in fabrication of carbide-graphite composites. However, another anomaly arose in that although powder lots NbC-13 and NbC-17 had the desired morphology, the results of the high temperature deformation test of composites made from them were greater than those for composite samples prepared from other NbC powder lots. The high temperature deformation test consists of loading a specimen to 2000 psi at 2700°C and holding for 30 min. The changes in dimension are then measured and used as a measure of the high temperature creep characteristics of the composite.

Since the fabrication procedures⁹ were essentially identical, it was believed that this difference in resistance to deformation was due to one or more contaminants in the as-received NbC powders. A quick look at the analytical results showed that Lots NbC-13 and NbC-17, which showed the least resistance to deformation, were substoichiometric, higher in oxygen and higher in iron content, than the other powder lots.

It was decided to plot the deformation results as a function of the various contaminants. The only trend as a function of increased contaminants was shown for iron. Figure 13, a plot of deformation for the various lots, shows an increase in deformation with increasing iron content.

This effect of iron, of course, was very undesirable, and our immediate concern was whether the

Fig. 9. Morphological structure "Type A." 1000X.

Fig. 10. Morphological structure "Type B." 1000 X.

Fig. 11. Morphological structure "Type C." 1000X.

Fig. 12. Morphological structure "Type D." 1000X.

TESTED AT 2700 °C, 2000 psi, 30 min.

Fig. 13. Effect of iron content in the as-received powders on resistance to deformation at elevated temperatures.

iron was combined in solution in the NbC or was present as a thin film on the particles from the crushing and milling operation. A 500-g sample from each of these two lots highest in iron was acid leached in hot HCl:H₂O-1:1, washed, and dried. Composite specimens of the same composition were hot pressed as before and deformation tested. It is apparent from the test results, also shown in Fig. 13, that the iron was present primarily as a result of the comminution process.

The effect of contaminants on high temperature deformation of composites is the subject of another IASL report.¹⁰ It is enough here to point out another effect related to particle size, namely, that the leaching operation changed the particle size distribution of the NbC powder significantly. The results are plotted in Fig. 14. It is apparent that the average particle size is reduced somewhat by the leaching operation and that a large amount of fines (particles less than 2 μ) are generated. This increase in fines created danger of undesirable aggregate formation during the blending operation.

The evaluation of many lots of NbC powder led to another revision in the NbC powder specification, and the specification, currently in use, is as follows:

-325 mesh NbC powder, highest purity available. The material shall have a nominal Fisher subsieve size of 4.0 to 5.0 μ , a minimum bulk density of 2.7 g/cc, and a minimum tap density of 4.25 g/cc. The iron content shall not be greater than 300 ppm, and the total of all other metallic contaminants shall not exceed 500 ppm.

A 5-lb sample shall be evaluated at IASL for qualification and characterization test before shipment of the remaining quantity will be approved.

Additional lots of powder were procured to this latest specification and evaluated. If the powders met the requirements of minimum bulk and tap density for a particular Fisher average particle size, the powder had the desired (Type B) morphology. This confirmed earlier observations and demonstrated the significance of morphology in development of a procurement specification even though it is not used in the specification supplied to the vendor. In fact, it is to be noted that the specification

Fig. 14. Effect on particle size distribution of leaching to remove iron.

includes only those parameters that can be determined routinely by the vendors in their manufacturing quality control.

The results of the study of powder morphology shed some light on a better correlation and understanding of the various characterization data determined. It was mentioned earlier that considerable scatter in the characterization data made it difficult and sometimes impossible to show a correlation between the different parameters determined. The work of A. L. Hipp¹¹ showed that the values for specific surface areas of tungsten powders obtained from the Fisher apparatus and calculated from the Sedibal data were remarkably consistent, considering the different principles involved. On the other hand, Hipp's work showed a comparison of the data on specific surface areas from the BET apparatus and the Sedibal data showed considerably more variation. The values obtained by the BET method were from three to 10 times greater than those obtained by other means. A similar trend was noted in the values of specific surface area obtained by the latter two methods for the powders discussed in this report.

A correlation between the various powder parameters seems apparent if one considers the morphology of the powder to be used. While more data on powders of varying average particle size and morphology will be necessary to confirm the observations reported herein, a significant correlation seems to exist, as will be illustrated in the following discussion.

A plot of bulk and tap density vs Fisher average particle size for the NbC powders of differing morphology is presented in Fig. 15. A scatter band is drawn to show the range of values obtained for the "Type B" powders. The breadth of this band is controlled by the particle size distribution and the variability of porosity within the powders.

Figure 16 is a plot of specific surface area as a function of Fisher average particle size. If one draws the scatter band for the "Type B" powders, a correlation exists. Again, if one studies all the data carefully, the breadth of this band is controlled by the particle size distribution and the porosity within the powders. Although only a limited amount of data was available for TaC powder, a similar correlation appears evident as is shown in Fig. 17. There was insufficient data on the ZrC

Fig. 15. Tap and bulk density vs Fisher average particle size for all NbC powder lots.

powders to show a similar plot; however, it is significant that two of the four morphological types observed to date were seen in these six powder lots.

SUMMARY AND CONCLUSIONS

A large amount of analytical and characterization data on ZrC, TaC, and NbC powders has been generated and presented in this report. While certain observations can be made after careful study of all of the data, this work is only a very small contribution to better understanding and practical application of the characterization data. The conclusions, relative to our aim, derived from this work follow.

1. Four basic morphological types of powders have been identified and associated with the manufacturing process used to make each powder.
2. Specifications of particular Fisher average particle size and minimum bulk and tap densities

Fig. 16. Specific surface area vs Fisher average particle size for all NbC powder lots.

resulted in powders having the desirable morphology relative to particle size, shape, and size distribution.

3. Using the data for a particular morphological structure, one can, indeed, show a correlation with a minimum scatter band when comparing the various characteristic parameters determined.

4. The chemical purity of the powder is important to the final high temperature properties in the fabricated product.

5. The requirements of Fisher average particle size, bulk, and tap density must be met by the final leached high purity powders because the particle

size can be influenced significantly by the leaching process.

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Fig. 17. Specific surface area vs Fisher average particle size for all TaC powder lots.

TABLE I

CHEMICAL ANALYSES OF COMMERCIAL CARBIDE POWDERS*

Niobium Carbide - NbC

<u>Elements</u>	<u>Lot 6</u>	<u>Lot 7</u>	<u>Lot 9</u>	<u>Lot 10a</u>	<u>Lot 10B</u>	<u>Lot 10C</u>	<u>Lot 10D</u>	<u>Lot 10E</u>	<u>Lot 11</u>
Nb	88.7%	88.2%	88.4%	88.4%	88.3%	88.7% $\pm$ 0.2%	88.7% $\pm$ 0.2%	88.5% $\pm$ 0.2%	88.6% $\pm$ 0.2%
Total C	11.2%	11.26%	11.08%	11.3%	11.2%	11.2%	11.2%	11.7%	11.51%
Free C	0.21%	0.23%	500 ppm	0.1%	0.1%	0.07%	0.12%	0.58%	0.47%
O ₂	0.64%	0.17%	895 ppm-0.21%	0.25%	0.19%	0.12%	0.27%	680 ppm-0.11%	365 ppm-0.11%
Li	3	3	3	3	3	3	3	3	3
Be	1	1	1	1	1	1	1	1	1
B	1	1	1	10	1	3	1	1	1
Na	3	3	3	3	3	3	3	3	3
Mg	1	1	1	20	1	3	1	1	1
Al	10	10	10	10	10	30	10	30	30
Si	10	10	10	10	20	10	10	10	10
K	30	30	30	30	30	30	30	30	30
Ca	5	10	20	30	30	10	5	5	5
Ti	10	10	300	10	10	10	10	10	10
V	50	50	50	50	50	50	50	50	50
Cr	10	10	200	10	30	30	30	10	10
Mn	3	3	3	3	10	3	3	3	3
Fe	50	300	500	1000	1000	300	300	1000	500
Co	10	10	20	10	30	100	100	10	10
Ni	10	10	50	10	30	100	30	10	10
Cu	10	10	10	10	10	10	10	10	10
Zn	300	300	300	300	300	300	300	300	300
Sr	30	30	30	30	30	30	30	30	30
Zr	300	100	100	300	300	300	300	300	300
Mo	100	100	100	300	100	100	100	100	100
Ag	1	1	1	1	1	1	1	1	1
Cd	-	3	3	-	-	-	-	-	-
Sn	50	50	50	50	50	50	50	50	50
Ba	10	10	10	10	10	10	10	10	10
Ta	500	500	500	500	500	500	500	500	500
W	1000	-	-	1000	1000	1000	1000	1000	1000
Pb	30	30	30	30	30	30	30	30	30
Bi	10	10	10	10	10	10	10	10	10

* All values ppm unless otherwise stated.

TABLE I (Cont.)

CHEMICAL ANALYSES OF COMMERCIAL CARBIDE POWDERS*

Niobium Carbide - NbC

Elements	Lot 12	Lot 13	Lot 13R**	Lot 14	Lot 15	Lot 17	Lot 17A	Lot 17AR**	Lot 18A
Nb	87.3% $\pm$ 0.2%	88.6% $\pm$ 0.2%	88.9%	87.6%	87.3%	87.1%	87.5%	88.5%	87.6%
Total C	10.97%	10.77%	10.9%	11.4%	11.32%	11.5%	11.42%	11.4%	11.45%
Free C	810 ppm	770 ppm	500 ppm	900 ppm	0.14%	0.23%	0.2%	0.2%	0.2%
O ₂	0.84%	0.26%	0.25%	700 ppm	0.12%	0.14%	0.14%	0.14%	0.13-0.14%
Li	3	3	3	3	3	3	3	3	3
Be	1	1	1	1	1	1	1	1	1
B	1	1	1	1	1	1	1	1	1
Na	10	3	3	3	3	3	3	3	3
Mg	200	1	1	1	30	1	1	1	1
Al	1000	10	10	10	10	200	200	10	10
Si	1000	10	30	20	10	1	10	30	10
K	1000	30	30	30	30	30	30	30	30
Ca	100	5	5	5	300	10	10	5	5
Ti	1000	10	10	500	10	10	10	10	10
V	50	50	50	50	50	50	50	50	50
Cr	30	10	10	30	30	10	10	10	10
Mn	3	50	3	30	3	30	30	3	30
Fe	500	3000	100	300	500	3000	3000	500	3000
Co	500	10	10	30	30	10	10	10	10
Ni	10	10	10	30	50	10	10	10	10
Cu	10	10	10	10	10	10	10	10	10
Zn	300	300	300	300	300	300	300	30	300
Sr	30	30	30	30	30	30	30	30	30
Zr	100	100	100	100	100	500	500	100	500
Mo	1000	100	100	100	100	100	100	100	100
Ag	1	1	1	1	1	1	1	1	1
Sn	50	50	50	50	50	50	50	50	50
Ba	10	10	10	10	10	10	10	10	10
Ta	500	500	500	500	500	500	500	500	500
W	1000	1000	1000	1000	1000	1000	1000	1000	1000
Pb	30	30	30	30	30	30	30	30	30
Bi	10	10	10	10	10	10	10	10	10

* All values ppm unless otherwise stated.

** Acid leached to remove iron.

TABLE I (Cont.)

CHEMICAL ANALYSES OF COMMERCIAL CARBIDE POWDERS*

Niobium Carbide - NbC

Elements	Lot 19	Lot 20	Lot 21A	Lot 21B	Lot 21C	Lot 22A**	Lot 22B**	Lot 22C**	Lot 23A**
Nb	88.6%	89.0%	88.0%	88.1%	87.4%	86.7%	86.7%	87.3%	84.1%-87.6%
Total C	11.4%	11.0%	11.9%	11.8%	11.6%	11.4%	11.4%	11.4%	11.3%
Free C	0.3%	0.1%	0.6%	0.6%	0.6%	0.2%	0.2%	0.2%	0.2%
O ₂	0.15%	0.15%	0.19%	0.27%	0.98%	0.31%	0.30%	0.28%	0.23%
Li	3	3	3	3	3	3	3	3	3
Be	1	1	1	1	1	1	1	1	1
B	1	1	1	1	1	1	1	1	1
Na	3	3	3	3	3	3	3	3	3
Mg	1	1	1	1	1	1	1	1	1
Al	10	100	10	10	10	10	10	10	10
Si	10	10	10	10	10	10	10	10	10
K	30	30	30	30	30	30	30	30	30
Ca	5	5	5	5	5	5	5	5	5
Ti	10	10	10	10	10	10	10	10	10
V	50	50	50	50	50	50	50	50	50
Cr	10	100	10	10	10	10	10	10	10
Mn	10	3	3	3	3	3	3	3	3
Fe	3000	300	200	30	30	500	500	500	1000
Co	10	30	10	10	10	10	10	10	10
Ni	10	30	10	10	10	10	10	10	10
Cu	10	10	10	10	10	10	10	10	10
Zn	300	300	300	300	300	300	300	300	300
Sr	30	30	30	30	30	30	30	30	30
Zr	100	100	100	100	100	100	100	100	100
Mo	100	100	100	100	100	100	100	100	100
Ag	1	1	1	1	1	1	1	1	1
Sn	50	50	50	50	-	50	50	50	50
Ba	10	10	10	10	10	10	10	10	10
Ta	500	500	500	500	500	500	500	500	500
W	1000	1000	1000	1000	1000	1000	1000	1000	1000
Pb	30	30	30	30	30	30	30	30	30
Bi	10	10	10	10	10	10	10	10	10

* All values ppm unless otherwise stated.

** Acid leached to remove iron.

TABLE I (Cont.)

CHEMICAL ANALYSES OF COMMERCIAL CARBIDE POWDERS*

Niobium Carbide - NbC

Elements	Lot 23B**	Lot 23C**	Lot 23AA**	Lot 23BB**	Lot 23CC-1**	Lot 23CC-2**	Lot 23CC-3**	Lot 24	Lot 25
Nb	86.5%-88.1%	86.8%	87.6%	88.1%	88.6% $\pm$ 0.2%	88.4% $\pm$ 0.2%	88.3% $\pm$ 0.2%	86.7%	88.4%
Total C	11.4%	11.4%	11.3%	11.24%	11.3%	11.5%	11.5%	12.0%	11.2%
Free C	0.3%	0.26%	-	0.19%	0.16%	0.27%	0.21%	0.64%	0.05%
O ₂	0.21%	0.23%	-	0.23%	0.20%	0.28%	0.24%	0.22%	0.11%
Li	3	3	3	3	3	3	3	3	3
Be	1	1	1	1	1	1	1	1	1
B	1	1	1	1	1	1	1	1	1
Na	3	3	3	3	3	3	3	3	3
Mg	1	1	1	1	1	1	1	1	1
Al	10	10	10	10	10	10	10	10	10
Si	10	10	10	10	10	10	10	10	10
K	30	30	30	30	30	30	30	30	30
Ca	5	5	5	5	5	5	5	10	5
Ti	10	10	10	10	10	10	10	10	10
V	50	50	50	50	50	50	50	50	50
Cr	10	10	10	10	10	10	30	10	50
Mn	3	10	5	5	3	10	3	10	3
Fe	500	1000	200	300	200	300	400	250	500
Cc	10	10	10	10	30	10	10	10	100
Ni	10	10	10	10	10	10	10	100	100
Cu	10	10	10	10	10	10	10	10	10
Zn	300	300	300	300	300	300	300	300	300
Sr	30	30	30	30	30	30	30	30	30
Zr	100	100	300	100	300	300	300	1000	300
Mo	100	100	100	100	100	100	100	100	100
Ag	1	1	1	1	1	1	1	1	1
Sn	50	50	50	50	50	50	50	50	50
Ba	10	10	10	10	10	10	10	10	10
Ta	500	500	500	500	500	500	500	500	500
W	1000	1000	1000	1000	1000	1000	1000	1000	2000
Pb	30	30	30	30	30	30	30	30	30
Bi	10	10	10	10	10	10	10	10	10

* All values ppm unless otherwise stated.

** Acid leached to remove iron.

TABLE I (Cont.)

CHEMICAL ANALYSES OF COMMERCIAL CARBIDE POWDERS*

Niobium Carbide - NbC

<u>Elements</u>	<u>Lot 26</u>	<u>Lot 27</u>	<u>Lot 28A</u>	<u>Lot 28B</u>	<u>Lot 28C</u>	<u>Lot 29</u>
Nb	88.2% $\pm$ 0.2%	88.7% $\pm$ 0.2%	88.1% $\pm$ 0.2%	88.1% $\pm$ 0.2%	87.7% $\pm$ 0.2%	87.8%
Total C	11.8%	11.4%	11.9%	11.9%	11.7%	11.9%
Free C	0.56%	0.08%	0.54%	0.56%	0.5%	0.7%
O ₂	0.17-0.21%	0.12%	0.14%	0.14%	0.15%	0.19%
Li	3	3	3	3	3	3
Be	1	1	1	1	1	1
B	1	1	1	1	1	1
Na	3	3	3	3	3	3
Mg	1	1	1	1	1	10
Al	30	10	10	10	10	10
Si	10	10	10	10	10	10
K	30	30	30	30	30	30
Ca	5	5	5	5	5	5
Ti	10	10	10	10	10	10
V	50	50	50	50	50	50
Cr	10	30	10	10	30	10
Mn	10	10	10	10	30	3
Fe	200	200	200	200	300	300
Co	10	30	10	10	10	10
Ni	100	50	100	100	30	30
Cu	10	10	10	10	10	10
Zn	300	300	300	300	300	300
Sr	30	30	30	30	30	30
Zr	300	300	300	300	300	300
Mo	100	100	100	100	100	100
Ag	1	1	1	1	1	1
Sn	50	50	50	50	50	50
Ba	10	10	10	10	10	10
Ta	500	500	500	500	500	500
W	1000	1000	1000	1000	1000	1000
Pb	30	30	30	30	30	30
Bi	10	10	10	10	10	10

* All values ppm unless otherwise stated.

TABLE I (Cont.)

CHEMICAL ANALYSES OF COMMERCIAL CARBIDE POWDERS*

Tantalum Carbide - TaC

<u>Elements</u>	<u>TaC-6</u>	<u>TaC-7</u>	<u>TaC-8</u>	<u>TaC-9</u>	<u>TaC-10</u>	<u>TaC-11</u>	<u>TaC-12</u>	<u>TaC-13A</u>	<u>TaC-13B</u>	<u>TaC-13C</u>
Ta	93.6%	93.6%	93.4%	93.5%	93.6%	93.3%	93.8 ± 0.3%	92.7 ± 0.3%	93.6 ± 0.3%	93.7 ± 0.3%
Total C	6.19%	6.27%	6.17%	6.26%	6.25%	6.25%	6.14%	6.15%	6.19%	6.29%
Free C	0.06%	0.03%	0.07%	0.10%	0.07%	0.08%	0.08%	0.05%	0.09%	0.07%
O ₂	0.14%	0.96%	0.22%	0.09-.20%	0.08-.21%	0.21%	0.29%	0.25%	0.37%	0.27%
Li	1	1	1	1	1	1	1	1	1	1
Be	1	1	1	1	1	1	1	1	1	1
B	3	3	3	3	3	3	3	3	3	3
Na	3	3	3	3	3	3	3	3	3	3
Mg	1	1	500	20	20	3	200	200	30	30
Al	3	3	3	3	3	3	3	3	3	3
Si	3	3	3	3	3	100	30	10	3	3
K	10	10	10	10	10	10	10	10	30	10
Ca	1	1	30	10	10	3	30	50	20	20
Ti	5	5	15	15	15	5	5	5	5	5
V	10	10	10	10	10	10	10	10	10	10
Cr	10	10	30	30	30	5	3	3	30	3
Mn	1	1	10	10	10	1	1	3	30	30
Fe	30	30	500	500	500	200	300	1000	500	500
Co	5	5	5	5	5	5	5	5	5	5
Ni	1	1	1	20	20	1	20	10	50	3
Cu	1	1	1	1	10	30	30	3	3	3
Zn	100	100	100	100	100	100	100	100	100	100
Sr	30	30	30	30	30	30	30	30	30	30
Y	30	30	30	30	30	30	30	30	30	30
Zr	30	30	30	30	30	30	30	30	30	30
Nb	100	100	2000	2000	2000	1000	100	200	100	100
Mo	10	10	10	10	10	10	10	10	30	30
Ag	1	1	1	1	1	1	1	1	1	1
Sn	3	3	3	3	3	3	3	3	3	3
Ba	10	10	10	10	10	10	10	10	10	10
W	30	30	30	30	30	30	30	30	30	30
Pb	3	3	3	3	3	3	3	3	3	3
Bi	5	5	5	5	5	5	5	5	5	5

* All values ppm unless otherwise indicated.

TABLE I (Cont.)

CHEMICAL ANALYSES OF COMMERCIAL CARBIDE POWDERS*

Elements	Tantalum Carbide - TaC				Zirconium Carbide - ZrC					
	TaC-16	TaC-17	TaC-18	TaC-19	ZrC-4	ZrC-5	ZrC-6	ZrC-7	ZrC-8	ZrC-9
TaC or ZrC	93.9% ± 0.3%	53.2 ± 0.5%	93.3%	92.8%	88.4%	88.1%	88.5%	86.5%	88.1%	87.4%
Total C	6.25%	6.52%	6.27%	6.35%	11.44%	11.83%	10.8%	10.7%	11.2%	11.7%
Free C	0.05%	0.05%	0.05%	0.05%	0.12%	0.46%	0.13%	0.05%	1.0%	0.9%
O ₂	0.036%	0.03-0.04%	0.16-0.22%	0.25%	0.15%	0.06-.2%	0.19%	0.18%	0.34%	0.13%
Li	1	1	1	1		10	10	10	10	10
Be	1	1	1	1		3	3	3	3	3
B	3	3	3	3		3	300	300	30	3000
Na	3	30	3	3		30	30	30	30	30
Mg	3	1	1	1		3	3	3	3	3
Al	30	3000	3	-		30	30	30	30	30
Si	20	10	30	20		10	200	10	100	30
K	10	10	10	10		30	30	30	30	30
Ca	3	10	1	10		30	30	30	30	100
Ti	500	3000	100	50		100	300	300	300	500
V	100	30	10	-		100	30	30	30	100
Cr	3	3	3	100		10	100	100	300	300
Mn	10	10	1	-		10	10	10	10	20
Fe	1000	30	300	100	200	30	300	300	100	1000
Co	5	5	2000	300		30	30	30	30	30
Ni	1	5	300	100		30	10	10	10	100
Cu	100	3	3	3		10	10	10	10	10
Zn	100	100	100	-		100	100	100	100	100
Sr	30	30	30	30		30	30	30	30	30
Y	30	30	30	30		-	-	-	-	-
Zr	30	30	30	-		-	-	-	-	-
Nb	2000	3000	2000	2000		1000	1000	3000	1000	1000
Mo	20	10	10	-		100	300	300	300	300
Ag	1	1	1	-		3	3	3	3	3
Sn	3	3	3	-		30	30	30	30	3
Ba	10	10	10	10		10	10	10	10	30
Ca	-	-	-	-		3	3	3	3	3
Hf	-	-	-	-	100	1000	-	200	200	1000
W	1000	30	300	-		-	-	-	-	-
Pb	3	3	3	-		100	100	100	100	100
Bi	5	5	5	5		30	30	30	30	30

* All values ppm unless otherwise indicated.

TABLE II
POWDER CHARACTERIZATION DATA

Powder Lot No.	Vendor	Density g/cc		Average Particle Size, μ	Specific Surface		Morphological Type
		Bulk	Tap		BET	Fisher	
ZrC-4	D	2.33	3.17	2.97	1.06	0.31	B
ZrC-5	E	1.98	2.78	5.4	0.84	0.17	C
ZrC-6	D	2.00	2.84	2.70	1.08	0.34	B
ZrC-7	D	2.05	3.13	2.75	1.19	0.33	C
ZrC-8	D	2.26	3.47	3.00	1.30	0.30	B
ZrC-9	D	2.41	3.70	5.27	0.734	0.17	B
TaC-6	E	4.83	6.61	5.40	0.29	0.77	B
TaC-7	E	5.51	7.32	10.20	0.60	0.04	B
TaC-8	A	4.69	6.70	2.18	0.75	0.19	B
TaC-9	A	5.13	7.44	2.10	1.01	0.20	B
TaC-10	A	5.26	7.47	2.10	0.89	0.20	B
TaC-11	F	5.19	7.33	2.25	0.89	0.18	B
TaC-12	A	2.88	3.36	7.00	0.825	0.06	C
TaC-13A	A	4.70	6.91	2.00	0.82	0.38	B
TaC-13B	A	4.98	6.21	1.35	1.26	0.31	B
TaC-13C	A	4.88	6.09	1.70	0.997	0.24	B
TaC-16	G	8.73	9.48	50.0	-	0.008	B
TaC-17	H	7.50	8.34	38.4	0.137	0.11	B
TaC-18	A	4.97	7.69	2.55	0.637	0.162	-
TaC-19	A	5.05	8.42	2.45	0.772	0.167	B
NbC-6	E	2.09	3.27	1.30	2.58	0.59	B
NbC-7	E	1.48	3.98	1.94	1.24	0.33	D
NbC-9	D	2.21	3.98	3.90	1.28	0.20	B
NbC-10A	A	2.72	3.77	3.60	1.12	0.21	B
NbC-10B	B	2.51	3.62	3.65	0.81	0.21	B
NbC-10C	C	2.53	3.77	3.87	0.84	0.20	B
NbC-10D	D	2.36	3.72	2.00	1.78	0.38	B & C
NbC-10E	E	2.36	3.72	3.48	1.43	0.22	B
NbC-11	E	2.38	3.38	3.00	0.88	0.26	B & D
NbC-12	E	2.62	3.90	2.80	1.22	0.27	B
NbC-13	E	2.90	4.24	3.60	0.92	0.21	B
NbC-13R	E	3.02	4.25	4.02	0.885	0.191	B
NbC-14	B	3.22	4.14	12.7	0.28	0.06	A
NbC-15	C	2.57	3.93	3.5	0.97	0.22	B
NbC-17	E	2.89	4.18	3.5	1.03	0.38	B
NbC-17A	E	2.65	4.14	3.65	0.87	0.21	B
NbC-17AR	E	3.13	4.26	4.11	0.788	0.187	B
NbC-18A	E	2.73	4.46	3.8	0.94	0.202	-
NbC-19	A	5.05	8.42	2.45	0.854	0.210	B
NbC-20	A	3.13	4.65	4.79	0.587	0.161	B
NbC-21A	E	1.56	2.44	1.30	2.160	0.590	B
NbC-21B	E	1.74	2.56	1.29	2.169	0.595	B
NbC-21C	E	1.60	2.35	1.29	1.224	0.595	B
NbC-22A	E	2.80	4.23	3.55	0.714	0.219	B
NbC-22B	E	2.69	4.31	3.90	0.813	0.197	B
NbC-22C	E	2.67	4.40	3.85	0.743	0.199	B
NbC-23A	E	3.00	4.46	3.91	0.714	0.196	B
NbC-23B	E	2.96	4.31	4.05	0.824	0.189	B
NbC-23C	E	2.98	4.35	4.05	0.843	0.189	B
NbC-23AA	E	2.88	4.38	3.55	-	0.216	B
NbC-23EB	E	3.11	4.28	4.0	0.864	0.192	B
NbC-23CC-1	E	3.20	4.56	4.25	-	0.181	B
NbC-23CC-2	E	2.71	4.20	3.62	-	0.209	B
NbC-23CC-3	E	2.89	4.30	3.60	-	-	-
NbC-24	E	2.82	4.32	3.01	1.072	0.255	-
NbC-25	A	2.97	4.74	4.50	0.622	0.171	-
NbC-26	E	2.71	4.27	3.35	-	-	B
NbC-27	A	2.87	4.49	4.20	-	0.183	B
NbC-28A	E	2.84	4.21	3.47	-	0.221	B
NbC-28B	E	2.61	3.99	3.42	-	0.224	B
NbC-28C	E	2.59	4.11	3.38	-	0.227	B
NbC-29	E	2.59	4.08	3.11	-	-	B