AGENCY FOR INTERNATIONAL DEVELOPMENT
WASHINGTON, D. C. 20523

BIBLIOGRAPHIC INPUT SHEET

FOR AID USE ONLY
Batch 89

1. SUBJECT CLASSIFICATION

A. PRIMARY: Development and economics — DM00-0000-0000

B. SECONDARY: Industries and industrialization

2. TITLE AND SUBTITLE

Plant requirements for manufacture of tungsten carbides

3. AUTHOR(S)

(101) U.S. Dept.of Commerce. Office of Technical Services

4. DOCUMENT DATE: 1955

5. NUMBER OF PAGES: ~~15p.~~ / 6p.

6. ARC NUMBER: ARC 673.734.C734

7. REFERENCE ORGANIZATION NAME AND ADDRESS

AID/TA/OST

8. SUPPLEMENTARY NOTES (Sponsoring Organization, Publishers, Availability)

(Plant requirement no.62)

9. ABSTRACT

10. CONTROL NUMBER

PN-AAF-824

11. PRICE OF DOCUMENT

12. DESCRIPTORS

Industrial plants
Manufacturing
Requirements
Small scale industries
Tungsten carbides

13. PROJECT NUMBER

14. CONTRACT NUMBER

AID/TA/OST

15. TYPE OF DOCUMENT

AID 590-1 (4-74)

[illegible] OF [illegible] GUIDES

TECHNICAL AIDS BRANCH

INTERNATIONAL COOPERATION ADMINISTRATION

Washington, D. C.

Tungsten Carbides.

INQUIRY: Information is requested on the process used in the manufacture of tungsten carbides, including plant layout, personnel and capital required.

INFORMATION SUBMITTED:

In its broad outline, the process on making tungsten carbide cutting tools, wire drawing dies, header dies, gages and other industrial products consists of preparing the powder carbide of tungsten, mixing it with a binder (such as cobalt) into compacts of desired forms, and sintering these to get the desired shapes, hardness, strength and other attributes.

Though this pattern appears simple, it soon becomes evident from a detailed study that the techniques involved are different from those generally used in powder metallurgy.

In a survey made among the manufacturers of the necessary equipment, and technicians experienced in carbide making, the following steps have been established as the pattern peculiar to this industry.

I. Making the Carbide Powder of Tungsten: The powders of commercial tungsten (w) and lampblack are weighed and mixed in a ball mill. (Indicated by Item I. on the plant layout). This mixture is carburized in a carbon resistor furnace or in a graphite crucible of high frequency. (Item 2 on the plant layout). The charge is gradually heated in the furnace until the flames die out, then hydrogen is introduced.

Heating continues until the temperature is gradually raised to 2500°F. and held there for about two hours. After this the furnace is allowed to cool slowly under the protection of hydrogen.

The mixture is then pulverized in a crusher (Item 3), then milled to finer powder (Item 4) and screened. (Item 5).

(For additional details see ADDENDUM A)

II. Mixing this carbide powder with a binder: In most cases the binder metal is cobalt, which is 99% pure, and should pass through a screen of 200 mesh.

Mixing is done by milling them together wet, to the desired pulverization in a ball mill. (Item 6). In this process each carbide molecule becomes coated with a continuous layer of the binder metal; a coating which aides in the necessary adhesion of the particles.

Each batch so mixed must be carefully checked with specially-built particle measuring instruments, and great care is needed in the mixing process to get a batch adequately coated. (Actual cementation takes place later in the final sintering process).

(For additional details see ADDENDUM (B) AND (C)

The compact so mixed is now passed through a dryer, to get rid of the fluid. (Item 7).

This is followed by fine screening, (Item 8) after which the powder is mixed with fine paraffin to facilitate handling and pressing. (Item 9).

III. Compacting the Mixture into Various Shapes: The mixture can now be formed into any variety of the shapes needed in industry. This is done in either of two ways: (a) By cold pressing the compacts into shapes in dies, then heating them in batches, and finally fusing them through a sintering process; or, (b) by a hot-pressing and sintering process cone simultaneously.

(a) In the cold-pressing process the powders are shaped in special dies, into rods, cylinders, flats, and hundreds of other forms. Or the mixture may be poured into crucibles and tamped into them loosely, avoiding carefully the creation of air pockets.

Pressure is applied either in a mechanical press, which may be cam-type, crank-type, a tuggle or a knuckle-joint type. (Item 10); and/or in a tableting machine for tool-tip shapes and similar articles. (Item 11).

As production increases with time, a hydraulic press may be acquired for larger pieces and heavier pressures.

The dies, molds and punches must be tailor-made to insure uniform density distribution in the compact, and they must be designed large enough to contain more powder than the ultimate article, (after final sintering); i.e. they must be from 15% to 18% oversize due to considerable shrinkage of the mixture during the pre-sintering operation which follows compacting. (The dies are indicated on the plant layout sheet as Item 12).

When the tableting machines are used, the compression-cycle for the "pills" is rather slow. This is found, however, to be the most effective method in carbide making, provided there is sufficient demand for each shape and size of the tablets.

(For additional information see (ADDENDUM D)

IV. Pre-Sintering: After the press had duly compacted the mixture in the dies to rough sizes and contours, the resultant forms are removed and pre-sintered in a furnace to get a cohesive structure similar to that of chalk, permitting easy shaping.

Pre-sintering is done in a box-type furnace, with the compacts surrounded by a protective atmosphere, such as hydrogen or dissociated amonia, at a temperature of 1000-1600°F.

The usual method is to place a batch of the compacted forms freely on a tray, pour inert, insulating powder such as alumina around them, put the trays in a container, and push one batch after another through the furnace. (Item 13).

After careful cooling, these rough, inaccurate pre-sintered forms of chalk-like consistency are removed from the furnace to the in-process storage, (Item 14) or are subjected to the final shaping operations. (Item 15). This may be done by facing, drilling, shaping, turning, grinding and other forming operations before the final sintering process.

(For additional information see ADDENDUM E)

V. Final Sintering: Starting operations on a small scale, for experimental purposes, the batch-type furnace used for pre-sintering (shown under Item 13) can be used. Later a continuous-feed arrangement and tube furnace may be added.

In this operation the parts are deposited in carbon boats, packed in granulated carbon of aluminum oxide, and pushed through the furnace, or the compacts may be placed on graphite discs while sintering is being done. The charges must be protected against decarburization and exidation by surrounding them with a neutral atmosphere, and heated at a temperature that varies between 2150° and 2550° F., and then moved to the cooling chamber.

(For further information see ADDENDUM F)

VI. Hot-Sintering: While the above described technique is regarded as the most efficient method of production, the hot-sintering method will produce somewhat harder and denser carbides with no loss to the ultimate strength of the product. Carbides produced by the first method have a Rockwell A hardness of 92, whereas the hot-pressed carbides have a Rockwell A hardness of 95.6, with a denser microstructure which gives the product superior strength, toughness and higher cutting properties.

The equipment used for hot-sintering is quite complicated in both design and construction. The powdered mixture coming from the ball mill is placed in a graphite mold, heated to sintering temperature for about fifteen minutes, while at the same time pressure is applied to the compact. Heating of the mixed powder and die may each be done separately, or the die alone may be heated and the powder get its heat by convection, or both may be heated simultaneously in an electric resistance furnace that surrounds the die, and placed inside the working space of the press. In all cases the process is enveloped in a non-oxidizing, protective atmosphere.

(For additional information see ADDENDUM G)

VII. Testing: Routine controls must be applied at each stage of the entire carbide making process. The physical testing of these hard metals is rather difficult and so far no standards have been accepted for certain phases of it.

Hardness is measured on a Rockwell A scale, equipped with a diamond indenter. (Item 17).

Variations in the combination of the ingredient powders, are determined by: (a) X-ray diffraction methods (Item 17) and/or by (b) measuring the electrical resistance of the carbide pieces. (Item 18).

Porosity is revealed by microscopic studies of the polished surfaces of hard metallographic specimens after the surfaces have been prepared with diamond polishing powder. (Item 19).

Density determinations are used to check the completeness of the sintering operation or to note possible changes in the composition and detrees of shrinkage. (Item 20).

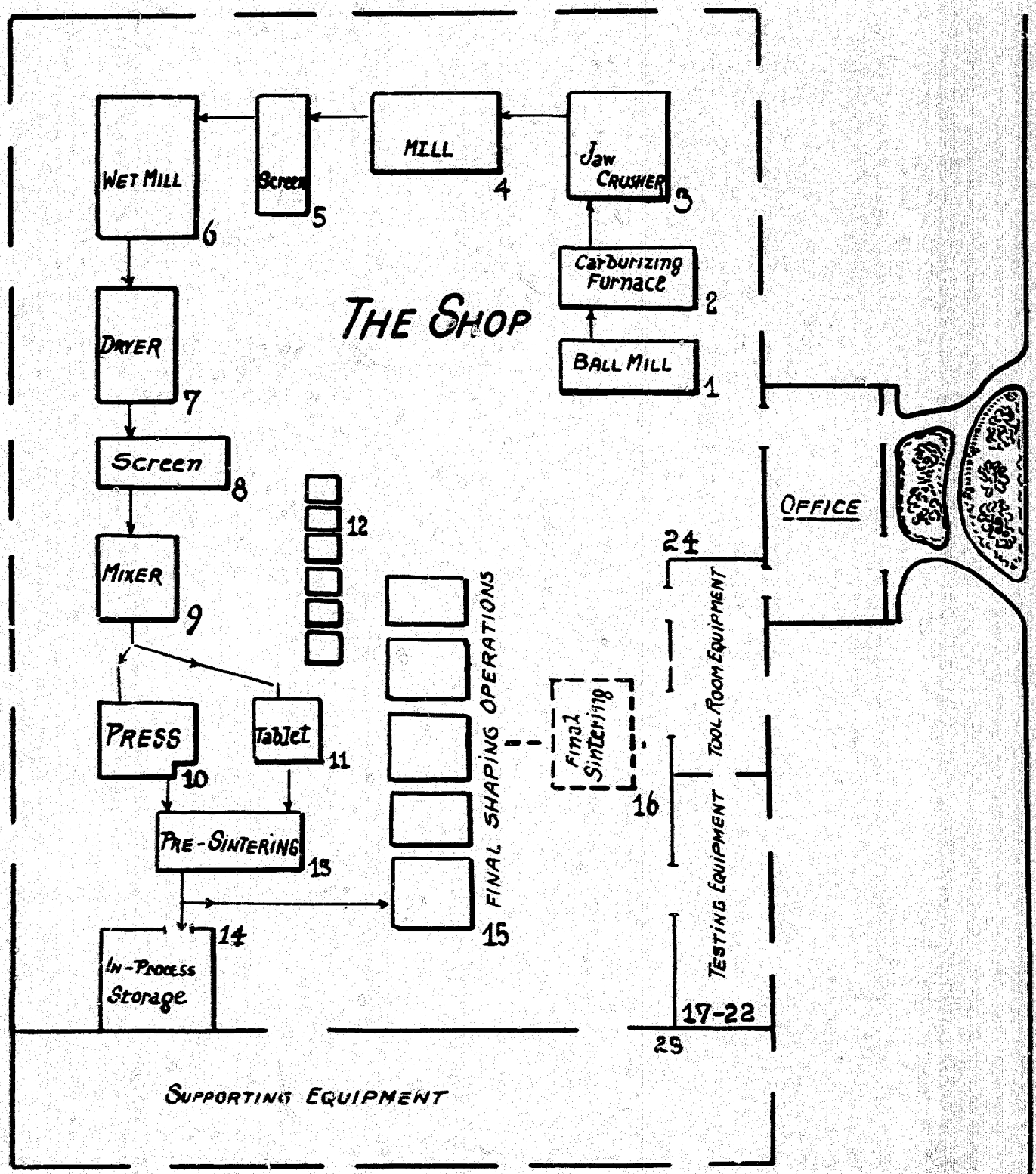
Chemical analyses are used to check the extent of the combined and free carbon in the carbide powder. (Item 22).

Cutting property tests of the tools are actually made on a lathe, by cutting cast iron and steels of known chip producing properties.

VIII. Supporting equipment: Hydrogen and ammonia gas dryer, ammonia dissociator, water cooler, current limiting device to protect molybdenum heating elements, liquid ammonia cylinders, control panels, automatic temperature regulators, optical pyrometer. (listed under Item 23).

IX. Tool Room equipment. (Item 24).

Item		Dollars
1	Ball mill	1500.00
2	Carburizing furnace	5000.00
3	Jaw crusher	2500.00
4	Pulverizing mill	2000.00
5	Screen	900.00
6	Wet ball milling equipment	2250.00
7	Steam dryer	1000.00
8	Fine screener	900.00
9	Paraffin mixing equipment	600.00
10	Compacting Press	8500.00
11	Tabelting machine	7500.00
12	Dies, 20 pieces	2000.00
13	Pre-sintering furnace	6000.00
14	In-process storage equipment	2000.00
15	Various machine tools for shaping operations	15000.00
16	Sintering machine (done on item 13)	00.00
17	to item 22 inclusive, for testing equipment	8000.00
23	Supporting equipment, including foundations, electrical installations, motors, generators, air conditioning, lighting, etc.	40000.00
24	Tool room equipment, gages, care and maintenance tools	25000.00
		130650.00



The above layout provides for the initial, experimental laboratory to manufacture TUNGSTEN CARBIDES

For details see next page

The building: There are approximately 3500 square feet of useable floor space. Naturally, there are many types of buildings that could be used in this instance.

X. Manpower requirements. In the few shops where carbides are made, there have been marked improvements in the process, due entirely to the fact that the technicians have studied carefully, on the spot, the many variables which determine the high quality required. And while there is no existing paper which covers in detail every phase of carbide manufacture, or all the improvements introduced during the recent past, there are available numerous instructive and illustrated details in the technical press, in symposiums on powder metallurgy, and in special reports on the results of investigations and tests made by experienced metallurgical scientists and carbide shop technicians. However, none of these empirical or technical data can take the place of the experienced and skill gained by actual practice.

Also, it should be remembered, that carbide technicians disagree in many respects on the advantages or disadvantages of certain processes in the making of carbides, and also, that in many respects the production of these hard metals is still a trade secret. There is, however, unanimous agreement on the single point, that successful manufacturing depends chiefly on the know-how acquired by the technicians charged with the management of the shop, and with the operation of each individual piece of equipment.

MANPOWER Requirements (for 50 lbs. per day):

General superintendent, per year	12000.00
Controller	8500.00
2 application technicians	16000.00
2 maintenance men	12000.00
8 machine operators in the shop	40000.00
5 mechanics in tool room	37500.00
2 instrument specialists	16000.00
4 helpers	12000.00
First year management and labor.....	154000.00

ADDENDUM A.

Pure Tungsten Powder: This can now be bought ready for use. It is made by reducing tungstic acid with sugar carbon. The metal so obtained is very pure and adequate for carbide making as it cements readily.

Carbon: The carbon powder (lampblack) analysis should prove to be 99.20% pure carbon, the rest being impurities of ash and moisture, with density 2. If same is produced by the decomposition of petroleum by heat, the maker should guarantee that it has been purified by benzine, alcohol or ether. If the incomplete combustion of a hydrocarbon-like acetylene was used, this may be accepted as a carbon ingredient, since such a mixture is even purer than lampblack. The usual porportion is 6.7 kg of lampblack to 100 kg of W. powder.

ADDENDUM B.

Binder Metal in the Mixture: The physical properties of the tungsten carbide-cobalt base mixture are greatly influenced by the quantity of the binder metal. Hardness decreases steadily in the finished product, with compositions up to 6% cobalt to lower values in metals with 20% cobalt. However, it should be noted, that the transverse rupture strength increases at the same time steadily with the rising cobalt content, until nearly 400,000 p.s.i. can be assured for compositions containing as much as 20% cobalt. For these extreme variations in the characteristics charts are now available so that carbide makers can furnish the proper carbides either with increased ductility and resistance to distortion, or with higher fatigue and impact strength. (See list of carbide manufacturers given)

When, for instance a special tool is required for machining tough, stringy steel materials, a mixture of titanium carbide (instead of tungsten carbide) powder with a cobalt binder is used. Or, where increasingly difficult demands are placed on certain cutting tools, titanium and/or tantalum carbides are sintered with iron-base alloys to lend them unusually high temperature - resistant properties.

ADDENDUM C.

Mixing by milling; The carbides of tungsten thus made, are now ready to be mixed and pulverized with powdered cobalt in a ball mill. The drum is lined with tungsten, Stellite or stainless steel, and the balls are usually made of cobalt, same as the binder metal. As a protective atmosphere either hydrogen or cracked propane is used, or the powders may be milled in the presence of water, or some other organic solvent to prevent oxidation of the powder.

Duration of milling: This may last from several hours to several days, until tests prove that the cobalt binder cannot be separated magnetically from the carbide particles. Powder so produced is now ready for the next processing, that of compacting into various shapes. Extreme care must be exercised during mixing, or local concentrations will show up in the finished carbides when checked on the photomicroscopic specimens.

ADDENDUM D

Compacting the mixture into various shapes: To insure maximum cohesive strength of the compacting atoms, as the pressure is applied on the dies, the operator is obliged to follow certain rules he himself must establish after repeated experiments. This is because the compacting technique is controlled by such variables as; (a) Size of the particles in the mixture, (b) Pressure applied in the dies, (c) Time required for the flow of the mixture in the dies, (d) Ratio between length and width of the dies, (e) Abrupt changes or projections in the dies, (f) Tolerances required.

There are presses which apply pressure in one direction or in two. In the latter case multiple punches are used with individual movements, velocities and pressures.

ADDENDUM E.

Pre-sintering: The heating of the compacts is done at a low-enough temperature, so that the binder metal-cobalt can produce the required pre-sintering effect. The temperature is gradualy raised to 1850°F to evaporate the paraffin lubricant used in the dies, without causing other changes in the compacts. To maintain the correct temperatures, retain the molds for the required time in the oven, to have a dry and pure reducing atmosphere is of utmost importance.

Very often the prepared compacts have been shaped into block-like forms, out of which a number of pieces can be readily cut. For this, instead of contoured wheels, diamond-tipped tools are used to overcome the destructive effects of abrasion. When the parts so shaped are sent through the final sintering operation described later, what little final shaping is required is done with diamond or boron carbides.

For large-scale operations continuous, mechanical transports operated hydraulically, push the trays through the tube furnace.

ADDENDUM F.

Sintering: Extreme care must be exercised in temperature control and timing. Making carbides for machining cast iron and non-ferrous metals the sintering temperature is somewhat lower than when making carbides for machining though steels. The temperature is controlled with a radiation pyrometer.

Also, it should be remembered that the strength of the finished carbide does not depend so much on the cementing action of the binder material (cobalt) as on the formation of a strong carbide skeleton during the final sintering. During this process the carbide particles are drawn together by surface tension forces. The extent to which ideal bonding takes place depends on particle size, their distribution in the grid and whatever may interfere with this action such as impurities, inclusions or oxide films that may be present.

The formation of the various patterns is very evident when the microstructures of different carbides are examined. For instance, the crystals of a tungsten carbide-cobalt mixture will show the tungsten particles with sharp edges as they are imbeded in the cobalt matrix, whereas the mixed crystals of Ti. C. & W.C. are nodular and darker in color while those of the Ta. C. have rounded corners.

Also, it should be noted, that in some shops the furnace with a molydbenum resistor element as well as the induction-type is used, because experience in the past indicate:

A furnace with a molydbenum resistor element, as used in our case, is a more efficient method, even though it is not considered by some carbids technicians as flexible as the industion-type. On the other hand, the latter had the advantage that the same power source can be used for all high-temperature operations.

When a box-type furnace is used for both pre-sintering and final sintering operations as in our plan, it is recommended by well-known authorities that a disasociated ammonia protective gas be used. This allows the high temperatures required for sintering, while at the same time a low-dew-point can be maintained.

ADDENDUM G.

This is done in a heated die, the powder being pressed into the die while the temperature is raised to 2500°F., and kept for several minutes. This method is expensive because the graphic dies are destroyed after each pressing.

The dies are usually heated in a carbon resistance furnace with the current passing directly through the dies, or through the punches. The die assembly may also be heated in a high-frequency induction-type furnace.

In another modification of the resistance heating process a current of high amperage is passed directly through the mixed powder while the necessary pressure is being applied.

Selection of one or the other type depends entirely on the thorough understanding of the relationship between pressure, heat and time.

In all cases protective atmosphere is essential.

ADDENDUM H.

The effectiveness of carbide manufacturing equipment is in direct ratio to the skill and experience of your shop technicians.

And while there is now available plenty of basic technical information on the subject, much of the equipment still has to be made on the spot to fit particular jobs.

BIBLIOGRAPHY.

Iron Age. Aug. 13/53. Article deals with some new improvements in the processing of carbides with titanium additives, their compositions and advantages of high temperature resistance.

Iron Age. Feb. 5/53. The importance of careful powder forming is emphasized, and the techniques of shaping, after pre-sintering are described.

Production Engineering. April 1953. Recent advances are shown, as described also in Stahl Und Eisen, Oct. 11/51., including the basic technology of carbide making, carburizing, applications of protective atmosphere, proper milling requirements and the advantages of hot-pressing.

Metallurgia. April 1953. The properties of tungsten carbide, di-tungsten carbide and titanium and tantalum carbides are enumerated. We are shown how tungsten carbide forms the basis of most carbides, how powders are mixed and the melting points of cementing (bending) are given. The weaknesses of iron and nickel bonding materials, as well as the main factors of bonding, shaping and final bonding are explained.

Metals Handbook. American Society for Metals. A brief review of the manufacture and the chief characteristics, bend tests, furnace equipment, protective atmosphere and use, care and maintenance of carbide tools.

Iron and Steel: July '53 (British) Brief review of the layout of a British manufacturing tungsten carbide tips and tools.

NObs 55219 - Nobs 5521, Nobs 31493, Noas 51005c. Research reports for the Navy Department, of tests on non-strategic metals as binders, of electrical resistance sintering, density and hardness measures, use of ball mills and machining tests.

Recommended Practices B.276, 52T, 1952 Book of ASTM Standards. Part 2, pg. 952.

Tungsten-Carbon-Cobalt System. Transactions AIME, Vol. 194 pg. 1045.

Treatise on Powder Metallurgy. By Claus G. Goetzel. PhD. Interscience Publ. Inc. 215-4th Ave., New York 3, N.Y. 3 volumes.

Catalogs which list the principal properties, characteristics and fields of application of all commonly available standard and specific grades, and how to select the proper grades:

Carboloy Dept. of General Electric Co. Detroit 32 Mich.

Carmet Sintering Carbides, Allegheny Ludlum Steel Co., 1500 Jarvis St., Ferndale, Detroit 20, Mich.

Kennametals Corp. Latrobe, Penna.

Vascoloy Ramet Corp., 210 E. 34th St., New York 16, N.Y.

Firth Sterling Inc., Pittsburgh, Pa.

Sintercast Corp., 136 Woodworth St., Yonkers, N.Y.

Patents. There is a most impressive list of hundreds of patents available on the carbide making process. Many of these are controversial but worth studying. It should be remembered, that in many respects carbide making is still a trade secret, since successful manufacturing depends chiefly on the know-how of the technicians managing the shop and the men operating each individual equipment.

This report was prepared by
Joseph de Feher
Arlington 1, Va.

11-55

Tungsten Carbide

INQUIRY: Information is requested on techniques and equipment for sintering and shaping tungsten carbide cutting tools and on tool designs.

INFORMATION SUBMITTED:

The tungsten carbide manufacturing plant of today is considerably different from the plant of a few years back. The present superior carbide plants, equipment and techniques permit the carbide to be produced to consistently high standards. The processes used today by the relatively small number of American manufacturers of carbides are basically similar. The first steps in making cemented tungsten carbides are the reduction of cobalt oxide to metallic cobalt and the carburization of tungsten. To reduce the cobalt oxide, the material is placed in "boats" of a special heat and corrosion resistant alloy which move through a continuous-type furnace. Here, high-purity hydrogen at elevated temperatures acts as the reducing agent.

In the carburization process a mixture of metallic tungsten powder and amorphous carbon combined by heating them together to the temperature of molten steel. Advanced methods of performing the carburizing operation depend on the use of a special electronic induction furnace. The intimately mixed tungsten and carbon powders are charged into a graphite crucible, and the temperature of the crucible and its contents is raised through a closely-controlled cycle.

The next major processing step is the mixing of tungsten carbide and cobalt in the proper proportions for the grade desired. The cobalt is a cementing agent and enables the formation of the carbide-to-carbide bond. Ball milling of the Co-TiC mixture follows. The mills are filled with naphtha after charging to exlude air and avoid oxidation. Water is also commonly used for this purpose. A final screening operation ensures the complete removal of large particles from the powdered mixture.

Two procedures are commonly used for producing useful carbide shapes ready for the sintering furnace. One of these is pill pressing to final shape in a tableting machine. The other is the production of relatively large-size billets for further shaping using hydrostatic impacting. The formed pieces, still in the soft stage, are then placed on a graphite tray and lowered into the sintering furnace. Recent developments in furnace design and operation use high vacuum techniques and electronic induction heating. A vacuum of the order of 50 microns is common in this operation. Sintering temperatures usually approach 3000°F. Close control of the heating and cooling cycle is gained by the use of an elaborate system of electronic equipment. The sintering cycle and the length of stay in the furnace are determined by the analysis of the material and the properties desired in the finished piece.

The fabrication of standard carbide cutting tools on a mass production basis requires modern plant layout and materials handling techniques. The sintered carbide, usually in the form of bar stock, is cut to length and then machined and ground to the desired shape and tolerance.

It will be seen that the methods used in the United States for the production

of tungsten carbide cutting tools are quite detailed, and complete information cannot be presented here. For more complete discussions of the techniques and equipment involved the inquirer is referred to references 1-3 of the appended bibliography. Illustrations of the various types of equipment required are included in these papers.

Literature on the design of specific types of cutting tools (for form grinding, milling, turning, etc.) are widely scattered throughout the various technical publications of the field, and it would require an extremely comprehensive bibliography to present a fairly complete cross-section of this information. For a detailed survey of this field, however, the inquirer is referred to an excellent publication (reference 4), which includes illustrations and design data on all major forms of cutting tools.

References

1. A.D. Stevens
   New Manufacturing Techniques Assure Maximum Carbide Utility
   Tooling and Production 20(11):91-95, February 1955

2. F.J. Lennon, Jr.
   Preforming Complex Carbide Shapes Lower Costs
   Iron Age 171(6):153-155, February 5, 1953

3. Mass Production of Standard Carbide Tools
   Machinery 58(10):182-185, June 1952

4. Metal Cutting Tool Handbook
   New York, Metal Cutting Tool Institute. 3rd edition, 1954. 689pp., $7.50
   Available from: Metal Cutting Tool Institute
   405 Lexington Avenue
   New York 17, New York